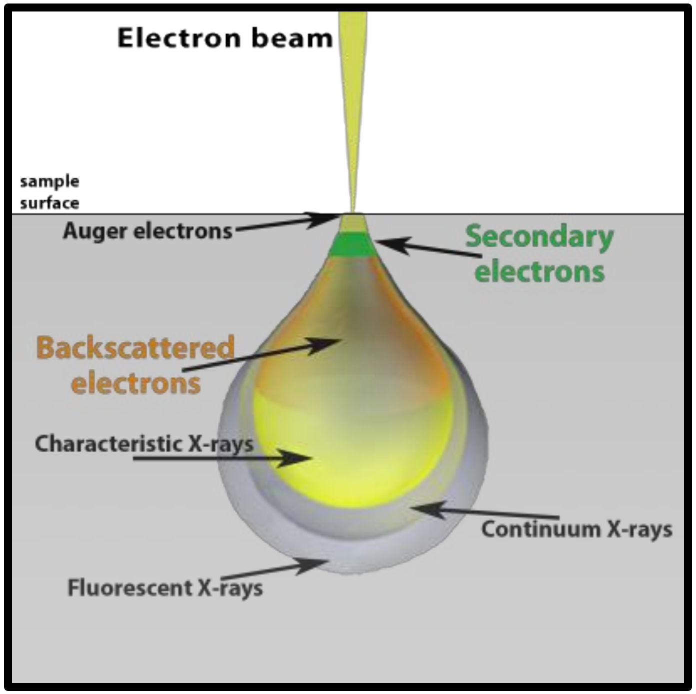
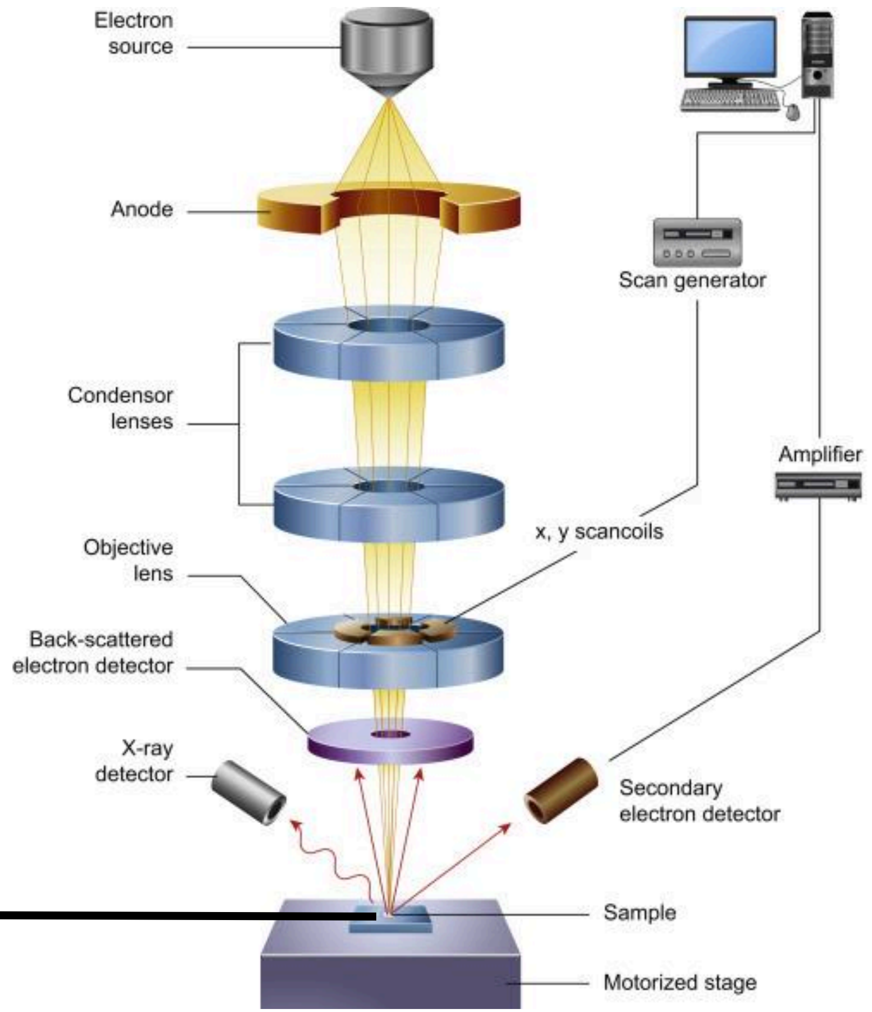
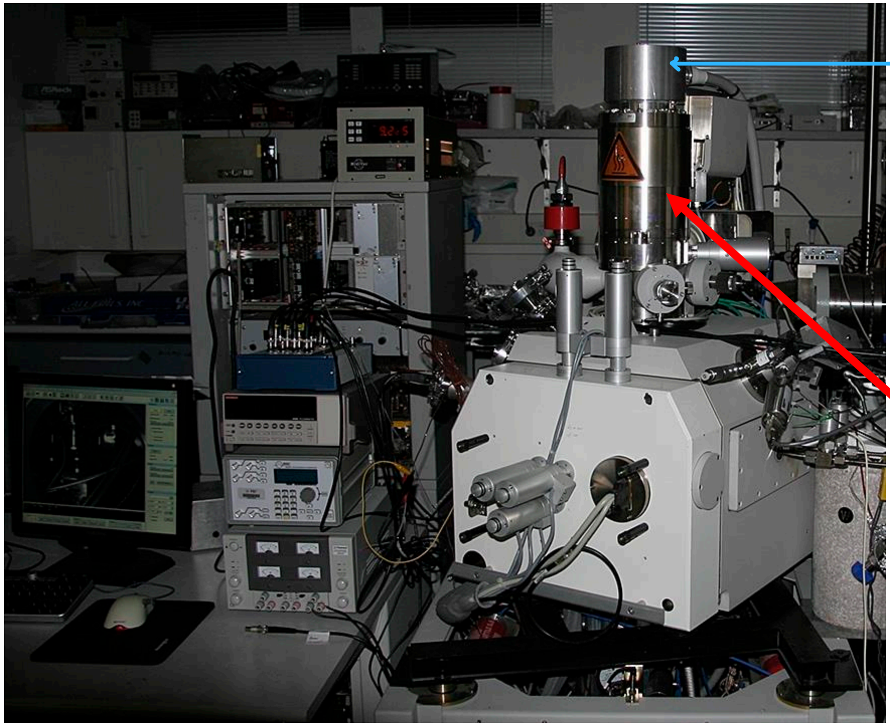
68320 Nanofabrication and Nanocharacterization Techniques

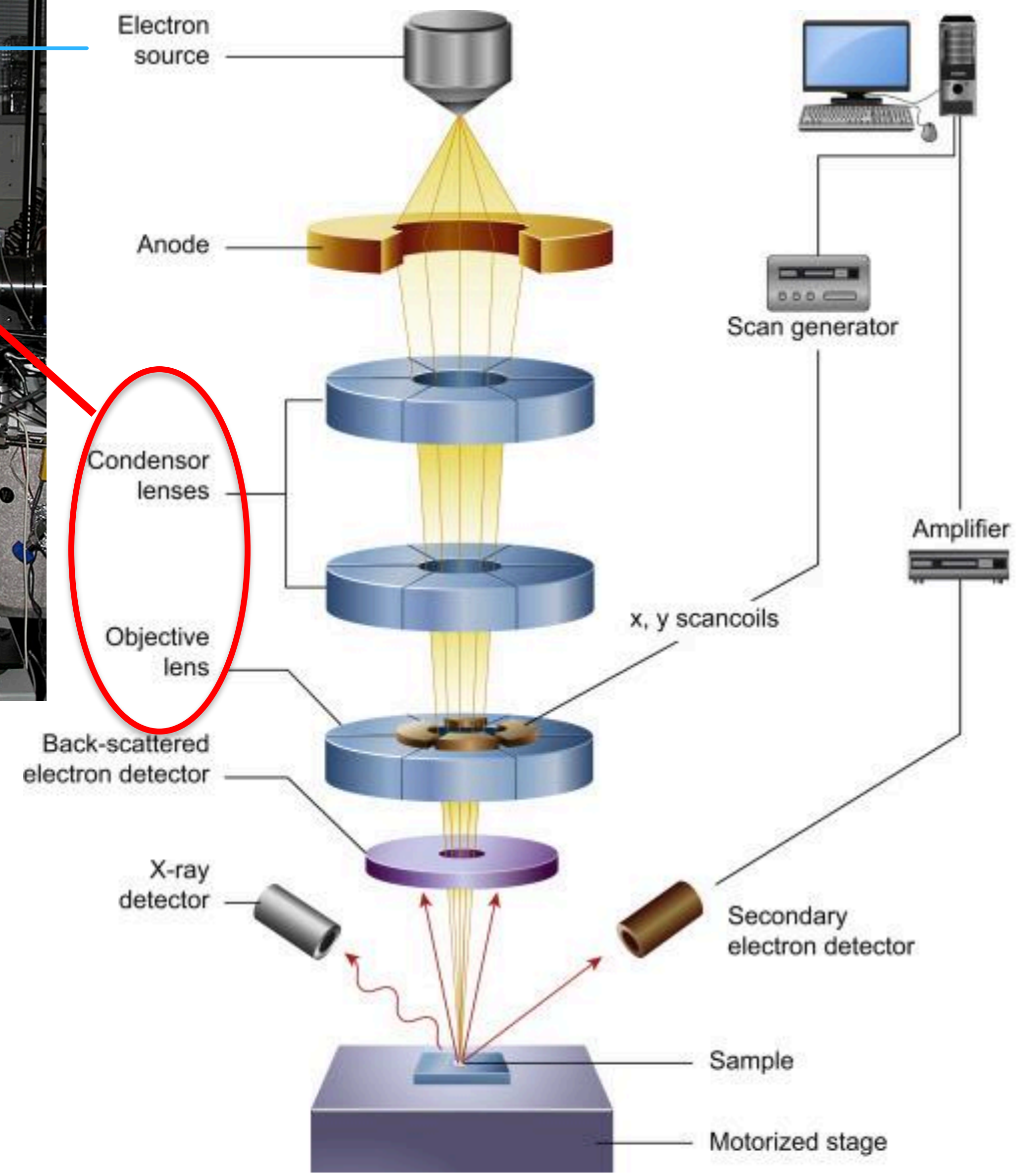
Lecture 2: Electron Optics and Electron-Solid Interactions





Electromagnetic lenses



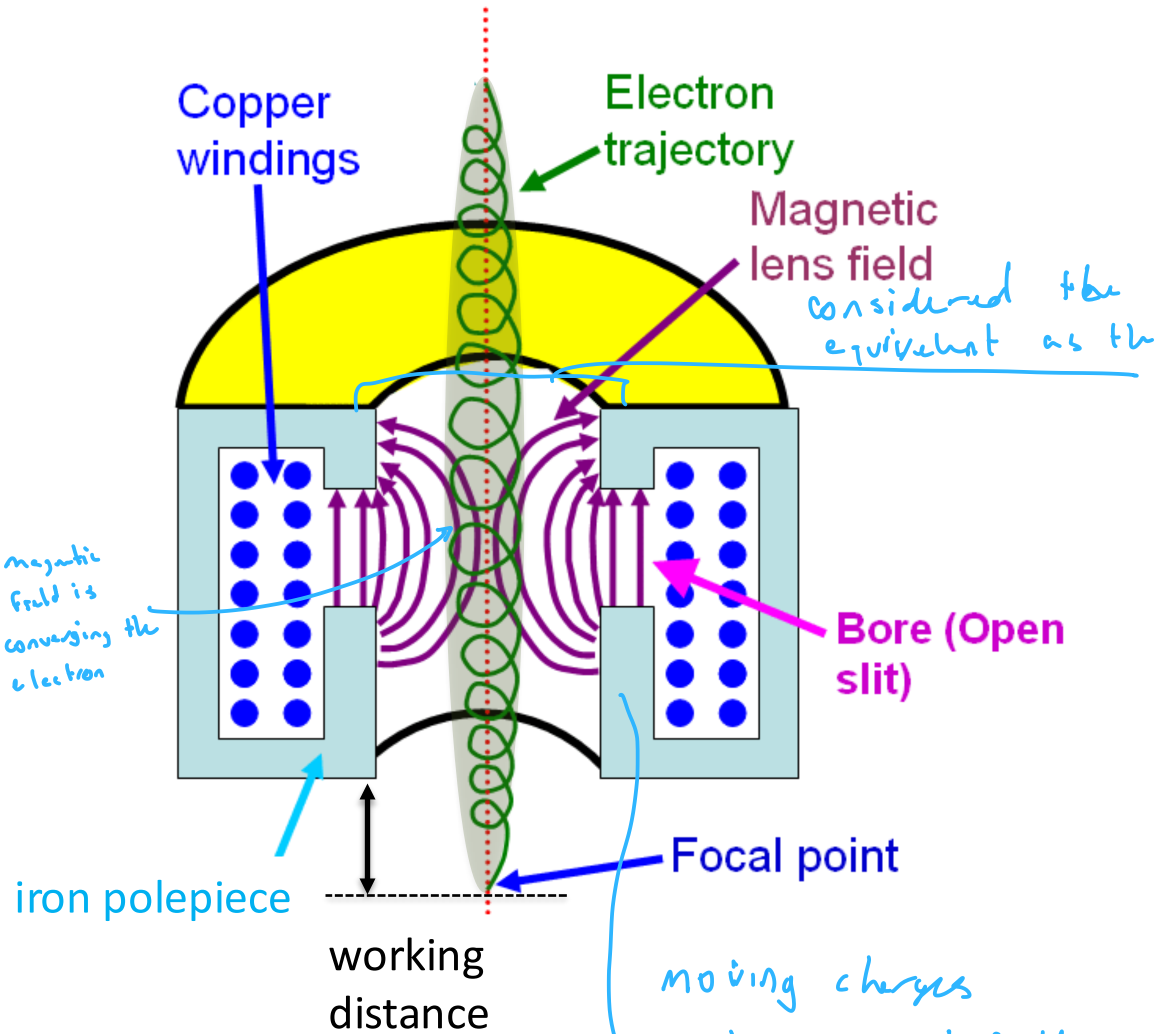


electromagnitism used to focus the electron beam

↳ magnetic field

NOTE: For clarity/simplicity, this column schematic does not show apertures (which are discussed later)

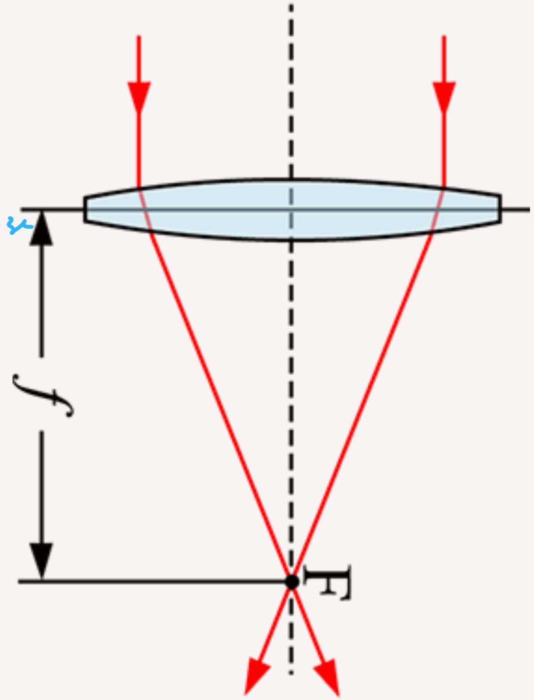
Electromagnetic lens



distance

focal length, $f = K (V/I^2)$

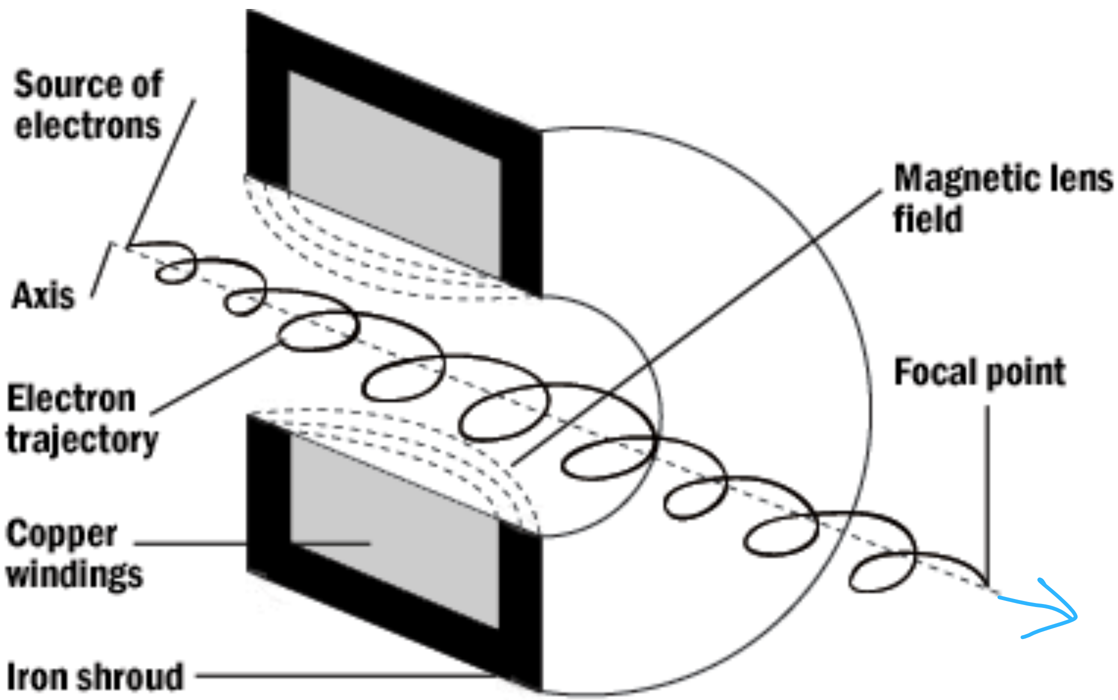
REVISION: light optics



lens: glass, refractive index n
focal length, f: fixed (sample is moved up/down to focus)

K = constant, V = accelerating voltage, I = current through the Cu coil (used to control focus)

Electromagnetic lens: Electron trajectories



used to focus on the sample
↳ changing the area the beam is focusing on

Force exerted on an electron:

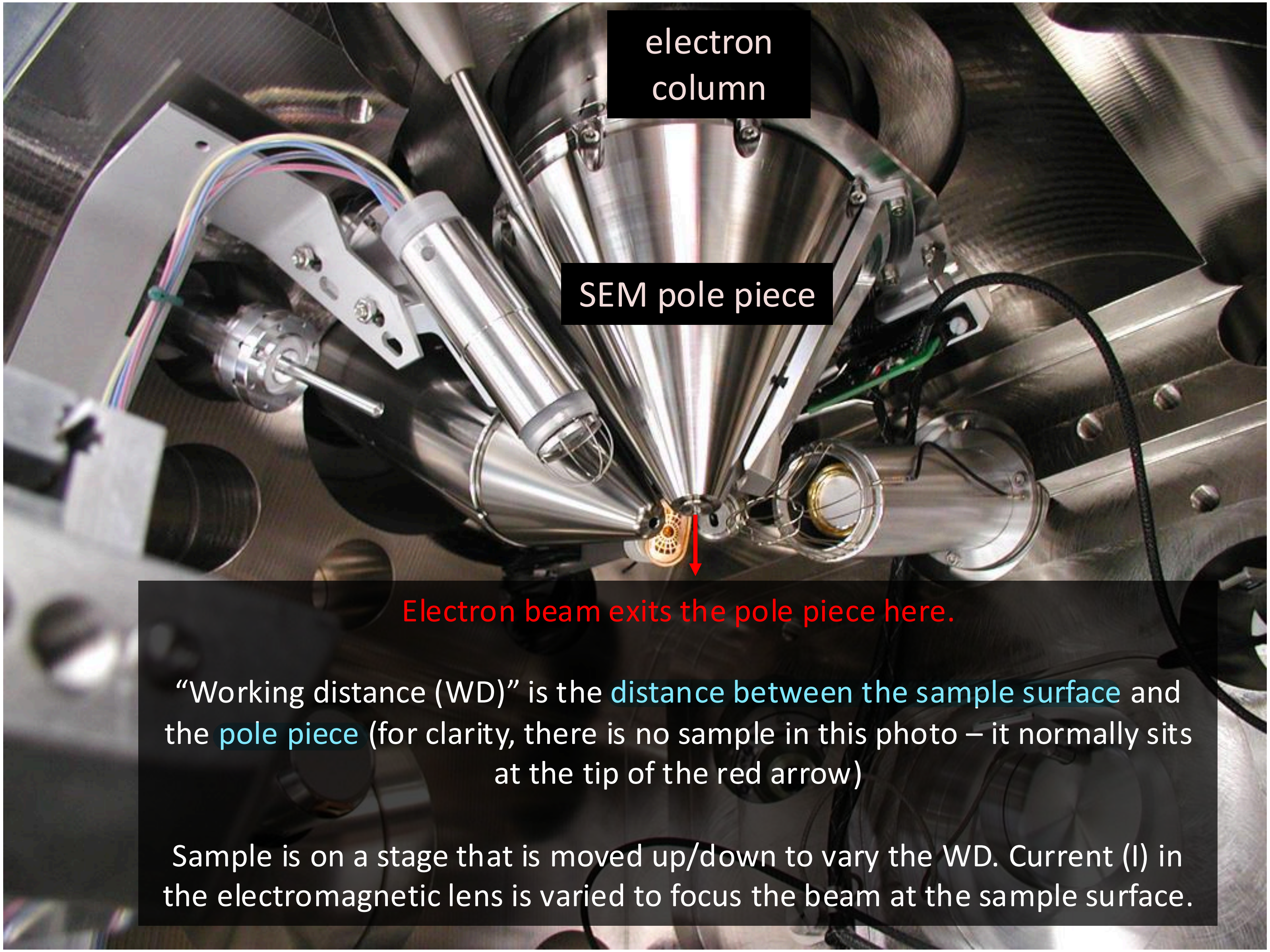
$$\mathbf{F} = q\ \mathbf{v} \times \mathbf{B}$$

go to wiki for revision on basic electrostatics

F = force on a charged particle (electron) N
q = charge (of an electron) C
v = velocity (of electron) m/s
B = magnetic field T

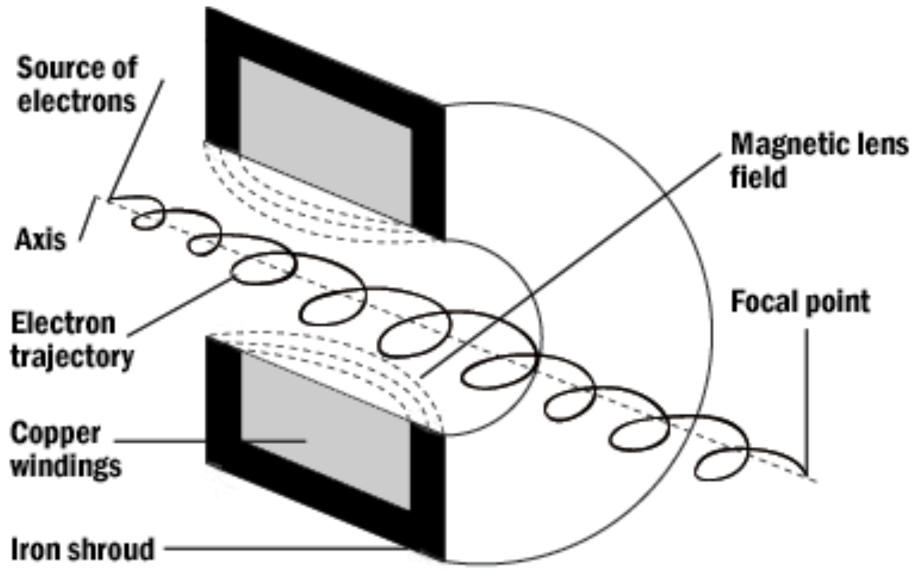
Working distance

Inside view of an SEM sample chamber



Sample class test question

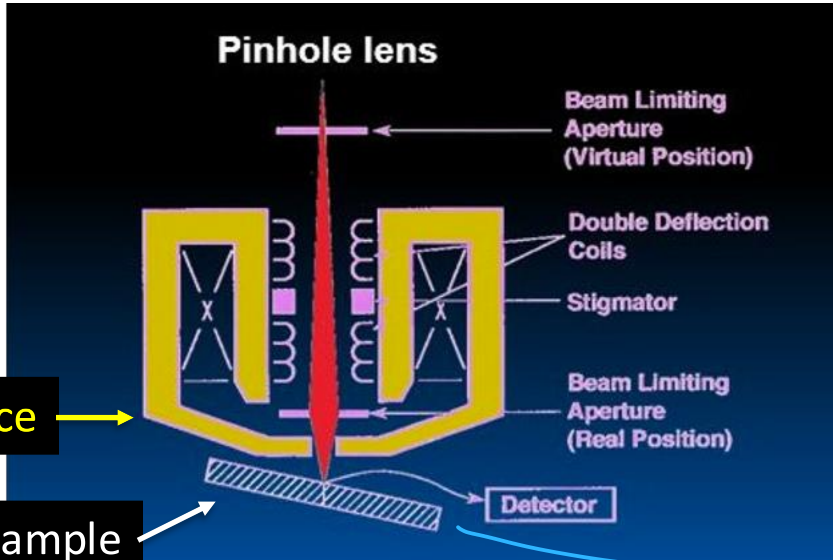
similar to class test question



The image shows the trajectory of an electron passing through an electromagnetic lens. **Which statement is correct?**

~~a)~~ The field increases the energy of the electron.
~~b)~~ The field increases the velocity of the electron.
~~c)~~ The field increases the momentum of the electron.
d) The field does not change the electron energy.
~~e)~~ The field decelerates the electron.

Electromagnetic lens designs



Pole piece

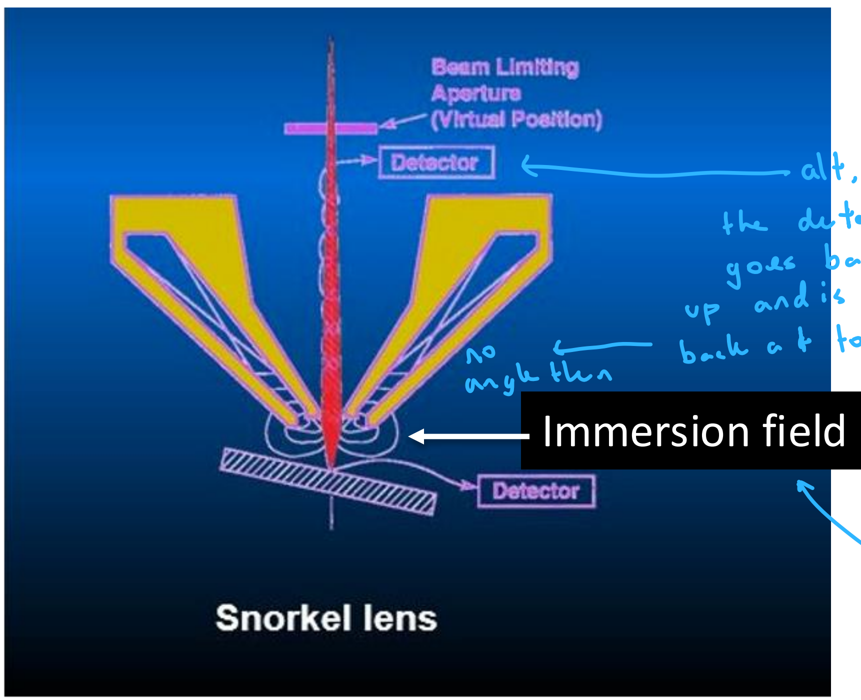
Sample

- cheaper, more basic

Pinhole lens
Magnetic field that focuses the beam is inside the pole piece.

minimise the focal length

angled to point sample to the detector
↳ there is a bias on the detector



alt, the detector goes back up and is collected back at top

no angle then

Immersion lens (a.k.a. snorkel lens)
Magnetic field extends beyond the pole piece, and "immerses" the sample. This reduces the effective working distance, reduces aberrations and improves resolution.

magnetic sample is BAD!

Electromagnetic lens: Aberrations

– convergence angle affects the aberrations

As with all lenses, electromagnetic lenses are subject to lens aberrations which increase the effective diameter of the probe - lowering spatial resolution

3 main limiters

- Spherical aberration: electrons near the edge of the lens need to be focused more strongly than those near the centre
- Chromatic: due to thermal contributions, electrons emitted from the source are not all at the same energy (wavelength) so there is a spread of focus (emitter → const. temp minimise the abrrations)
- Diffraction: electrons being wave-like, small apertures can broaden the probe via diffraction effects (wavelength of the electron, like the double slit experiment ↳ apature causes the wavelength to diverge around (in picometres))
- Astigmatism: lack of radial symmetry of lens - non-circular probe

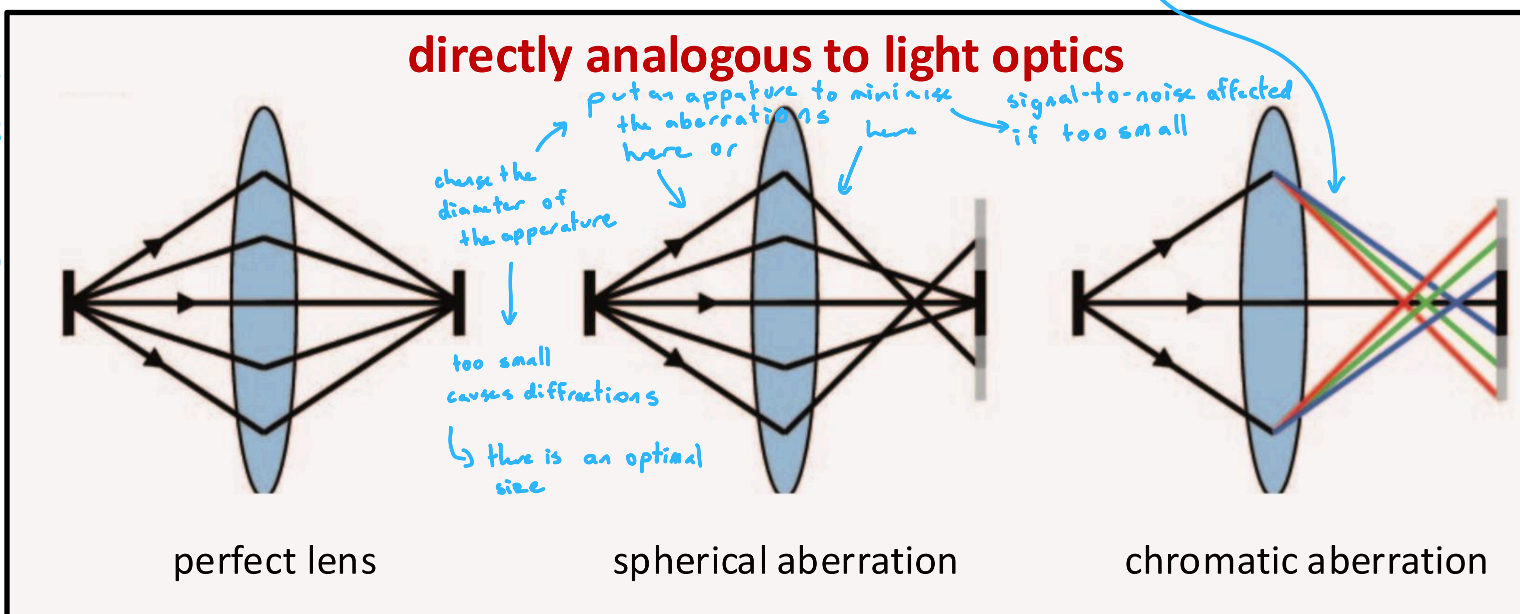
reduce the energy produced in the tip

atoms oscillate from temp causing collision of electron, changing the velocity

when beam is not the right shape → from circle to elipse shape

diff energies/ wavelengths

free electrons hit the insulators and create electro fields that will affect the electron beam



put an appature to minimise the aberrations here or here

signal-to-noise affected if too small

change the diameter of the apperature

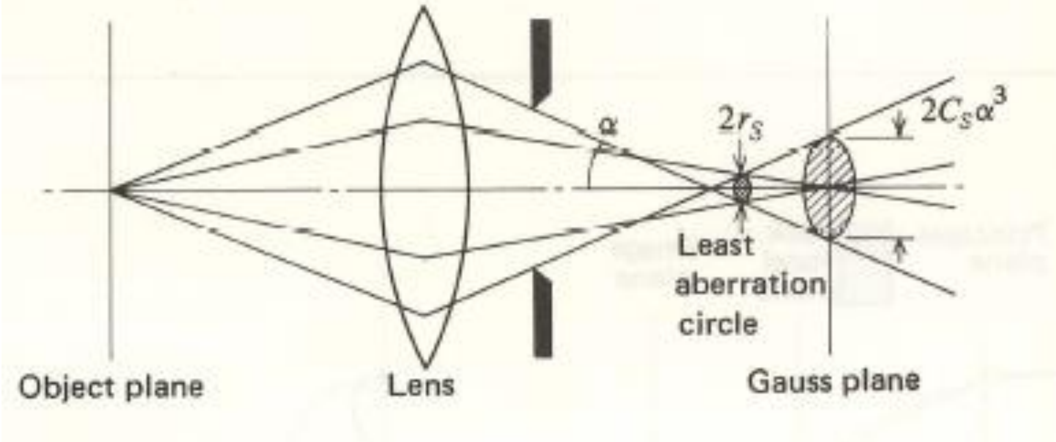
too small causes diffractions

↳ there is an optimal size

Increases the speed changes the focal length, but decrease the working distance

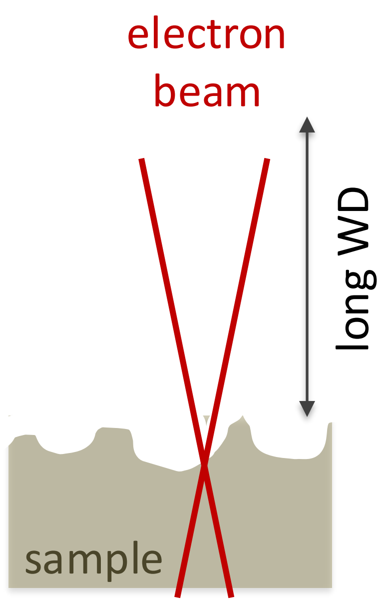
Spherical aberration, C_s

aperture: rejects (blocks) high angle electrons

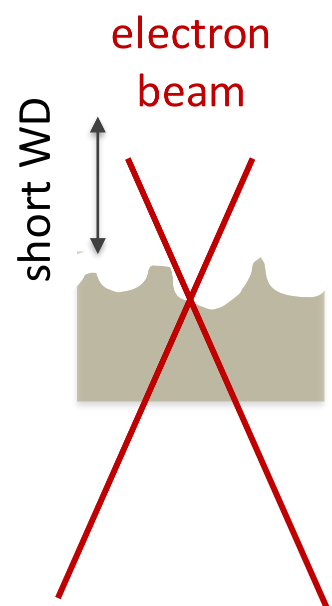


C_s reduced by <u>decreasing</u> aperture diameter, which also limits the electron beam current.

An SEM operator can use the aperture size to control resolution and electron beam current.



superior depth of field

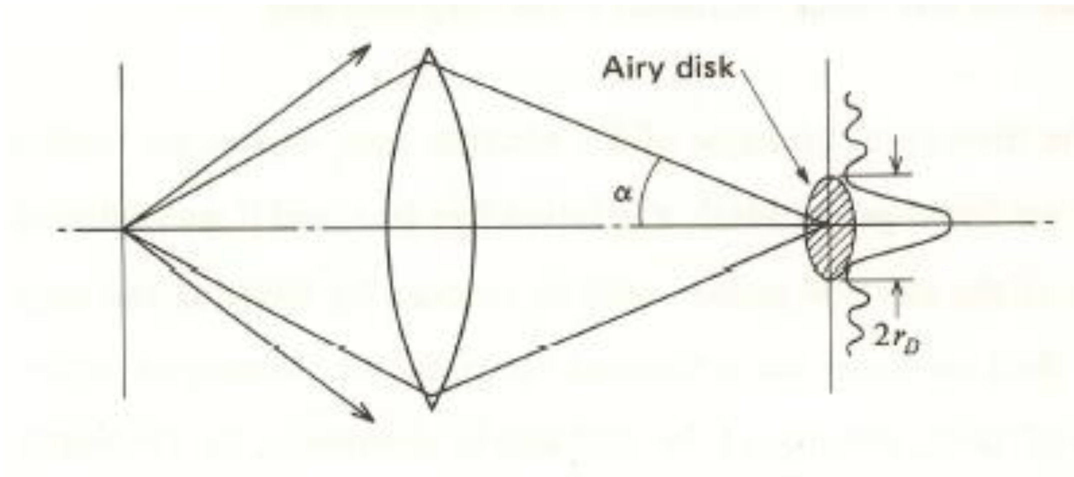


superior resolution

C_s is also reduced by increasing the magnetic field of the electromagnetic lens (ie: by increasing the beam convergence angle and operating the SEM at short "working distance", WD).

An SEM operator can use the working distance to trade off resolution for depth of field.

Diffraction aberration

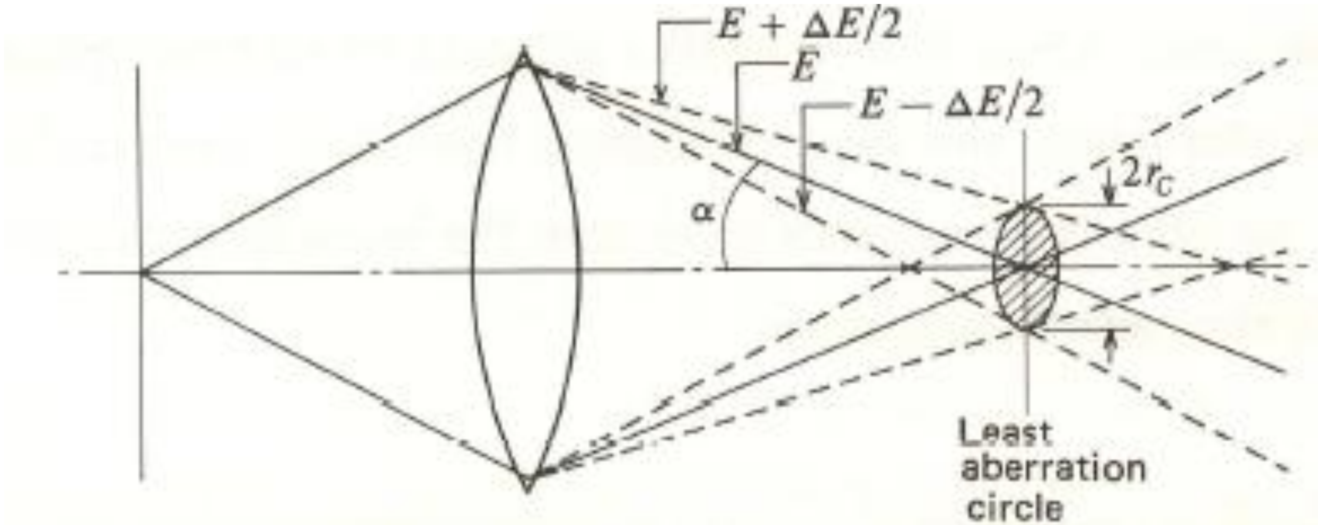


As the aperture diameter decreases, electrons will diffract due to their wave-like properties

r_D reduced by <u>increasing</u> aperture diameter (i.e., if the aperture is too small, resolution is limited by diffraction)

there is an optimum aperture diameter (typically ~ 10 μm in an SEM) that yields best resolution (ie: smallest electron beam diameter) by optimising both diffraction & spherical aberrations

Chromatic aberration, C_c

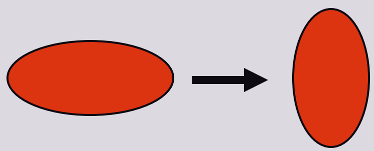


- Electrons have a <u>range of energies</u> due to the temperature of the electron source (and also fluctuations in the accelerating voltage caused by noise in the power supply)

- Examples: cheap W electron gun: 1 eV to 3 eV; cold field emission gun: ~ 0.3 eV

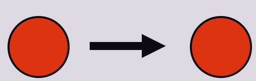
Astigmatism aberration

Astigmatism Present

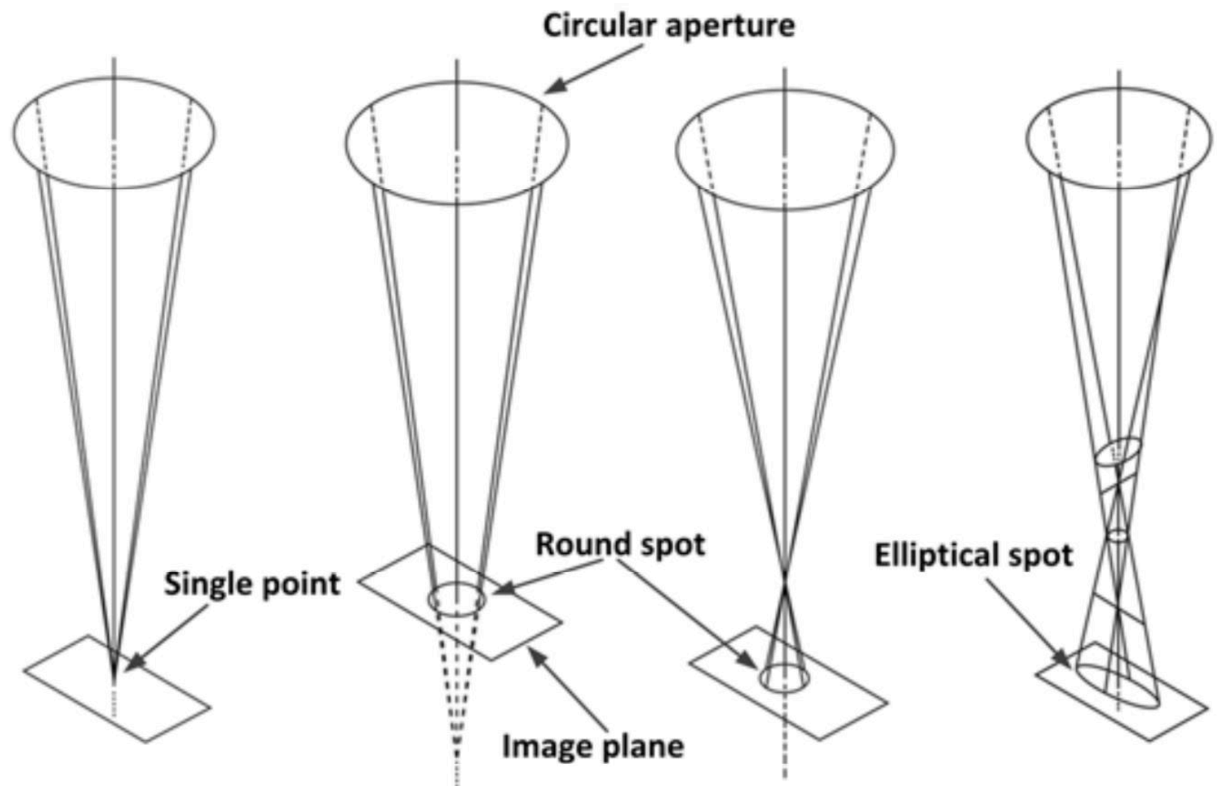


rocking focus

Astigmatism Corrected



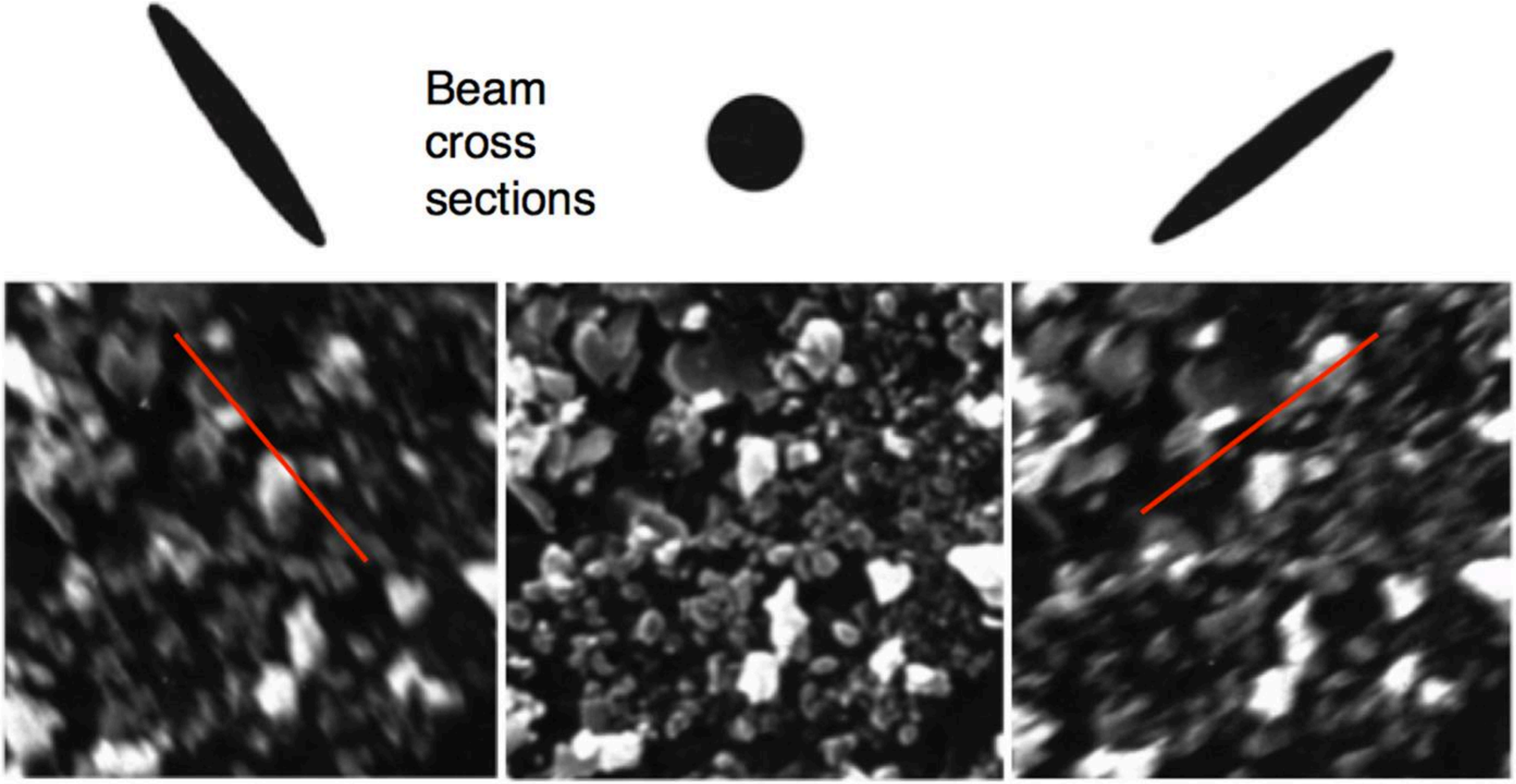
rocking focus



(a) In-focus (b) Underfocus and Overfocus (c) Astigmatism

electrodes around the beam in x or y
to change the shape of the beam

Astigmatism aberration

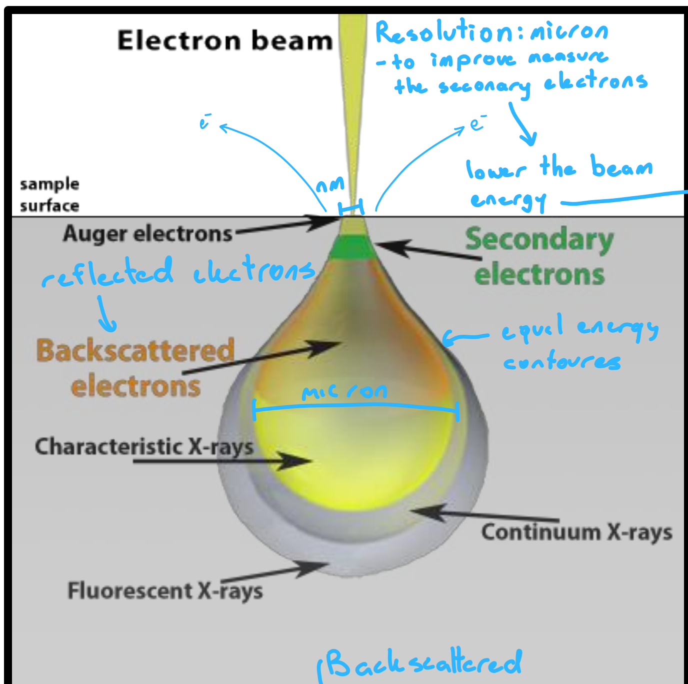


- caused by lack of radial symmetry in the lens
- produces an elliptical electron beam spot rather than circular
- identified by 90^{O} streaking as image rocked through focus
- corrected by stigmators that re-shape beam to a circular spot – either r, θ or x, y control to adjust orientation and strength of the correction field

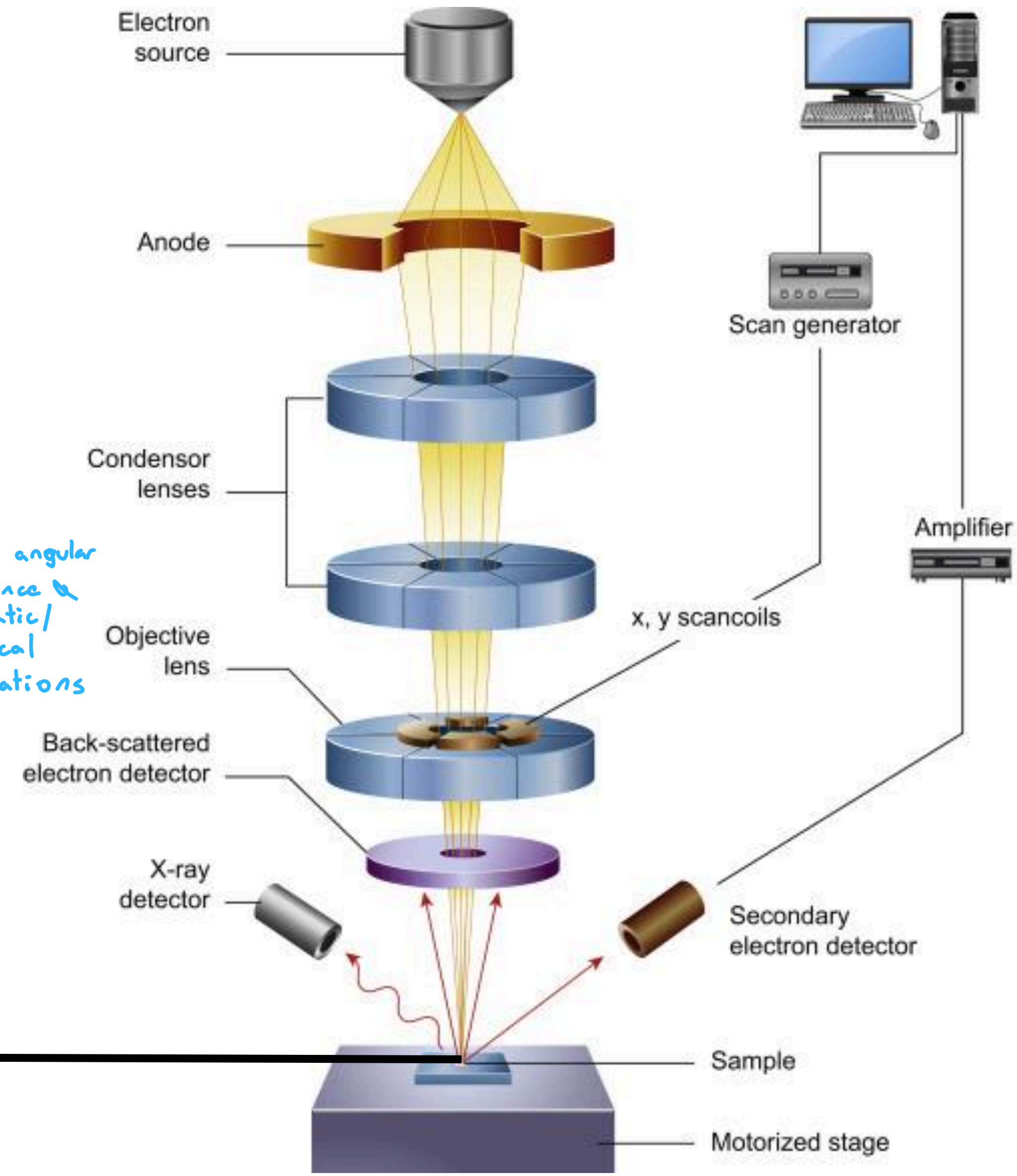
Electron imaging & electron-solid interactions

electron-solid interactions affect electron image:

- resolution &
- information content



however angular divergence & chromatic/ spherical aberrations



· density
· crystalline structure

The atomic mass

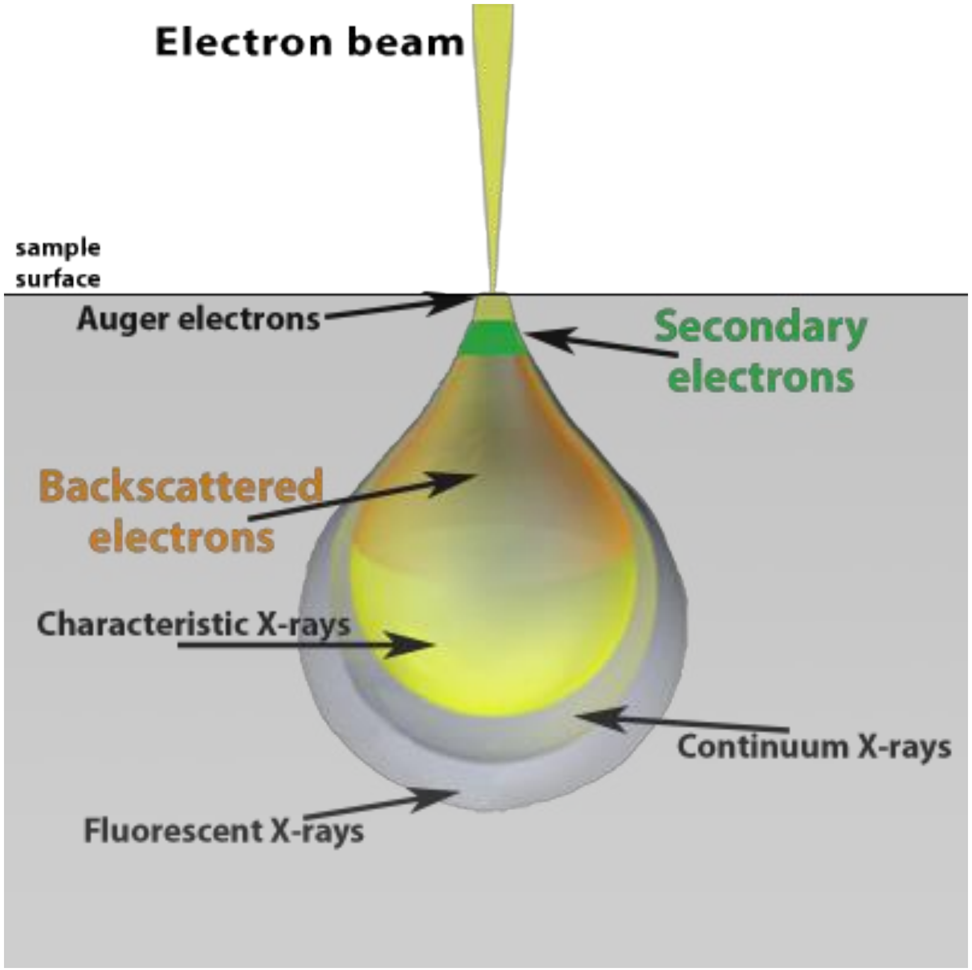
↳ electron colliding with atom of higher atomic mass is deflected

Interaction of the electron beam/probe and the sample

Each emissive signal originates from a different depth within the specimen

this is covered in this week's tutorial

- the total energy of the incident electrons must be dissipated (in a bulk specimen) and energy must be conserved
- energy loss processes generate emission signals that can used to form an image as the electron beam scans across the specimen
- each of these signals carries different information about the specimen



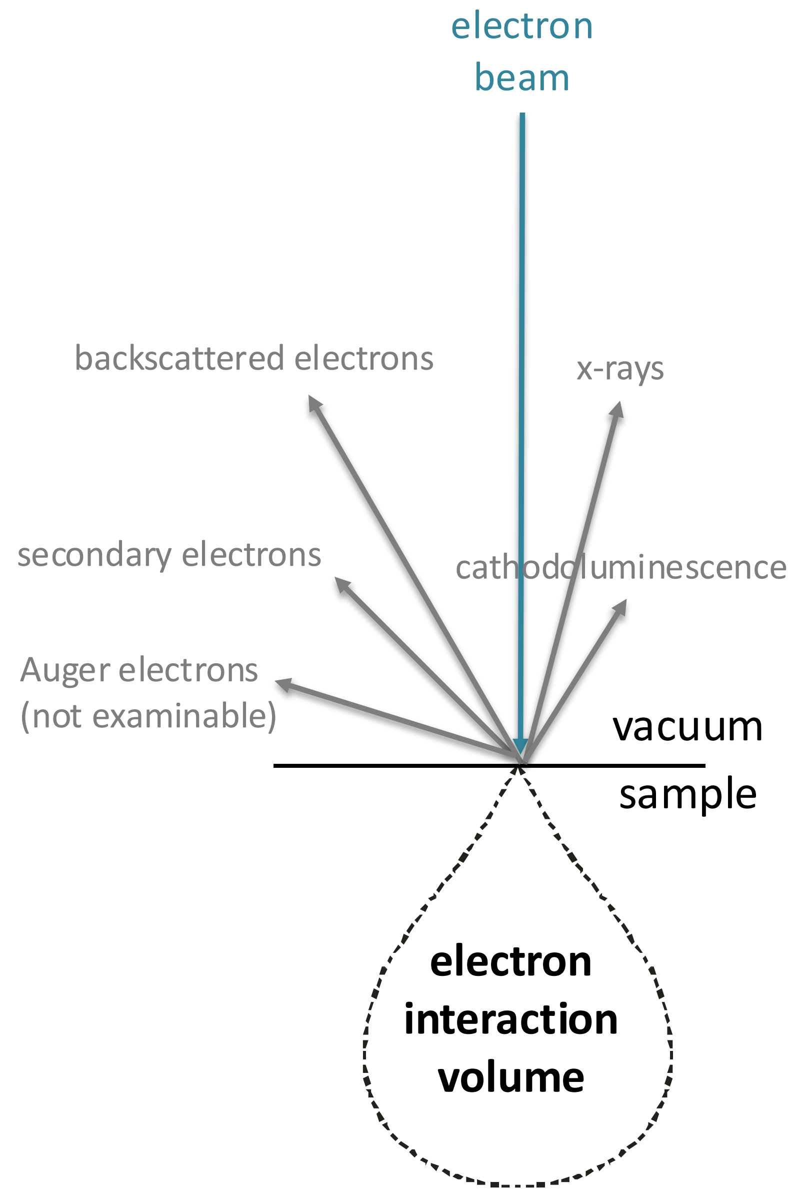
Main energy loss processes in solids

this is covered in this week's tutorial

- ***specimen heating*** *- generation of phonons* - highest cross-section

- **breaking of chemical bonds that bind atoms** (1 -10 eV): produces secondary electrons and cathodoluminescence

- **ionization of core shell electrons** (keV): produces X-rays



Electron Scattering Mechanisms

Elastic Scattering (due to interactions with nuclei)

Change in electron trajectory with virtually no loss in energy

Causes electron spreading in specimen & backscattered electrons

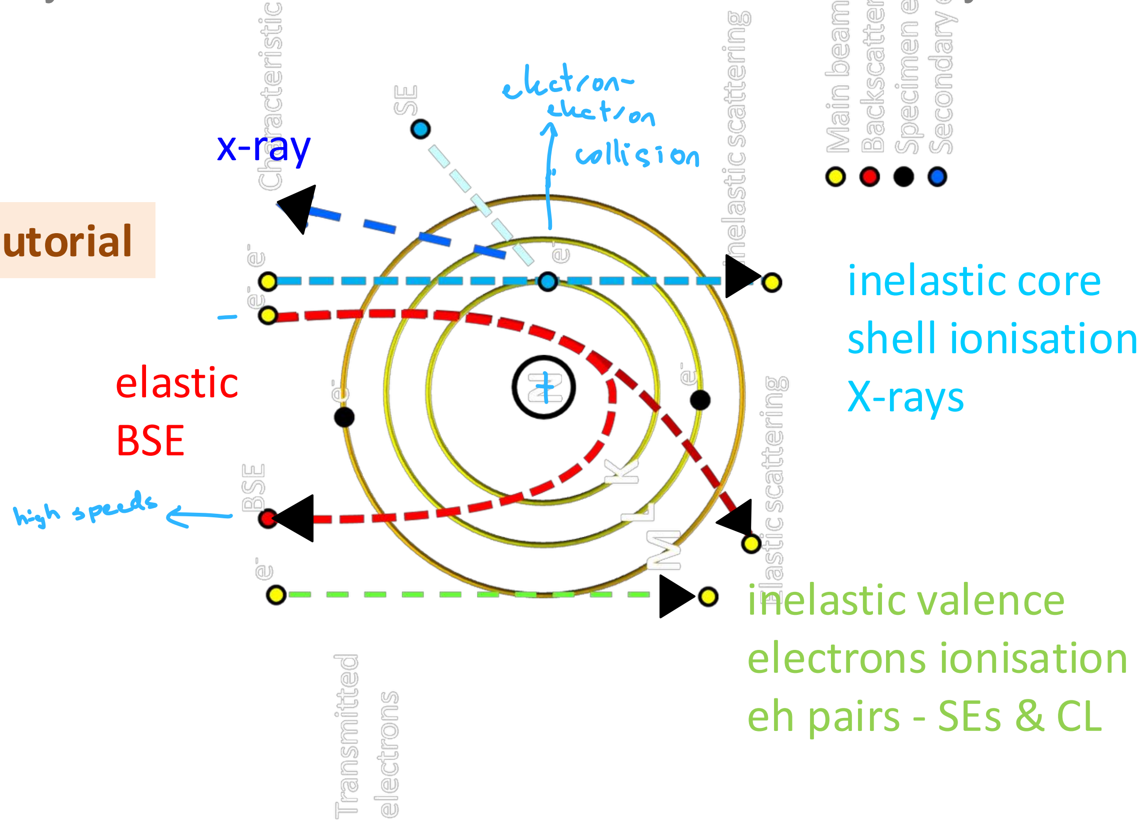
Inelastic Scattering (due to electron-electron scattering)

Loss of energy with no change in trajectory

Generation of heat, secondary electrons, cathodoluminescence and X-rays

this is covered in this week's tutorial

Illustrates the types of collisions →



Primary ("beam") electron range (R_e)

this is covered in this week's tutorial

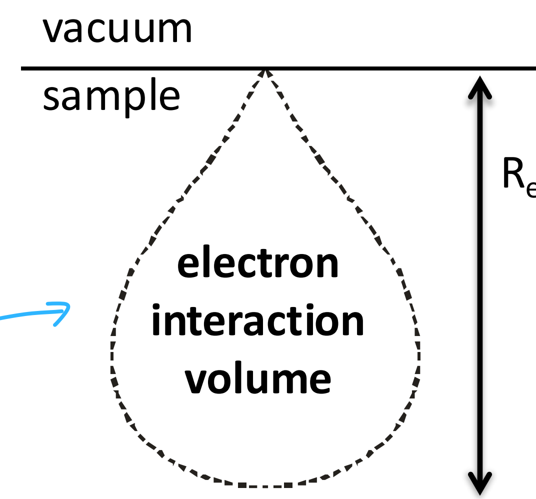
atomic weight [g/mol]

$$R_e(nm) = \frac{27.6 A E_o^{5/3}}{Z^{8/9}\, r}$$

energy of beam [keV]

atomic number

density g/cm^3



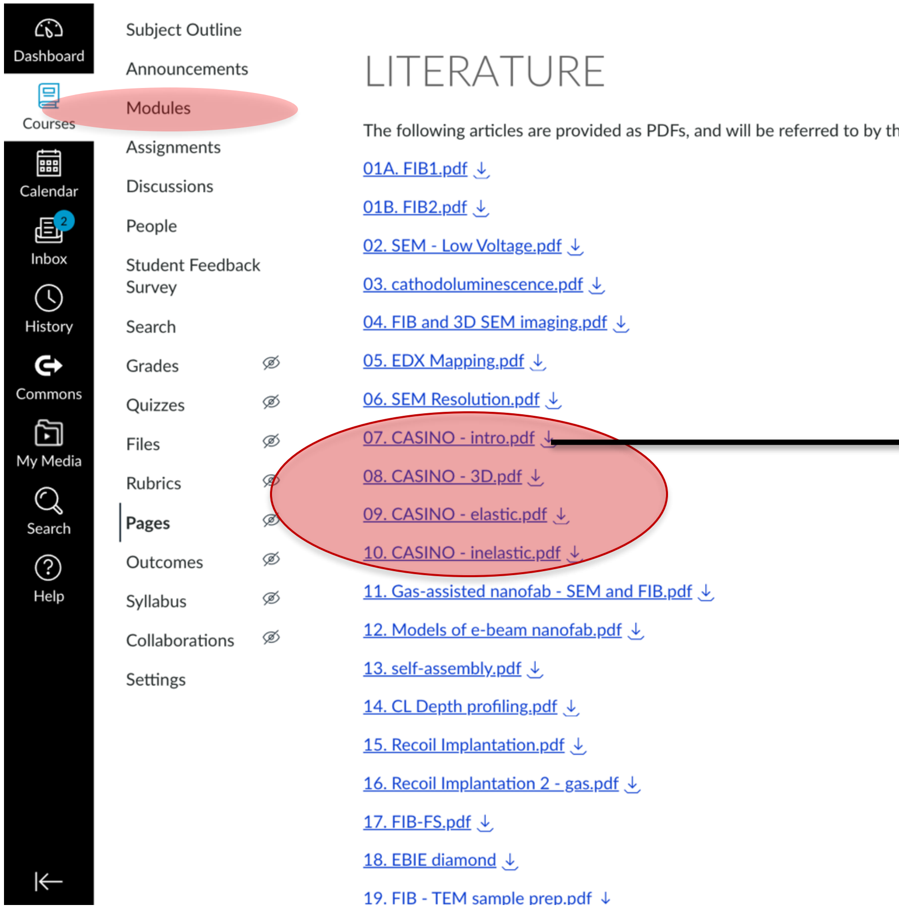
Original paper on this: Kanaya & Okayama, JPD 5, 43(1972)

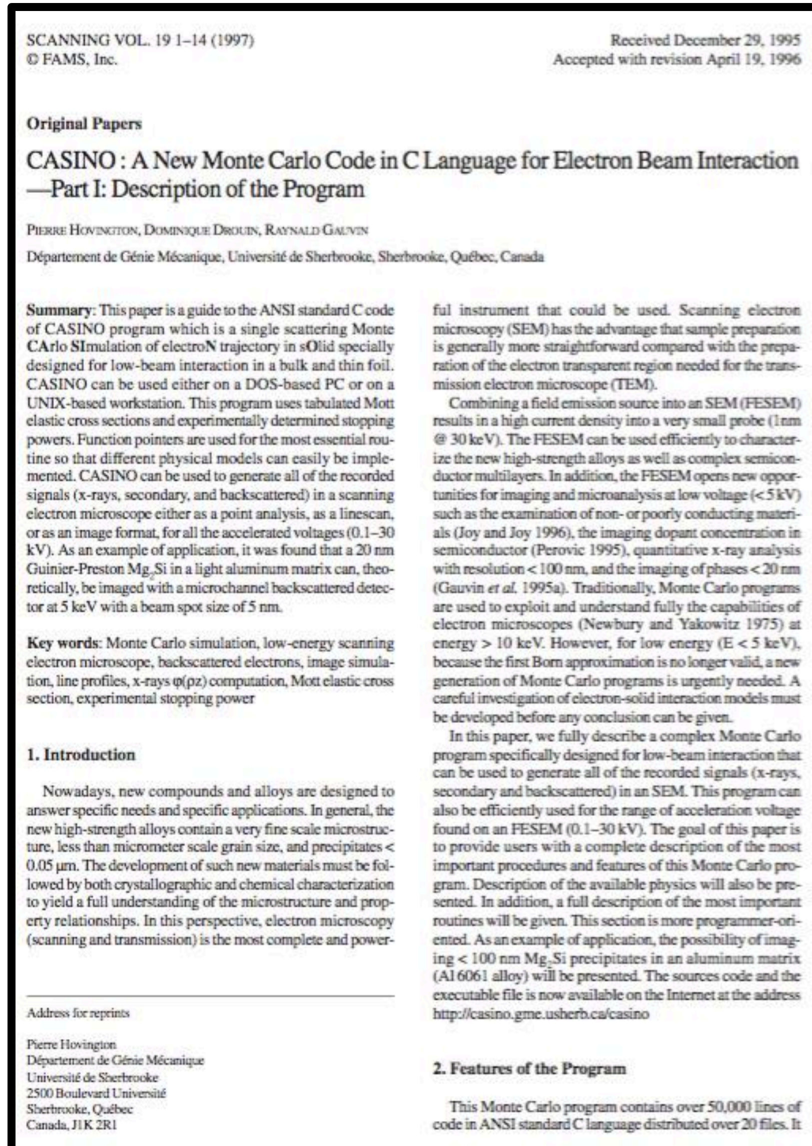
Electron emission:

Backscattered Electron (BSE) imaging in Scanning Electron Microscopy (SEM)

Papers on Canvas (useful for today’s tute)

\Canvas\Modules\Tutorials…\Tute 2…\07. CASINO - intro.pdf





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Original Papers

CASINO : A New Monte Carlo Code in C Language for Electron Beam Interaction —Part I: Description of the Program

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Summary: This paper is a guide to the ANSI standard C code of CASINO program which is a single scattering Monte CArlo SImulation of electroN trajectory in sOlid specially designed for low-beam interaction in a bulk and thin foil. CASINO can be used either on a DOS-based PC or on a UNIX-based workstation. This program uses tabulated Mott elastic cross sections and experimentally determined stopping powers. Function pointers are used for the most essential routine so that different physical models can easily be implemented. CASINO can be used to generate all of the recorded signals (x-rays, secondary, and backscattered) in a scanning electron microscope either as a point analysis, as a linescan, or as an image format, for all the accelerated voltages (0.1–30 kV). As an example of application, it was found that a 20 nm Guinier-Preston Mg_2Si in a light aluminum matrix can, theoretically, be imaged with a microchannel backscattered detector at 5 keV with a beam spot size of 5 nm.

Key words: Monte Carlo simulation, low-energy scanning electron microscope, backscattered electrons, image simulation, line profiles, x-rays $\varphi(\rho z)$ computation, Mott elastic cross section, experimental stopping power

1. Introduction

Nowadays, new compounds and alloys are designed to answer specific needs and specific applications. In general, the new high-strength alloys contain a very fine scale microstructure, less than micrometer scale grain size, and precipitates < 0.05 µm. The development of such new materials must be followed by both crystallographic and chemical characterization to yield a full understanding of the microstructure and property relationships. In this perspective, electron microscopy (scanning and transmission) is the most complete and powerful instrument that could be used. Scanning electron microscopy (SEM) has the advantage that sample preparation is generally more straightforward compared with the preparation of the electron transparent region needed for the transmission electron microscope (TEM).

Combining a field emission source into an SEM (FESEM) results in a high current density into a very small probe (1 nm @ 30 keV). The FESEM can be used efficiently to characterize the new high-strength alloys as well as complex semiconductor multilayers. In addition, the FESEM opens new opportunities for imaging and microanalysis at low voltage (< 5 kV) such as the examination of non- or poorly conducting materials (Joy and Joy 1996), the imaging dopant concentration in semiconductor (Perovic 1995), quantitative x-ray analysis with resolution < 100 nm, and the imaging of phases < 20 nm (Gauvin *et al.* 1995a). Traditionally, Monte Carlo programs are used to exploit and understand fully the capabilities of electron microscopes (Newbury and Yakowitz 1975) at energy > 10 keV. However, for low energy (E < 5 keV), because the first Born approximation is no longer valid, a new generation of Monte Carlo programs is urgently needed. A careful investigation of electron-solid interaction models must be developed before any conclusion can be given.

In this paper, we fully describe a complex Monte Carlo program specifically designed for low-beam interaction that can be used to generate all of the recorded signals (x-rays, secondary and backscattered) in an SEM. This program can also be efficiently used for the range of acceleration voltage found on an FESEM (0.1–30 kV). The goal of this paper is to provide users with a complete description of the most important procedures and features of this Monte Carlo program. Description of the available physics will also be presented. In addition, a full description of the most important routines will be given. This section is more programmer-oriented. As an example of application, the possibility of imaging < 100 nm Mg_2Si precipitates in an aluminum matrix (Al 6061 alloy) will be presented. The sources code and the executable file is now available on the Internet at the address http://casino.gme.usherb.ca/casino

2. Features of the Program

This Monte Carlo program contains over 50,000 lines of code in ANSI standard C language distributed over 20 files. It

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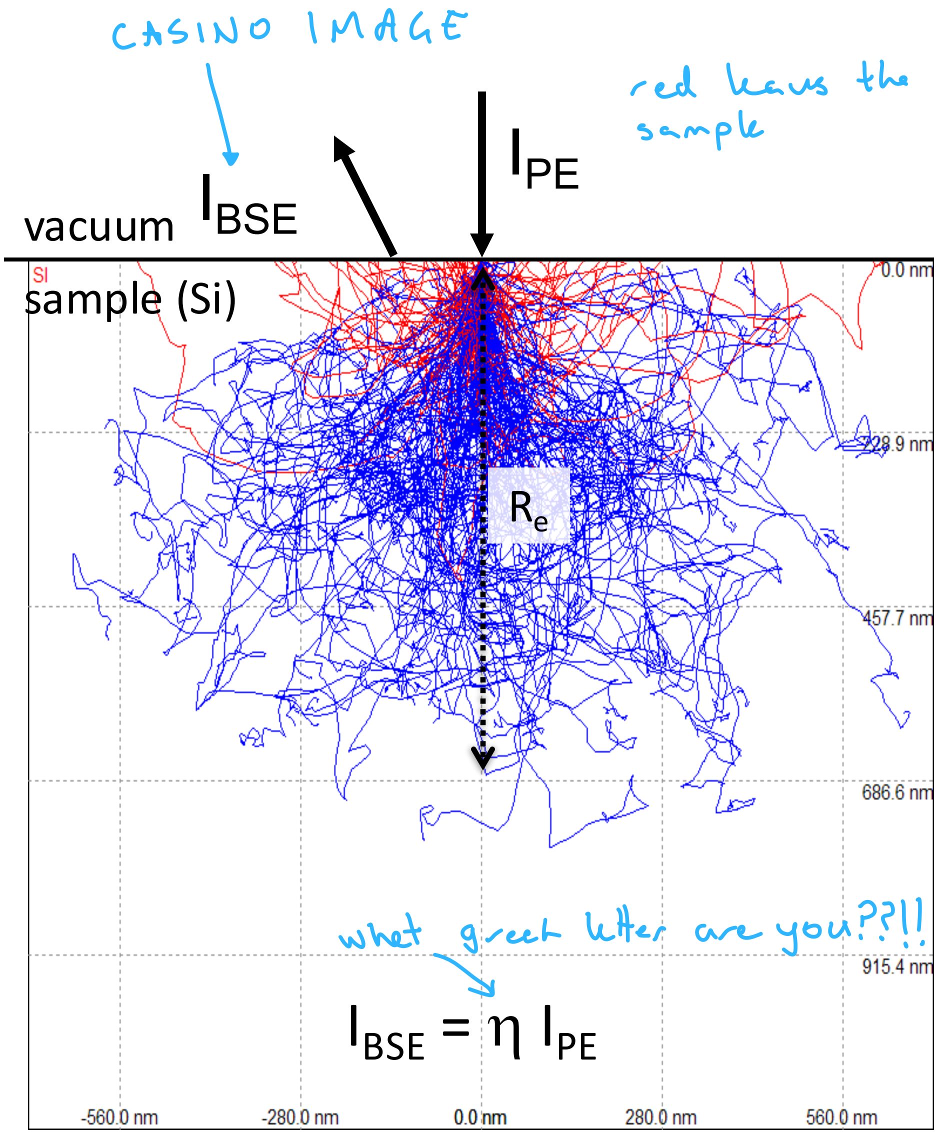
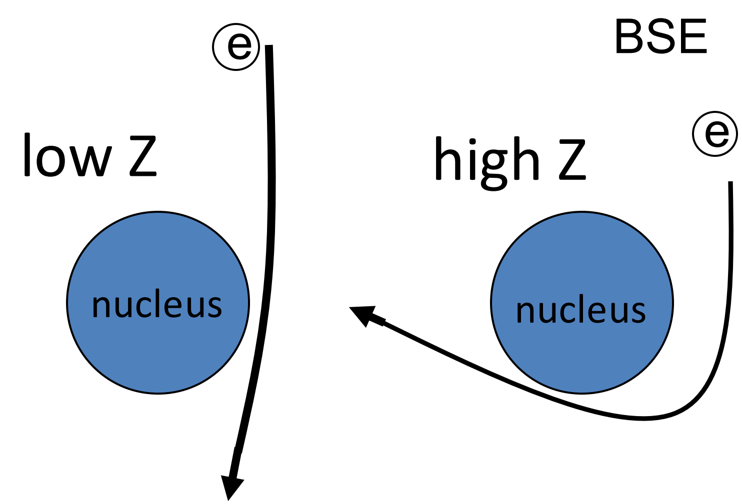
Backscattered Electrons: compositional imaging

this is covered in this week's tutorial



η is the BSE yield

Red trajectories are primary electrons that have been back-scattered out of the specimen.



larger Z increases the electron scattering efficiency, so η increases with mean Z

BSE compositional Imaging

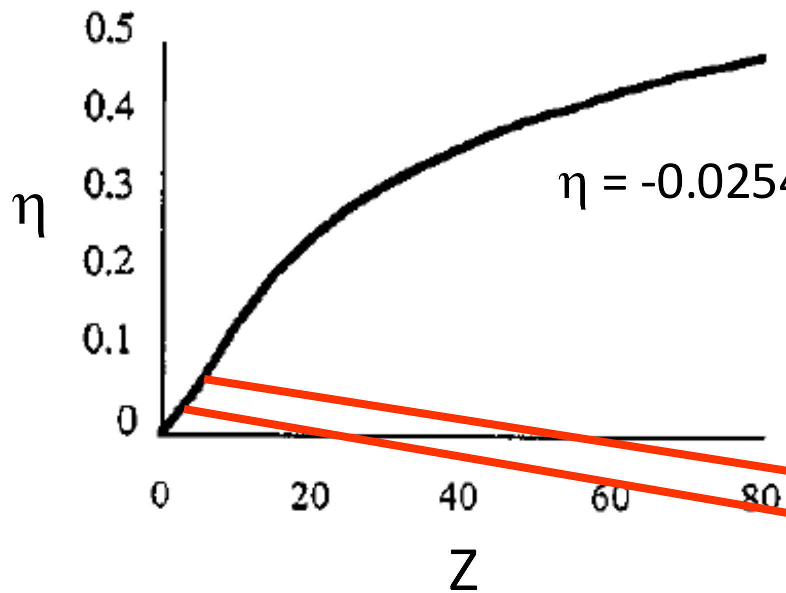
this is covered in this week's tutorial

$I_{BSE} = \eta \, I_{PE}$

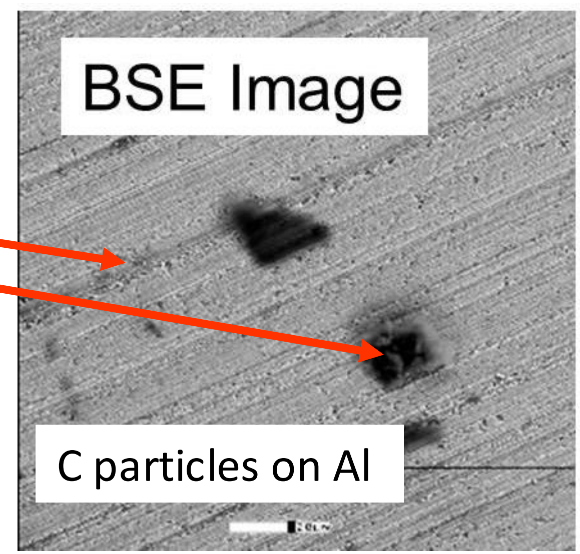
$\eta = I_{BSE} / I_{PE}$

$\mathbf{I_{PE}}$ = primary electron beam current
$\mathbf{I_{BSE}}$ = backscattered electron (BSE) current
$\boldsymbol{\eta}$ = BSE coefficient (a.k.a. "BSE yield")

η increases with increasing atomic number (Z)

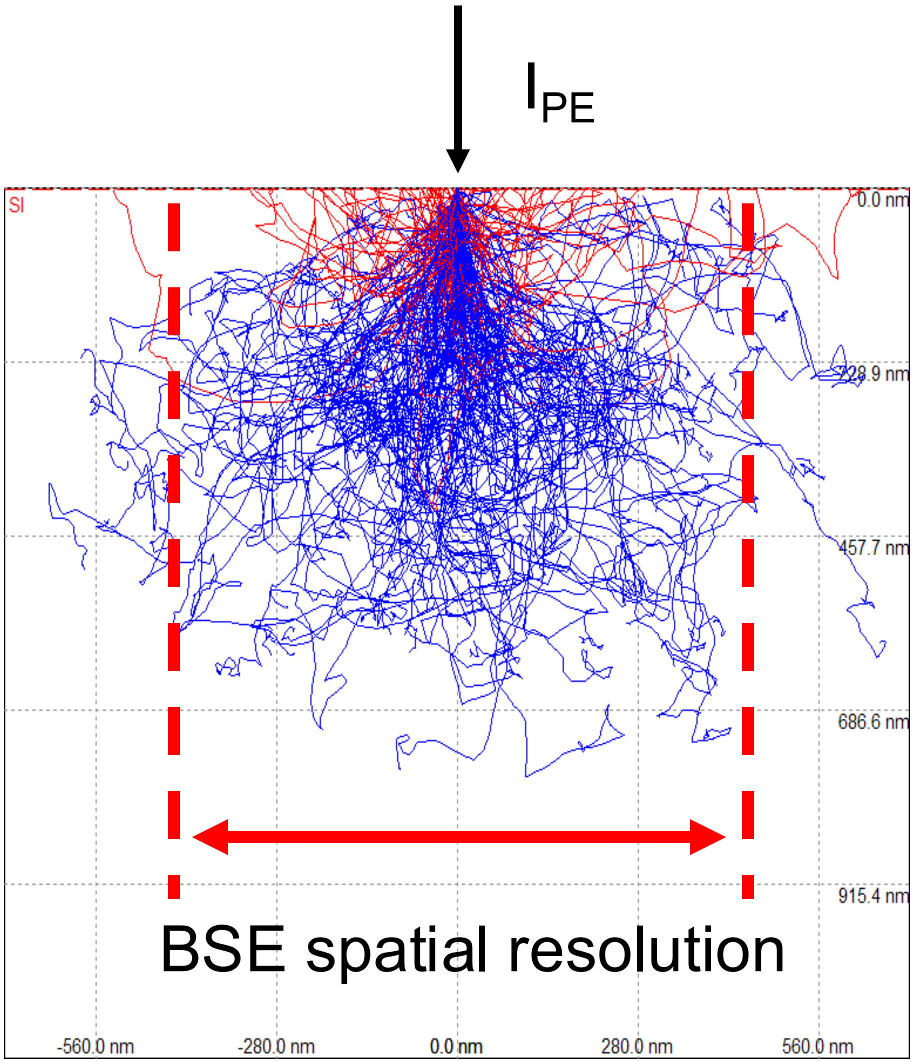


$$\eta = -0.0254 + 0.016\,Z - 1.86\text{x}10\text{-}4\,Z^2 + 8.3\text{x}10\text{-}7\,Z^3$$



Dark carbon particles in BSE image have lower average Z than Al substrate

BSE Compositional Imaging: Resolution



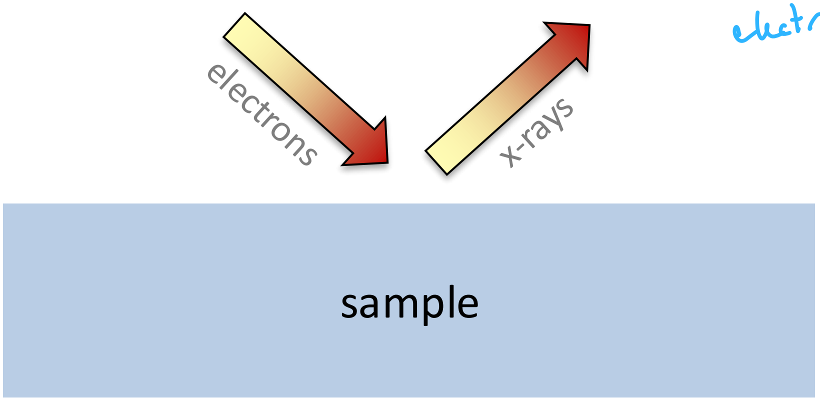
BSE image resolution is approximated by the lateral dimensions of the interaction volume rather than the electron probe diameter

this is covered in this week's tutorial

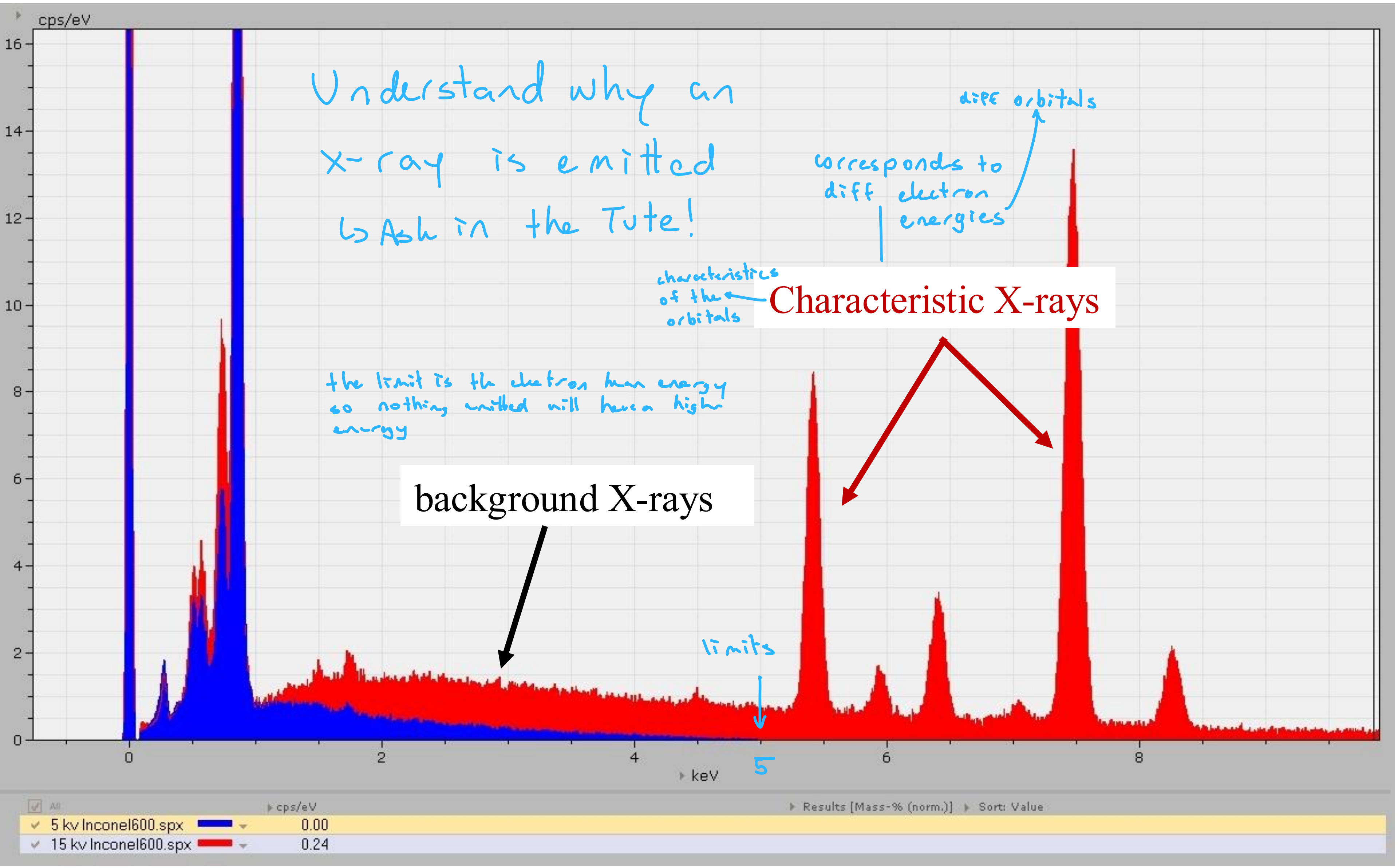
Relevant to Week 3 & 4 (depending on which group) content!

X-ray analysis in a scanning electron microscope: Energy-dispersive x-ray spectroscopy (EDX)

reflected electrons are called backscattered electrons



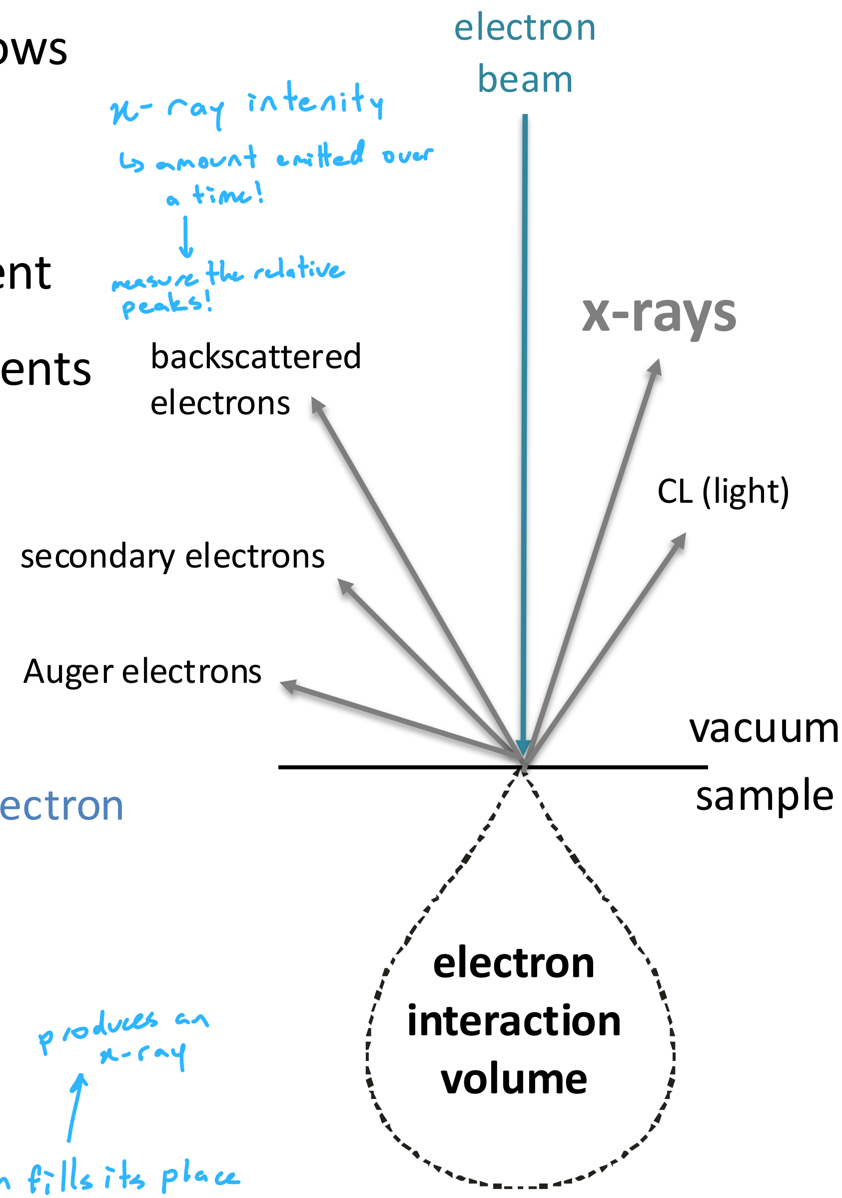
EDX of steel: 5keV (Blue) & 15 keV (red)



Characteristic x-rays: Microanalysis

- electron beam induced x-ray emission allows selective area elemental analysis down to nanoscale volumes (by measuring the electrons:
  - spectra → corrponds to the peaks
  - Qualitative - What elements are present
    ↳ but does not tell how much of it?
  - Quantitative - Concentrations of elements

x- ray intenity
↳ amount emitted over a time!
↓
measure the relative peaks!



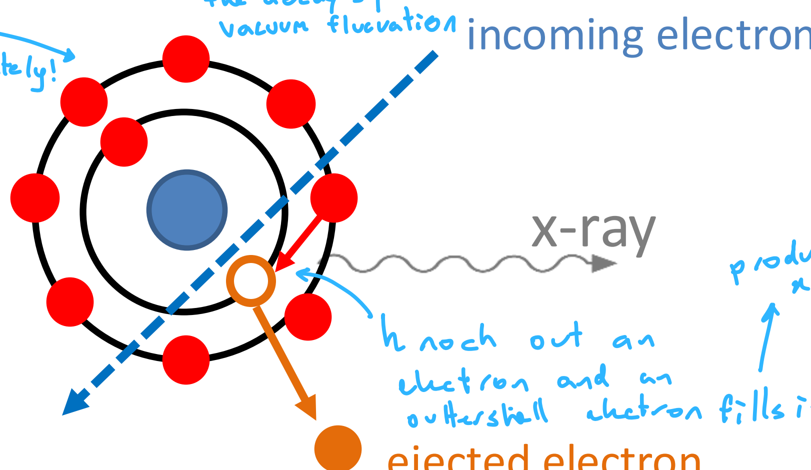
ask more about this!

vacuum flucuation causes the outer electron to decay
↳ Uncertainty principle

The electron is defined by a blur in the electron cloud that cannot be defined!

This principle causes the decay by the vacuum flucvation

does not stay ionised
-though does not happen immediately!



knoch out an electron and an outtershell electron fills its place

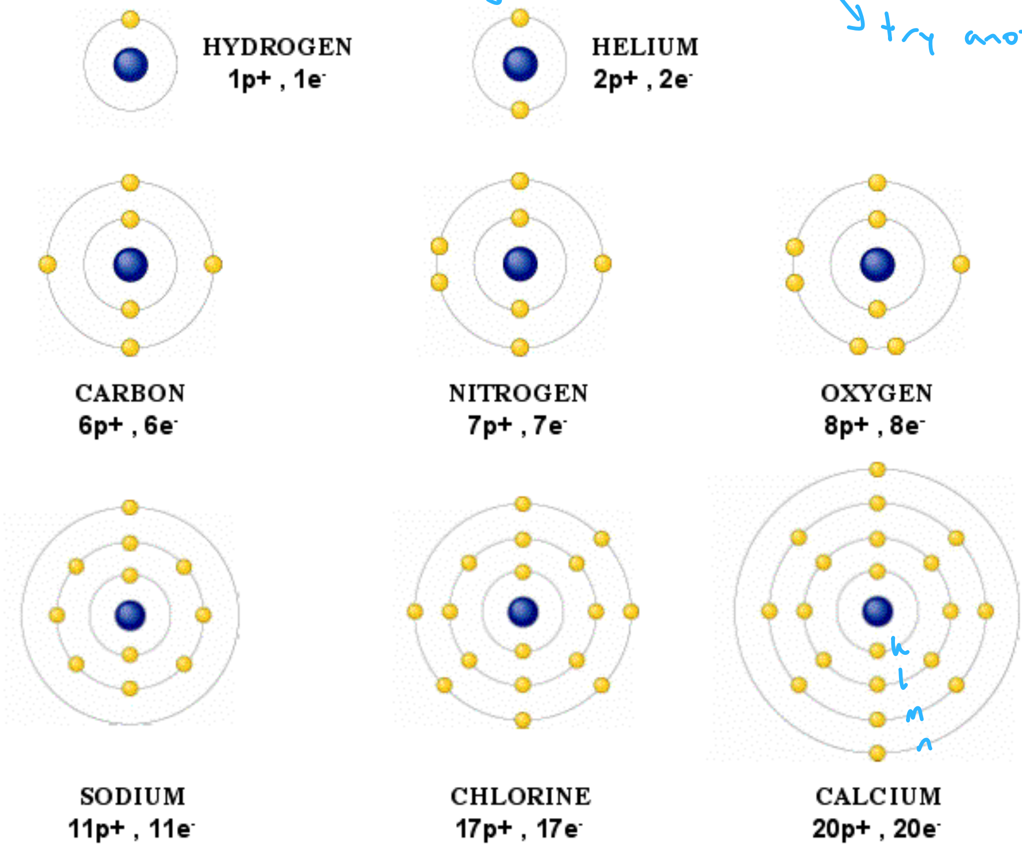
produces an x-ray

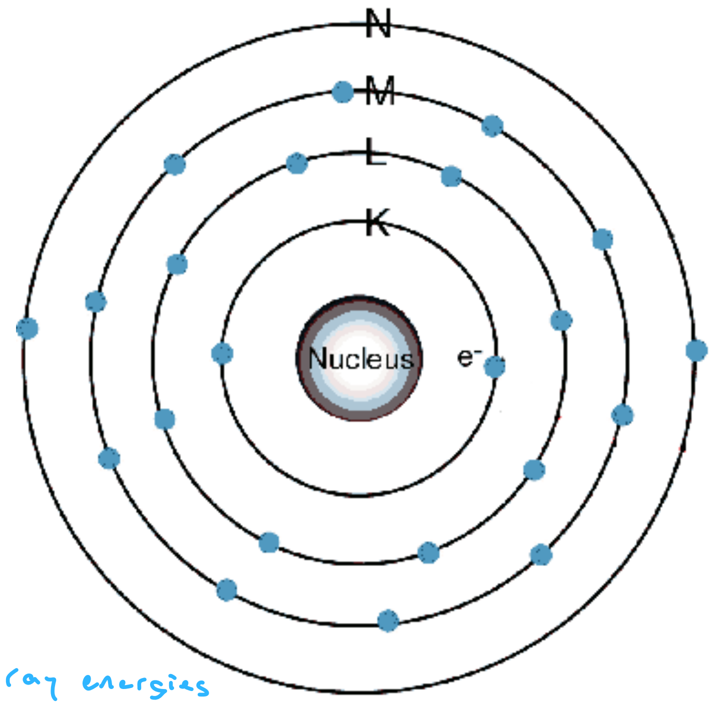
Atomic structure

nothing to do measure these 2

no outer shell electron so no spectra

try another technique





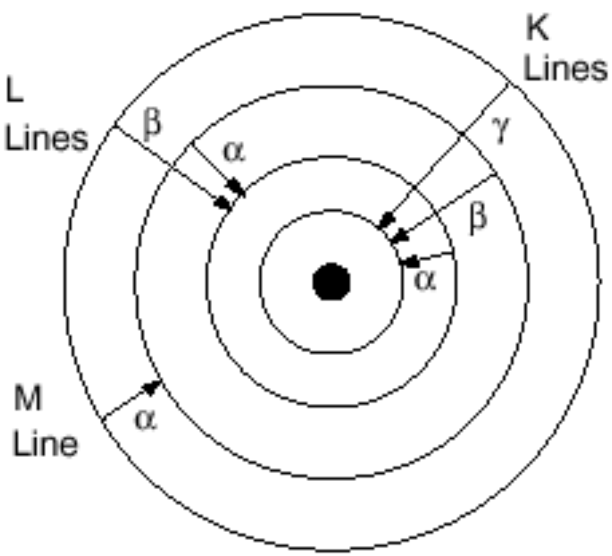
x-ray energies are given by energy difference

X-ray lines are labelled K, L, M… These letters denote the orbital that is ionised (ie: the orbital that an electron transitions to upon x-ray generation)

Energy required to remove the electron from an orbital is known as the ionisation potential (a.k.a. critical edge energy, E_C) generally around 0.1 to 50 keV

Characteristic x-rays

- energy of the X-ray is determined by the energy difference between the atomic levels involved in the electronic transition
- each element has a characteristic set of X-rays and X-ray spectra can be used to identify elements from nanometer sized volumes



X-ray notation

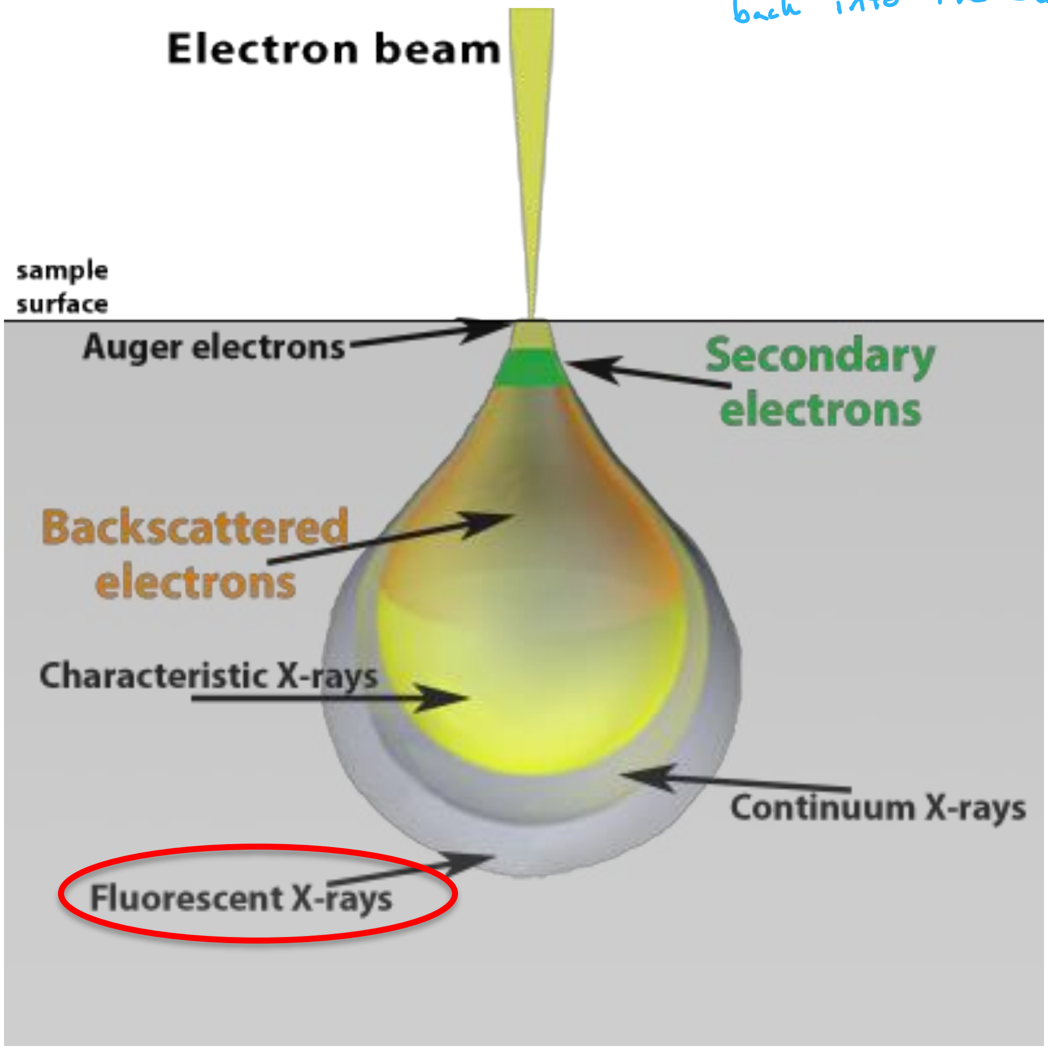
K x-rays are transitions that terminate on **1s level**

L x-rays are transitions that terminate on **2s, 2p levels**

M x-rays are transitions that terminate on **3s, 3p, 3d level**

X-ray absorption (in a sample)

x-rays can be absorbs back into the sample



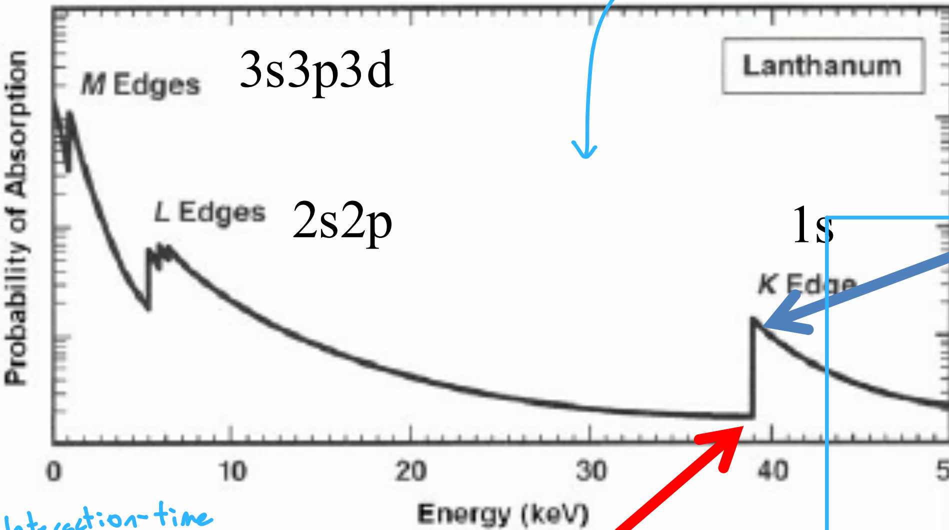
the x-rays generated within the interaction volume are absorbed by electrons as they travel through the sample

each x-ray photon is either absorbed entirely or it is emitted from the sample

X-ray absorption & “absorption edges”

low energy absorbs easily

absorption spectrum



maximum absorption immediately above K X-ray absorption edge

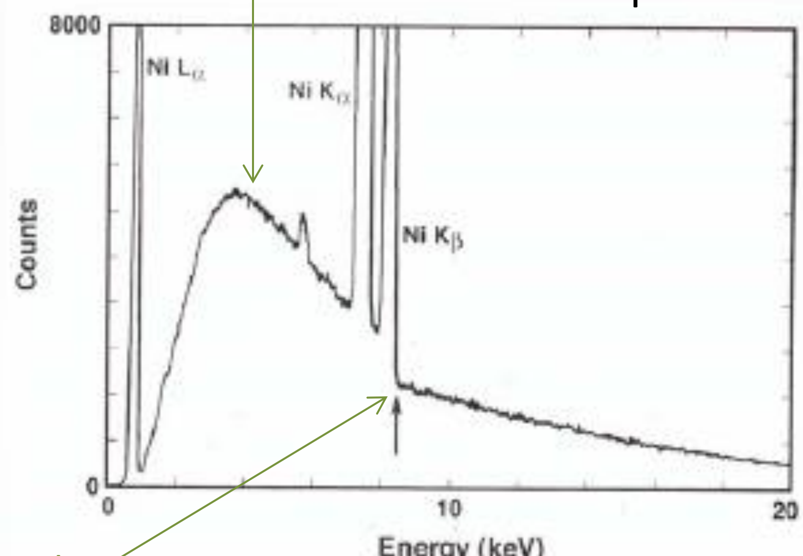
interaction-time
↳ ask to re-explain this

'wavefunction collapse'

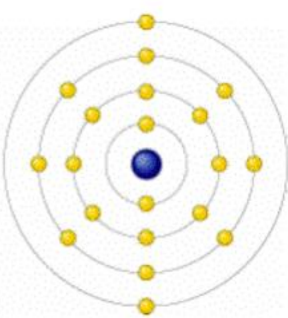
No K x-rays produced (by x-rays) when energy is below K Ec

background X-rays

emission spectrum



reflect how it reflects two graphs



abrupt decrease in background intensity above the K edge due to absorption by Ni

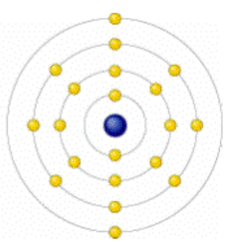
Absorption edge

- each electron in an atomic orbital (1s, 2s, 2p, 3s etc.) has a specific binding energy with the nucleus
- the closer the electron orbital is to the nucleus the higher its binding energy, $E_{\text{K-lines}} > E_{\text{L-lines}} > E_{\text{M-lines}}$
- the minimum energy required to completely remove an electron from orbital is known as the **critical edge** energy E_C (a.k.a. “ionisation potential”)
- electron beam energy must be greater than the critical edge, E_c to produce the X-ray

To generate x-rays must give min energy to 'knock out'

to overcome the binding energy

↳ highest energy is from the outermost energy



Platinum	Critical edge (keV)	X-ray Energy (keV)
K	78.39	66.83
L	11.56	9.442
M	2.133	2.051

X-ray self-absorption by a sample

Most x-rays must travel through the sample before leaving the surface

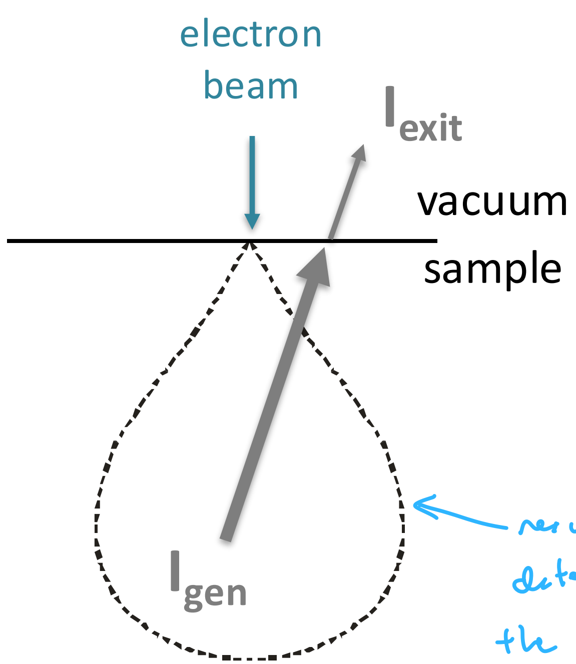
X-ray absorption follows Beer's law

$$I_{exit} = I_{gen} e^{-\mu S}$$

μ = mass attenuation coefficient
S = x-ray path length

In the lab!

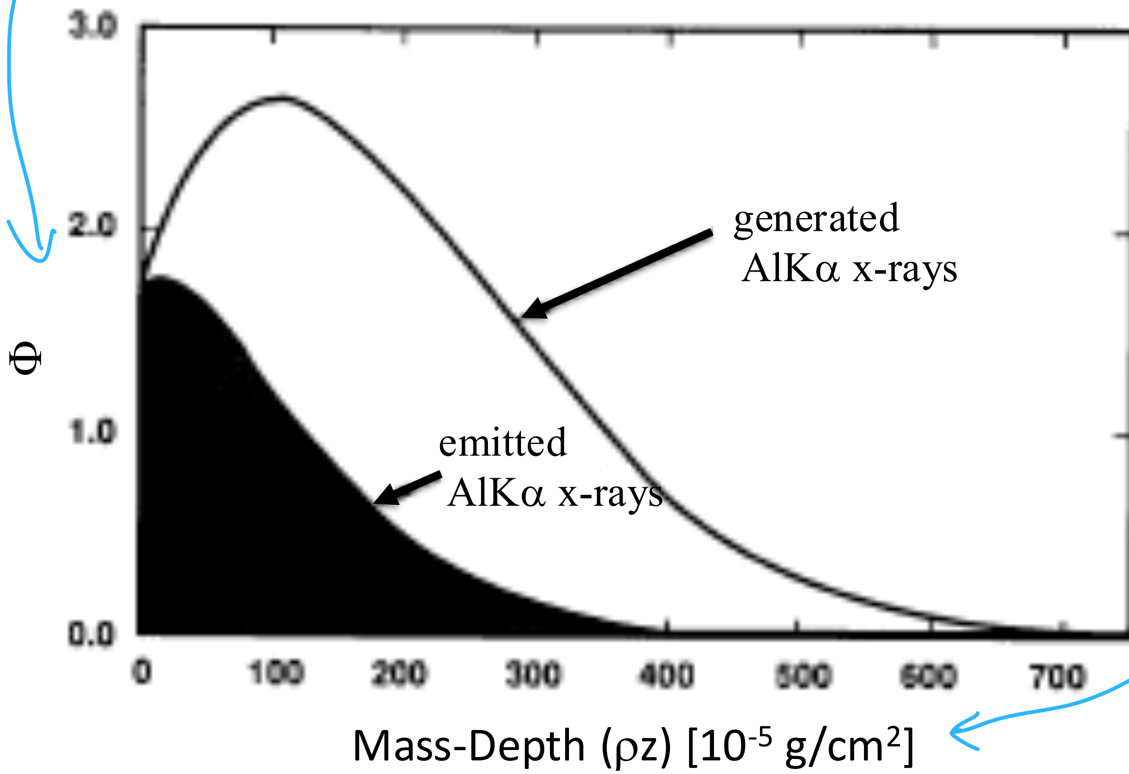
μ depends on energy of emitted X-ray and ionisation energies of atoms in sample

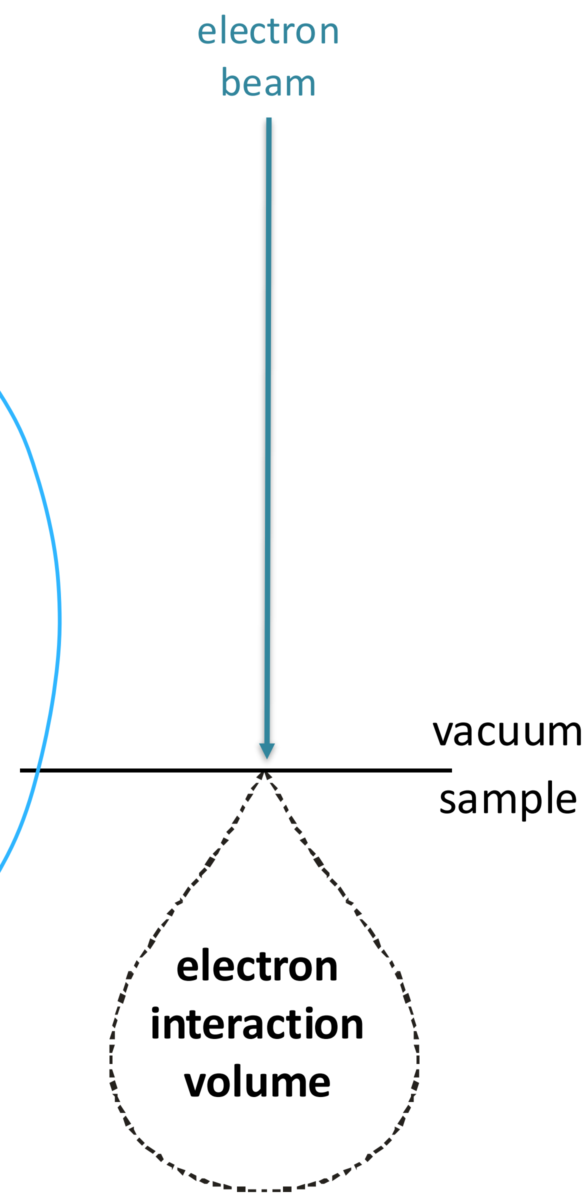


resolution is determined where the x-ray absorbtion interaction volume

$\Phi(\rho z)$ curves

Distribution of generated and emitted x-rays versus "mass-depth"





$\phi = f\,(\rho z)$

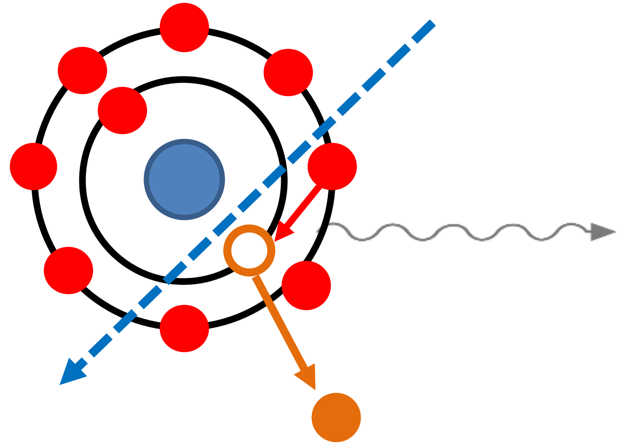
ρ = mass density [g/m^3]
z = depth [m]

X-ray generation probability

Next slides for revision

1. ionisation probability
2. fluorescence yield (probability of x-ray emission upon de-excitation of ionized atom)
3. secondary fluorescence (due to x-ray self-absorption)

x-ray probability



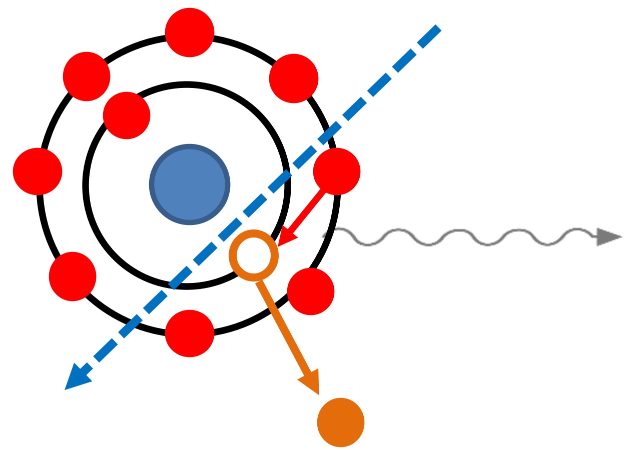
Ionisation cross-section, Q [units: m^2]

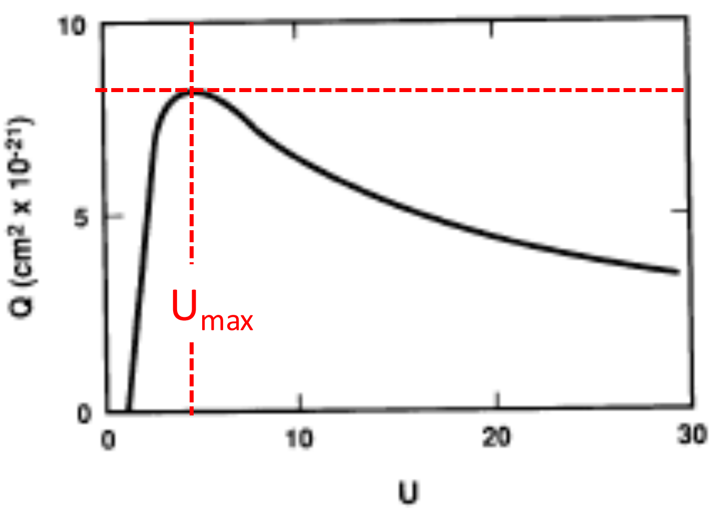
measure of ionisation probability quantified

$$Q = \frac{C_1}{UE_C^2}\ln(C_2 U)$$

where U is the overvoltage ($U = E_o/E_c$)

C_1 and C_2 are constants,
E_c = critical edge,
E_o = beam energy





maximum number of ionisations when U ~ 3 to 4 times E_c

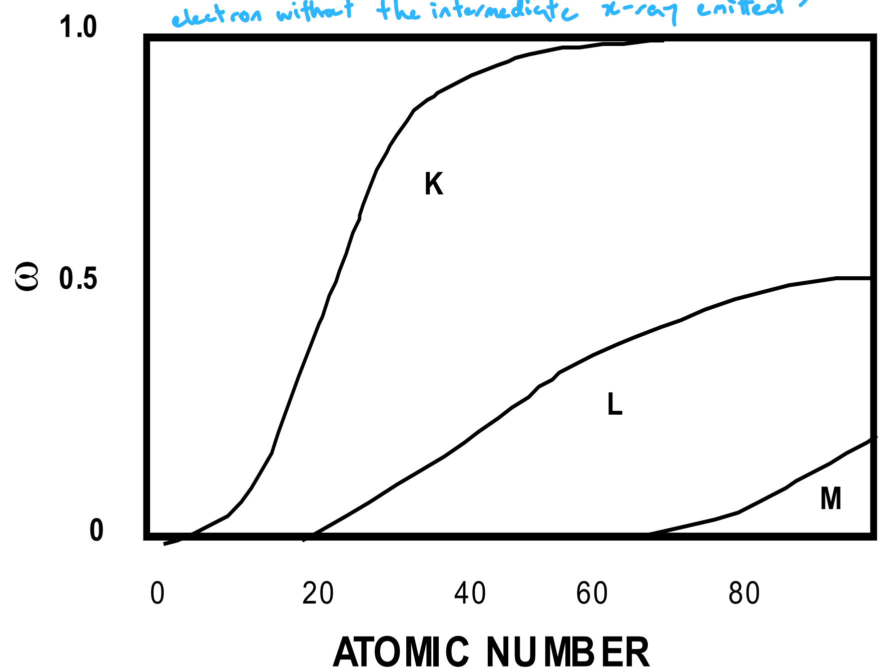
Fluorescence Yield, ω [unitless]

- not all ionisations result in the production of an x-ray
- there is a certain probability that an excited atom will relax via a radiationless transition in which energy is dissipated via emission of an energetic outer shell electron, known as an <u>Auger electron</u>

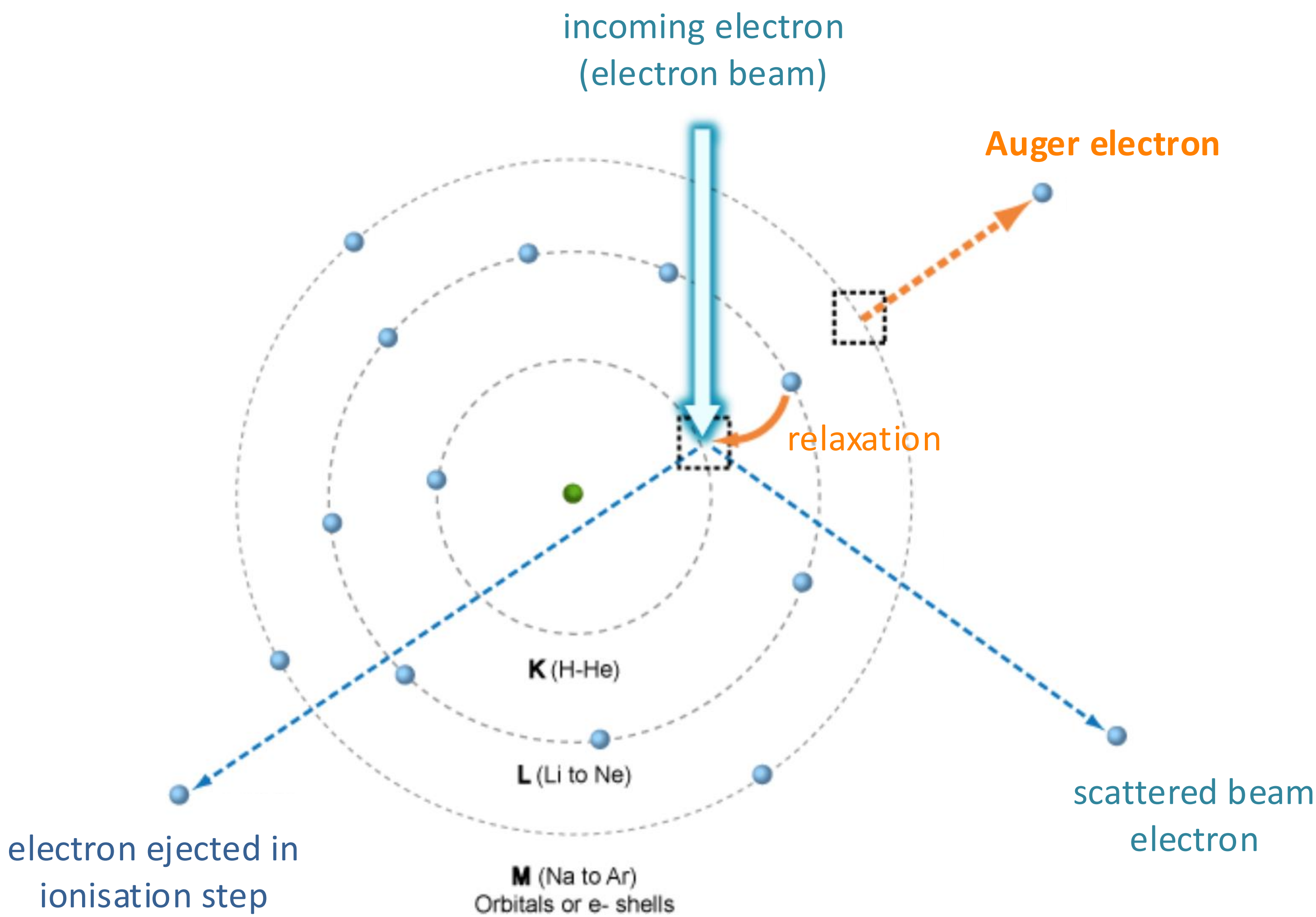
this is called:

something else can take up the energy instead of x-ray
↳ kinetic energy → electron of an outer shell electron without the intermediate x-ray emitted

- ω = the probability that an ionised atom will release a x-ray photon
- (1-ω) = probability that an ionised atom will release an Auger electron
- eg:
  - light elements (eg: C, N, O), ~ 1 in every 100 ionisation produces a K x-ray
  - transition metals (eg: Fe), ~ 1 in every 2 ionisations produces an x-ray

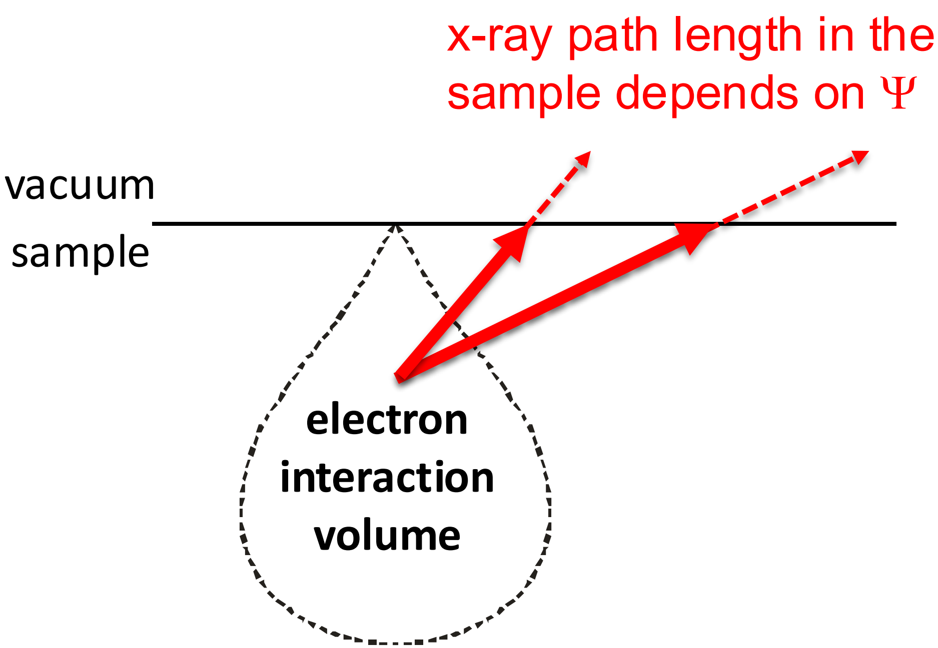


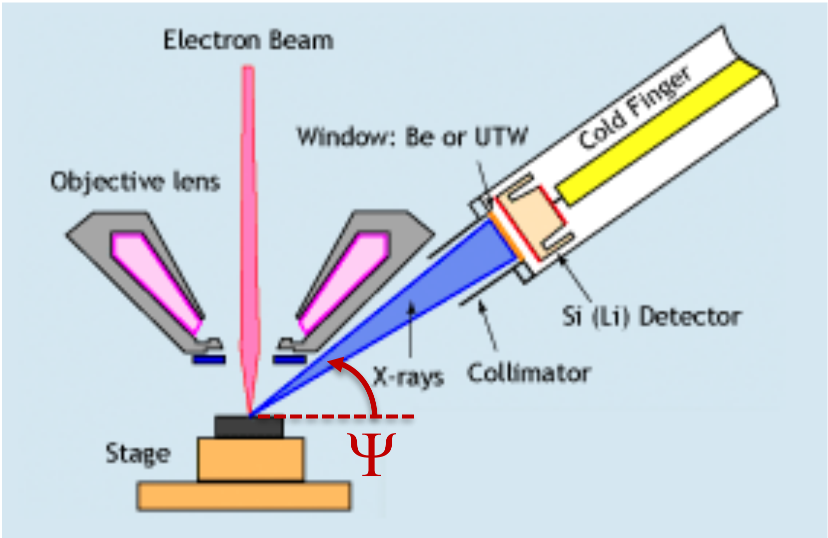
Auger electron emission



Secondary Fluorescence

- x-rays produced by other characteristic and background x-rays
- reduces the intensity of absorbed x-rays and increases the intensity of fluoresced x-rays
- affected by the x-ray escape depth, x-ray energy, sample composition and position of x-ray detector





- Ψ = "take-off angle"
- larger Ψ = less x-ray absorption
- Ψ limited by the the pole piece

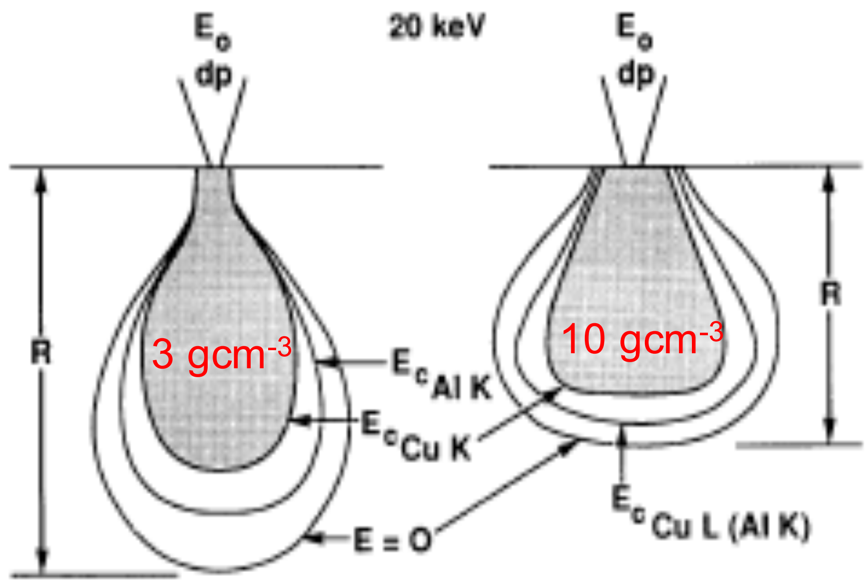
X-ray analysis spatial resolution

- determined by the x-ray range, not the beam diameter
- X-ray range (ie: maximum depth at which x-rays are generated) was determined by Anderson and Hasler:

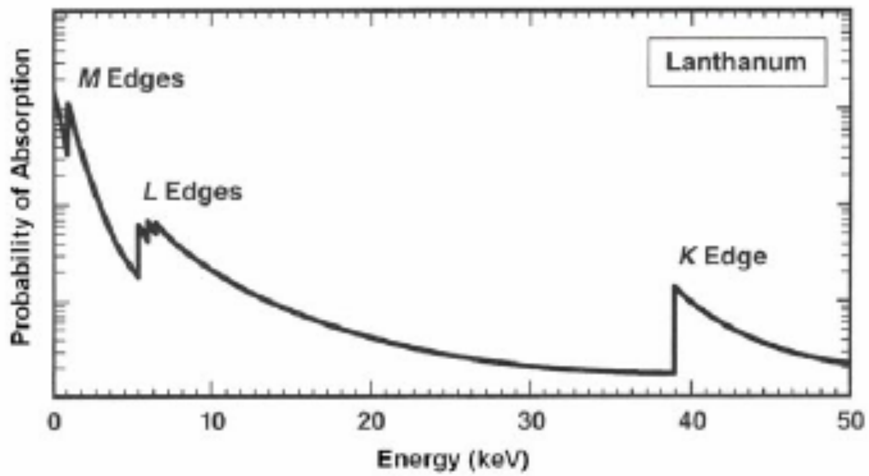
$$R_x = \frac{0.064\left(E_0^{1.68} - E_c^{1.68}\right)}{\rho}$$

each x-ray line has its own generation volume

R_x (μm) = x-ray range, E_o(keV) = e beam energy, E_c(keV) = critical edge, ρ (gcm^{-3}) = density



X-ray absorption



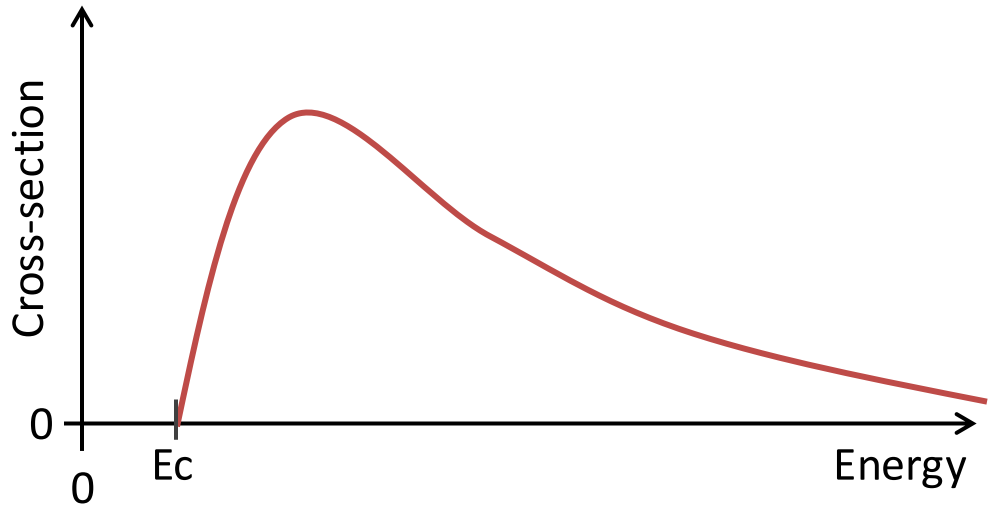
This x-ray absorption spectrum shows a number of so-called x-ray “absorption edges” for the element lanthanum. The edges are labelled K, L and M.

The K absorption edge is at energy Ea and the K x-ray has energy Ex.

Which statement is true?

a) Ea is equal to the energy of Auger electrons emitted from lanthanum atoms.
b) Ea is equal to Ex.
c) Ea greater than Ex.
d) Ea is smaller than Ex.

Cross-sections



The graph shows a cross-section that pertains to x-ray analysis in an electron microscope. Which statement is true?

a) A cross-section can not equal to zero below the energy Ec. This is physically impossible and hence this plot must be incorrect.
b) The units of a cross-section are the same as the units of probability (i.e., it is unitless).
c) This is a cross-section for an electron relaxation transition that gives rise to x-ray emission.
d) This is a cross-section for ionisation of an atom by an electron.
e) The quantity on the horizontal axis is the energy of the emitted x-ray.

What’s next?

Today	**Tutorial 2:** 12 PM
Thurs	**Prac 1:** [see next slide]

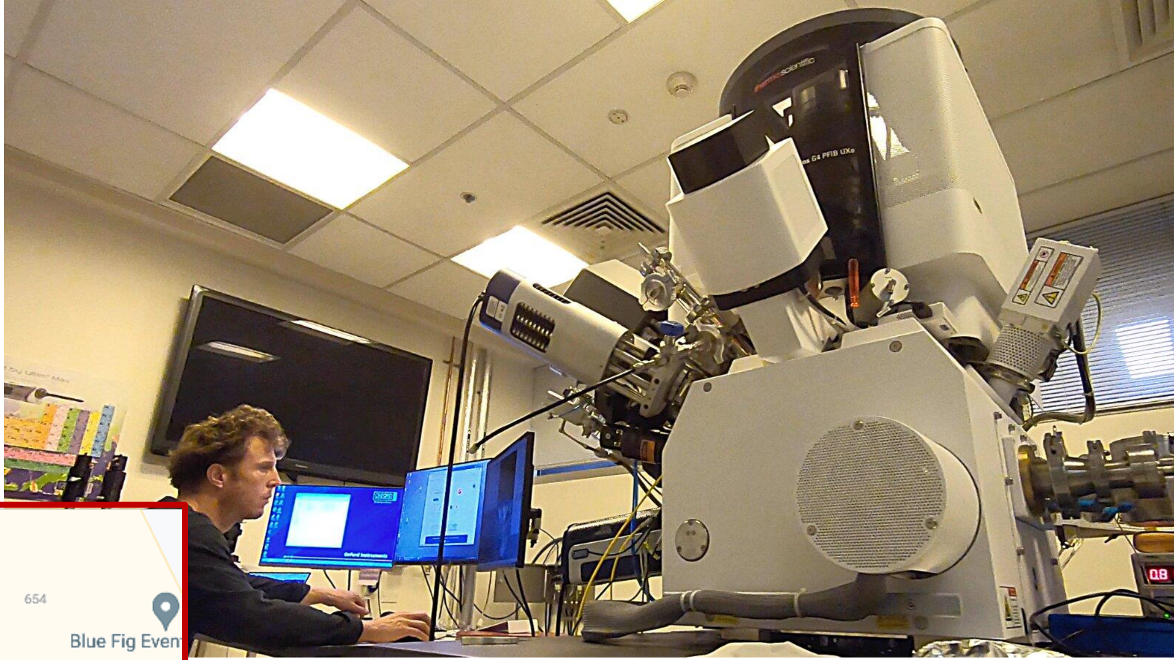
Next week	**Lecture 3:**	Tues
	Tutorial 3:	Tues
	Lab 2:	Thurs

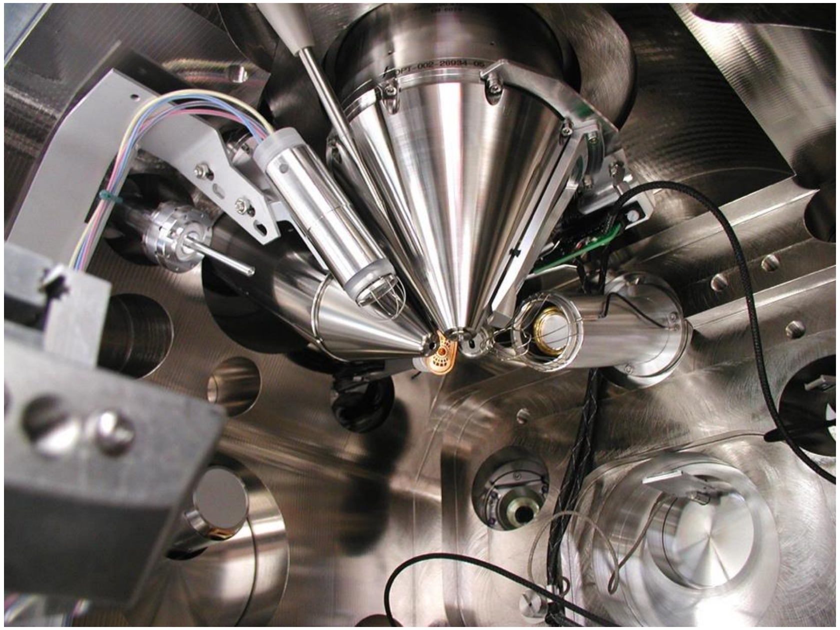
Program

Week/Session	Dates	Description
1	5 Aug	Lecture 1: Introduction Tutorial 1: Nano-scale imaging basics
2	12 Aug	Lecture 2: Electron optics and electron-solid interactions Tutorial 2: Electron-solid interactions Practical 1: Scanning electron microscopy
3	19 Aug	Lecture 3: Ion beams and electron/ion beam nanofabrication Tutorial 3: Ion-solid interactions Practical 2: Electron/ion beam nanofabrication
4	26 Aug	**CLASS TEST 1**
5	2 Sept	Lecture 4: Cathodoluminescence & x-ray analysis Tutorial 4: Cathodoluminescence simulations Practical 3: Nanocharacterisation techniques
6	9 Sept	Lecture 5: Advanced nanocharacterisation Practical 4: Nanocharacterisation techniques
7	16 Sept	Lecture 6: Ion-solid interactions and gas-assisted nanofabrication (advanced) **Portfolio 1 due this week** (practicals) **Portfolio 2 due this week** (tutorials)
8	23 Sept	**CLASS TEST 2**

Pracs

CB04.02.310
Corner of Harris & Thomas St
Near the UTS gym





Pracs: 1hour on Thursdays

- You will use 2 electron/ion beam labs
- Lab safety rules: Max 8 students per lab
- Class is split into 4 groups; find your group at [Canvas → People → Groups]
- Groups 1 & 2 start at 1PM
- Groups 3 & 4 start at 2 PM