Scanning electron microscopy techniques

MS-633 Materials Science and Engineering (EDMX)

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http://cime.epfl.ch

Outline

- **Introduction to electron microscopy (EM)** (by E. Oveisi)
  - **Why use electrons?**
  - **Wavelength and resolution**
  - **Types of interactions**

- **EM setup** (by E. Oveisi)
  - **Electron sources**
  - **Lenses**
  - **Vacuum system**
  - **Detection system**

- **SEM** (by E. Oveisi & M. Cantoni)
  - **Operation, Signals**
  - **Contrast mechanism**
  - **Interpretation of images, Challenges**
  - **Related techniques**
  - **Advanced and high-resolution SEM**

- **Chemical analysis and Monte Carlo simulations** (by M. Cantoni)

- **Focused ion beam** (by M. Cantoni)

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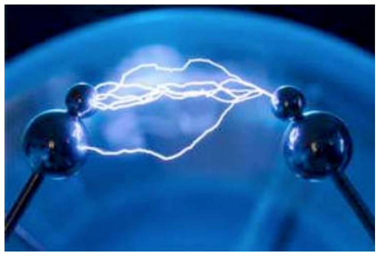
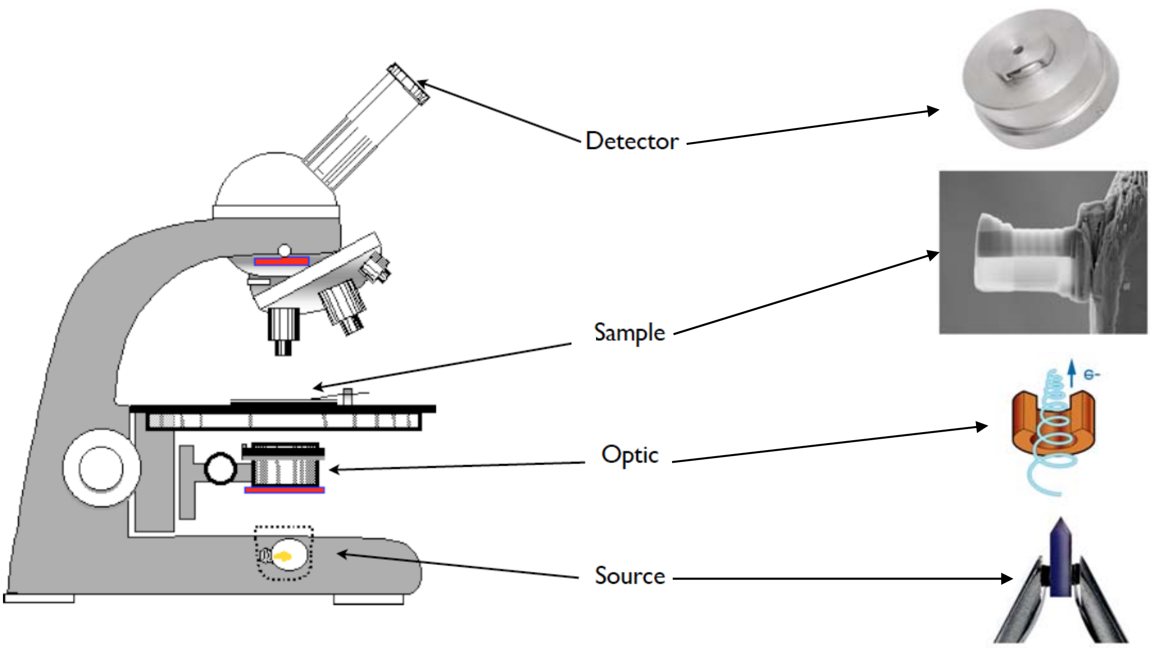
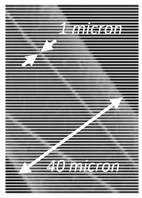
What is an electron microscope?



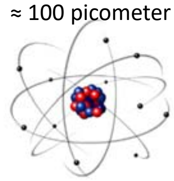


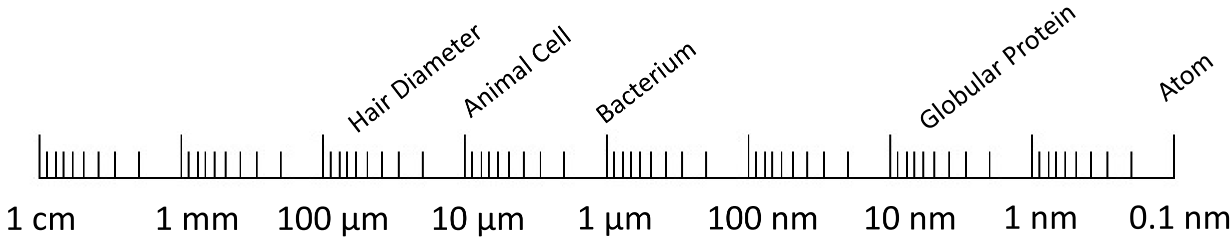
Why use electrons as probe?



DNA helix

Size of an atom
≈ 100 picometer





Naked eye

Electron microscopes

Conventional light microscopes

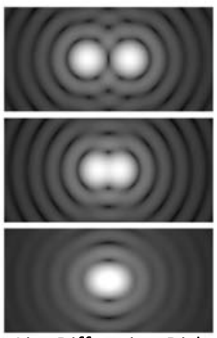
Visible light wavelength: 400-700 nm

Electron wavelength: 0.0387 nm @ 1 keV
0.0019 nm @ 300 keV

Developed by Ernst Ruska in 1931 (Nobel Prize in Physics 1986)
Electrons have wave-like properties (De Broglie 1924)
The electron wavelength depends on its energy
Magnetic lenses can be used to focus electrons

Why use electrons as probe?

Definition of resolution: The minimum distance that two point-source objects have to be in order to distinguish the two sources from each other. There are two closely related values for the diffraction limit, the Abbe and Rayleigh criterions.



Airy Diffraction Disks

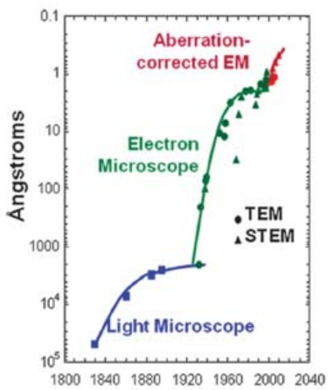
Abbe's definition of maximum resolution of an optical system states that the smallest feature resolved is limited by diffraction.

- Abbe diffraction limit for the light microscope

(around 1000 atomic diameter) around half of the wavelength

- Equivalent for the electron microscope

25-50 x wavelength



* NOTE: Lens aberrations limit spatial resolution
Spherical and Chromatic Aberration corrections allow for 0.05 nm resolution at 300 kV

Why use electrons as probe?

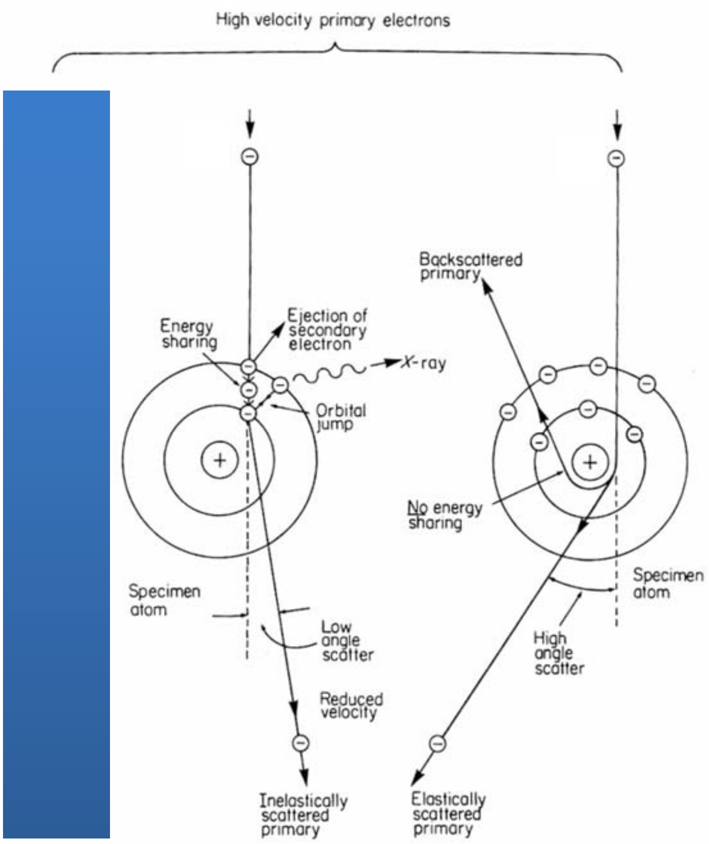
	Advantageous	Disadvantageous
Visible light	Not very damaging Easily focused Eye detector	Long wavelengths (400 nm)
X-rays	Small wavelength (Angstrom) Good penetration	Hard to focus Damage sample
Neutrons	Low sample damage Small wavelength (pm)	How to produce? How to focus?
Electrons	Small wavelength (pm) Can be focused	Damage sample Poor penetration

- High en
- Easy to
- Easily m
- **Interact**

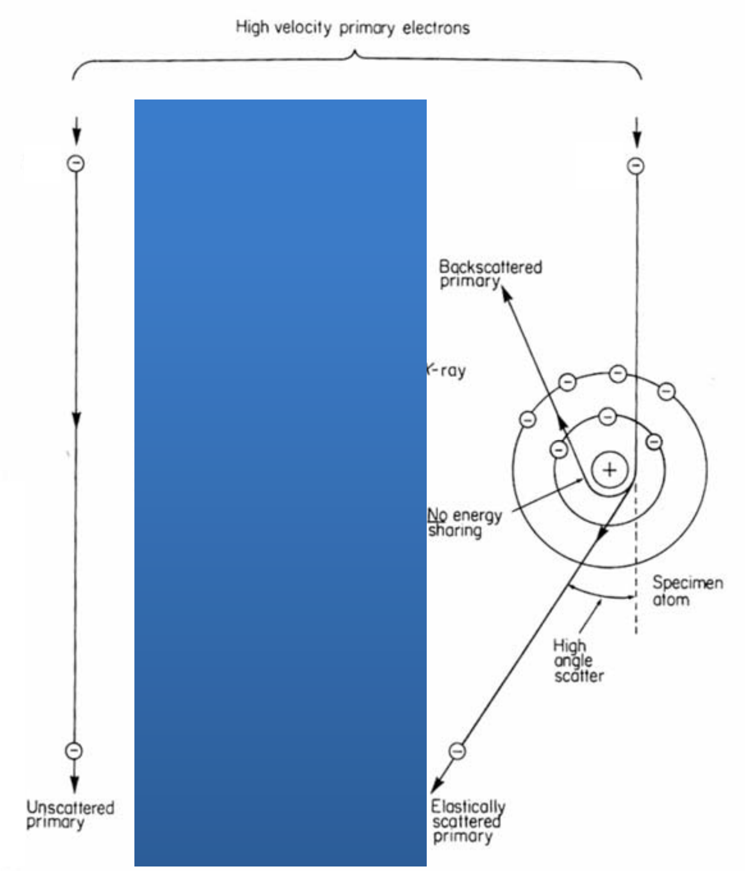
Electron microscopes are used not only for obtaining good resolution images but also:

- **can be used as a diffractometer**
- **for chemical analyses**
- **for imaging/measuring strain field in the sample**
- **etc.**

Three classes of scattering outcomes



Three classes of scattering outcomes



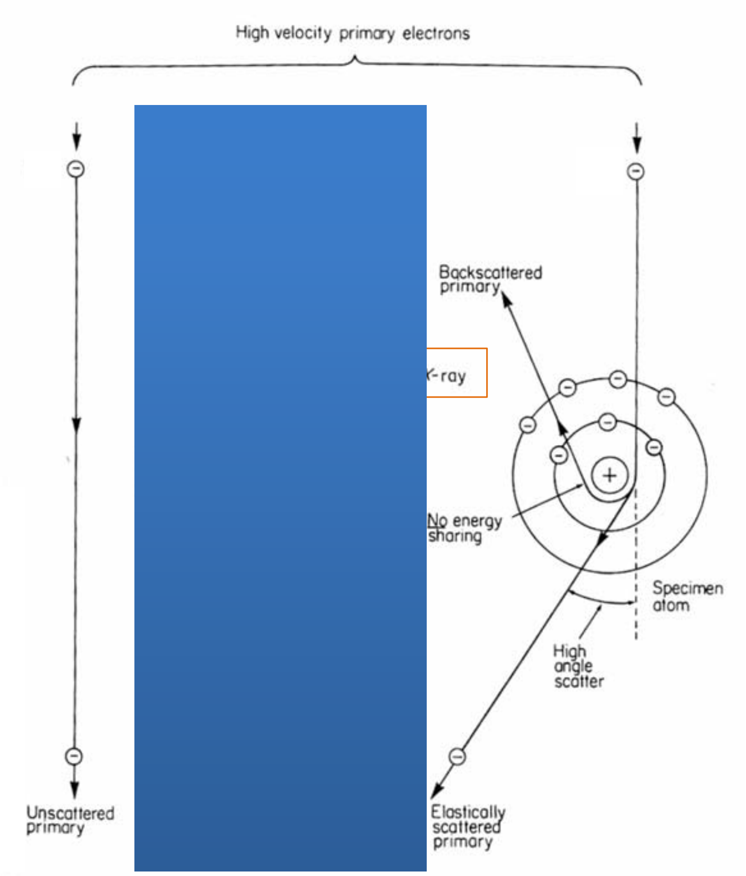
Inelastic events occur when the electron beam interacts with the electric field of a specimen atom electron.

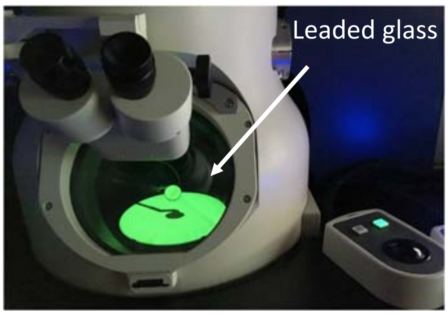
The result is a **transfer of energy** to the specimen atom and a potential expulsion of an electron from that atom as a **secondary electron (SE)**. SEs by definition are less than 50 eV.

If the vacancy due to the creation of a secondary electron is filled from a higher level orbital, an X-Ray characteristic of that energy transition is produced.

Energy transfer (loss) - reduced velocity -> Will be focused more strongly by the lenses,
at a plane higher up in the microscope than the electron that are scattered/elastically scattered

Three classes of scattering outcomes

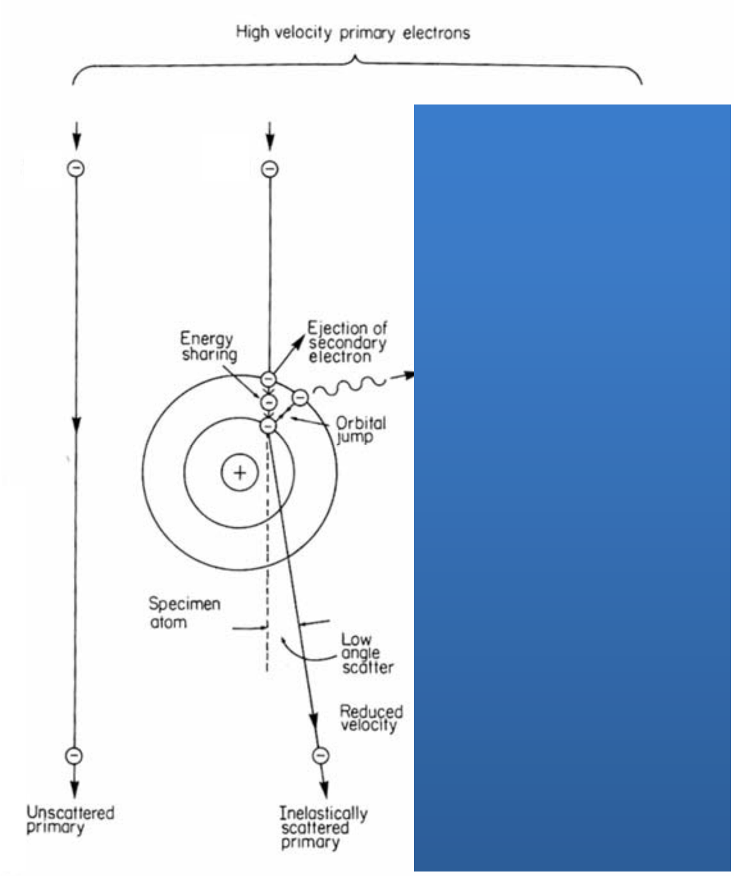




That's why TEMs are shielded!

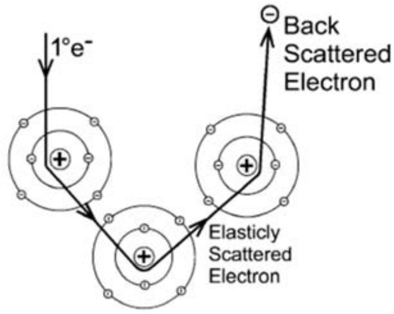
Reduced velocity -> Will be focused more strongly by the lenses,
at a plane higher up in the microscope than the electron that are scattered/elastically scattered

Three classes of scattering outcomes



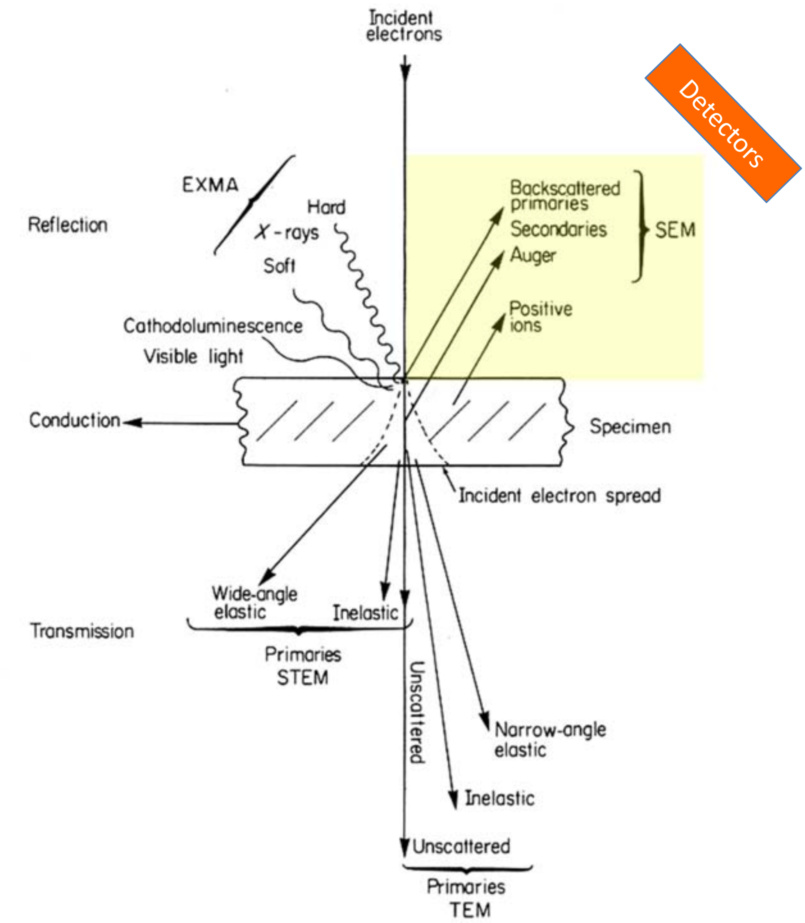
Elastic events occur when a beam electron interacts with the electric field of the nucleus of a specimen atom, resulting in a change in the direction of the beam electron **without a significant change in the energy** of the beam electron (< 1 eV).

If the elastically scattered beam electron is deflected back out of the sample, the electron is termed a **backscattered electron (BSE)**.



No (negligible) energy transfer
High angle scattering for elastically scattered electrons

Types of electron microscopes

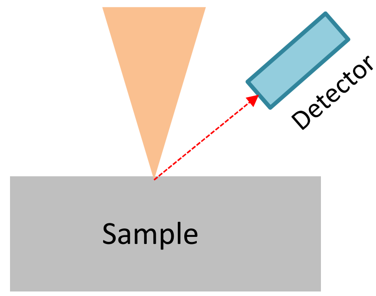


Reflected signal from:

- back-scattered electrons
- secondary electrons (emitted by the sample)
- Positive ions
- etc.

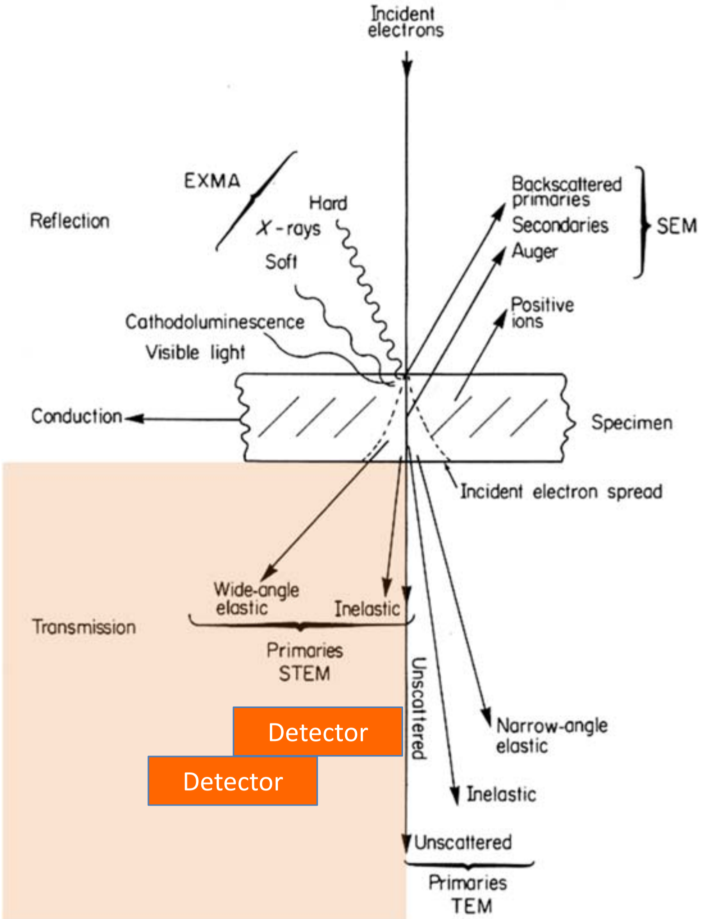
Scanning Electron Microscopy

Electron beam (0.2-30 keV)



Convergent illumination (= probe)
Scanning (moving beam) mode

Types of electron microscopes

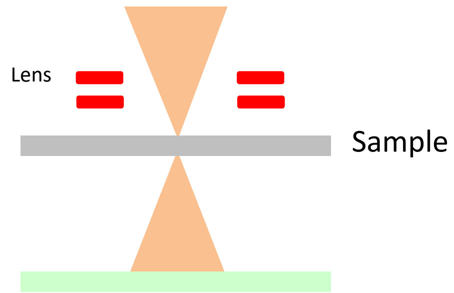


Transmitted signals from:

- Elastically scattered electrons
- inelastically scattered electrons
- Diffuse scattering events
- etc

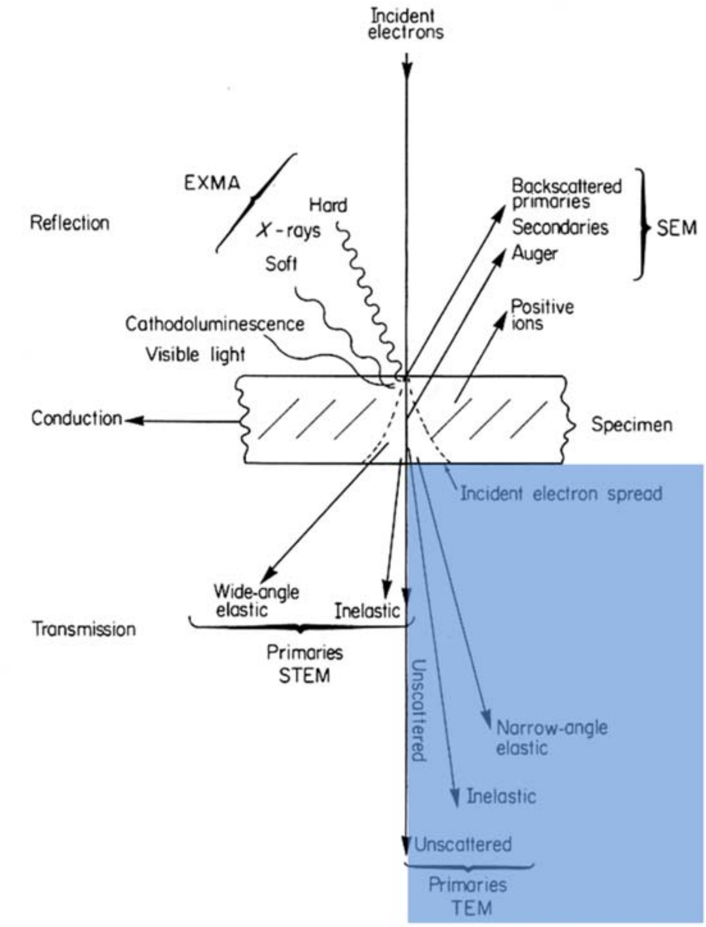
Scanning Transmission Electron Microscopy

Electron beam (60-300 keV)



Convergent illumination
Scanning mode

Types of electron microscopes

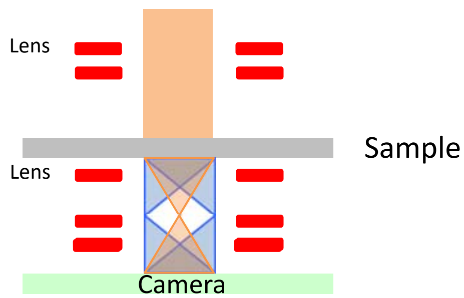


Images is created through the interference between the:

- Unscattered primary electrons (transmitted beam)
- Narrow-angle elastically scattered (diffracted) electrons

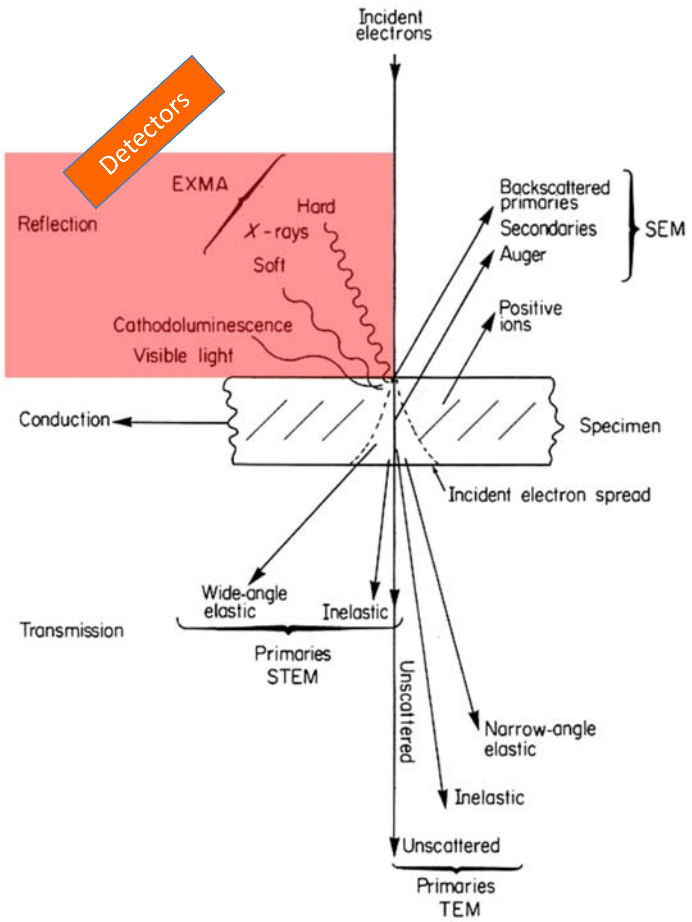
Transmission Electron Microscopy

Electron beam (60-300 keV)



Parallel illumination

Types of electron microscopes



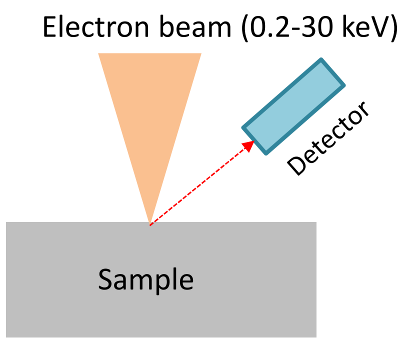
Inelastic events will ionize atoms, when they relax they emit:

- X-ray signals (characteristic X-rays)
- Photons (IR, UV)
- Visible light (Cathodoluminescence)
- Etc.

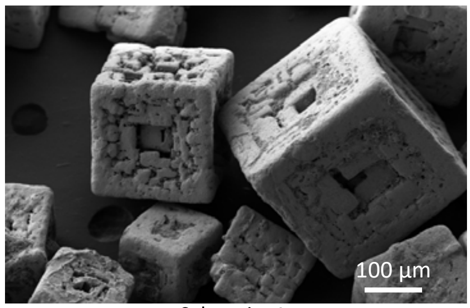
EXMA: Electron Probe X-ray Microanalyser

Types of electron microscopes

Scanning electron microscope (SEM):

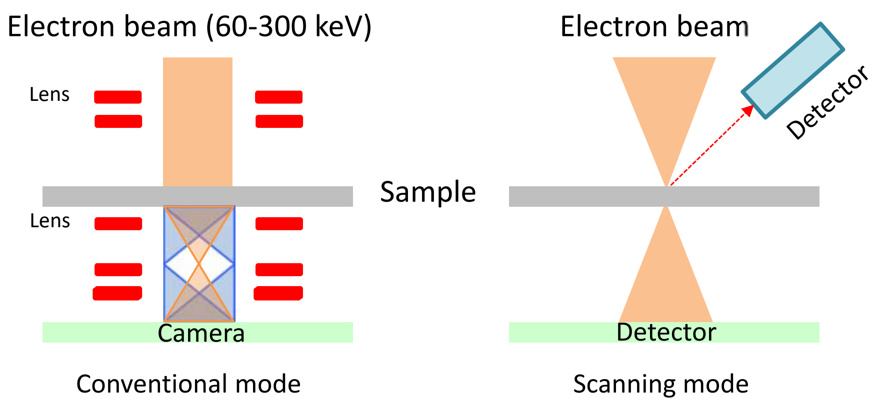


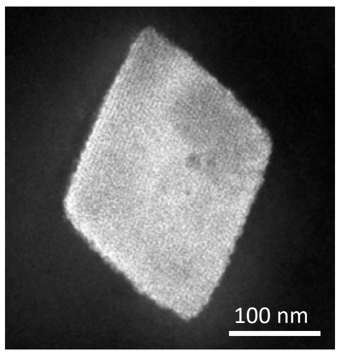
Signals:
Secondary electrons,
Back-scattered electrons,
X-ray, Auger, …



Salt grains [a]

Transmission electron microscope (TEM):

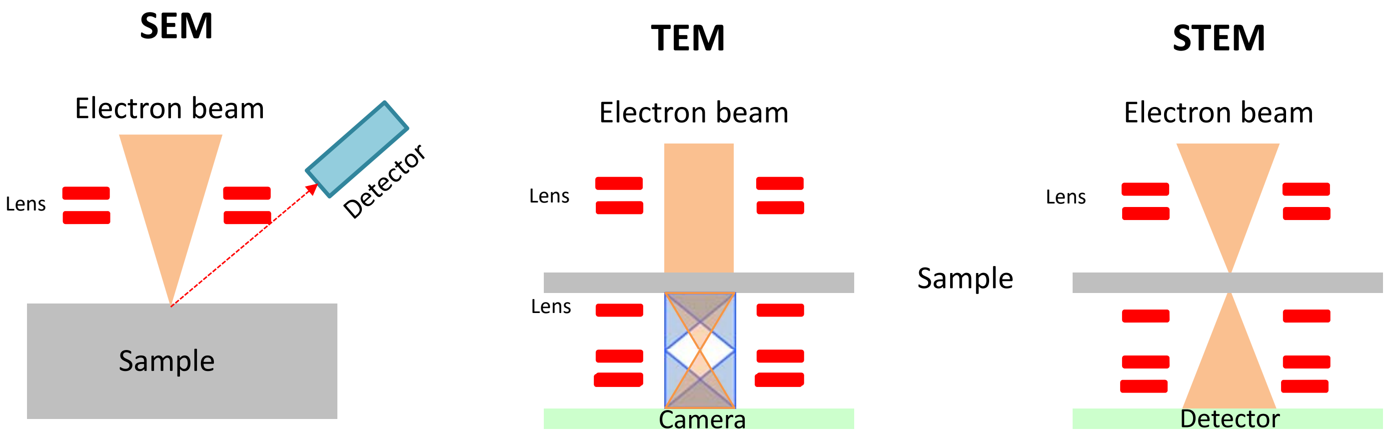




Cu-based metal-organic framework [b]

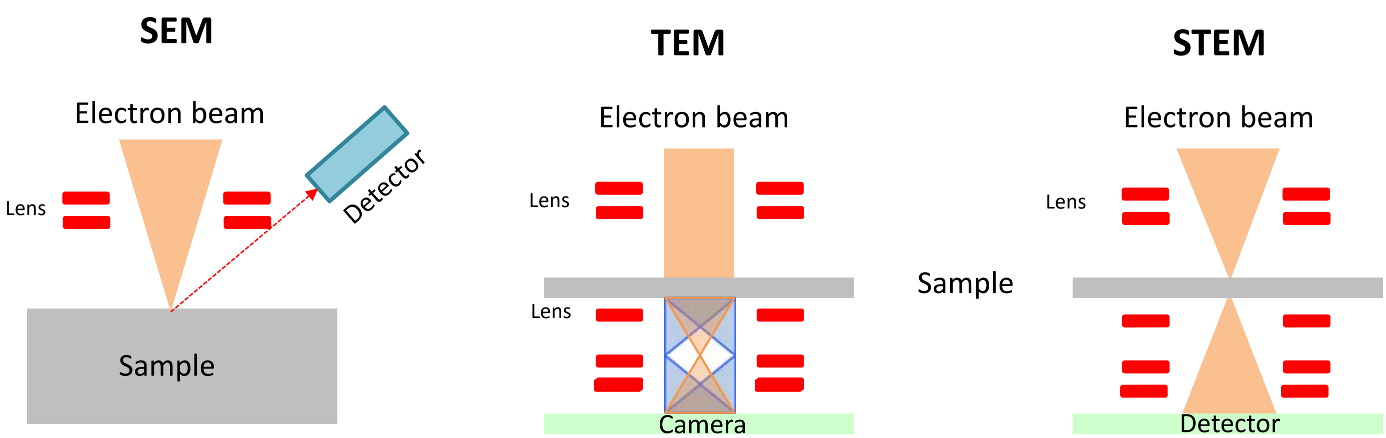
[a] http://www.trente.eu
[b] D. Sun et al., ACS Central Science 2018.

Components of an electron microscope



Vacuum system
Electron gun
Lenses
Detectors

Components of an electron microscope – Vacuum system



1. Electron propagation is only possible through vacuum!
2. Need a good vacuum system to reduce contamination!

Vacuum scale

To clean the microscope room!	Primary vacuum	Secondary vacuum	Corse UHV (High vacuum)	UHV

760 — 10^{-1} — 10^{-3} — 10^{-6} — 10^{-9} — *Torr*


Hoover, …


Rotary vane pump

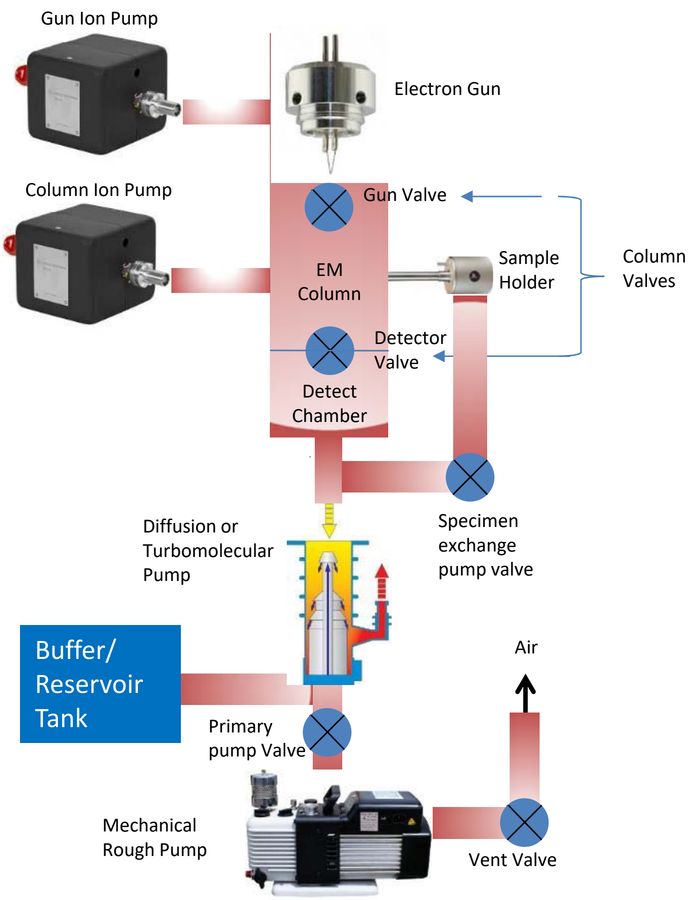

Oil diffusion pump


Turbomolecular pump


Ion getter pump

Different kinds of vacuum pumps have different range where they are effective

Vacuum system



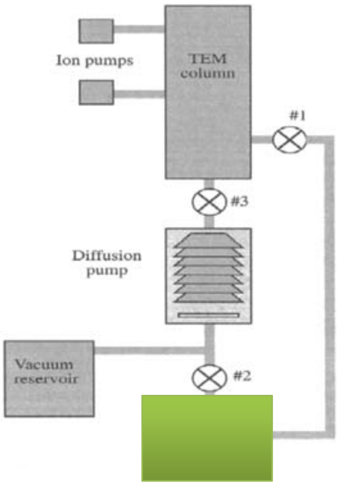
- **Primary vacuum (>0.1 Pa)**
  - Mechanical pump
- **Secondary to high vacuum**
  Detector or viewing chamber (<10^{-4} Pa)
  - Oil diffusion pump
  - Turbomolecular pump
- **High and ultra-high vacuum**
  Gun & specimen area (<10^{-6} Pa)
  - Ion getter pump
  - Cold trap

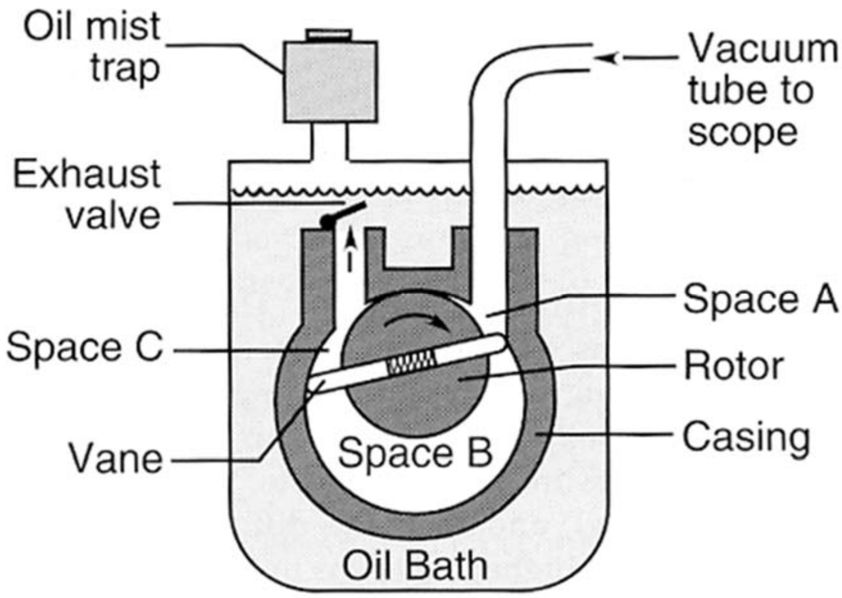
Vaccum level in space:
1 Pa at 100km
above earth surface

Vacuum system – Primary vacuum

Rotary vane pump

- Uses oil
- noisy

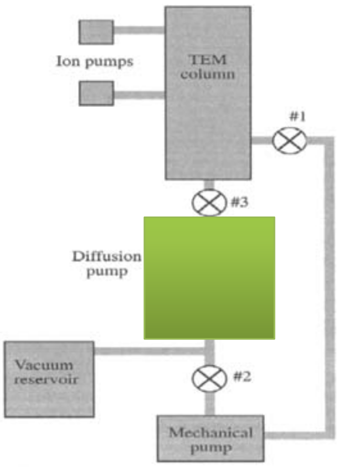


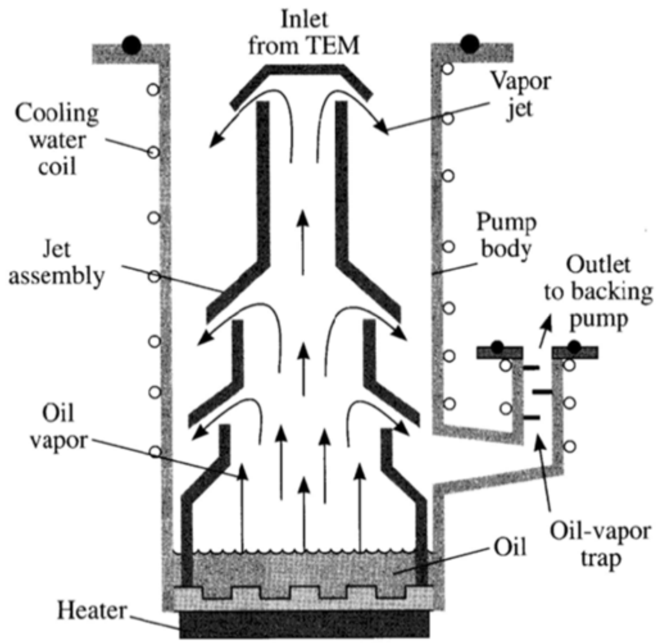


Vacuum system – Secondary vacuum

Oil diffusion pump

- Vibration free
- Contamination possible oil vapor
- High pumping capacity (>500 l/s)
- Best with cold trap

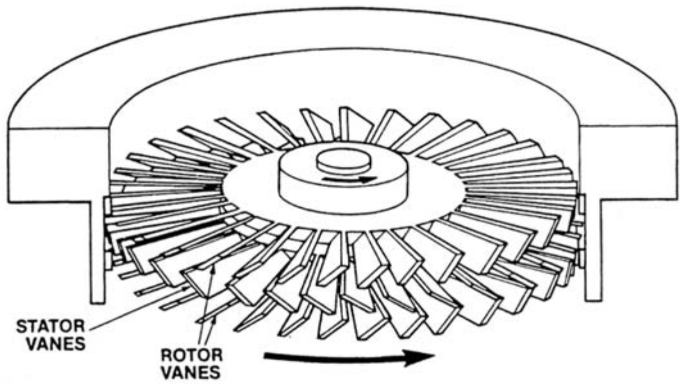


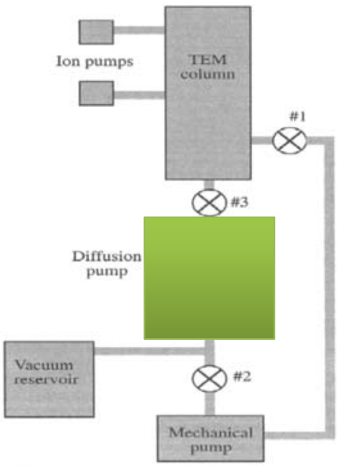


Vacuum system – Coarse UHV

Turbomolecular pump

- Rotation speed 20-50'000 rpm
- Magnetic bearings
- Pumping volumes 50-500 l/s

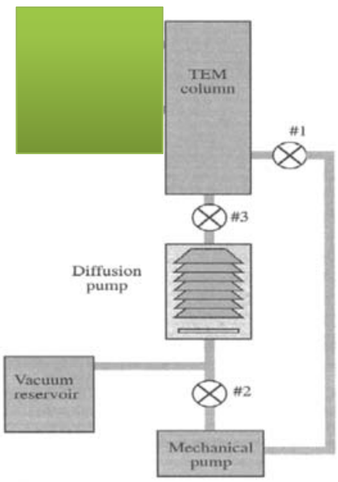


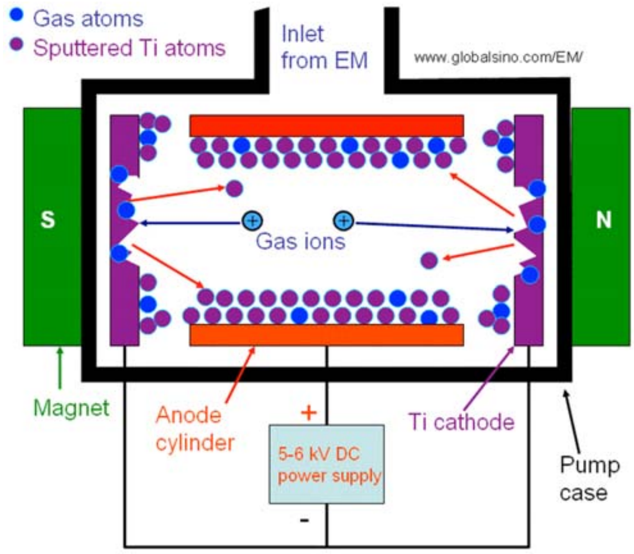


Vacuum system – Ultra-high vacuum

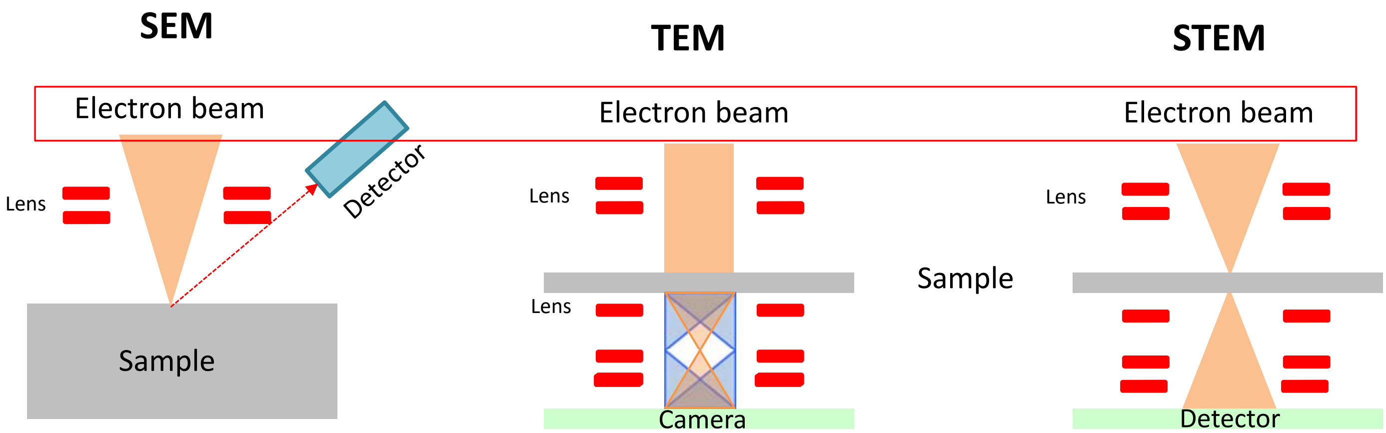
Ion getter pump

- no vibrations
- No exit:
  improves vacuum !

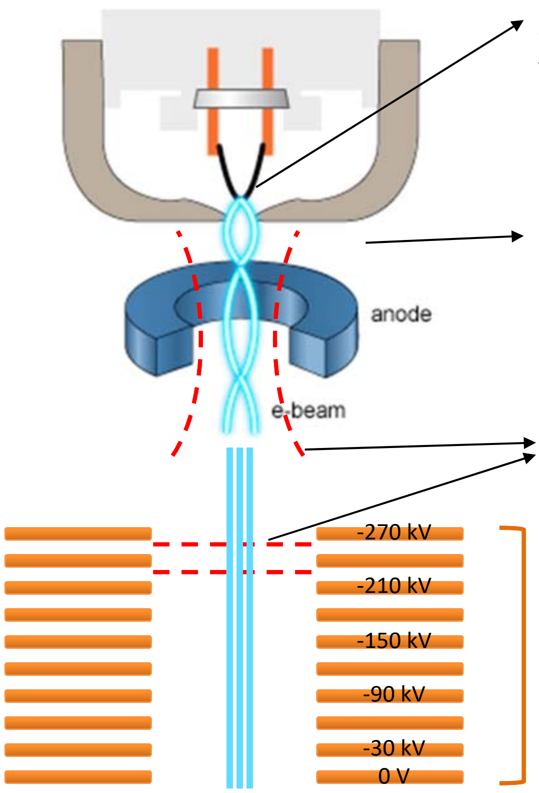




Components of an electron microscope – Electron gun

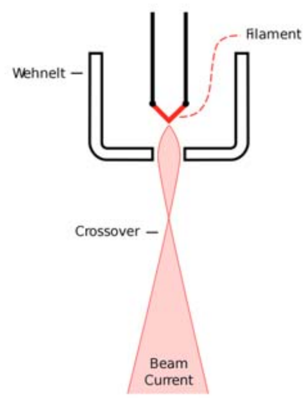


Electron gun



Filament
Electrons receive enough energy to escape the filament (work function of the filament)
The filament is hold at a negative voltage e.g. – 300 kV

Wehnelt cup and anode
Produces a small negative bias (e.g. -300 kV – Δ) to focus electrons to crossover



Electrostatic field

Accelerator
Lets give the electrons some speed

Is isolated by SF_4 gas -> Heavier than air

Electron gun

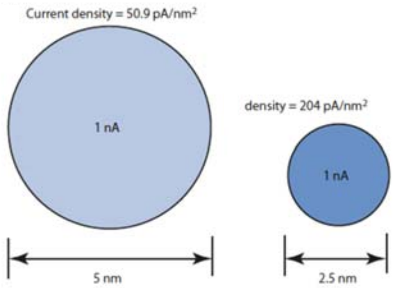
Intensity | Brightness

Spatial coherency:
Do all the electrons com form the same direction?
An electron beam emanating from a small source size is said to have high **spatial coherency.**

Temporal coherency:
Do all the electrons have exactly the same speed/energy?
A beam with high **temporal coherency** will have electrons of the same wavelength.

Important parameters

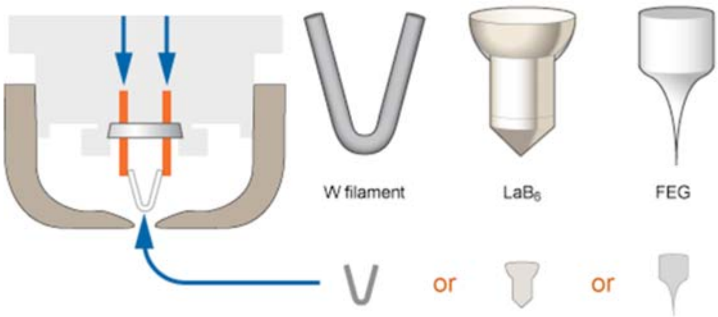
- Source and crossover size: determines the probe size (→ resolution)
- Energy spread: temporal coherency
- Emitted current and current density
- **Brightness**: current per surface unit and per solid angle
- Current stability
- Vacuum needed

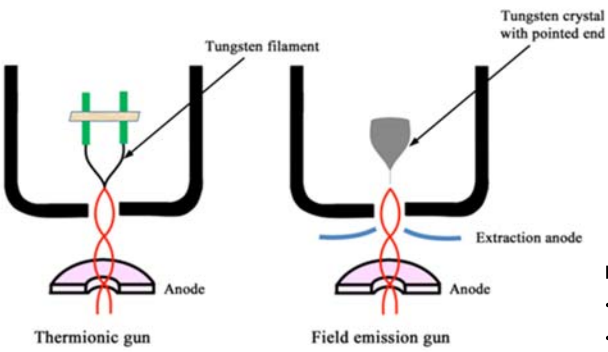


Electron gun

Two types of emission guns:

- **Thermionic gun**
  - W or LaB_6 crystal
- **Field emission gun (FEG)**
  Cold FEG
  Schottky FEG



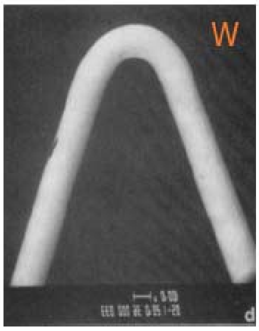


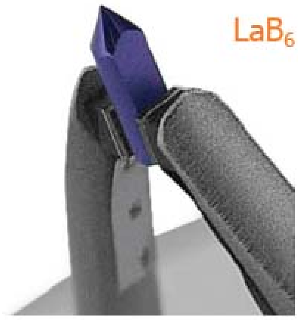
In a FEG:

- **High field E≈10^9V/m**
- **First anode = extractor (always constant)**
- **Second anode = accelerator**

Electron gun – Thermionic gun

- Filament is heated to overcome the work-function to release electrons to vacuum level.
- Tungsten wire heated to ~2800K
- LaB_6 crystal heated to 1900K





Advantages
Simple to use
Cheap
No ultra-high vacuum required
Maintenance friendly

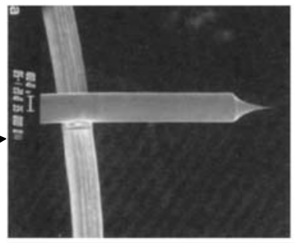
Disadvantages
Low brightness
High energy spread
Large source size (10-100 □m)

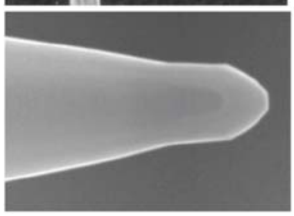
Thermionic gun
Analogous to volcano
More electrons form a large tip (10-100 □m)
Different energies
Different directions

Electron gun – Field emission gun

- The strength of an electric field E is considerably increased at sharp points.
- Lowers the work-function barrier so that electrons can tunnel out of the filament (usually tungsten).
- Surface has to be pristine (no contamination or oxide)
- Ultra-high vacuum condition (Cold FEG) or poorer vacuum if tip is heated (”thermal” FEG; ZrO surface treatments → Schottky emitters).

  - **Cold field emission** ($E \approx 10^9$ V/m)
    W mono-crystal with sharp tip
    tip radius ~100nm
  - **Thermally assisted emission**:
    Shottky effect
    W/ZrO tip at 1700-1800K





Advantages
Small energy dispersion (< 0.4 eV)
High coherence
High brightness
→ higher resolution at lower energies

Disadvantages
Expensive
Ultra-high vacuum necessary
Cold FEG needs flushing (cleaning) after ~8 hrs

FEG
Analogous to child’s slide
Electrons from a very sharp tip radius ~100nm
Same energy
Same direction

Electron gun

Characteristics of principal electron sources at 200 kV

	W	LaB6	FEG Schottky (ZrO/W)	FEG cold (W)
Crossover size (nm)	**>10^5**	10^4	**10-100**	3
Emission current (μA)	**100**	20	**100**	20~100
Current density (A/m^2)	**5**	10^2	**10^5**	10^6
Brightness B (A/m^{2}sr)	**5×10^9**	5×10^{10}	**5×10^{12}**	10^{13}
Energy spread ΔE (eV)	2.3	1.5	0.6~0.8	0.3~0.7
Current stability (%/hr)	<1	<1	<1	5
Vacuum pressure (Pa)*	10^{-3}	10^{-5}	10^{-7}	10^{-8}
Vacuum temperature (K)	2800	1800	1800	300

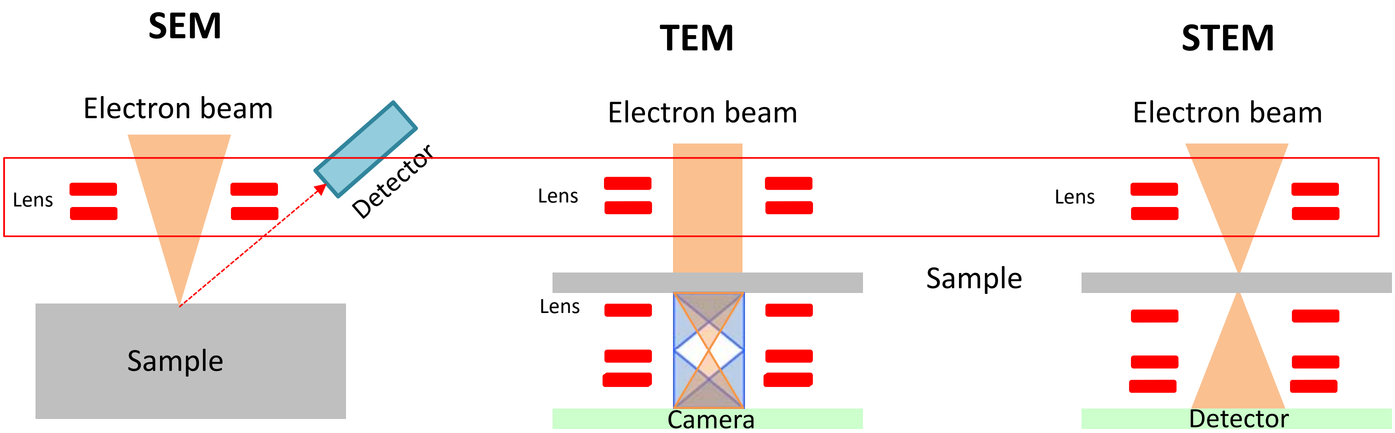
* Might be one order lower

Electron gun

W Advantages:	LaB_6 advantages:	FEG advantages:
Rugged and easy to handle	High brightness	Extremely high brightness
Requires only moderat vacuum	High total beam current	Long life time, more than 1000 h.
Good long time stability	Long life time (500-1000h)	
High total beam current		

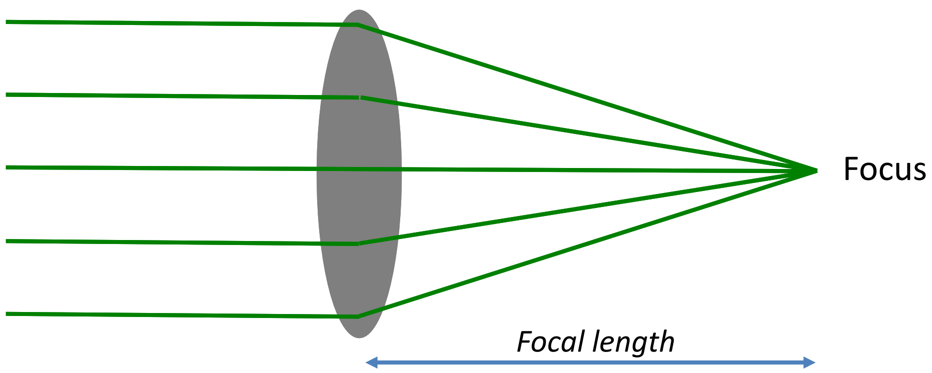
W disadvantages:	LaB_6 disadvantages:	FEG disadvantages:
Low brightness	Fragile and delicate to handle	Very fragile
Limited life time (100 h)	Requires better vacuum	Current instabilities
	Long time instabilities	Ultra high vacuum to remain stable

Components of an electron microscope – Lenses

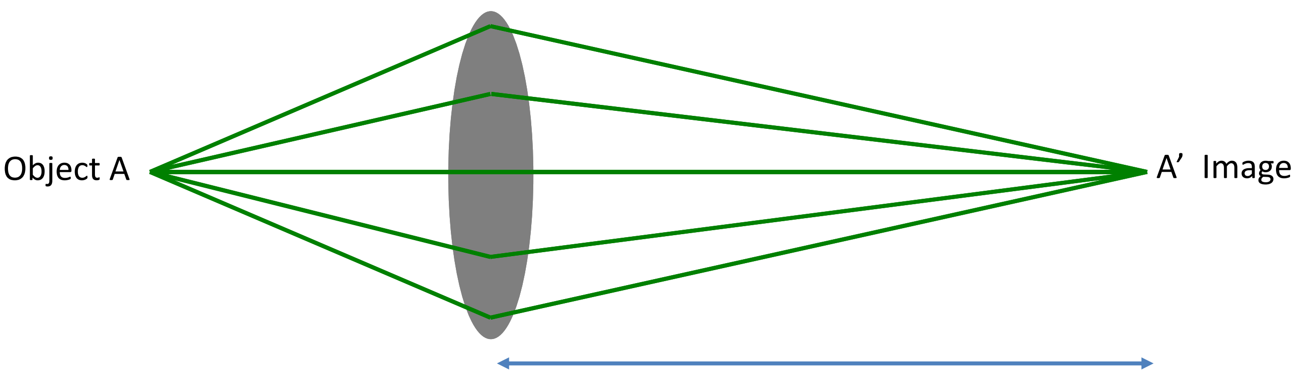


Properties of the lenses

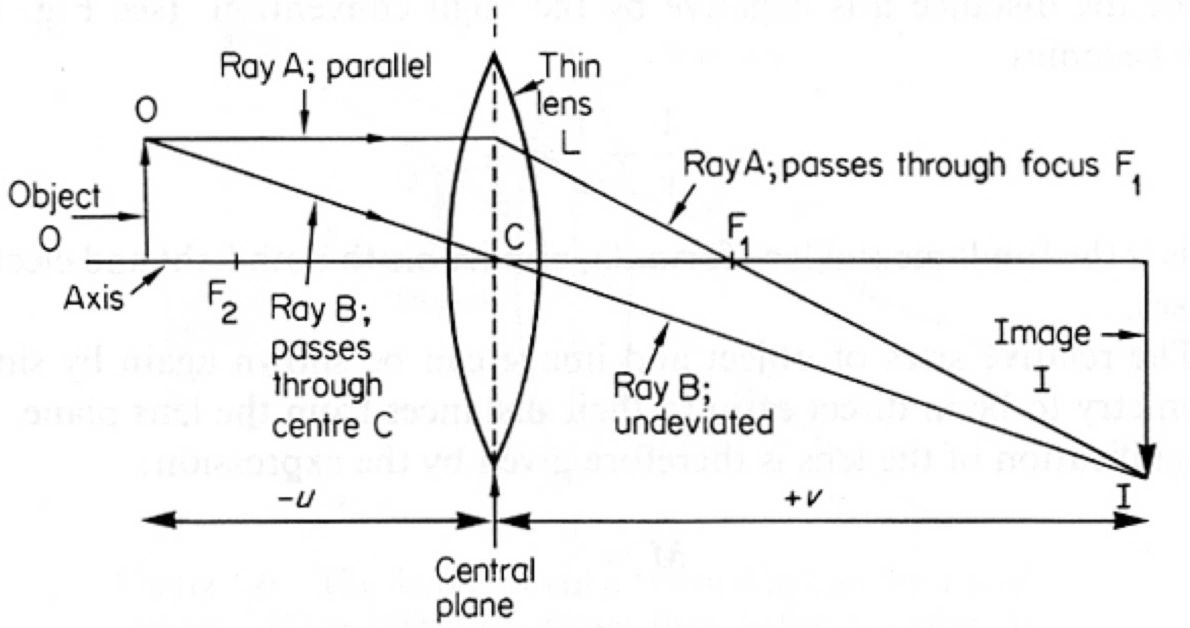
Lens bends beams to focus it to a point.



The rays emanating from a point in the object plane come to one common well defined point in image plane.

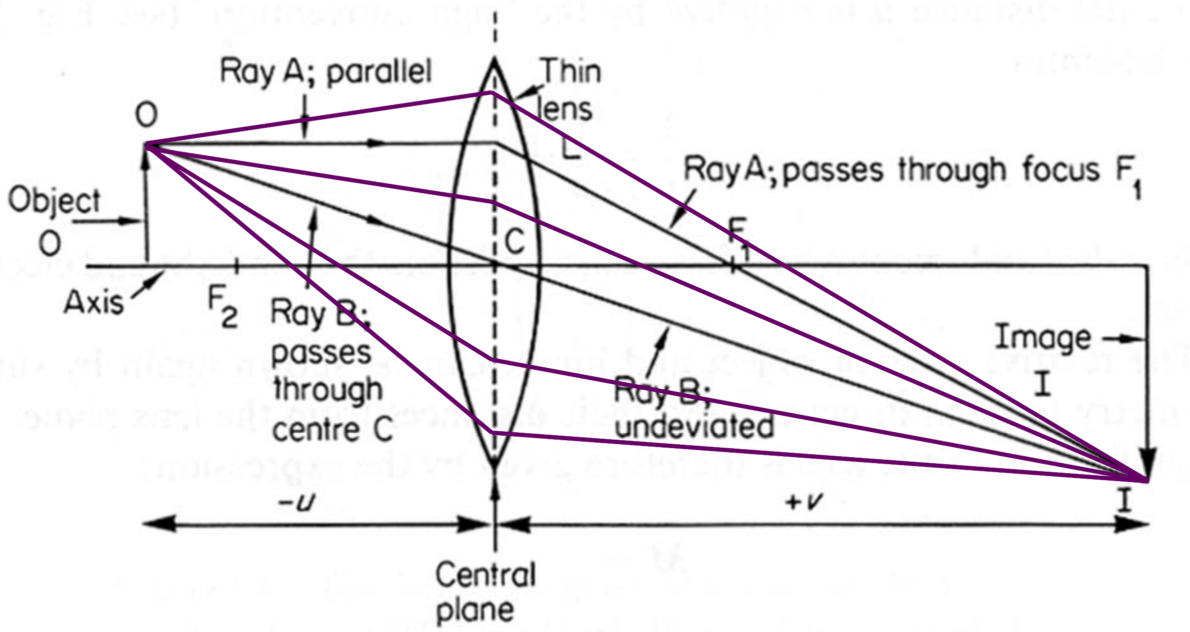


Lens produces a magnified (or de-magnified) image

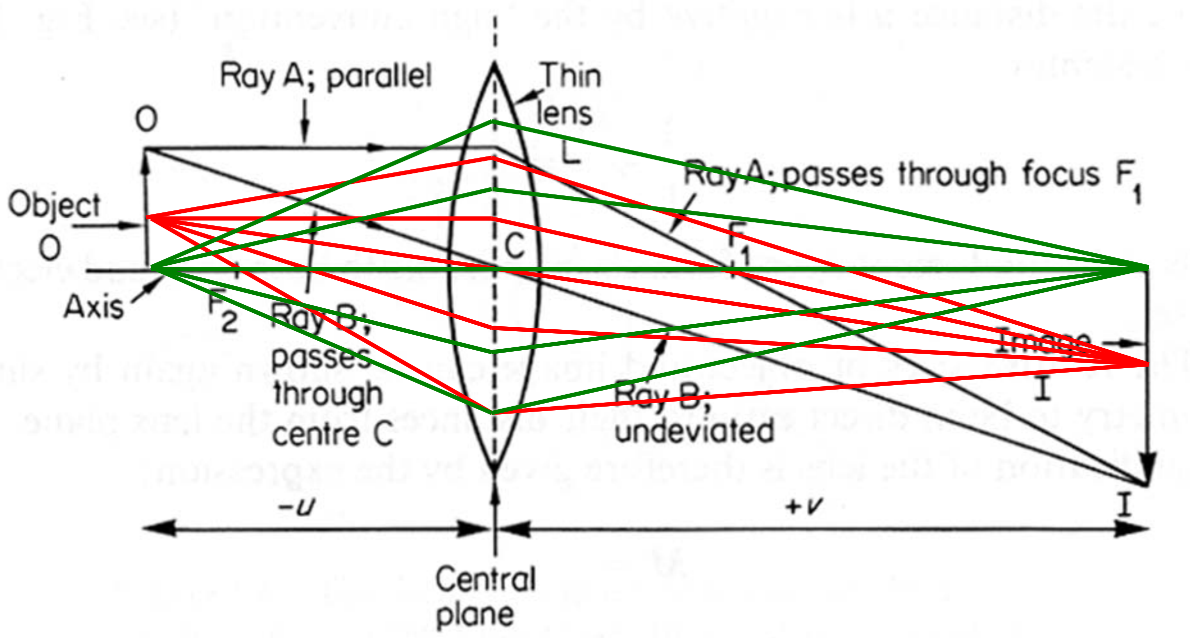


Electron optics - Lenses

Lens produces a magnified (or de-magnified) image



Lens produces a magnified (or de-magnified) image



Thin lens equation: $1/f = 1/u + 1/v$

Magnification = v/u

Light:

Glass or polymer lenses

Deflection of light through changing refraction index

Charged particles:

Lorentz Force!

Electrostatic lenses

Magnetic lenses

Particularity:

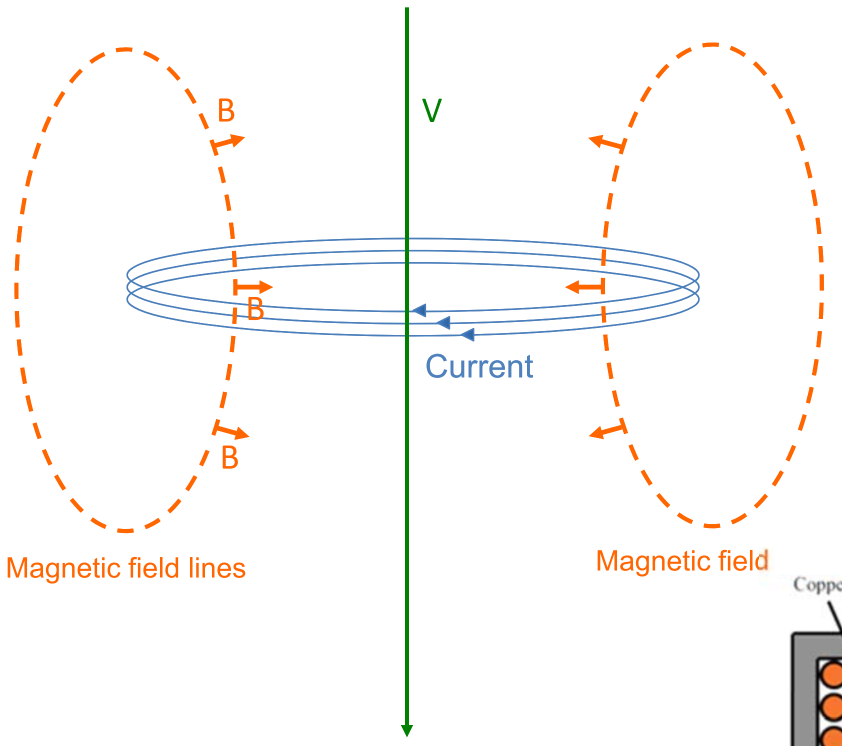
Variable focus

Tuneable correctors (astigmatism)

NOTE: Electron microscopes have more than one electromagnetic lens. Under this circumstance the image plane of the the **n_{th}** lens becomes the object plane of the **$(n+1)_{th}$** lens.

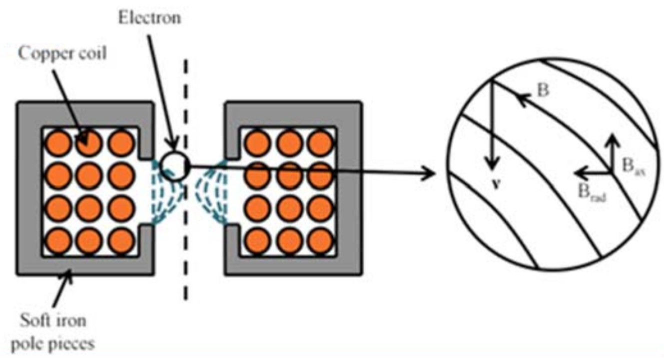
The total magnification is the product of the magnification of all the lenses.

Electron optics - Lenses

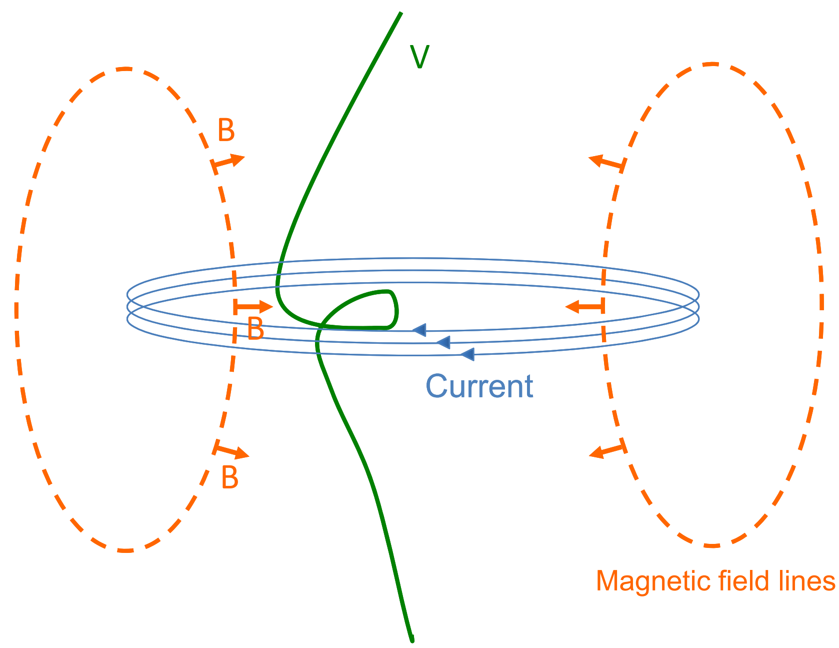


$$\vec{F}=q.(\vec{V} \times \vec{B})$$

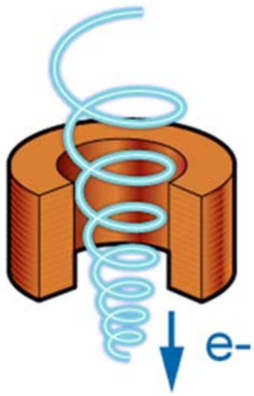
An electromagnetic lens consists of a coil of copper wires inside an iron pole piece. A current through the coils creates a magnetic field (symbolized by orange lines) in the bore of the pole pieces which is used to converge the electron beam.

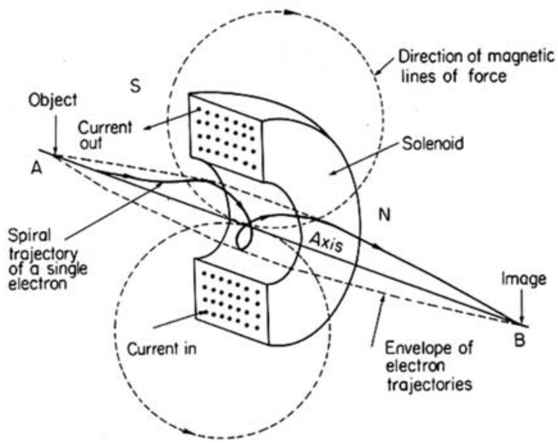


Electron optics - Lenses



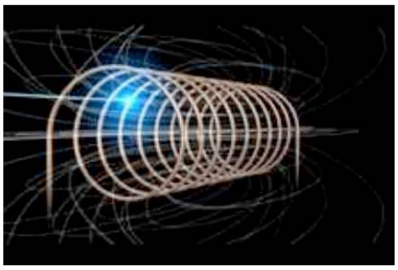
$$\vec{F}=q.(\vec{V} \times \vec{B})$$



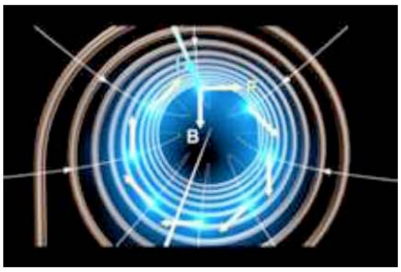


Electron optics - Lenses

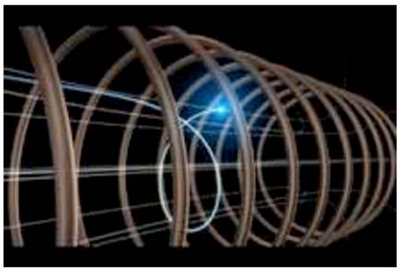
An electron enter inside a solenoïd



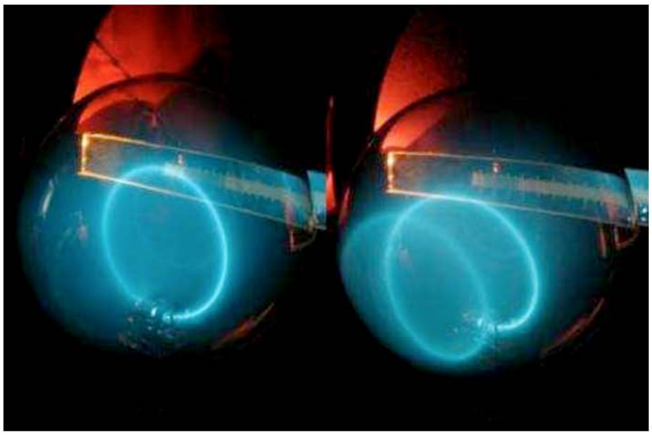
The Lorentz force is then applied

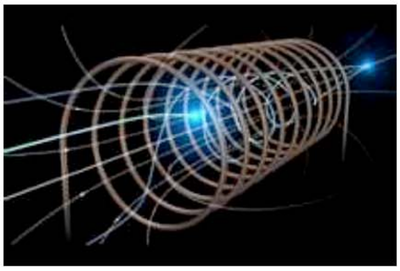


Due to the force, the trajectory is helical

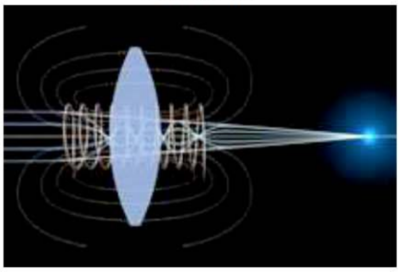


An example with angle (v,B)=90 and (v,B)=90



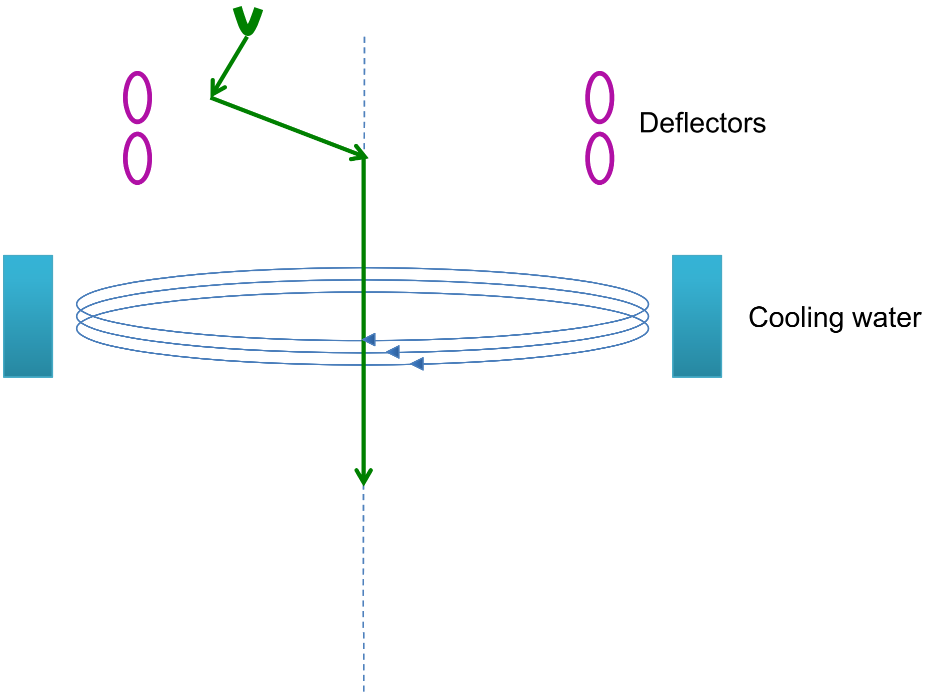


Due to the field shape, the electrons are focused !!!!

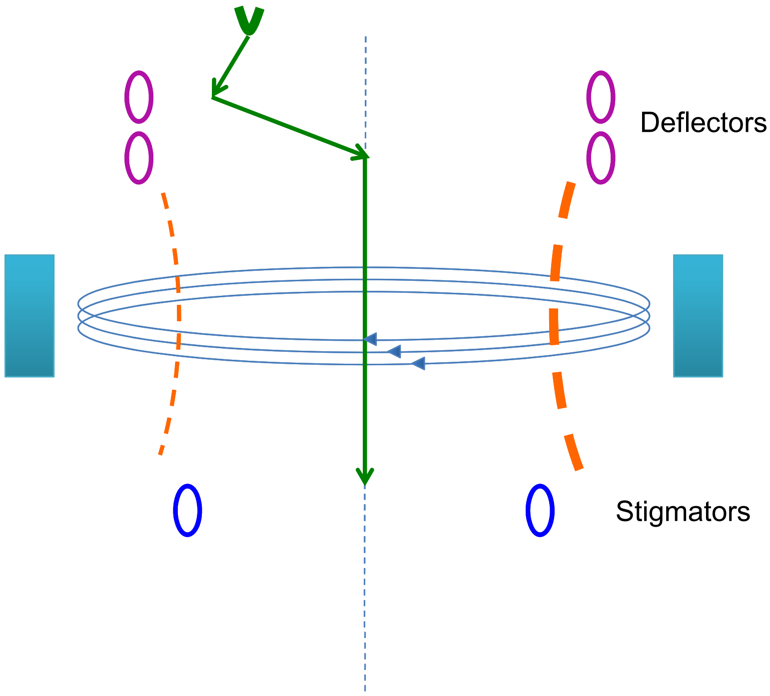


Slide from F. Houdellier, CNRS-France

Electron optics - Lenses

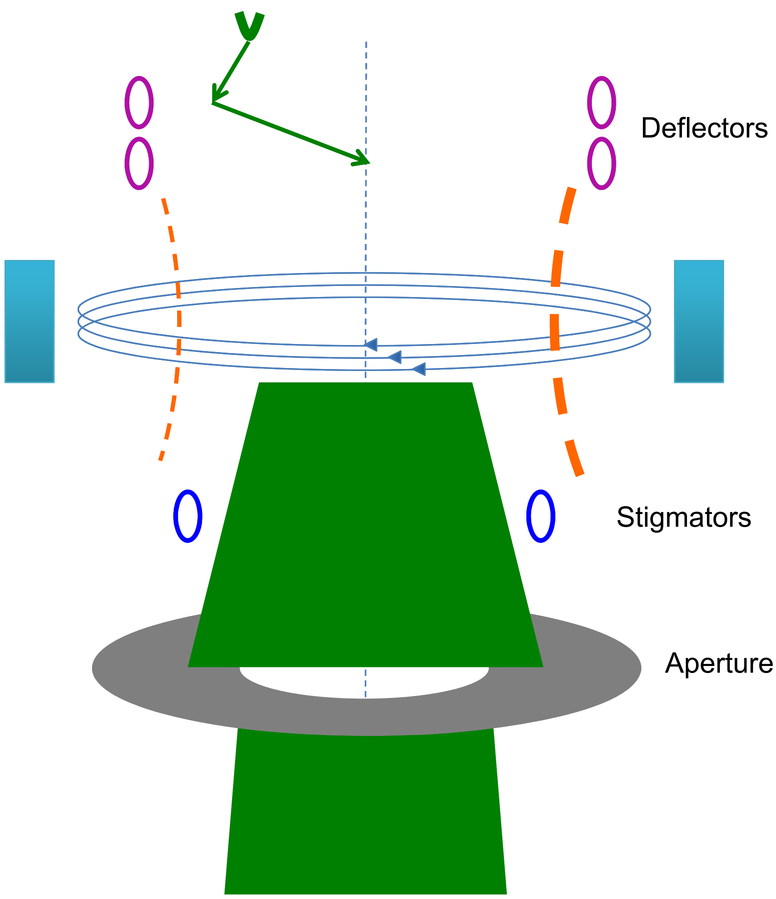


Electron optics - Lenses



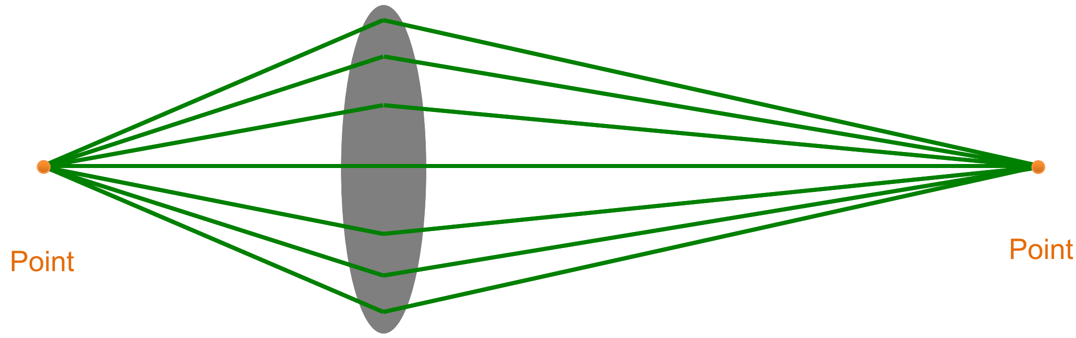
Local temperature changes
Non uniform wiring
Non-uniform current/Magnetic field

Electron optics - Lenses



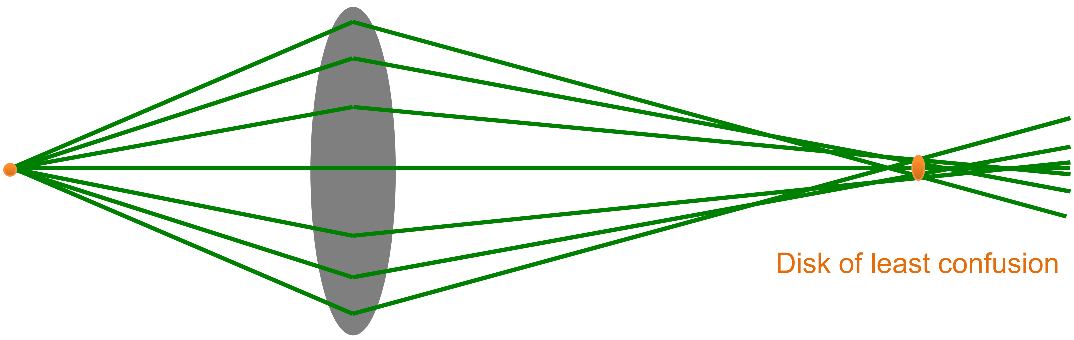
Electron optics - Aberrations

Ideal lens



A point source is focused to a point

Real life

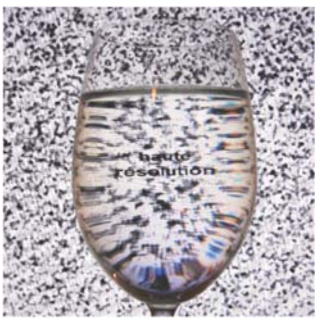


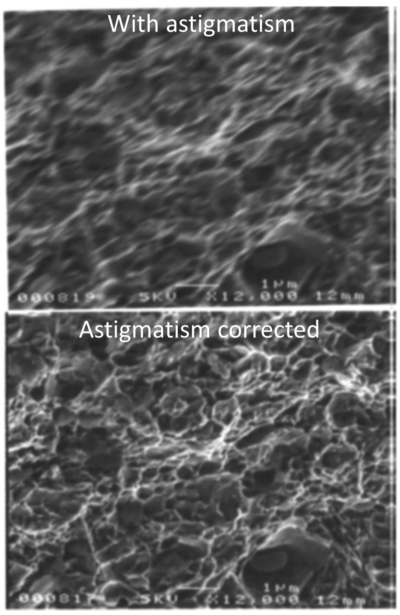
A point source is focused to a disk

Lens aberrations reduce resolution!

Electron optics - Aberrations

- **Lens aberrations**
  - Chromatic aberration
  - Spherical aberration
  - Astigmatism
  - Diffraction effect

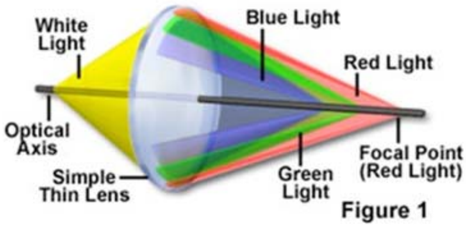


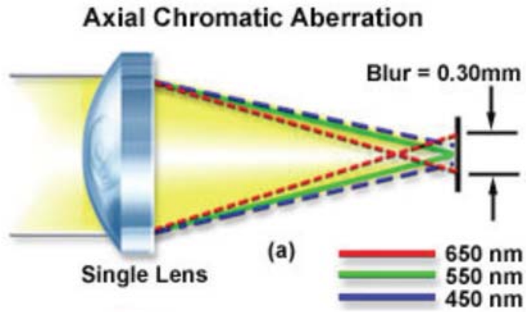


Lens aberrations are one of the main limitations to obtaining high spatial resolution.
BUT astigmatism can be easily corrected.

Electron optics: Chromatic aberrations

- Chromatic aberration is distortion that occurs when there is a failure of a lens to focus all colors (wavelengths) to the same convergence point.
- Electrons of lower energy will be bent more strongly.
- Correcting the aberration is necessary, otherwise the resulting image would be blurry and delocalized, a form of aberration where periodic structures appear to extend beyond their physical boundaries.
- Increases with source energy spread.
- Decreases with increasing electron energy (E_0)



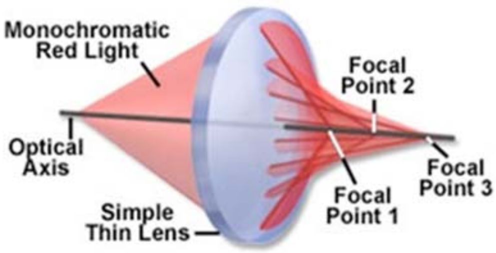


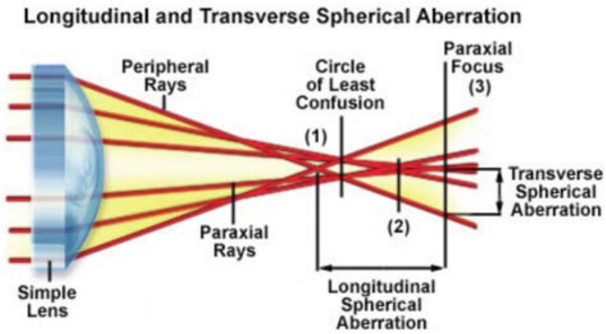
Critical for non-monochromatic beams (e.g. Thermionic and Shottky sources)

Electron optics: Spherical aberration

Core of the galaxy M100 ©NASA

- Spherical aberration occurs when parallel illumination rays that pass through the central region of the lens focus farther away than the illumination rays that pass through the edges of the lens.
- Result is multiple focal points and thus a blurred image.
- The diffraction and the spherical aberration limits on resolution have an opposite dependence on the angular aperture of the objective





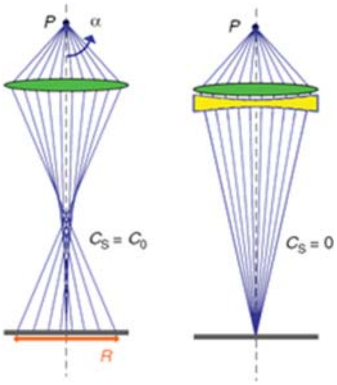
Focal length depends on the distance from optical axis

Image of the object is dispersed along the optical axis

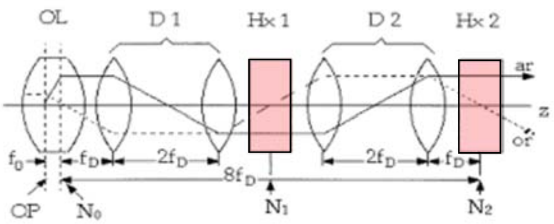
Circle of least confusion $d_s = \frac{1}{2} C_s \lambda^{3ar}$

Electron optics: Spherical aberration correctors

Light optics: Correction with combination of convex and concave lenses



Electron optics: Correction with hexapole or quadrupole and octopole lenses



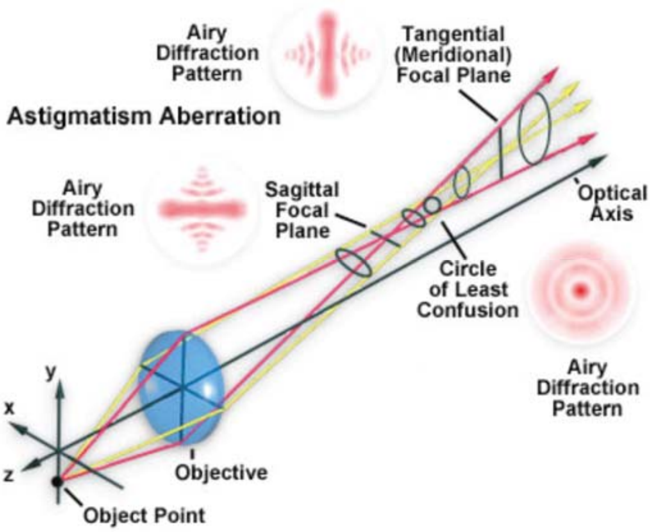
Cs-corrector

http://www.sfc.fr/Material/hrst.mit.edu/hrs/materials/public/ElecMicr.htm

Electron optics: Astigmatism

Focal length varies for different axes of the lens.

Image will appear "stretched" with changing the focus



Under focus image

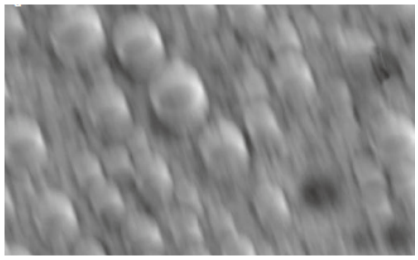
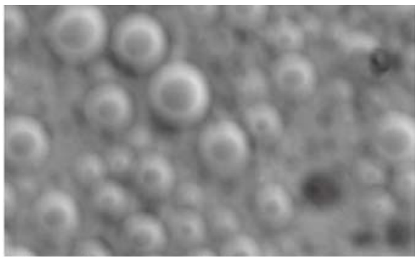
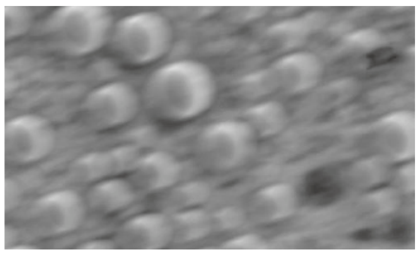


Image in focus



Over focus image

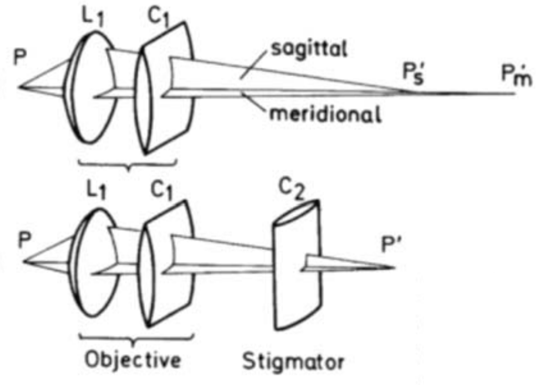


Caused by:

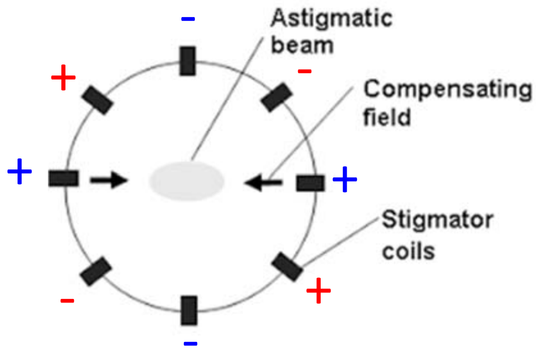
- imperfections in the manufacturing of the pole-piece and the copper windings
- Stray magnetic field

Electron optics: Astigmators

Light optics: correction with cylindrical lenses



Electron optics: Correction with quadrupole lenses
2 quadrupole lenses under 45 degree allow to control strength and direction of correction



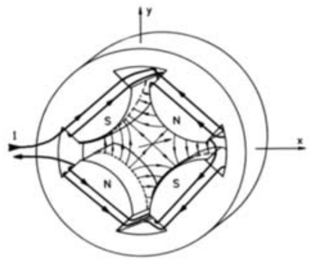
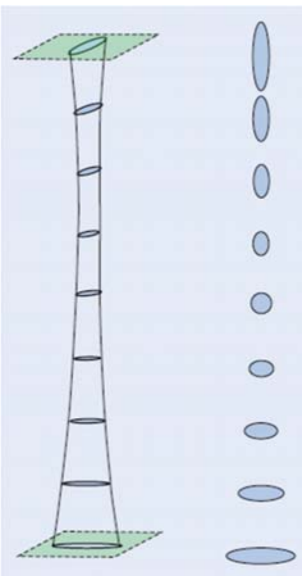
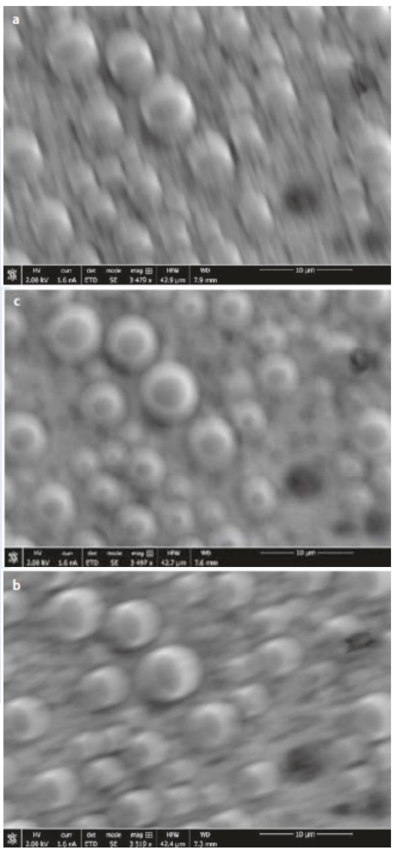


Fig. 2.12. Construction of a quadrupole lens

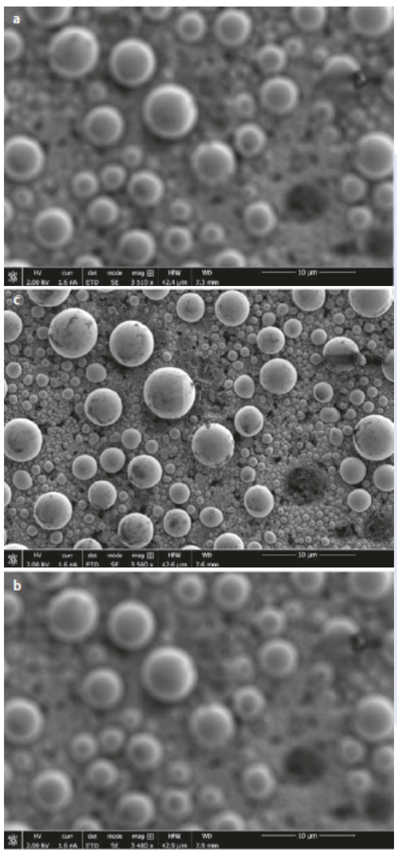
Electron optics: Astigmatism



With astigmatism

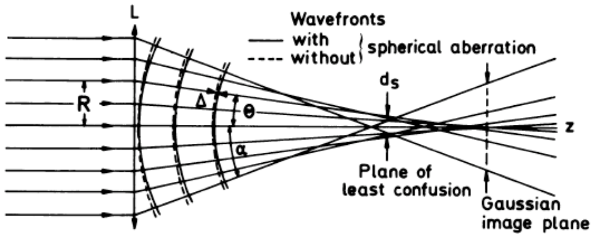


Astigmatism corrected

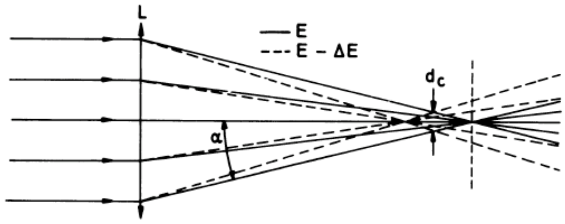


Electron optics: Aberrations

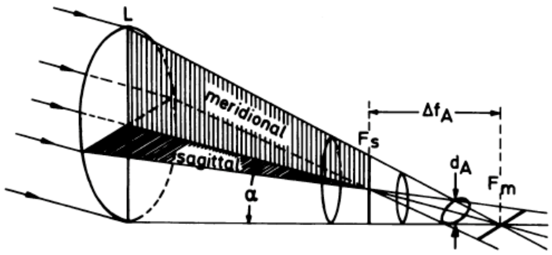
Spherical aberration

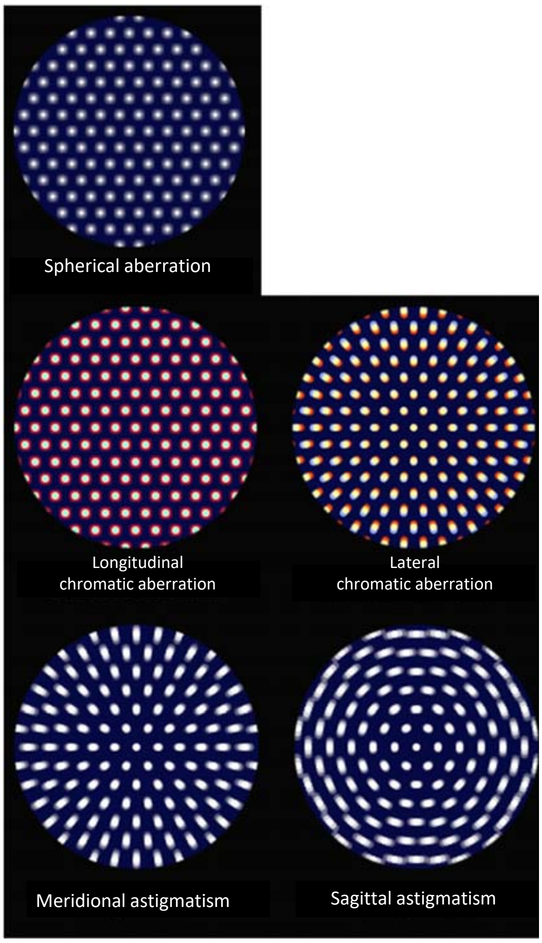


Chromatic aberration



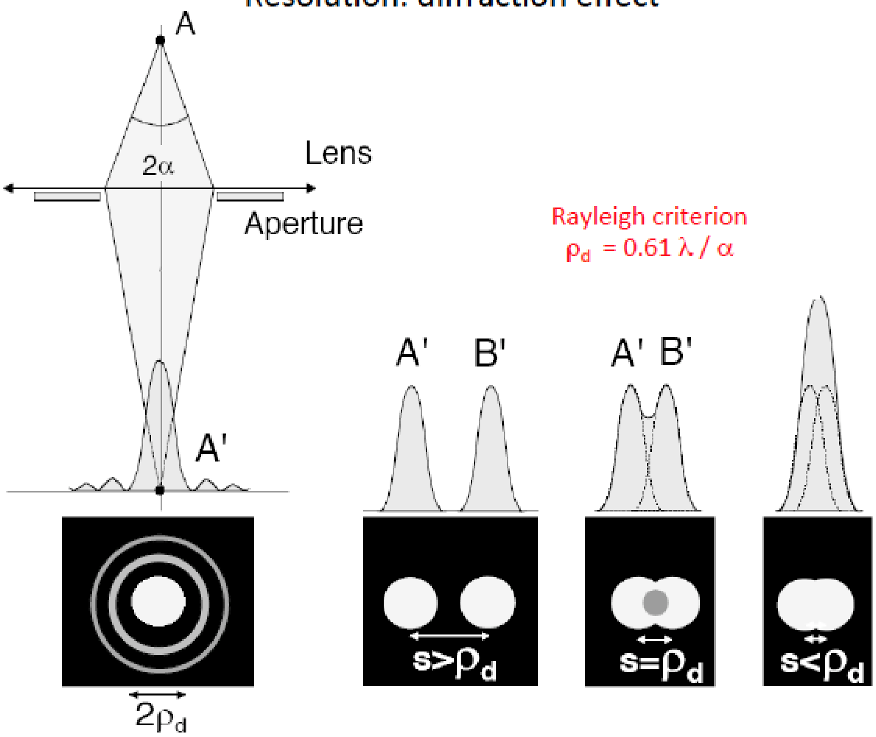
Astigmatism





Electron optics - Aberrations

Resolution: diffraction effect



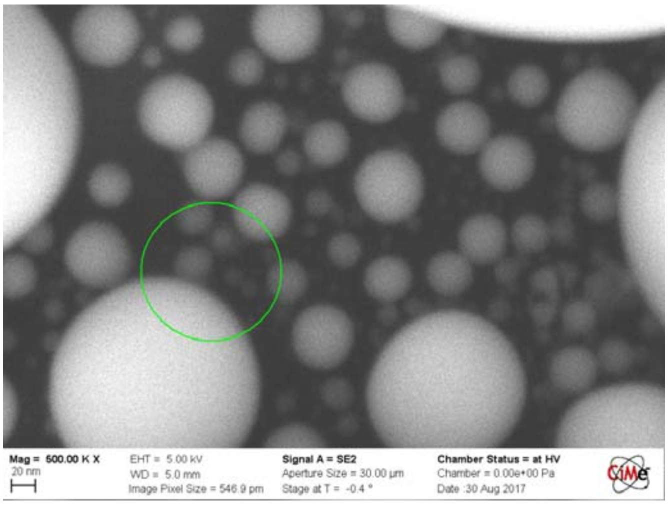
Rayleigh criterion

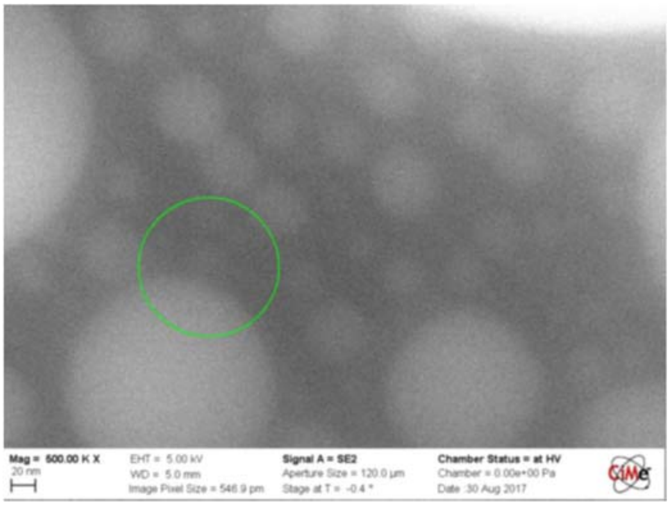
$\rho_d = 0.61\ \lambda / \alpha$

The diffraction and the spherical aberration limits on resolution have an opposite dependence on the angular aperture of the objective lens.

Electron optics - Aberrations

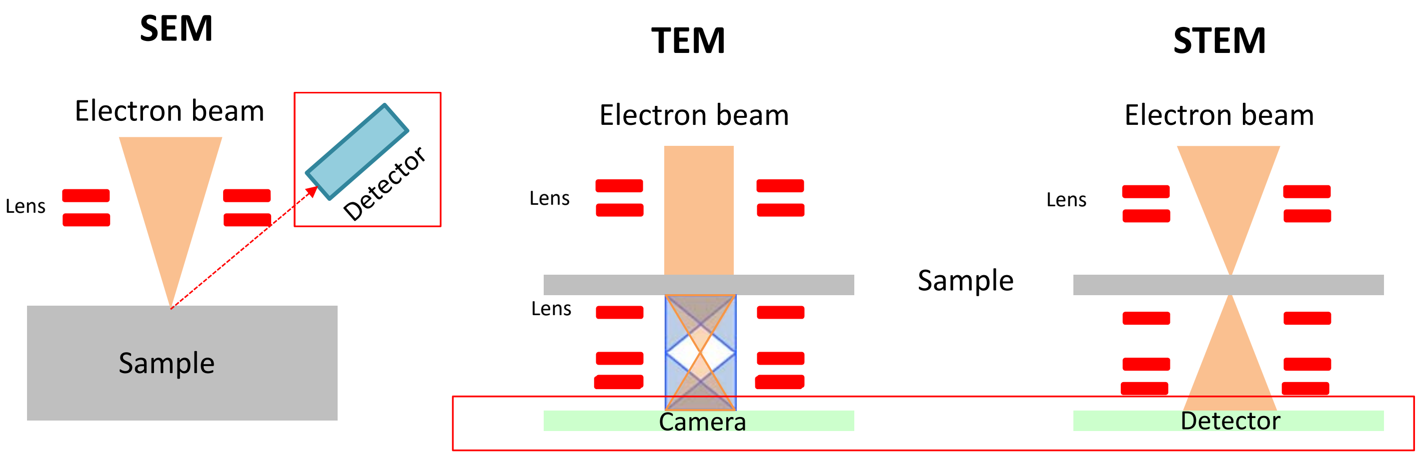
Optimal Aperture size (30µm) | Large Aperture size (120µm)





Though large apertures produce larger convergence angles (less diffraction effect), spherical aberration increases probe size and reduces resolution

Components of an electron microscope - Electron detection system



Photomultiplier
- Everhart-Thornley detector

Semiconductor BSE detector
- Silicon diode with a p-n junction

BSE Robinson detector
- Large scintillator/fiber optic

In-lens detectors

Phosphor screen

Films and image plates

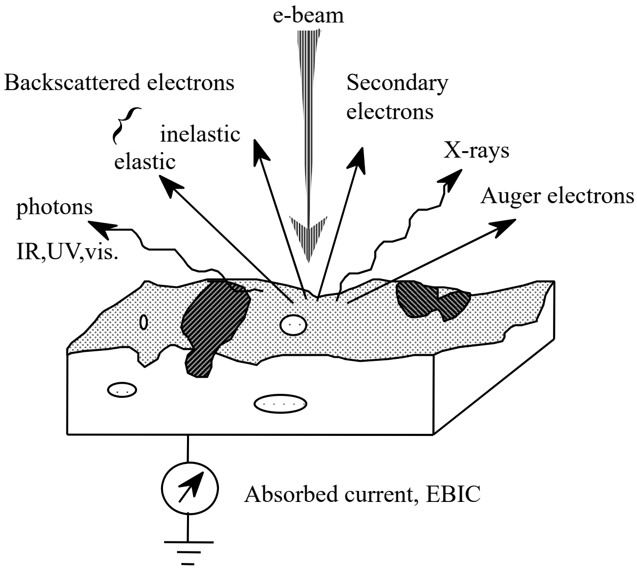
Cameras:
- semiconductor charge-coupled devices (CCD)
- complementary metal–oxide–semiconductor (CMOS)
- Direct electron

Energy filters and spectrometers + CCD

Disk/ring shape detectors

Pixelated detectors

SEM signals



Electron signals:

Secondary electrons SE
Electrons ejected from material at low energies
Topography, low energy ≈ 5-50 eV

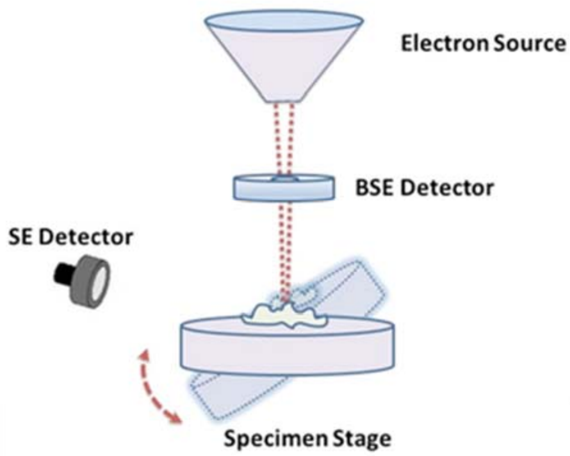
Backscattered electrons BSE
Incident electrons that elastically scatter and leave the sample
Atomic number Z
Energy ≈ eV_0 (range from 50 eV to an energy close to initial energy)

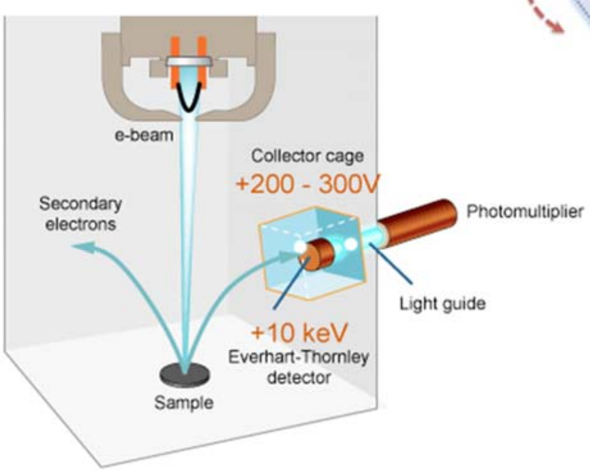
Auger electrons
Ejected electrons with an energy characteristic of target elements
Not detected in conventional SEM, surface analysis

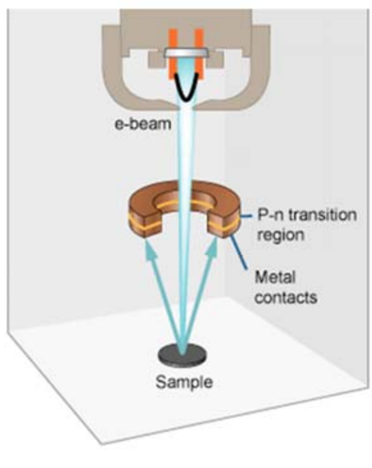
Other signals:

Electromagnetic radiations etc.
Phonos: visible, UV, IR
Absorbed current, electron-holes pairs creation, EBIC
Plasmons
Sample heating (phonons)

Electron detectors



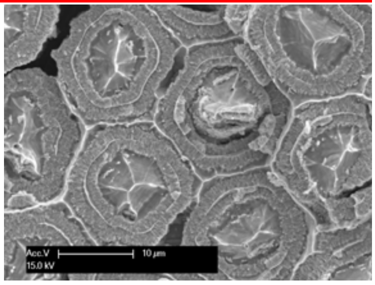


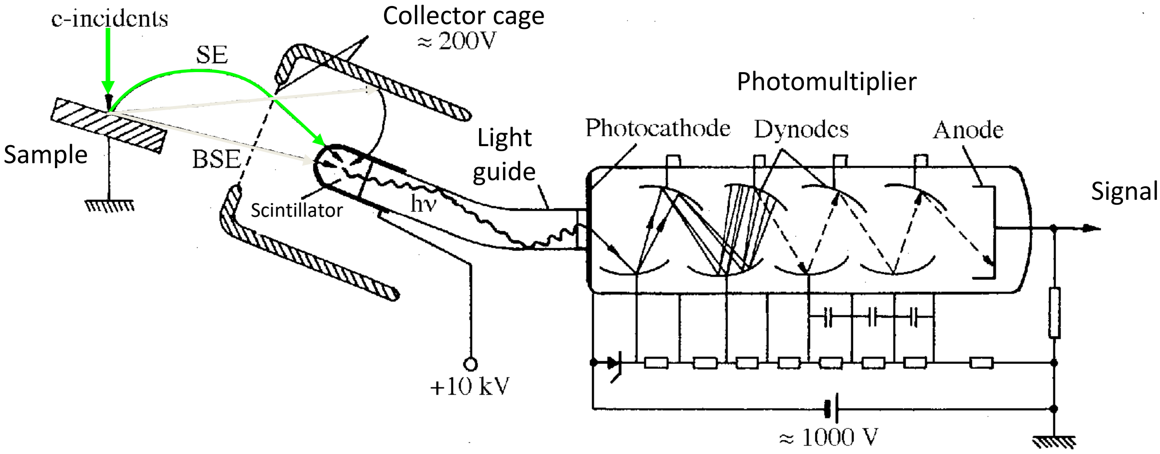


https://myscope.training/legacy/sem/background/whatissem/detectors.php

Detection of low energy electrons

- Photomultiplier
- Everhart-Thornley detector

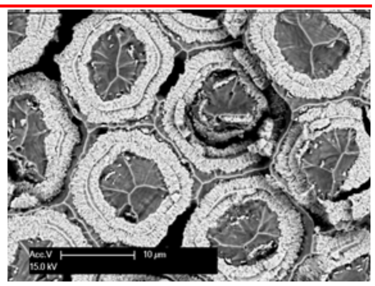


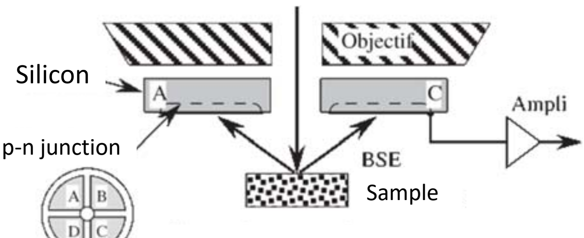


Collects and detects low energy (<100eV) electrons: SEM secondary electrons

Detection of high energy electrons

- Semiconductor detector





Semiconductor detector

BSE semiconductor detector: a silicon diode with a p-n junction close to its surface collects the BSE (3.8eV/ehole pair)

- large collection angle
- slow (poor at TV frequency)
- some diodes are split in 2 or 4 quadrants to bring spatial BSE distribution information

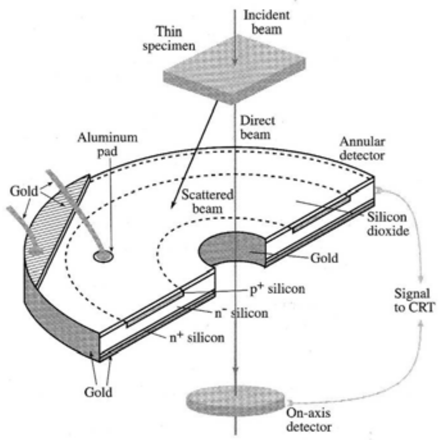


Figure 7.1. Semiconductor detector of the surface-barrier type, shown in a configuration where it would be used to detect high-energy forward-scattered electrons. The direct beam is detected by a small circular detector on the optic axis surrounded by a concentric wide-angle annular detector, which detects any scattered electrons.

Detects higher energy (>5kV) electrons: SEM backscattered electrons