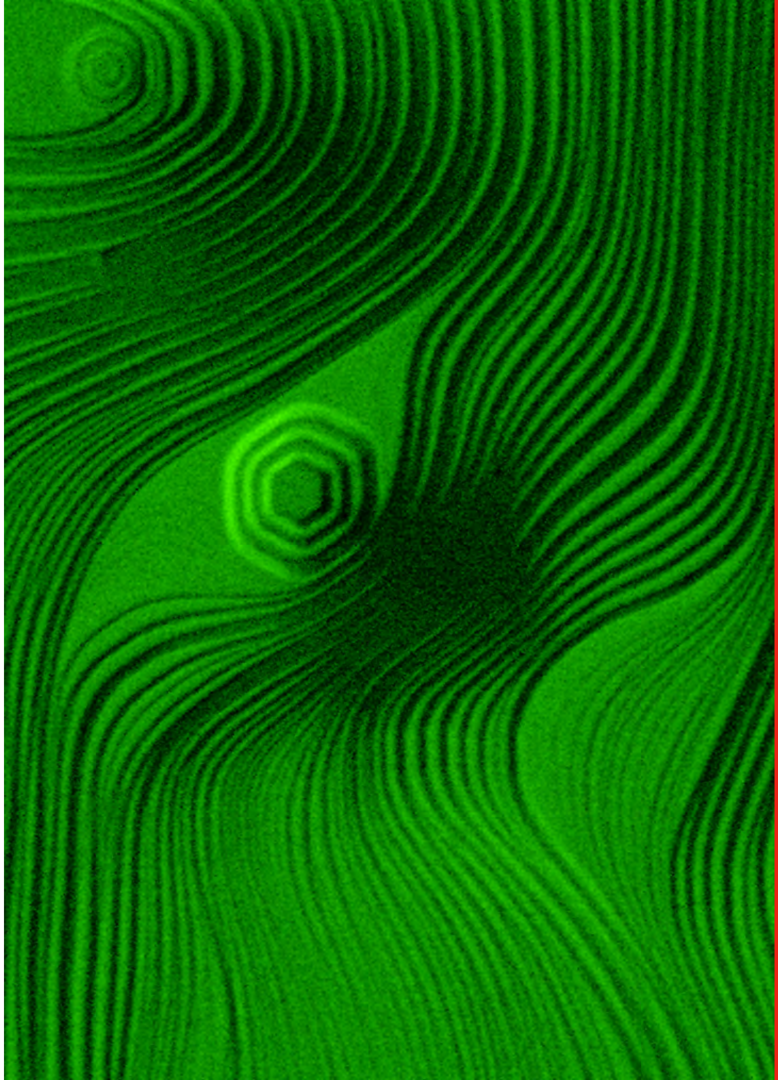
EPFL



Emad Oveisi

Scanning Electron Microscopy Techniques

2019-2020

Outline

- **Introduction to electron microscopy** (by E. Oveisi)
  - **Electron matter interaction**
- **SEM setup**
  - **Electron sources**
  - **Lenses**
  - **Vacuum system**
  - **Detection system**
- **Imagining with SEM**
  - **Operation, Signals**
  - **Contrast mechanism**
  - **Interpretation of images, Challenges**
  - **Related techniques** (By M. Cantoni)
  - **Advanced and high-resolution SEM**
- **Chemical analysis and Monte Carlo simulations**
- **Focused ion beam**

Why use electrons?

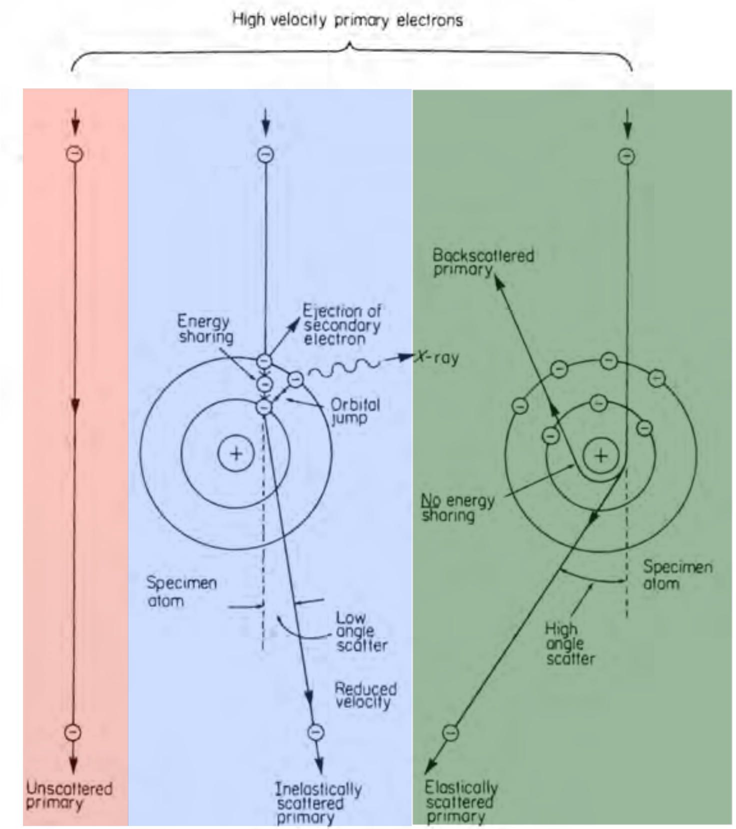
	Advantageous	Disadvantageous
Visible light	Not very damaging Easily focused Eye detector	Long wavelengths (400 nm)
X-rays	Small wavelength (Angstrom) Good penetration	Hard to focus Damage sample
Neutrons	Low sample damage Small wavelength (pm)	How to produce? How to focus?
Electrons	Small wavelength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

High energy electrons have a short wavelength
Easy to produce high brightness electron beams
Easy to manipulate : focused
Interact strongly with matter

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

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

e- matter interactions

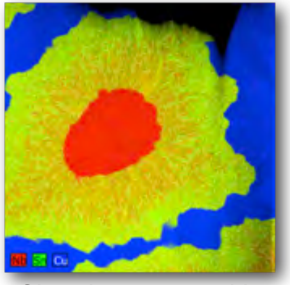


Inelastic events: The result is a **transfer of beam energy** to the specimen atom (**energy loss**) and a potential expulsion of an electron from that atom as a **secondary electron (SE)**.

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.

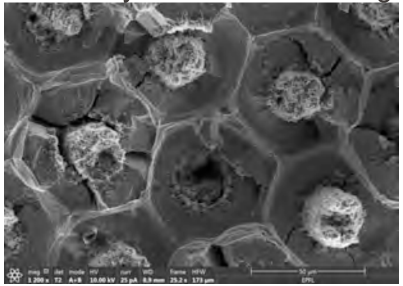
That's why TEMs are shielded!

Characteristic X-rays



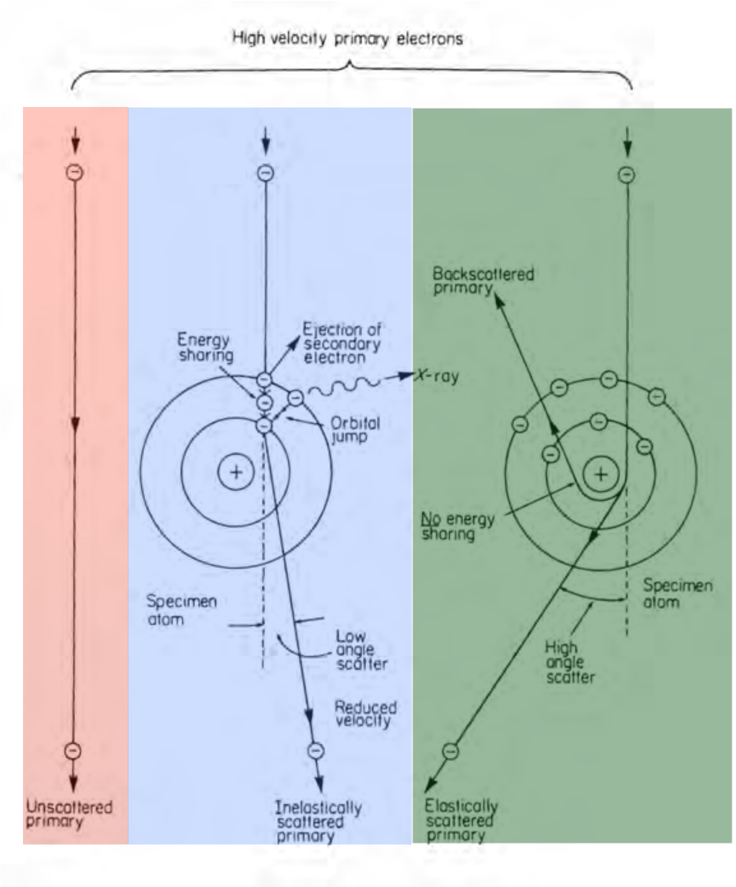
Chemical composition

Secondary electron SEM image



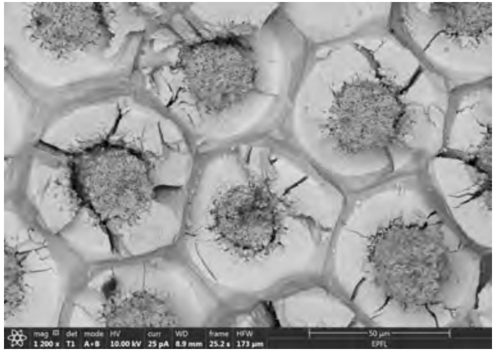
Topography

e- matter interactions



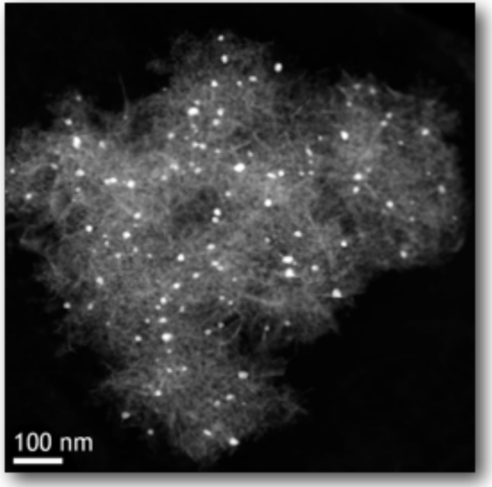
Elastic events occur when a beam electron interacts with the electric field of the nucleus of a specimen atom, resulting in a change in the direction of the beam electron **without a significant change in the energy** of the beam electron (< 1 eV).

Heavier atom => More backscattering



Mass contrast
Back-scattered SEM image

Larger nucleus => More scattering



Pt catalyst particles on Al_2O_3

Mass/thickness contrast
HAADF-STEM image

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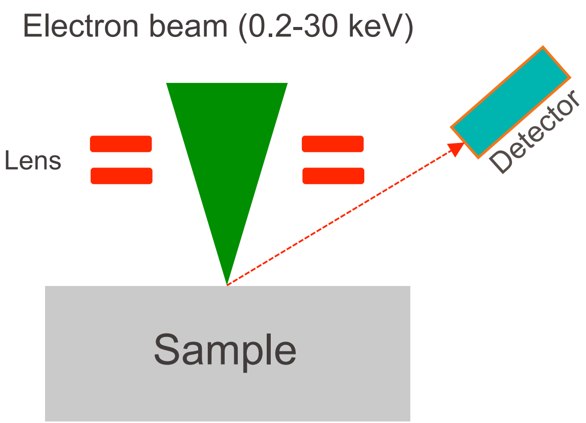
Types of electron microscopes

Scanning Electron Microscope (SEM)



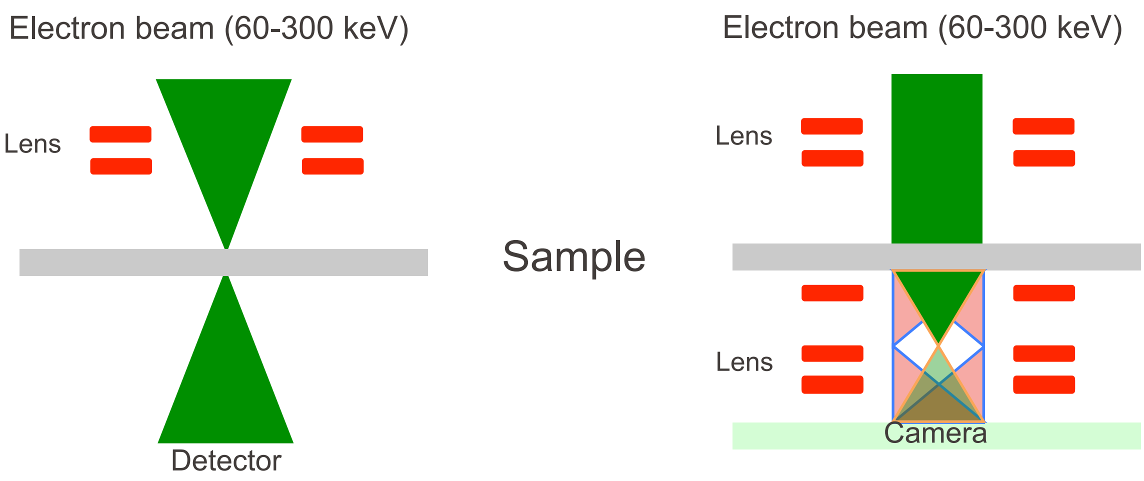
Signals:
Secondary electrons,
Back-scattered electrons, X-ray, Auger, …

Salt grains

Types of electron microscopes

Transmission Electron Microscope (TEM)

- Scanning mode (STEM)
- Conventional mode (CTEM or TEM)

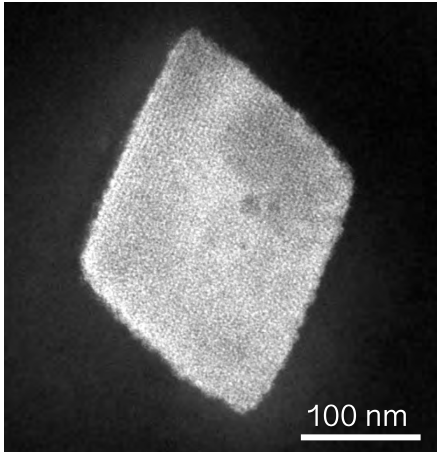


Types of electron microscopes

- SEM

Salt grains

- TEM



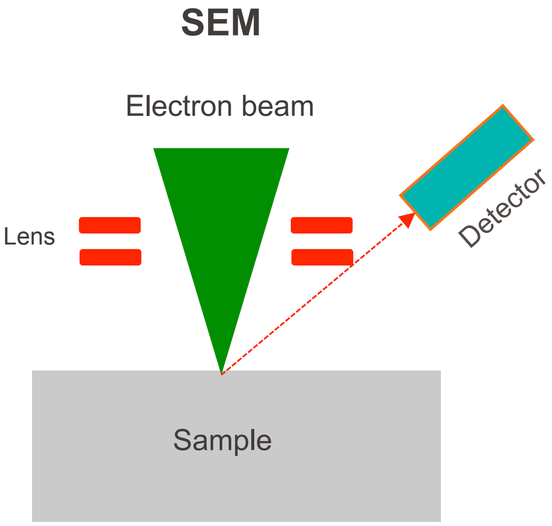
Cu-based metal-organic framework

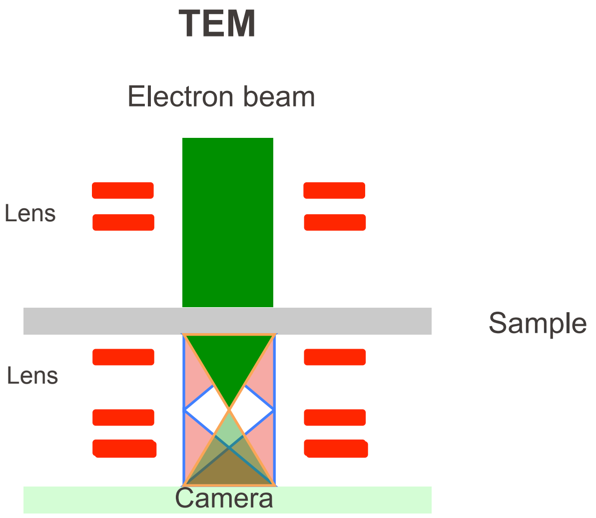
Types of electron microscopes

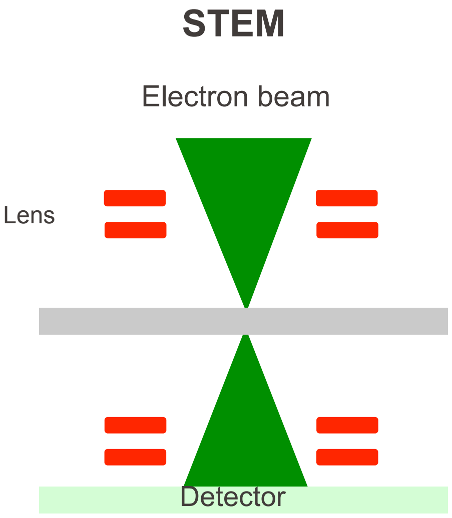
- SEM

- TEM

Components of an EM

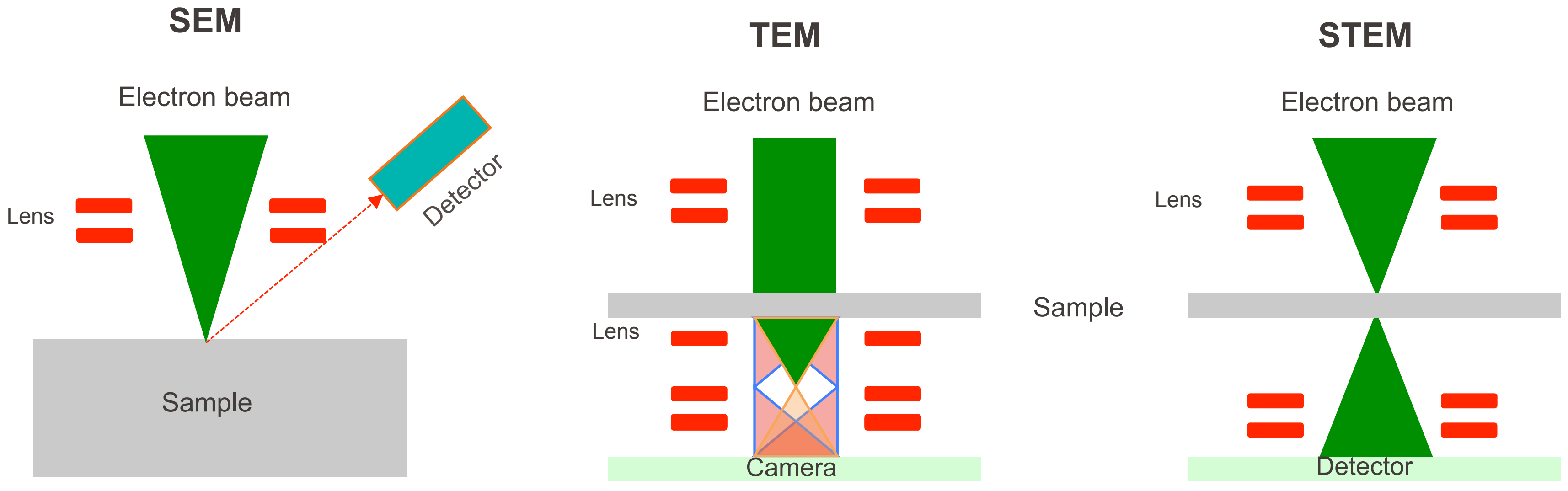






Vacuum system
Electron gun
Lenses
Detectors

Components of an EM – Vacuum system



Why we need vacuum?

1. Electron propagation is only possible through vacuum (e^- interacts heavily with the matter)
2. Need a good vacuum system to reduce contamination and surface modification

Vacuum system

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

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

Vacuum cleaner!

Rotary vane pump

Oil diffusion pump

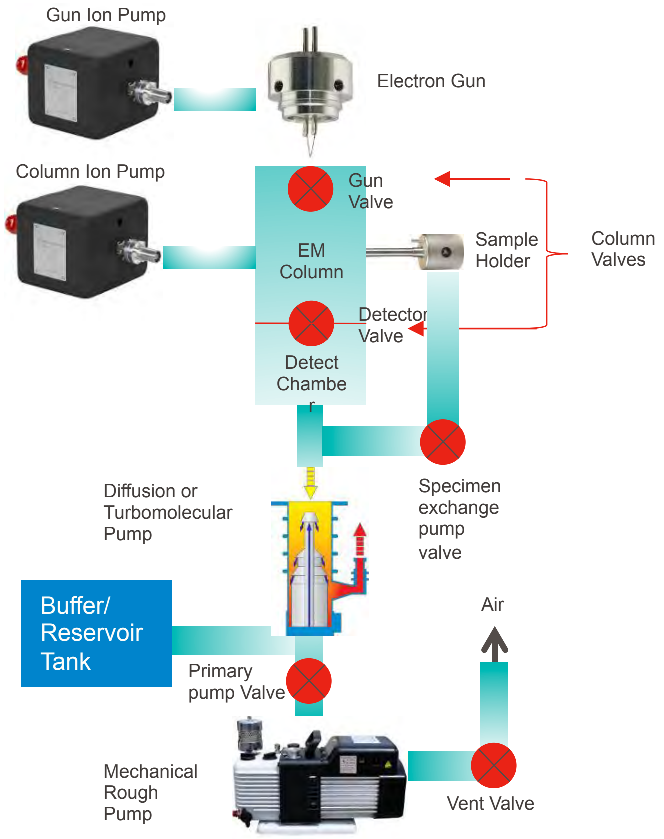
Turbomolecular pump

Ion getter pump

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

760 Torr = 1 Atmosphere = 1.013 Bar = 101.325 KPa

Vacuum system



UHV is not needed in all parts of the microscope

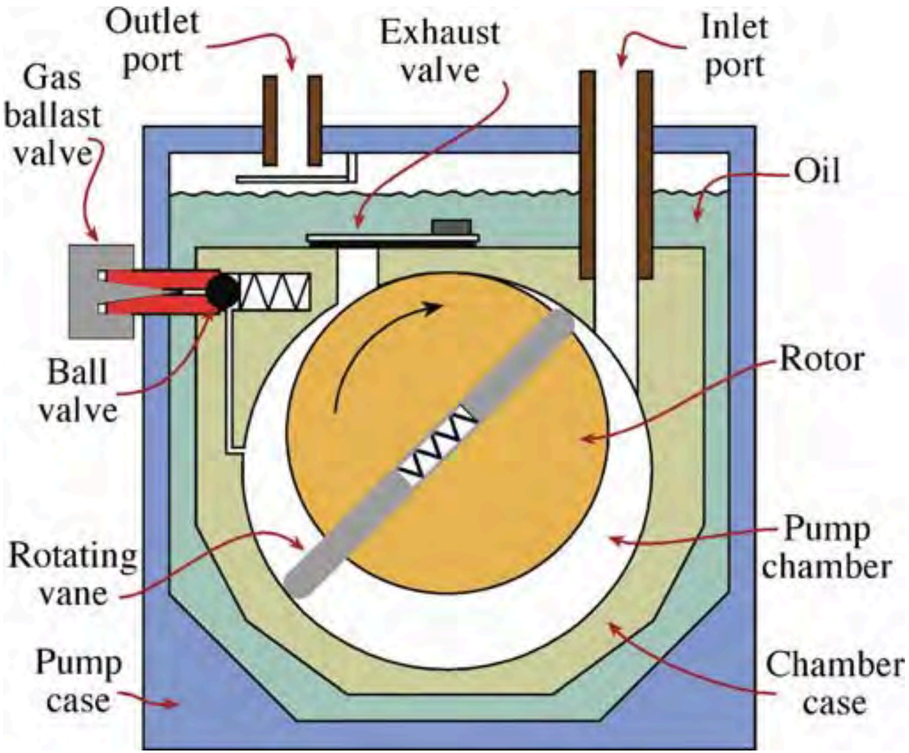
- **Primary vacuum (>0.1 Pa)**
  - Mechanical pump
- **Secondary to high vacuum**
  Detector or viewing chamber (<10^{-4} Pa)
  - Oil diffusion pump
  - Turbomolecular pump
- **High and ultra-high vacuum**
  Gun & specimen area (<10^{-6} Pa)
  - Ion getter pump
  - Cold trap

Vaccum level in space: 1 Pa at 100km above earth
760 Torr = 1 Atmosphere = 1.013 Bar = 101.325 KPa

Vacuum system

Rotary (mechanical) vane pump

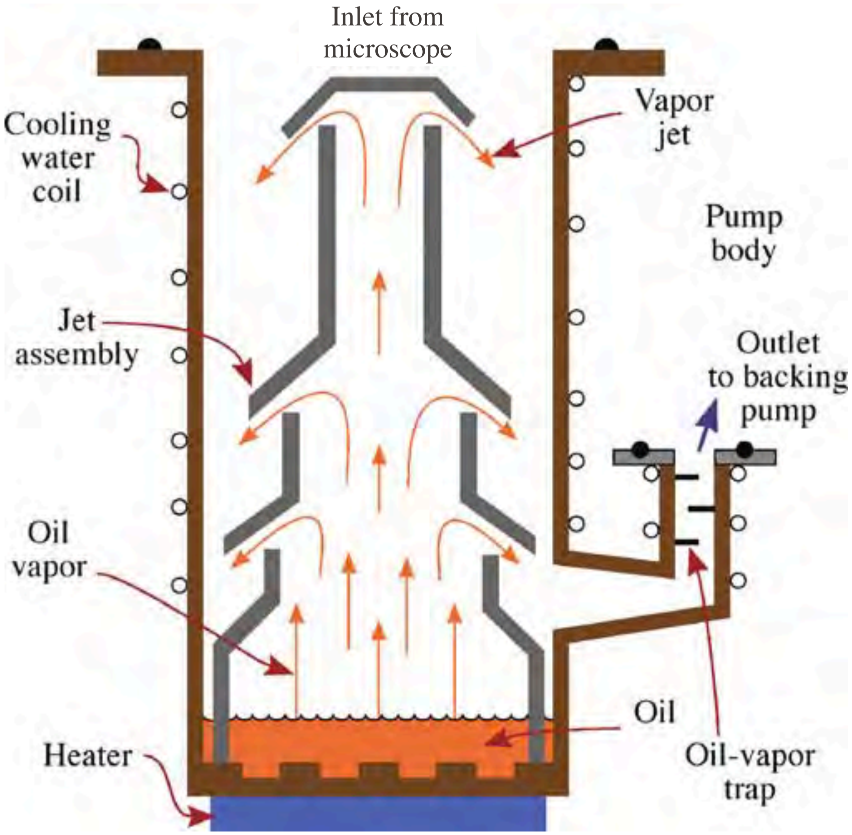
- Uses oil
- Noisy



Vacuum system

Oil diffusion pump

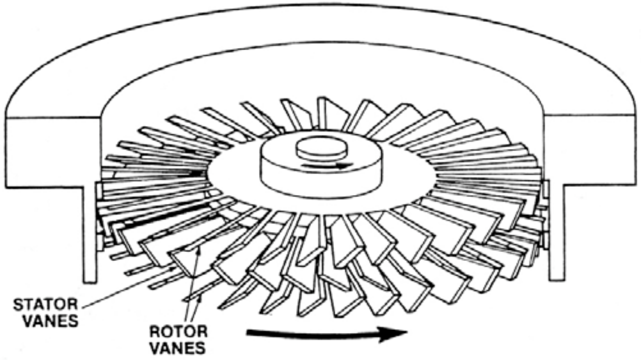
- Vibration free
- Contamination possible oil vapor
- High pumping capacity



Vacuum system

Turbomolecular pump

- Uses a turbine to force gases from the microscope
- Rotation speed 20’000-50’000 rpm
- Magnetic bearings
- Pumping volumes 50-500 l/s
- Can start (slowly) at ambient pressures, increasing speed as the pressure is lowered
- Ultimately providing UHV conditions at high enough speeds



EPFL

Vacuum system

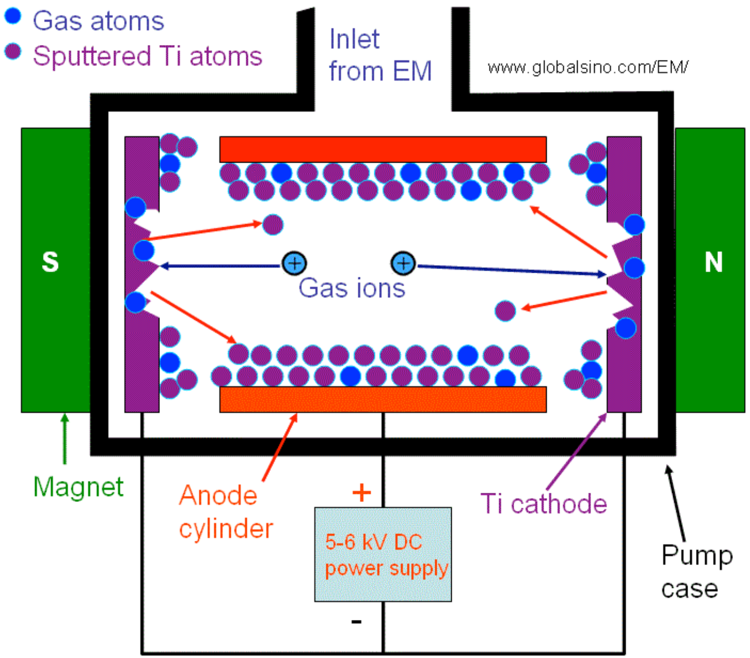
Ion getter pump

- No vibrations
- No exit = trapping: improves vacuum !

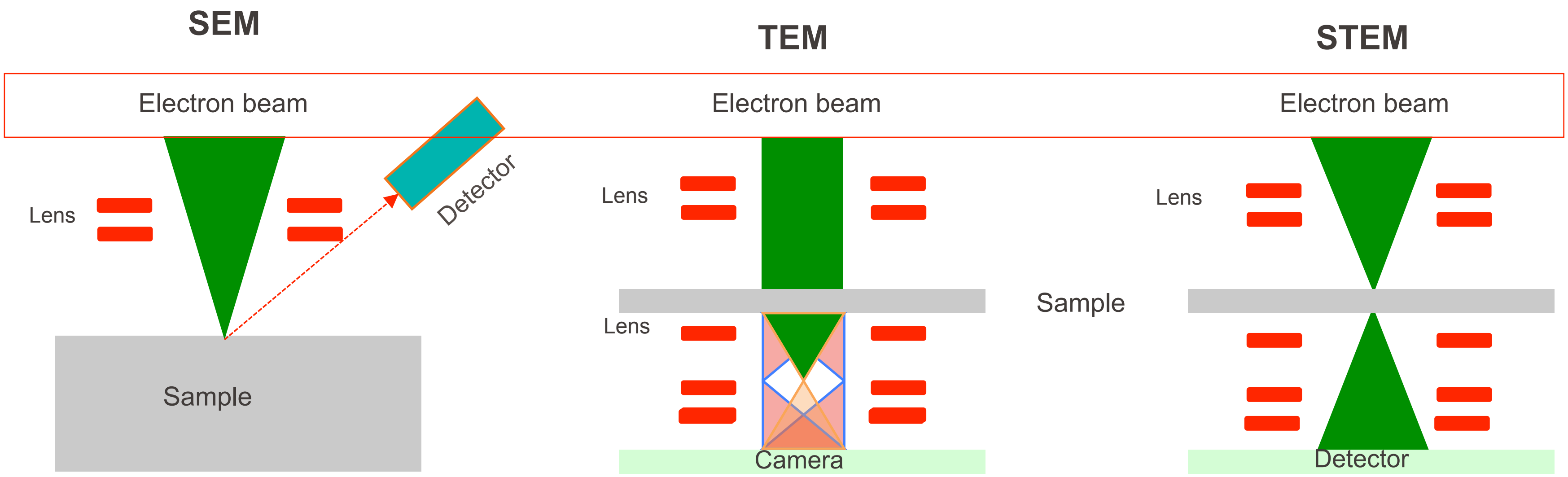
The ion pump emits electrons from a cathode. These ions spiral in a magnetic field and ionize air molecules, which are then attracted to the cathode. The energetic gas ions sputter Ti atoms from the cathode and they condense throughout the pump chamber, mainly on the cylindrical anode, trapping gas atoms.

Thus ion pumps remove gas atoms in two ways;

- by chemisorption on the anode surfaces
- by electrical attraction to the cathodes.



Components of an EM



Vacuum system
Electron gun
Lenses
Detectors

Electron gun

- Purpose: To create a narrow intense beam of electrons

- 3 types of electron guns:
  - Thermionic (Thermal)
    Heat only
  - Cold field emission
    Electric field: Potential (voltage) difference
  - Heat assisted field emission: Schottky emitter
    Heat + Electric field

Thermionic gun

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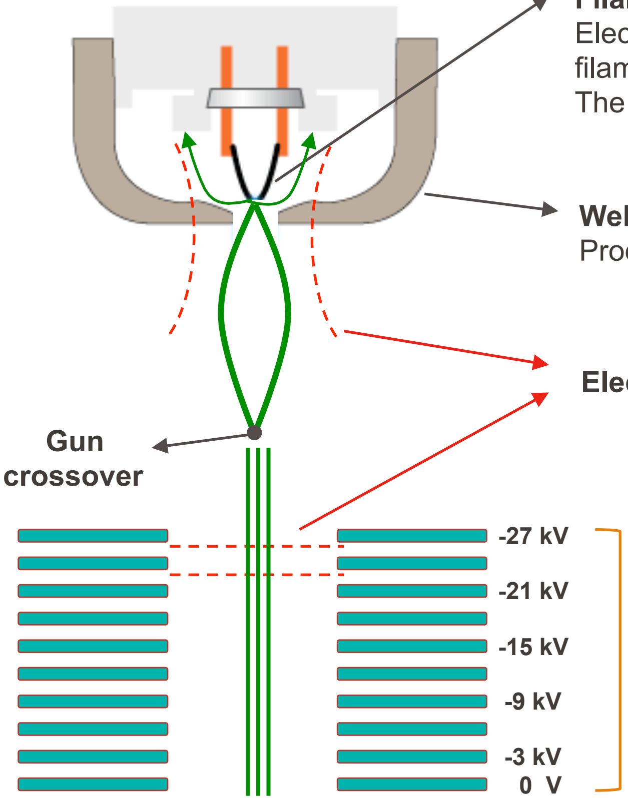


Filament (cathode) @ V_0
Electrons receive enough energy to escape the filament (work function of the filament)
The filament is hold at a negative high voltage e.g. – 30 kV

Wehnelt cup and anode @ $V_0 + V_{bias}$
Produces a small negative bias (e.g. -31 kV) to focus electrons to crossover

Electrostatic field

Accelerator

To give the electrons some speed

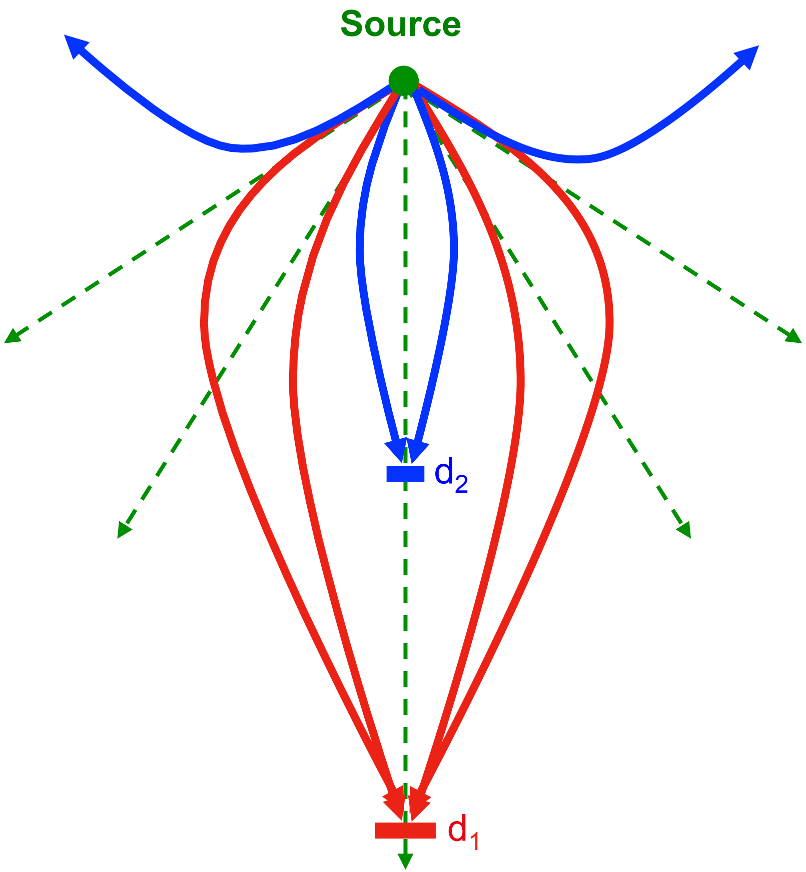
Stacks are isolated by SF_4 gas → Heavier than air

Thermionic gun

- Filament as a light bulb : W wire or LaB_6 crystal
  - 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
  - Must heat slowly otherwise burn out the filament or crystal damage

- Heating current (filament current) is NOT beam current!

- Saturation point of the filament
  - Optimized electron output | Filament life time



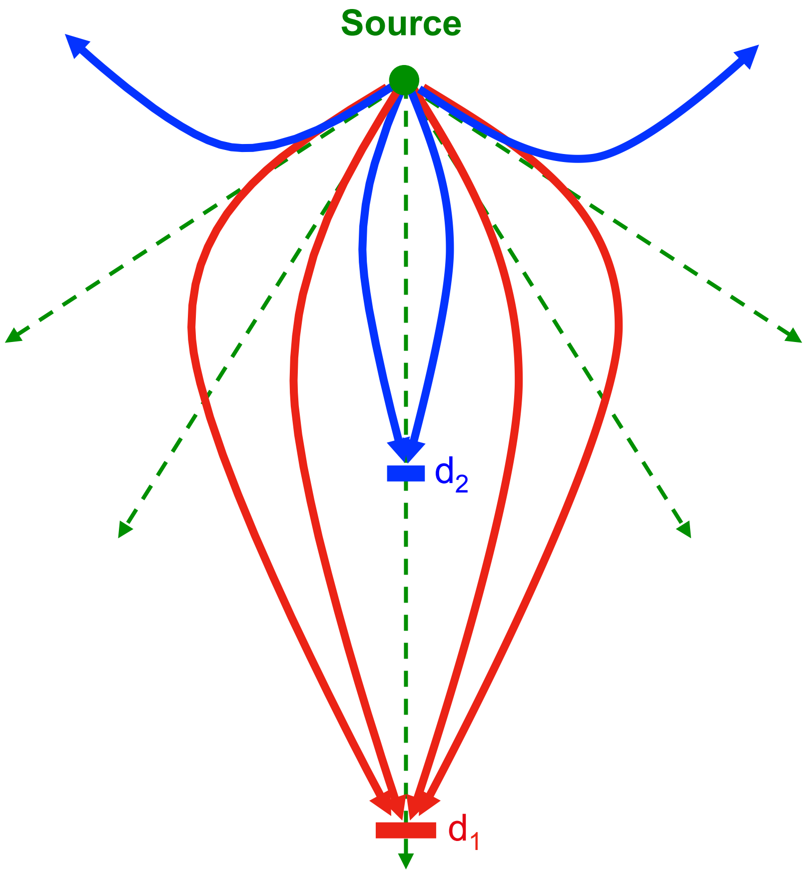
Bias condition #1

- Large collection angle
- Large cross over (d_1)
- Large probe

Bias condition #2

Stronger = more negative

- Narrower collection angle
- Smaller cross-over (d_2)
- Smaller probe
- Less total beam current

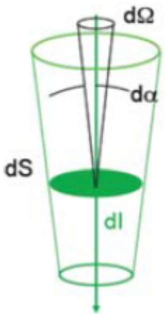


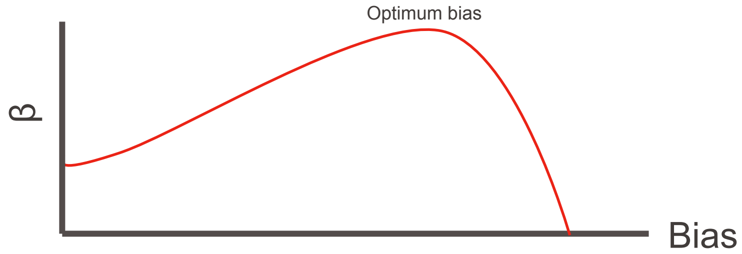
Current density

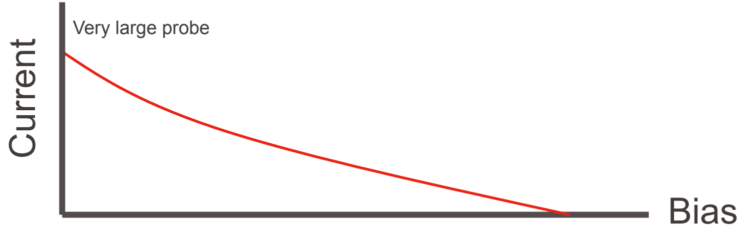
$$\beta \mid \text{brightness} = \frac{\text{Beam current}}{(\text{Area})\ (\text{Solid angle})}$$

Crossover size

Collection angle





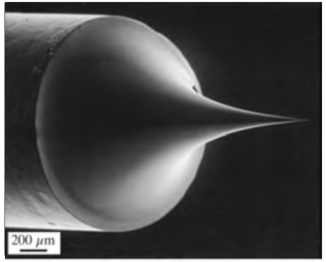


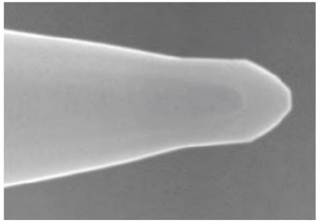
Field Emission Gun (FEG)

By applying an electric field of very high field strength at the surface of a metal, electrons are emitted even without heating the metal: *Cold field emission*

- Sharp tip is needed (less than 100 nm)
  - The strength of an electric field E is considerably increased at sharp points.
- Electrons can tunnel straight from the Fermi level out of the filament (usually tungsten).
- Surface has to be pristine (no contamination or oxide)

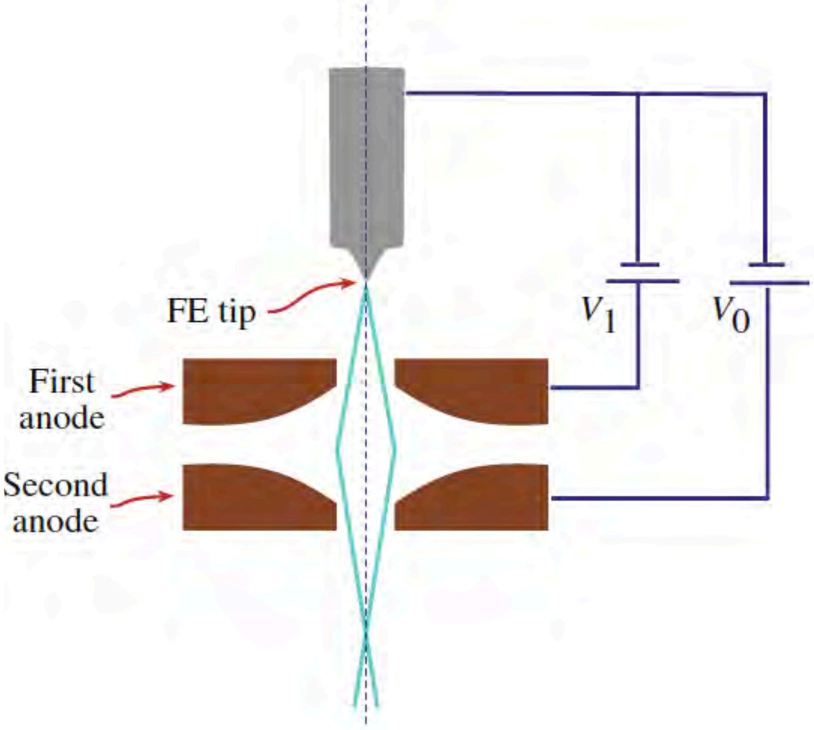
- 2 types of FEGs:
  - Cold FEG (Ultra-high vacuum condition needed)
    - $E \approx 10^9$ V/m
    - W mono-crystal with sharp tip (radius ~100 nm)
  - Heat assisted FEG: Schottky effect* (high vacuum is usually enough)
    - W crystal with ZrO surface treatments to lower the work-function
    - Can work with slightly poorer vacuum





*Schottky effect is the effective decrease of the work function when an external field is applied at the metal surface.

Field Emission Gun (FEG)



- Anode 1 provides the extraction voltage to pull electrons out of the tip.
- Anode 2 accelerates the electrons to the desired voltage.

The electrons are accelerated through the appropriate voltage by the second anode.

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Electron gun

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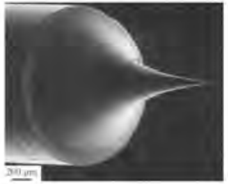
Intensity

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.**

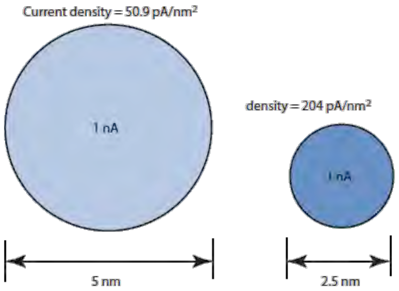
Temporal coherency:
Do all the electrons have exactly the same speed/energy?
A beam with high **temporal coherency** will have electrons of the same wavelength.

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

$$\beta \mid \text{brightness} = \frac{\text{Beam current}}{(\text{Area})\ (\text{Solid angle})}$$

- Current stability
- Vacuum needed



Electron guns

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

- Simple to use & maintenance friendly
- Cheap
- Requires only moderate vacuum
- High total beam current
- Low brightness
- High energy spread
- Large source size (10-100 μm)
- Limited lifetime (~1000h for LaB_6)

- FEG
  - Analogous to child's slide (toboggan)
  - Electrons from a very sharp tip radius ~100nm
  - Same energy
  - Same direction

- High coherence (both spatial and temporal)
- Small energy dispersion (< 0.4 eV)

→ higher resolution at lower energies
- High brightness
- Long lifetime >1000h
- Expensive
- Ultra-high vacuum necessary
- Cold FEG needs flushing after ~8 hrs

Electron gun

	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^2sr)	**$5x10^9$**	$5x10^{10}$	**$5x10^{12}$**	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

Components of an EM

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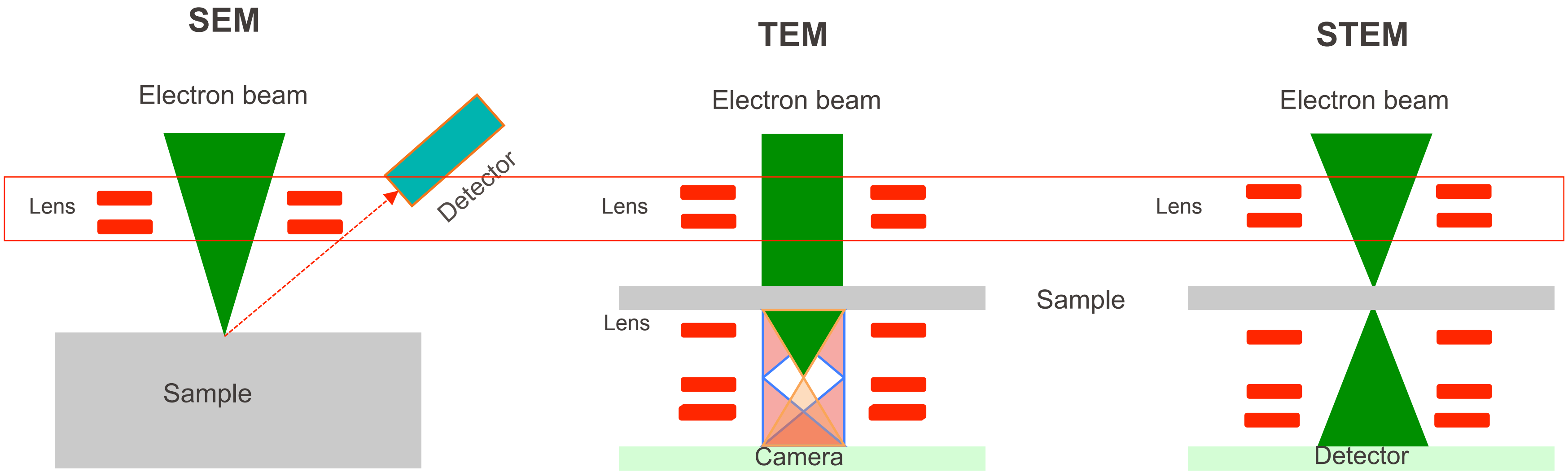
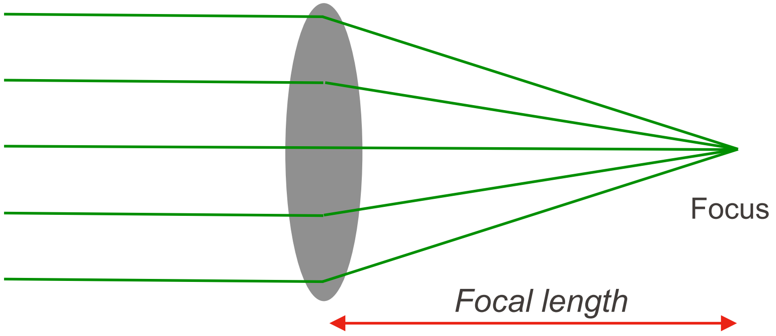


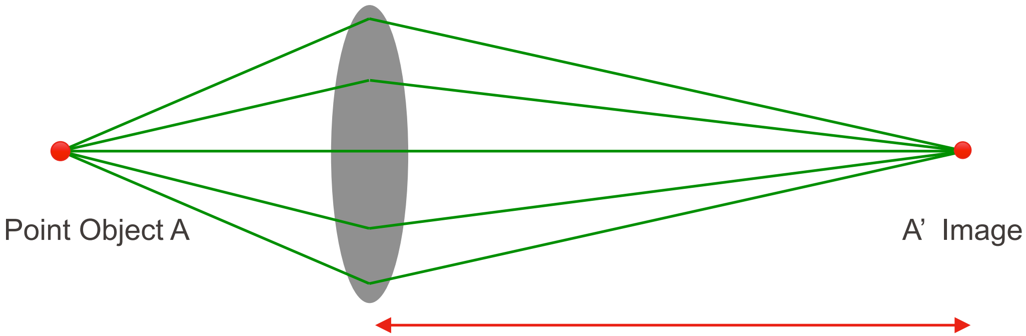
Vacuum system
Electron gun
Lenses
Detectors

Lenses

Lens bends beams to focus it to a point.

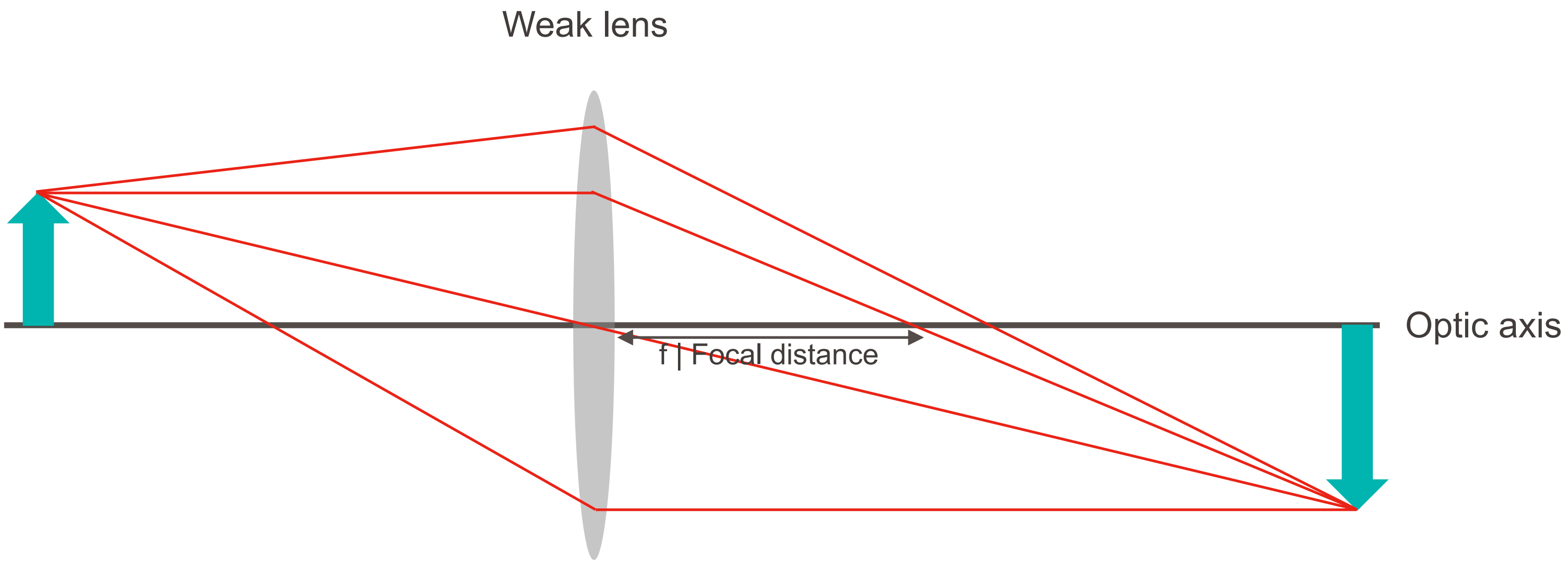


The rays emanating from a point in the object plane come to one common well defined point in image plane.



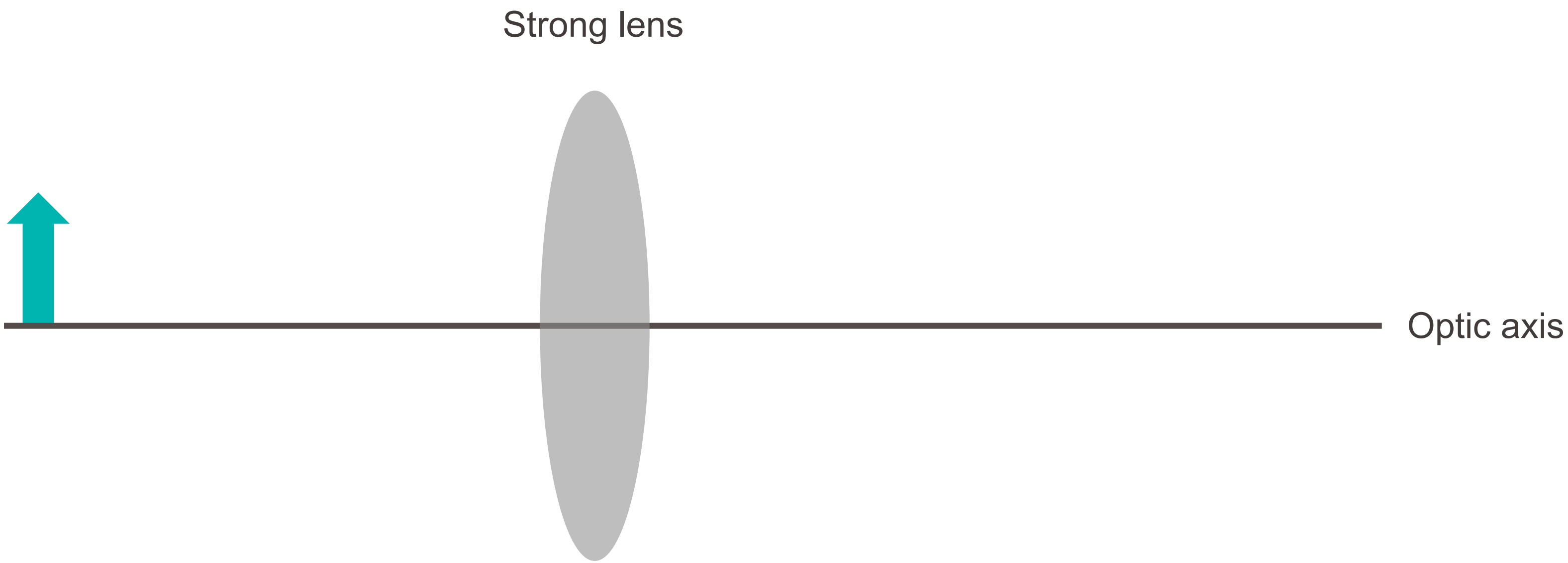
Lenses

Lens produces a magnified (or de-magnified) image



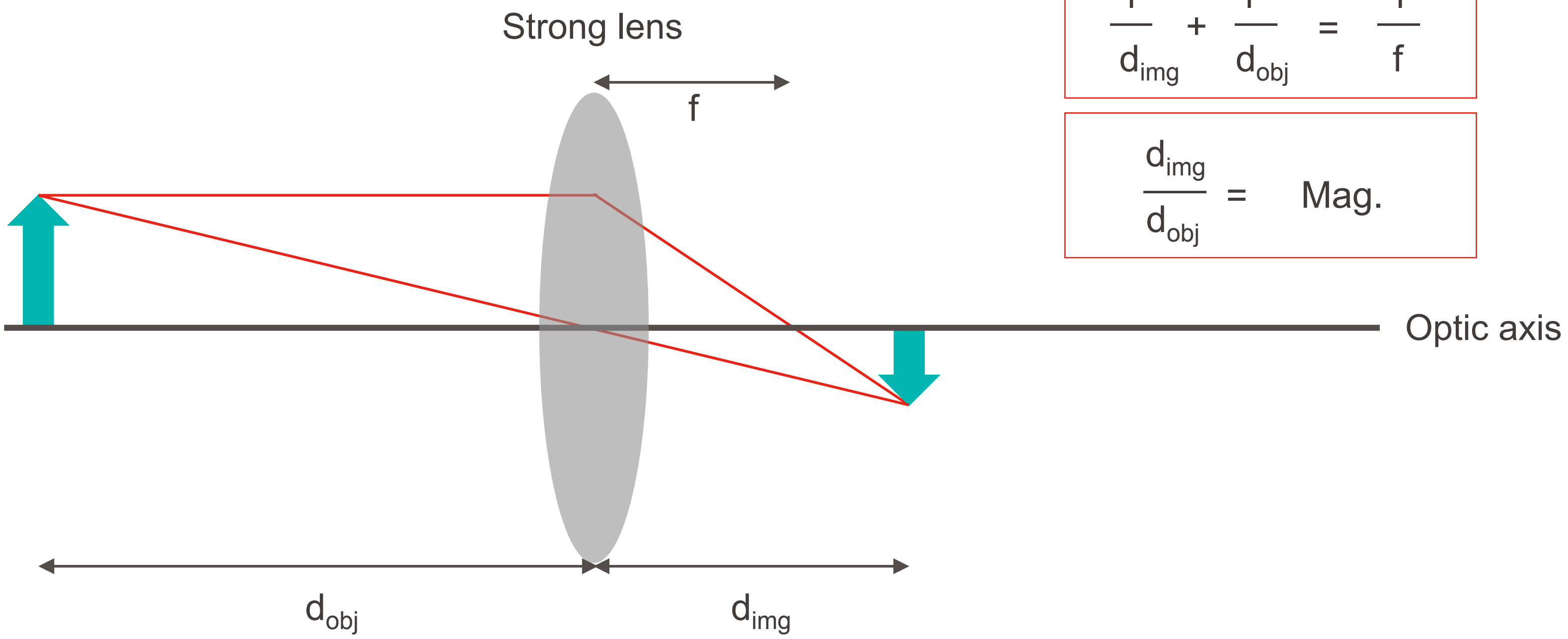
Lenses

Lens produces a de-magnified (or magnified) image



Lenses

Lens produces a de-magnified (or magnified) image

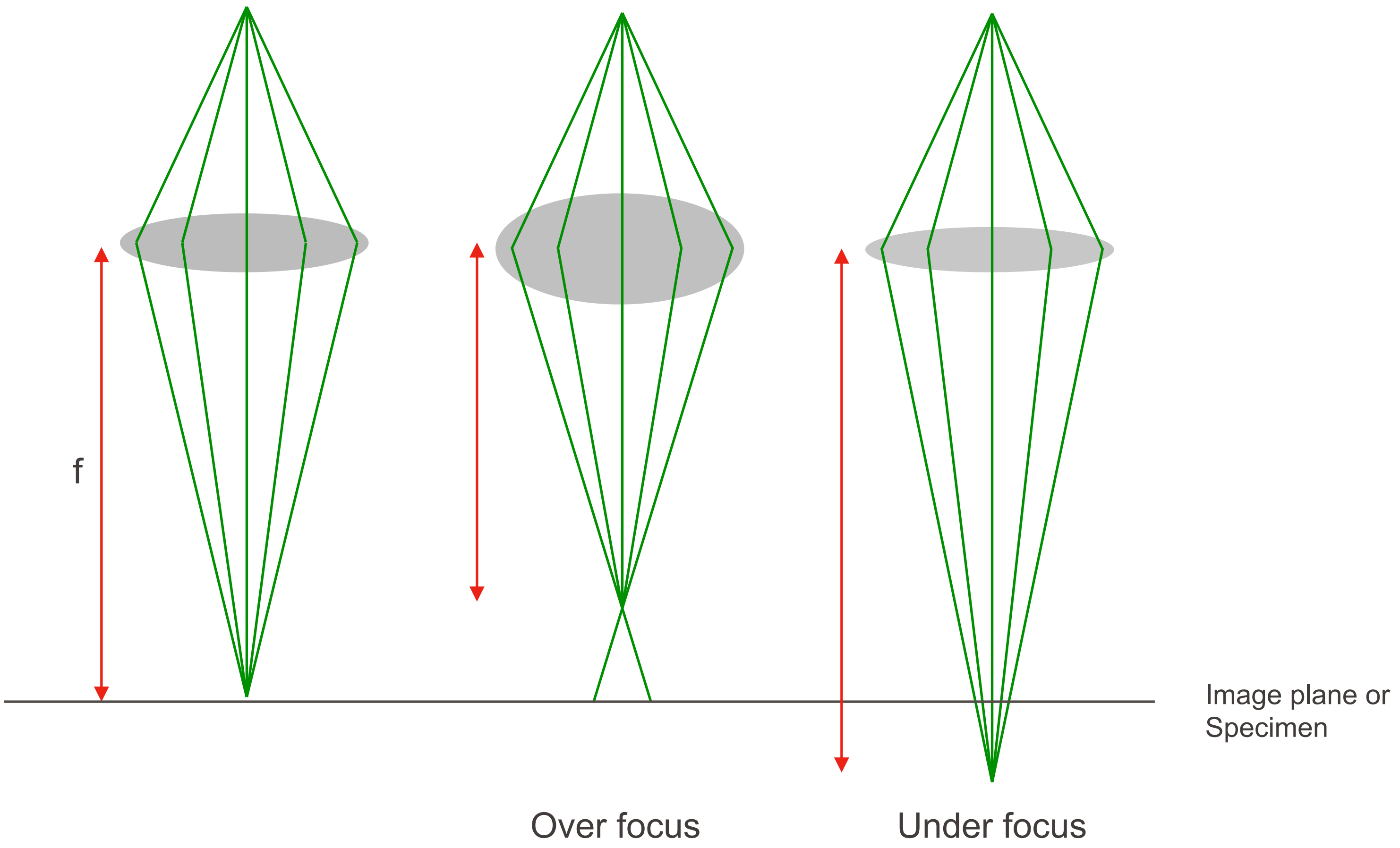


$$\frac{1}{d_{img}} + \frac{1}{d_{obj}} = \frac{1}{f}$$

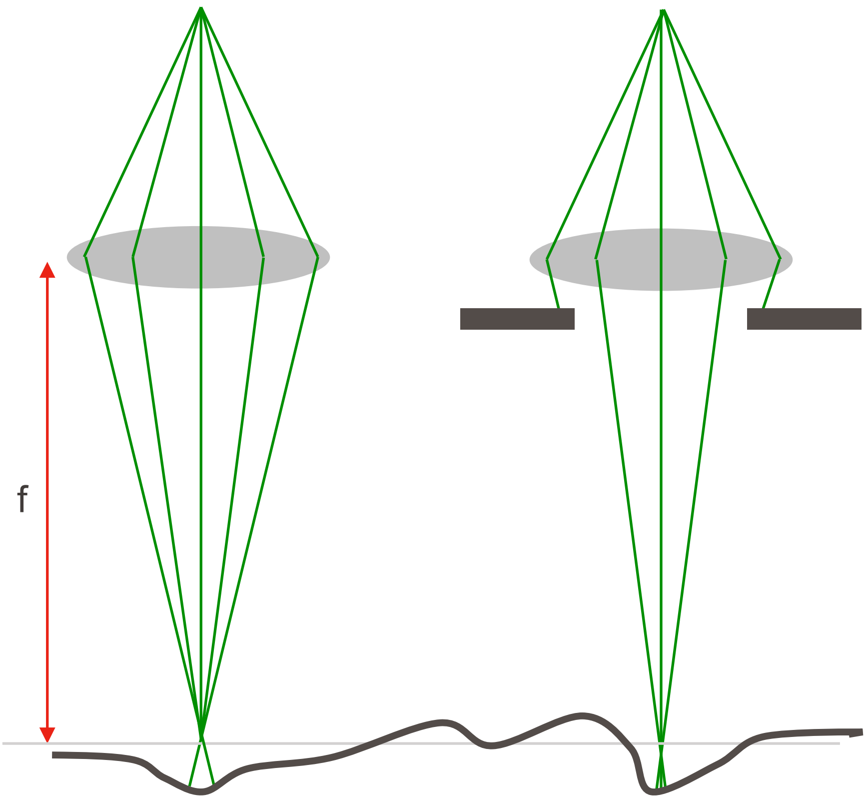
$$\frac{d_{img}}{d_{obj}} = \text{Mag.}$$

NOTE:
Electron microscopes have more than one lens. Under this circumstance the image plane of the the $\mathbf{n_{th}}$ lens becomes the object plane of the $\mathbf{(n+1)_{th}}$ lens. The total magnification is the product of the magnification of all the lenses.

Lenses



Lenses



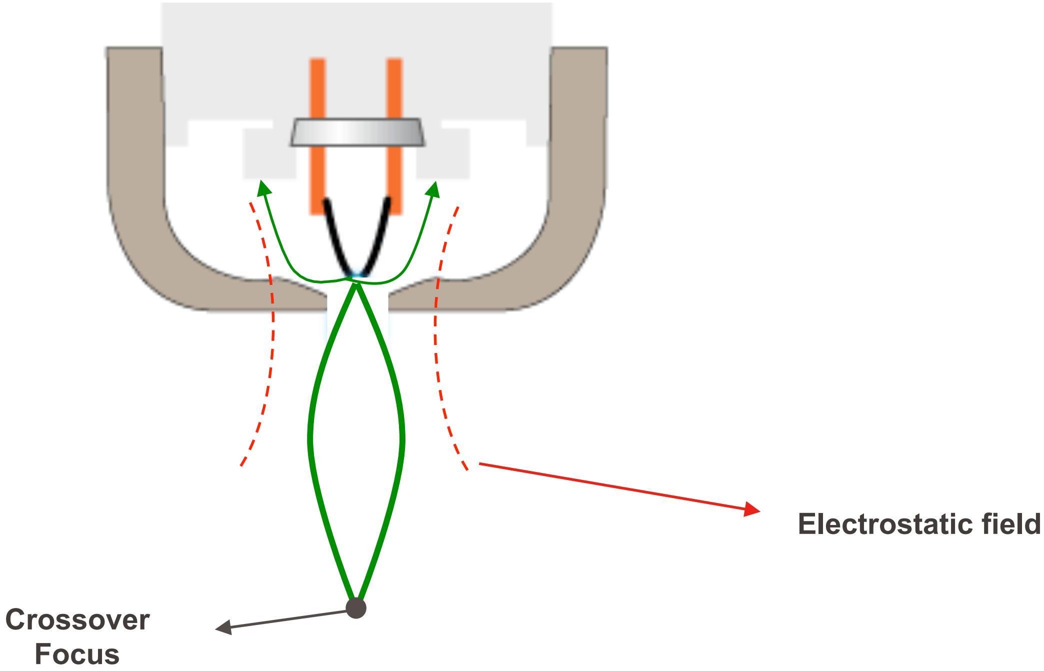
Smaller convergence angle
Less probe current
Larger depth of field?

Lenses

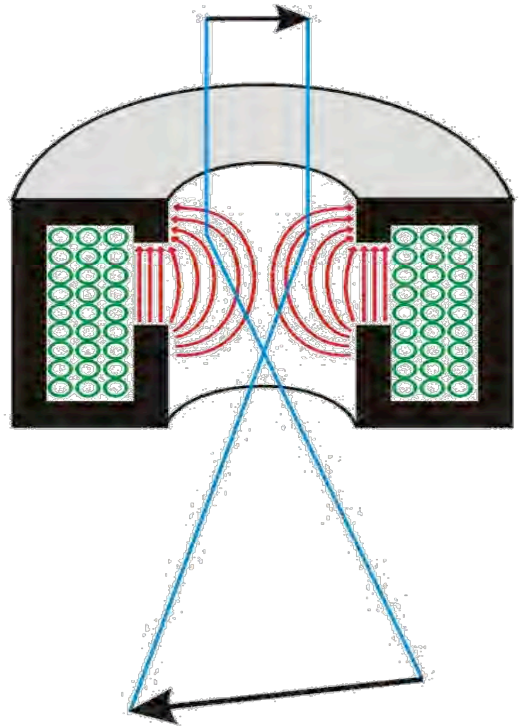
- Lenses for light
  - Glass or polymer lenses
  - Deflection of light through changing refraction index

- Lenses for electrons
  - Variable focus
  - Electrostatic
  - Electromagnetic: Lorentz force

Lenses - Electrostatic



Lenses - Electromagnetic



An electromagnetic lens consists of a coil of copper wires inside an iron pole piece.

A current through the coils creates a magnetic field in the bore of the pole pieces which is used to converge the electron beam.

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

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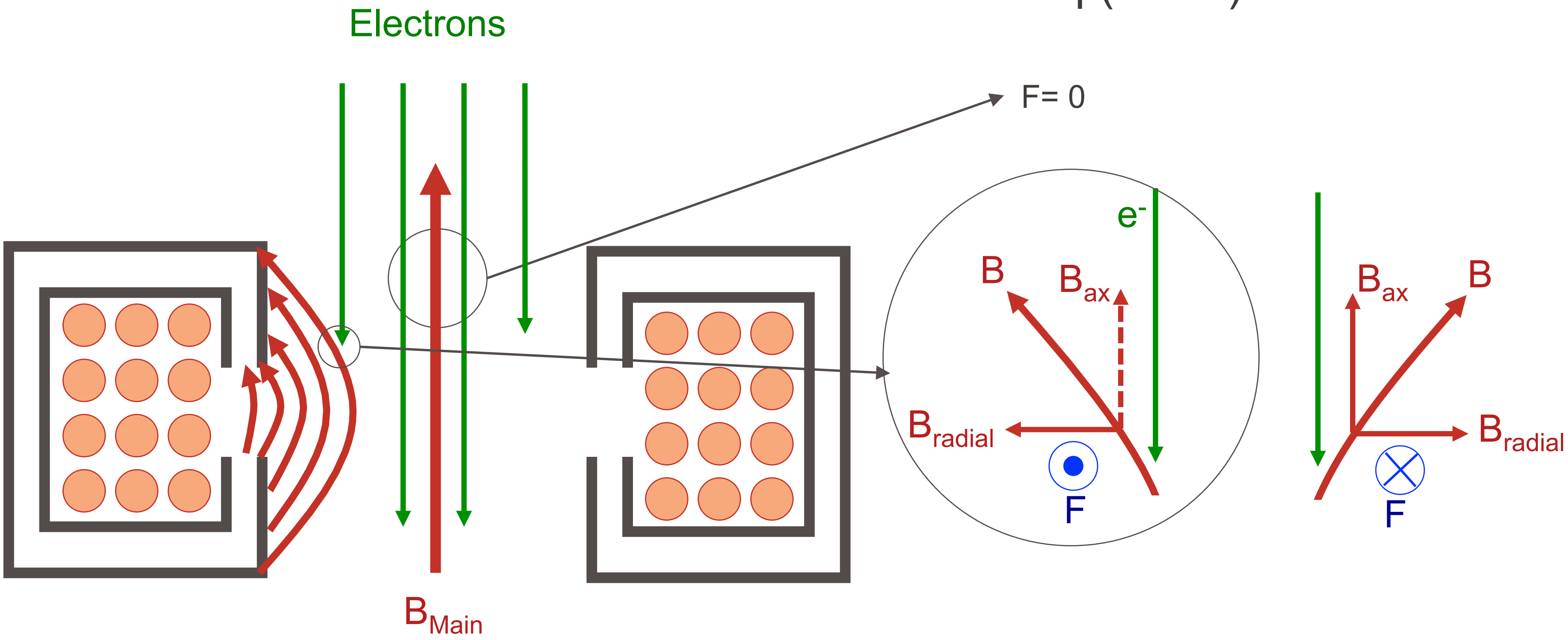
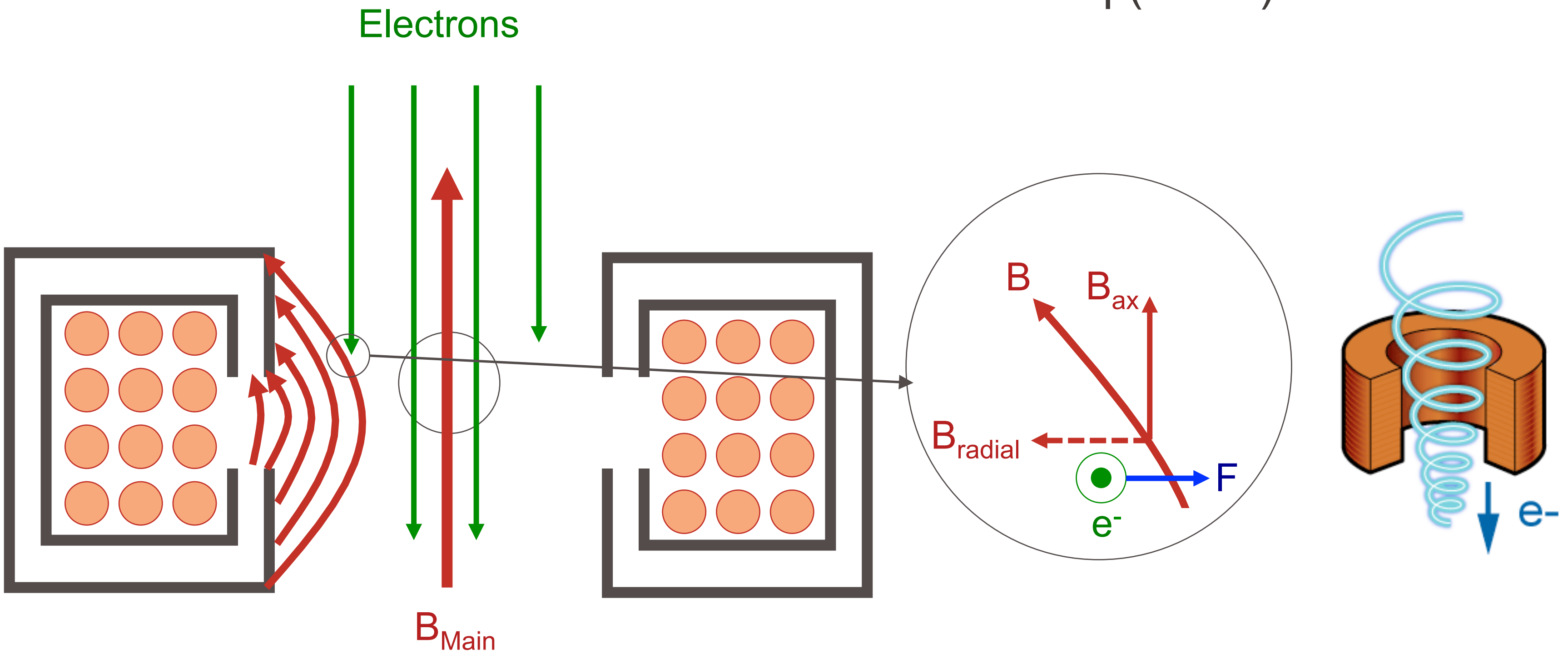
Lenses - Electromagnetic

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

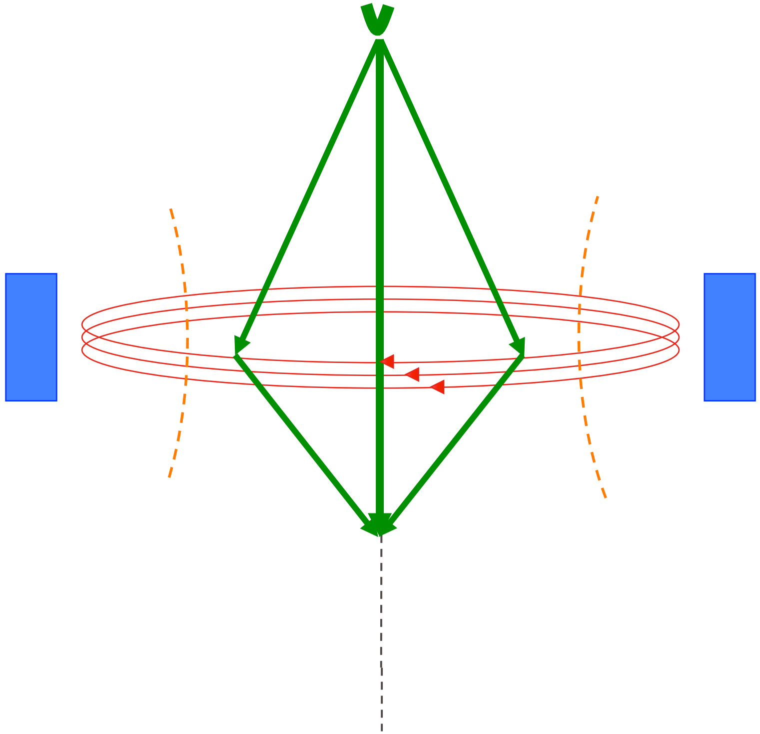


Lenses - Electromagnetic

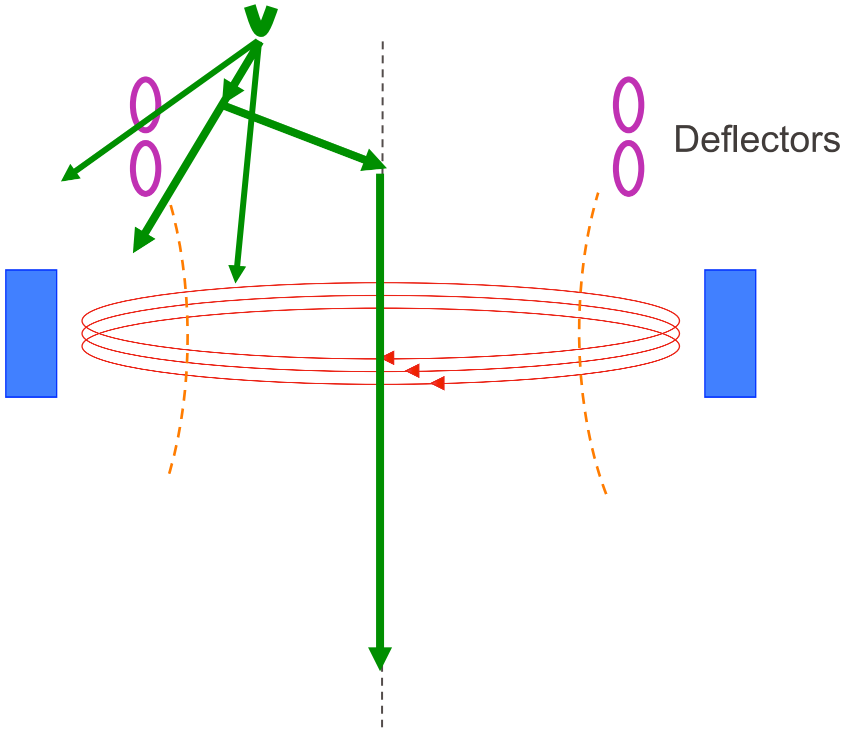
Lorenz force: $\vec{F}=q.(\vec{V} \times \vec{B})$



Electron optics

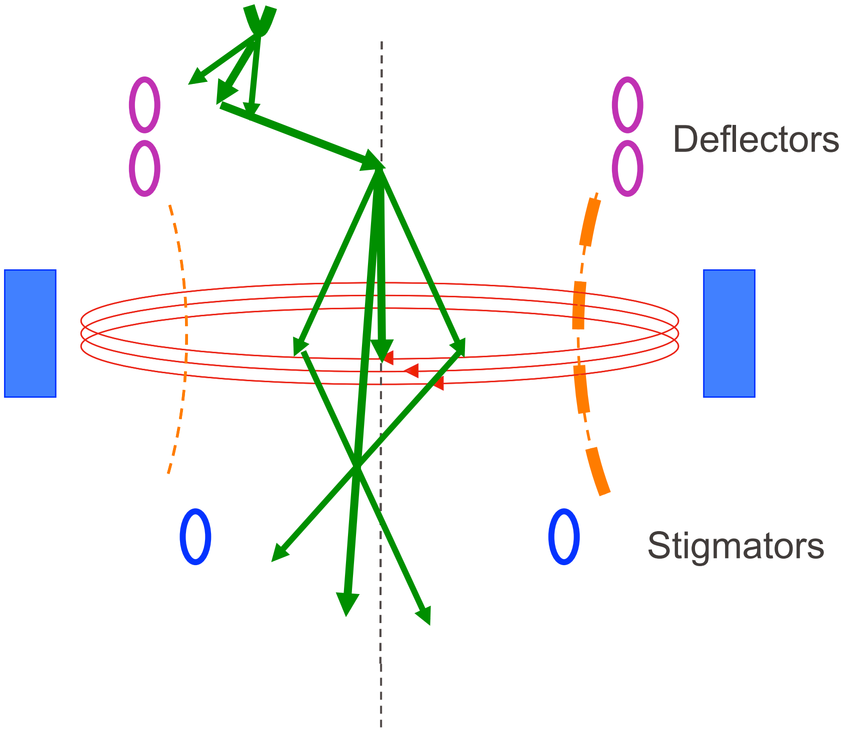


Electron optics

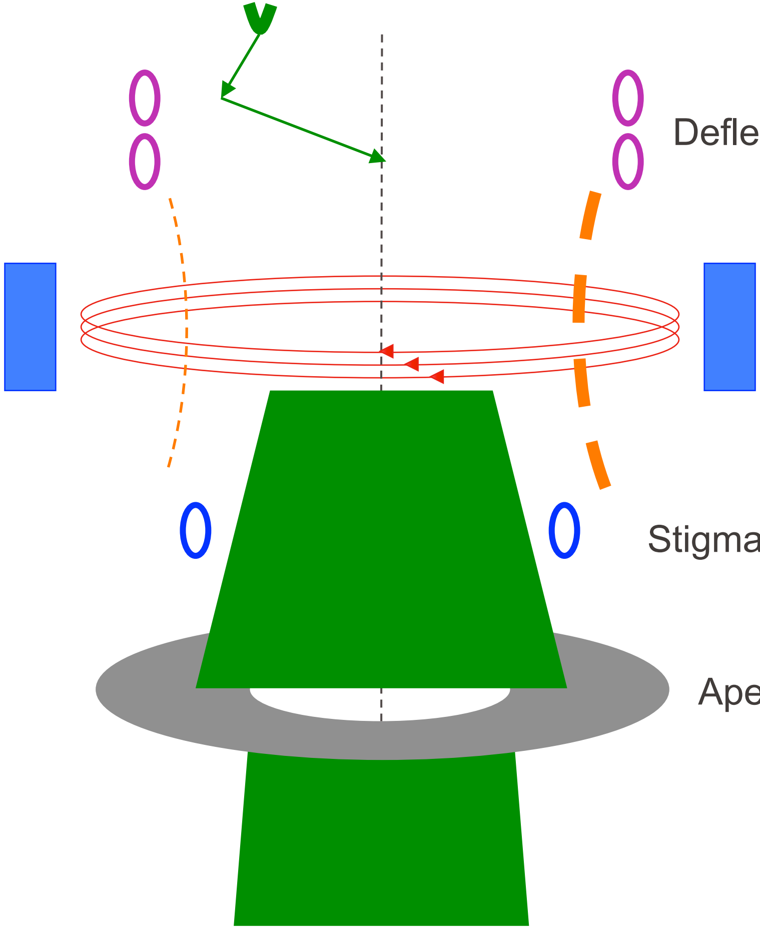


Electron optics

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



Electron optics



Electron optics | Scanning mode

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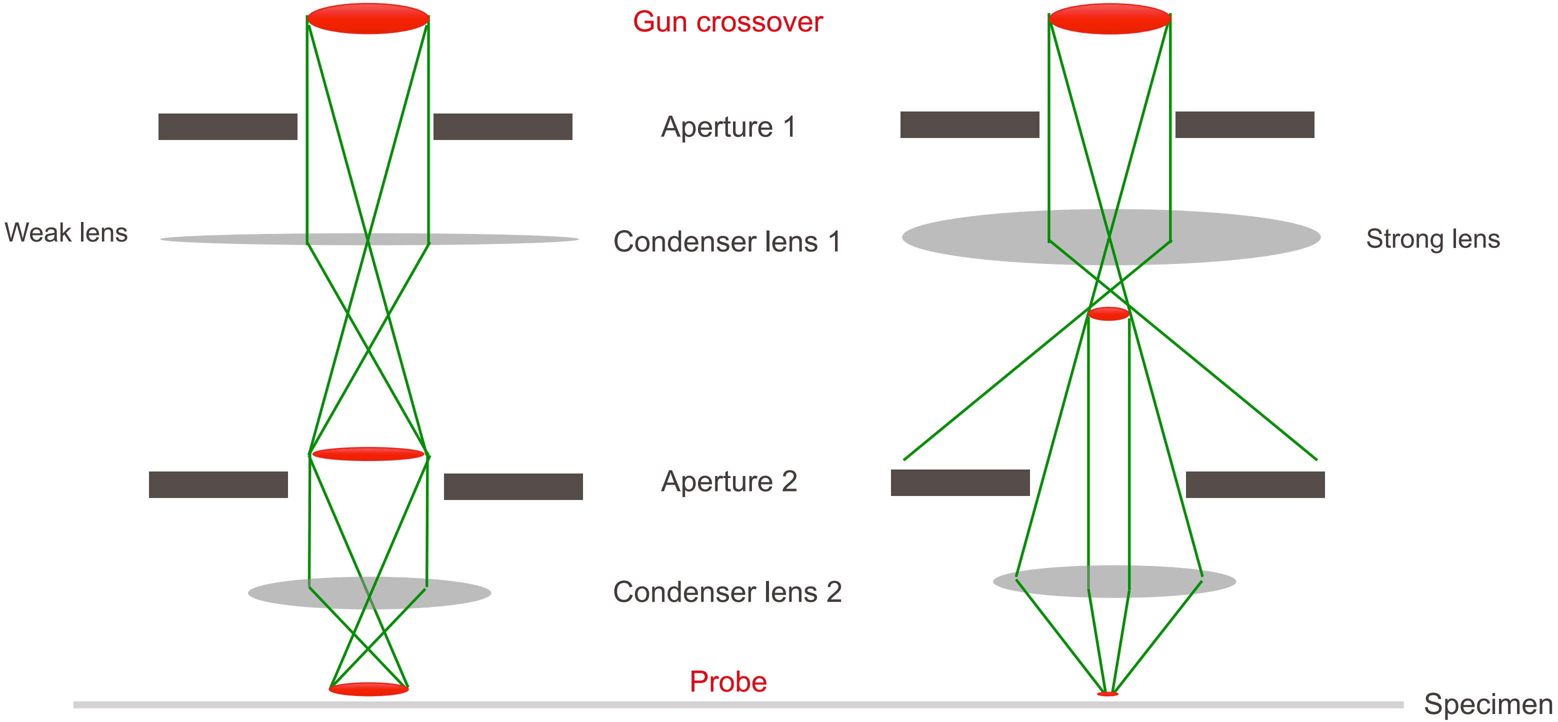
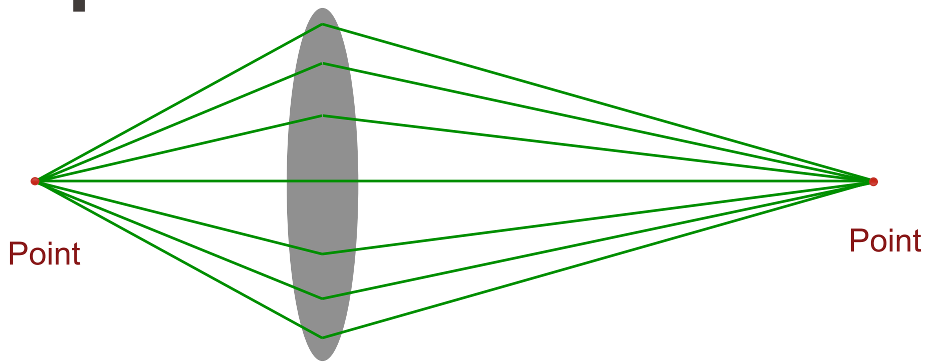


In scanning mode we need a narrow and convergent illumination.
In fact, we use the lens system to de-magnify the image of the gun.

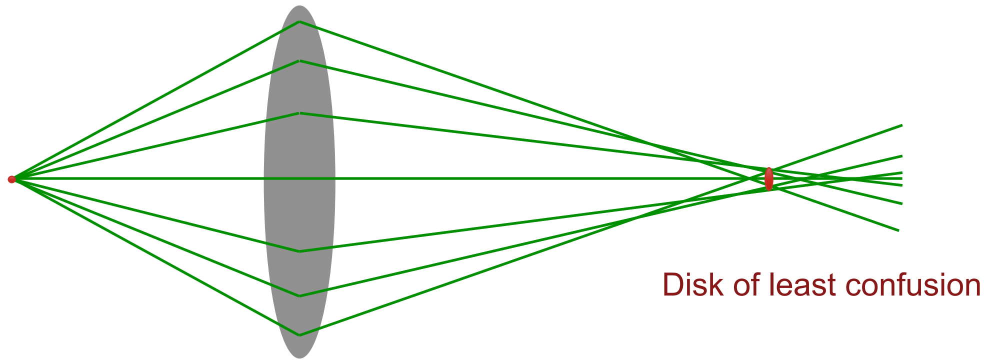
Electron optics

Ideal lens



A point source is focused to a point

Real lens

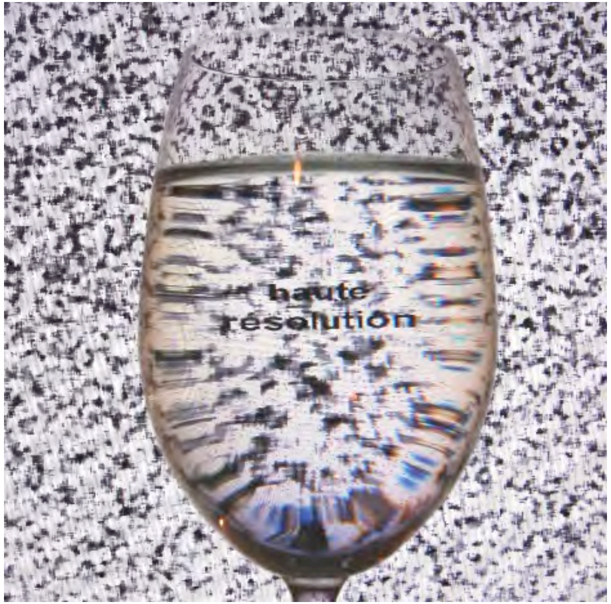


A point source is focused to a disk

Lens aberrations reduce resolution!

Electron optics

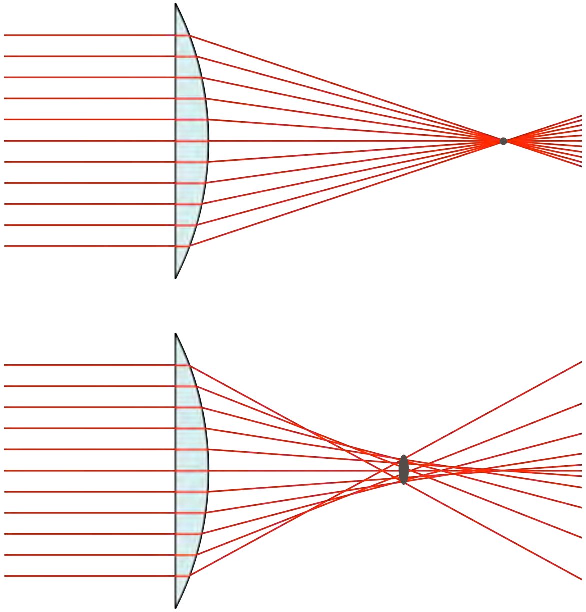
- **Lens aberrations**
  - Spherical aberration
  - Chromatic aberration
  - Astigmatism
  - Diffraction effect



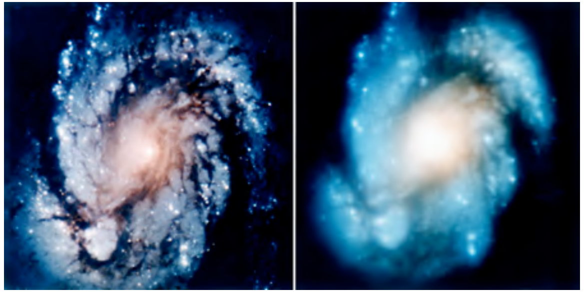
Lens aberrations are one of the main limitations to obtaining high spatial resolution.

Electron optics – Aberrations

- Spherical aberration (Cs)



- Parallel rays that pass through the central region of the lens focus farther away than the rays that pass through the edges of the lens.
- Results in multiple focal points and thus a blurred image.
- Larger probe and lower resolution.


Core of the galaxy M100 ©NASA

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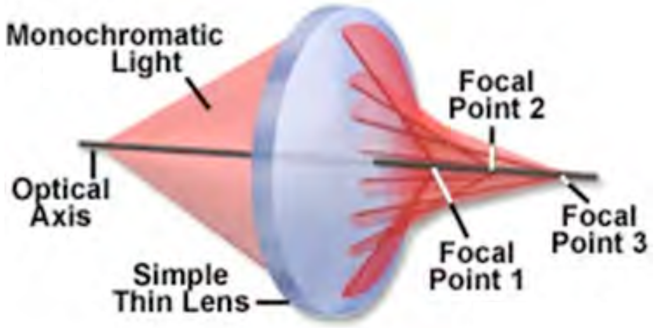
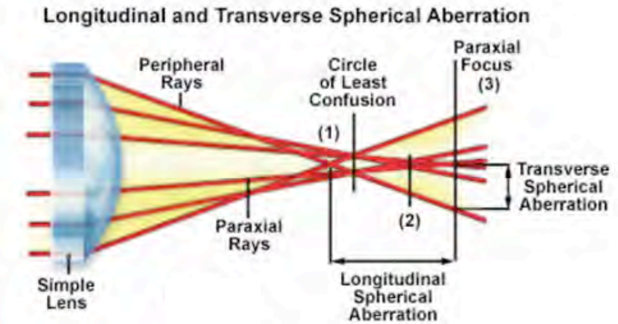
Electron optics – Aberrations

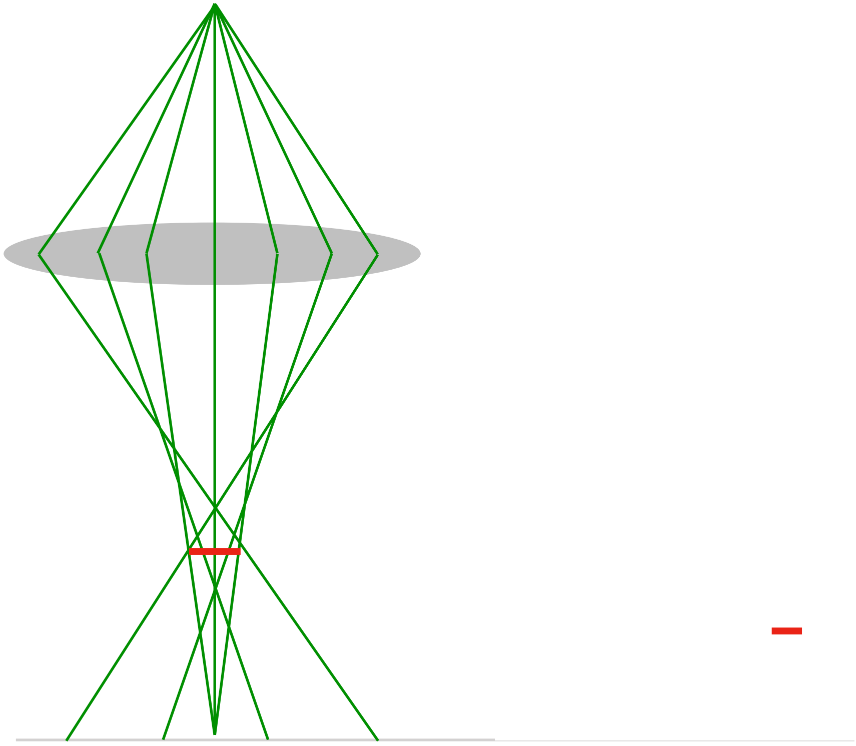
- Spherical aberration (Cs)



- Focal length depends on the distance from optical axis
- Image of the object is dispersed along the optical axis
- Circle of least confusion $d_s = ½ C_s λ^3$



Electron optics – Aberrations



How to lower the effect of spherical aberration?

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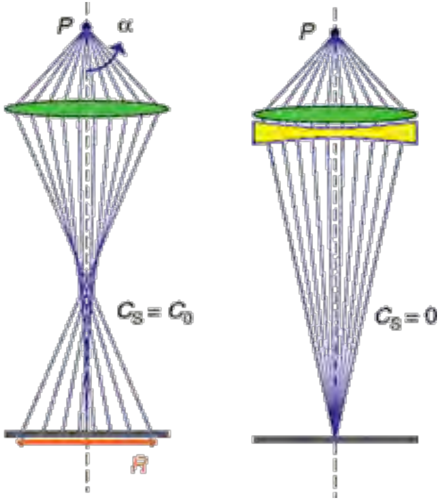
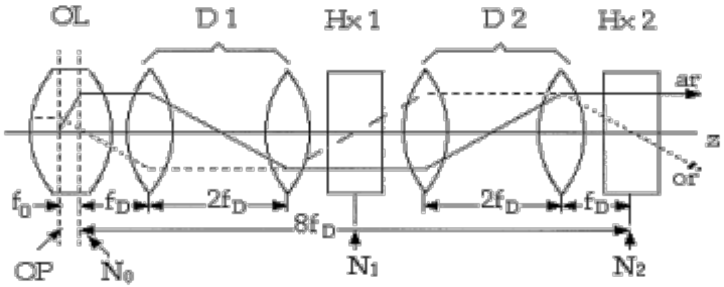
Electron optics – Aberrations

- Cs correction in light optics
  - Correction with combination of convex and concave lenses



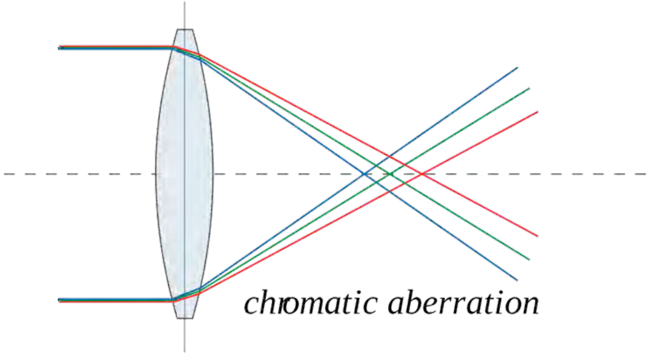
- Cs correction in electron optics
  - Correction with hexapole or quadrupole and octopole lenses



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

Electron optics – Aberrations

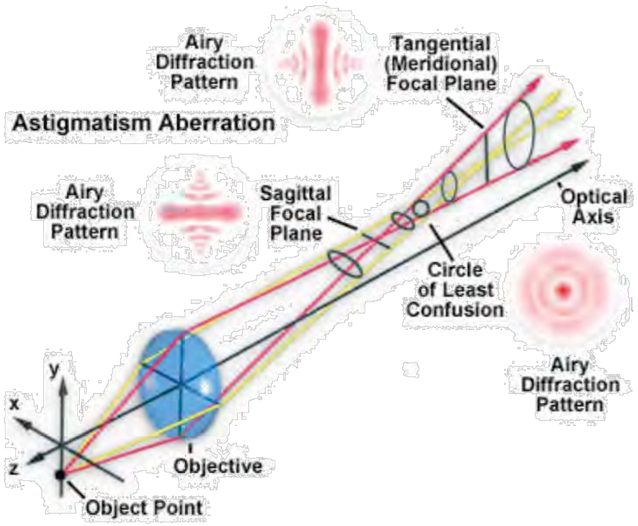
- Chromatic aberration (Cc)



- Lens cannot focus all energies (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.
- Cc increases with source energy spread.

Electron optics – Aberrations

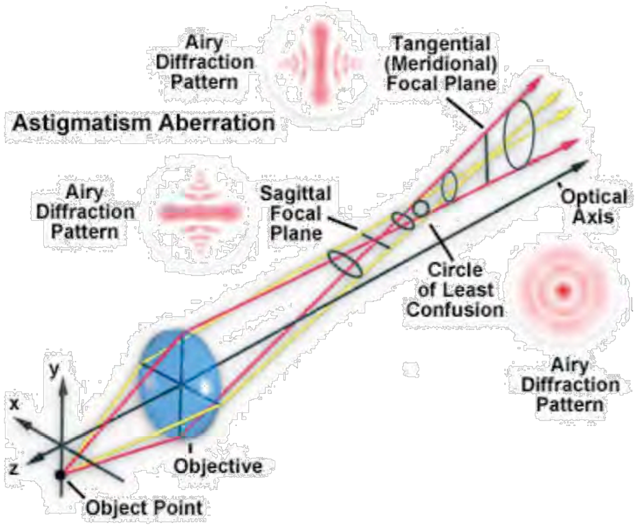
- Astigmatism



- Focal length varies for different axes of the lens.
- Image will appear “stretched” with changing the focus
- Caused by:
  - imperfections in the manufacturing of the pole-piece and the copper windings
  - Stray magnetic field

Electron optics – Aberrations

- Astigmatism



Under focus image

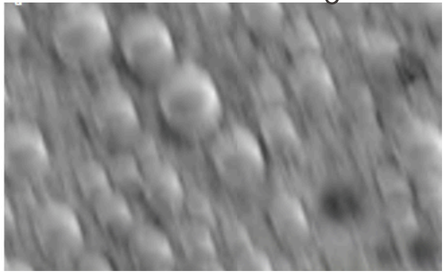
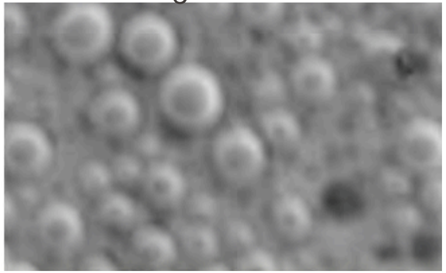
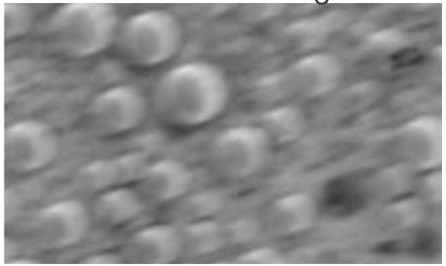


Image in focus



Over focus image



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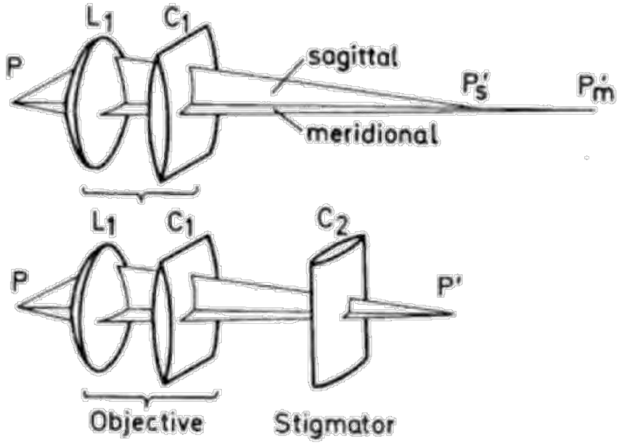
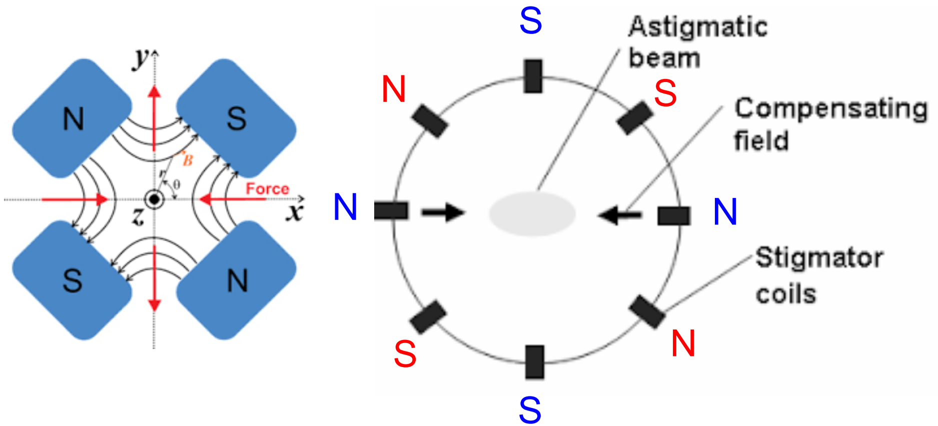
Electron optics – Aberrations

- Astigmatism in light optics
  - Correction with cylindrical lenses



- Astigmatism correction in electron optics
  - Correction with quadrupole lenses
  - 2 quadrupole lenses under 45 degree allow to control strength and direction of correction



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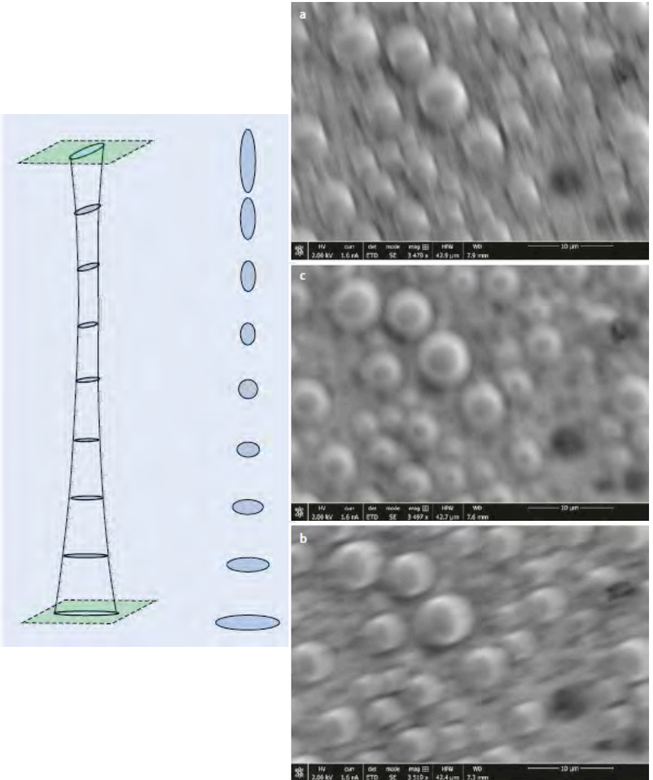
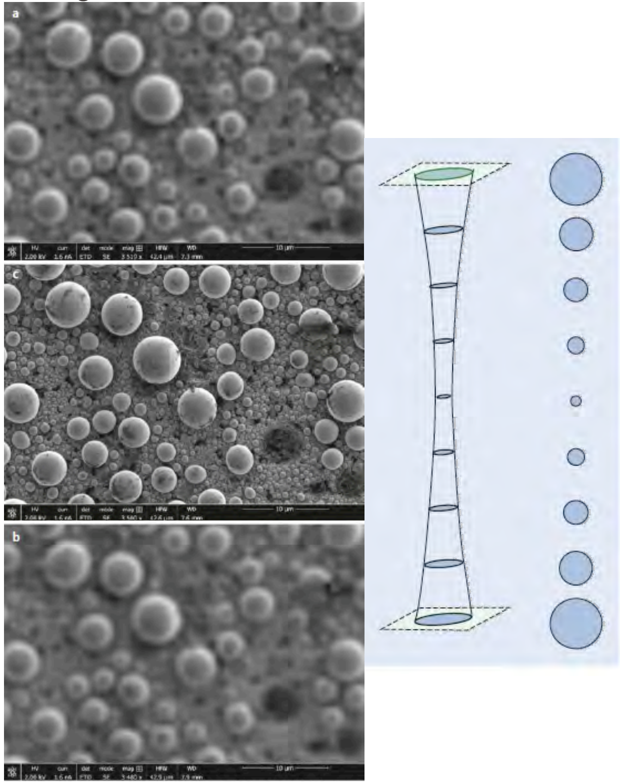
Electron optics – Aberrations

With astigmatism



Astigmatism corrected

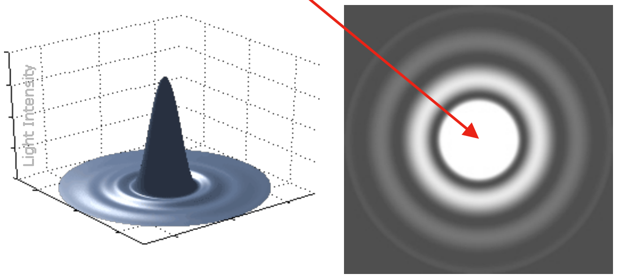


Electron optics – Aberrations

- Diffraction effect
  - Light/electron rays passing through a small aperture will begin to diverge and interfere with one another
  - → Diffraction | Airy disks

Disk/circle of confusion

Airy disk



Large aperture

Small aperture

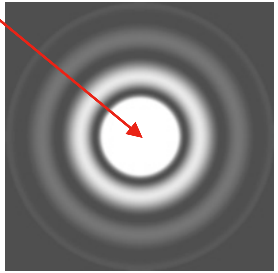
https://www.cambridgeincolour.com/tutorials/diffraction-photography.htm

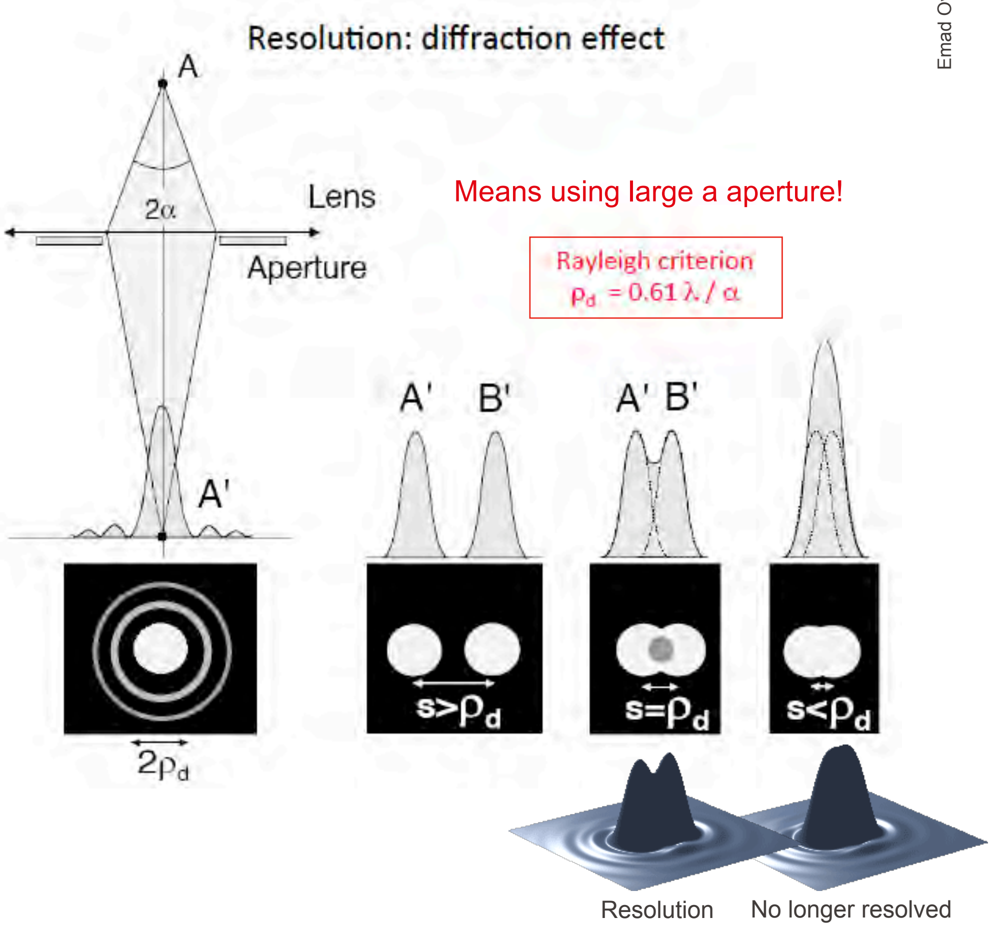
Electron optics – Aberrations

- Diffraction effect
  - Airy disks when light/electrons passing through a small opening (such as your camera's aperture)

Disk/circle of confusion

Airy disk

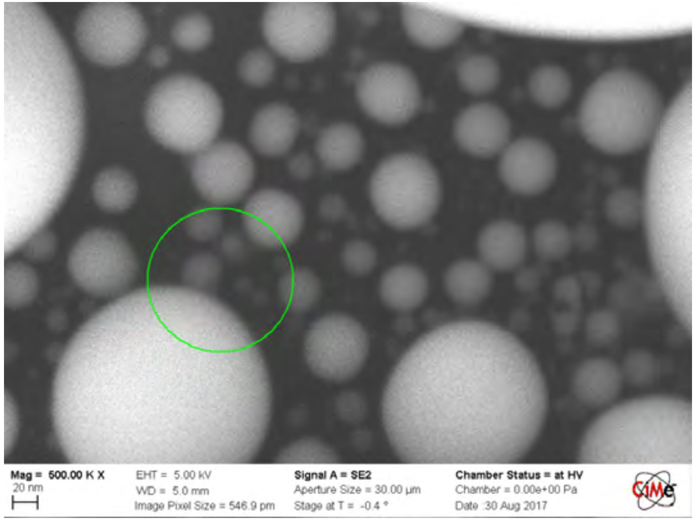




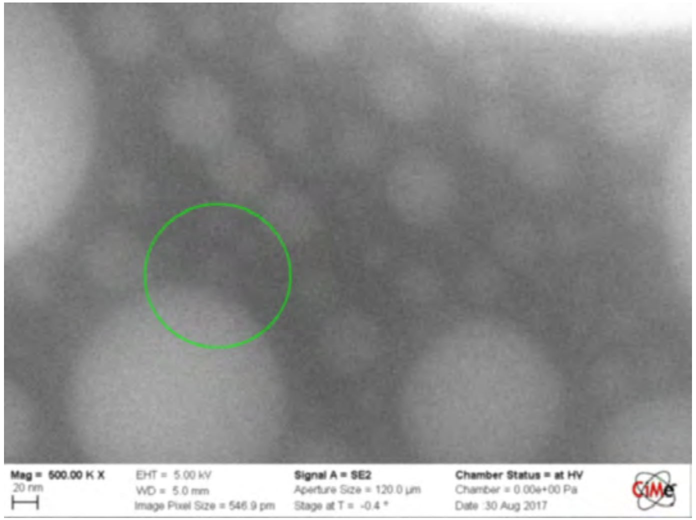
https://www.cambridgeincolour.com/tutorials/diffraction-photography.htm

Electron optics – Aberrations

Optimal Aperture size (30µm)

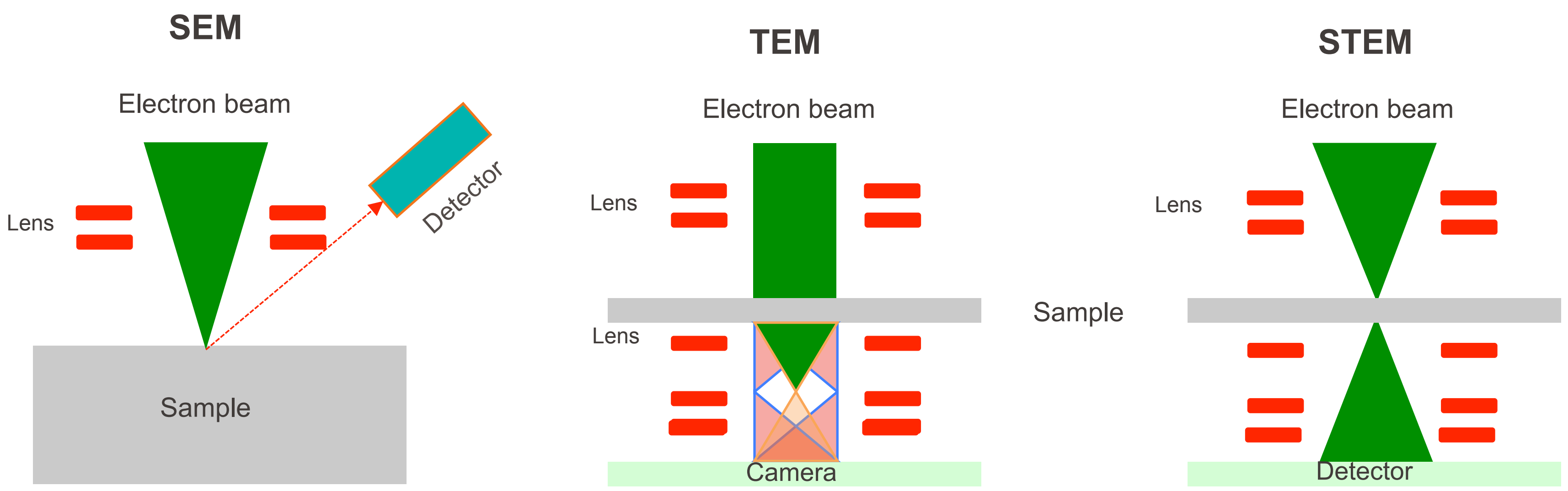


Large Aperture size (120µm)



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

Components of an EM



Vacuum system
Electron gun
Lenses
Detectors

Components of an EM

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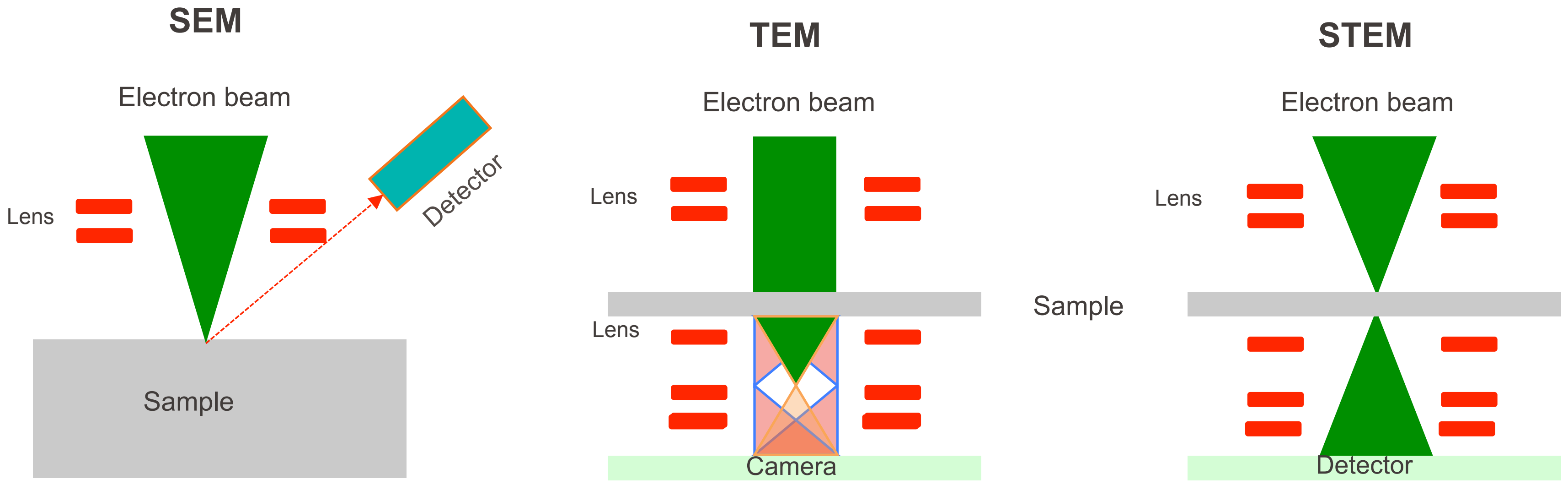


Scintillator/Photomultiplier
- Everhart-Thornley detector

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

Phosphor screen

Negatives and image plates

Semiconductor charge-coupled devices (CCD)

Complementary metal–oxide–semiconductor (CMOS)

Direct electron detectors

Disk/ring shape semiconductor detectors

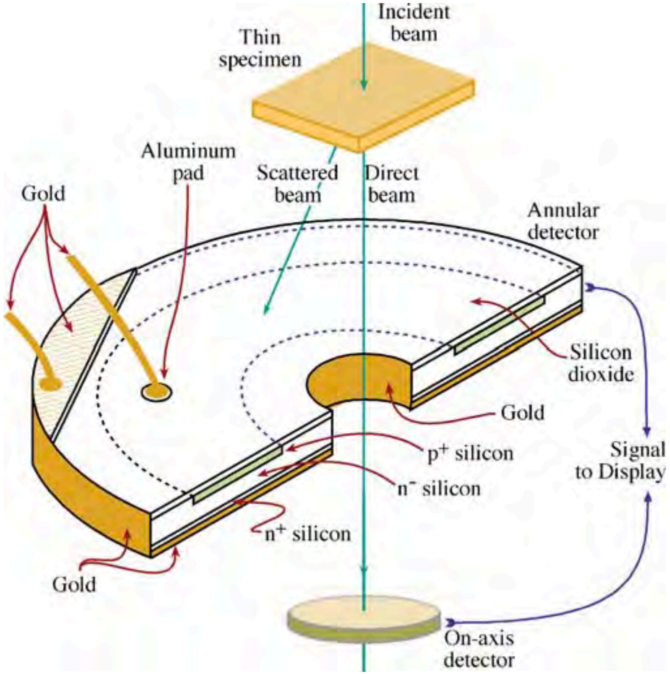
Pixelated detectors

Detectors
How to "See" electrons

- **Semiconductor detectors**
- **Scintillator-Photomultiplier detectors**
- Charge-Coupled Device (CCD) Detectors
- Complementary metal-oxide semiconductors (CMOS)
- Direct electron cameras

Electron detectors
Semiconductor detectors

- Si diode with a p-n junction close to its surface collects
  - By doping the Si (e.g., by ion implantation of n-type impurity atoms into p-type Si or vice versa).
  - n-type | Gives free electrons to semiconductor
  - By evaporating a thin layer of Au on the surface of high-resistivity n-type Si, or evaporating Al onto p-type Si (i.e. surface-barrier detector or a Schottky diode).

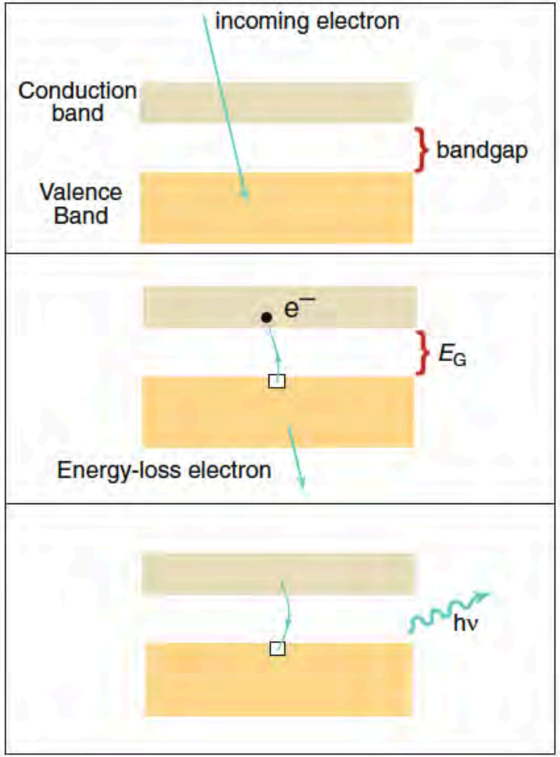


Electron detectors
Semiconductor detectors

- When struck by the high-energy electrons, most of the beam energy is transferred to valence-band electrons in the Si which are excited across the band gap into the conduction band thus creating electron-hole pairs (3.6 eV / electron-hole pair).

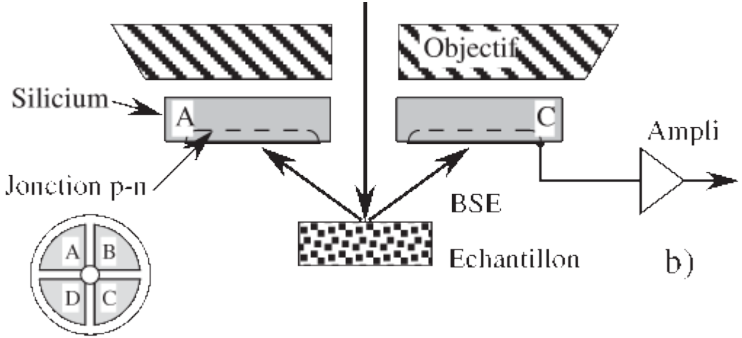
- 10 kev → ~2800 electrons

- Thus, the incoming electron signal is converted to a current in the external circuit between the surface contacts.



Electron detectors
Semiconductor detectors

- Very efficient at picking up and amplifying electron signals
- Large collection angle
- Cheap and easily fabricate
- Slow | Not responsive to rapid changes in signal intensity (poor at TV frequency)
- Some diodes are split in 2 or 4 quadrants to bring spatial electron distribution information



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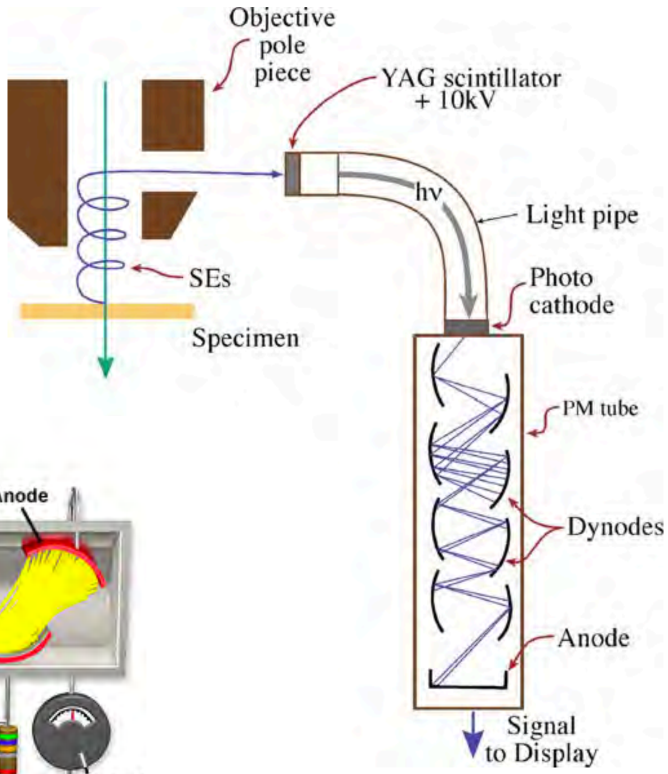
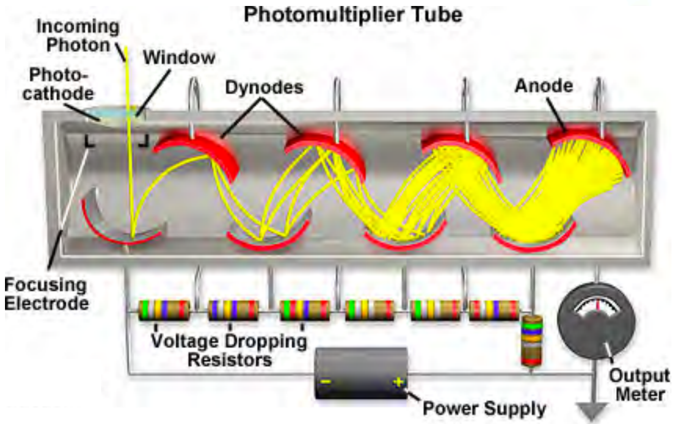
Electron detectors Scintillator-Photomultiplier

- A scintillator emits visible light when struck by electrons
- the light from the scintillator is amplified by a photomultiplier (PM) system, attached to the scintillator via a light pipe
- A collector (Faraday) cage with positive potential can be used to attract low-energy e$^-$





Electron detectors
Scintillator-Photomultiplier

- Faster an with lower noise level compared with the semiconductor detectors
- → low-intensity images and TV-rate images are easily displayed
- Not as robust as the semiconductor detector, being even more susceptible to radiation damage, particularly after long-time exposure to the beam.
- Scintillator-PM combination is substantially more expensive and bulky compared to semiconductor