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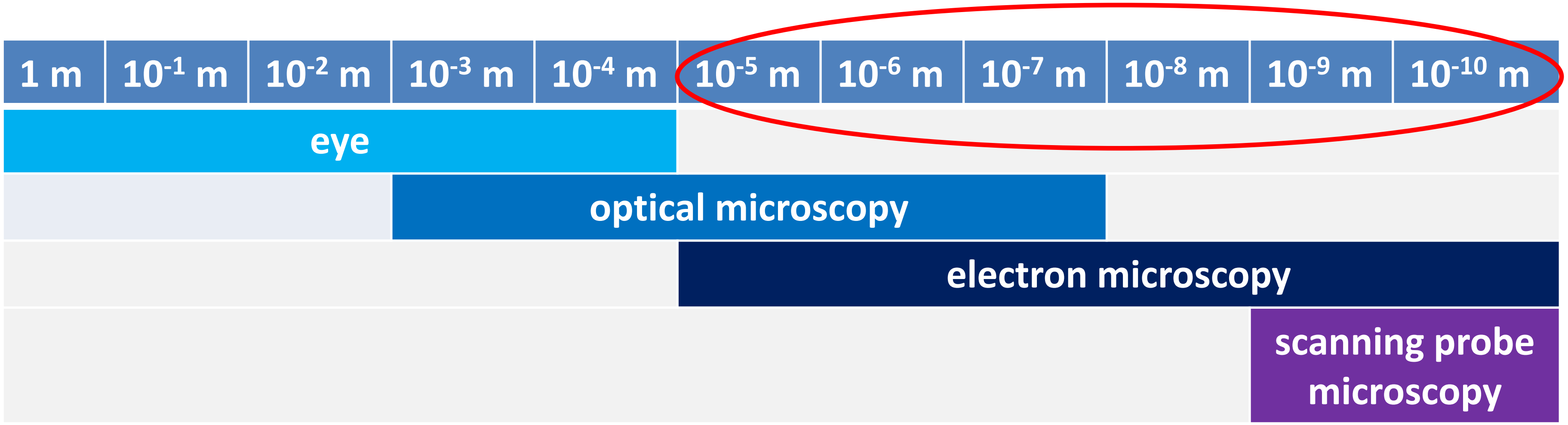
Physical and Chemical Analyses of Materials

Electron sources

Introduction

⇨ Electron sources are needed in electron microscopy and in electron induced chemical analyses as it is the case for XRMA and AES.

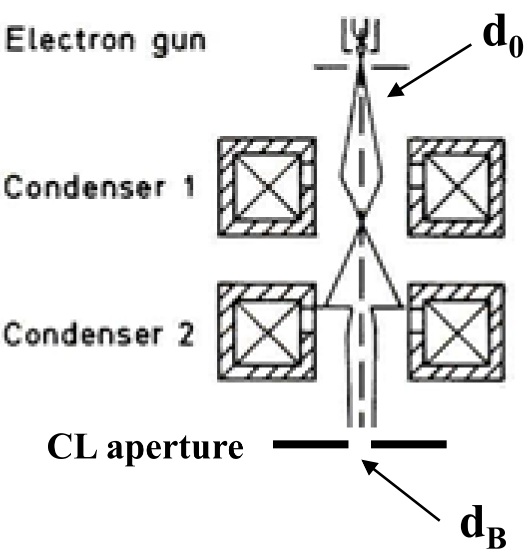
⇨ Conventional electron microscopes use an electron beam energy from 10 to 100 keV which is achieved using an acceleration tension from 10 to 100 kV. These electron energies allow the studies of samples from 10^{-5} to 10^{-10} meter size.



↳ The imaging of such tiny objects can be achieved only with a high lateral resolution which strongly depends on the electron beam size shone on the studied sample.

⇨ For chemical analyses, the electron source must be as stable as possible.

⇨ An electron source is made of an electron gun and many often two condensers, sometimes a third "mini" condenser is added to control the convergence angle of the electron beam in recent TEM:



⇨ The electron gun produces an electron beam of given intensity $\mathbf{I_0}$ and brightness **B** with a beam diameter $\mathbf{d_0}$, the initial cross-over. $\mathbf{d_0}$ depends on the acceleration voltage applied at the electron gun and suffers from the interactions between the emitted electrons that increase $\mathbf{d_0}$.

⇨ The condensers are made of electromagnetic lenses which de-magnify the electron beam emitted by the electron gun from $\mathbf{d_0}$ to the electron beam size $\mathbf{d_B}$.

⇨ The fine tuning of the condenser lenses (CL) aperture allows the optimisation of $\mathbf{d_B}$.

Electron gun and condensers

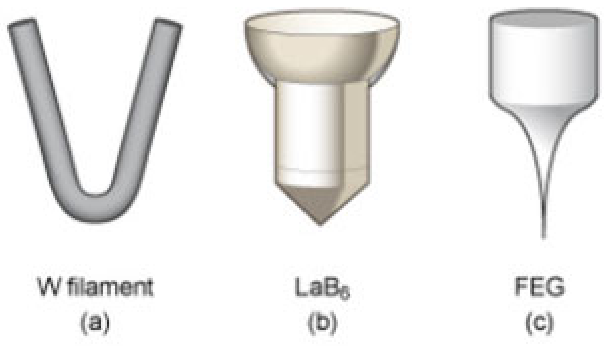
Electron gun emitters

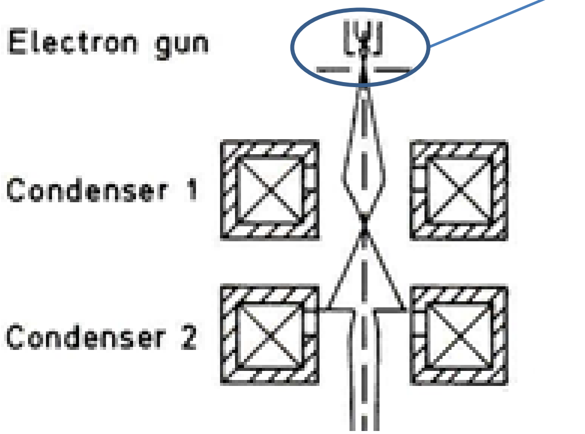
⇨ The electron beam can be produced by thermionic, Schottky or field-emission. Thermionic and field-emission are the most commons.

↳ Thermionic emission is provided by a tungsten (W) or a LaB_6 cathode heated at 2500 - 3000 K and 1400- 2000 K respectively (**a** and **b** on the scheme).

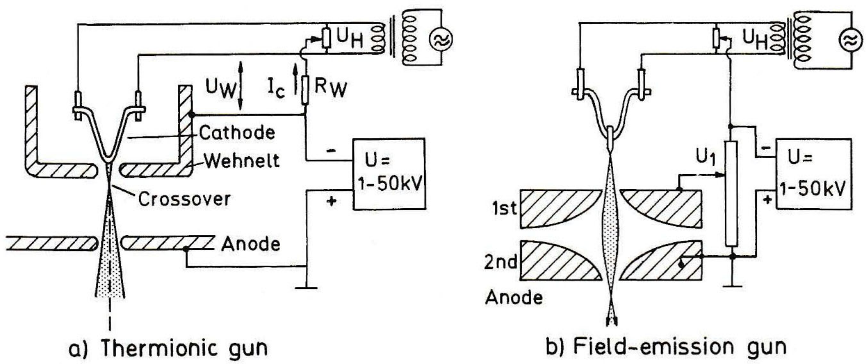
↳ Schottky emission is obtained from the heating of a ZrO/W tip at 1700 K under an electric field of $\approx 2\times10^8$ V·m^{-1}. This electron source is called S-FEG.

↳ Cold Field emission source uses a W tip at 300 K under an electric field of $\approx 5\times10^9$ V·m^{-1}. This electron source is called C-FEG.





⇨ When emitted by the filament or the tip (cathode), the electrons meet two electrodes:



⇨ In a thermionic gun, the Wehnelt cathode is more negative than the emitting cathode (few hundred of volts) and the anode is grounded.

⇨ In a field emission gun, the system is made of two anodes.

⇨ This series of electrodes results in the electron acceleration in both systems giving a beam at the desired energy.

Electron gun characteristics

⇨ The current density J_0 ($A \cdot m^{-2}$) of the electron emitters depends on the chemical nature of the cathode.

$$J_0 = AT_c^2 e\left(\frac{-\Phi_w}{kT_c}\right)$$

↳ In this equation, **A** is a constant which depends on the chemical nature of the cathode ($A \cdot m^{-2} \cdot K^{-2}$), T_c is the temperature of the cathode (K), Φ_W is the cathode work function (eV) and **k** is the Boltzman constant (8.62×10^{-5} $eV \cdot K^{-1}$).

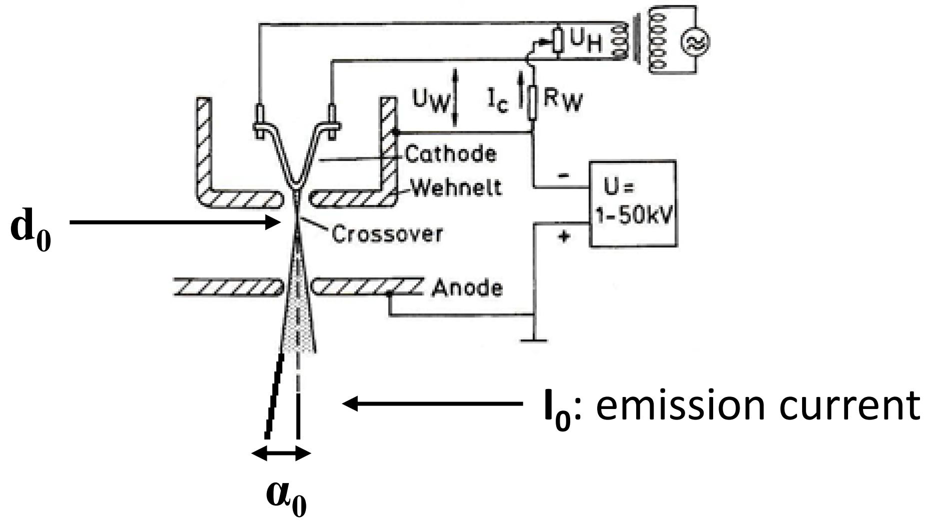
	thermionic gun		field emission gun (FEG)	
			S-FEG	C-FEG
Cathode material	W	LaB_6	W/ZrO	W
Vacuum (mBar)	1×10^{-4}	1×10^{-6}	1×10^{-8}	1×10^{-11}
T_c (K)	2700	1700	1700	300
A ($A \cdot m^{-2} \cdot K^{-2}$)	6×10^{5}	4×10^{5}	1.2×10^{6}	-*
Φ_W (eV)	4.5	2.7	2.7	4.5
J_0 ($A \cdot m^{-2}$)	1×10^{4}- 3×10^{4}	2×10^{5}- 5×10^{5}	5×10^{6}	1×10^{9} - 1×10^{10}

* The equation used to access to the current density is more complex than the presented one.

⇨ The Brightness **B** of the electron beam is the most important parameter to consider as it remains constant along the electron beam path even though it undergoes a focusing or defocusing through a series of lenses.

↳ Measuring **B** ($A \cdot m^{-2} \cdot sr^{-1}$) allows the determination of the initial cross-over diameter $\mathbf{d_0}$. **B** is given per solid angle units as:

$$\mathbf{B} = \frac{I_0}{\pi\left(\frac{d_0}{2}\right)^2 \pi(\alpha_0)^2} = \frac{4I_0}{(\pi d_0 \alpha_0)^2}$$



↳ The diameter $\mathbf{d_0}$ (m), the divergence half-angle $\boldsymbol{\alpha_0}$ (rad) and the beam current of emission $\mathbf{I_0}$ (A) are defined at the point at which the electrons are focused after leaving the source.

↳ The initial cross-over diameter $\mathbf{d_0}$ is then:

$$d_0^2 = \frac{4I_0}{B\pi^2\alpha_0^2} \Rightarrow d_0 = \frac{2}{\pi}\sqrt{\frac{I_0}{B}}\frac{1}{\alpha_0}$$

↳ **B** can be expressed as a function of the energy $\mathbf{E}_0$ of the electron beam (eV):

$$\mathbf{B} = \frac{\mathbf{E_0 J_0}}{\pi \mathbf{k T_c}}$$

↳ In this equation, $\mathbf{J_0}$ ($A \cdot m^{-2}$) is the electron current density, $\mathbf{T_c}$ (K) is the temperature of the cathode and **k** ($1{,}38 \times 10^{-23}$ $J \cdot K^{-1}$) is the Boltzmann constant.

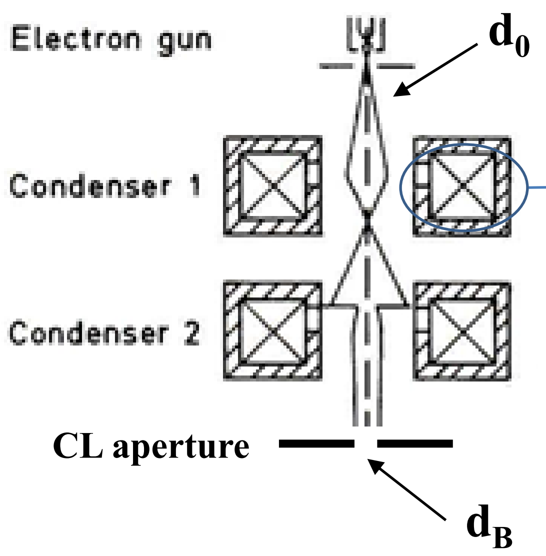
	thermionic gun		field emission gun (FEG)	
			S-FEG	C-FEG
Cathode material	W	LaB_6	W/ZrO	W
Brightness **B** at 30 kV ($A \cdot m^{-2} \cdot sr^{-1}$)	$2 - 10 \times 10^{8}$	2.5×10^{10}	$10^{11} - 10^{12}$	$10^{12} - 10^{13}$
Brightness **B** at 100 kV ($A \cdot m^{-2} \cdot sr^{-1}$)	1×10^{10}	5×10^{11}	5×10^{12}	1×10^{13}
Initial cross-over $\mathbf{d_0}$	20 - 50 µm	10 - 20 µm	15 nm	2.5 nm

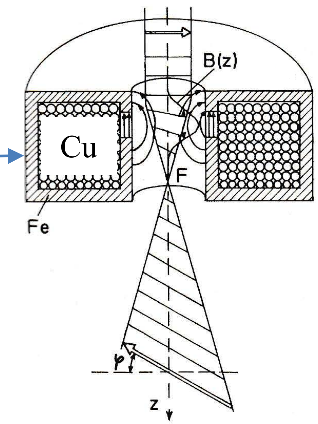
↳ Practically, the brigthness **B** can be either tuned through the beam energy $\mathbf{E_0}$, that is changing the acceleration tension of the electrons, or by changing the chemical nature of the cathode material through $\mathbf{J_0}$.

Condensers

⇨ The condensers are made of electromagnetic lenses, the role of which is to de-magnify the cross-over beam diameter $\mathbf{d_0}$ to the final beam diameter $\mathbf{d_B}$.

⇨ The condensers lenses de-magnify the cross-over beam from 1000 to 10 000 folds.

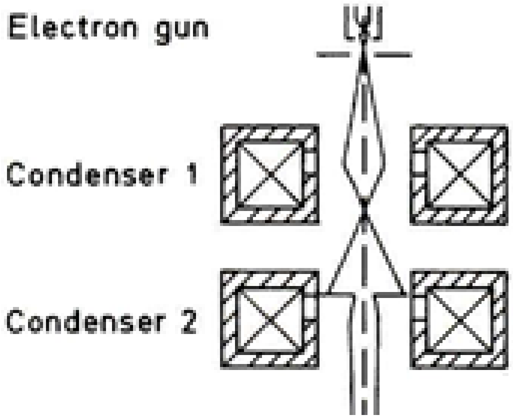




↳ Electromagnetic lenses are made of Fe/Cu and are tuneable.

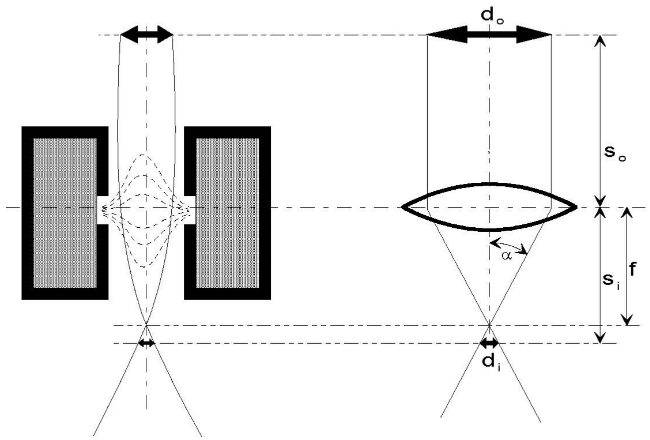
↳ The focusing effect of the lens increases with the magnitude of the **B** field controlled by the current passing through the coils.

↳ Convergent electromagnetic lenses have a tunable focal distance **f** thanks to the intensity of the current circulating in the lens.



⇨ Condenser 1 defines the probe size. Condenser 2 controls the illumination area, *i.e.* the intensity shone on the sample.

⇨ In recent TEMs, a third "mini" condenser is used to control the convergence angle of the electron beam.



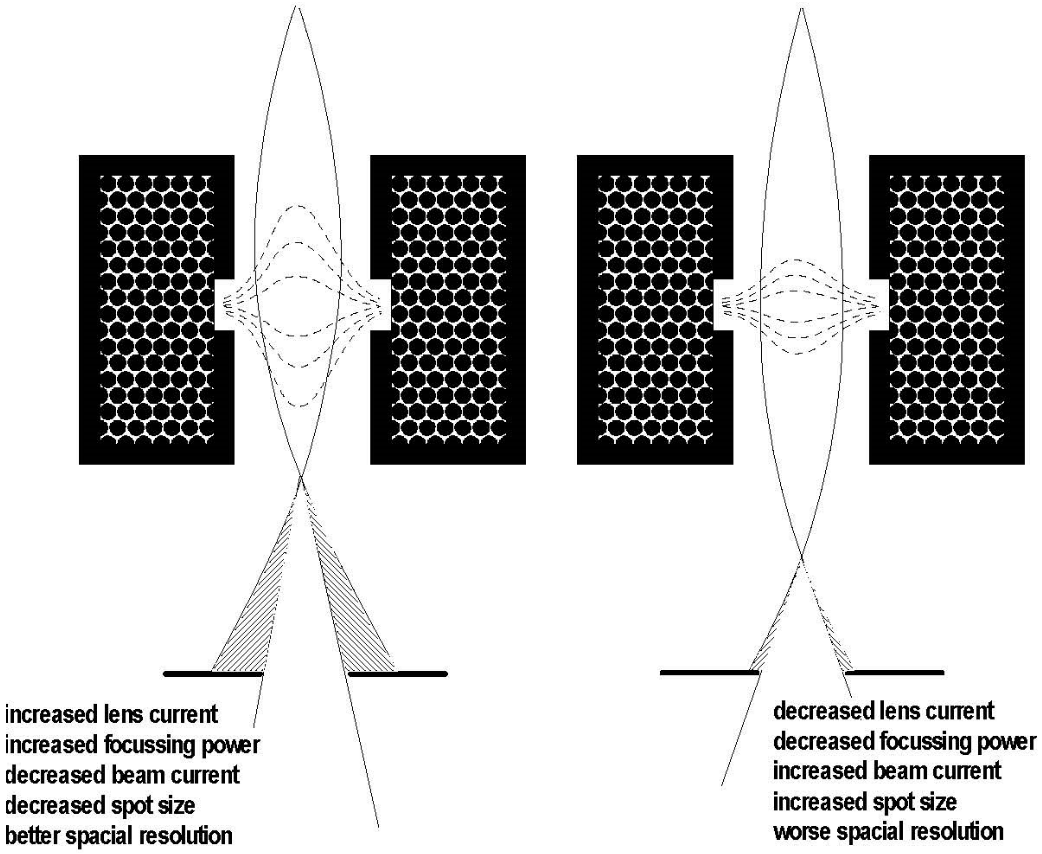
⇨ Let's consider an object of diameter $\mathbf{d_0}$ and its image of diameter $\mathbf{d_i}$.

⇨ The focal distance **f** (m) of the electromagnetic lens is calculed as follows:

$$\frac{1}{f} = \frac{1}{S_0} + \frac{1}{S_i} \Rightarrow f \propto \frac{E_0}{I_B^2}$$

⇨ In this case, for a given electron beam energy $\mathbf{E_0}$, the focal distance **f** of the condenser lenses will impose the current of the electron beam $\mathbf{I_B}$ shone onto the studied sample.

⇨ The magnification **M** of the beam is defined as follows:

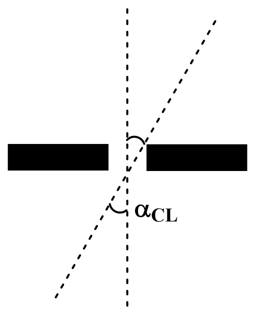


$$\mathbf{M} = \frac{\mathbf{d_i}}{\mathbf{d_0}} = \frac{\mathbf{S_i}}{\mathbf{S_0}}$$

Condenser lenses aperture

⇨ After passing through the condenser lenses aperture, of half-angle $\boldsymbol{\alpha}_{CL}$ (rd), $\mathbf{d_i}$ becomes $\mathbf{d_G}$, usually called the diameter of the gun.

CL aperture



As the condensers lenses decrease the initial beam intensity from $\mathbf{I_0}$ to $\mathbf{I_B}$ to downsize the beam diameter thus $\mathbf{d_G}$ is given by:

$$\mathbf{d_0} = \frac{\mathbf{2}}{\pi}\sqrt{\frac{\mathbf{I_0}}{\mathbf{B}}}\frac{\mathbf{1}}{\alpha_0} \Rightarrow \mathbf{d_G} = \frac{\mathbf{2}}{\pi}\sqrt{\frac{\mathbf{I_B}}{\mathbf{B}}}\frac{\mathbf{1}}{\alpha_{CL}}$$

⇨ As the brightness **B** is conserved along the electron optics, the beam diameter $\mathbf{d_G}$ is tuneable through the beam current $\mathbf{I_B}$ and the condenser lens aperture (half angle $\boldsymbol{\alpha}_{CL}$).

⇨ The final electron beam diameter $\mathbf{d_B}$ is also affected by the aberrations caused by the electron optics.

Electron optics

Introduction

⇨ As for light optics, electron optics is subjected to various aberrations that influence the beam diameter $\mathbf{d_B}$.

⇨ There are five possible isotropic aberrations as for light optics:

↳ spherical aberration

↳ astigmatism

↳ field curvature

↳ distortion

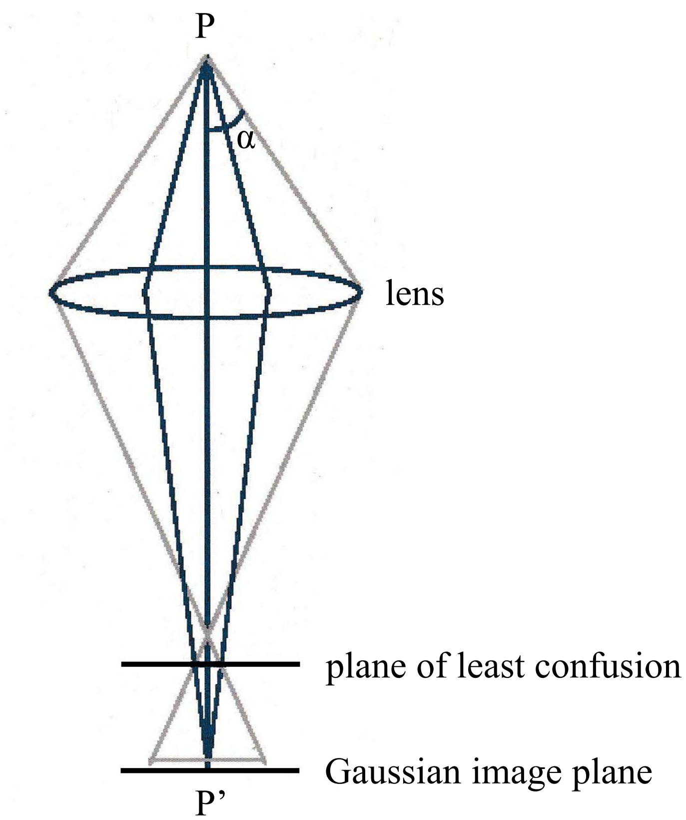
↳ coma

⇨ There is also anisotropic abberations, the most important are chromatic aberrations.

↳ These ones are largely due to an non-homogenous electron energy that could come from an insufficient stabilisation of the acceleration voltage, an energy spread of the electron gun or energy losses during the electron travelling though the optics or a combination of all.

⇨ Diffraction aberration has to be considered also because the electron beam passes through an aperture.

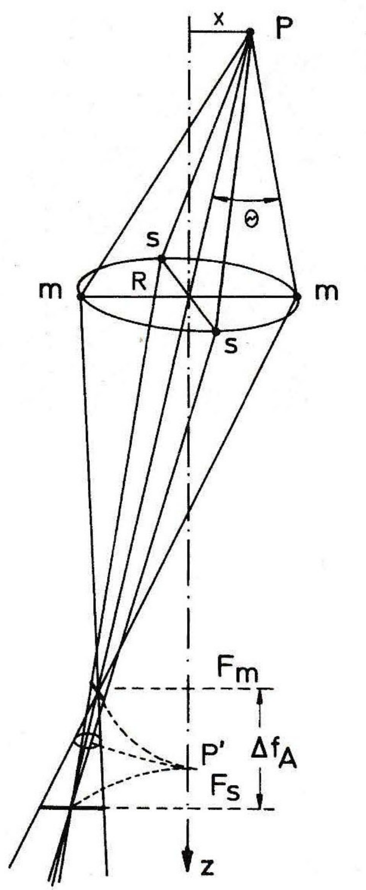
Spherical aberration



⇨ Spherical aberration has the effect of reducing the focal length for electrons passing through outer zones of the lens (gray path).

⇨ The Gaussian plane image is the position of the image when very small apertures are used (small **α**).

Astigmatism and field curvature

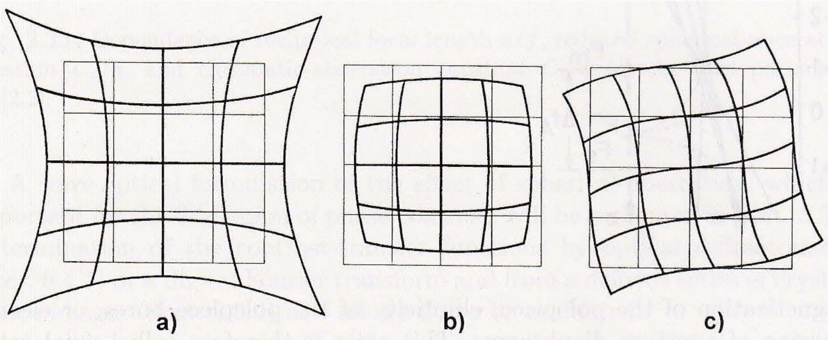


⇨ For astigmatism and field curvature, one can observe a shift from the propagation axis **x**.

⇨ This kind of deformation is minimalised when using small apertures (small **α**).

⇨ Small **α** values decrease also spherical aberration.

Distortion

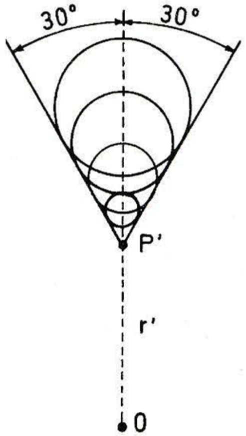


➩ Distortion causes a displacement in the Gaussian image plane.

➩ Distortion can be indirectly due to the spherical aberration.

Distortion of a square: pin cushion (**a**), barrel (**b**) spiral distortion (**c**)

Coma



➩ Here is presented the effect of coma on the image **P'** of a specimen point **P** situated at 0.

➩ The Gaussian image plane is situated at **r'**.

➩ The size of **P'** increases as a function of the distance between the Gaussian image plane and the detector.

Diffraction aberration

⇨ Diffraction aberration is due to the inherent wave nature of the beam.

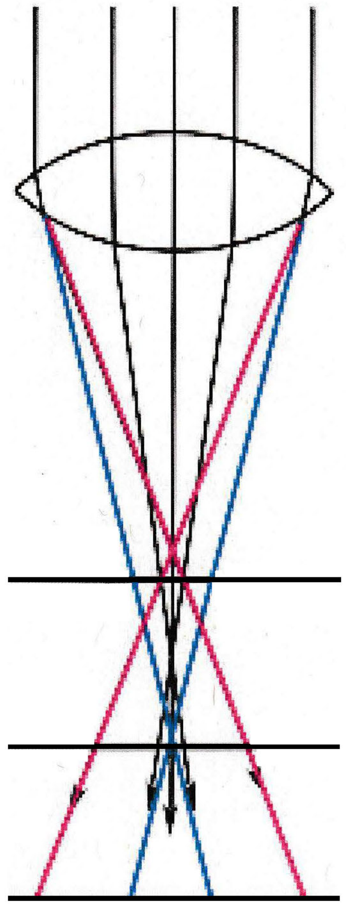
⇨ As a radiation passes through an aperture, it scatters or is said to diffract.

↳ The long wavelengths are the most impacted, so electrons are less affected by diffraction aberration than photons.

Chromatic aberration

⇨ Chromatic aberation occurs for non-homogeneous electron beams. The electron energy peak broadering **ΔE** involves a wavelength spread.

	thermionic gun		**field emission gun (FEG)**	
			S-FEG	C-FEG
Cathode material	W	LaB_6	W/ZrO	W
Energy spread **ΔE** at 100 kV (eV)	1.5 - 3	1 - 2	0.3 - 0.7	0.2 - 0.7



⇨ This phenomenon causes a variation in the focal length and influences $\mathbf{d_B}$.

Electron sources features

Introduction

⇨ In transmission microscopy, the objective lens is positioned after the sample. The diameter of the beam shone on the sample that is the diameter of the probe $\mathbf{d_P}$ is equal to $\mathbf{d_B}$.

↳ As the objective lens is positioned after the sample, the observed spot diameter $\mathbf{d_{spot}}$ on the detector is conditioned by the objective lens and is different from $\mathbf{d_P}$.

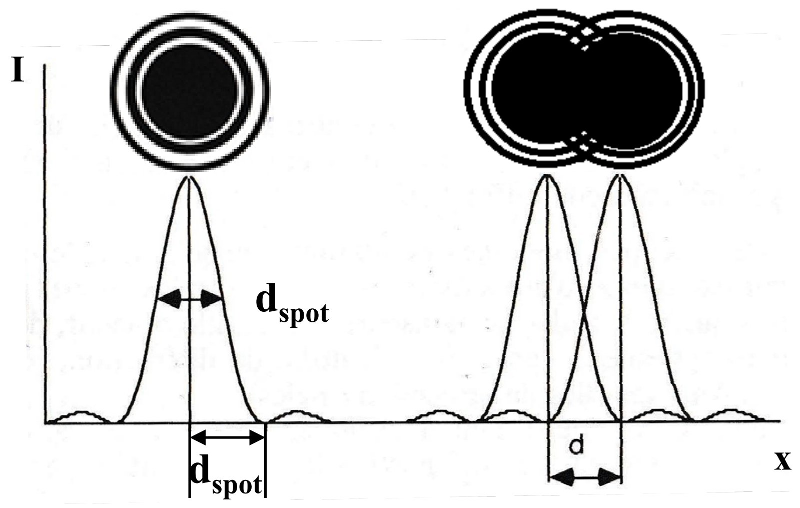
⇨ In reflexion microscopy, the objective lens is positioned before the sample. The diameter of the beam shone on the sample that is the diameter of the probe $\mathbf{d_P}$ is different from $\mathbf{d_B}$.

↳ As there is no optics between the sample and the detector, the observed spot diameter $\mathbf{d_{spot}}$ on the detector is equal to $\mathbf{d_P}$.

Electron beam spot

⇨ Because of the diffraction aberration caused by the presence of several apertures along the electron pathway, the electron beam's spot is made of confusions disks: a Airy pattern.

↳ The following scheme depicts an electron beam's spot represented by the intensity of the beam **I** as a function of the distance **x**.



↳ The spot size $\mathbf{d_{spot}}$ is taken at the full width of half maximum (FWHM) of the principal disk.

↳ Two spots are distinguishable when the distance between them is $\mathbf{d = d_{spot}}$.

↳ The lateral resolution of an electron microscope is $\mathbf{d = d_{spot}}$.

↳ $\mathbf{d_{spot}}$ depends on the electron source, *i.e.* $\mathbf{d_B}$ and on the series of lenses and apertures situated between the electron source and the detector.

The beam diameter d_B of the electron source

⇨ The electron beam diameter $\mathbf{d_B}$ is affected by the diameter $\mathbf{d_G}$ of the beam produced by the electron gun and by the electron optics aberrations.

⇨ Considering an electron spot as an electron density distribution which follows a Gaussian distribution, $\mathbf{d_B}$ results from the quadratic mean of all the contributions associated to the electron gun $\mathbf{d_G}$ and to the electron optics: the spherical aberration $\mathbf{d_S}$, the chromatic aberration $\mathbf{d_C}$ and the diffraction aberration $\mathbf{d_D}$:

$$\mathbf{d_B = \sqrt{d_G{}^2 + d_S{}^2 + d_C{}^2 + d_D{}^2}} \quad \text{with} \quad \mathbf{d_S = \frac{1}{2} C_S \alpha_{CL}{}^3 \;,\; d_C = \frac{\Delta E}{E_0} C_C \alpha_{CL} \;,\; d_D = \frac{1.51}{\alpha_{CL}\sqrt{E_0}}}$$

⇘ In these equations, $\mathbf{C_S}$ (nm) and $\mathbf{C_C}$ (nm) are the coefficients for spherical and chromatic aberrations respectively. $\mathbf{\Delta E}$ represents the energy peak broadening of the electron beam (see the table in slide 17, energy spread) and $\boldsymbol{\alpha_{CL}}$ is the CL aperture half angle.

$$d_B = \sqrt{\left(\frac{4I_B}{\pi^2 B} + \frac{2.28}{E_0}\right)\frac{1}{\alpha_{CL}{}^2} + \frac{1}{4}C_s{}^2\alpha_{CL}{}^6 + \left(\frac{\Delta E}{E_0}\right)^2 C_c{}^2\alpha_{CL}{}^2}$$

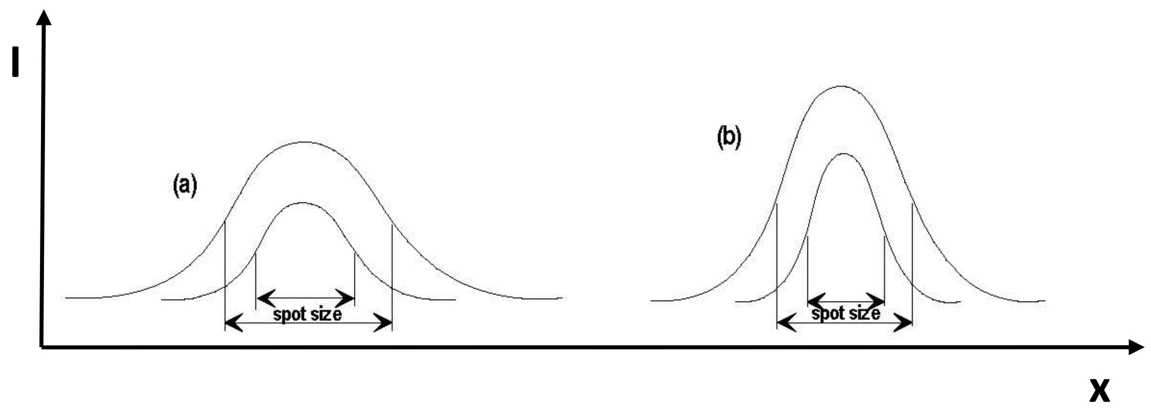
- Astigmatism which presents a ellipsoidal disk of confusion instead of a pure circle is not taken into account in the previous equation as there is no expression to qualify this aberration.

- The ultimate contribution of astigmatism to $\mathbf{d_B}$ is usually ignored assuming that the electron focusing system is not astigmatic or corrected. Small degree of astigmatism can be corrected with a stigmator.

- Working at a CL aperture half angle $\boldsymbol{\alpha_{CL}}$ from 10^{-3} to 10^{-2} rd leads to an optimal reduction of the influences of the aberrations but increases the influence of the electron gun on $\mathbf{d_B}$.

- To decrease the influence of the electron gun, one has to prefer a FEG source with a greater brightness **B**, $\mathbf{d_B}$ is then limited by both spherical and chromatic aberrations which can be corrected by using a suitable combination of lenses.

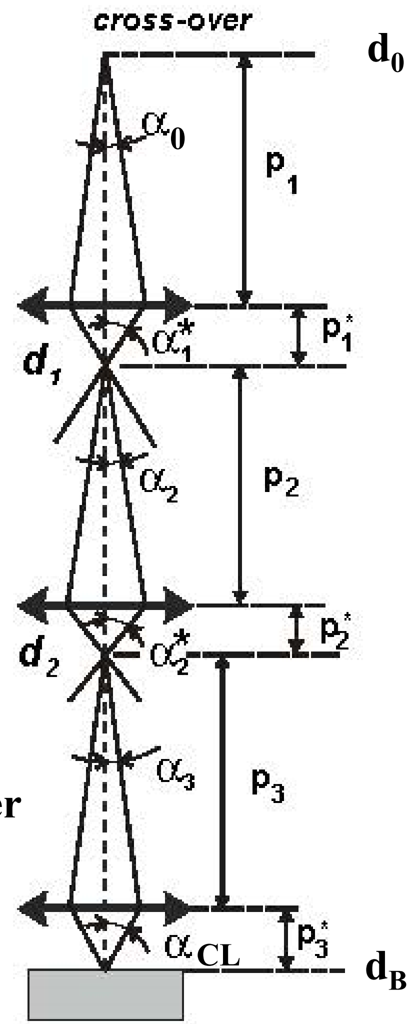
⇨ The influences of the beam intensity $\mathbf{I_B}$ and the brightness **B** of an electron source are presented below. Figure **b** stems from a brighter electron source than that of figure **a**.



↳ As shown on figures **a** and **b**, a beam intensity increase causes the broadening of the electron spot.

↳ As expected, the increase of the brightness of the beam (figure **b**) decreases the spot size.

The magnification of the electron column



⇨ The magnifications $\mathbf{m_1}$, $\mathbf{m_2}$ and $\mathbf{m_3}$ of the different condensers are:

$$\mathbf{m_1 = \frac{p_1^*}{p_1} = \frac{\alpha_0}{\alpha_1^*} \Rightarrow d_1 = m_1 d_0}$$

$$\mathbf{m_2 = \frac{p_2^*}{p_2} = \frac{\alpha_2}{\alpha_2^*} \Rightarrow d_2 = m_2 d_1}$$

$$\mathbf{m_3 = \frac{p_3^*}{p_3} = \frac{\alpha_3}{\alpha_{CL}} \Rightarrow d_B = m_3 d_2}$$

⇨ The magnification **M** of the electron column (**M << 1**) is then:

$$\mathbf{d_B = m_1 m_2 m_3 d_0}$$