Microstructural Characterization of Materials

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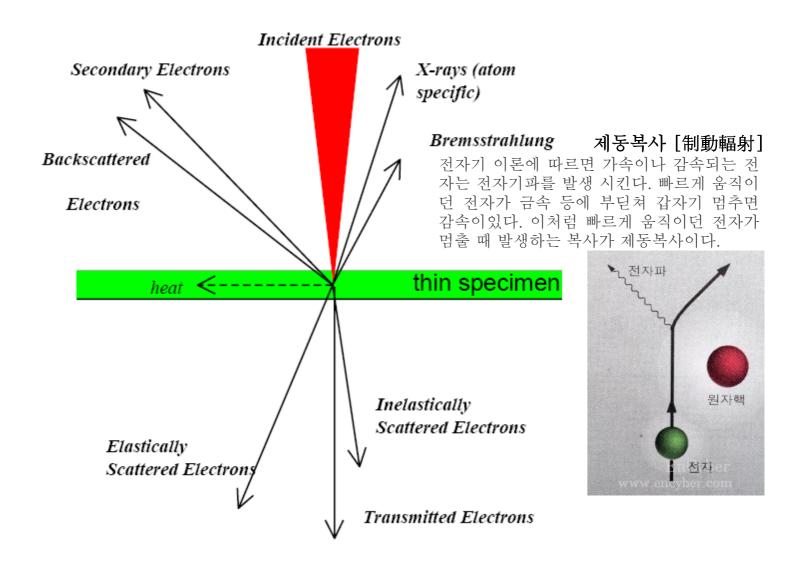
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Interaction of high energy (~kV) electrons with (solid) materials-II

Interaction with a thin specimen (TEM & STEM)

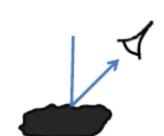


reminder

2 different approaches:



Backscattered and secondary electrons





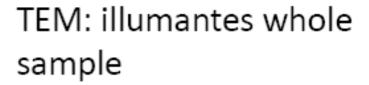


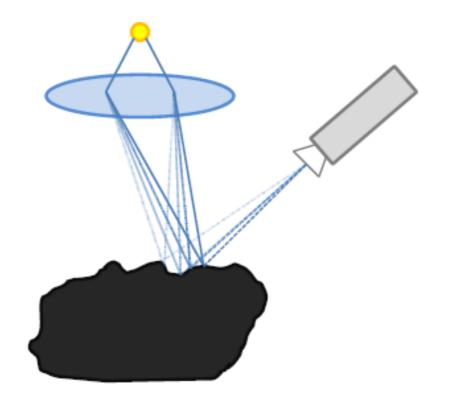
Transmitted electrons

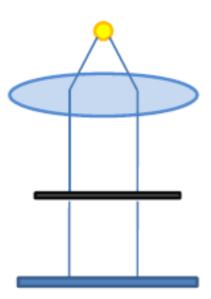


reminder

SEM: scans with a focused point







TEM Construction

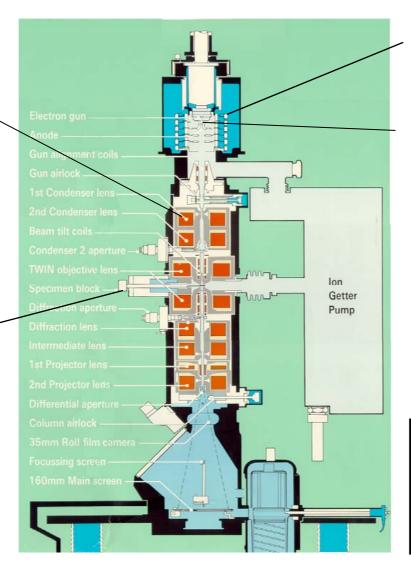
Cross-sectional view of CM12

Electro-magnetic lens

Plus many more Electromagnetic lens... Apertures...

Sample loadlock

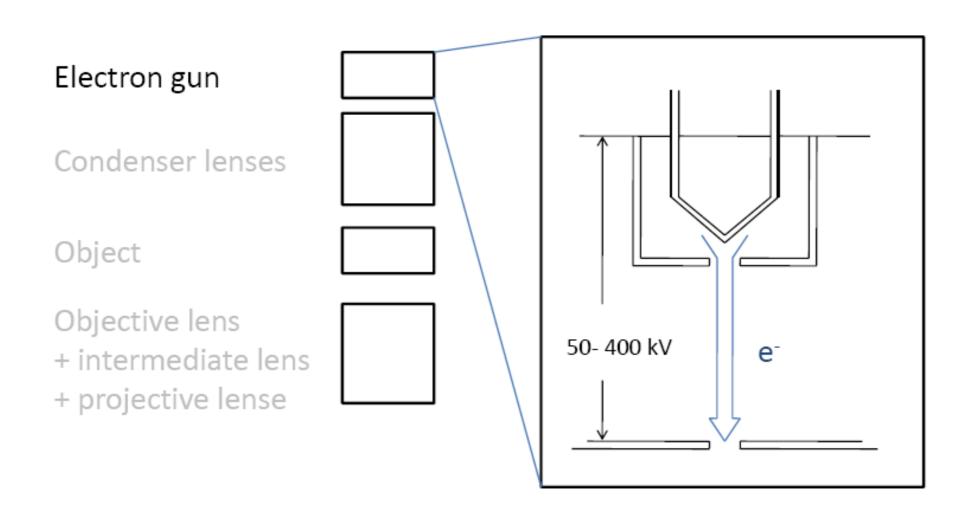
Energetic electron to visible light conversion



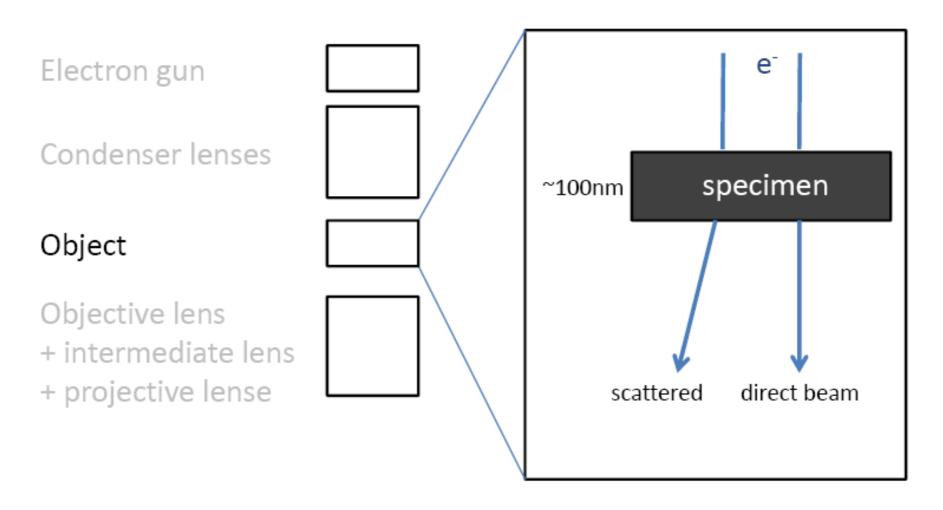
High voltage insulation

Electron Source

Image recording system



Electron gun Condenser lenses Object Objective lens + intermediate lens + projective lense



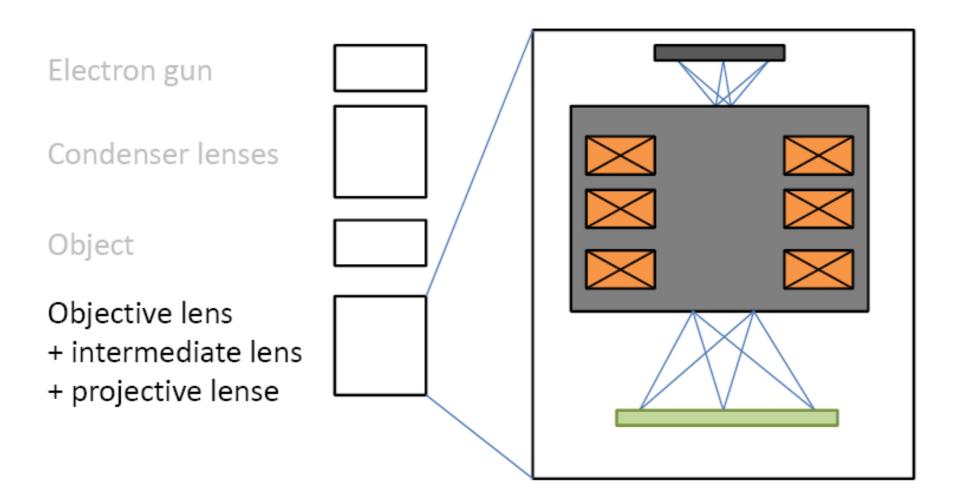
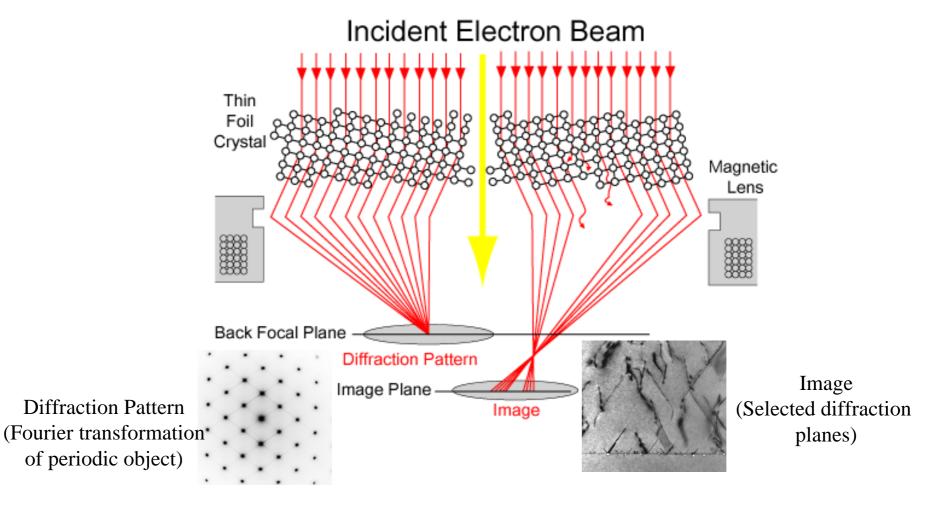
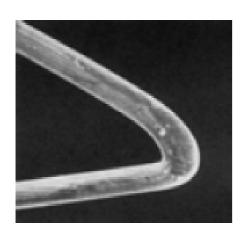


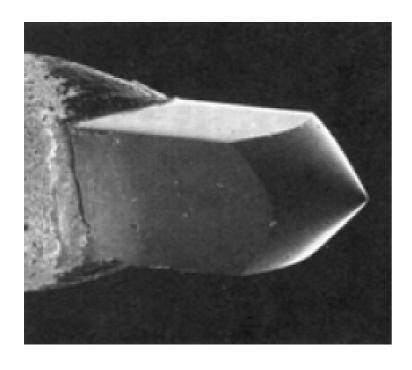
Image and Diffraction

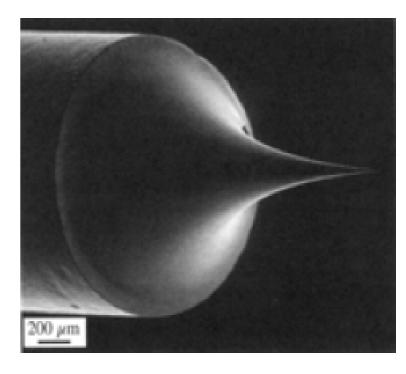


• Diffraction occurs from the planes nearly parallel to the beam direction

$$\lambda = 2d \sin \theta_B$$
, where $\theta_B < 0.5$ degree







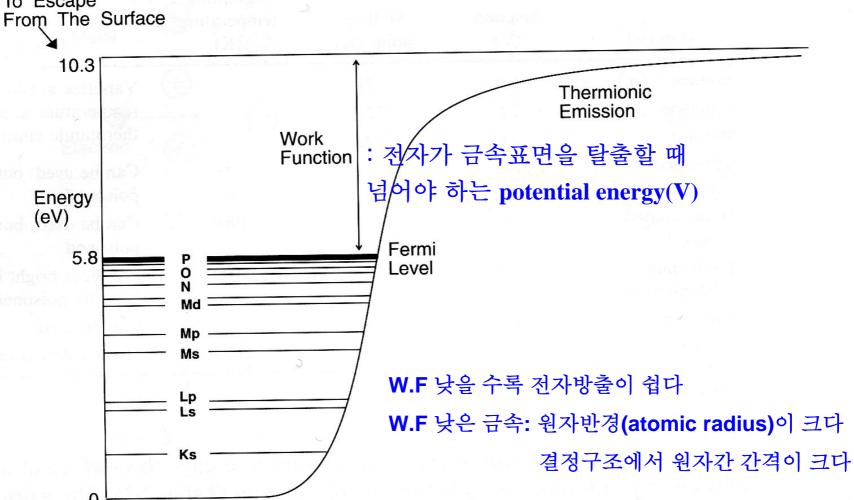
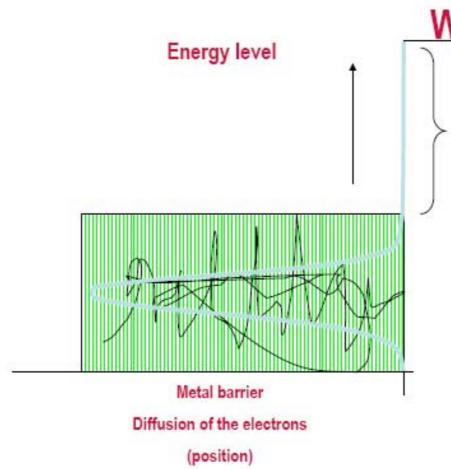


Figure 2-3 Thermionic emission curve of tungsten. Diagram of the energy of the orbital electrons of tungsten showing that the work potential is the difference between the energy necessary for the electrons to escape from the surface of the metal and the energy of the highest-orbital electrons.

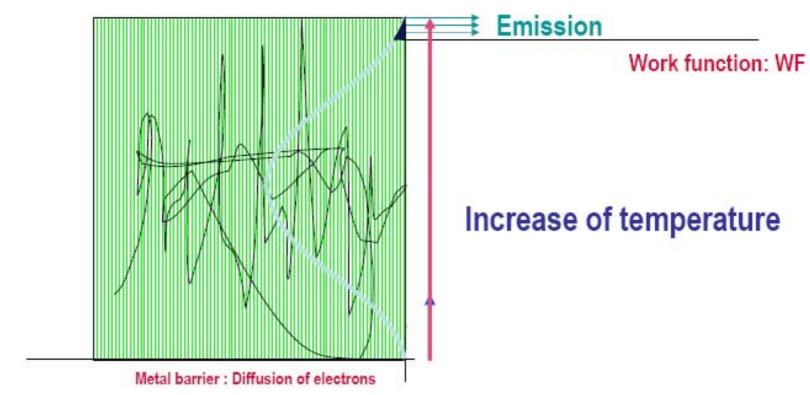
How to get electron emission?



Work function: WF

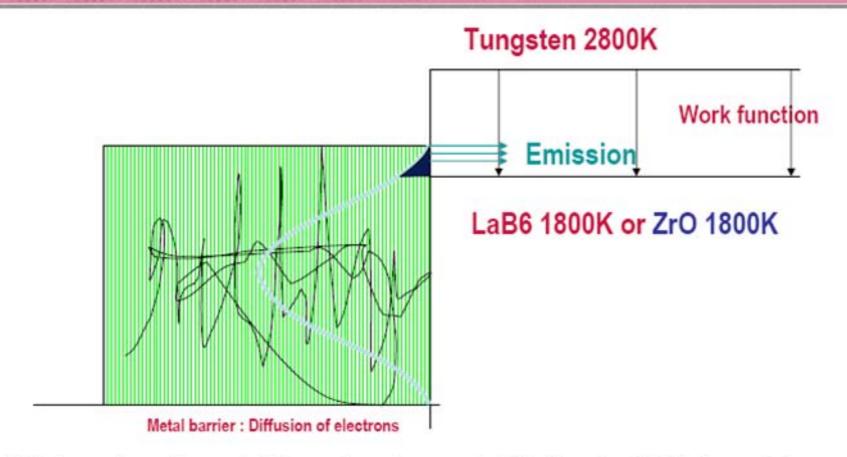
For emission this energy level is too short

Thermal Emission



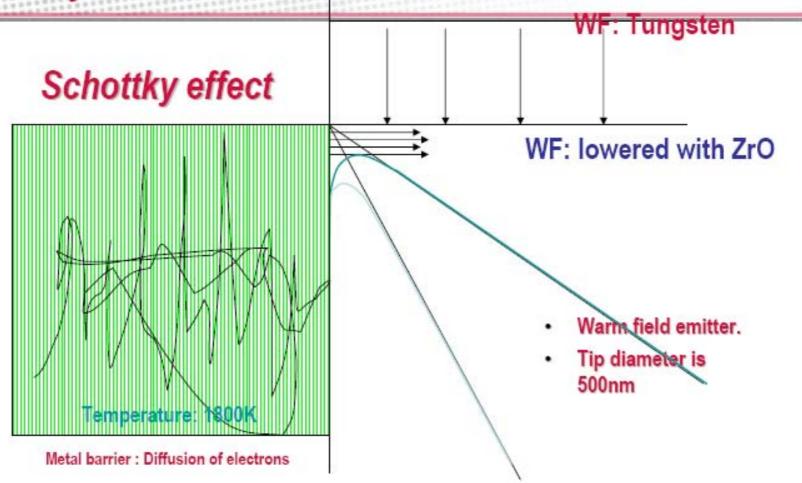
Electrons need to have more energy than the work function (WF) to leave the emitter. Their energy depends on the temperature, so heating can be used to cause emission (thermionic emitter like W or LaB6).

A lower work function



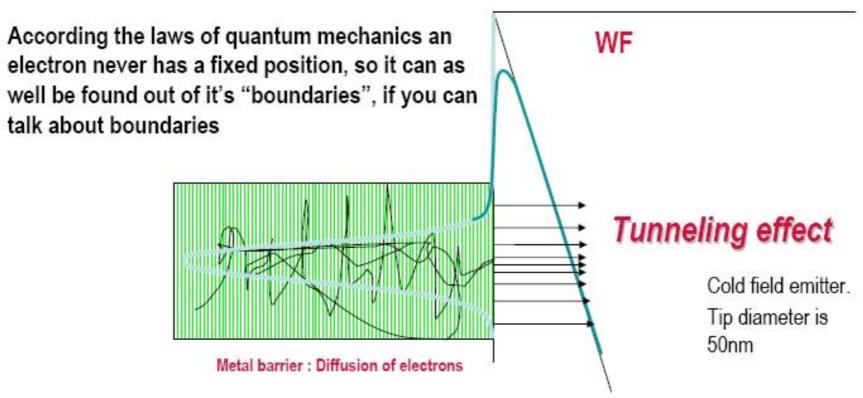
The WF depends on the material, so choosing a material with a low WF helps emisison (LaB6 or ZrO layer on FEG emitter). It also depends on the *crystal orientation* (FEG).

Schottky emitter



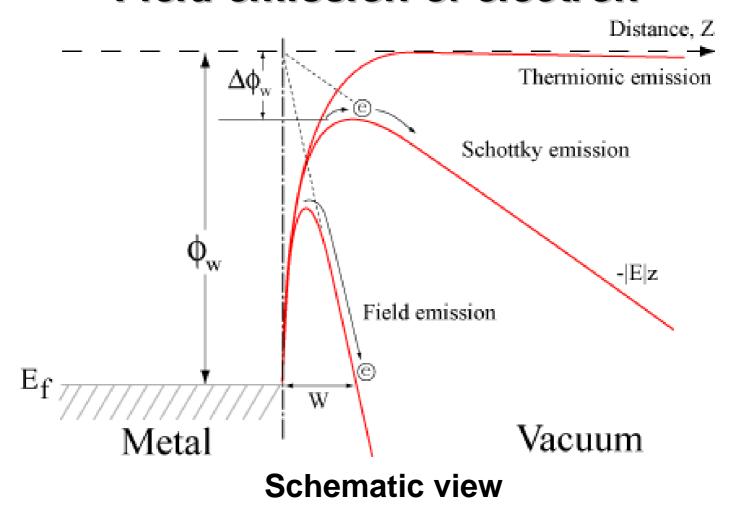
The Schottky effect is that the WF becomes lower when a strong electric field is applied on the emitting surface(used in SFEG).

Tunneling effect



Field emission occurs at even higher electric fields. In this case the electrons tunnel through the now very thin barrier(used in CFEG).

Field emission of electron



W 원자의 가장 높은 궤도의 전자들은 실온에서 energy barrier를 넘기 힘들다.

그러나 금속이 강한 전기장(electric field) 이 놓이면 궤도전자들은 potential barrier energy를 통해 tunneling해서 금속 표면에서 방출된다.

Cold field Emission

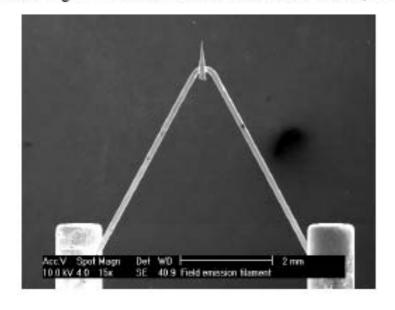
advantages: small spot size (1-2nm) → high resolution low △E → improving of low volt. Operation.

Tip 에 high electric field 를 주어 electron 을 방출

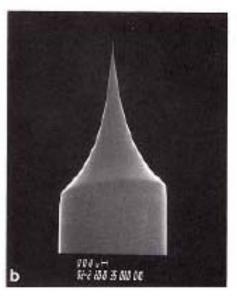
W<100> & W<310> single crystal 10e-10 torr 이상의 고진공

Flashing (burn out) 필요:

고진공 상에서도 tip 의 표면에 기체원자들이 흡착될 수 있어 emission 을 적게 만들므로 tip 을 고온으로 가열하여 흡착물을 제거 Flashing 후 안정화 까지 기다려야 하며, 또한 tip 수명 단축







Thermal Field Emission advantages: No flashing, brightness is similar to cold FE

W<100> single crystal

Continuous Heating (1300 ~ 2000K)

No Flashing

Lower vacuum 10e-9 torr

단 점 : energy spread 가 높음 (chromatic abberation 증가) (larger ΔE)

Schottky Field Emission

1800 K

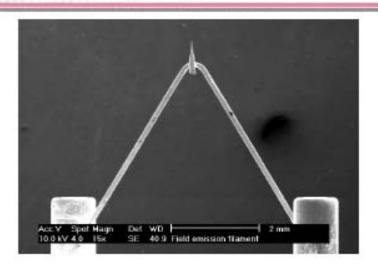
ZrO2 를 W 의 <100> 면에 코팅하여,

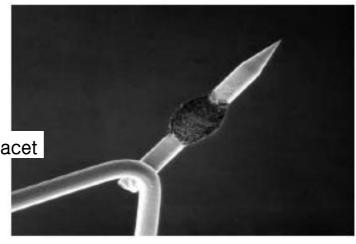
일함수를 4.5 Ev 에서 2.8 Ev 로 낮추기 때문에,

즉 schottky barrier 가 낮아지는 효과를 이용

장점:

good emission stability: flat emitting area on <100> facet good energy spread

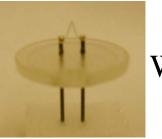




Electron sources

Table 2.1. Some features of electron sources

Emission	Thermionic	Thermionic	Schottky	Field emission
Material	W	LaB_6	$\rm ZrO/W$	W
Work function (eV)	4.5	2.7	2.5	4.5
Working temperature (K)	2800	1400-2000	1400-1600	300
Emission current density (A/cm^2)	1	10^{2}	10^{3}	10^{5}
Gun brightness $(A/(cm^2 \cdot sr))$	10^{5}	10^{6}	108	10^{8}
Crossover diameter (μm)	30	10	1	0.01
Energy width (eV)	1-2	0.5 - 2.0	0.3 - 1.0	0.2-0.4
Life (h)	~ 50	$\sim \! 1000$	$\sim 25,000$	$\sim \! 1000$
Vacuum requirements (Pa)	$10^{-2} - 10^{-3}$	$10^{-3} - 10^{-4}$	$10^{-7} - 10^{-8}$	$10^{-8} - 10^{-9}$



W



LaB₆



FEG

Basic electron optics

- Electrons and ions are <u>charged particles</u>, they can be accelerated in a E field.
- The trajectory of an accelerated charged particle can be deflected by E and/or B field.
- According to de Broglie, the accelerated (highenergy) particles also behave like waves.

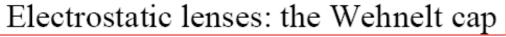
Provide means to (de)focus the electron beam on the specimen to focus the image,
 (3)to change the magnification, and to switch between image and diffraction

Round Lenses

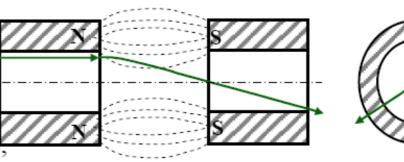
Magnetic lenses

▶ change the direction of electrons

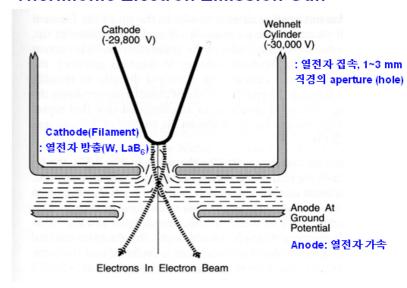
- ▶ magnifying (diverging) 발산
- ▶ diminishing (converging) 수렴
- condenser lenses, objective lenses
- intermediate lenses, projection lenses



- Advantage
- ▶ rotation free
- Disadvantage
- high precision in construction
- ▶ high precision in alignment
- extreme cleanliness

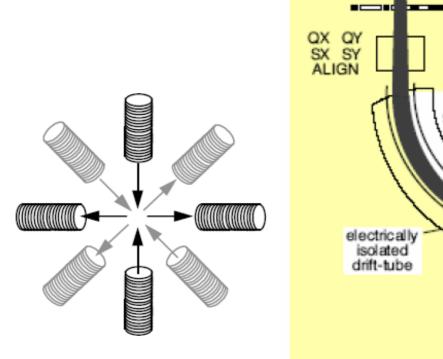


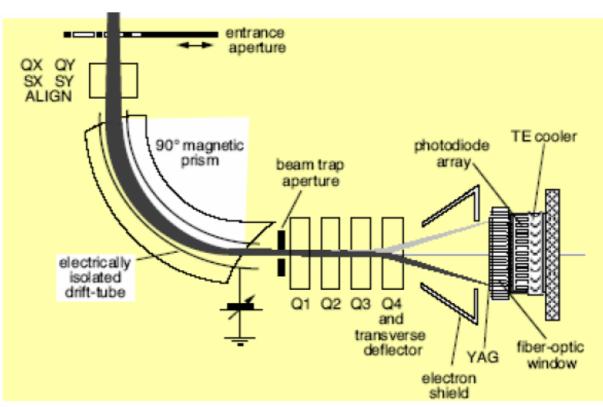
Thermionic Electron Emission Gun



Pole Lenses

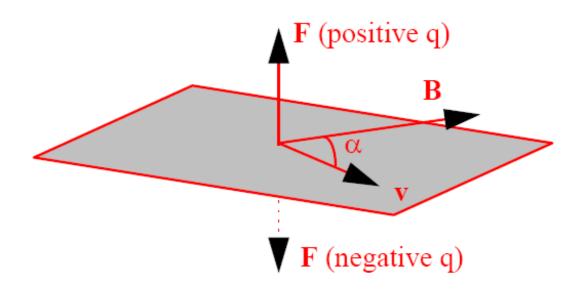
- ▶ Pole lenses are all electromagnetic, no electrostatic
- Different magnifying power in X, Y direction is possible
- The construction is just like the stigmators
- ▶ Usually seen in Cs correctors and EELS (Electron Energy Loss Spectroscopy)
- Qudrapole, Hexapole, Octupole lenses are common.





 Electromagnetic lenses are based on the fact the moving electrons are forced into a spiral trajectory, i.e. focused into one point

- Working Principle: Lorenz Force
 - electrons are only deflected by magnetic fields



Electromagnetic Lenses for Electrons

- Focus
- Magnification and demagnification
- Electron trajectory changed by magnetic field
- $\mathbf{F} = -\mathbf{e} \mathbf{v} \times \mathbf{B}$
- $F = evB sin\theta$
- If v // B, F = 0

$$R = \frac{m_0 v}{eB}$$

 the focal length is given by:

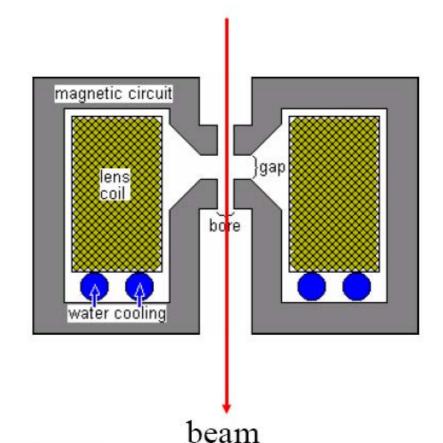
$$f = \frac{K \cdot U}{(N \cdot I)^2}$$

K : constant

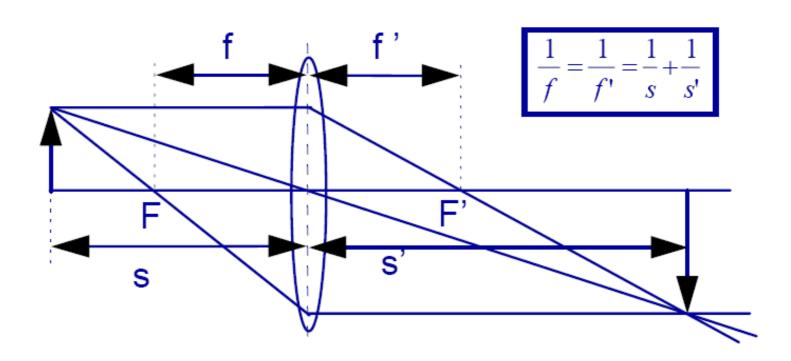
U : accelerating voltage

N : windings ≥=

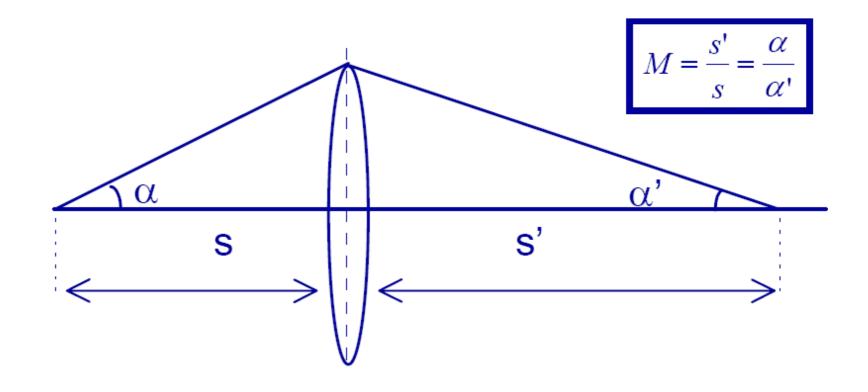
I : lens current



Gaussian Law



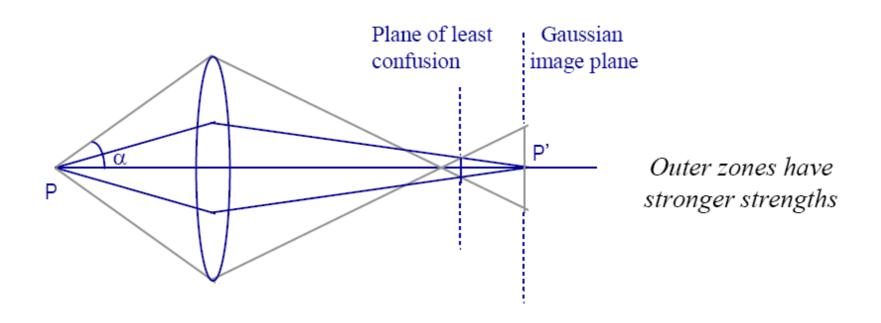
Gaussian Law



Lenses Spherical Aberration

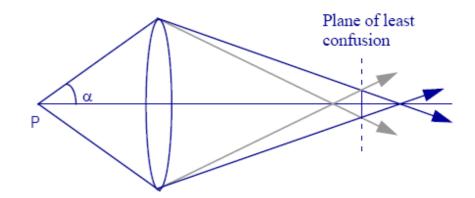
$$\delta_s = C_s \alpha^3$$

 Lens imperfections lead to different focal lengths in centre and at edges of lens



Lenses Chromatic Aberration

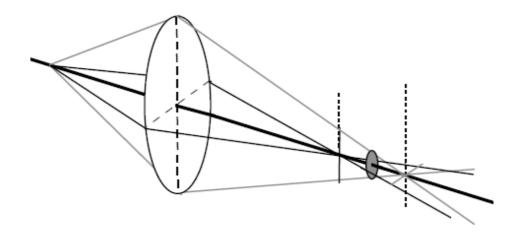
 Blurring due to energy spread in electron beam and lens current fluctuations



$$\delta_c = C_c \alpha \left(\frac{\Delta E}{E} + \frac{2\Delta I}{I} \right)$$

Lenses Astigmatism

Lens defect caused by magnetic field asymmetry



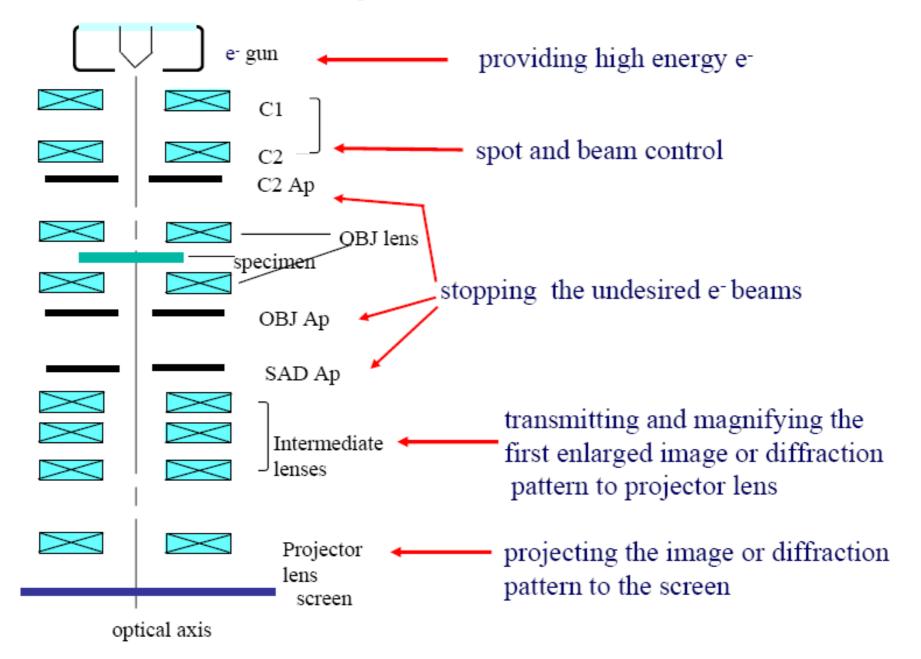
can be corrected using stigmators!

Lens System

- Condenser C1Lens
- Condenser C2 Lens
- Objective Lens

- Imaging Lenses (TEM)
 - diffraction (1st intermediate lens)
 - intermediate
 - projector

Lens System of TEM



Lens System Condenser C1 and C2

- C1
 - strong demagnifying lens
 - spotsize setting
- C2
 - weak lens
 - intensity control

Lens System & Microscope Resolution

- Microscope resolution is governed by: (for TEM)
 - wavelength of electrons
 - C_s of objective lens
 - other lenses are less crucial
 (α/M)

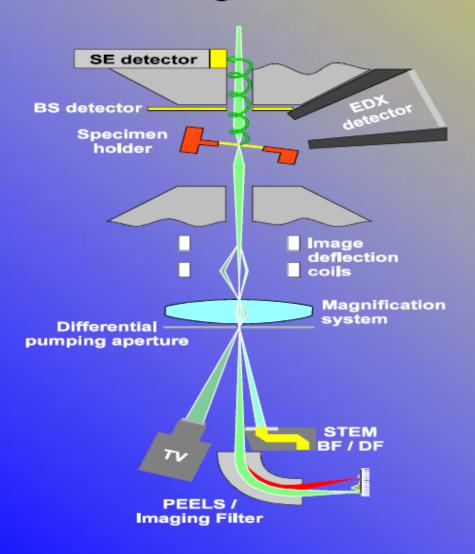
$$\mathcal{S} = 0.66 \times C_s^{1/4} \lambda^{3/4}$$

Stigmators

 Provide means to correct for deficiencies in the magnetic lenses

- EM stigmators:
 - At condenser, objective and diffraction lens (TEM)
 - At condenser, objective (SEM)
 - closely positioned to the lenses

Signals and Detectors



- In TEM
 - Energy Filter
 - TV / CCD camera
 - Plate camera
- In STEM
 - BF / DF
 - HAADF
 - BS & SE (SEM)
- In STEM and TEM
 - EDX and PEELS