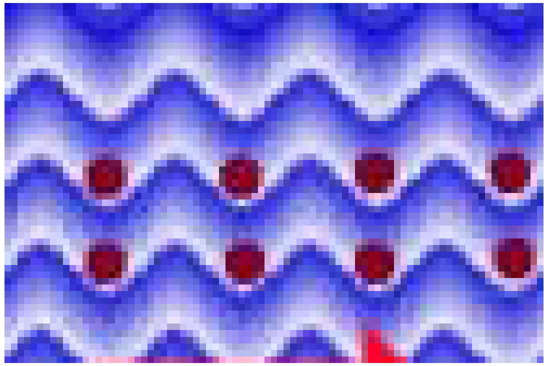
Chap 4. Scanning Electron Microscopy

- Seeing the Nano World: Because visible light has wavelengths that are hundreds of nanometers long we can not use optical microscopes to see into the nano world. Atoms are like boats on a sea compared to light waves.

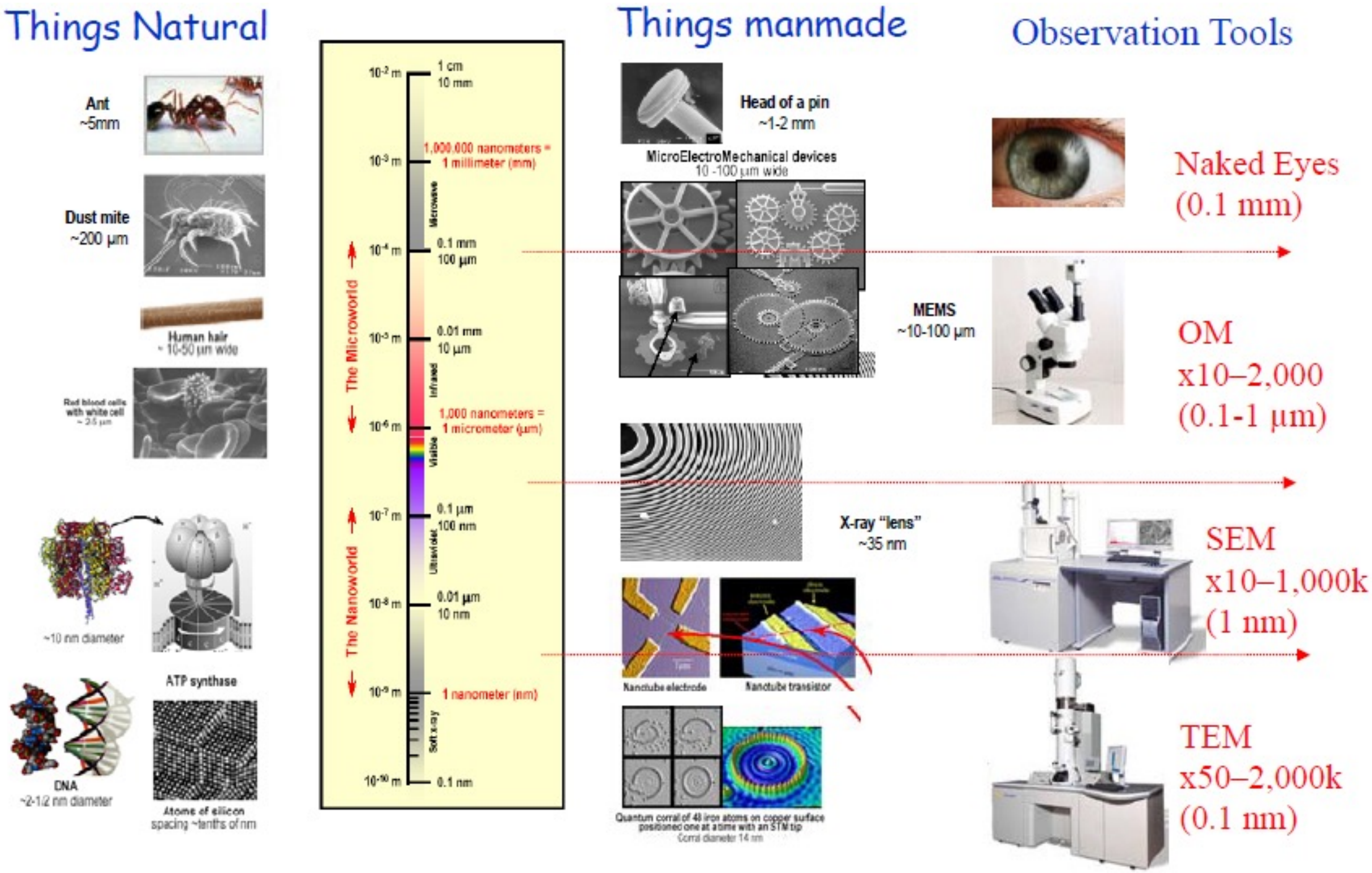


- The first electron microscope was produced by Max Knoll and Ernst Ruska in the 1930's.
- There are many types of electron microscopes. These include
  - TEM transmission electron microscope
  - SEM scanning electron microscope

Outline

- General introduction to Electron Microscopy
- Theoretical background
- Instrumentation
- How to get a good image? A Guide to SEM operation
- SEM Attachments (Composition analysis)

The Scale of Things



Observation Tools

Naked Eyes
(0.1 mm)

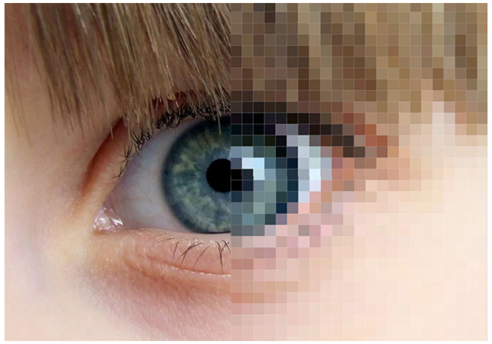
OM
x10–2,000
(0.1-1 µm)

SEM
x10–1,000k
(1 nm)

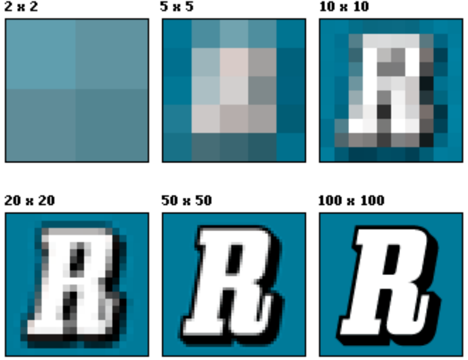
TEM
x50–2,000k
(0.1 nm)

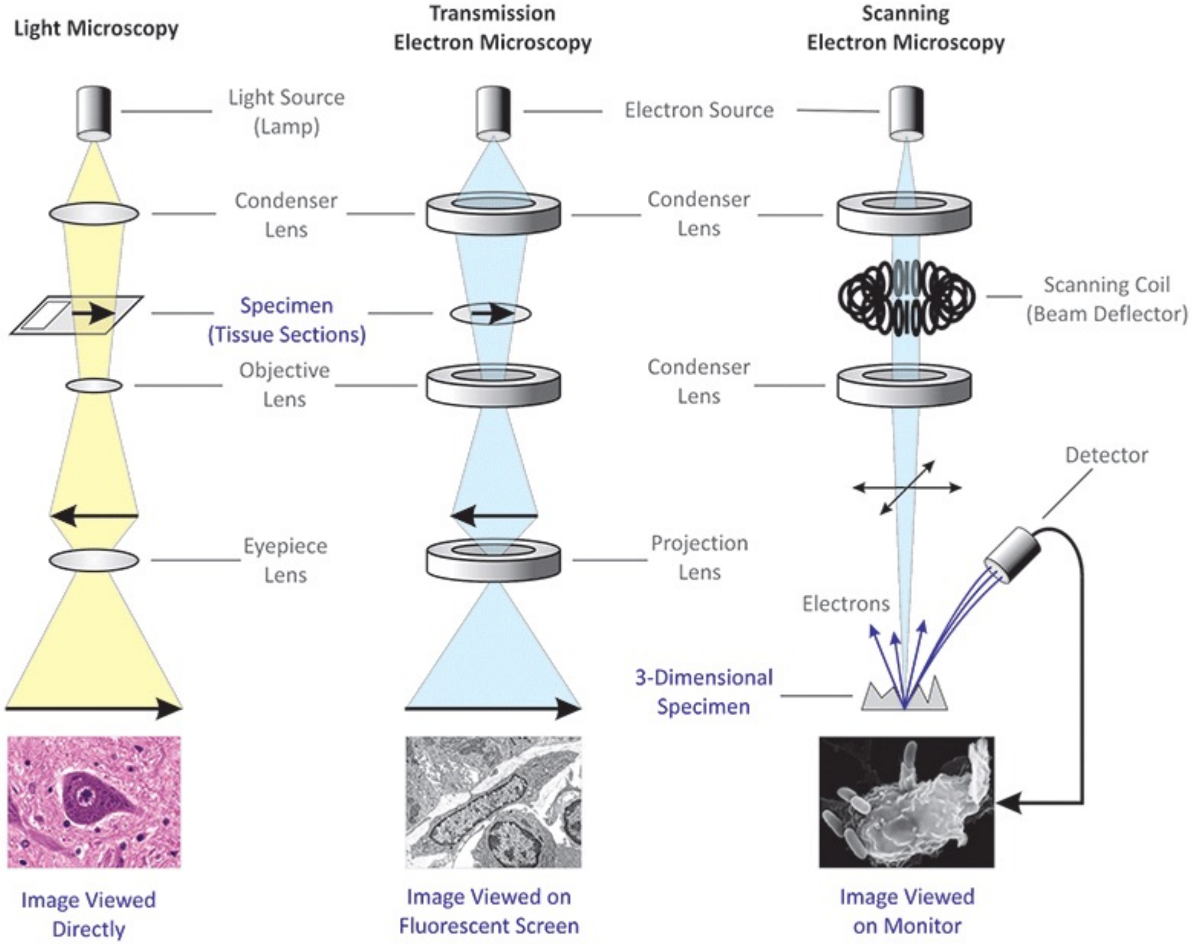
Imaging of Nanomaterials

Limits of human vision



Spatial resolution



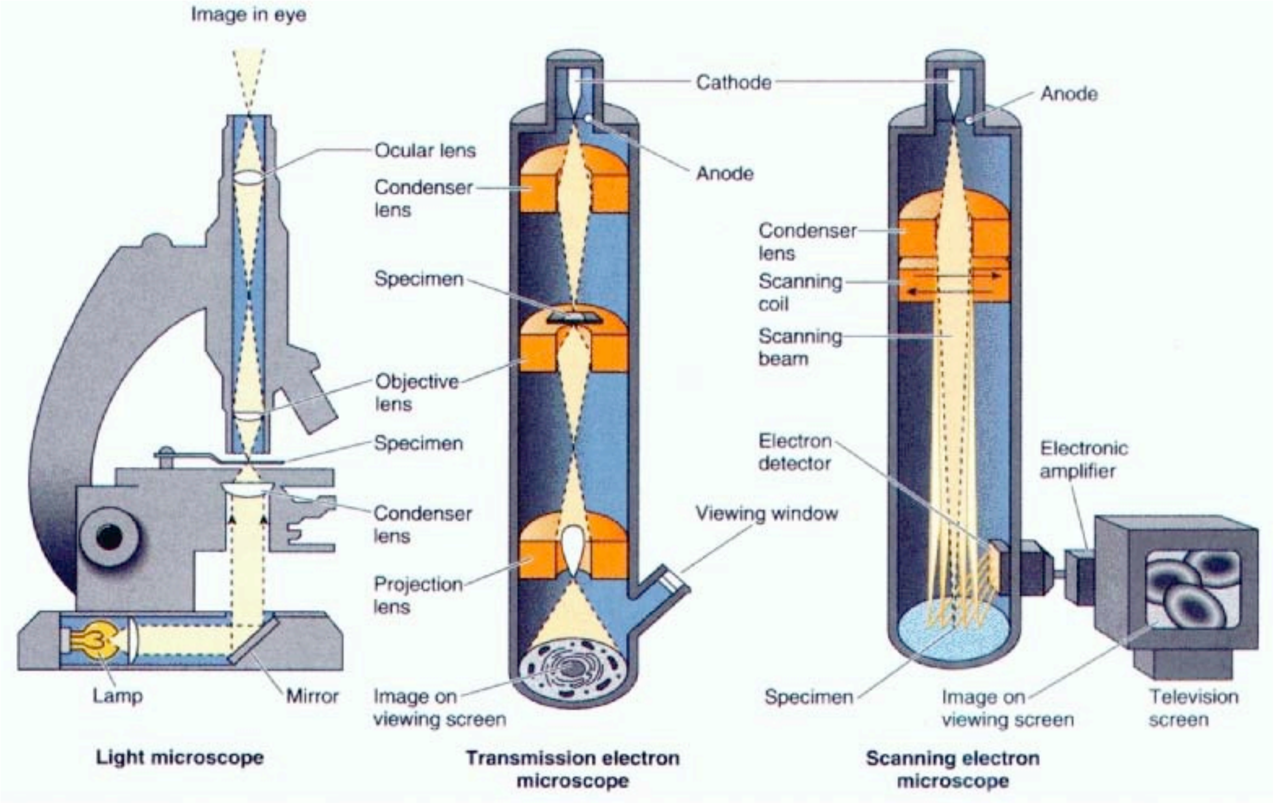


Comparison of Microscopy

OM
(Optical Microscopy)

TEM
(Transmission Electron Microscopy)

SEM
(Scanning Electron Microscopy)

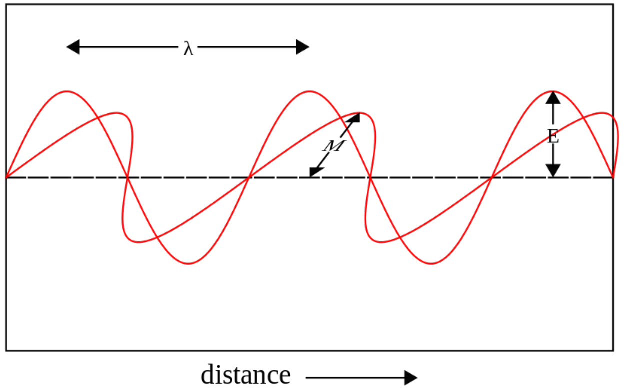


Comparison of Microscopy

	OM	SEM	TEM
Illuminating Beam	Light beam	Electron beam	Electron beam
Wavelength	200~750 nm	~0.086 nm (20 kV)	~0.0025 nm (200 kV)
Resolving Power	~0.1~1 μm	1 nm	0.1 nm
Magnification	10 ~ 2000 X	20 ~ 1,000,000 X	50 ~ 2,000,000 X
Medium	Atmosphere	Vacuum	Vacuum
Lens	Optical (glass)	Electromagnetic	Electromagnetic
Focal Length	Fixed	Variable	Variable
Dept of Focus	Small	Large	Large
Contrast	Reflection/ Absorption	Scattering/ Diffraction	Scattering/ Diffraction

Theoretical Background: Rayleigh’s Theory

- Light wave

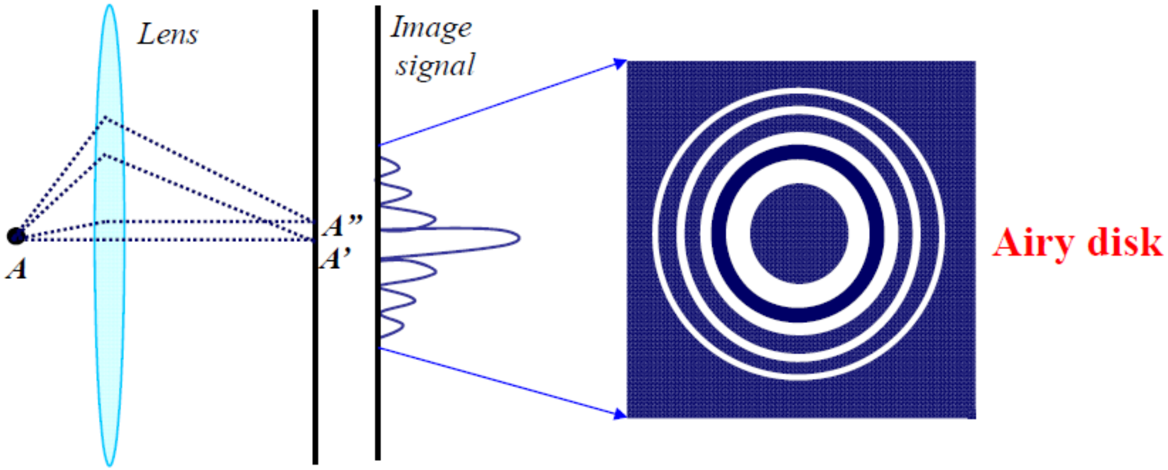


λ = wave length

E = amplitude of electric field

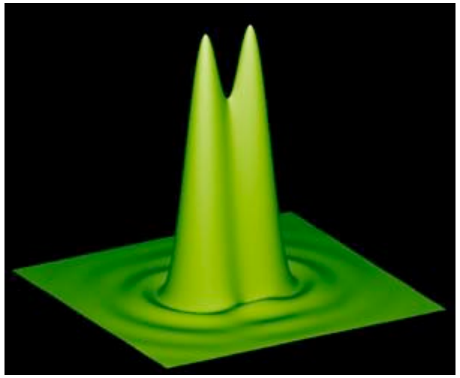
M = amplitude of magnetic field

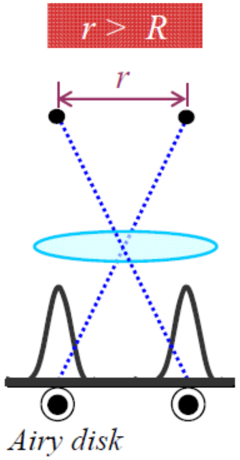
- Light diffraction



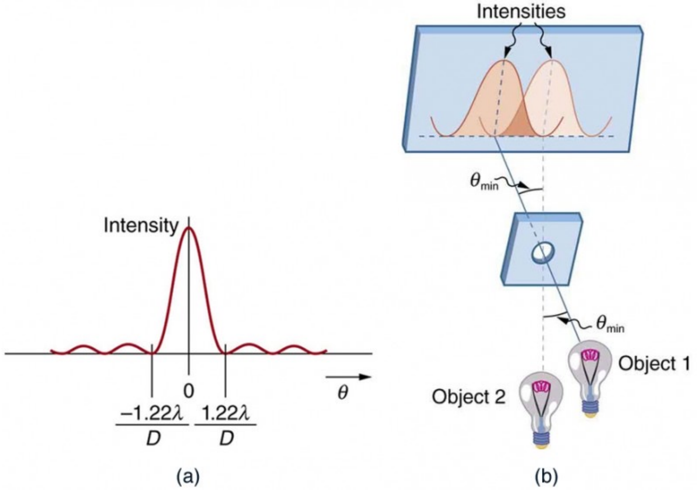
Theoretical Background: Rayleigh's Theory

- Reyleigh Limit

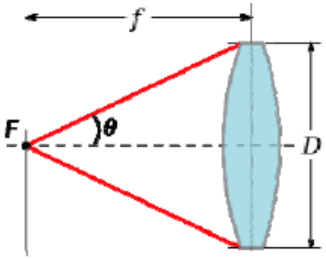




R: Resolution limit → **Rayleigh Limit**



- Resolution of Optical Microscopy (R)



$$R = \frac{0.61\lambda}{N_A} = \frac{0.61\lambda}{n_0 \sin\theta} \qquad \frac{0.61}{n_0 \sin\theta} \cong 0.5 - 1 \quad \text{and} \quad \lambda_{min} \cong 400 \sim 800\ nm$$

- λ : wavelength of light
- n_0 : index of refraction
- θ : convergence angle

➡ $R \geq \sim 200\ nm$

This is termed Far Field Optics!

(numerical aperture)=R/f

Theoretical Background: De Broglie’s Theory

- Wave-particle duality

:All matter (any object) has a wave-like nature

$$\lambda = \frac{h}{p} \qquad p = mv$$

- ➢ λ : wavelength of light
- ➢ h : index of refraction
- ➢ p : convergence angle

- Resolution of electron microscope

$$R \cong \frac{\lambda}{\beta_{objective}}$$

$$\lambda = \frac{h}{p} = \frac{h}{mv} \leftarrow E_{kinetic} = eV = \frac{1}{2}mv^{2}$$

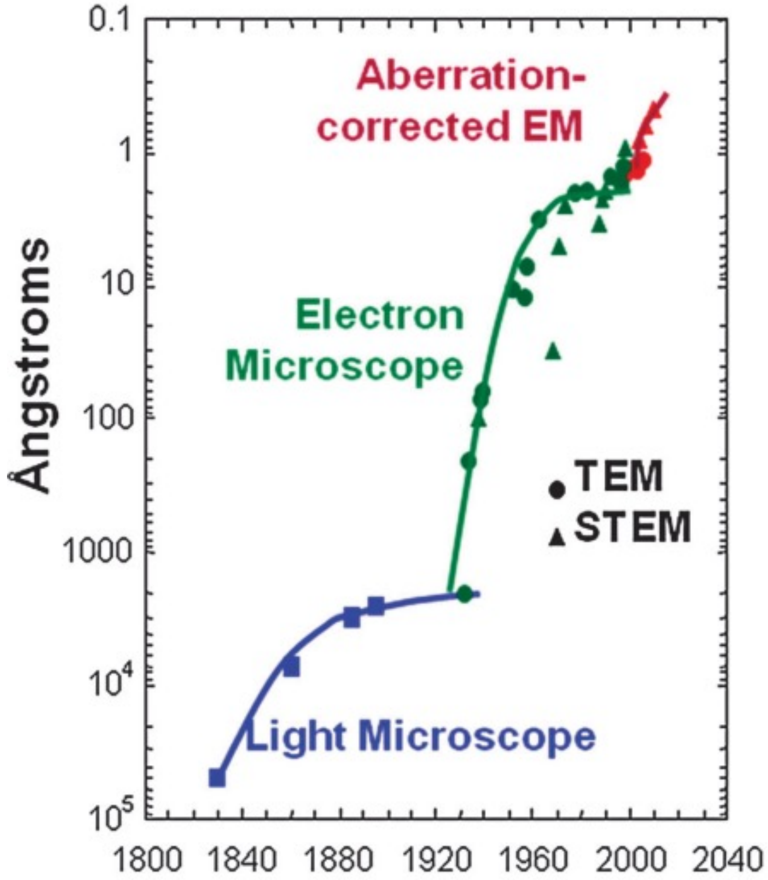
$$= \frac{h}{\sqrt{2meV}} \cong \sqrt{\frac{150}{V}}\,[\text{Å}]$$

Wave of electrons in the electron microscopy !!

If $V = 150$ V, then $\lambda = 0.1$ nm
Resolution is limited by the lens defects
If $\lambda = 2$–10 Å, then $R = 1$–3 Å (atomic resolution limit)

Why Imaging by Electron?

	Photon	Electron
Energy	2 eV	10 keV
Wavelength	600 nm	0.012 m,
NA	1.2	0.01
Resolution	> 500 nm	~ nm



- Spatial resolution versus year for microscopes

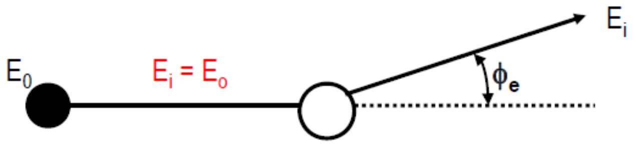
Elastic and Inelastic Scattering

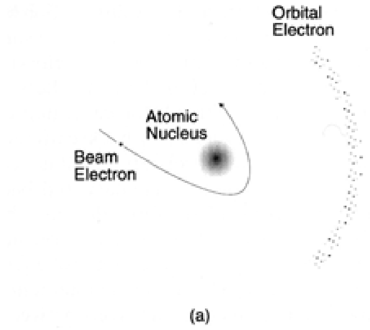
- Elastic scattering: backscattered electron, diffraction

  : results from the energetic electrons with the nuclei of the atoms, partially screened by bound electron

  : affect the trajectories of the beam electrons

  : doesn't alter significantly the electron energy



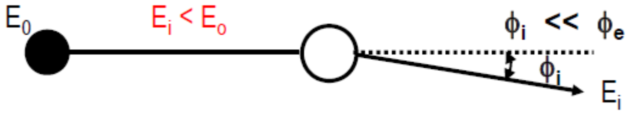


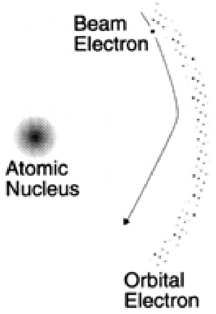
- Inelastic scattering: Secondary electron, Auger electron, X-ray, CL

  : result in a transfer of energy to the solid

  : limit the range of travel of the electron

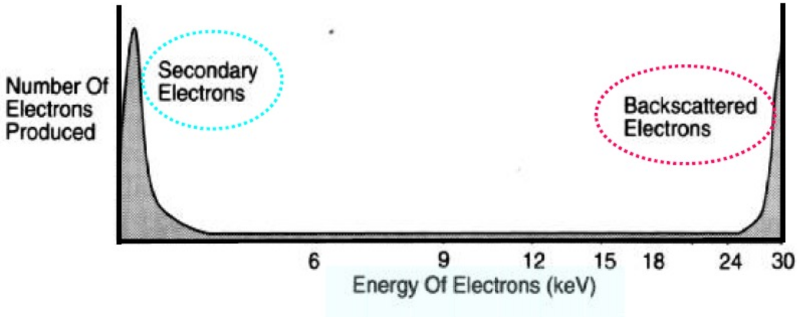
  : excitation of conduction electrons leading to secondary electron emission

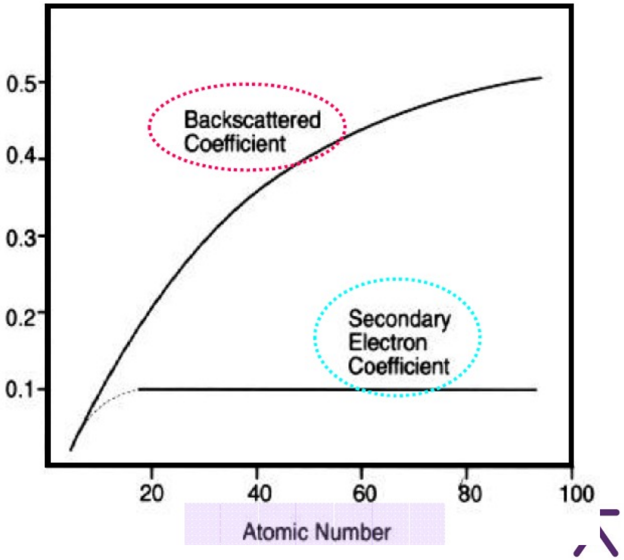




Backscattered Electrons & Secondary Electrons

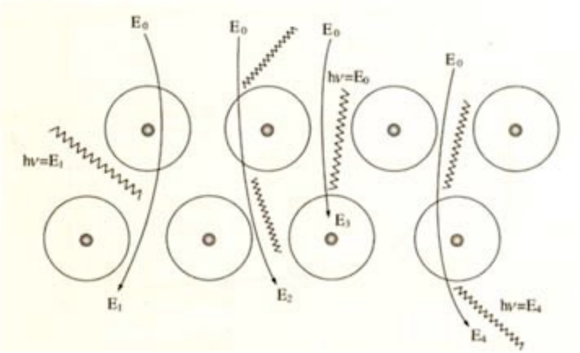
- Backscattered Electrons
  - Produced by interaction between incident electrons and nucleus
  - Brightness directly related to mass of elements
  - **Compositional information**
- Secondary Electrons
  - Produced by energy loss from deflected electrons (Energy less than 10-50 eV)
  - Signals from nanometer layer
  - Edges look bright, flat planes look dull
  - **Morphological information**



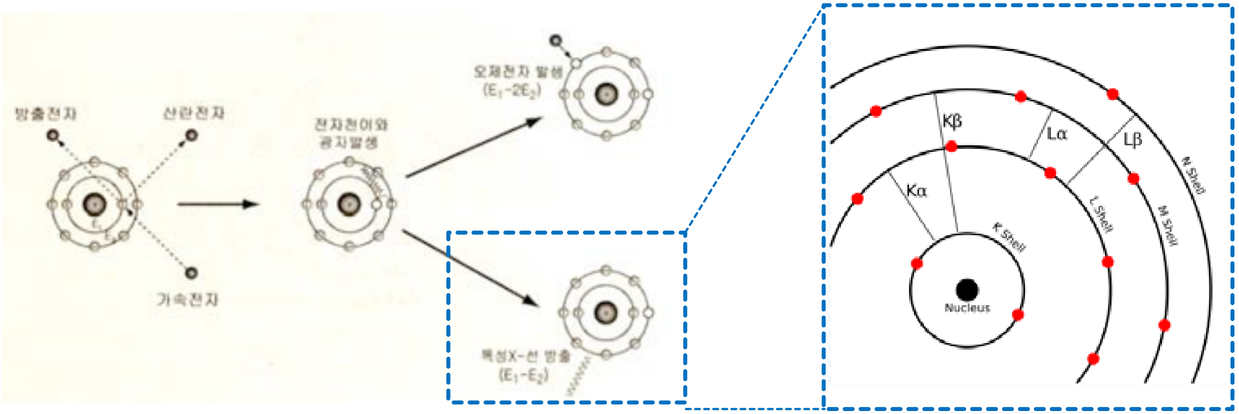


X-Ray

- X-rays
  - Produced by inelastic collision between primary beam electrons and atoms approximately 1000 nm deep within the specimen
  - Continuous X-ray
  - Characteristic X-ray: **elemental Analysis**

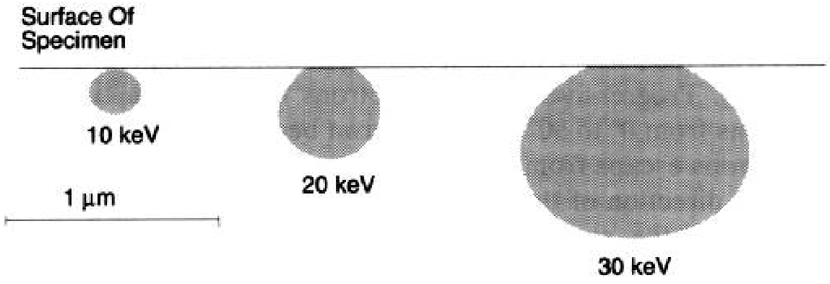


Continuous X-ray

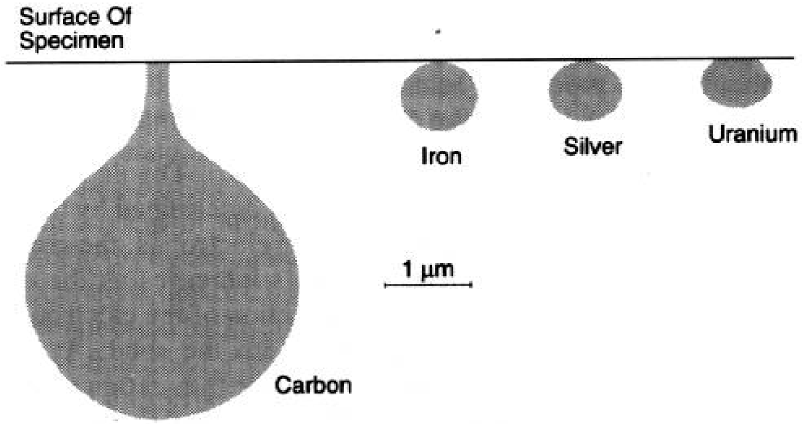


Interaction Volume

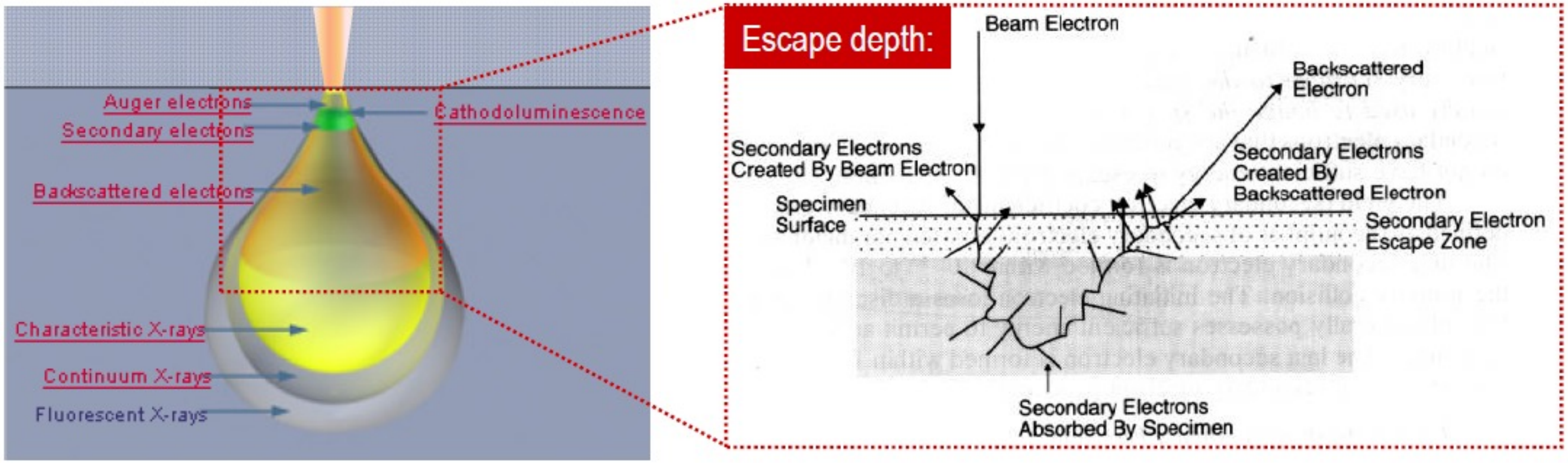
- Acceleration Voltages



- Effect of atomic numbers

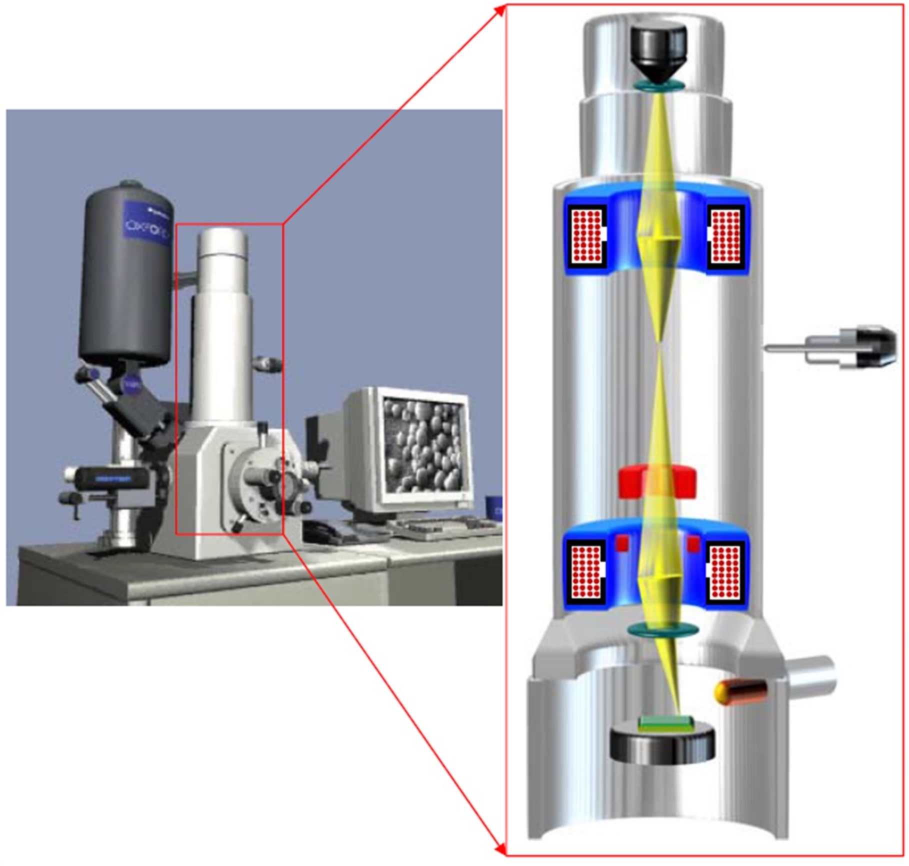


Depth of Generation of Various Quanta and their Spatial Resolutions



Signal / Mode	Purpose of SEM	Information
• Secondary electrons (SEI)	Topographical observation of surface	Morphology
• Backscattered electrons (BSE)	Compositional observation of surface	Atomic composition
• X-rays (EDS or WDS)	Elemental analysis of specimen	Atomic number
•Cathodoluminescence (CL)	Internal Characteristics observation	Bandgap, impurities

Instrumentation: Structure of SEM



Column

- Electron Gun
- Optic
  - Condenser Lens (CL)
  - Apertures
  - Scanning coils
  - Objective Lens (OL)

Chamber

- Detectors
- Specimen Stage

진공장치

- Vacuum system

영상처리장치

- Display Screen

제어장치

- Electronics and Controls

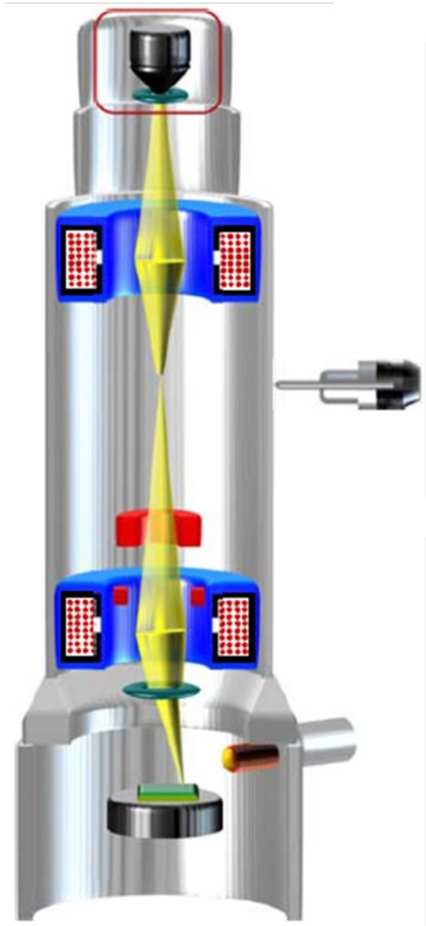
SEM in YouTube

http://www.youtube.com/watch?v=bfSp8r-YRw0&feature=related

http://www.youtube.com/watch?v=lrXMIghANbg&NR=1

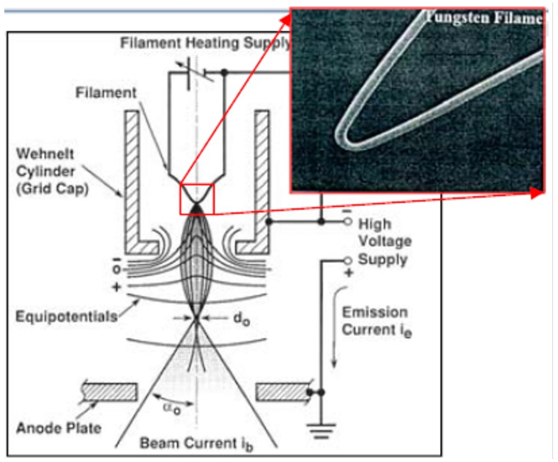
Pt 1~6 of 6
http://www.youtube.com/watch?v=c7EVTnVHN-s&NR=1

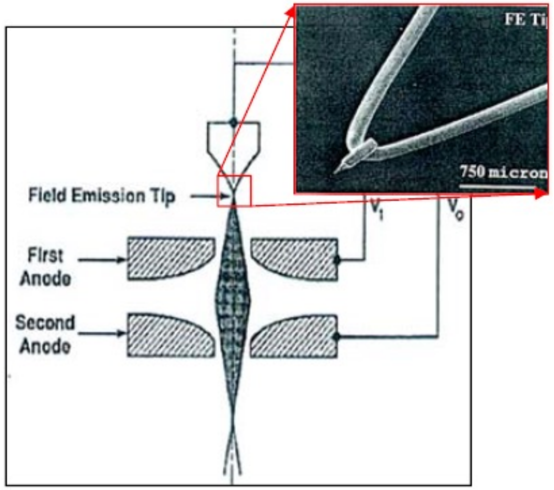
Electron Gun



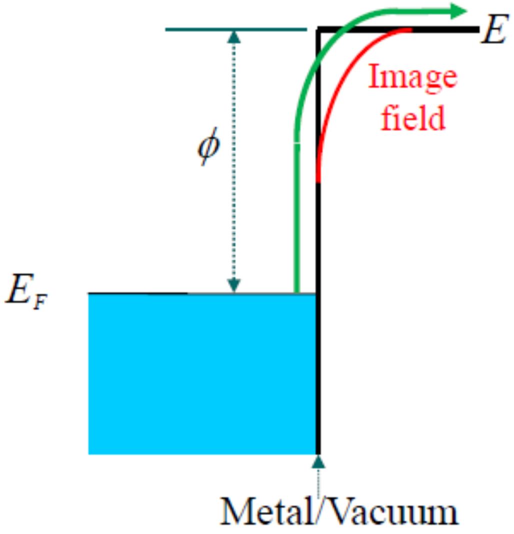
- Thermoionic emission gun:
- Electron beam is produced by heat emission.
- Low cost & Simple cathode
- Minimal Vacuum 10^{-4} torr

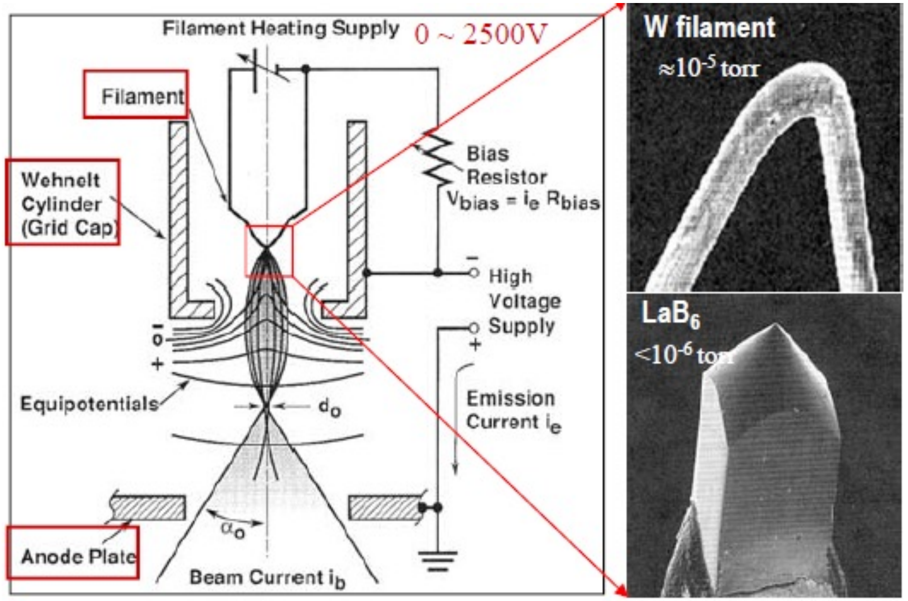
- Field emission gun:
- Electron beam is produced by electron tunneling.
- Sharp tip point
- Several order brighter
- Ultra high vacuum 10^{-10} torr
- Cold/Thermal/Schottky FE





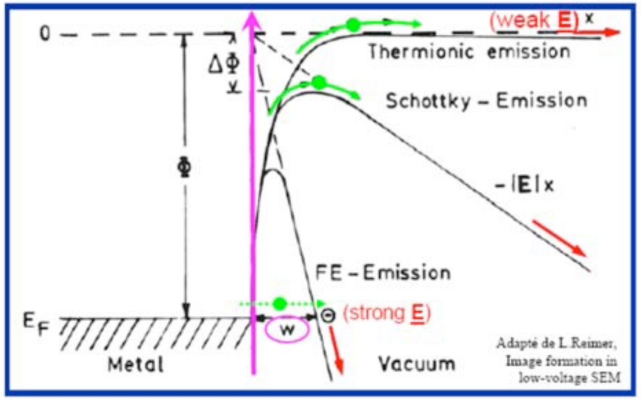
Electron Gun: Thermionic Emission



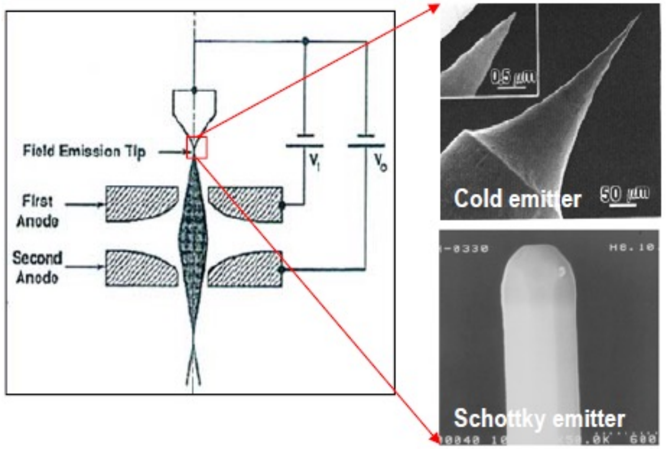


Low work function High melting point Stable in mild vacuum	Materials	EW (eV)	T(operation) (K)	T(melt) (K)
	W	4.5	2700	3643
	LaB6	2.5	1800	2800

Electron Gun: Field Emission Gun (FEG)



Adapté de L Reimer, Image formation in low-voltage SEM



- Field emission gun: Cold emitter

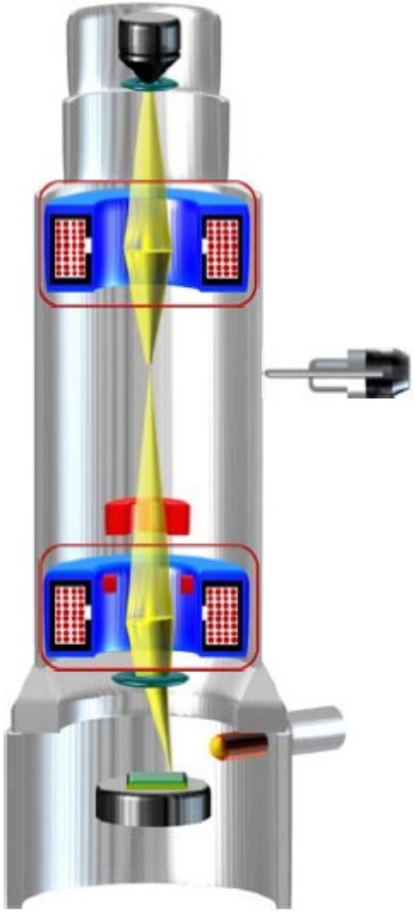
  Electrons "tunnel" through the potential barrier (w) when the applied electric field **E** reaches some 10^9V/m (quantum mechanic effect)

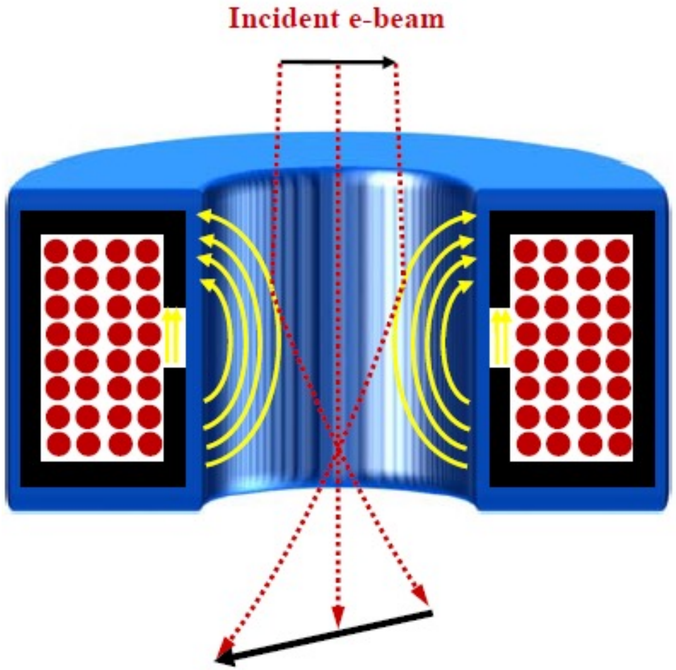
- Field emission gun by Schottky effect: Schottkey emitter

(intermediate case between thermionic and field emission):

No tunneling any more (W is too large): emission is similar to the thermionic gun, but lowering $\Delta\Phi$ of the barrier height by the electric field (Schottky effect) The cathode is also enhanced by adding ZrO_2 to lower the value of Φ.

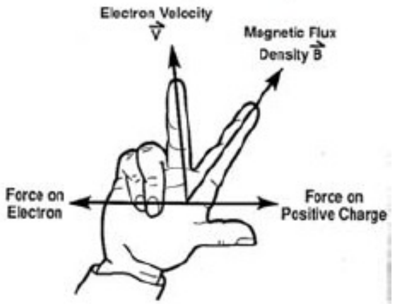
Electromagnetic Lens



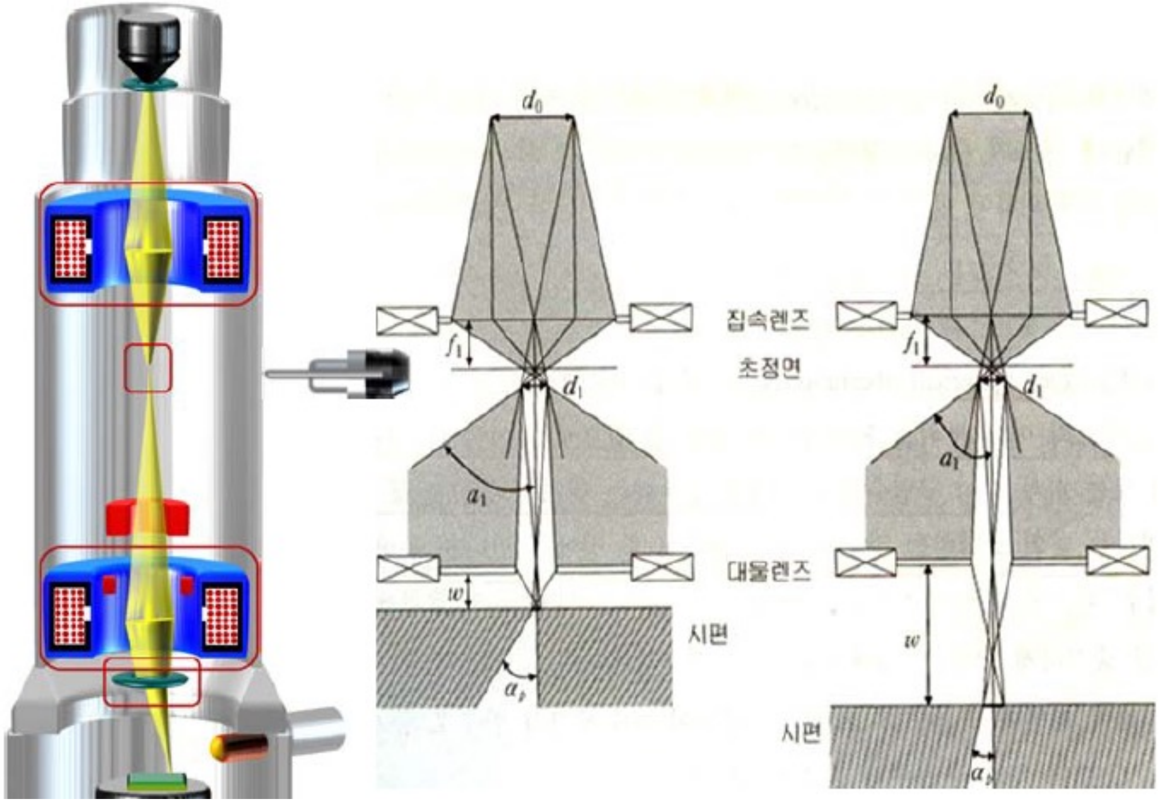


Lorentz force
by magnetic flux

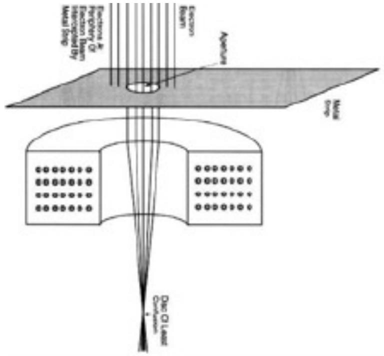
$F = -e(v \times B)$



Electromagnetic Lens: Ray Traces and Apertures



- **Aperture**

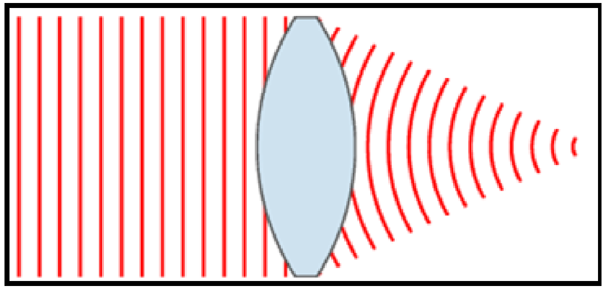


이탈하는 전자빔 제거
전류량 제한
전자빔 집광

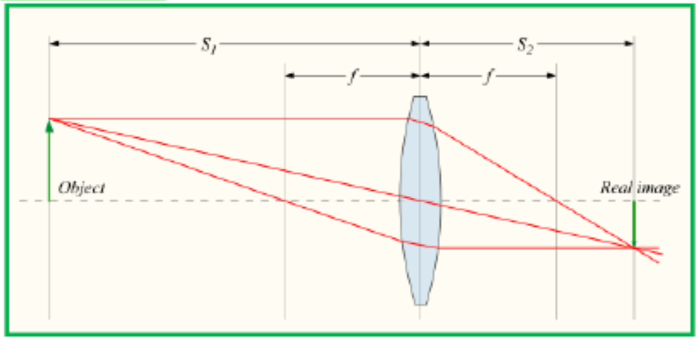
The aperture decreases the beam angle a1 diverging from the condenser lens to a smaller angle α_a for the electrons emerging the OL

Electromagnetic Lens Defects: Aberrations

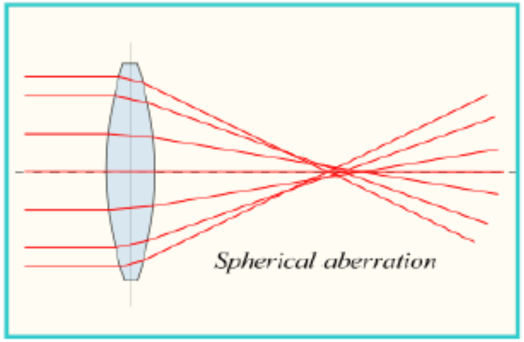
Lenses can be used to focus light. Ideal lens



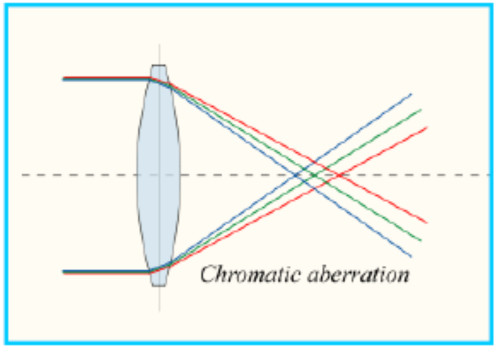
Ideal lens



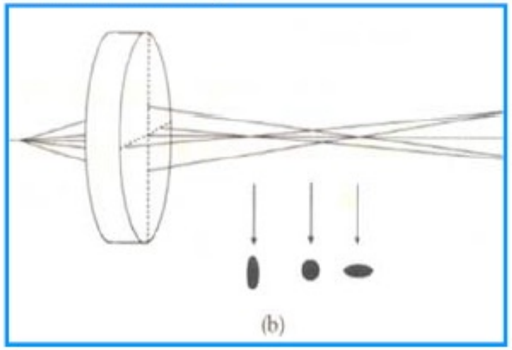
Spherical aberration



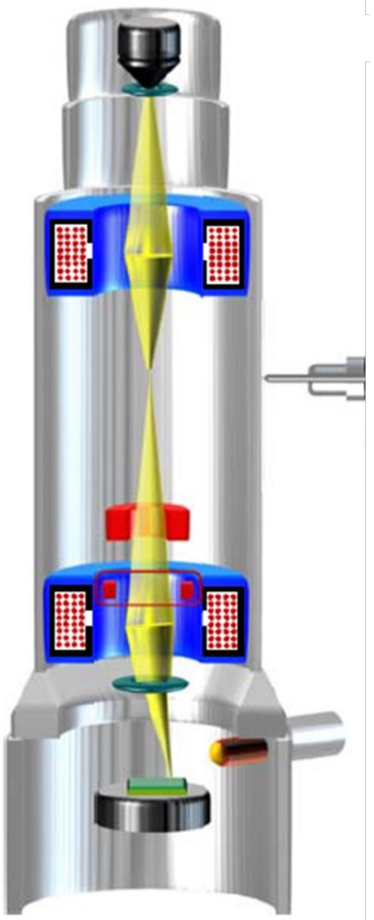
Chromatic aberration

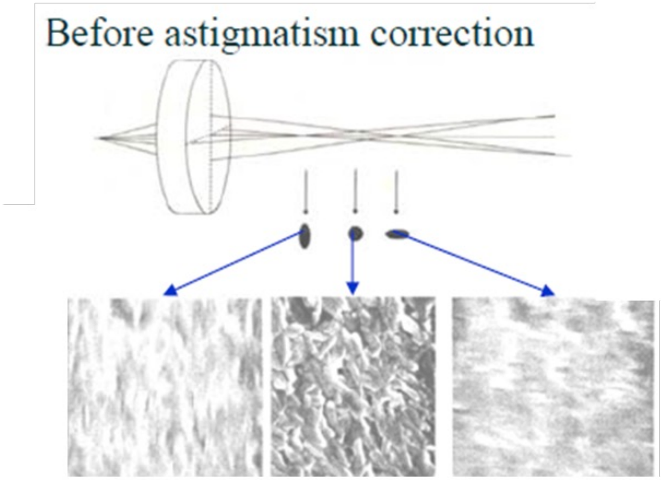


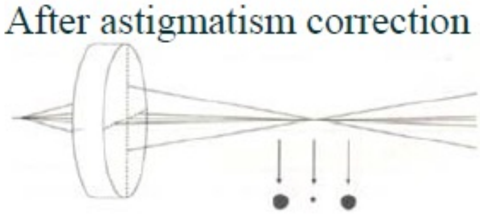
Astigmatism

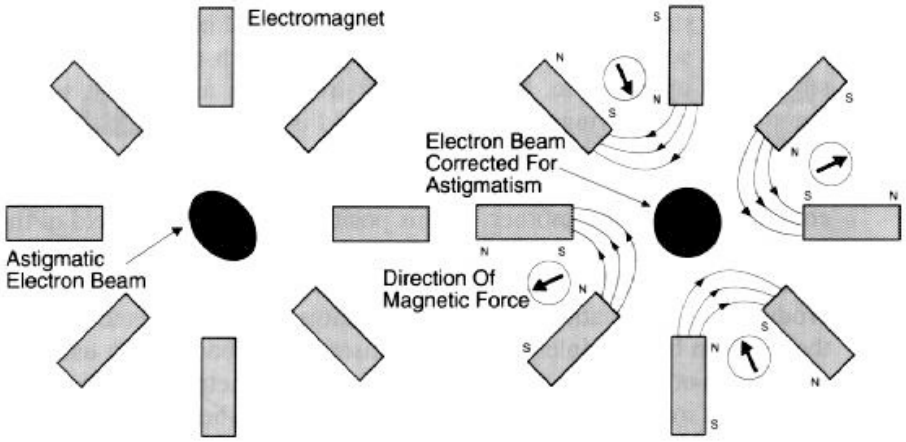


Astigmator




Before astigmatism correction

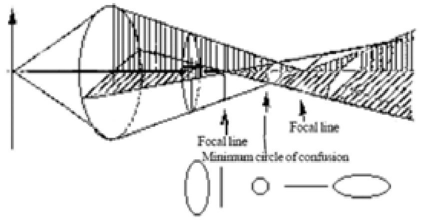

After astigmatism correction



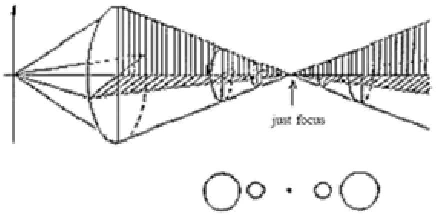
http://www.youtube.com/watch?v=NP5VckJfv04

Influence of Astigmatism on Image Quality

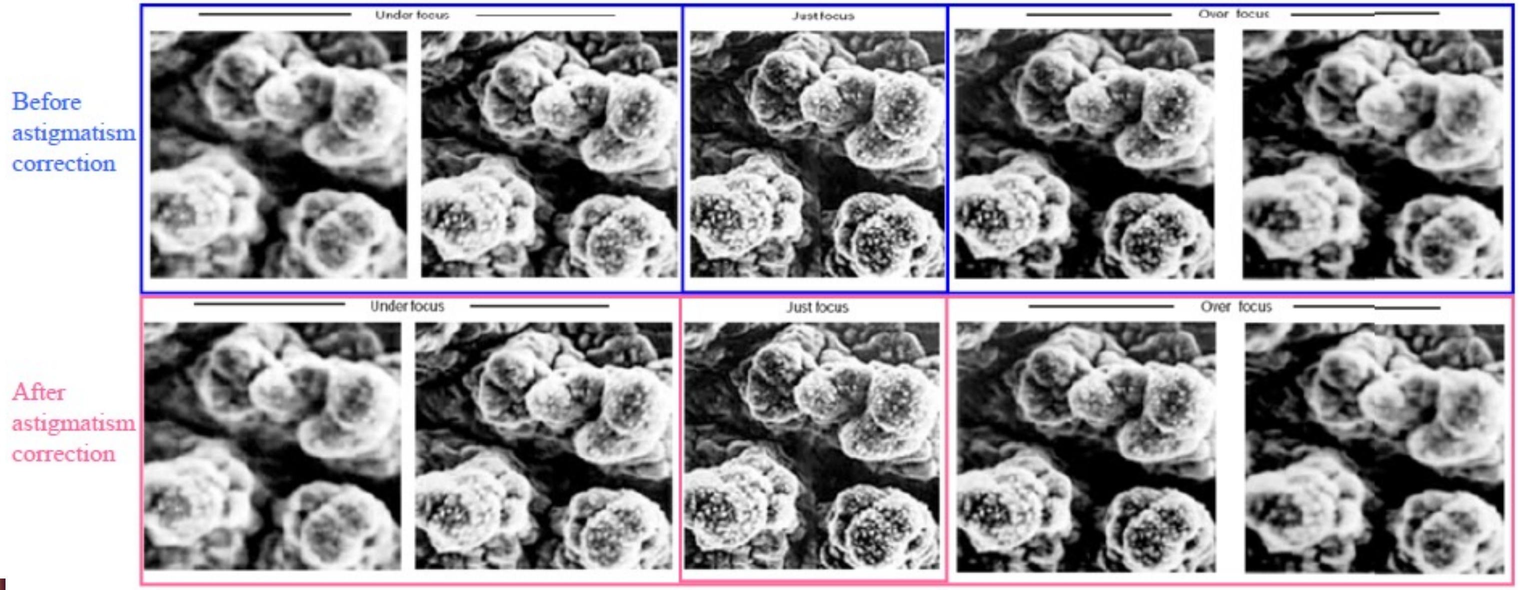
- Astigmatism: the aberration caused by the machining accuracy and materials of the pole piece



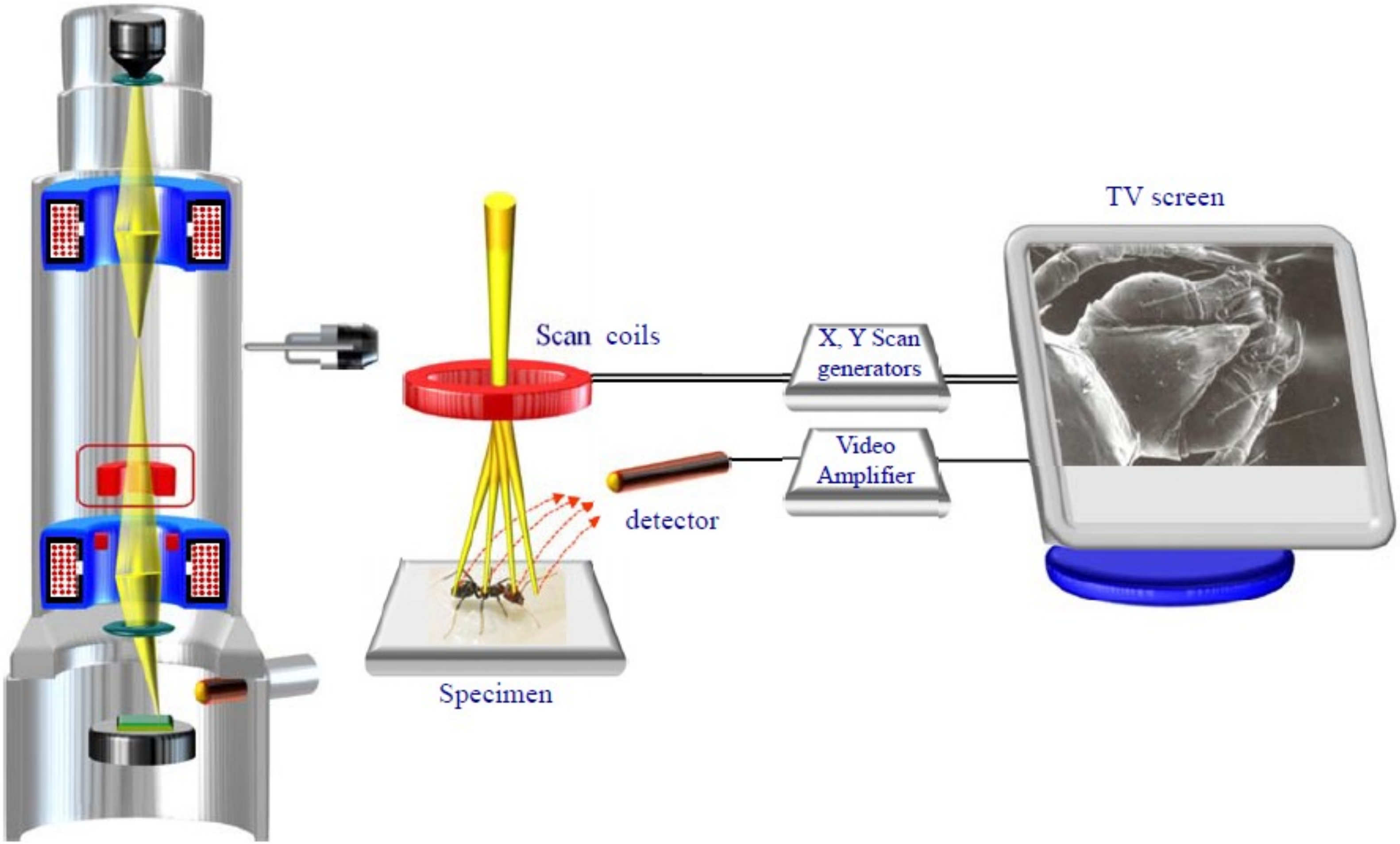
(a) Shape changes in electron beam when there is astigmatism



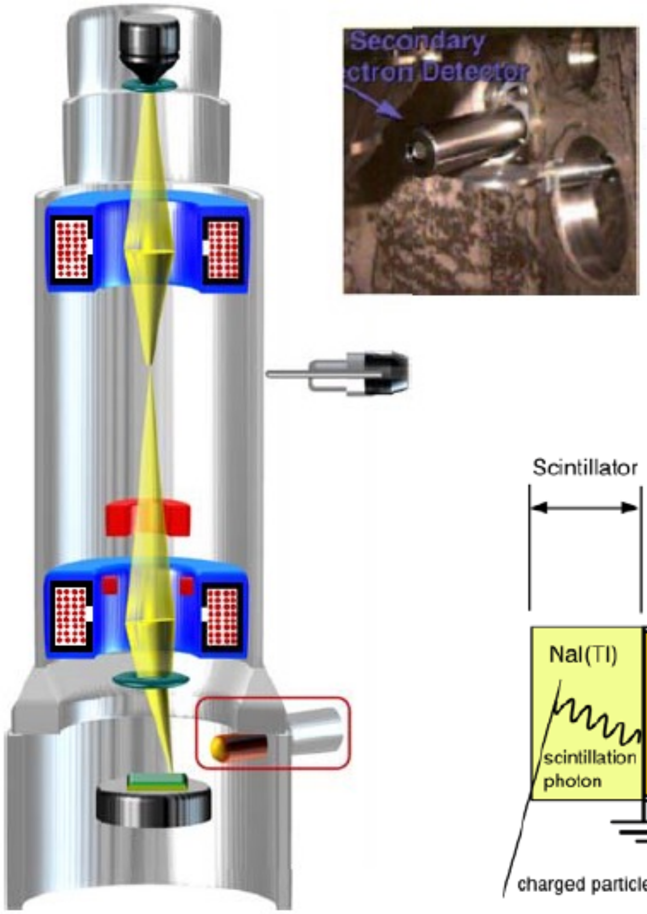
(b) Shape changes in electron beam when astigmatism is corrected

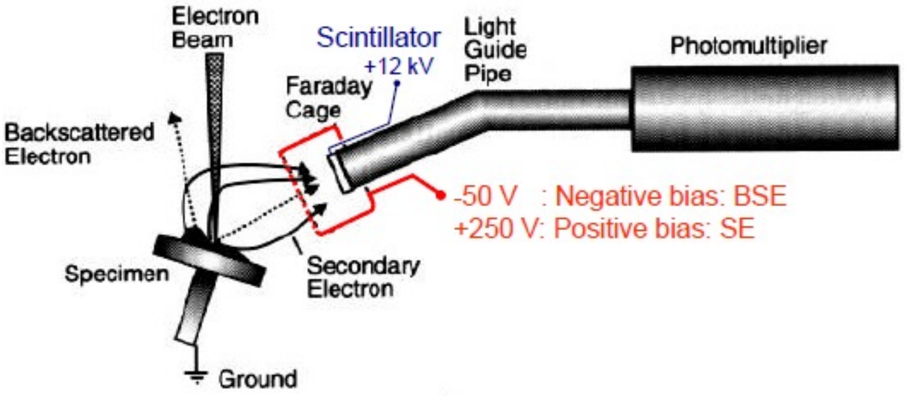


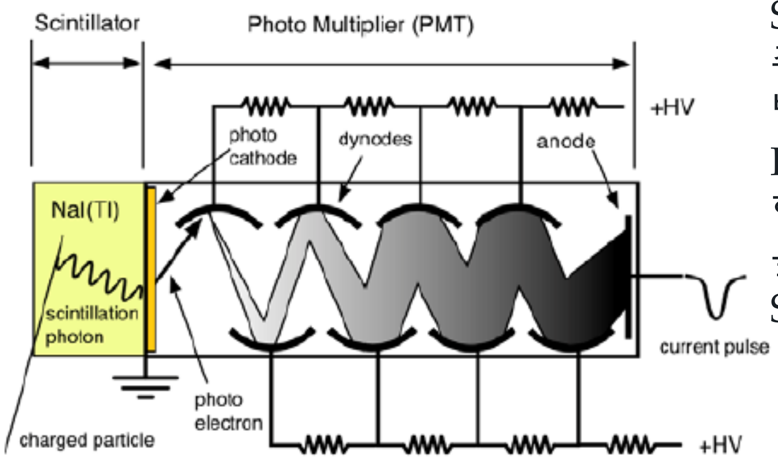
Scan coil: Scanning and Image Acquisition



Detection of Secondary Electrons





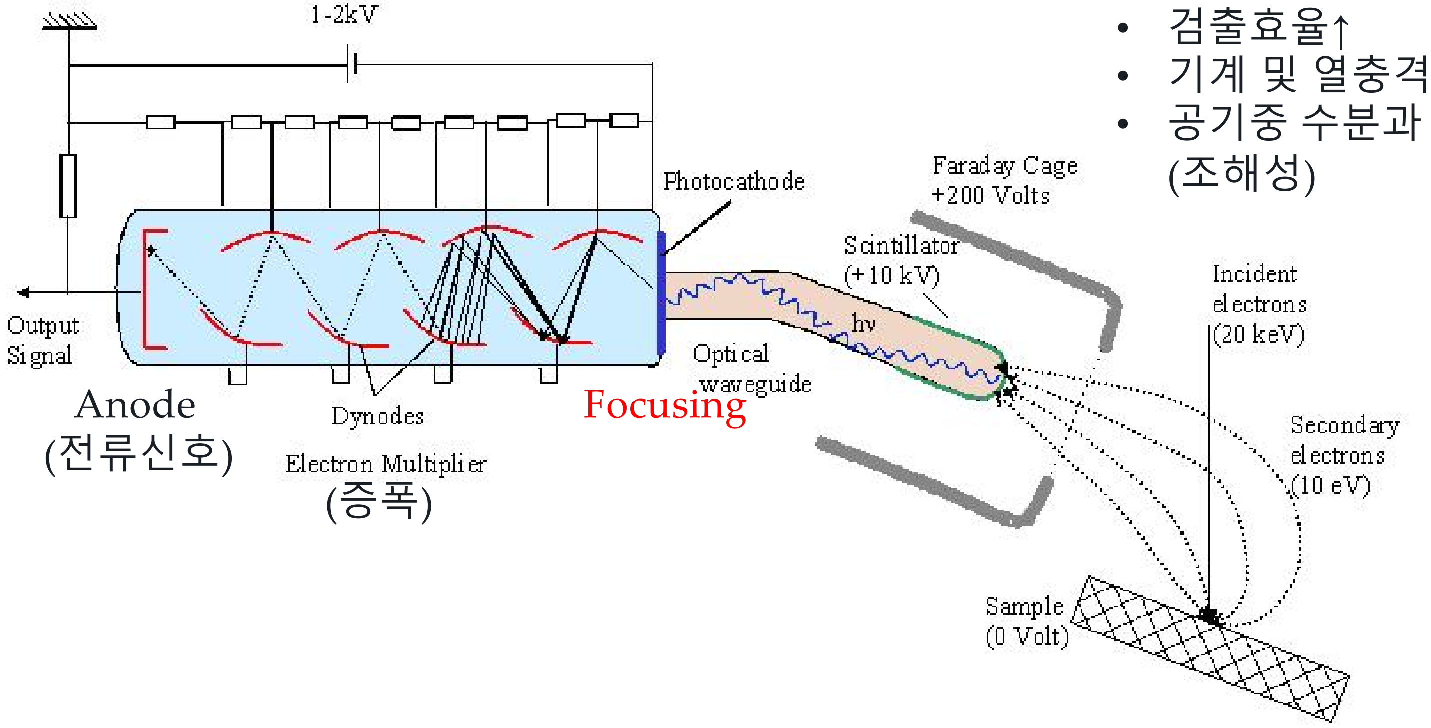


Scintillator: 형광체로써 물질의 준안정성 때문에 방사선을 받을 시 발광.

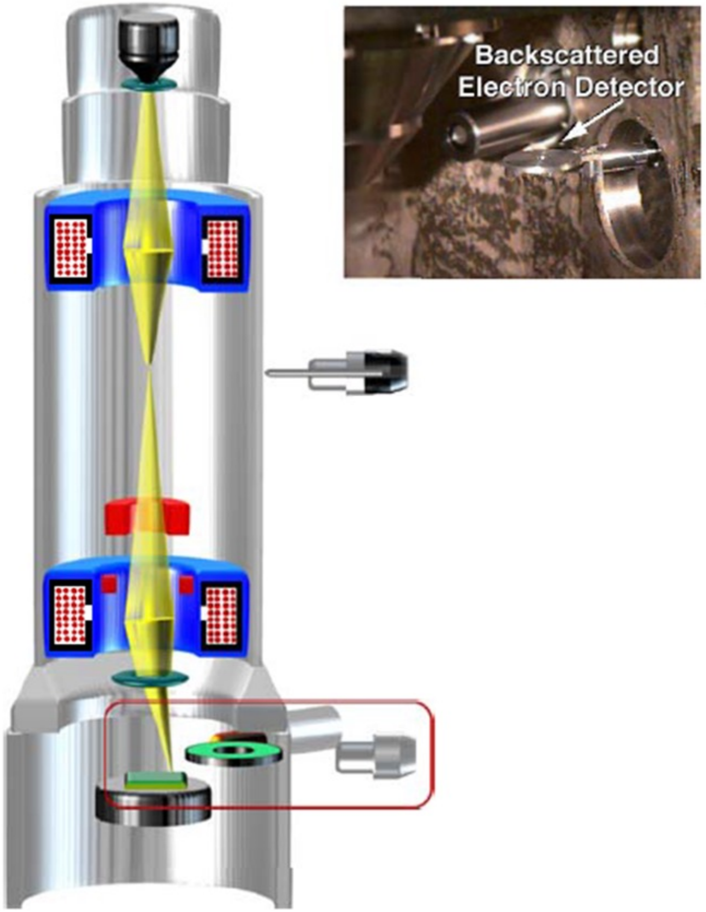
PMT: 입자물리학에서 많이 사용되는 형태로,
높은 E 빛 ➔ 가시광선 ➔ 증폭 ➔ Signal의 순서

Scintillator ➔ Nd (Tl)

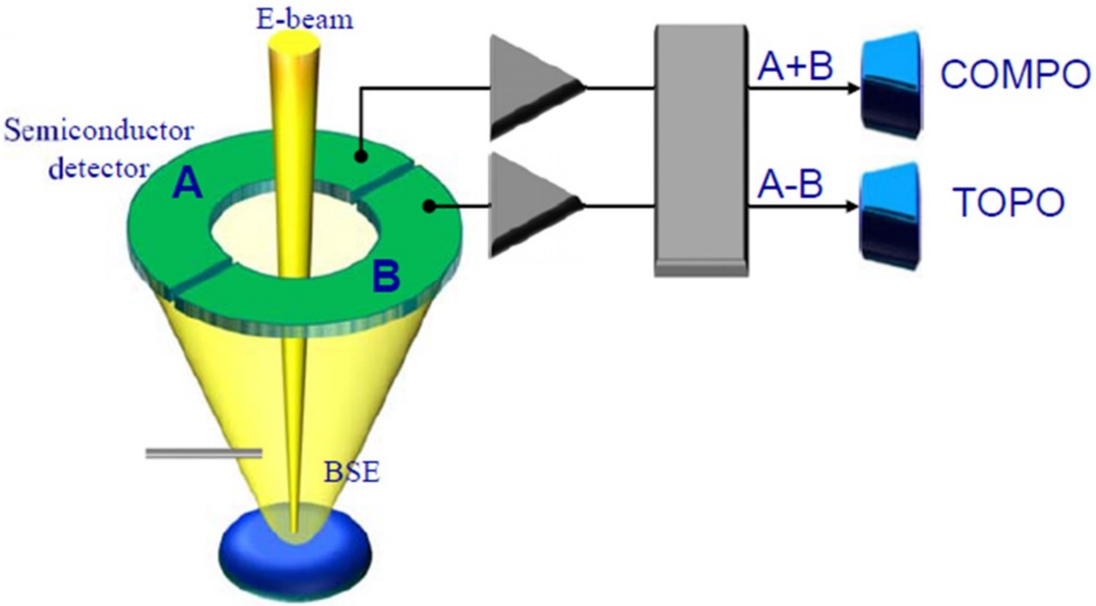
- 검출효율↑
- 기계 및 열충격 ↑
- 공기중 수분과 반응시 용해됨 (조해성)



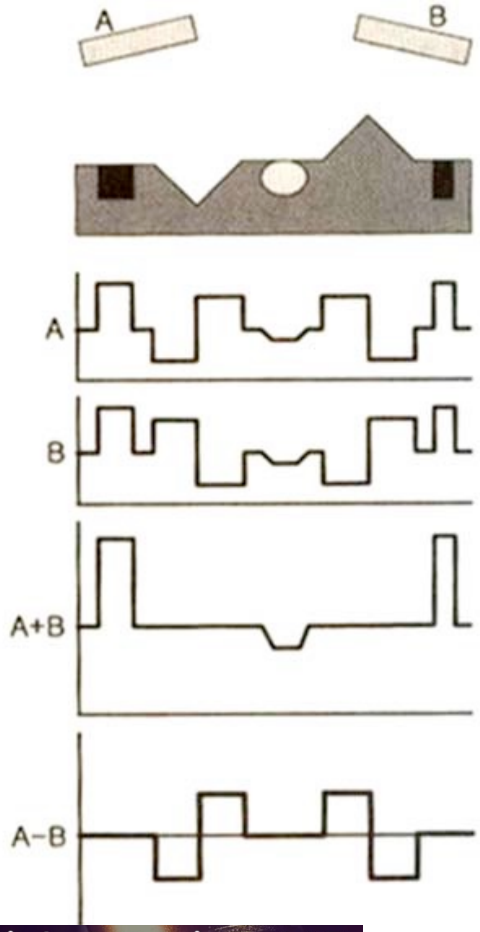
Detection of Backscattered Electrons



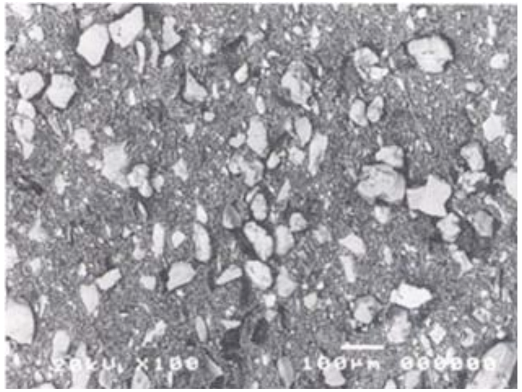
To collect electrons, the backscatter detector moves under the lens so the electron beam can travel through the hole in its center.



Detection of Backscattered Electrons to “Aid in Analysis”



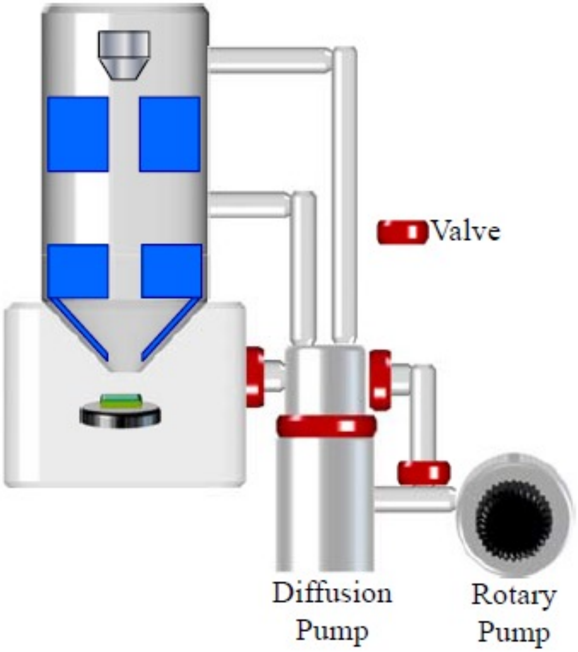
- **A+B: COMPO**

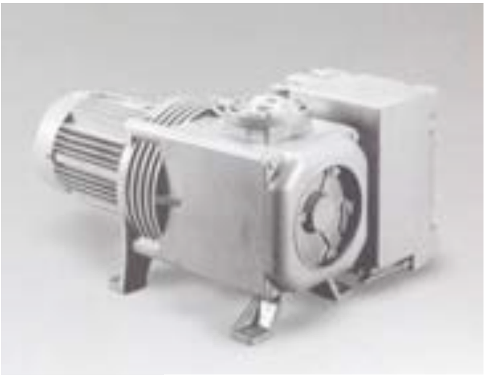


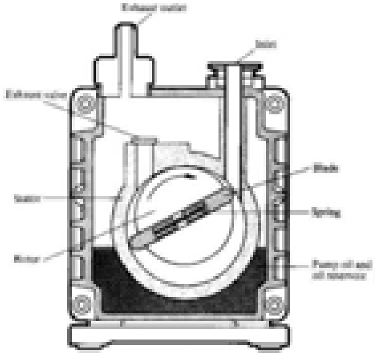
- **A-B: TOPO**

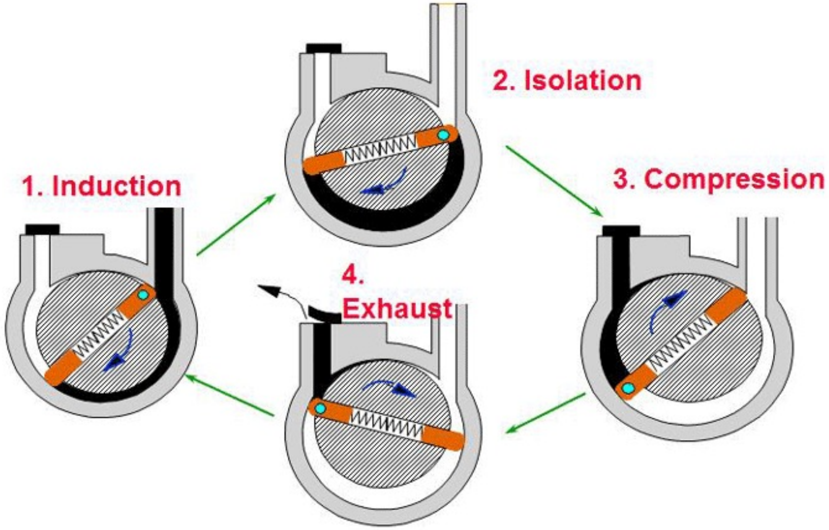
Vacuum System

Positive displacement pumps : Rotary vane pump, the most common



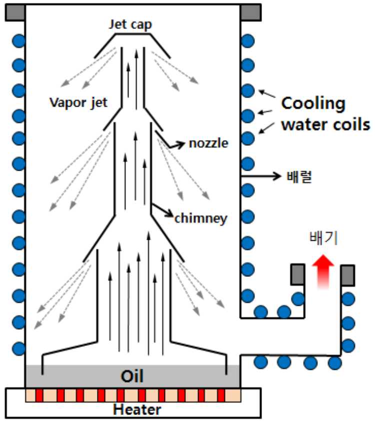




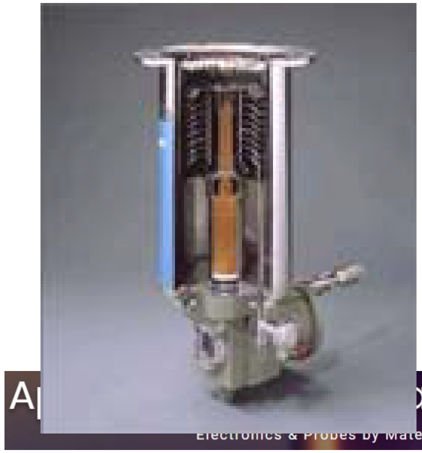


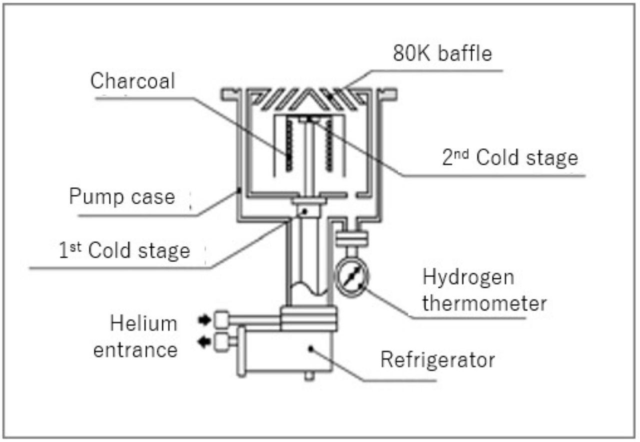
Vacuum System

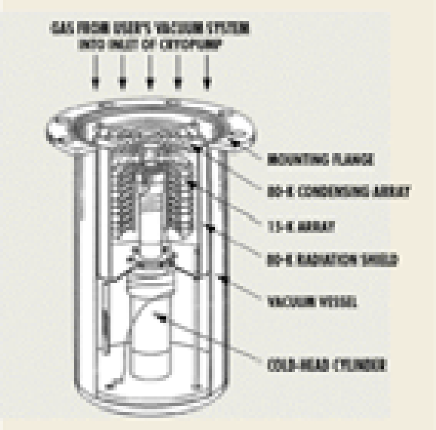
Momentum transfer pumps: Diffusion pump, Turbomolecular pump

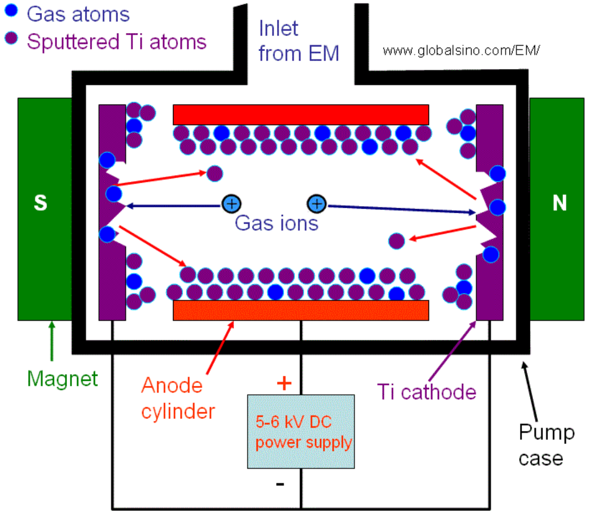


Entraps pumps: Cryopump, Ion pump

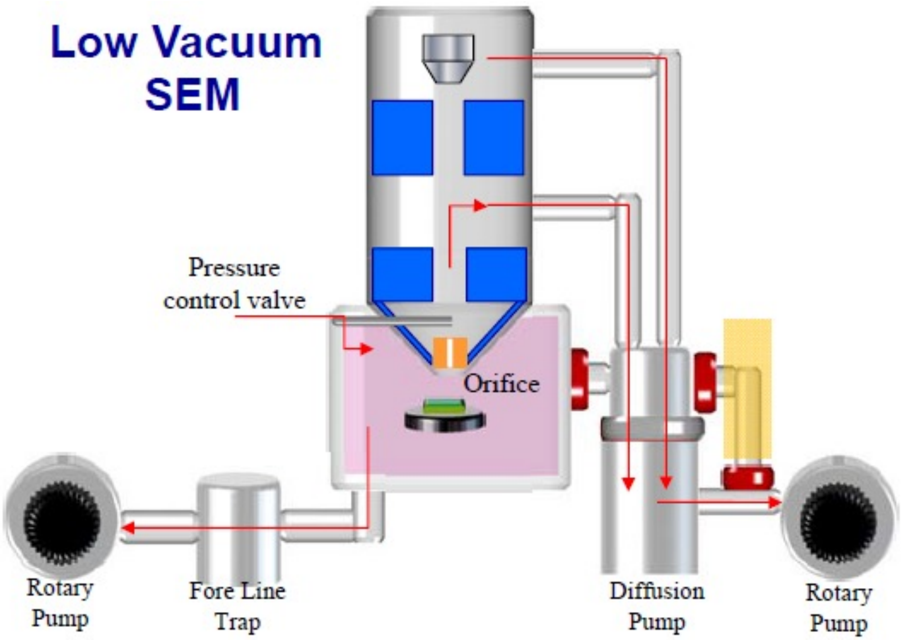


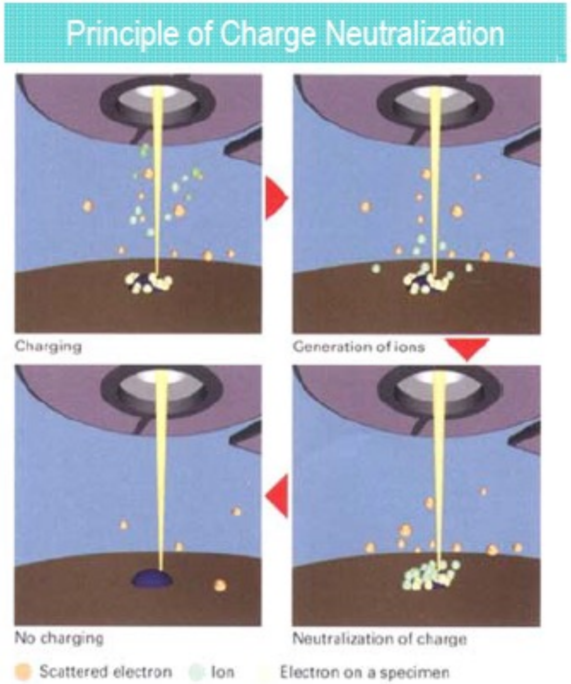






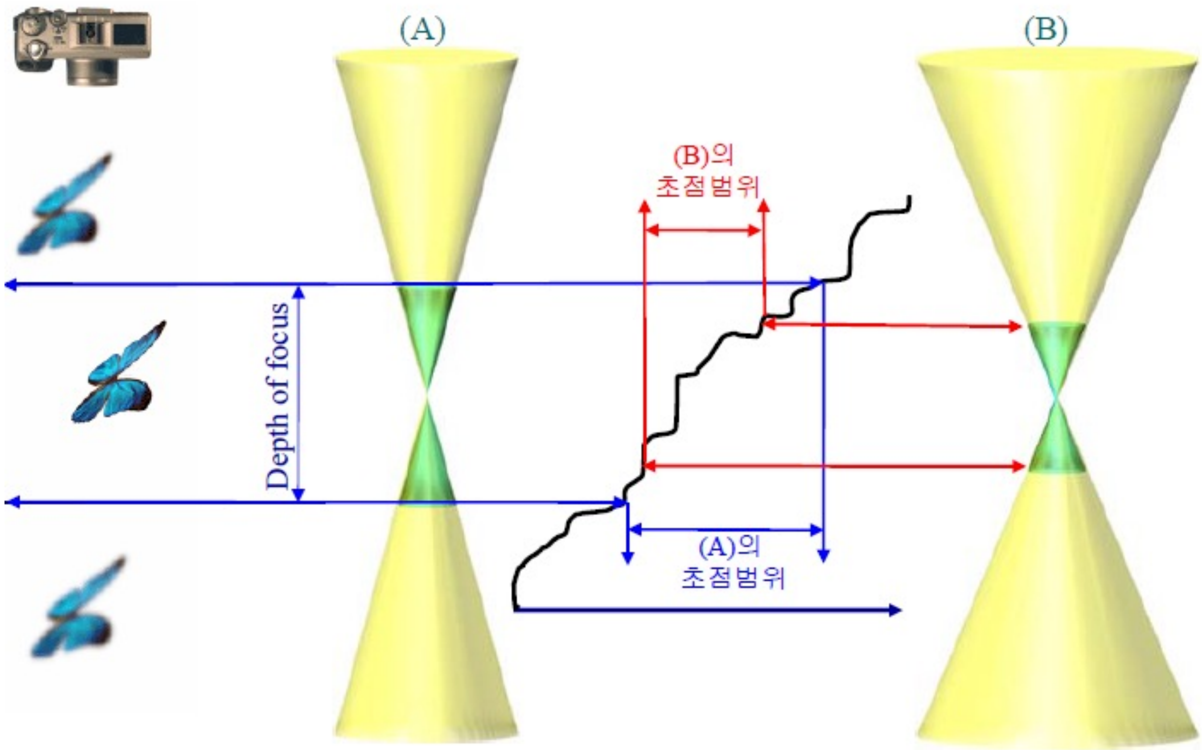
Low Vacuum SEM





- HVSEM samples have to be electrically conductive and not produce vapors in a vacuum.
- LVSEM is a new innovation specifically designed to study wet, oil bearing, or insulating materials (EX: Biological cells, plants, concrete, wood, asphalt and liquid suspensions)

Depth of Focus

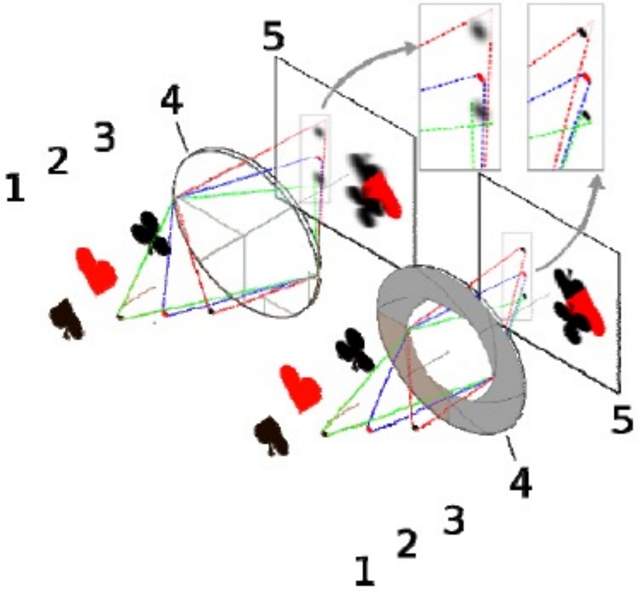


The area within the depth of field appears sharp while the areas in front of and beyond the depth of field appear blurry.

Effect of Objective Aperture and Working Distance on the DOF

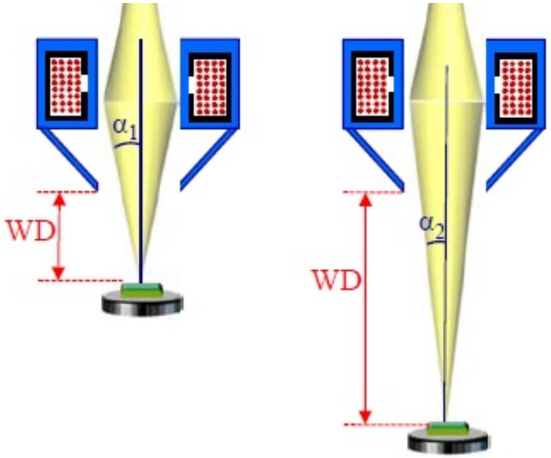
- Effect of aperture size

If Aperture size ↓ , then DOF ↑



- Effect of working distance (WD)

If WD ↑ , then DOF ↑



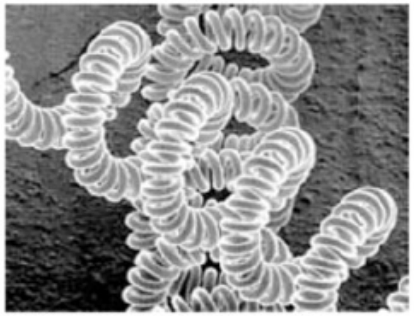
$\alpha_1 >> \alpha_2$

Influence of Working Distance and Objective Aperture on Images

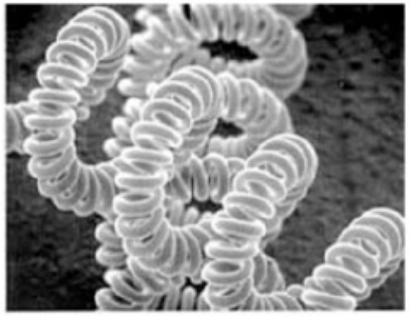
- Working distance effect :

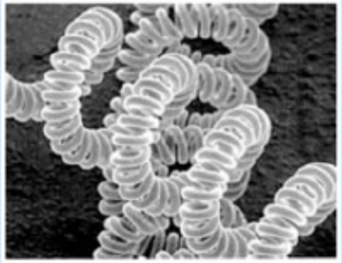
WD ↑, Depth of focus ↑

→ **Resolution ↑**


(d) OL aperture diameter: 200μm WD: 38mm

WD

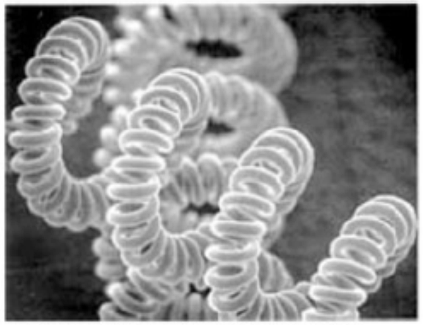

(b) OL aperature diameter: 200μm WD: 10mm


(c) OL aperture diameter: 200μm wd:20mm

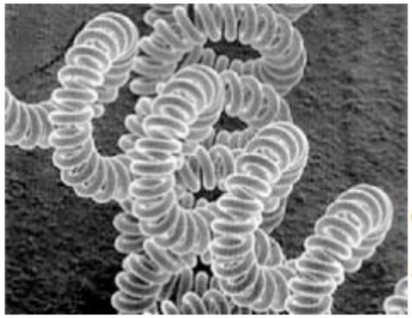
- Objective aperture effect :

OL aperture ↑, Depth of focus ↓

→ **Resolution ↓**

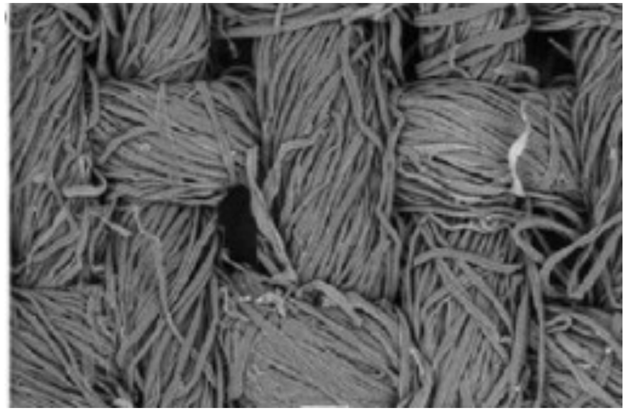

(a) OL aperature diameter: 600μm WD: 10mm

OL aperture

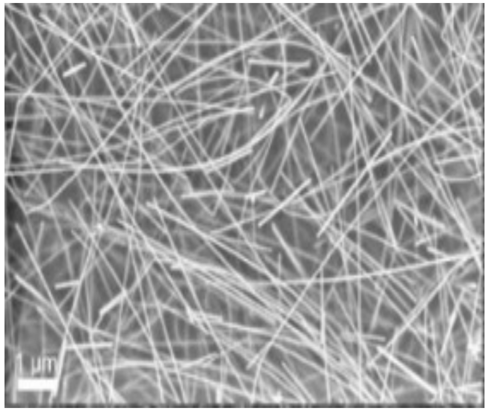

(e) OL aperture diameter: 100μm WD: 38mm

SEM Images

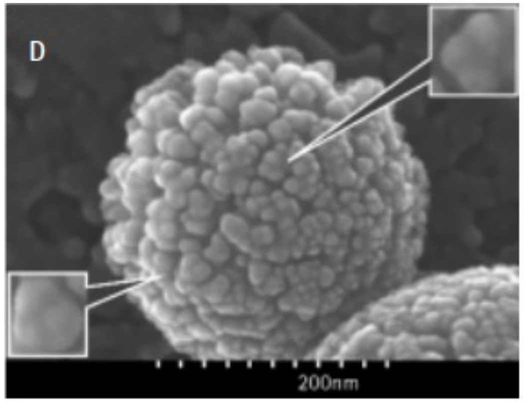
Untreated fabric



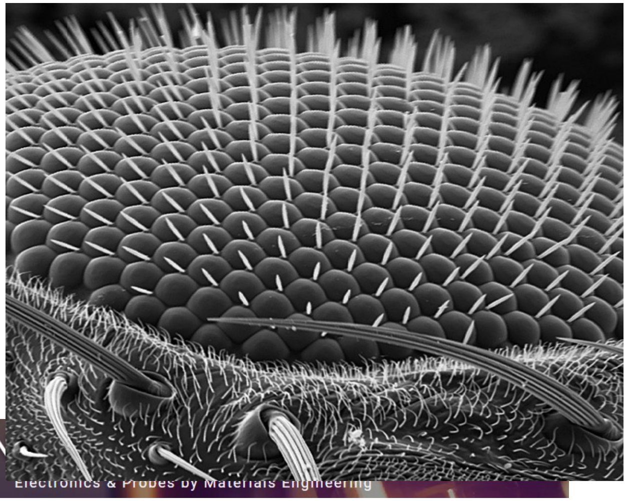
Silver nanowires



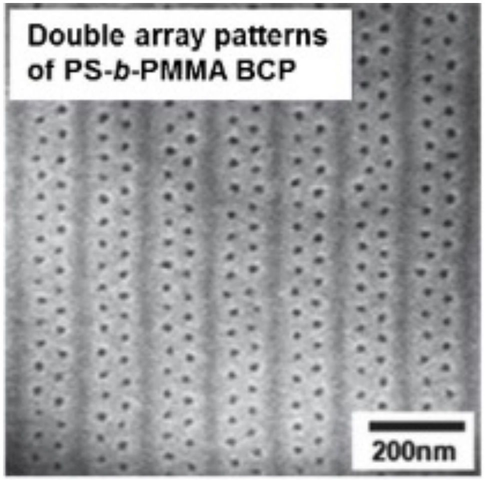
SARS coronavirus



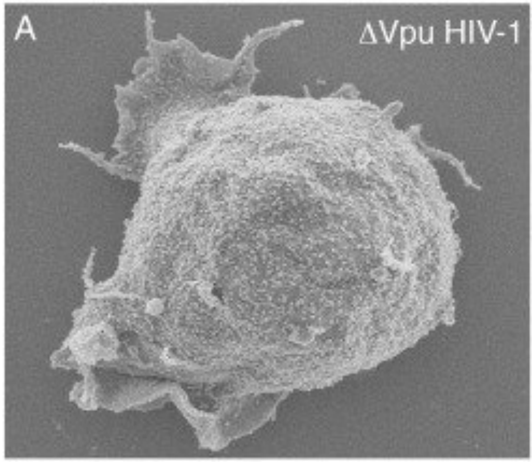
Fly eyes



Polymer pattern



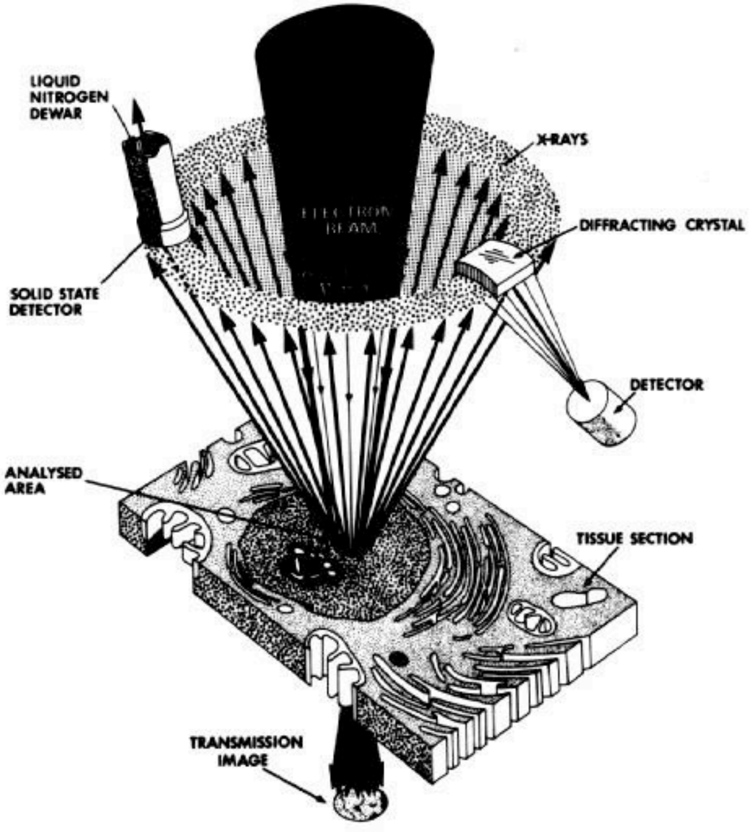
HIV-1



SEM Attachments (Composition Analysis)

- Energy dispersive x-ray Spectrometer (EDS)
- Wavelength dispersive x-ray Spectrometer (WDS)

X-Ray Microanalysis



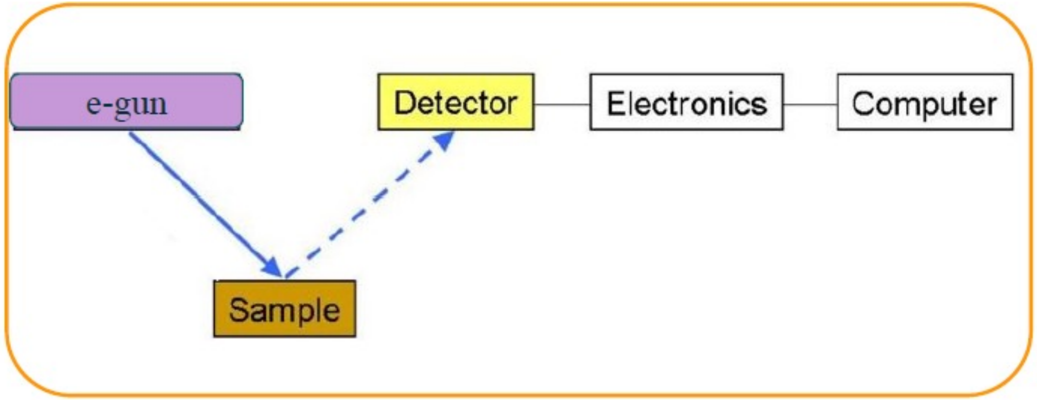
- X-ray microanalysis (composition analysis): X-rays, which are produced by the interaction of electrons with the atoms in the sample, may be detected in an SEM equipped.

EDS :
Energy Dispersive x-ray Spectroscopy
WDS :
Wavelength Dispersive x-ray Spectroscopy

$$E = \frac{hc}{\lambda}$$

EDS vs WDS

Energy dispersive x-ray Spectrometer (EDS)


e-gun
Detector
Electronics
Computer
Sample

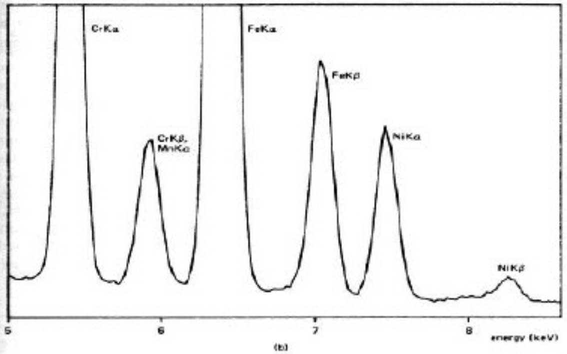
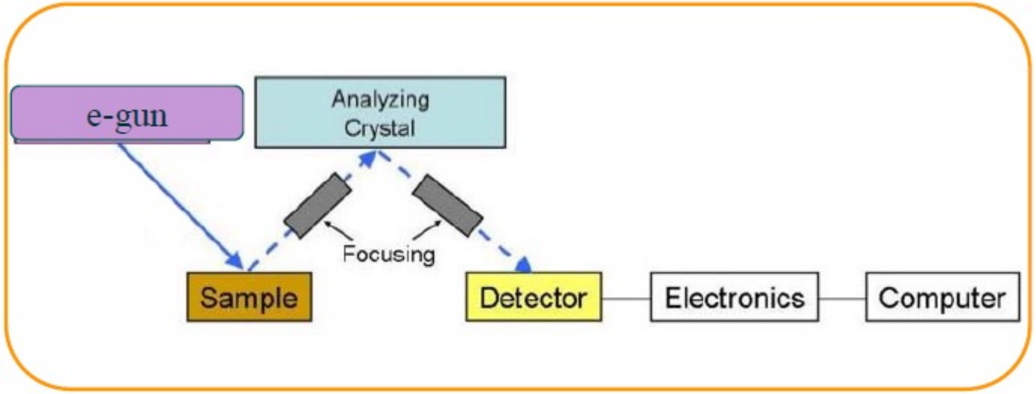
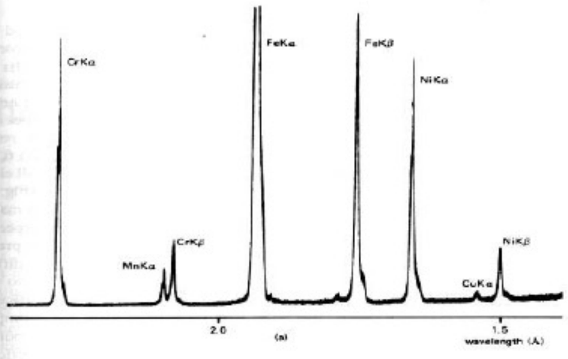


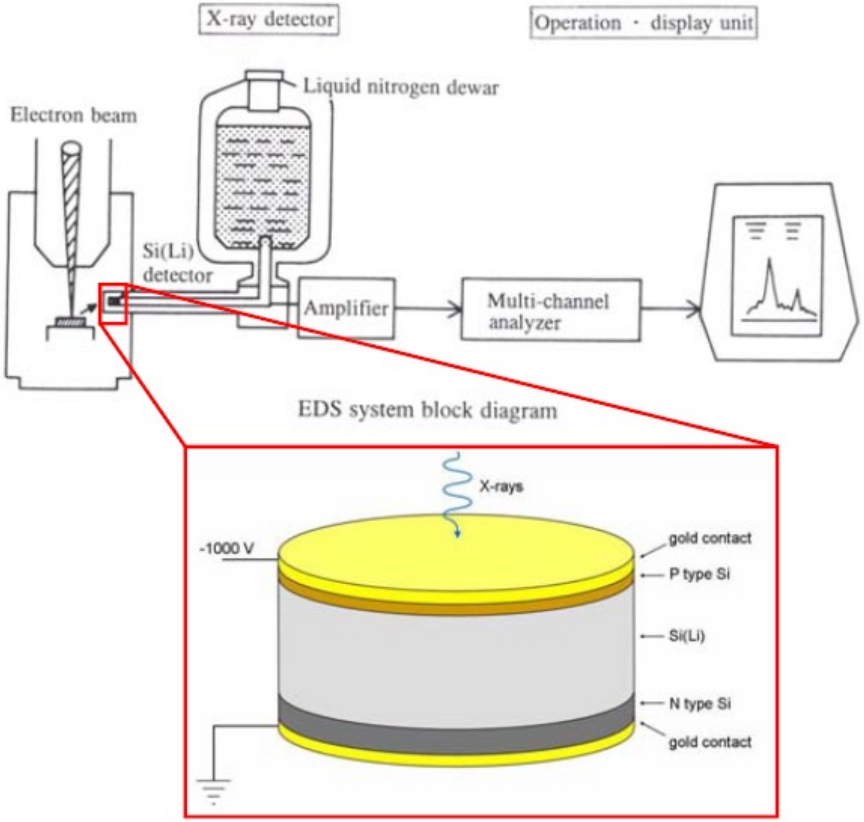
Fig. 6.7 – Comparison of spectra from a steel (1.7 wt % manganese) at 20 kV taken using (a) WDS and (b) EDS; note the manganese Kα peak is not resolved with EDS.

Wavelength dispersive x-ray Spectrometer (WDS)


e-gun
Analyzing Crystal
Focusing
Sample
Detector
Electronics
Computer

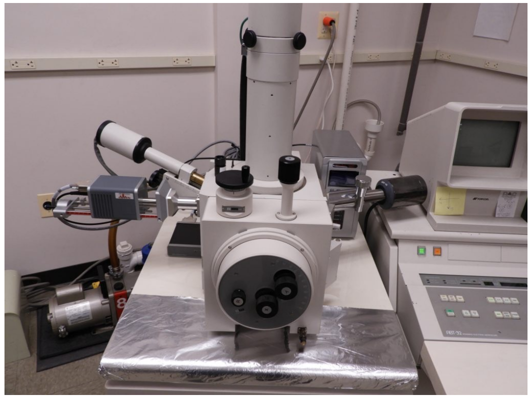


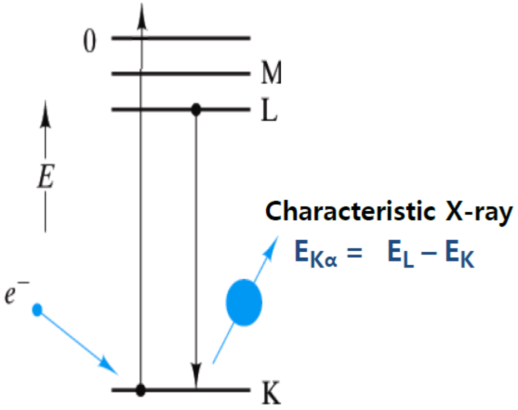
EDS : Energy Dispersive X-Ray Spectroscopy

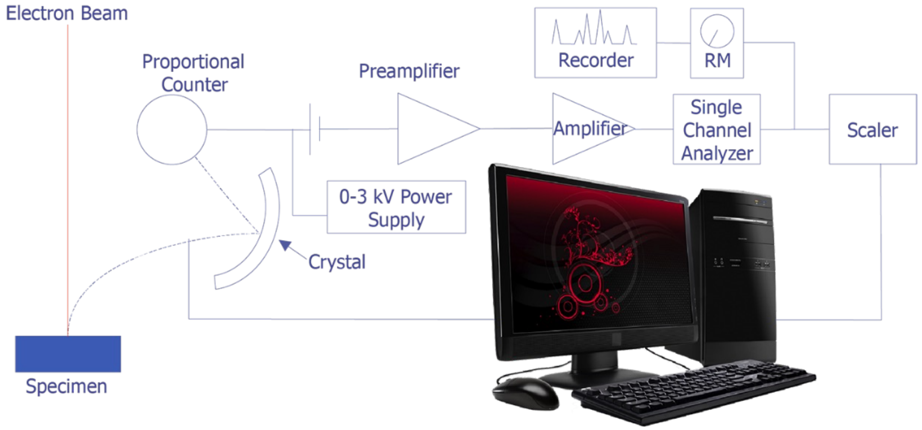


- Cooled in LN_2 temps, Si crystal converts X-ray photon into charge by ionization.
- Charge is integrated through the FET and is proportional to X-ray energy.

Energy-Dispersive X-ray Spectroscopy (EDS, EDX)

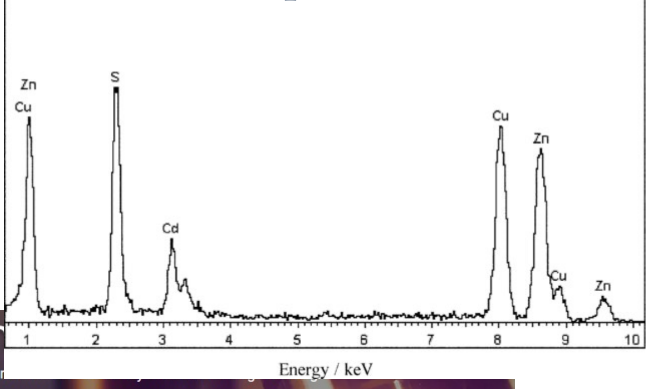






Depth resolution	Lateral resolution
0.1 ~ 5 μm	0.1 ~ 5 μm

EDS spectrum



- **Solid state X-ray detector: Si(Li) → large solid angle & compact**
- X-ray inside the solid state Si detector

➔ emission of photoelectron by photoelectric absorption

➔ generation of electron-hole pairs by inelastic collision with the photoelectron

➔ measurement of the induced current

➔ measurement of the energy of the characteristic X-ray

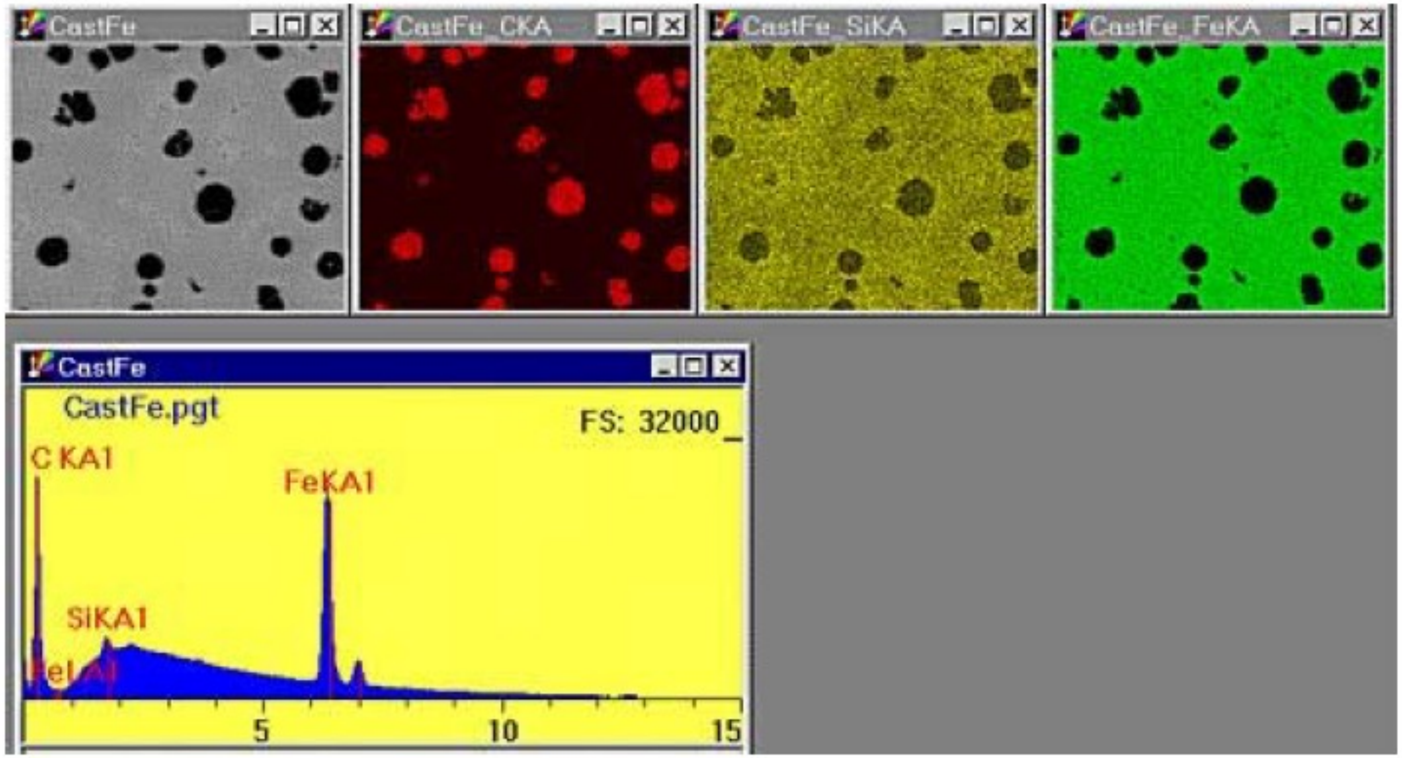
- **In EDS, it is difficult to detect the elements below Na (EK<1keV)**

- small x-ray emission yield
- large noise (large Bremsstrahlung x-ray)
- large absorption at low energy by Be window

EDS Example

A cast iron sample

SEM C map Si Map Fe map

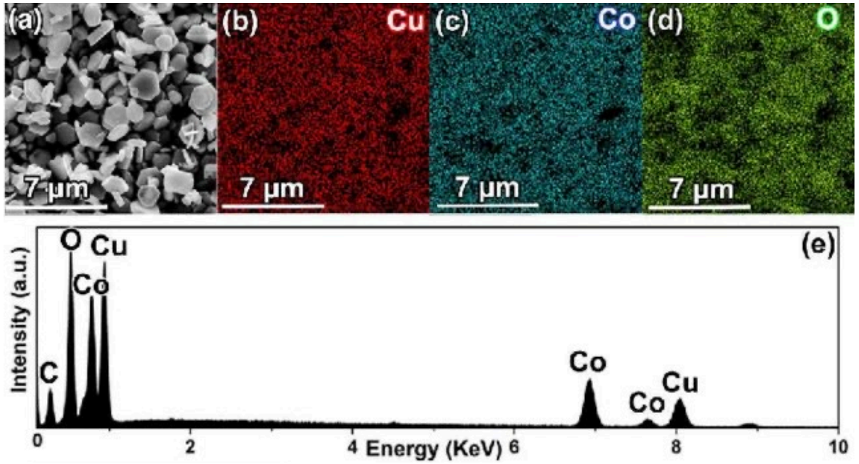


Small Area Analysis and Element Mapping by EDS

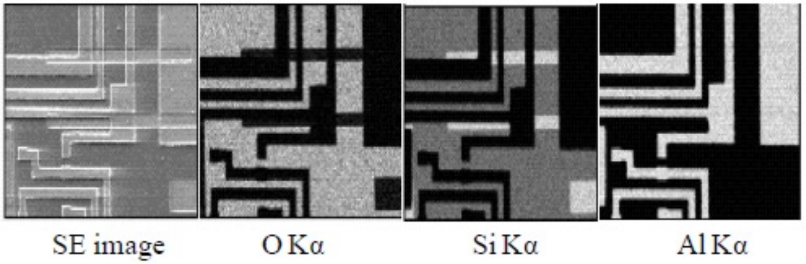
Depth and lateral resolution of EDS

➔ 0.1~5μm depending on E_0 , θ, detection element and material density.

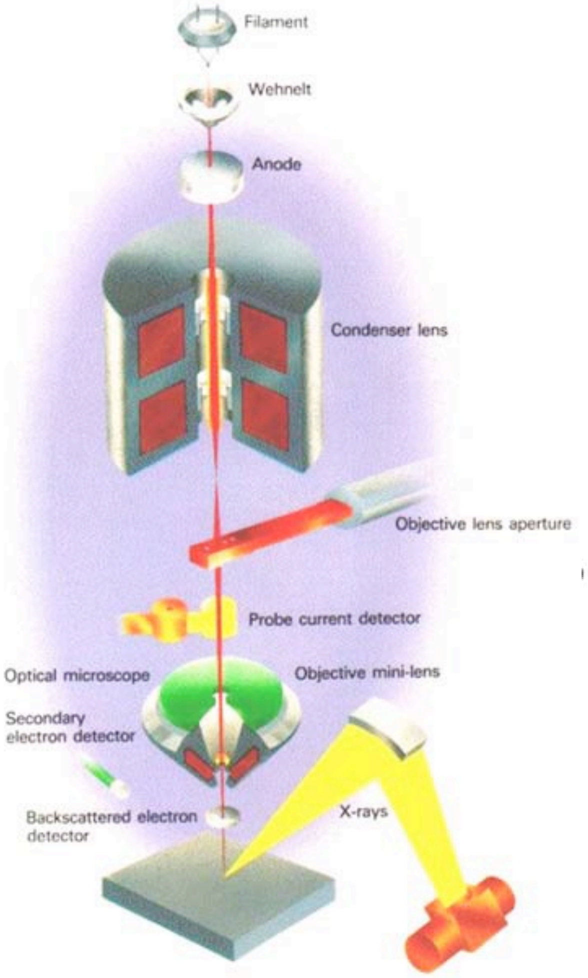
- Particle or small area analysis is possible with EDS
- Before particle or small area analysis, the lateral resolution must be calculated.

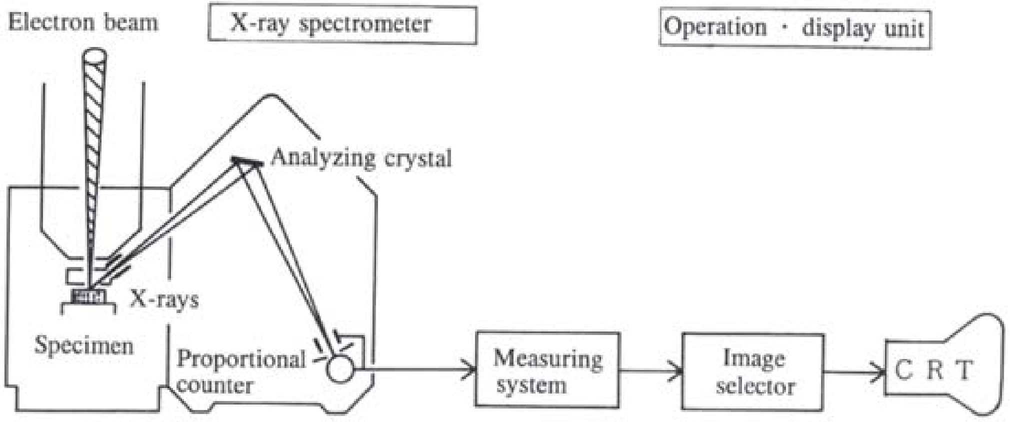


Device imaging



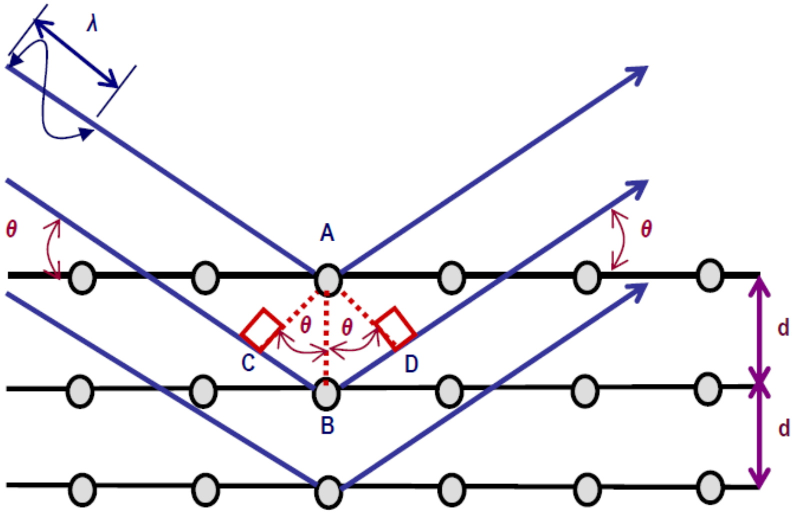
Operation Principle of WDX





WDS system block diagram

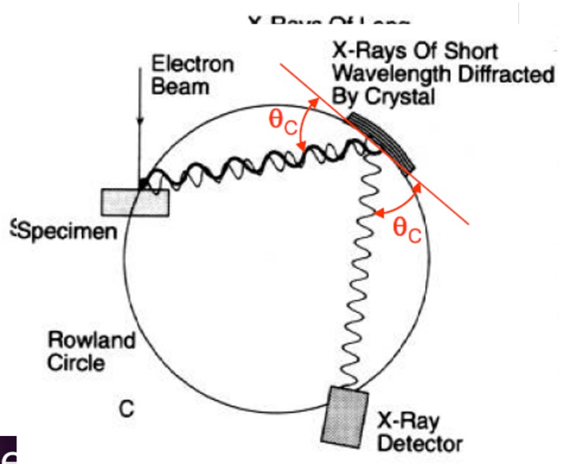
Operation Principle of WDX

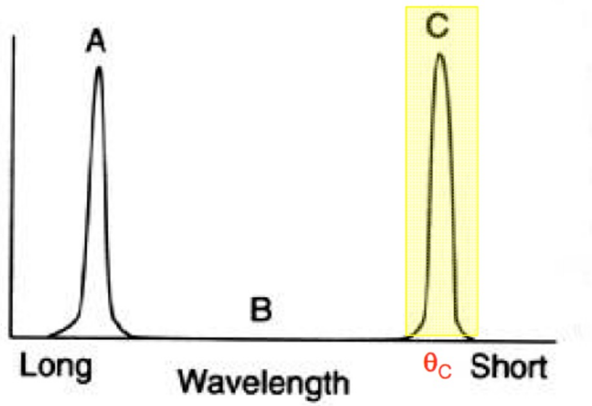


Bragg's Diffraction Law

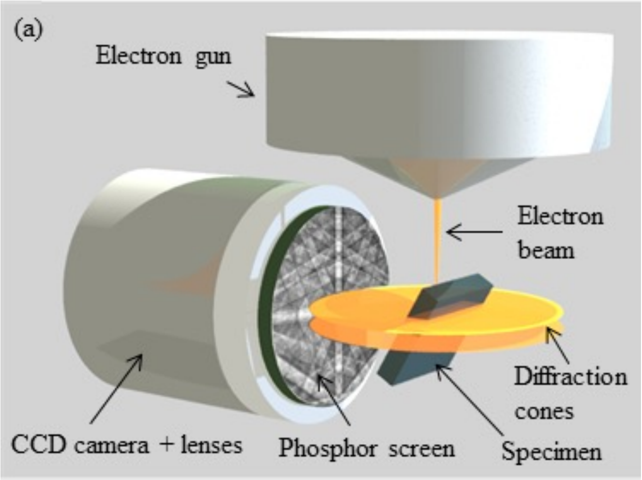
$$n\lambda = 2d\sin\theta$$

n : Diffraction index
Θ: Incident angle
λ: Wavelength:
d : Interspacing distance



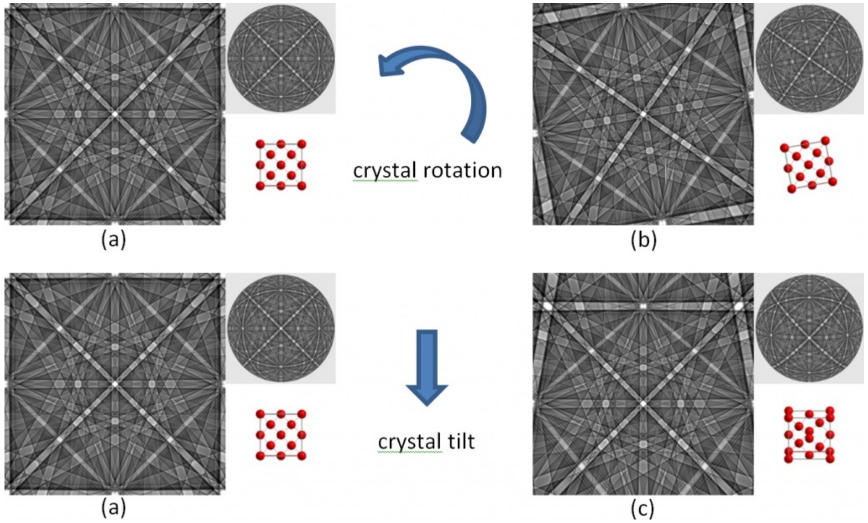


Electron Backscatter Diffraction (EBSD)

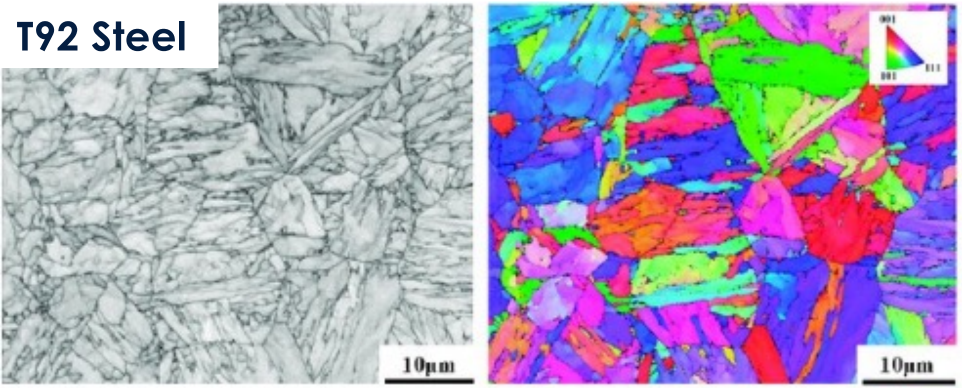


EBSD can be used to confirm the orientation of polycrystalline

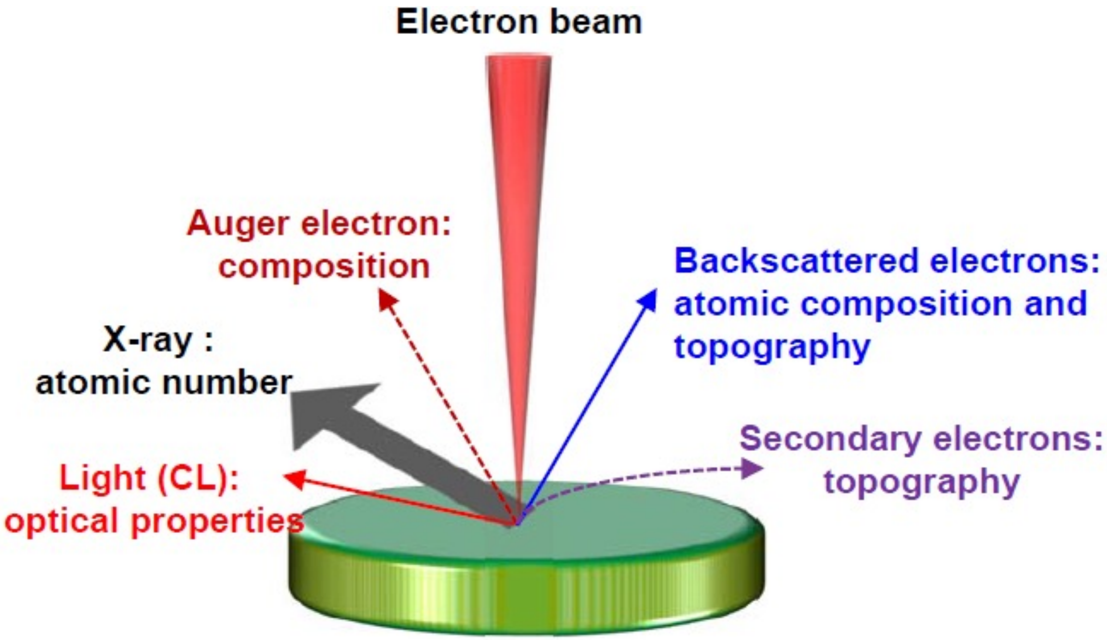
- Effect of the rotation and tilt of single crystal silicon on the EBSD pattern



T92 Steel



Summary I



- The SEM is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern.
- The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as optical property, and electrical conductivity.