

2009 spring

***Microstructural Characterization
of
Materials***

04. 08. 2009

Eun Soo Park

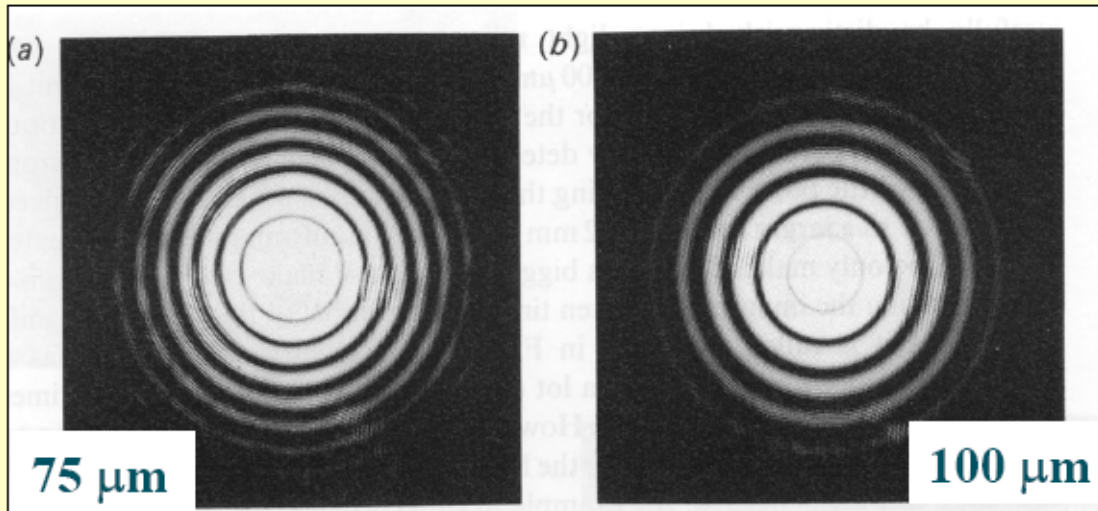
Office: 33-316

Telephone: 880-7221

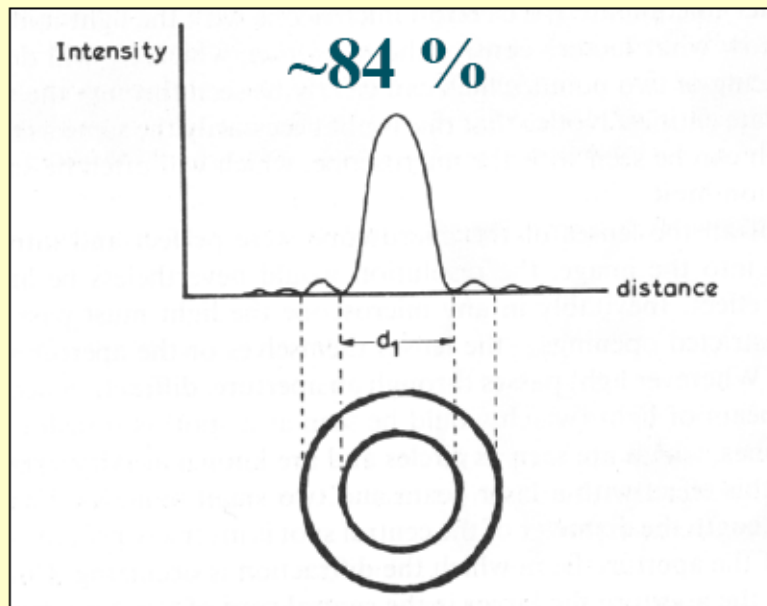
Email: espark@snu.ac.kr

Office hours: by an appointment ¹

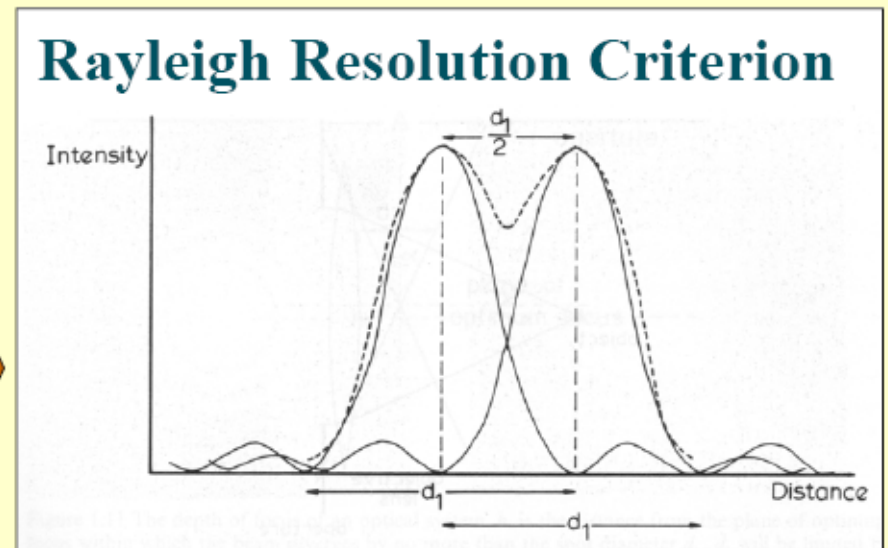
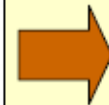
Resolution ... Airy Discs



Laser beam
Diffraction
through a
pinhole

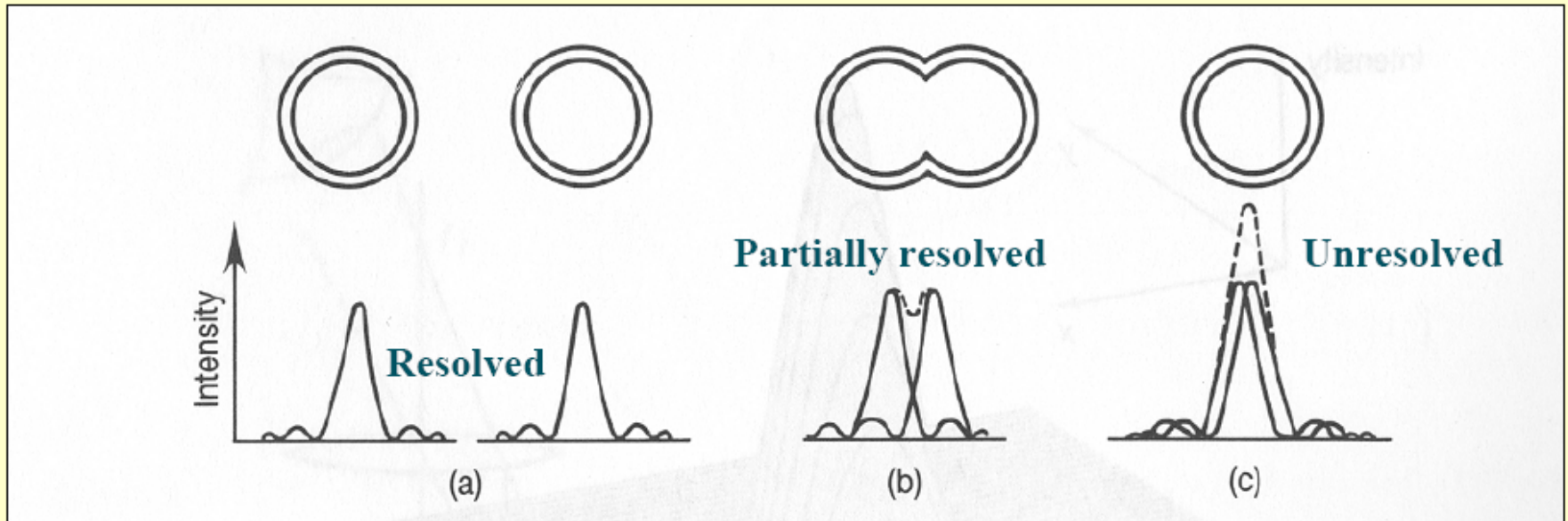


$d_1 \sim 1/\text{aperture-diameter}$



$$R_1 = d_1/2 = 0.61\lambda/n\sin(\alpha) = 0.61\lambda/NA$$

Diffraction limited Resolution



Thus the smallest separation is determined by the N.A. ($1/2f\#$)

Typically the best objective has N.A $\approx 1.6 \Rightarrow$ resolution ≈ 170 nm

For $\lambda \sim 400$ nm (green light)

\Rightarrow Decrease λ

Electron Microscopy - Decreasing The Wavelength

$$\Rightarrow E = \frac{p^2}{2m} = eV \quad \text{Energy Conservation}$$

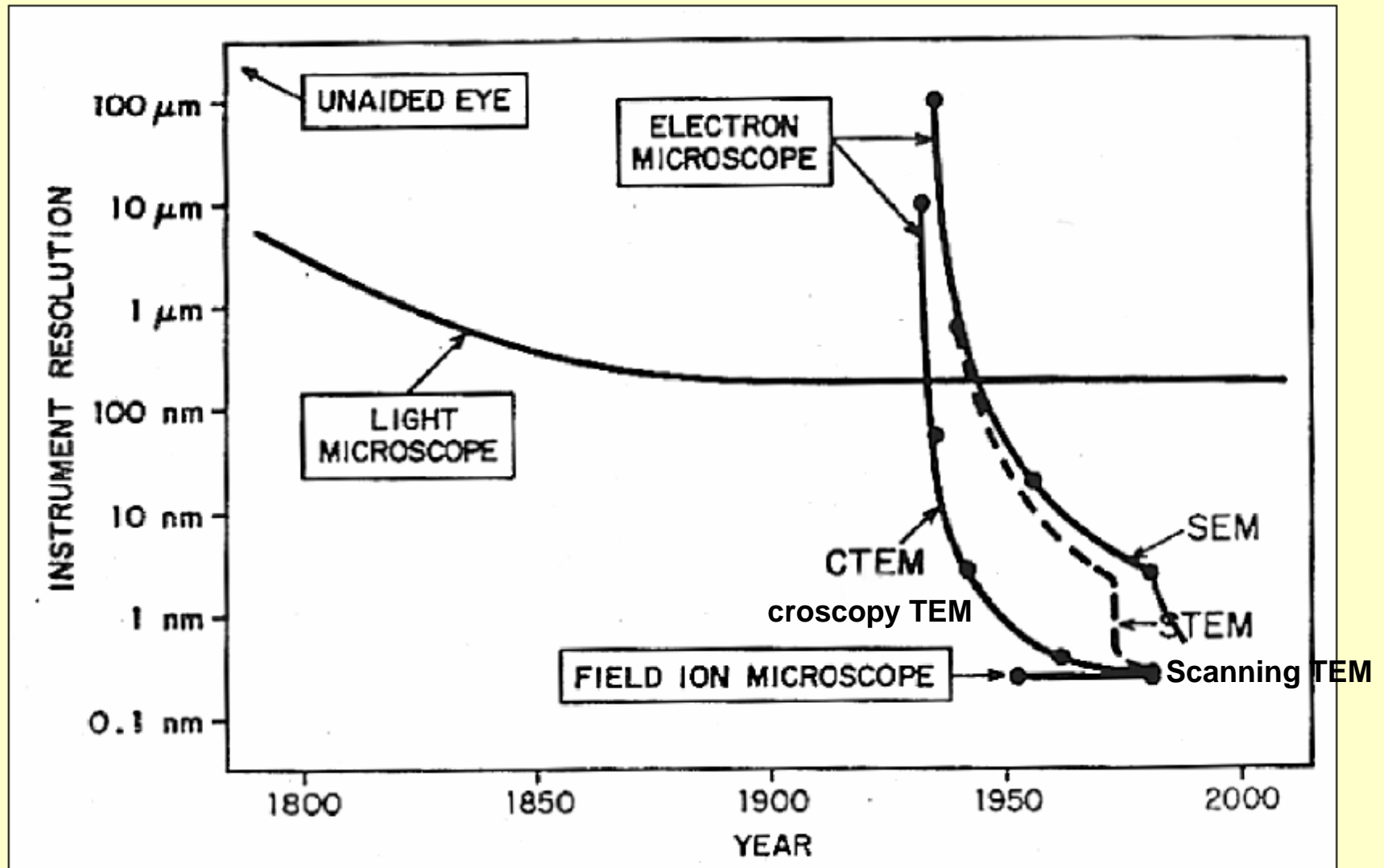
$$\Rightarrow p = \sqrt{2meV}$$

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

$$\lambda = \frac{h}{p} = \frac{h}{\sqrt{2meV}} = \frac{6.6 \cdot 10^{-34}}{\sqrt{2 \cdot 9.1 \cdot 10^{-31} \cdot 1.6 \cdot 10^{-19} \cdot 50000}} \approx 0.05 \text{ \AA}$$

Resolution (50 kV): $R_1 = 0.61\lambda/NA \sim (0.6 \bullet 0.05)/1.6 \sim 0.2 \text{ \AA}$

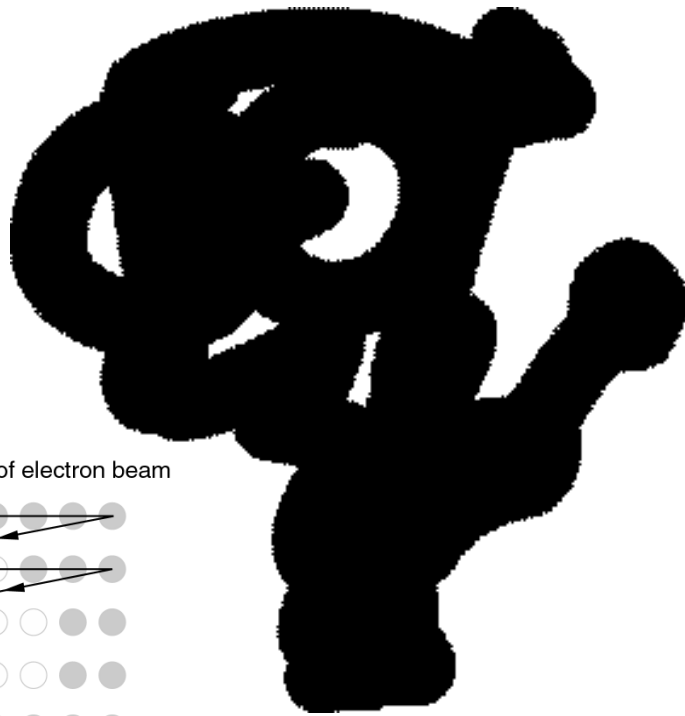
The Evolution of Resolution



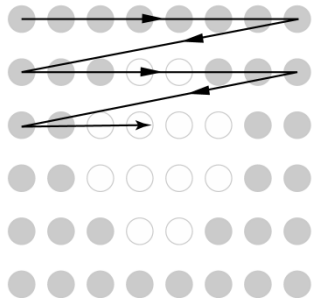
광학현미경과 전자현미경의 비교

항 목	전자현미경	광학현미경
광 원	전자빔	가시광선
파 장	0.0859 Å (20KV) ~ 0.0251 Å (200KV)	7,500 Å(가시광선)~ 2,000 Å(자외선)
전파매질	진 공	공 기
렌즈(방법)	전자기렌즈	유리렌즈
분해능	점분해능 : 3.5 Å 회절격자분해능 : 1.4 Å	가시광선 : 2000 Å 자외선 : 1000 Å
사용배율	100X ~ 450,000X	10X ~ 2,000X
초점조절	전기적	기계적

Effect of Probe Size : Impact on Resolution



Discrete movement of electron beam

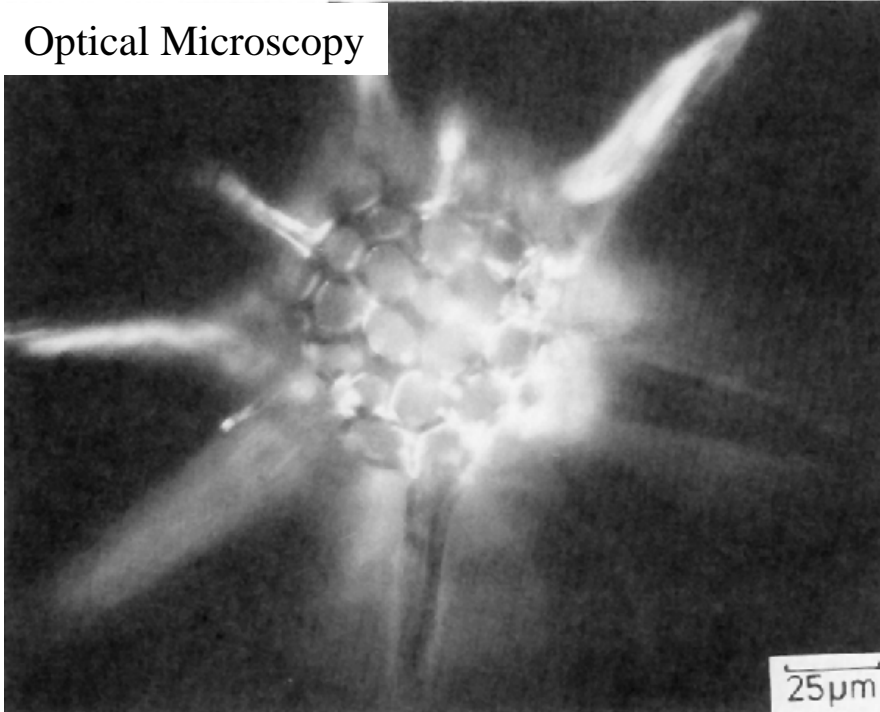


With large probe

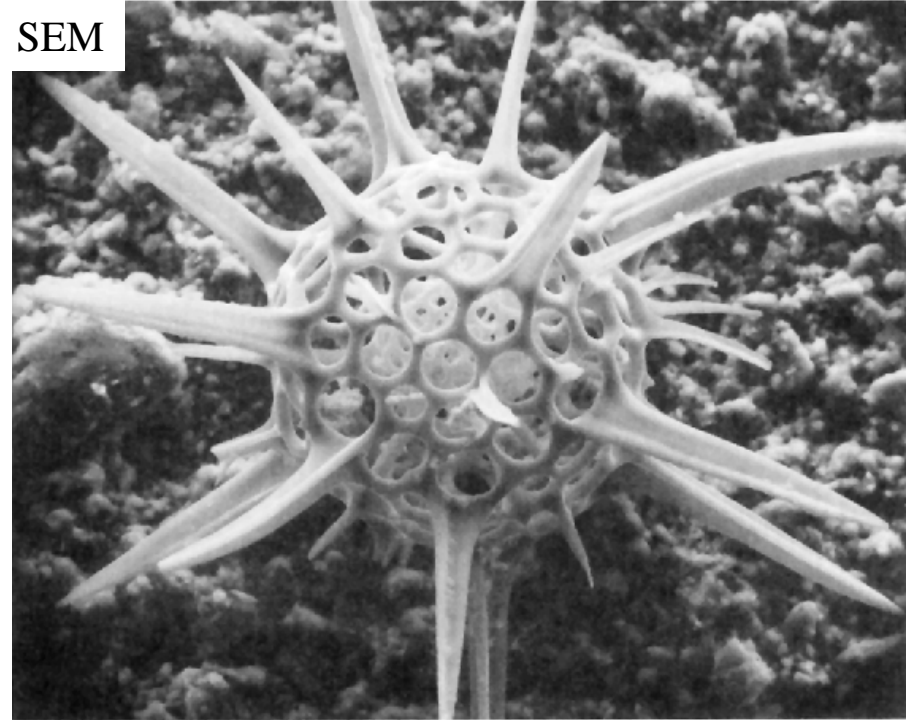
With small probe

Optical vs. Electron Microscopy

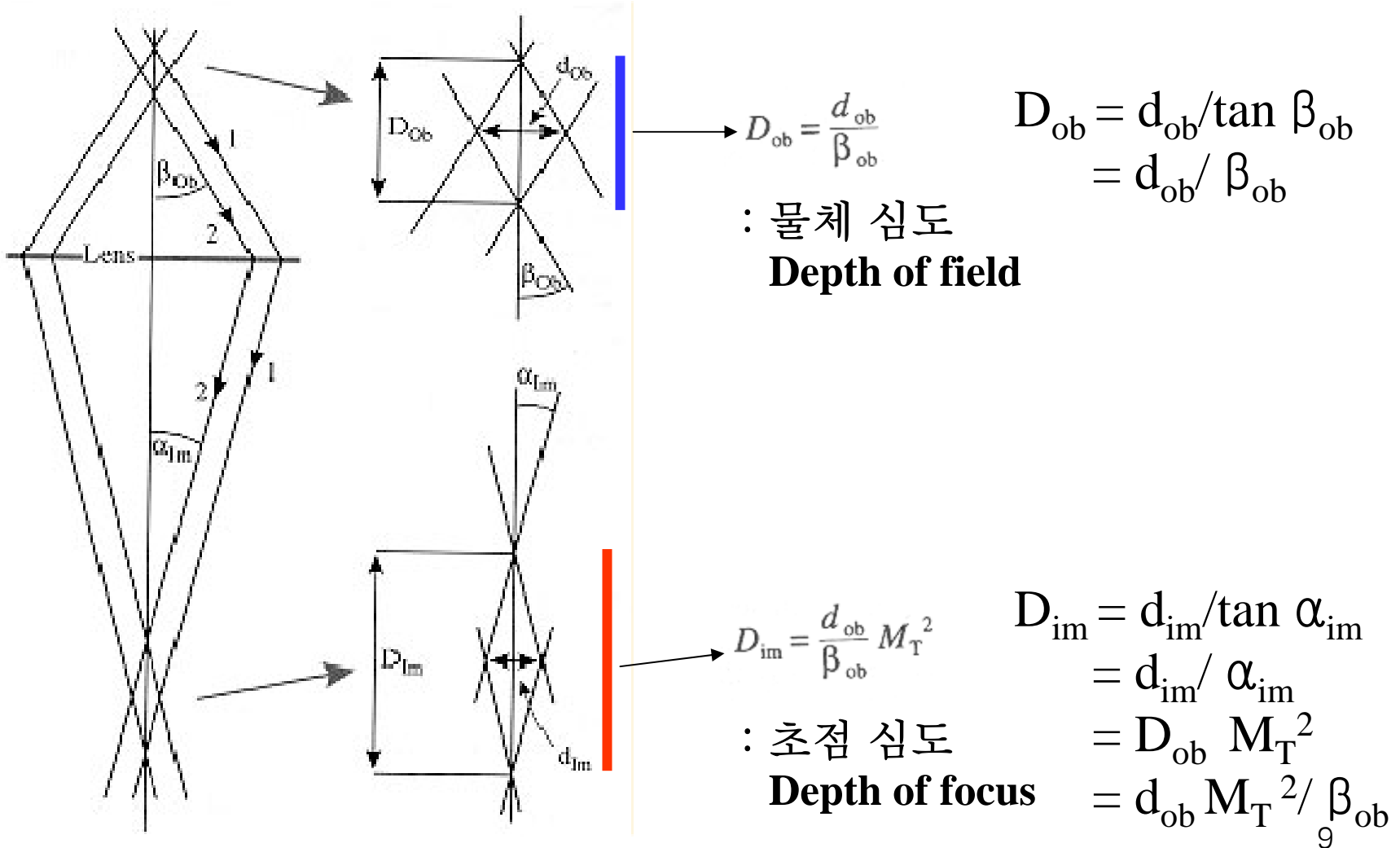
Optical Microscopy



SEM



Depth of focus



Depth of focus

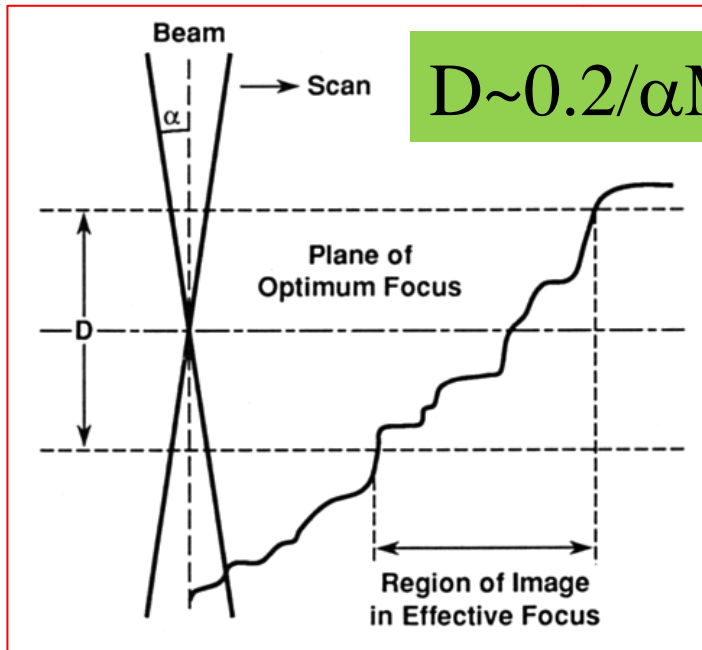
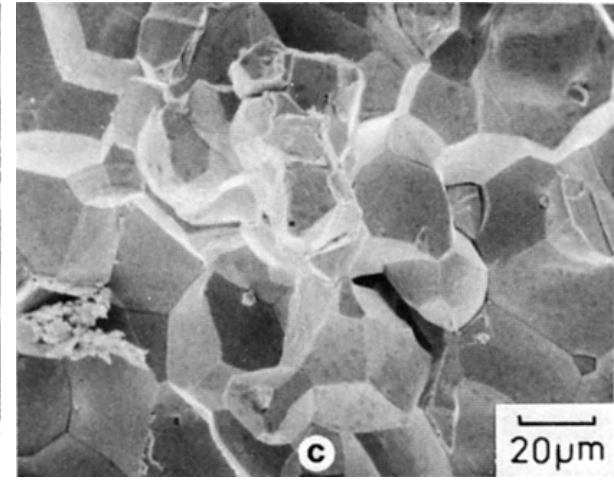
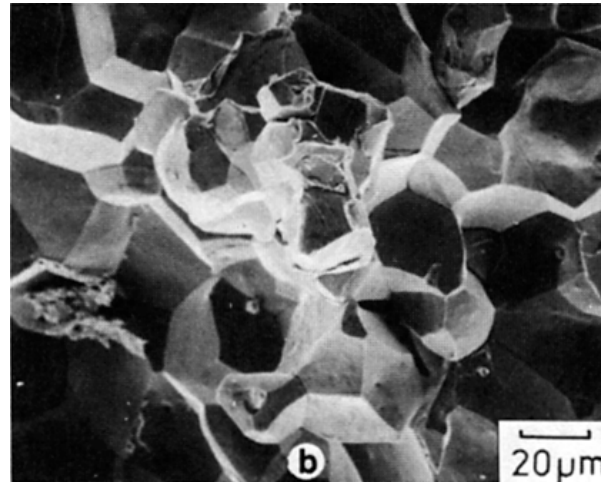
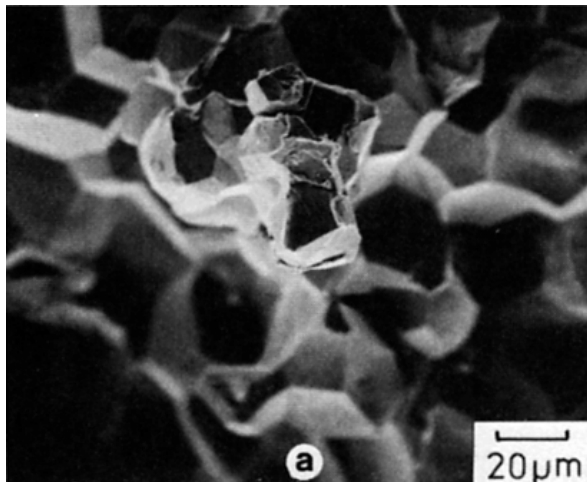


Table 4.3. Depth of Focus (Field) in μm

Magnification	α (rad)		
	5×10^{-3}	1×10^{-2}	3×10^{-2}
10X	4,000	2,000	670
50X	800	400	133
100X	400	200	67
500X	80	40	13
1,000X	40	20	6.7
10,000X	4	2	0.67
100,000X	0.4	0.2	0.067



전자기렌즈에 의한 분해능의 한계

Ideal Res. Limit : **Rayleigh limit**

Practical Res. is determined by **various Lens aberrations**

$$d = \sqrt{d_d^2 + d_s^2 + d_c^2 + d_a^2} \quad \text{Negligible } d_c, d_a \quad d = \sqrt{d_d^2 + d_s^2}$$

$$d_d = \frac{0.61\lambda}{\alpha}$$

Diffraction 수차

$$d_s = C_s \alpha^3 M$$

C_s : 구면수차 계수

$$d_a = \alpha^3 \Delta f$$

Astigmatism: stigmator로 보정

$$d_c = C_c \alpha \frac{\Delta E}{E}$$

C_c : 색수차 계수
 ΔE : 0.2-0.3eV for cold FE

Spherical aberration and aperture diffraction vary in opposite directions with α . This leads to the need to find an optimum aperture angle α_{opt} .

SEM - scanning electron microscopy

tiny electron beam scanned across surface of specimen

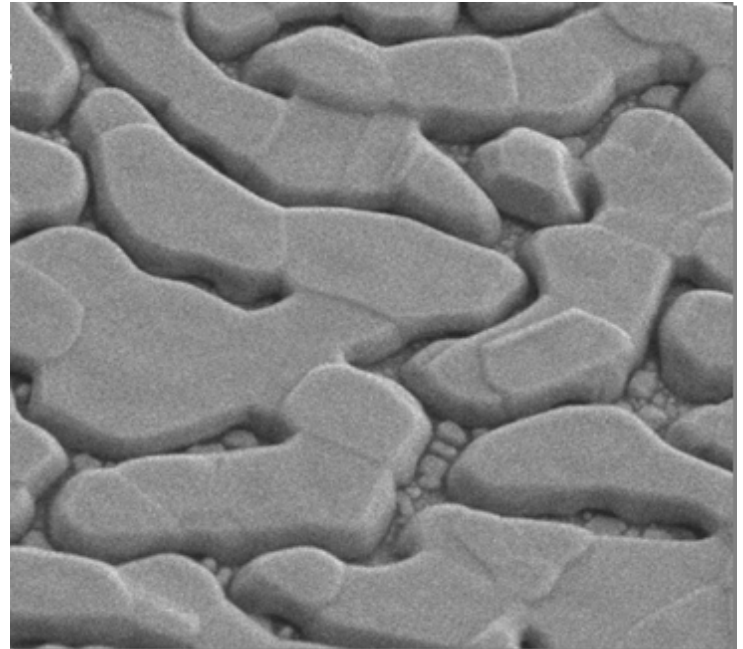
backscattered or secondary electrons detected

Magnification range 15x to 200,000x

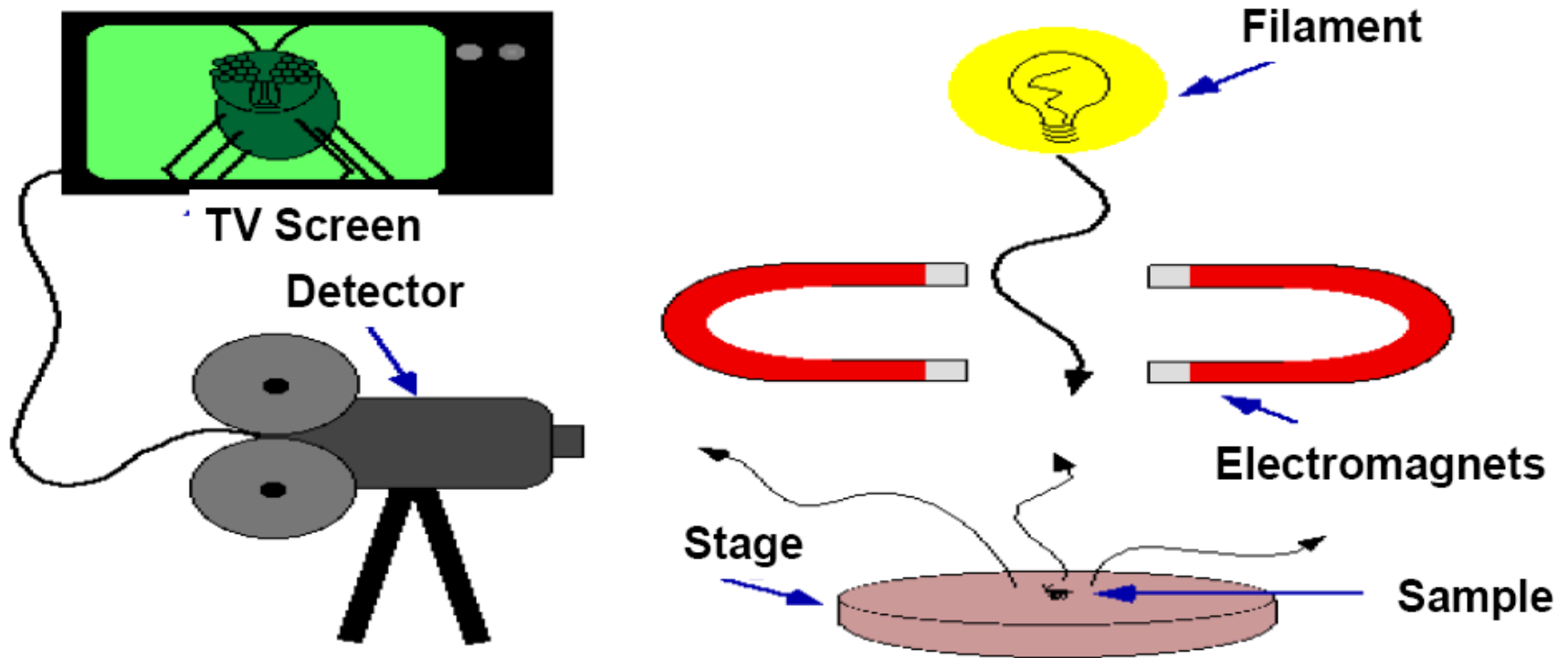
Resolution of 50 Å

Excellent depth of focus

Relatively easy sample prep

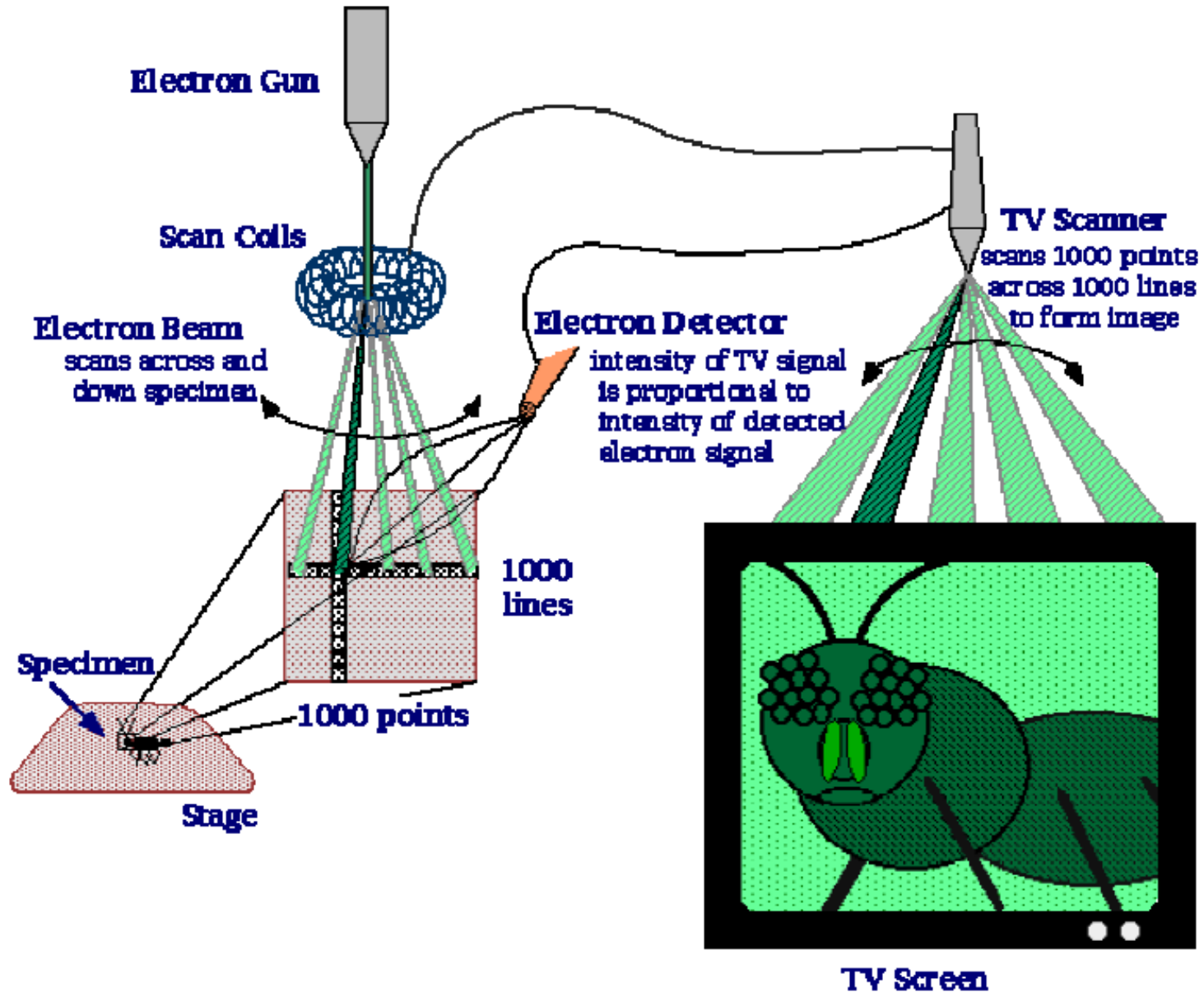


Breakdown of an electron microscope

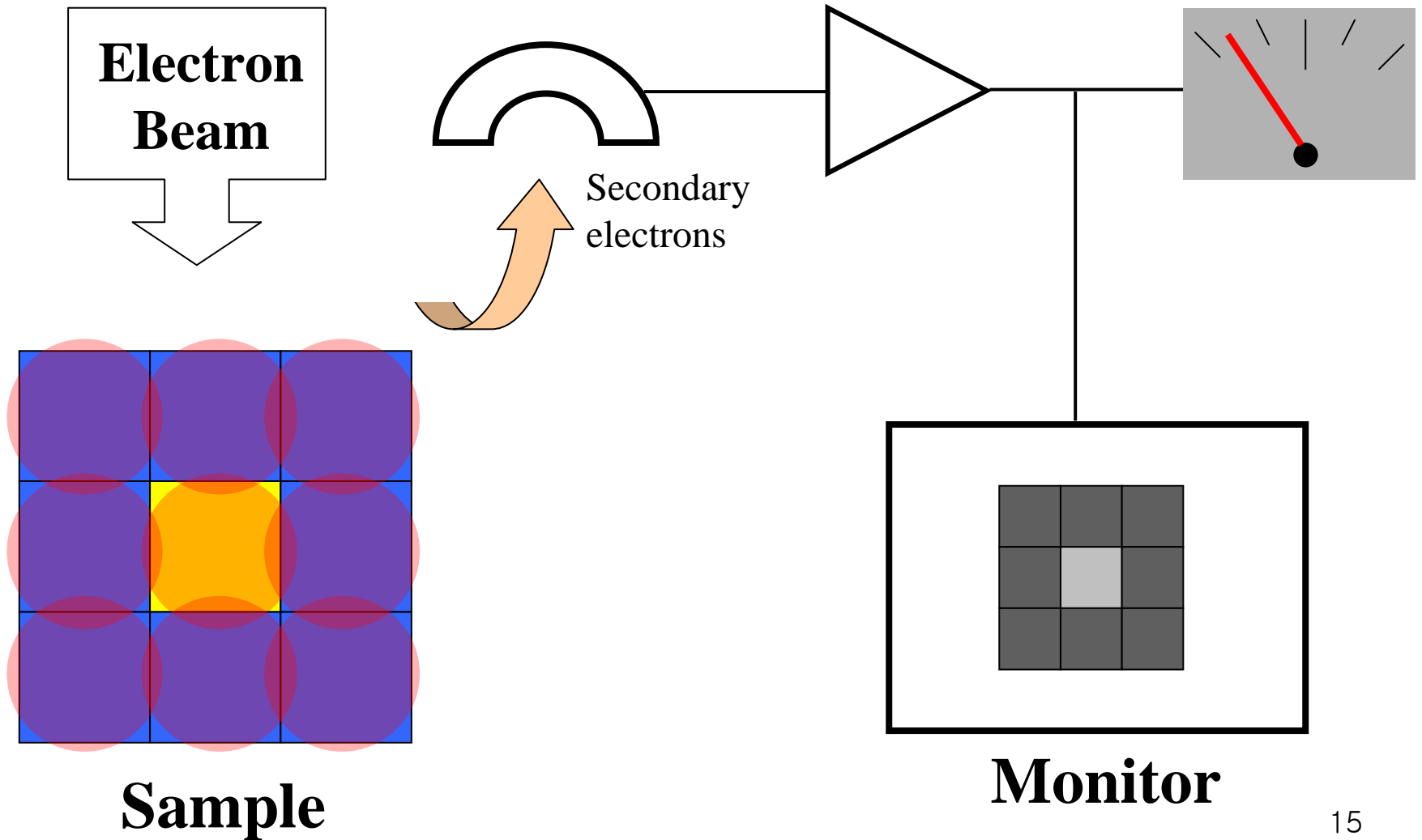


In simplest terms, an SEM is really nothing more than a television. We use a filament to get electrons, magnets to move them around, and a detector acts like a camera to produce an image.

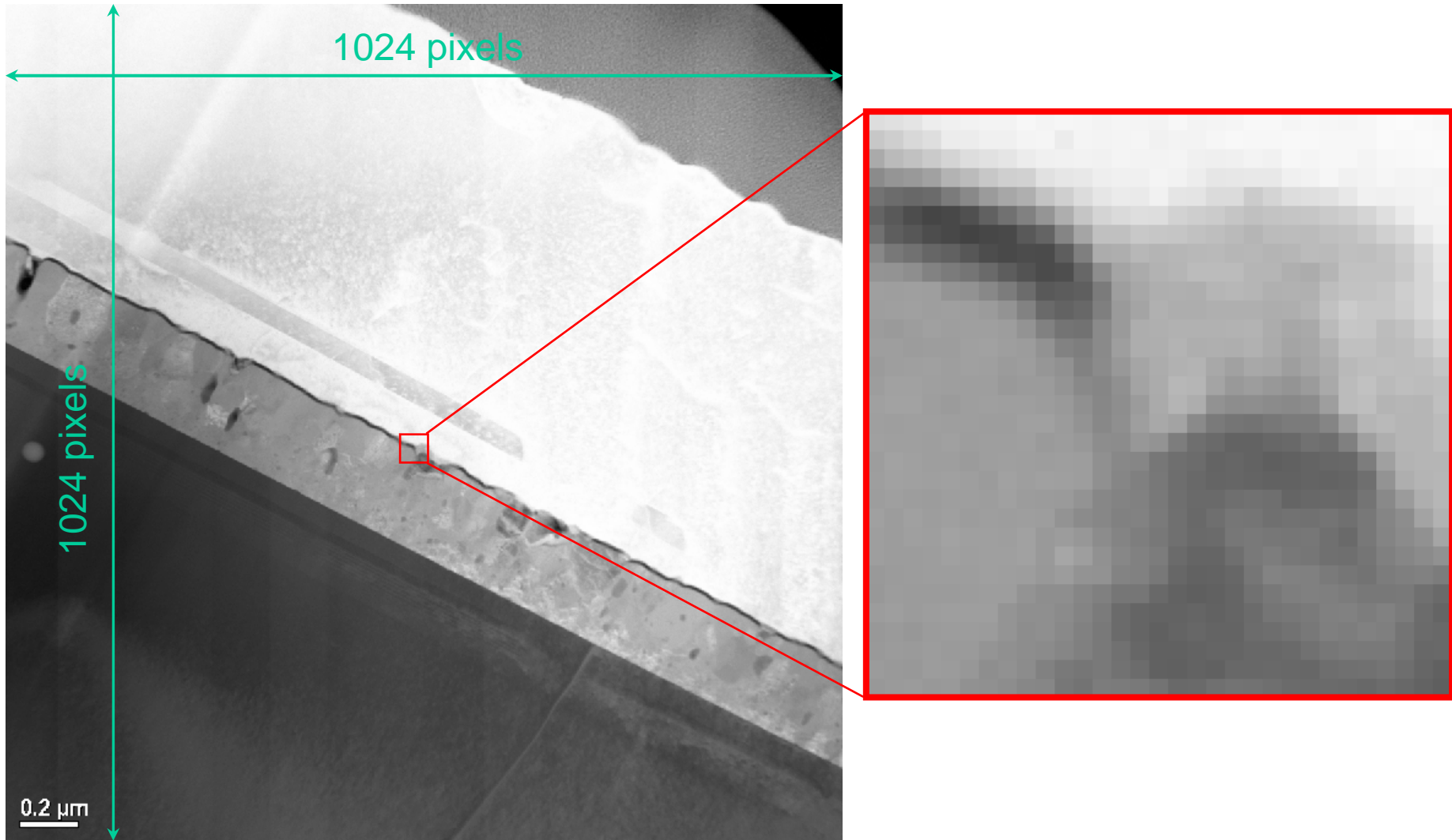
SEM Operation



Scanning Technology

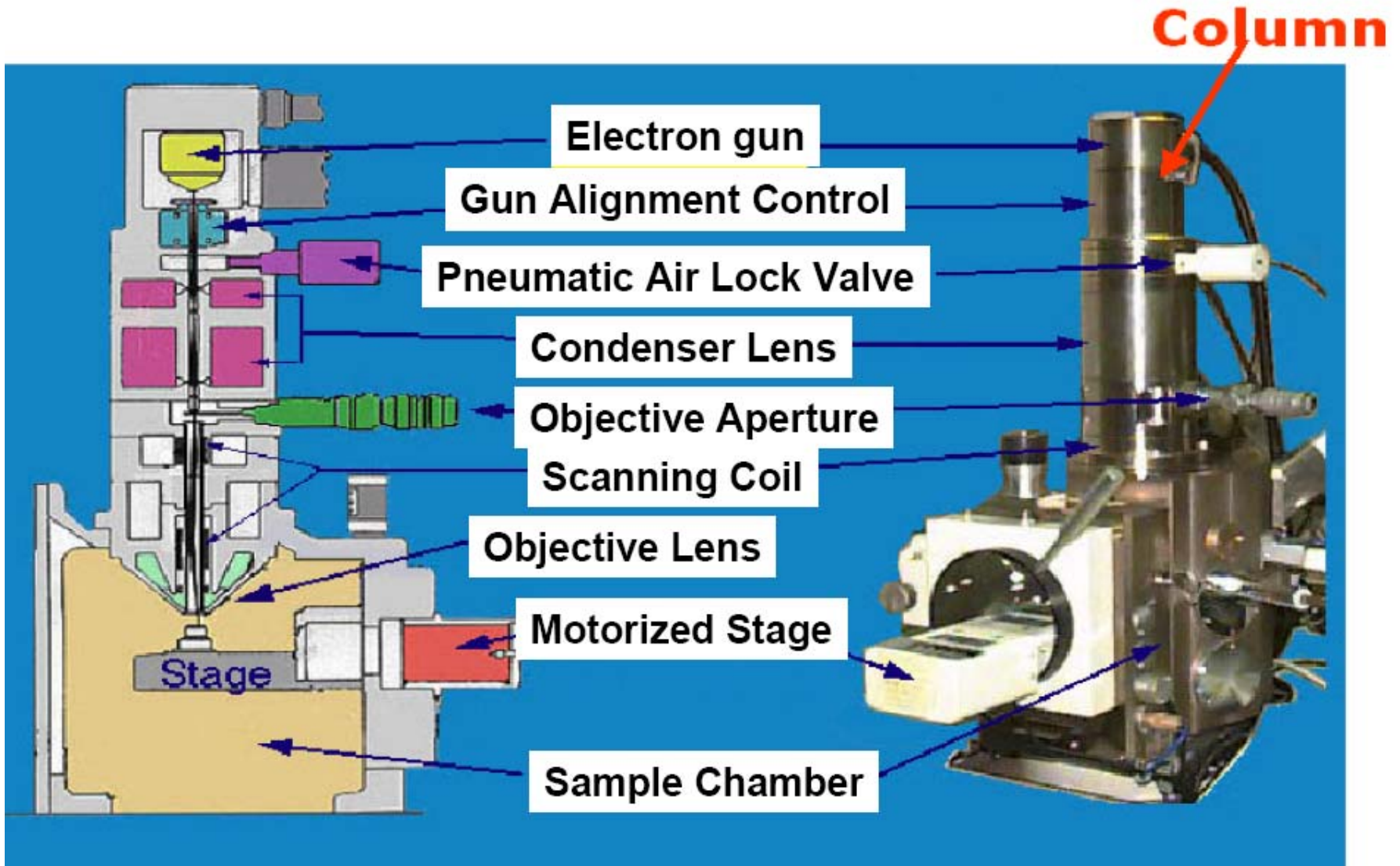


Pixel by pixel image



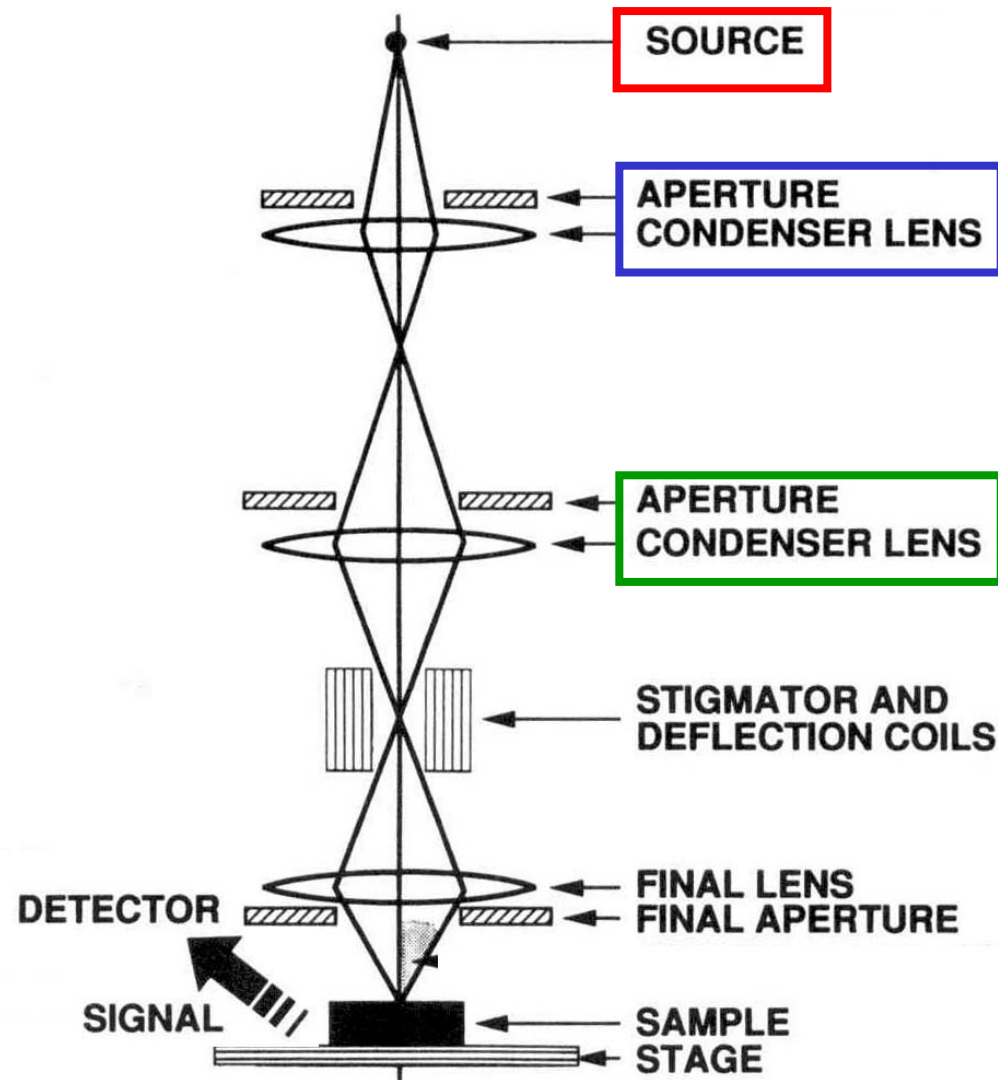
Digital Z-contrast image

A look inside the column



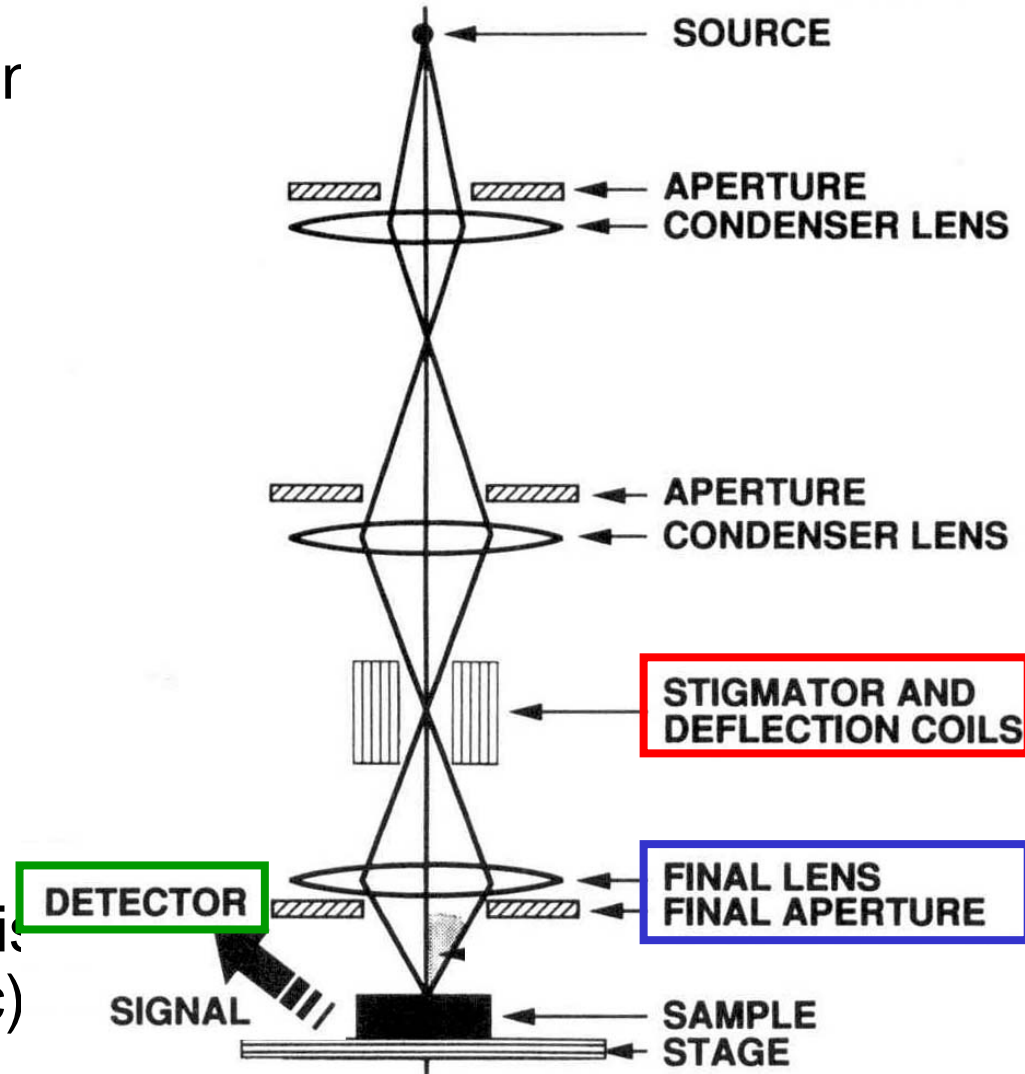
SEM: Optics #1

- Electron gun produces beam of monochromatic electrons.
- First condenser lens forms beam and limits current ("coarse knob").
 - Condenser aperture eliminates high-angle electrons.
- Second condenser lens forms thinner, coherent beam ("fine knob").
 - Objective aperture further eliminates high-angle electrons from beam.



SEM: Optics #2

- Beam "scanned" by deflector coils to form image.
- Final objective lens focuses beam onto specimen.
- Beam interacts with sample and outgoing electrons are detected.
- Detector counts electrons at given location and displays intensity.
- Process repeated until scan is finished (usu. 30 frames/ sec)



Electron Gun (전자빔)

Electron source



Condenser lens



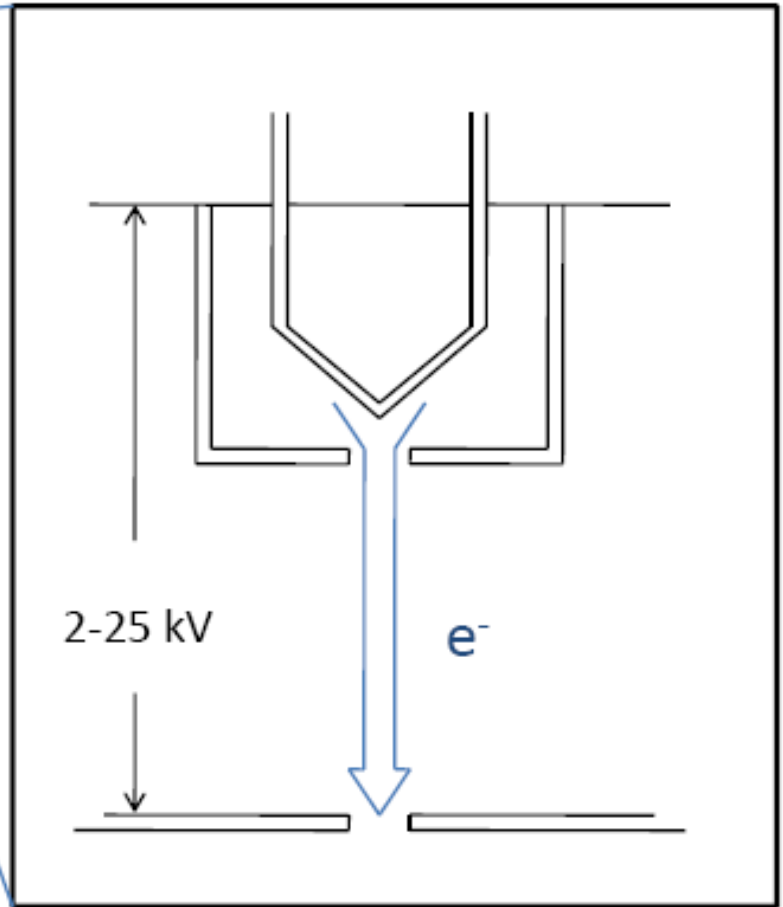
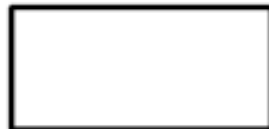
Scan coil



Objective lens



Specimen
+ Detectors

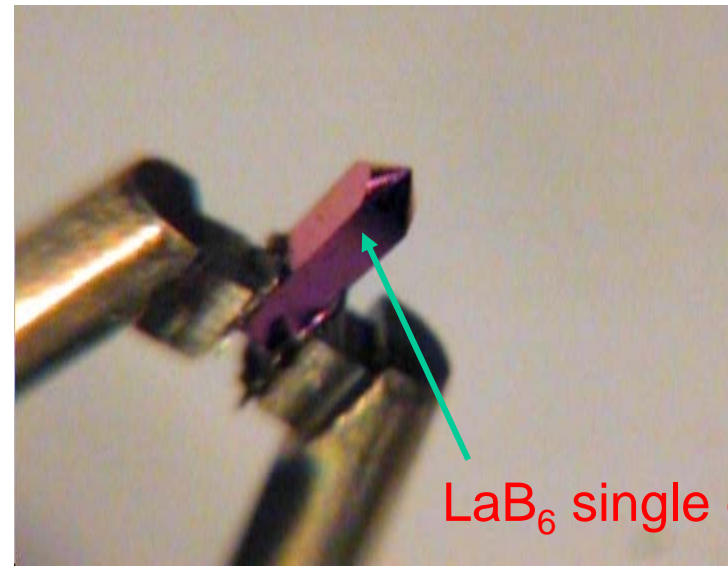
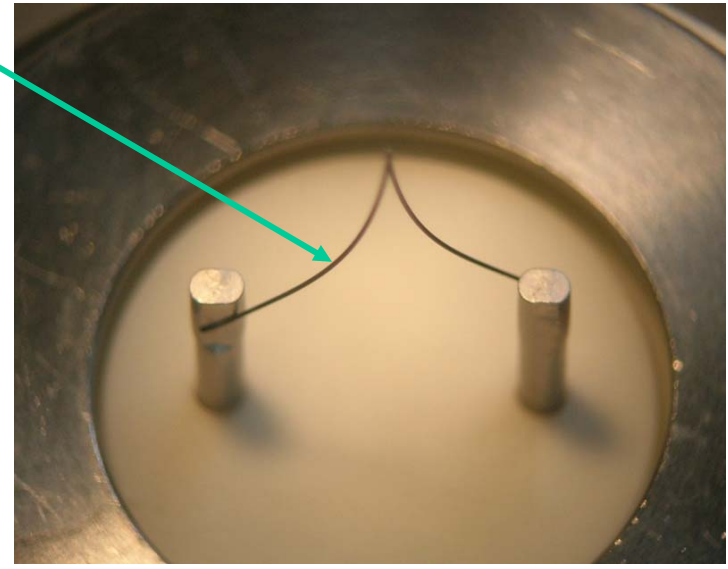
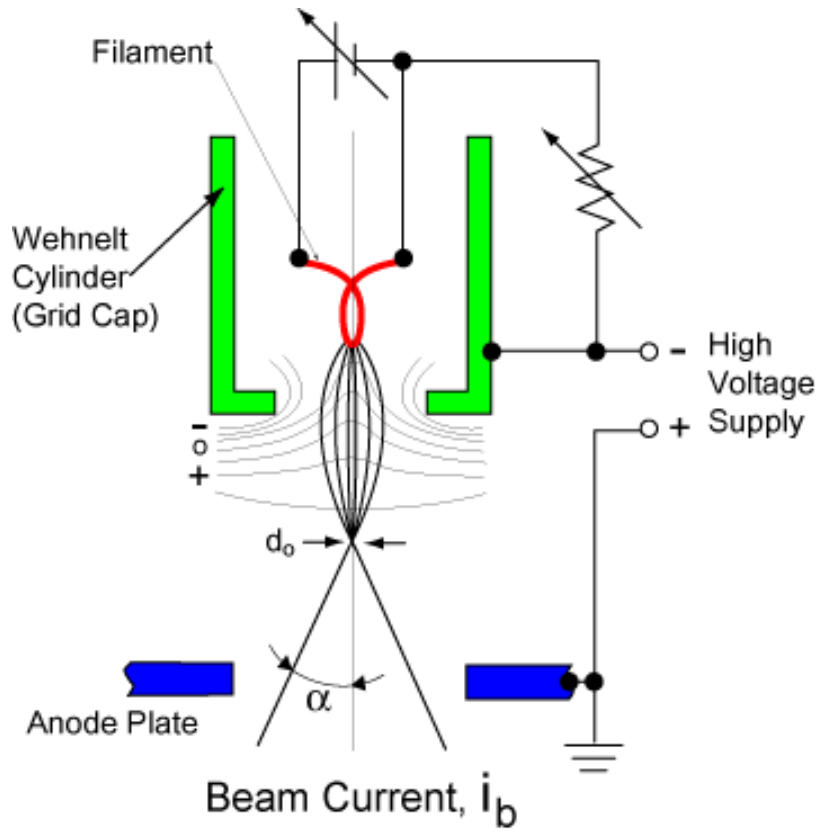


Electron Emission-electron gun

- Cathode of the gun is the source of electrons for the beam in the electron microscope
- Thermionic emission – heat
- Field emission – strong electrical field
- Photoelectric emission – electromagnetic radiation

Thermionic Emission

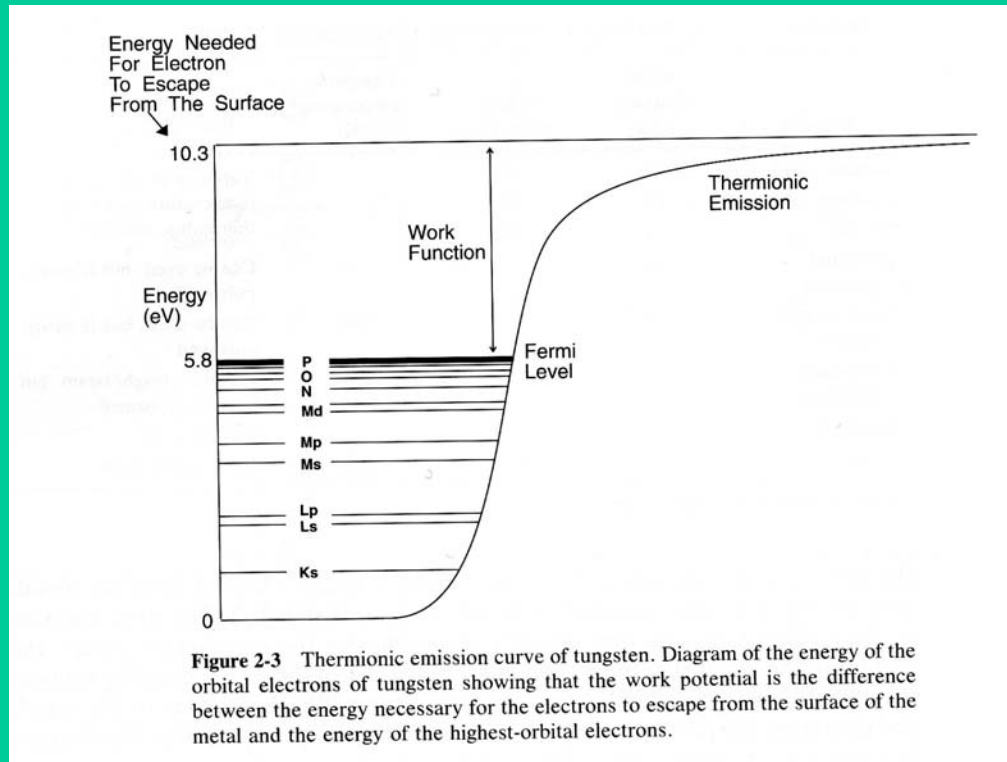
Tungsten Hairpin



Self biasing from a resistor

LaB₆ single crystal

Thermionic Emission(열전자 방출)



Work Function: 전자가 금속표면을 탈출할 때 넘어야 하는 potential energy(V)

W.F 낮을 수록 전자방출이 쉽다

W.F 낮은 금속: 원자반경(atomic radius)이 크다

결정구조에서 원자간 간격이 크다

Orbital Electrons in Tungsten(W)

TABLE 2-1 CONFIGURATION OF THE ORBITAL ELECTRONS IN LANTHANUM AND TUNGSTEN ATOMS

	Atomic number	K		L		M			N				O			P
		<i>s</i>	<i>s</i>	<i>p</i>	<i>s</i>	<i>p</i>	<i>d</i>	<i>s</i>	<i>p</i>	<i>d</i>	<i>f</i>	<i>s</i>	<i>p</i>	<i>d</i>	<i>s</i>	
Lanthanum	57	2	2	6	2	6	10	2	6	10		2	6	1	2	
Tungsten	74	2	2	6	2	6	10	2	6	10	14	2	6	4	2	

The electrons that are circled are far from the atomic nucleus and are the main contributors to thermionic emission.

Negative electron은 원자의 positive nuclei 주위의 전자궤도 (orbit)에 속박되어 있다.

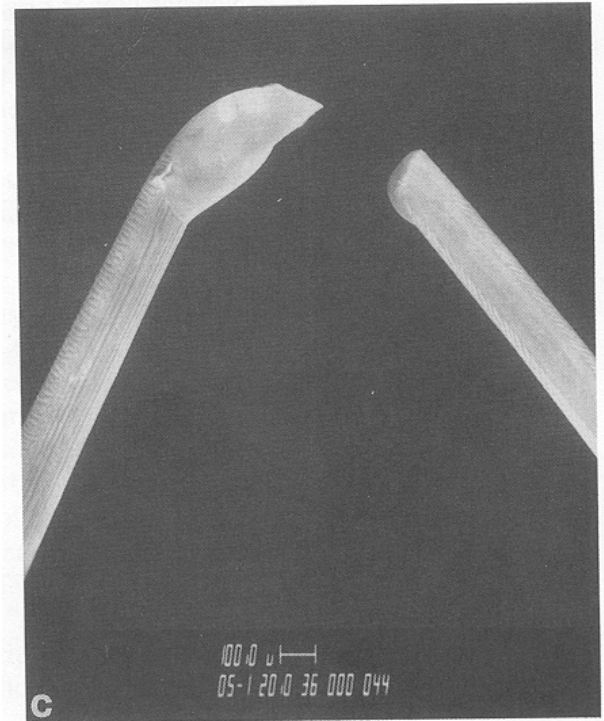
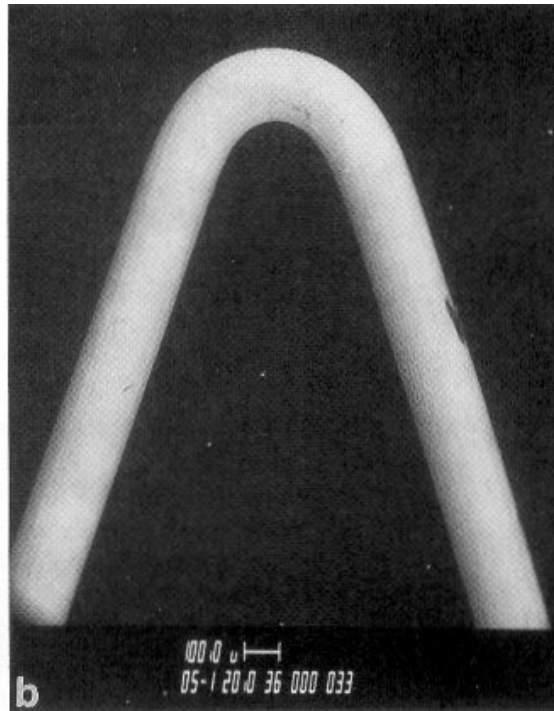
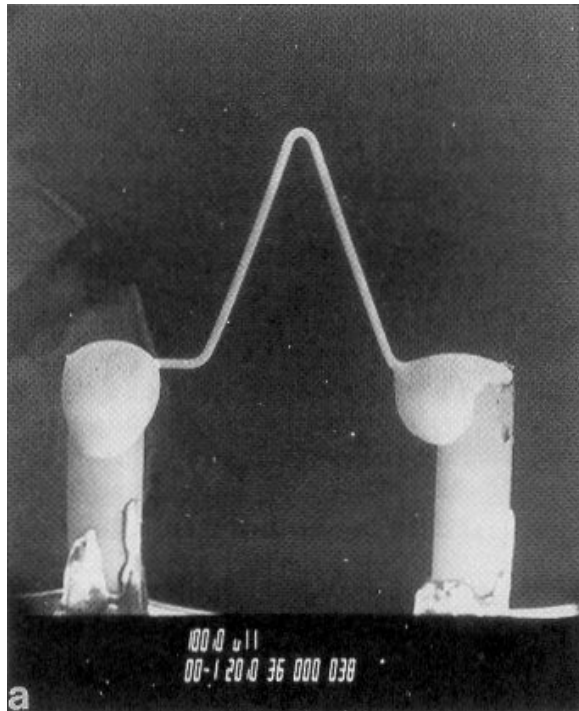
가장 바깥궤도에 있는 전자: valence electrons

작은 holding force와 높은 에너지를 가지므로 상대적으로 떼어내기 쉬우나, 낮은 표면탈출에 필요한 운동에너지가 적음

1. Tungsten Hairpin Electron Gun

Inexpensive, low mag.

high current (x-ray microanalysis), low vacuum(10^{-5} torr), $t=30-100$ h



V-shaped hairpin tip of $d = 100\mu\text{m}$

대부분의 금속 : 열전자 방출 전에 용융, 용점이 높은 W 사용

Electron source assembly

Hitachi



JEOL



Removed Wehnelt cap



Electron Gun(W-filament)

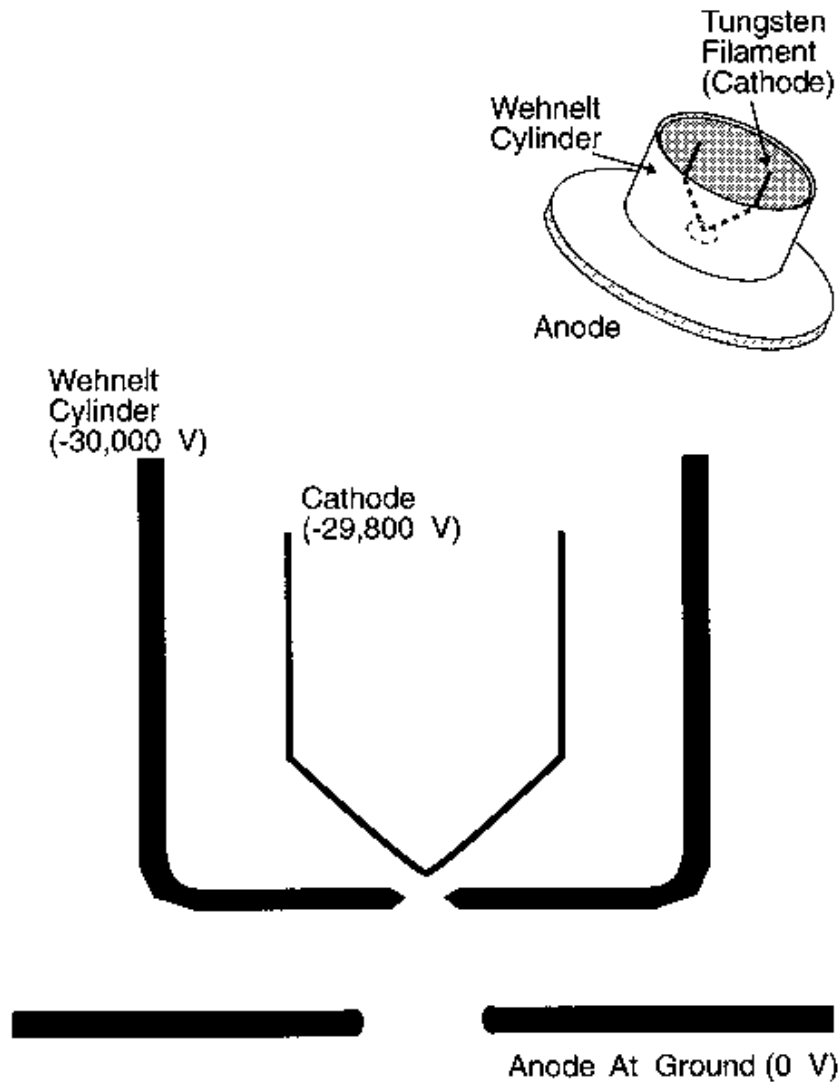
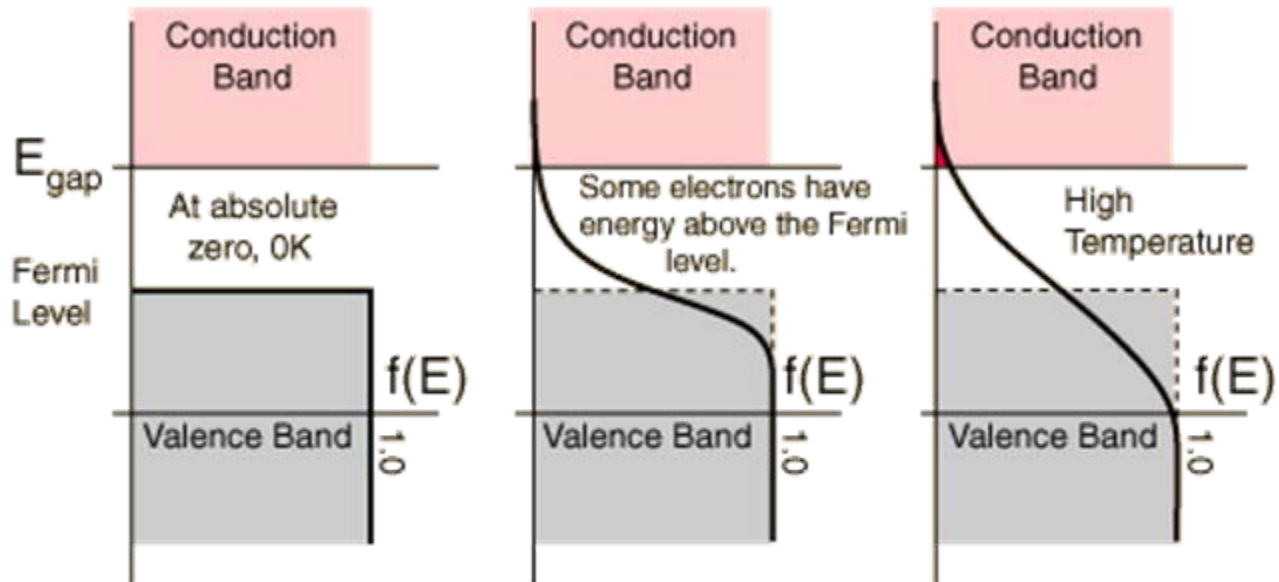


Figure 2-4 Biased electron guns, showing the position of the cathode, Wehnelt cylinder, and anode.

Thermal emission



No electrons can be above the valence band at 0K, since none have energy above the Fermi level and there are no available energy states in the band gap.

At high temperatures, some electrons can reach the conduction band and contribute to electric current.

$$f(E) = \frac{1}{\exp\left(\frac{E - E_f}{kT}\right) + 1} \approx \exp\left(-\frac{E - E_f}{kT}\right)$$

Operation of the Self-biased Gun

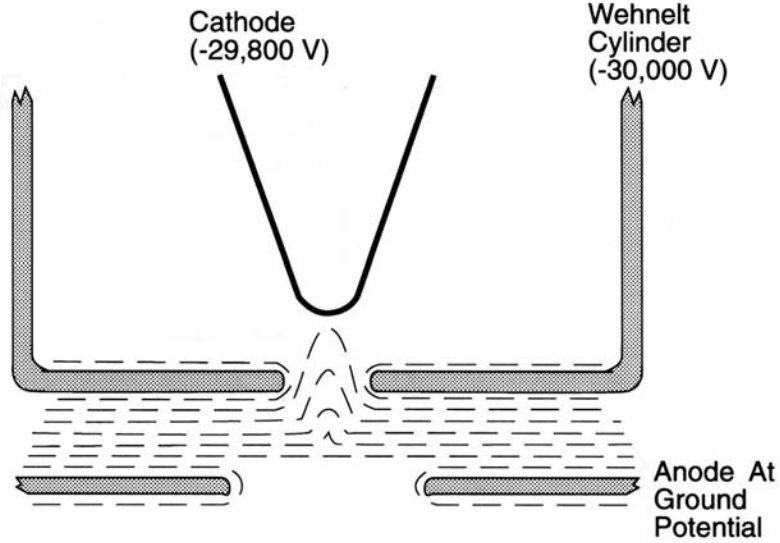


Figure 2-10 Equipotential lines of electrostatic force in a biased gun.

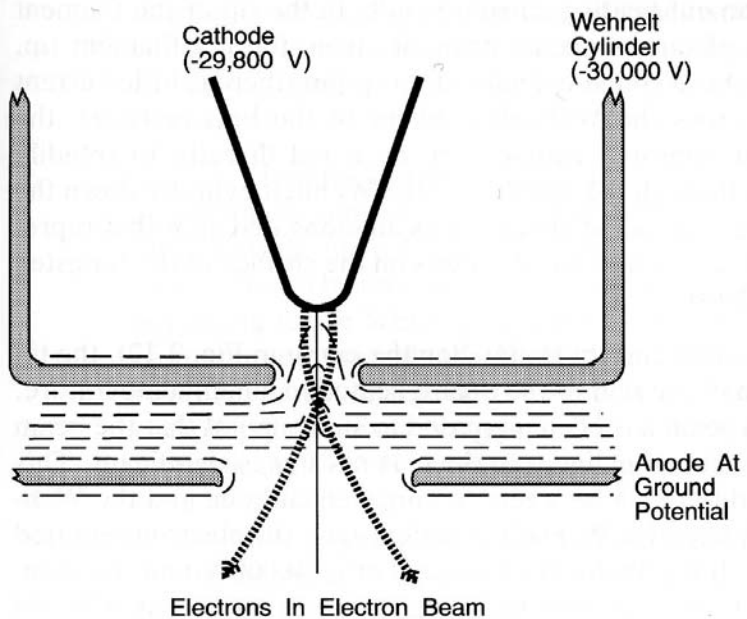


Figure 2-11 Path of the be electrons in a self-biased gt

열전자 방출 전자총

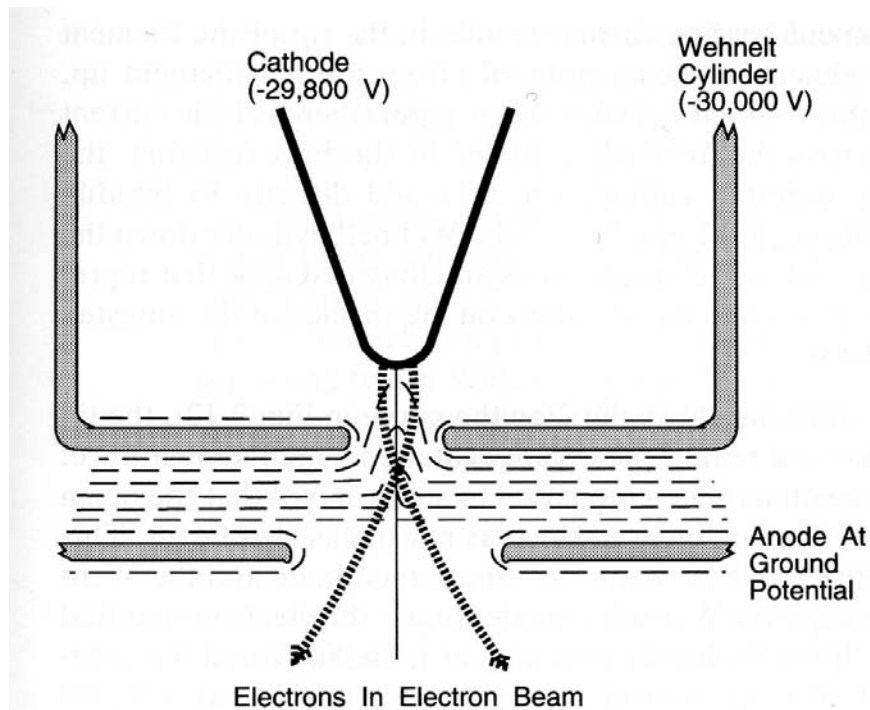


Figure 2-11 Path of the be electrons in a self-biased gu

Cathode(Filament): 열전자 방출(W, LaB₆)

Wehnelt: 열전자 집속, 1~3 mm 직경의 aperture (hole)

Anode: 열전자 가속

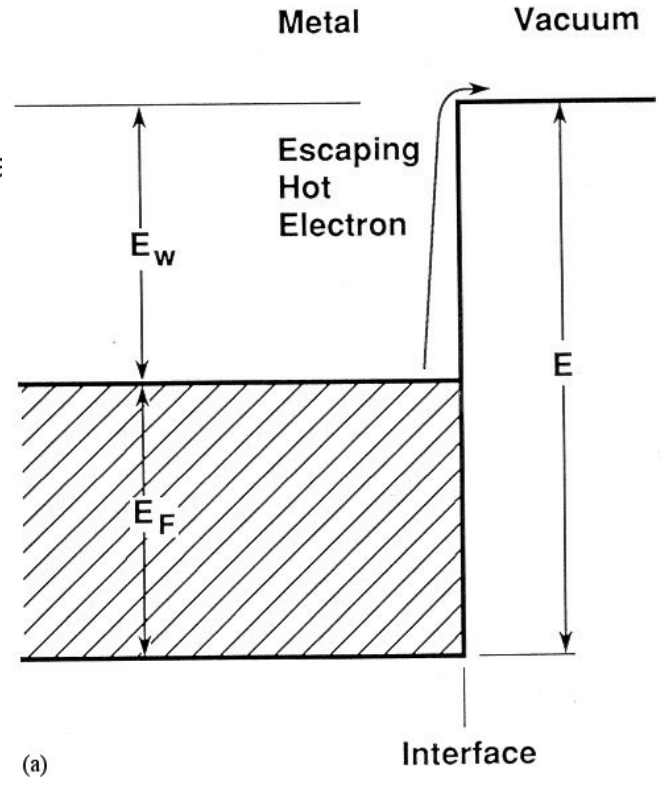
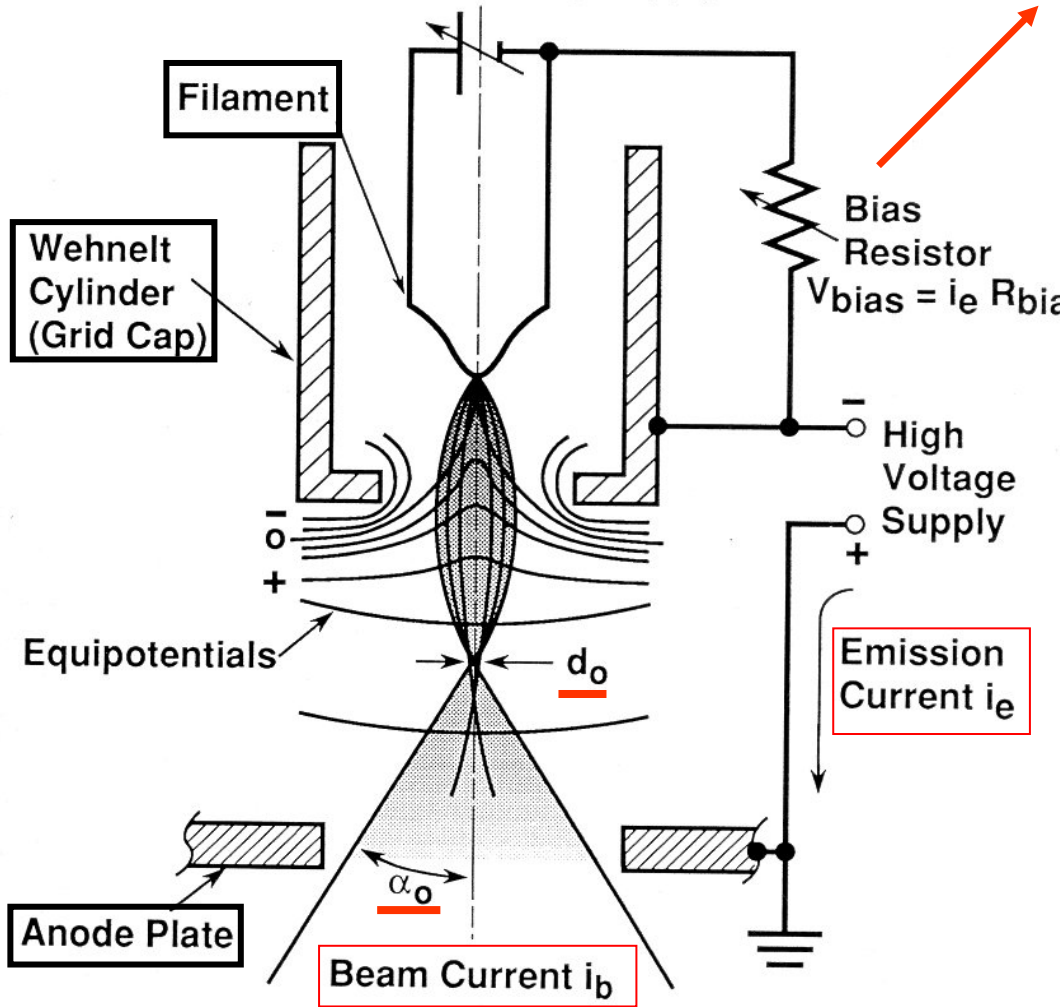
가속전압(Accelerating Voltage): cathode와 Anode 사이의 potential 차이

- 보통 30,000V (30kV)

Filament current (i_f)

Filament Heating Supply

Self Biased Electron Gun



Richardson's law

work function

$$J = A_c T^2 \exp(-E_w / kT) \quad : \text{방출 전자 전류밀도 (A/cm}^2\text{)}$$

◆ Brightness : concept of current density

$$\beta = \frac{\text{current}}{\text{area} \times \text{solid angle}} = \frac{4i_b}{\pi^2 d^2 \alpha^2} \text{ A/cm}^2 \text{sr}$$

Brightness $\uparrow \Rightarrow$ current \uparrow in same beam size
 \Rightarrow beam diameter \downarrow in same current

Maximum theoretical Brightness (Langmuir eq'n)

$$\beta_{\max} = \frac{J_c e V_o}{\pi k T} \text{ A/cm}^2 \text{sr for thermionic gun} \sim 9.2 \times 10^4 \text{ A/cm}^2 \text{sr}$$

$$\beta_{\max} = \frac{J_c e V_o}{\pi \Delta E} \text{ A/cm}^2 \text{sr for field emission gun}$$

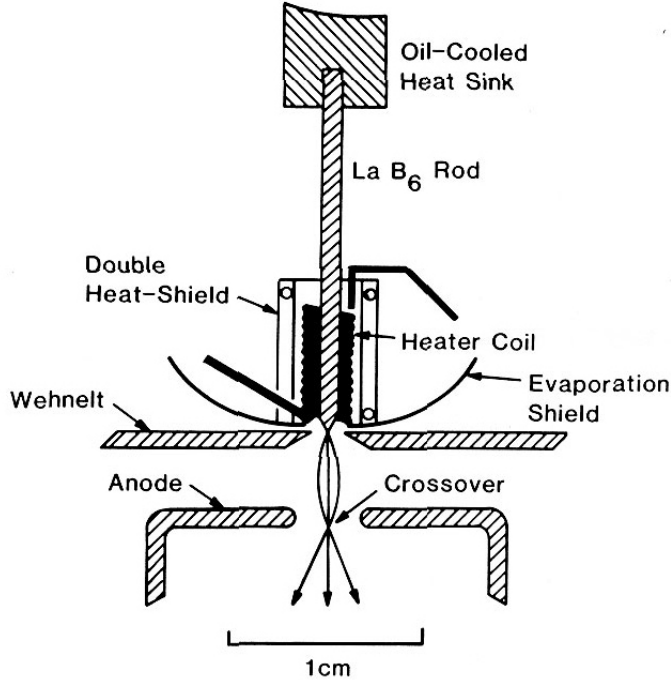
$\rightarrow \Delta E$ 0.3eV for cold emission

$$\beta_{\max} \sim 2 \times 10^9 \text{ A/cm}^2 \text{sr}$$

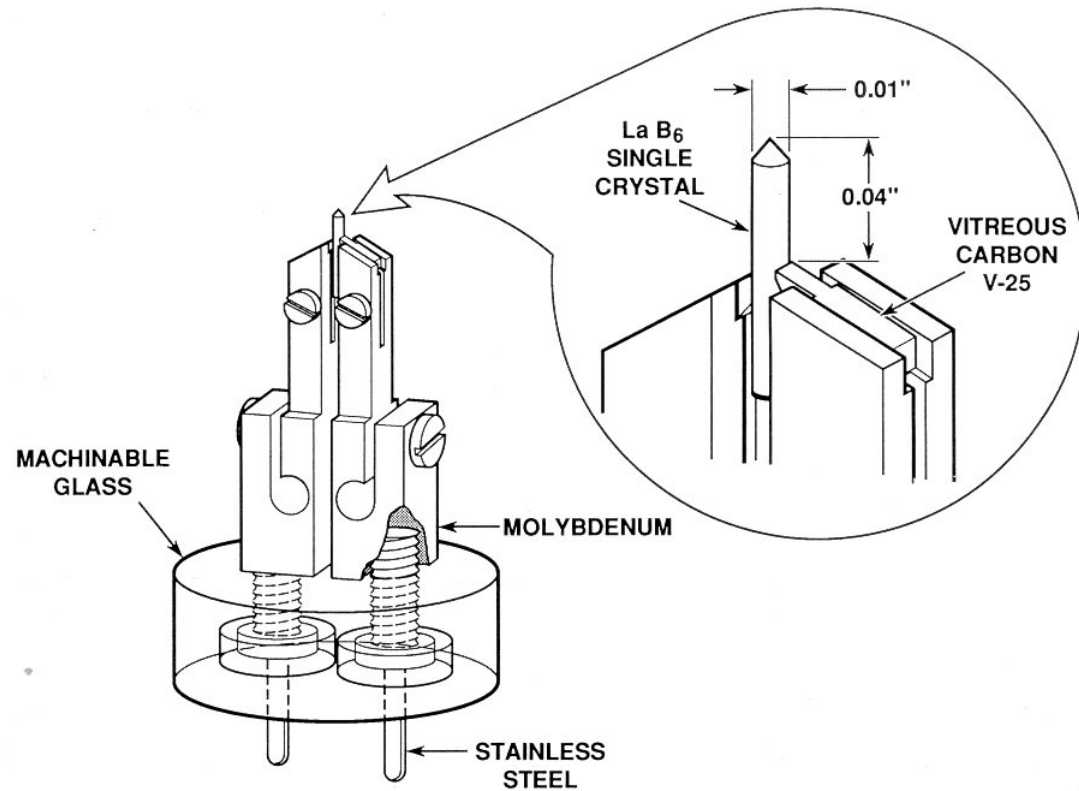
2. LaB₆ Electron Gun

High brightness(5-10times) : lower work function(2.5eV : 4.5eV for W), expensive but longer lifetime(1000hr), high vacuum(10⁻⁷torr)

$$\beta_{\max} = 10^5 \text{ A/cm}^2 \text{sr}$$



간접 가열 방식



직접 가열 방식

Heating current of LaB_6 filament

LaB6_heating

