

2009 spring

***Microstructural Characterization
of
Materials***

04. 20. 2009

Eun Soo Park

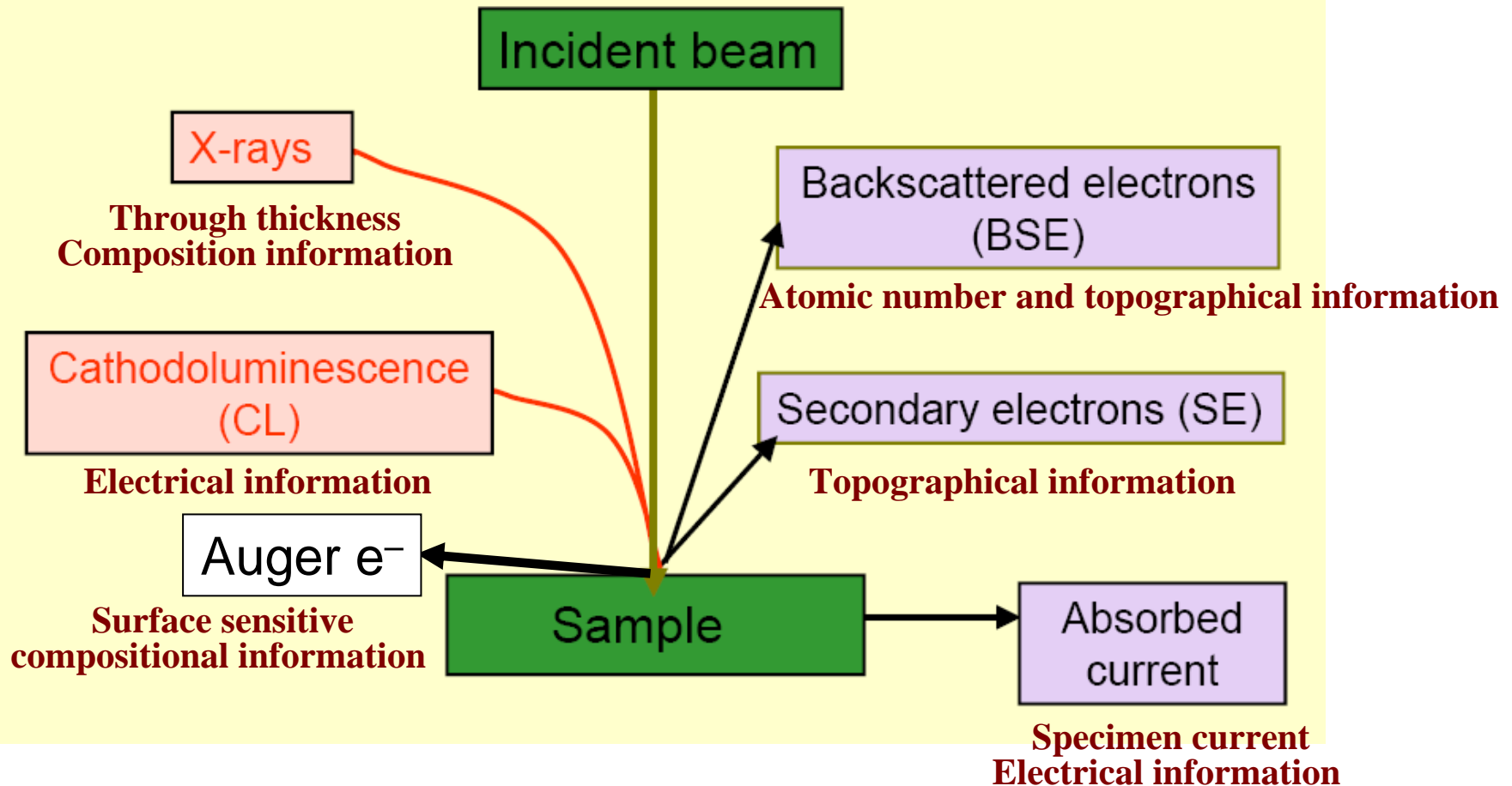
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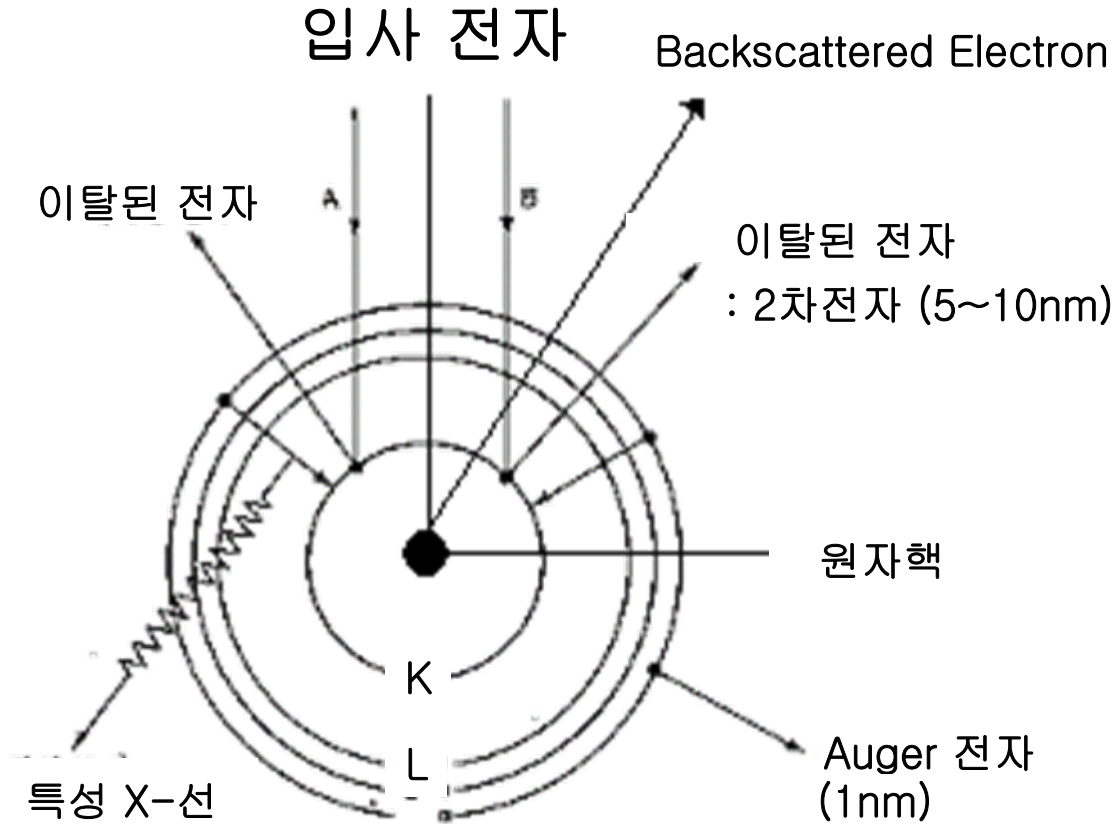
Office hours: by an appointment

Collected Signals in SEM



While all these signals are present in the SEM, not all of them are detected and used for information. The signals most commonly used are the Secondary Electrons, the Backscattered Electrons and X-rays

전자빔과 원자의 반응



1. 2차 전자
 - 입사전자와 충돌하여 이탈된 전자 (표면 형상 정보 전달)

2. 후방산란전자(BSE)
 - 원자핵과 충돌 후 후방으로 산란되는 전자 (원자 번호와 연관 ⇒ 조성정보, EBSD)

3. 특성 X-선
 - 입사빔에 의해 전자각의 전자가 튀어나간 경우 여기된 전자가 아래각으로 내려오면서 발생시키는 X-선 (EDS, WDS를 통해 조성분석)

4. Auger 전자
 - 입사빔에 의해 전자각의 전자가 튀어나간 경우 여기된 전자가 방출 (경량원소에서 많이 나타남, 표면 성분 분석)

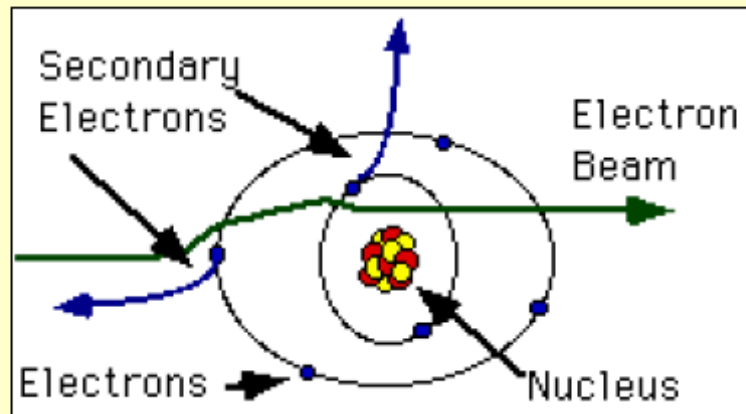
Basic SEM Modes of Operation - Summary

Signal/Mode	Information	Material	Resolution
Secondary electrons (SE)	Morphology	All (*)	~1 nm
Backscattered electrons (BSE)	Atomic number	All (*)	0.1-0.5 μm (**)
X-ray (EDS or WDS)	Atomic composition	All (flat)	~1 μm
(CL)Cathodoluminescence	Bandgap, impurities, lifetimes	Insulators and semi-conductors	~ 1 μm

(*) usually sizes of 1cm, dependent on SEM configuration

(**) voltage and Z dependent

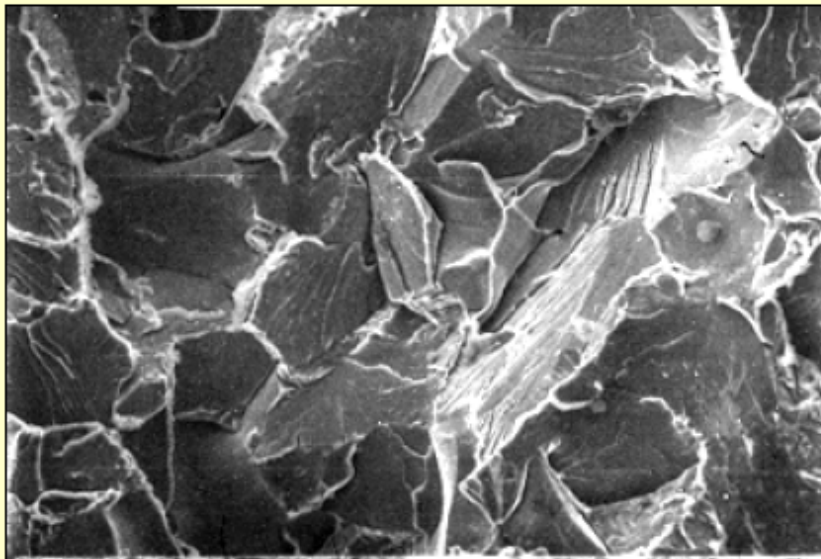
Secondary Electrons



1. **Secondary electrons** are predominantly produced by the interactions between energetic beam electrons and weakly bonded conduction-band electrons in metals or the valence electrons of insulators and semiconductors.
2. There is a great difference between the amount of energy contained by beam electrons compared to the specimen electrons and because of this, only a small amount of kinetic energy can be transferred to the secondary electrons.

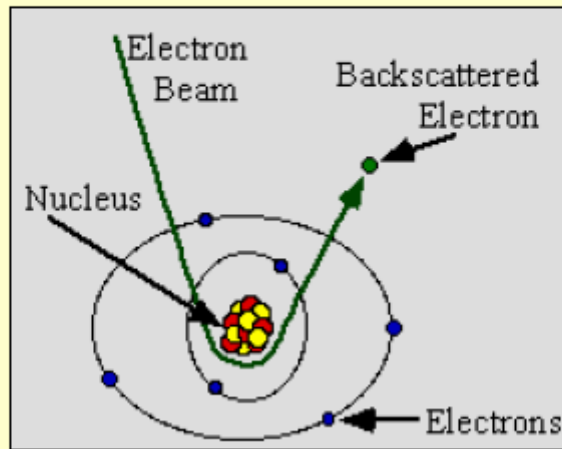
Secondary Electrons and Detection

SE are specimen electrons that obtain energy by **inelastic** collisions with beam electrons. They are defined as electrons emitted from the specimen with energy less than 50 eV



A broken surface of a piece of metal, formed using **SE** imaging

Elastic Scattering – Backscattering



As the name implies, **elastic scattering** results in little (<1eV) or no change in energy of the scattered electron, although there is a change in momentum. Since momentum, $p=mv$, and m doesn't change, the direction of the velocity vector must change. The angle of scattering can range from 0-180 degrees, with a typical value being about 5 degrees.

Elastic scattering occurs between the negative electron and the positive nucleus. This is essentially Rutherford scattering. Sometimes the angle is such that the electron comes back out of the sample. These are **backscattered electrons**.

Backscattered Electron

시편을 구성하고 있는 원소의 종류에 따라 입사된 전자가 후방산란 (back scattering)에 의해 되돌아오는 정도가 다름을 이용하여 이미지를 구성 - (원자번호 ↑, 후방산란전자 ↑)

입사전자의 약 30% 정도

후방산란은 입사된 전자가 한번의 충돌에 의해 입사된 방향의 반대방향으로 되돌아오는 현상

보통의 경우에 후방 산란되는 전자는 여러 번의 탄성산란을 겪고 방출

· Backscatter coefficient η :
$$\eta = \frac{n_{BSE}}{n_B} = \frac{i_{BSE}}{i_B}$$

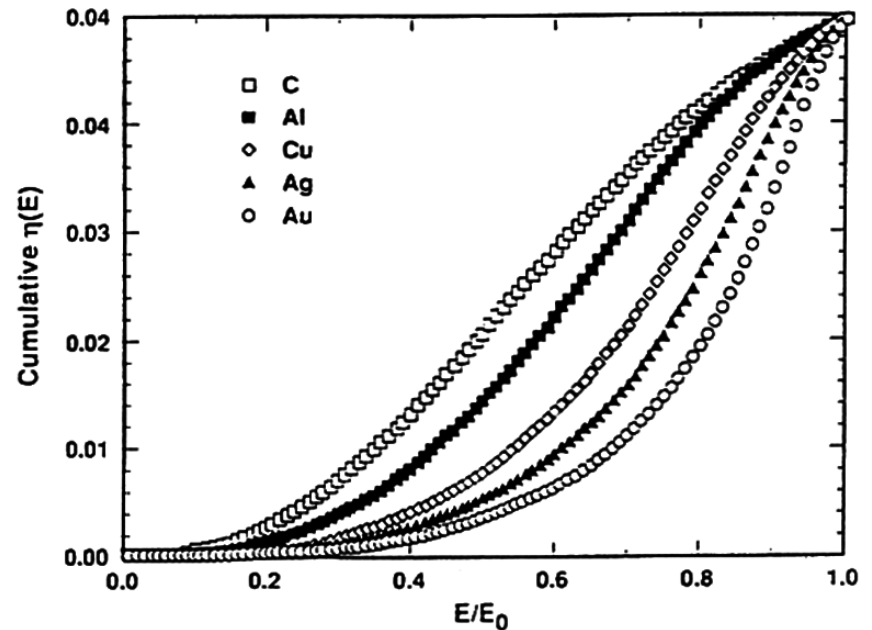
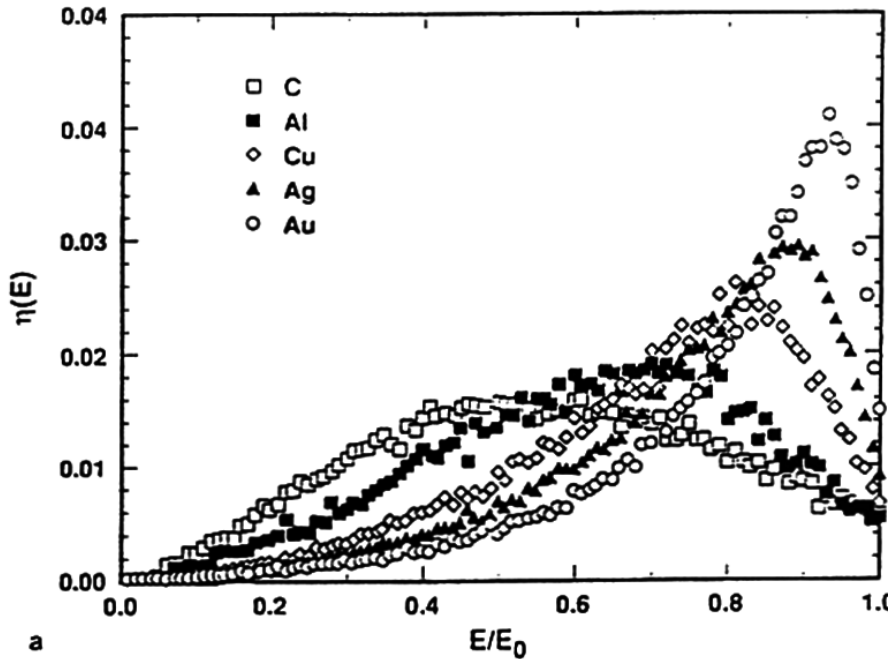
BSEs respond to composition (atomic number, compositional contrast), local specimen surface inclination (topographic or shape contrast), crystallography (electron channeling), and internal magnetic fields (magnetic contrast)

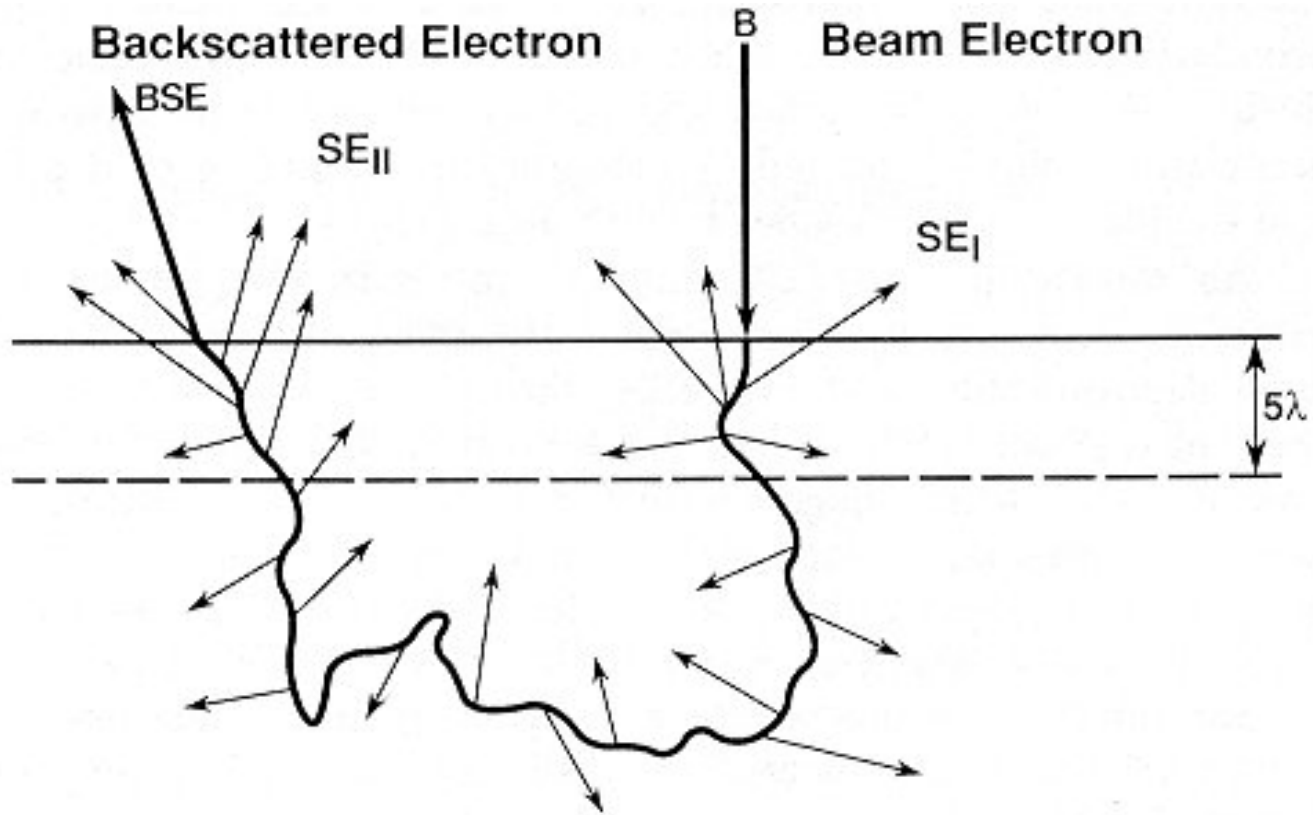
Energy Distribution

후방산란전자는 에너지 손실이 거의 없는 경우에서부터 모든 에너지를 손실하는 경우까지 모두 가능한 분포

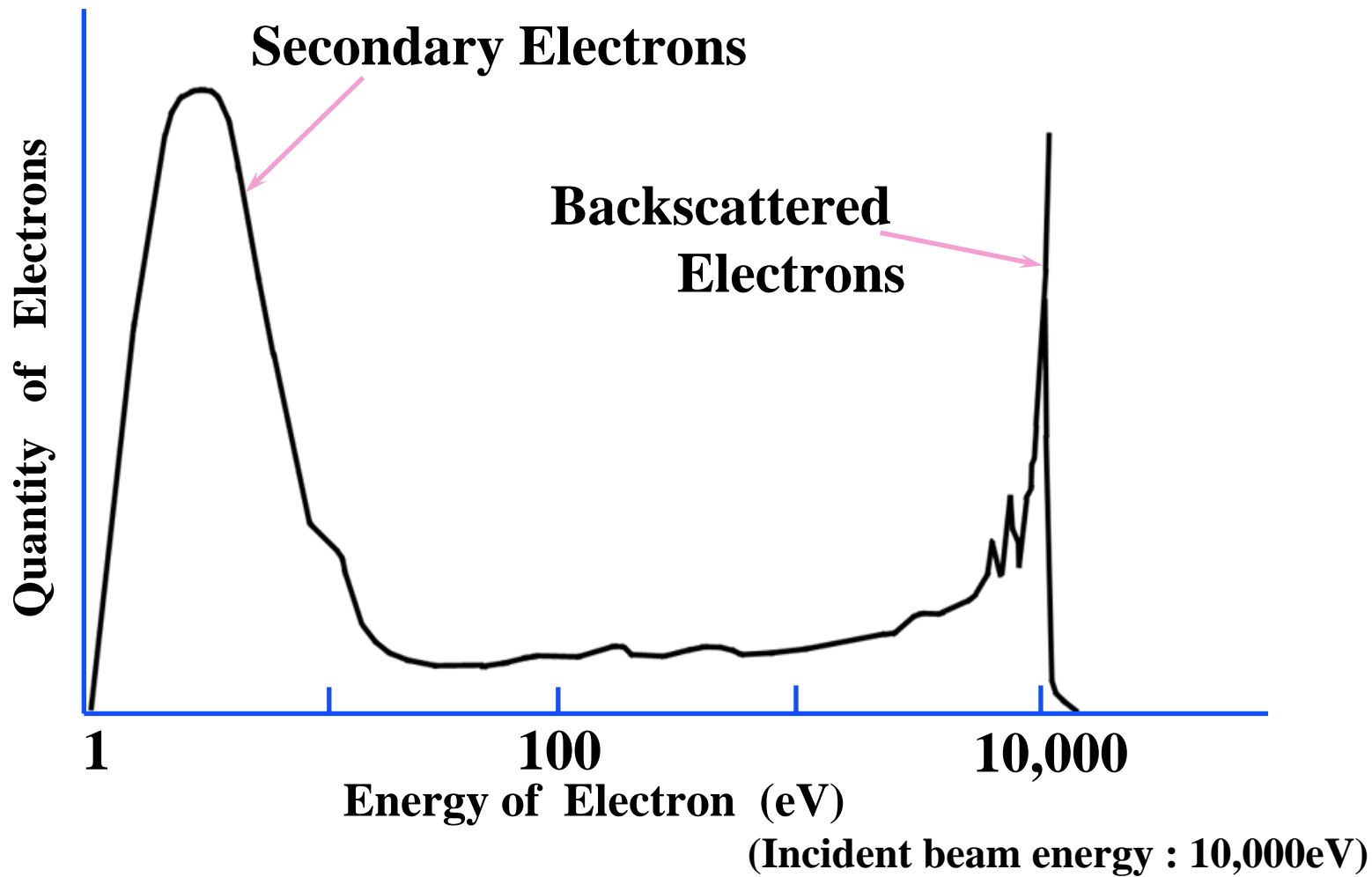
에너지 손실이 약 50% 이내의 전자들이 대부분

에너지 분포 곡선에서 peak이 나타나는 점이 존재하며 이러한 peak point는 원자번호가 증가할수록 E/E_0 값이 1에 가까워진다





- 시편에 입사된 전자가 표면에서부터 5λ 내의 진행과정에서 형성되는 것으로 이를 SE_I 라고 하며 높은 해상도를 나타냄
- 시편에 입사된 전자가 시편 내부에서 많은 충돌과정을 경험하고 시편 밖으로 후방 산란되는 과정 중에 2차 전자를 발생, SE_{II} 라하며 후방산란 전자와 함께 방출



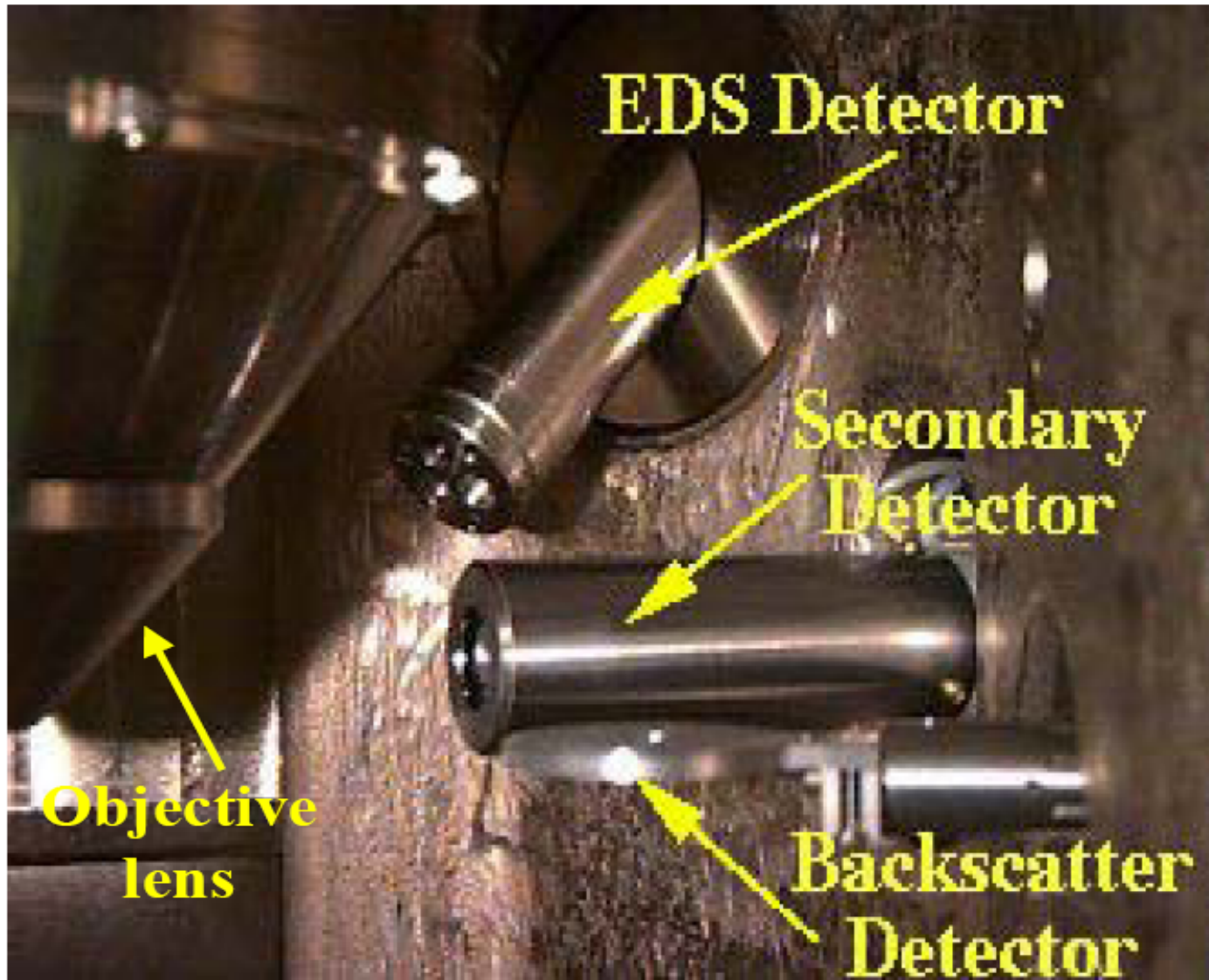
Energy spectrum of the electrons emitted from a specimen

Detection

An electron **detector** is used with the SEM to convert the radiation of interest into an **electrical signal** for manipulation and display by signal processing electronics, which is much like a television. Most SEM's are equipped with an **Everhart-Thornley (E-T)** detector. It works in the following manner:

The scintillator material is struck by an energetic electron. This collision produces photons which are conducted by total internal reflection in a light guide to a photomultiplier. These photons are now in the form of light so they can pass through a vacuum environment and a quartz glass window. The photon is then converted back into an electron current where a positive bias can attract the electrons and collect them so that they will be detected.

Electron Detector



SEM detectors (SE)

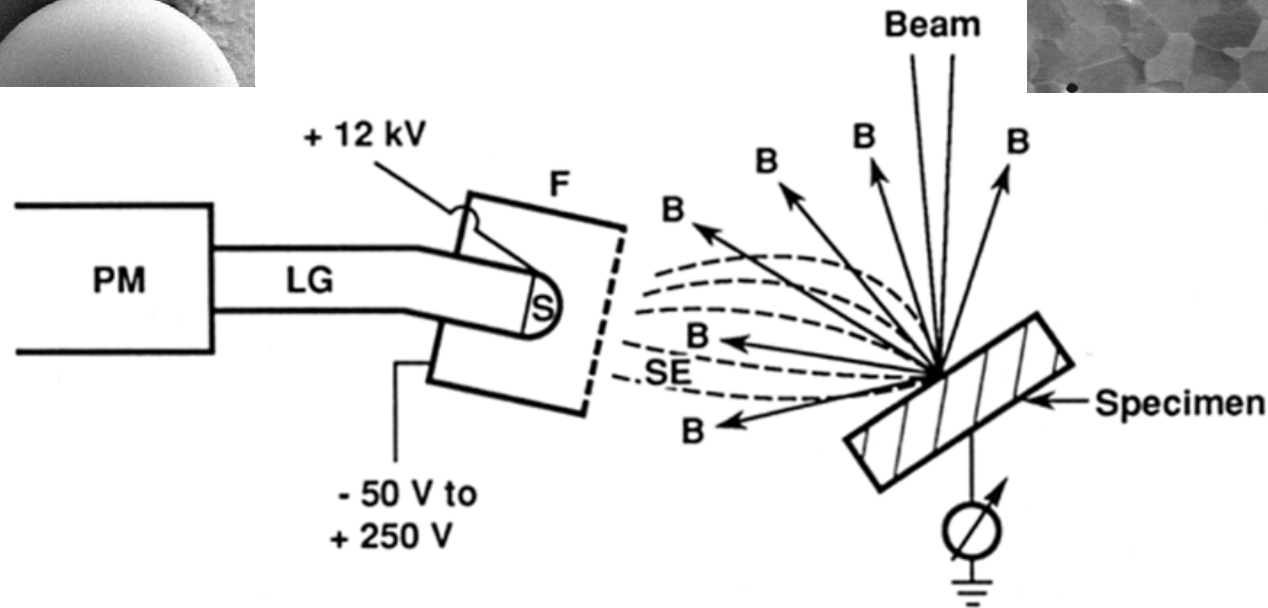
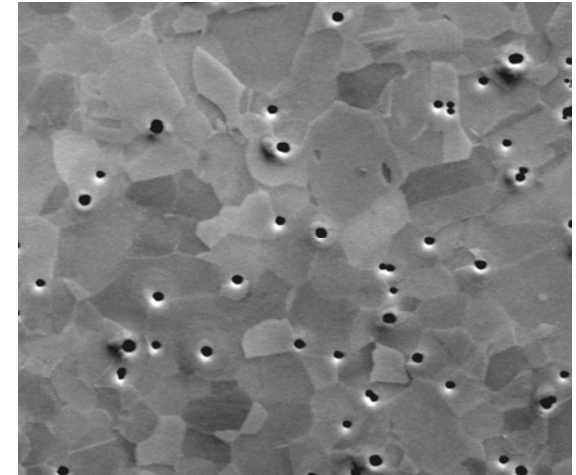
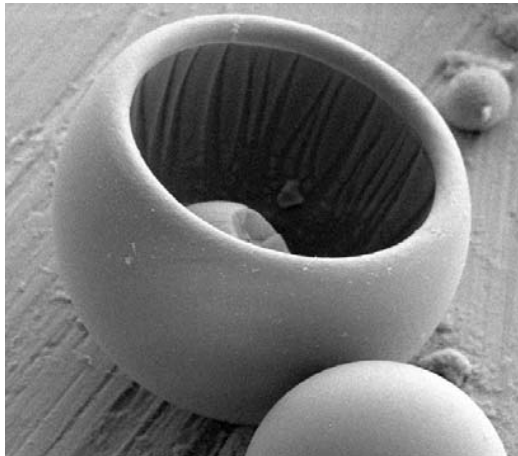
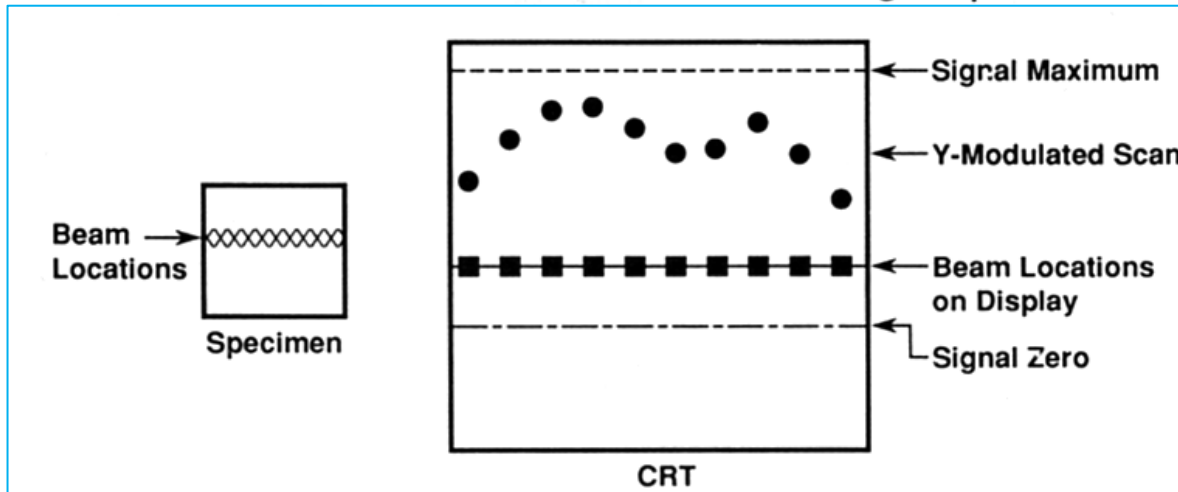
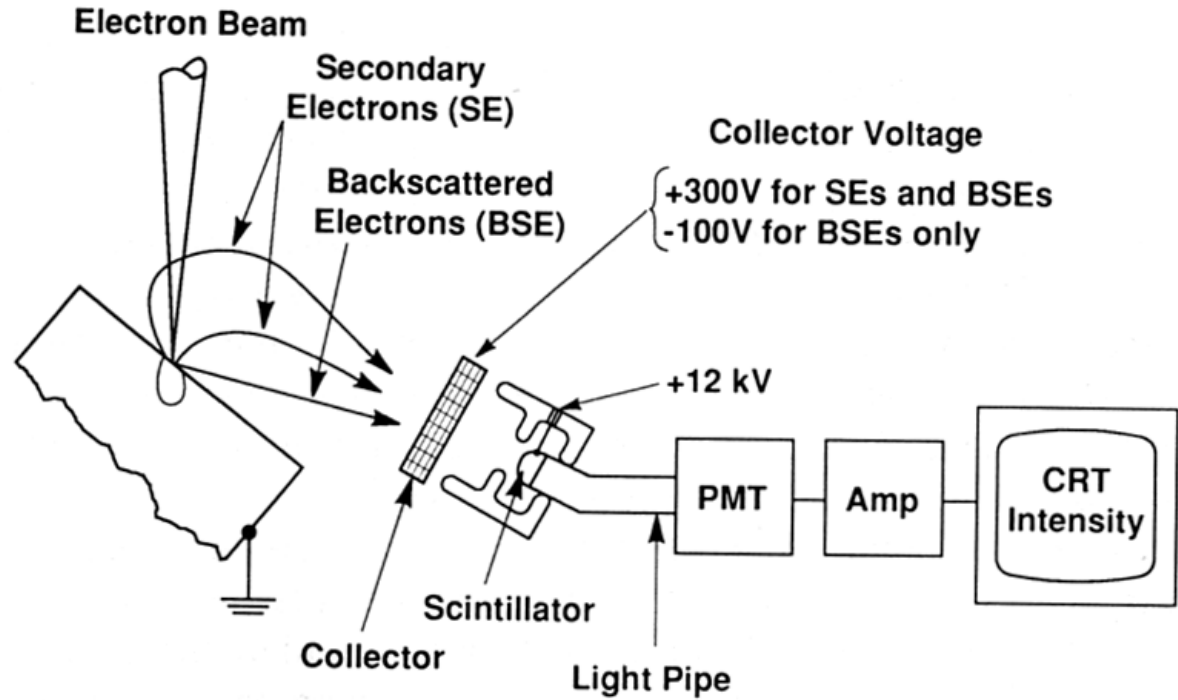
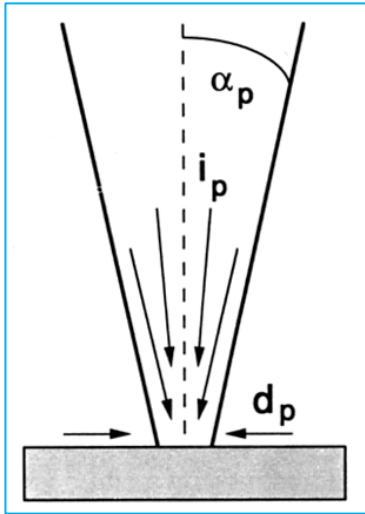
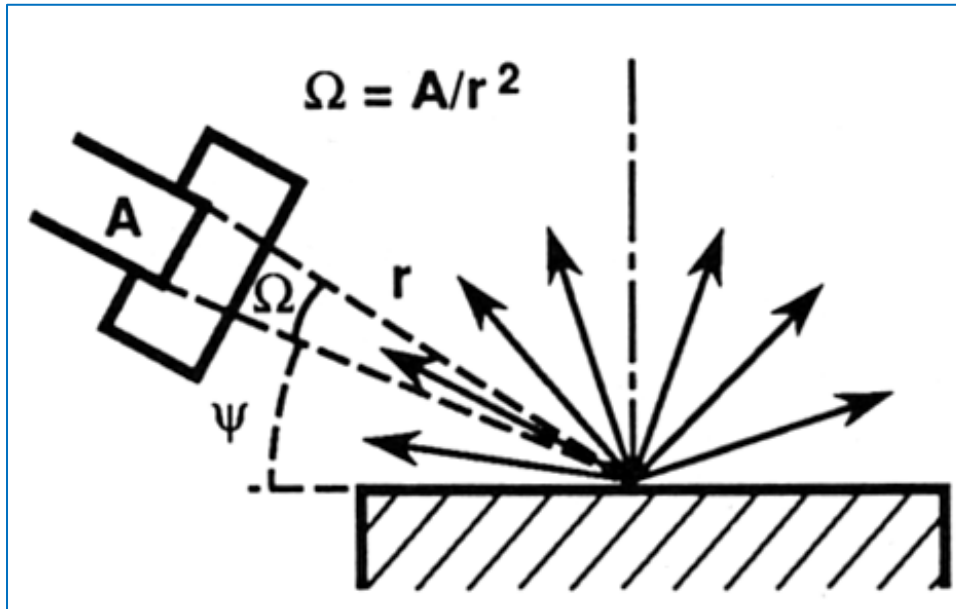


Figure 4.18. Schematic diagram of the Everhart–Thornley detector: B, backscattered-electron trajectories; SE, secondary-electron trajectories; F, Faraday cage (bias range -50 V to $+250\text{ V}$); S, scintillator, with thin metallic coating; high bias ($+12\text{ kV}$) supply to the scintillator coating; LG, light guide; PM, photomultiplier.

Image formation

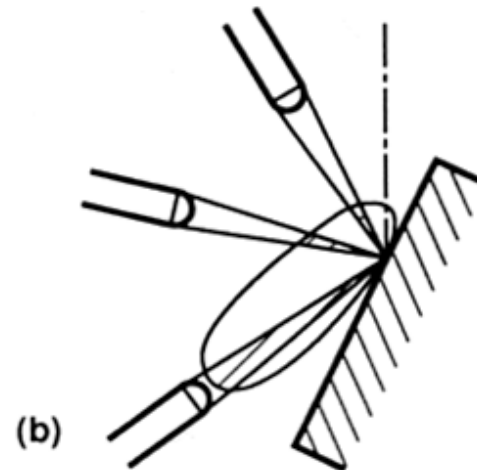
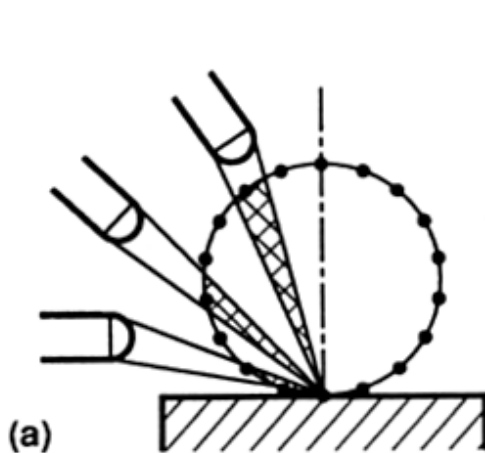


Collection efficiency

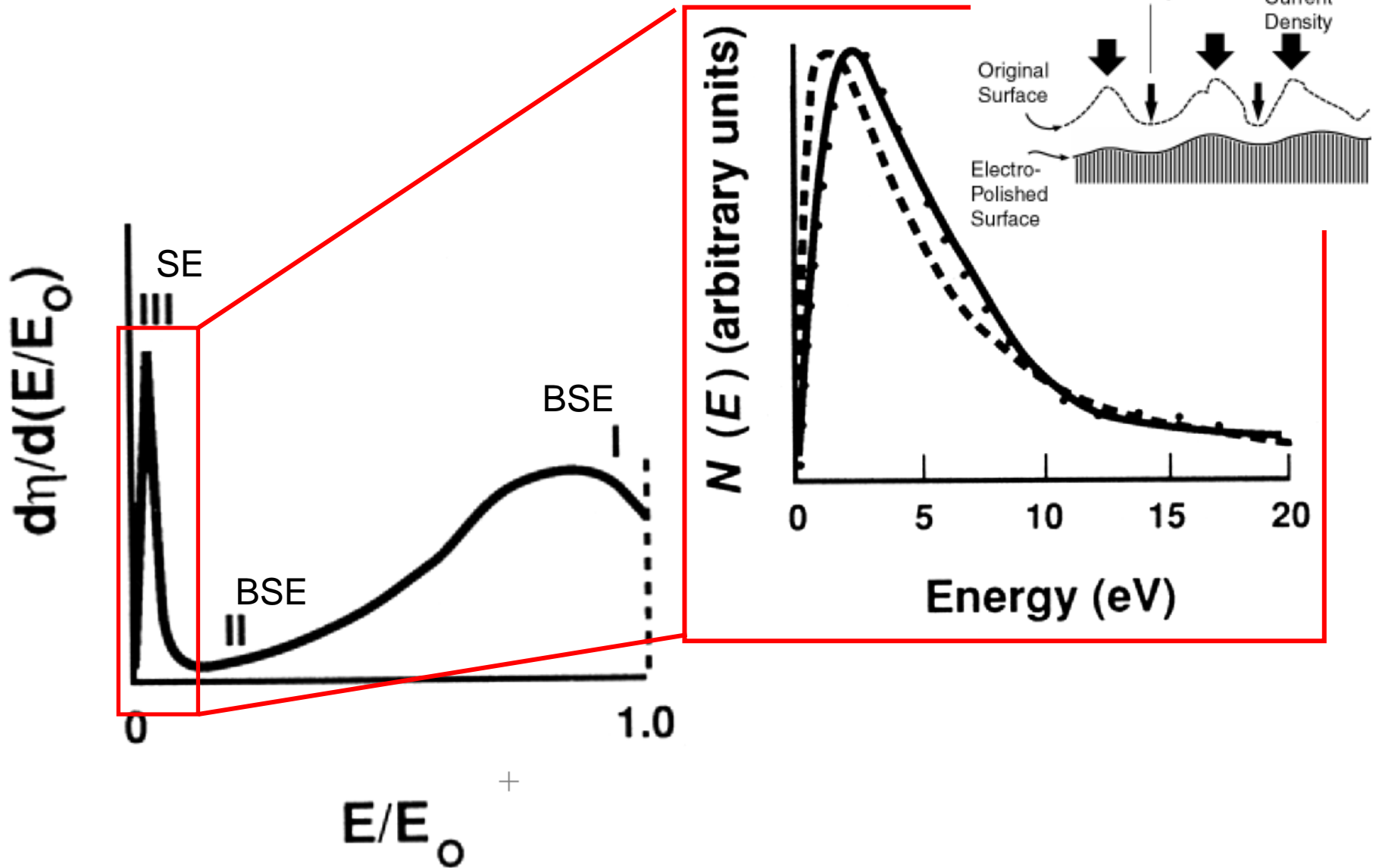


일반적인 A ~ 10-30 mm²

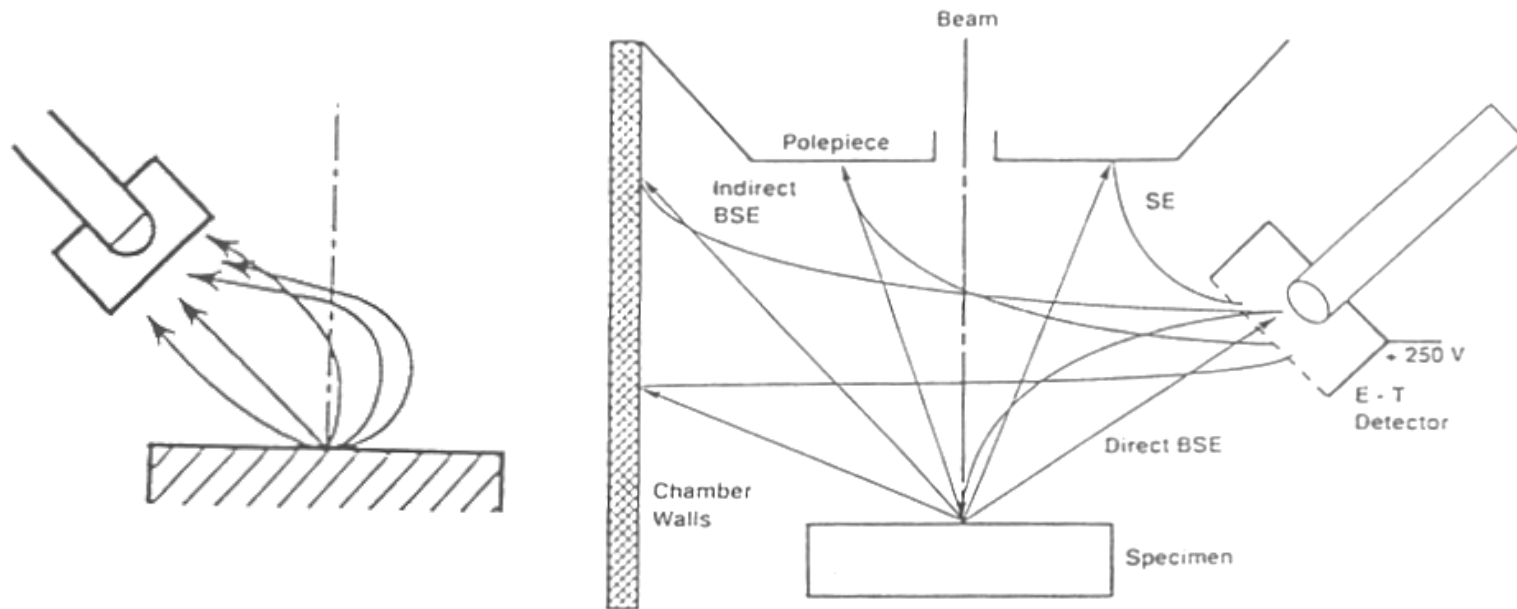
Detector in SEM
- SE detector
- BSE detector



Energy distribution



Positive Bias

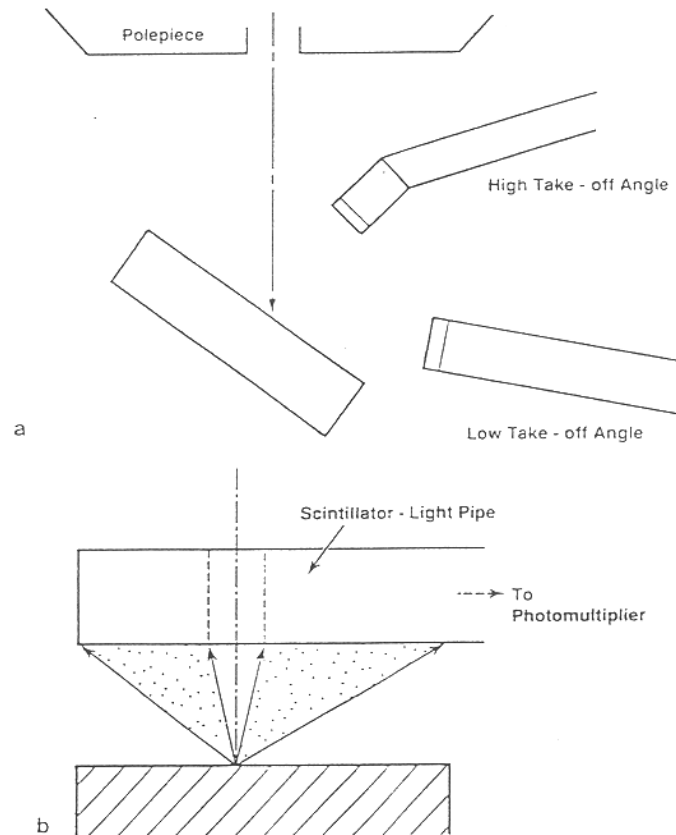


Signal	Source	Percentage of total
SE _I	Beam-produced secondaries	9%
SE _{II}	Backscatter-produced secondaries	28%
SE _{III}	Remote backscatter-produced secondaries	61%
SE _{IV}	Beam-produced secondaries from aperture	2%

Dedicated BSE detector

Scintillator Backscatter detector

SE와 BSE와의 energy 차이에 의해 BSE만 효과적으로 검출할 수 있도록 고안

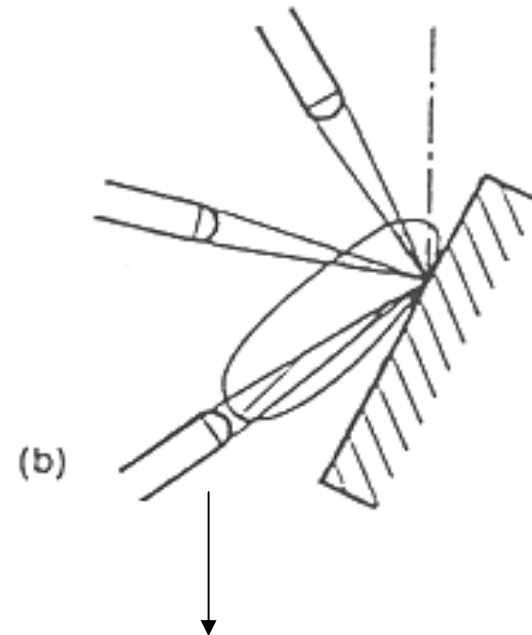
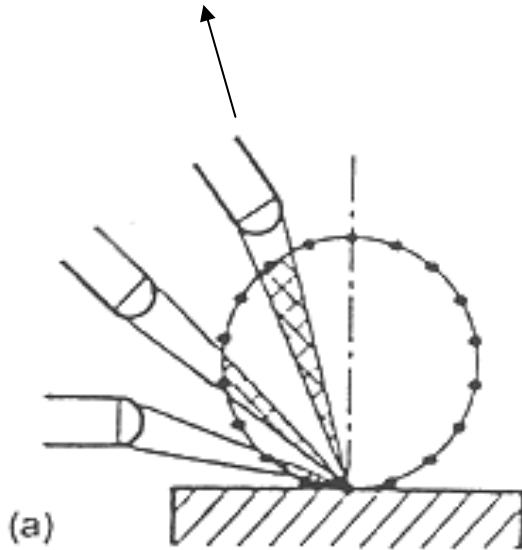


SE는 에너지가 낮아 Scintillator에 부딪쳐도 photon를 생성시키지 못한다.

Negative Bias

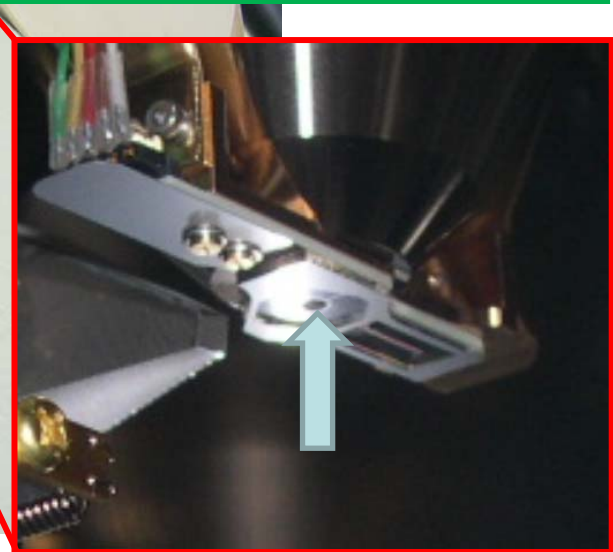
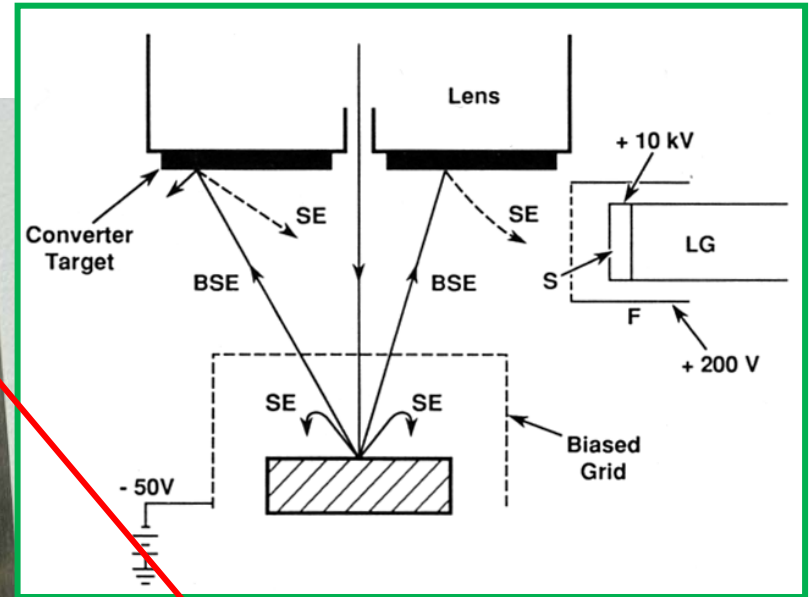
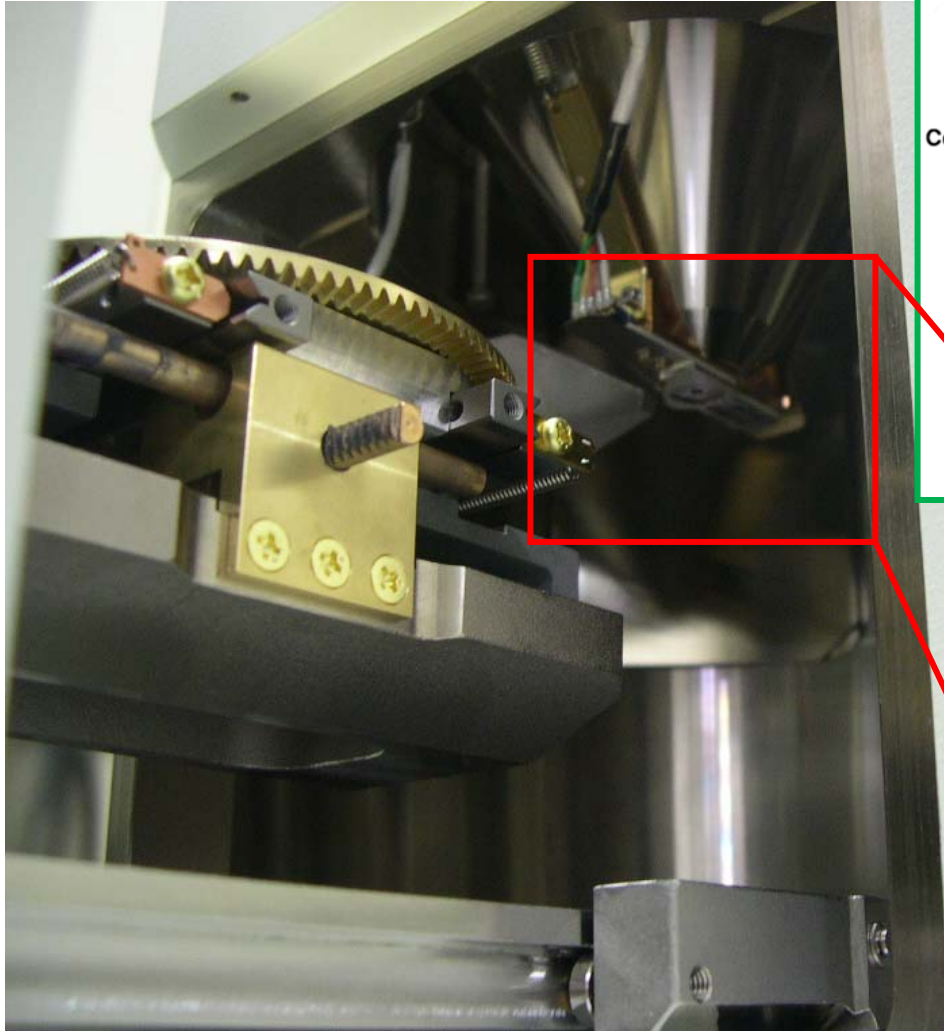
Only BSE are detected(SE rejected)

BSE가 가장 많이 collected



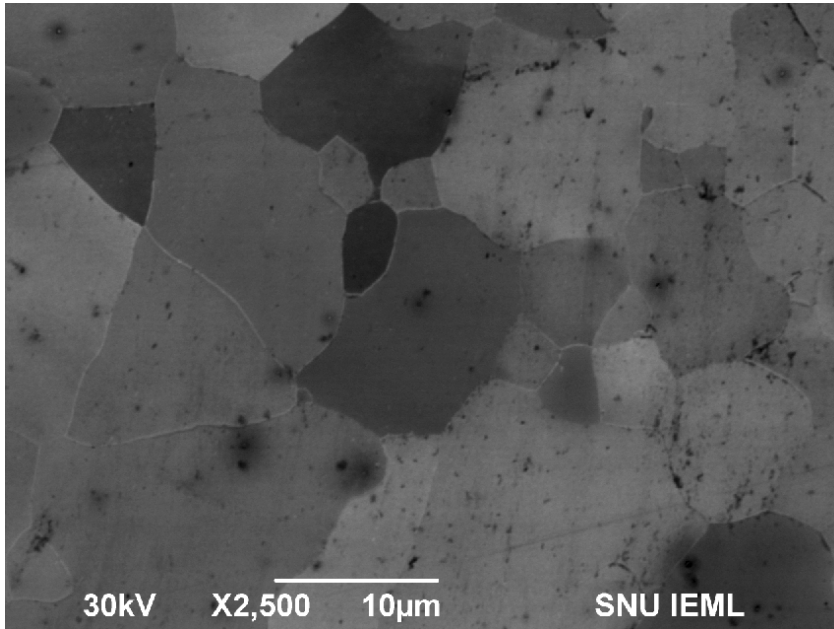
BSE가 가장 많이 collected

SEM detectors (BSE)

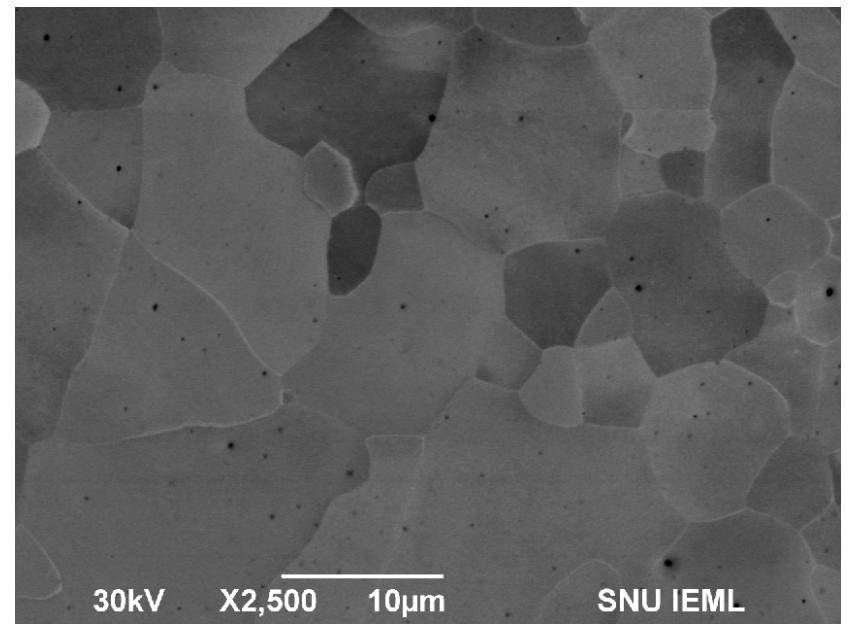


In-situ Lab JSM 6390 performance test

SE

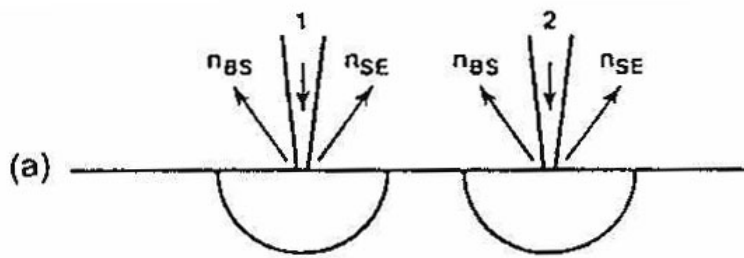


BSE



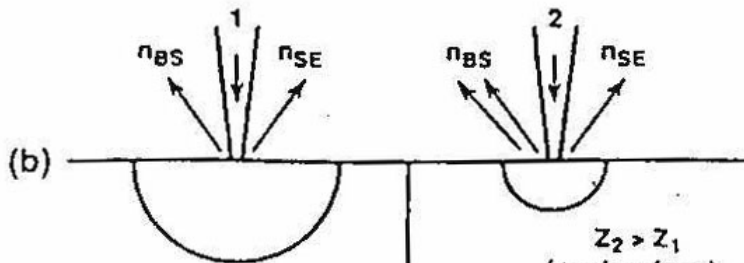
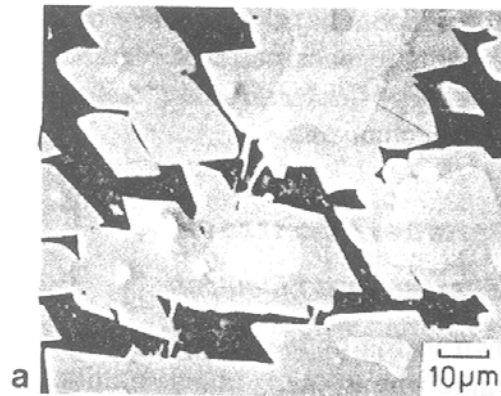
Compositional contrast

secondary electron coefficient는 원자번호에 따른 차이가 거의 없다.
backscattered electron coefficient는 원자번호가 클수록 증가
 BSE mode에서 시편을 구성하는 조성에 따른 contrast를 얻을 수 있으며
 원자번호가 큰 것이 더 밝게 보인다.



$$(n_{BS})_1 = (n_{BS})_2$$

$$(n_{SE})_1 = (n_{SE})_2$$



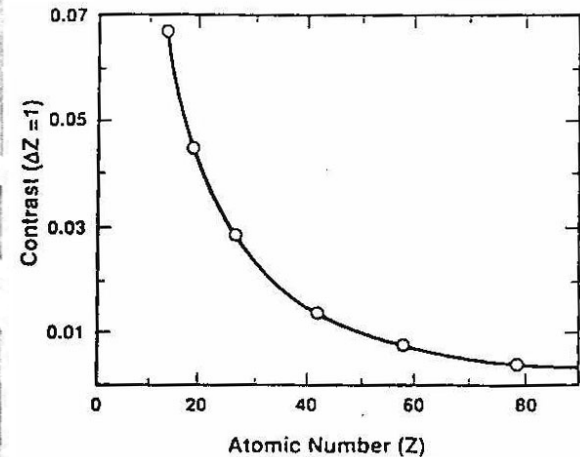
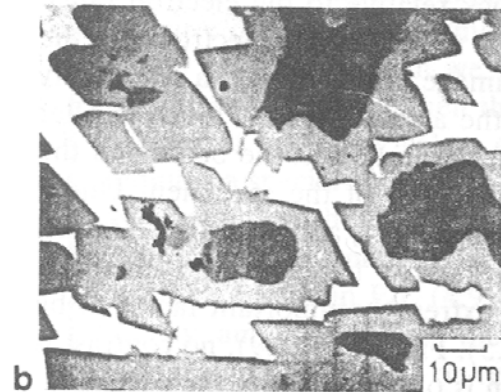
$$Z_2 > Z_1$$

$$(n_{BS})_2 > (n_{BS})_1$$

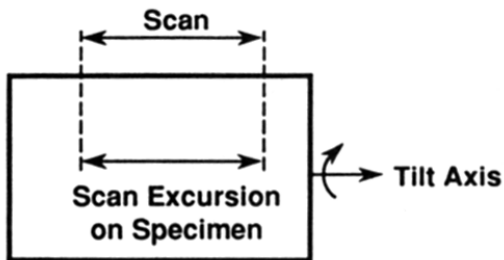
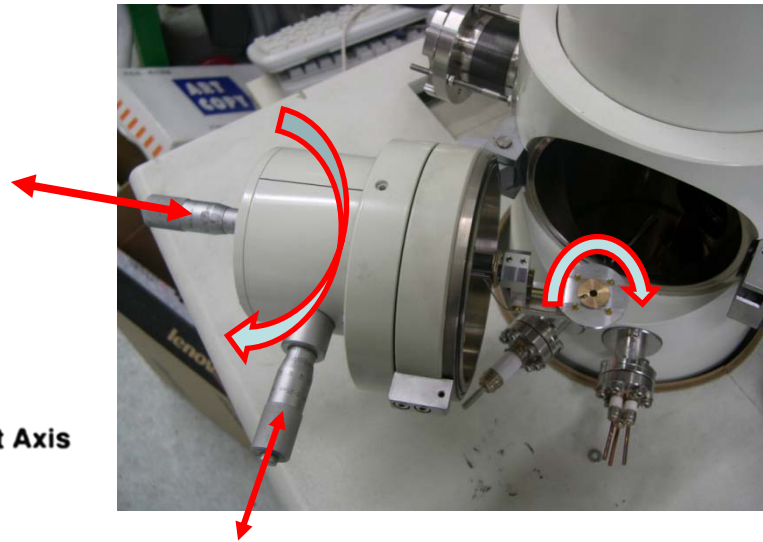
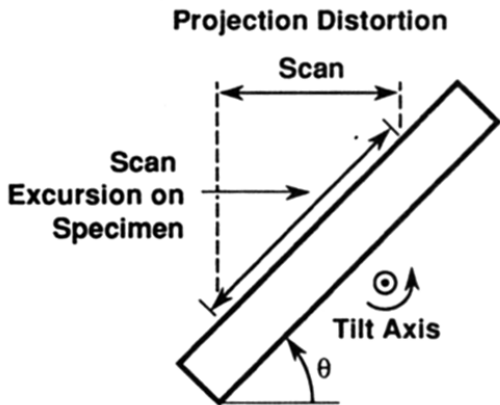
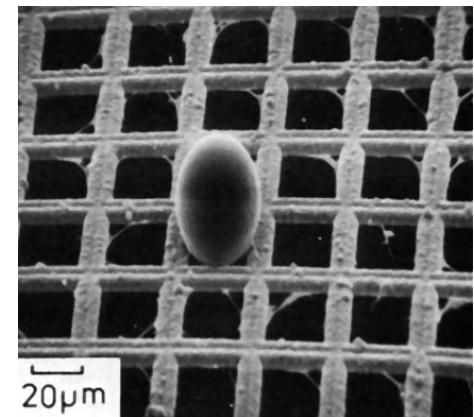
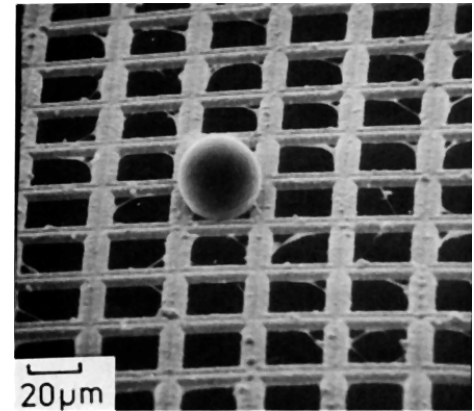
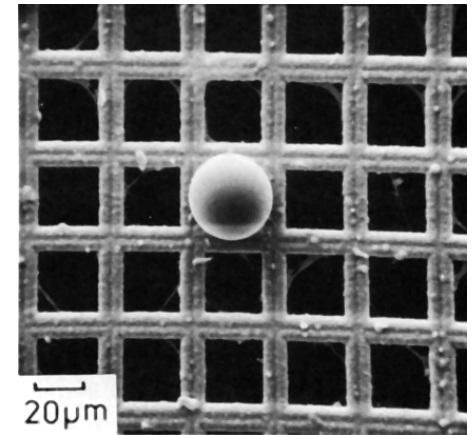
$$(n_{SE})_2 \approx (n_{SE})_1$$

light

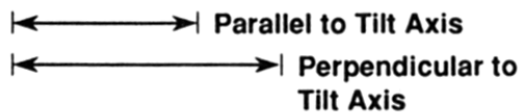
heavy



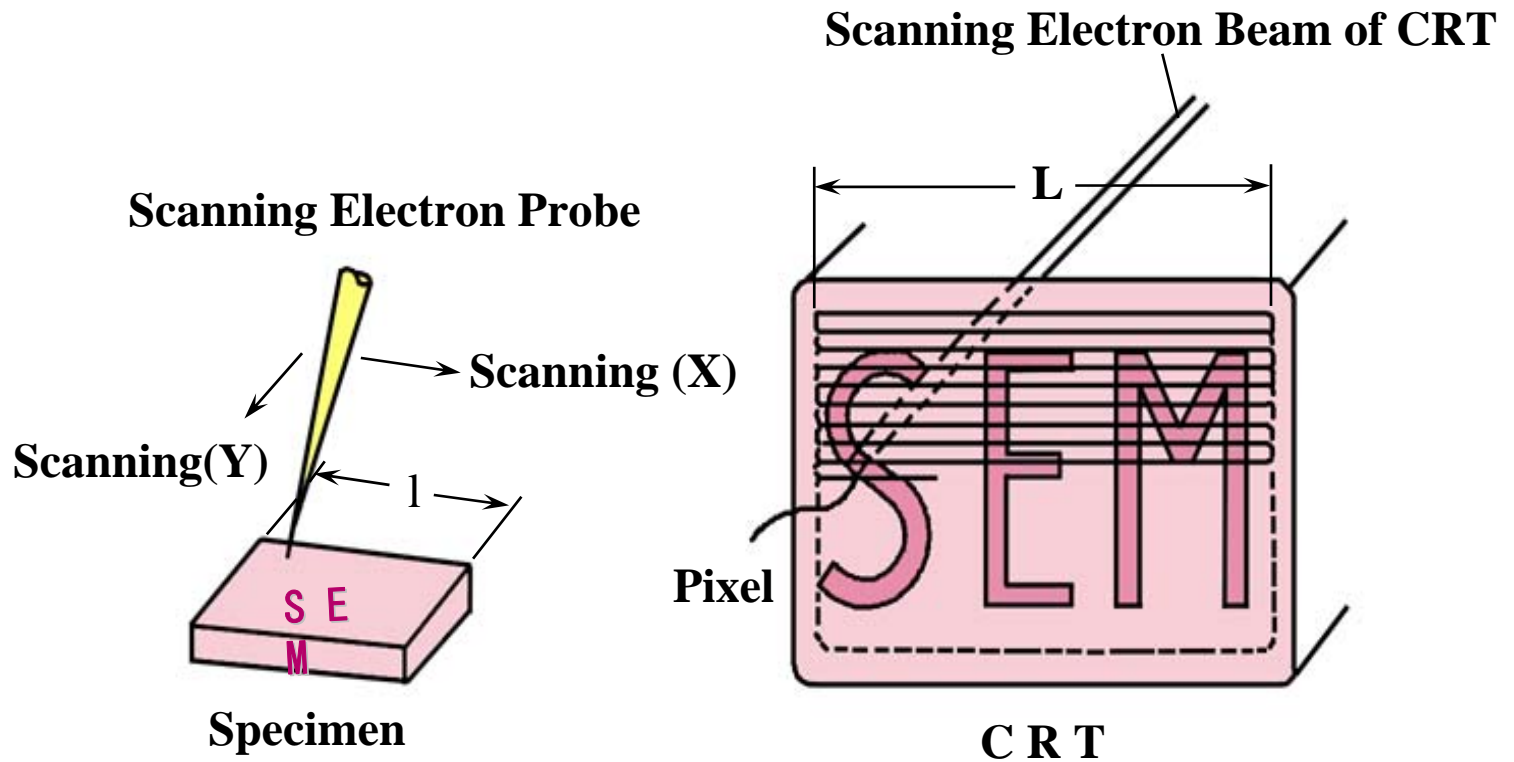
Effect of tilting



Comparison of Scan Excursions for $\theta = 45^\circ$



Theory of Scanning Electron Microscope



$$\text{Magnification : } (M) = L / l$$

Magnifying mechanism in the SEM

Magnification

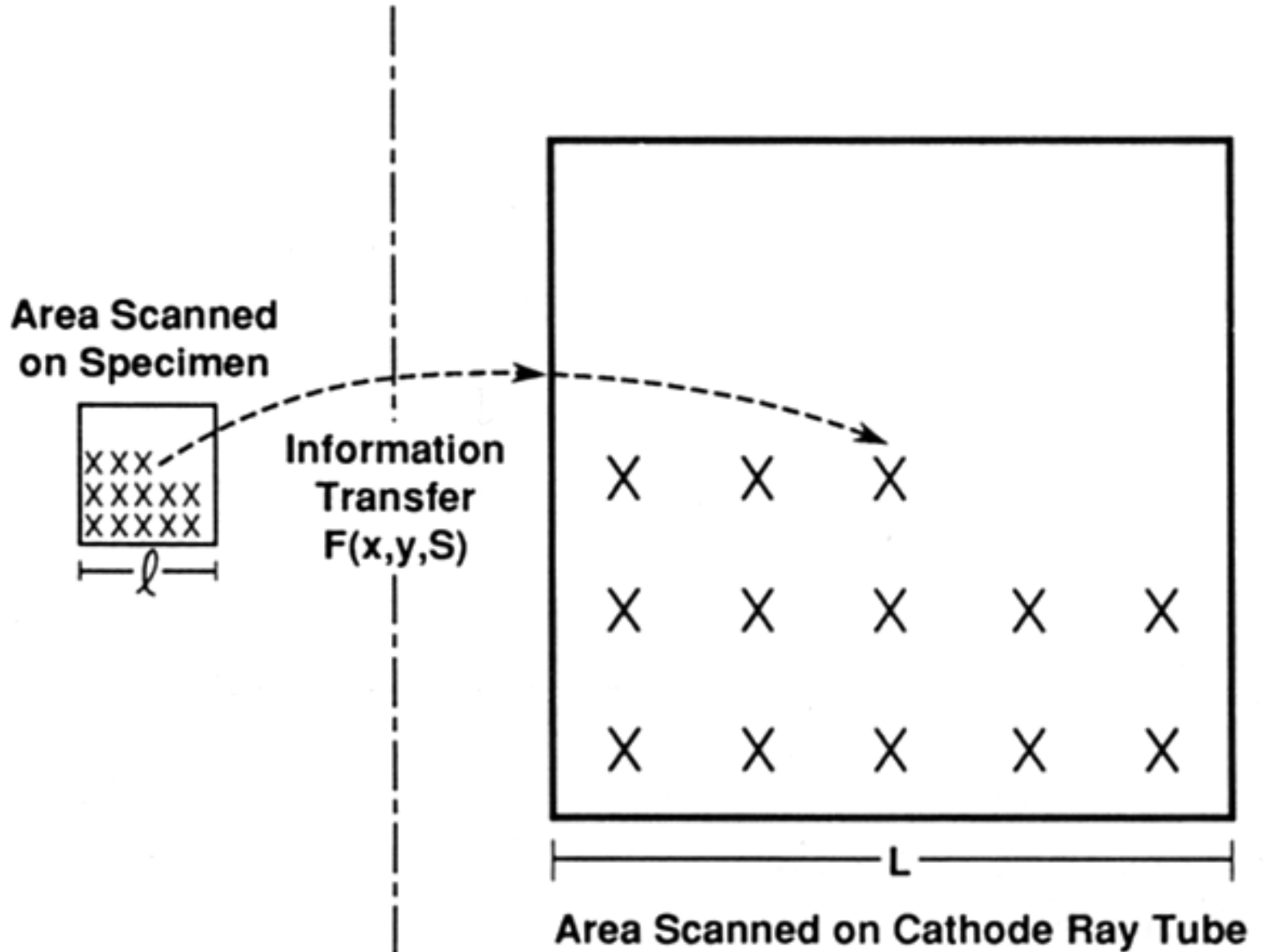
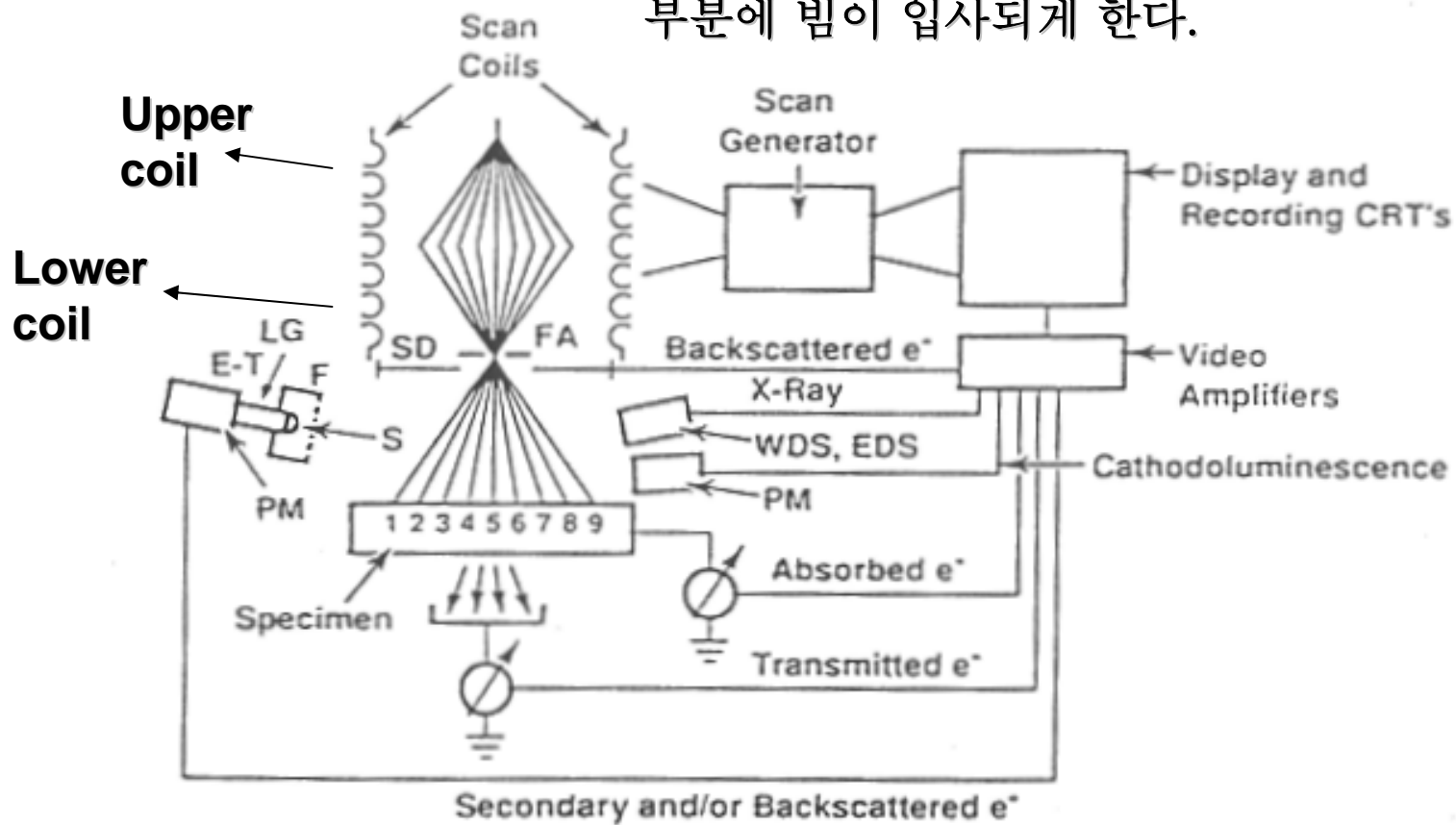


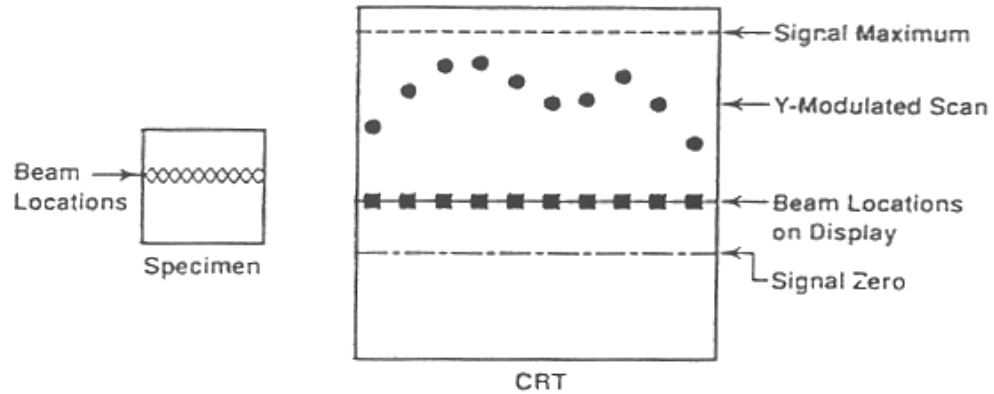
Image formation in the SEM

Scanning action

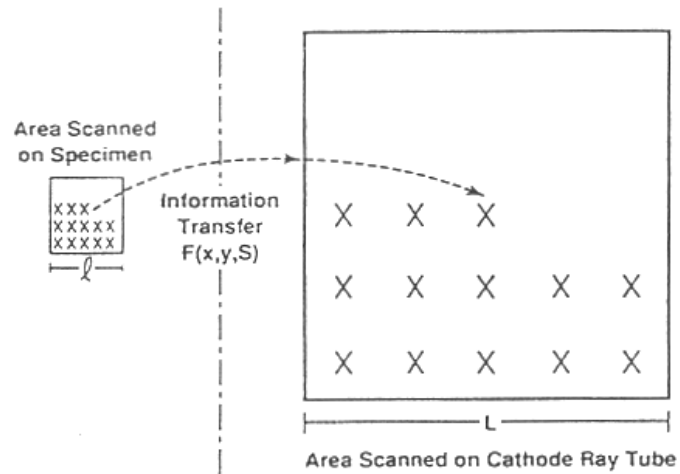
전류의 변화로 인해 시편의 특정 부분에 빔이 입사되게 한다.



Line scan



Area scan



시편의 특정 위치에서 나온 정보를 **CRT**의 특정 위치에 옮기는 **mapping operation**에 의해서 형성된 이미지

Area sampled as a function of magnification

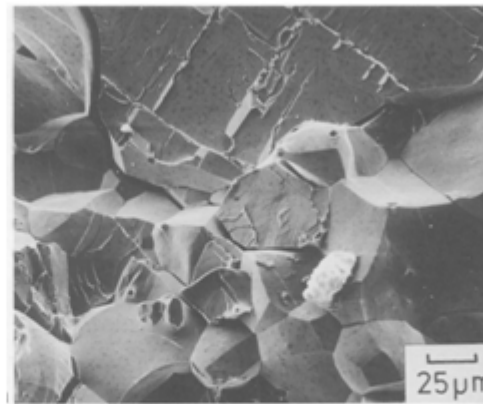
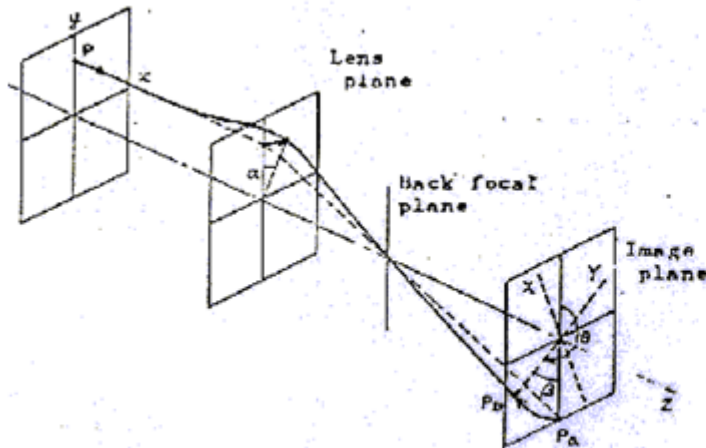
Magnification

$$M = \frac{L_{CRT}}{L_{spc}}$$

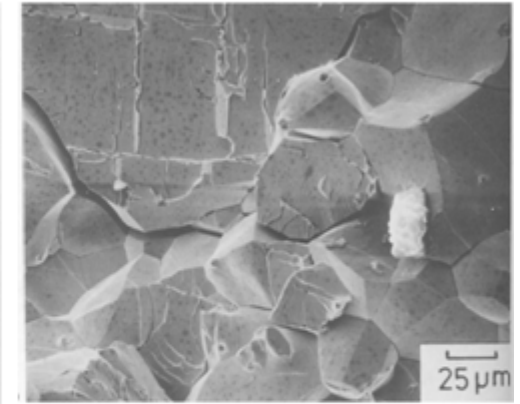
Magnification	Area on sample
10X	(1 cm) ²
100X	(1 mm) ²
1,000X	(100 μm) ²
10,000X	(10 μm) ²
100,000X	(1 μm) ²
1,000,000X	(100 nm) ²

^a Assumes CRT screen measures 10 cm × 10 cm.

Working distance 변환에 따른 시편의 회전



15mm



45mm

Scan coil에 의한 전자빔 입사의 회전

Picture element(pixel) size

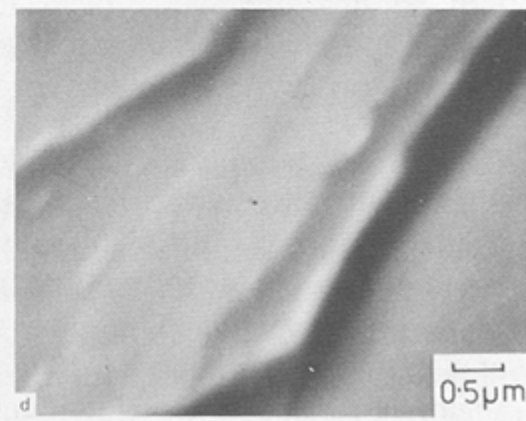
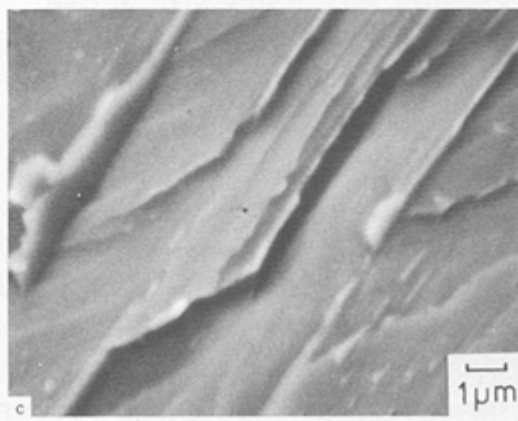
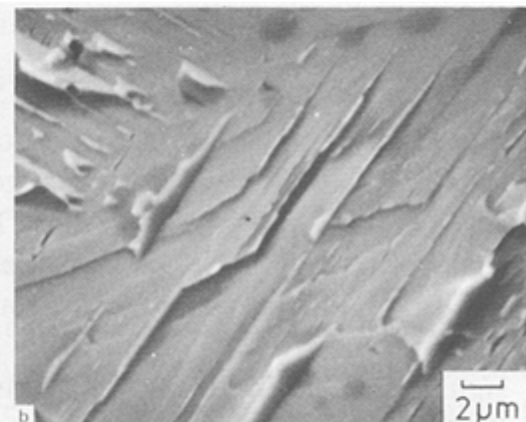
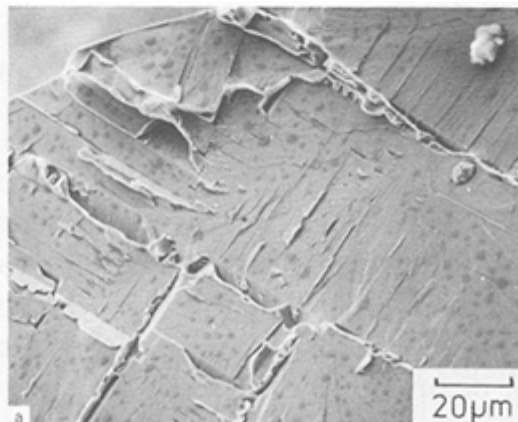
: 시편의 정보가 화면으로 옮겨지는 시편에서의 면적의 크기

$$D_{PE} = \frac{L_{spc}}{N_{pe}}$$

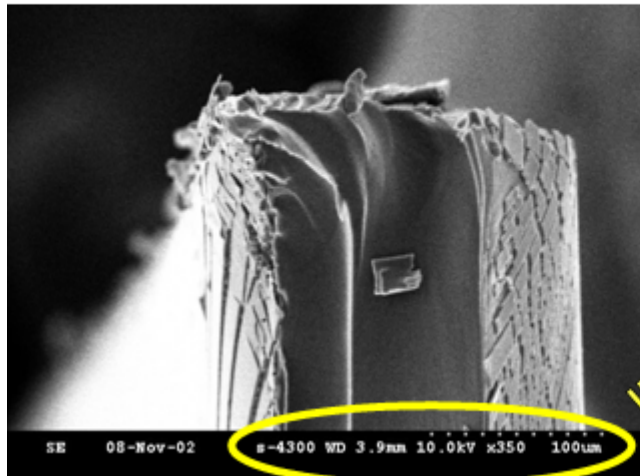
N_{pe} : Number of discrete location along the scan line

Magnification	Edge of picture element
10X	10 μm
100X	1 μm
1,000X	0.1 μm (100 nm)
10,000X	0.01 μm (10 nm)
100,000X	1 nm

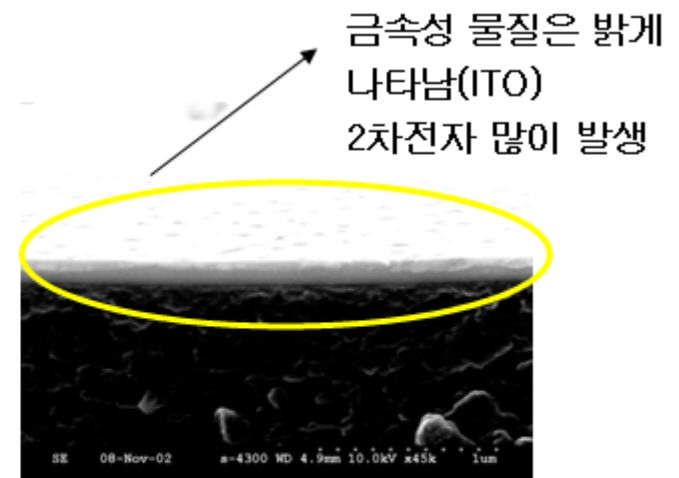
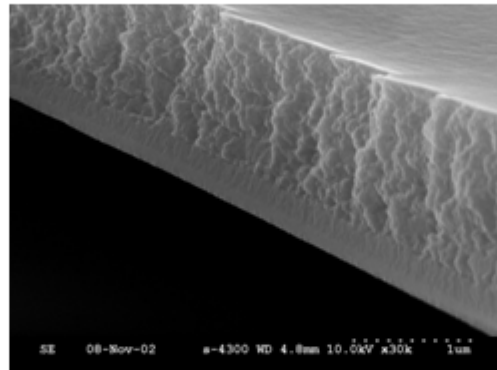
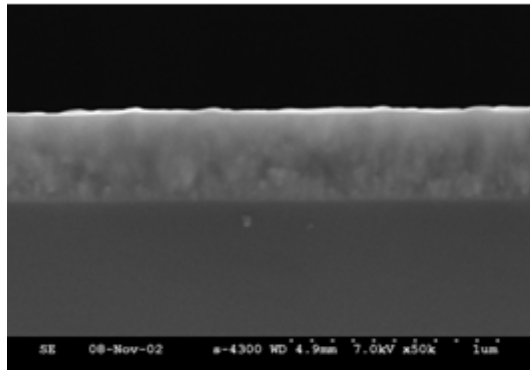
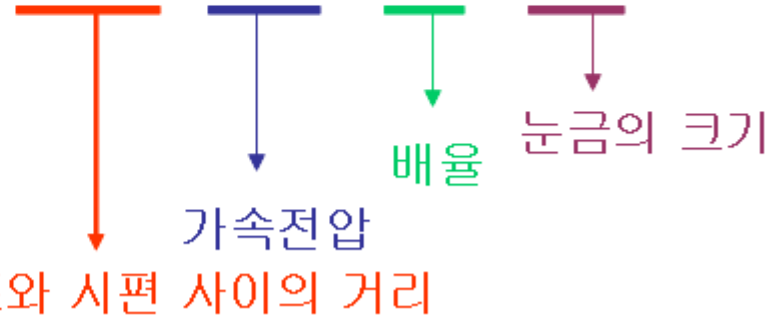
^a 1000 \times 1000 scan matrix; 10 cm \times 10 cm display on CRT.



Example



s-4300 WD 3.9mm 10.0kV x350 100um



같은 가속전압과 같은 배율로 시편을 관찰하였으나 다른 양상을 보여주고 있다. 즉 조건이 같더라도 시편에 종류에 따라 영상의 질은 달라진다.!!!