2009 spring

Microstructural Characterization of Materials

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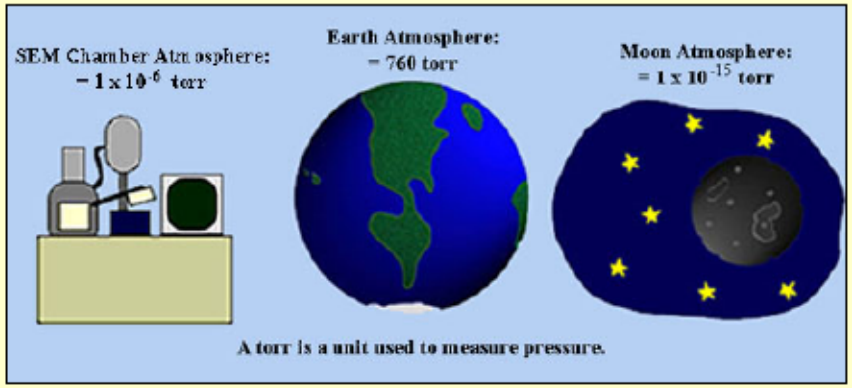
Vacuum



When a SEM is used, the column must always be at a vacuum. There are many reasons for this. If the sample is in a gas filled environment, an electron beam cannot be generated or maintained because of a high instability in the beam. Gases could react with the electron source, causing it to burn out, or cause electrons in the beam to ionize, which produces random discharges and leads to instability in the beam. The transmission of the beam through the electron optic column would also be hindered by the presence of other molecules. Those other molecules, which could come from the sample or the microscope itself, could form compounds and condense on the sample. This would lower the contrast and obscure detail in the image.

A vacuum environment is also necessary in part of the sample preparation. One such example is the sputter coater. If the chamber isn't at vacuum before the sample is coated, gas molecules would get in the way of the argon and gold. This could lead to uneven coating, or no coating at all.

The Sputter Coater

C, Cr, Au-Pd

The sputter coater is used to coat non-metallic samples (bugs, plants, human hair, etc.) with a thin layer of gold. This makes them conductive, and ready to be viewed by the SEM. If the samples are metallic, they can simply be mounted and placed in the SEM

A purpose of coating

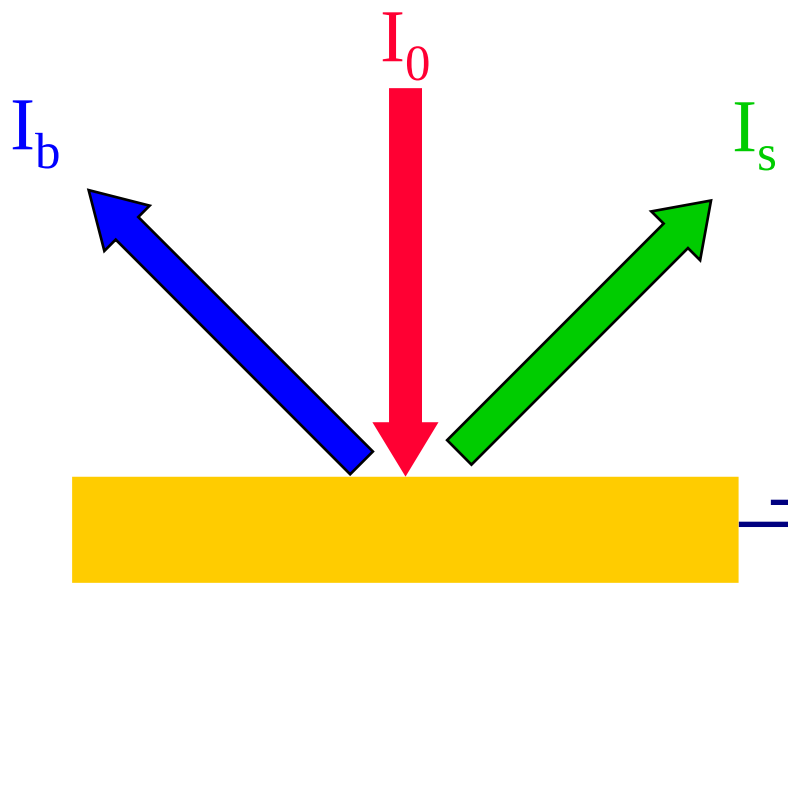
1. Give a conduction to specimen surface → Prevention of charge-up

2. Increase the efficiency of secondary electron → Improvement of Signal to Noise (S/N) ratio

3. Protect the specimen surface against primary electron beam → Reduction of damage

Theory of Scanning Electron Microscope

$I_0 \neq I_s + I_b + I_a$ ⇨ **Charge-up**



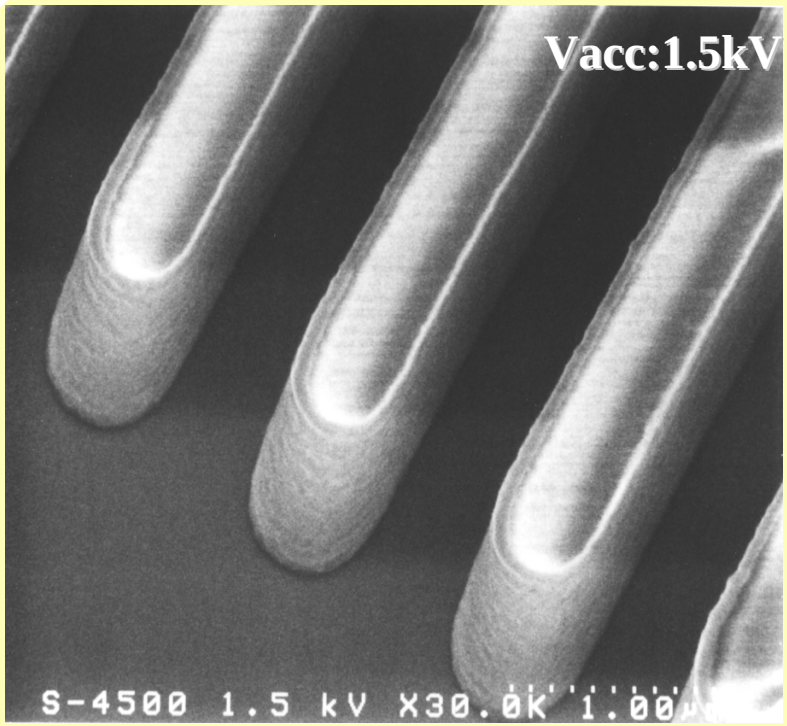
I_0:Primary Beam

I_s:Secondary Electron

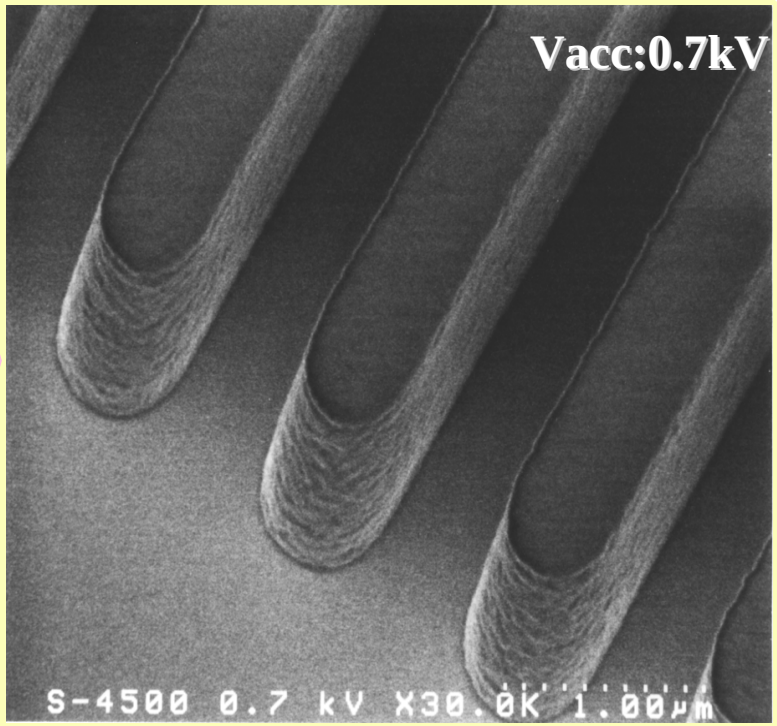
I_b:Back scattered Electron

I_a:Specimen Current

Observation at lower accelerating voltages

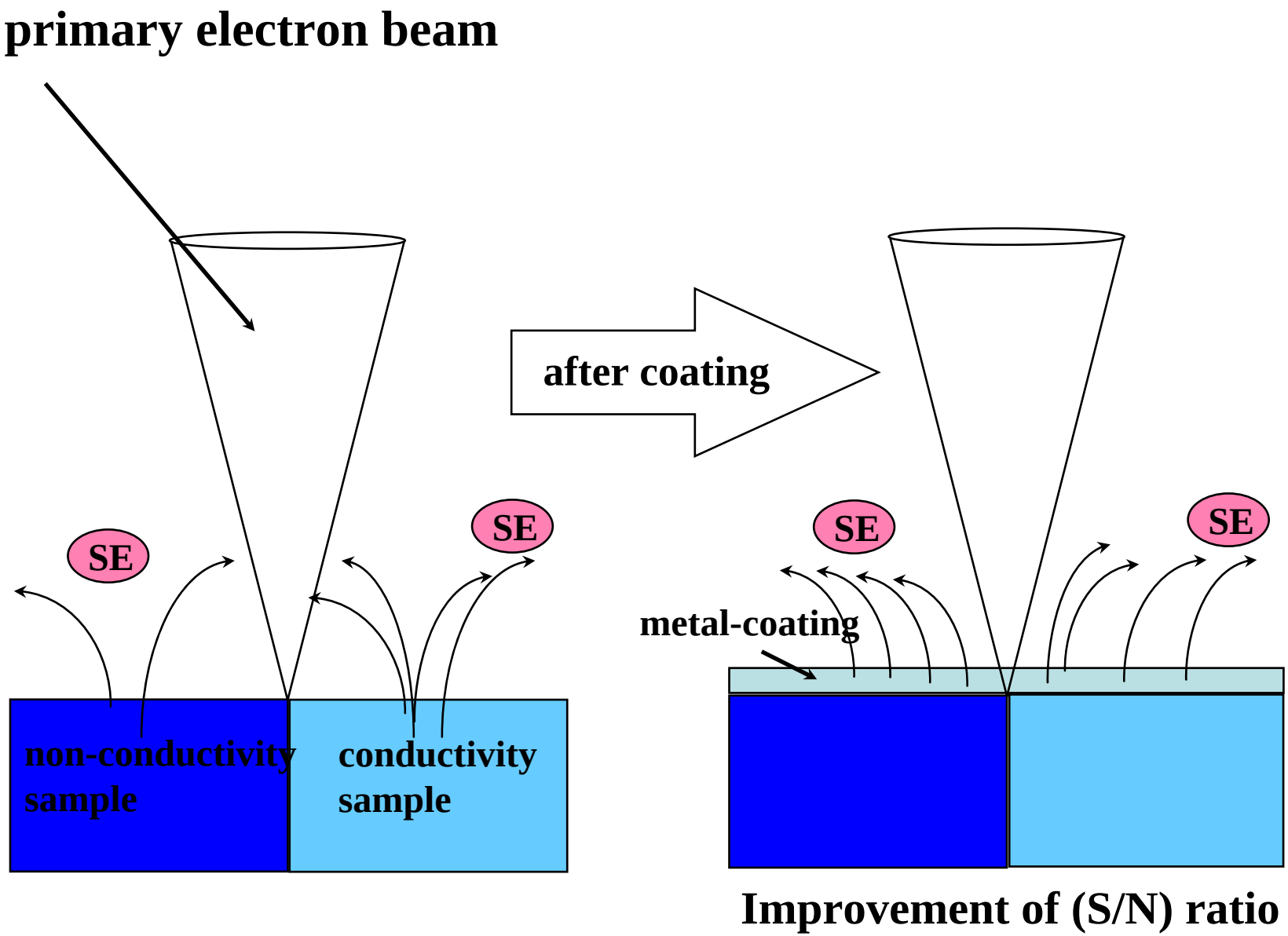


Charge-up Phenomena



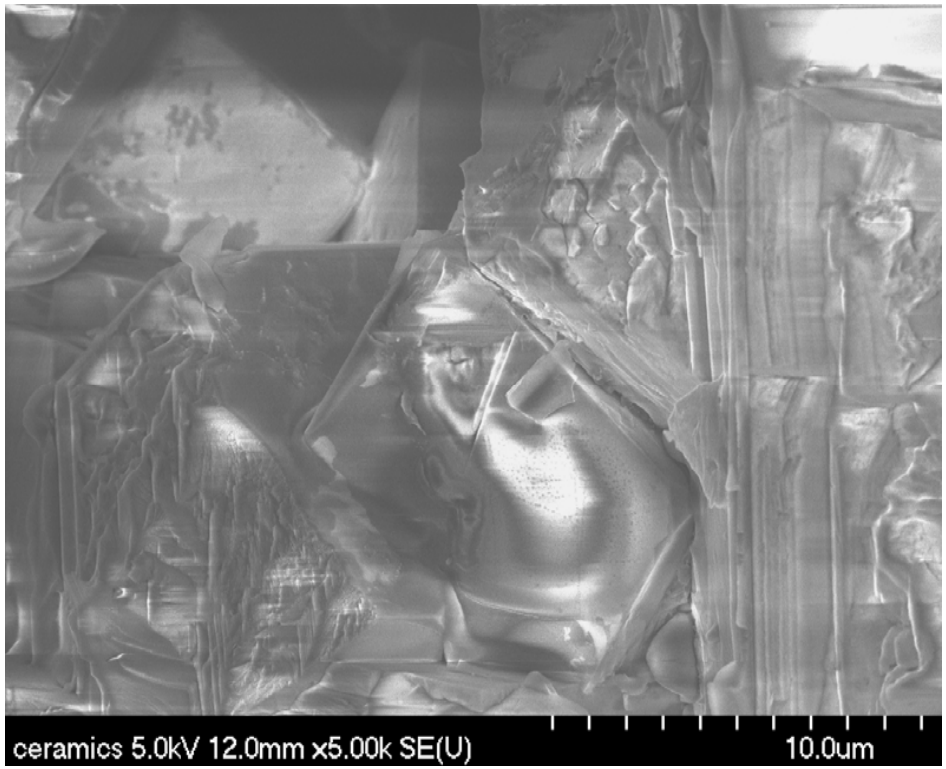
Eliminate Charge-up Phenomena

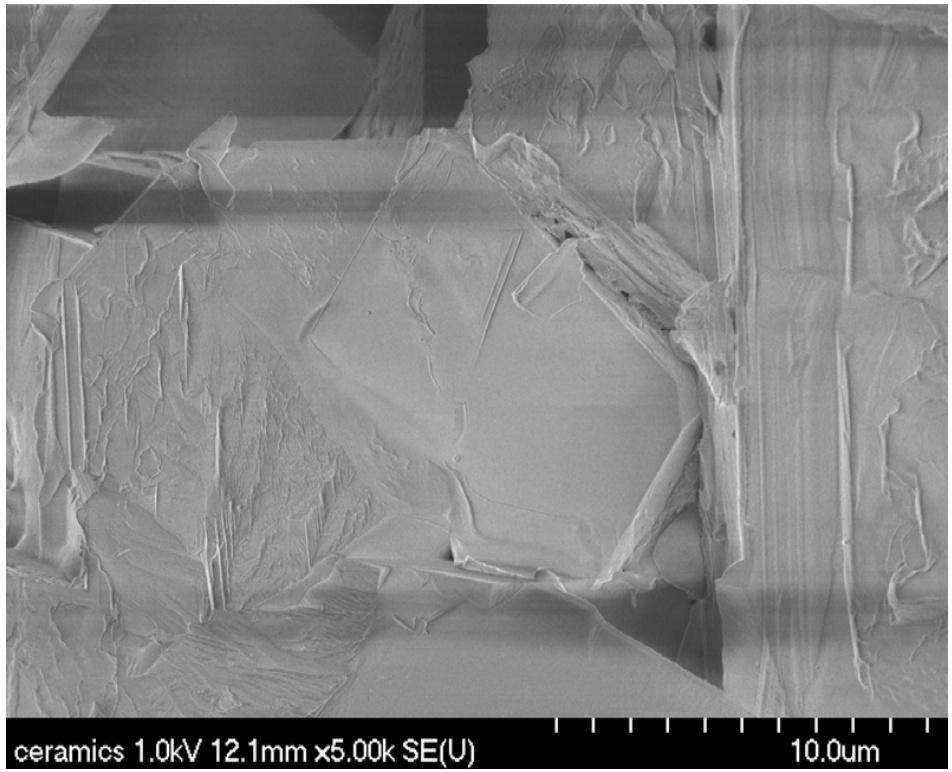
Specimen : SiO_2 on Photo Resist Line Pattern



Improvement of (S/N) ratio

Non-Conductive Samples Observation



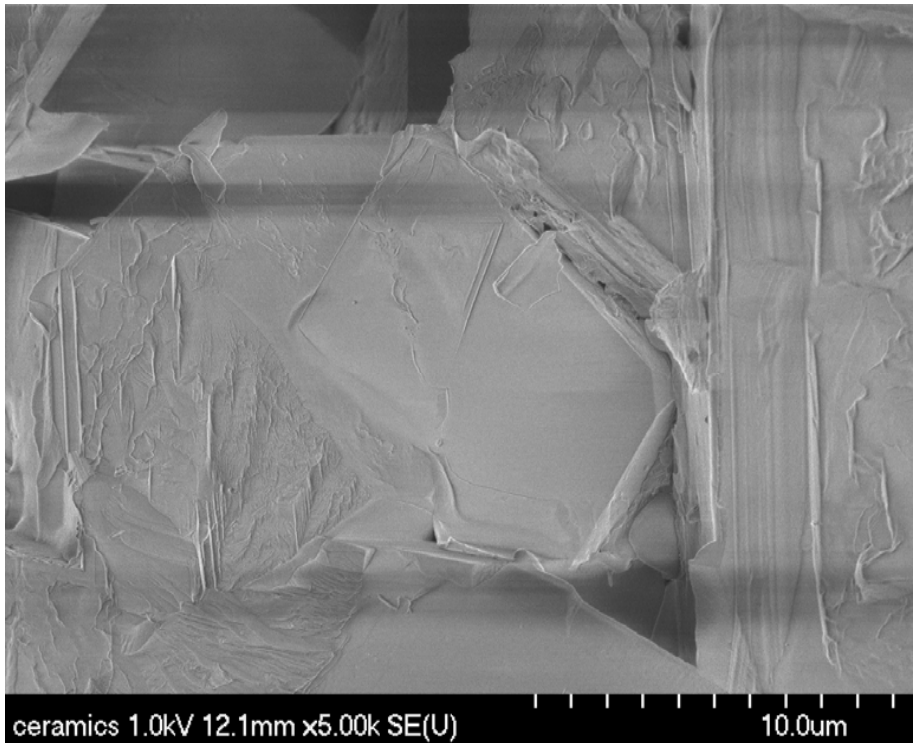


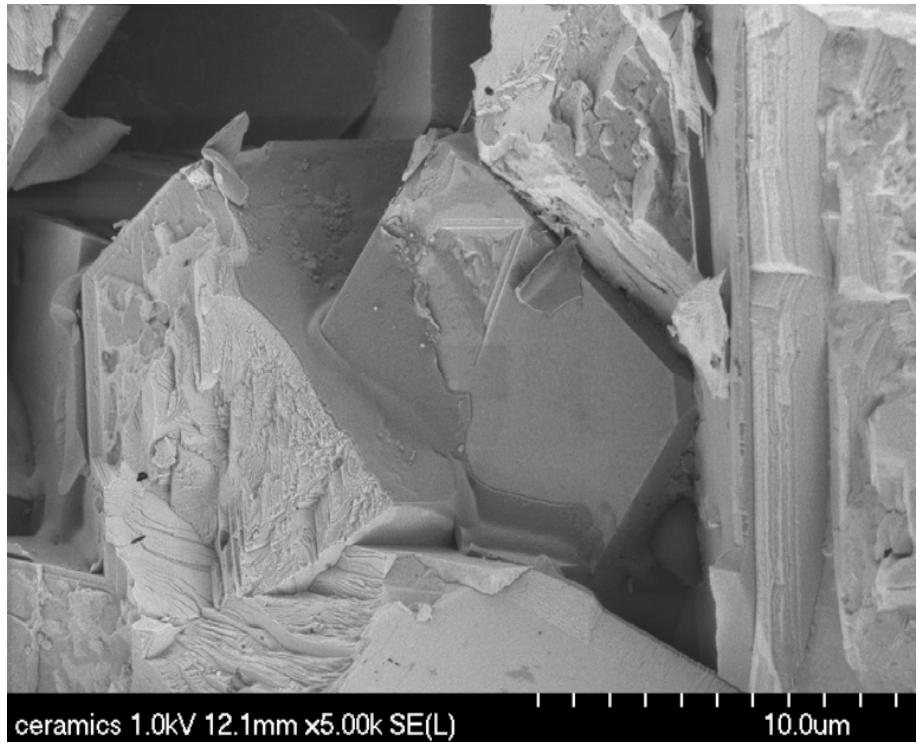
Vacc : 5kV
Upper Detector

Vacc : 1kV
Upper detector

Specimen : Ceramics

Non-Conductive Samples Observation





Vacc : 1kV
Upper Detector

Vacc : 1kV
Lower Detector

Specimen : Ceramics

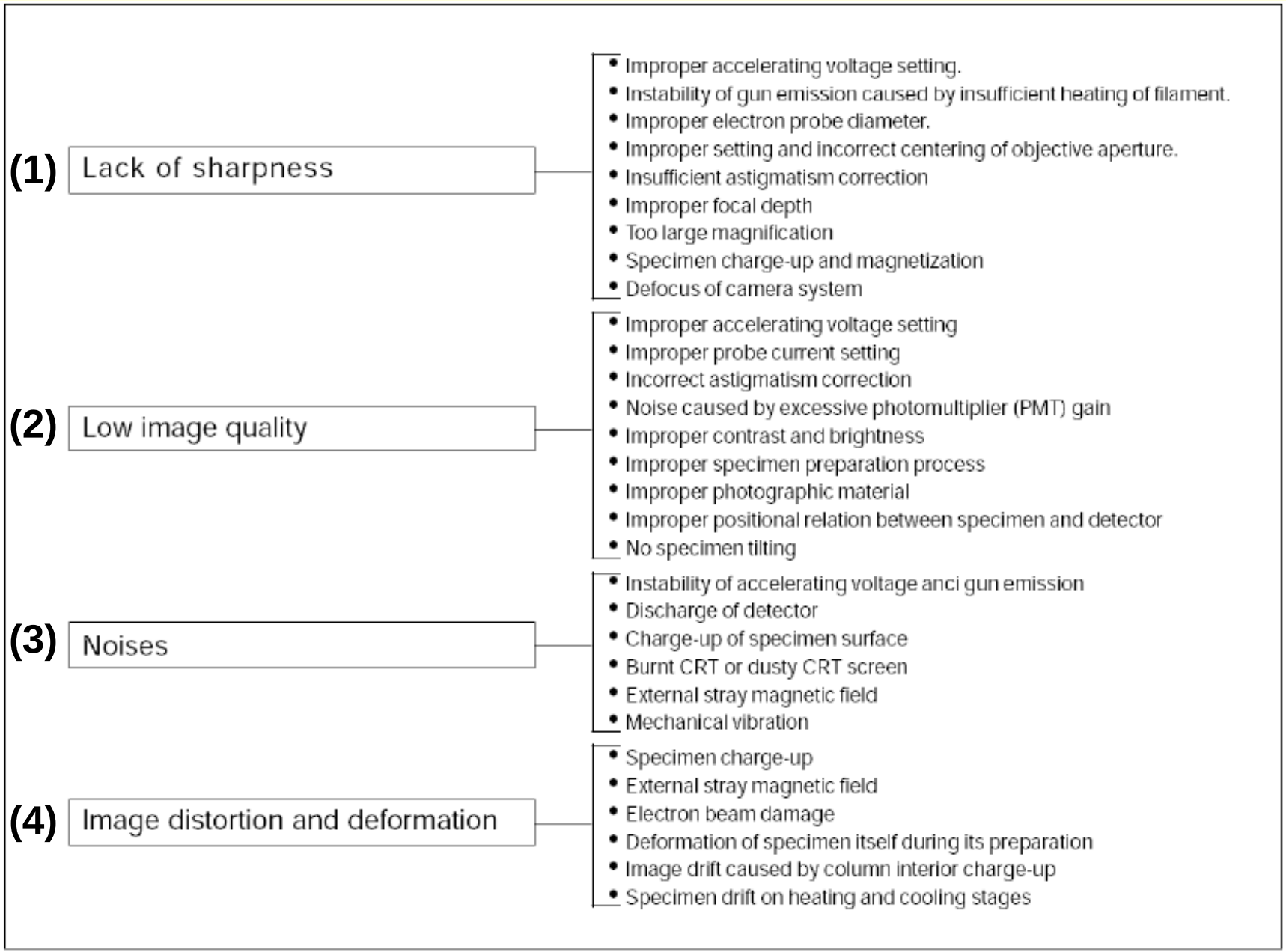
Disturbances

Types of Imaging Disturbances

Image disturbances can be classified by the following expressions:

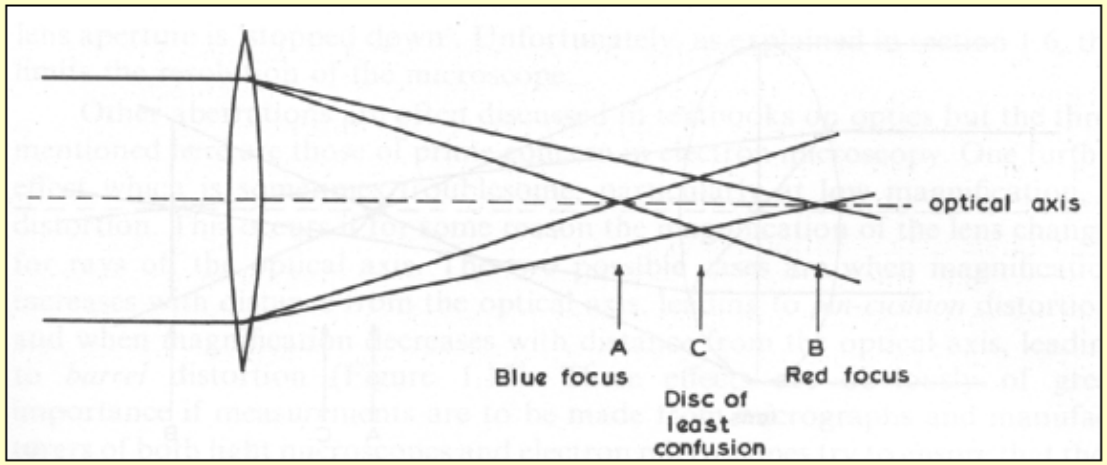
- **Chromatic aberrations**
- **Images lacking sharpness and contrast**
- **Unstable images**
- **Generally poor-quality images**
- **Noisy images**
- **Images showing jagged edges**
- **Unusual-contrast images**
- **Distorted or deformed images.**

Image Disturbances and Their Causes

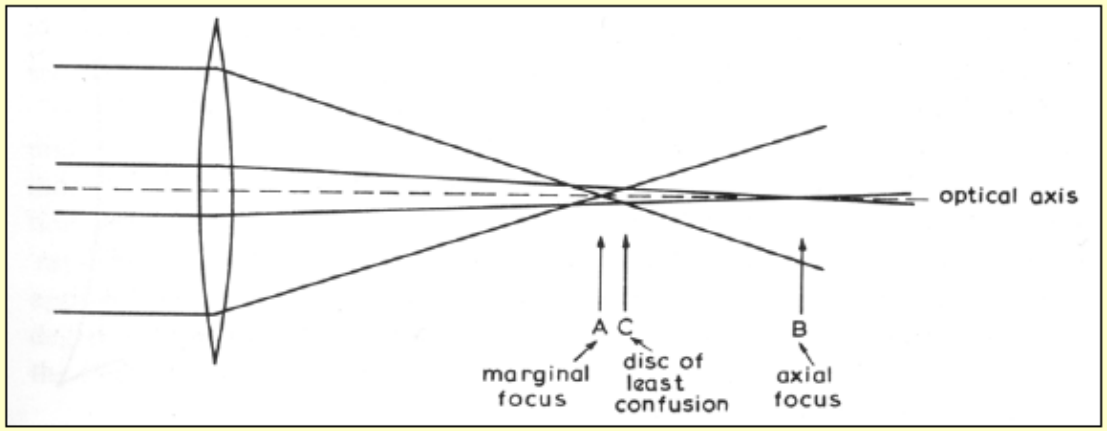


Aberrations

Chromatic



Spherical



$r_1 = 0.61\lambda/\alpha \quad r_2 = C_s\alpha^3 \Rightarrow r = r1 + r2$

optimized $\Rightarrow \alpha_{opt} = 0.67\lambda^{1/4}C_s^{-1/4} \quad r_{opt} = 1.21\lambda^{3/4}C_s^{1/4}$

Image Changes Caused by Interactions Between Electron Probe and Specimen

Influence of accelerating voltage on image quality:

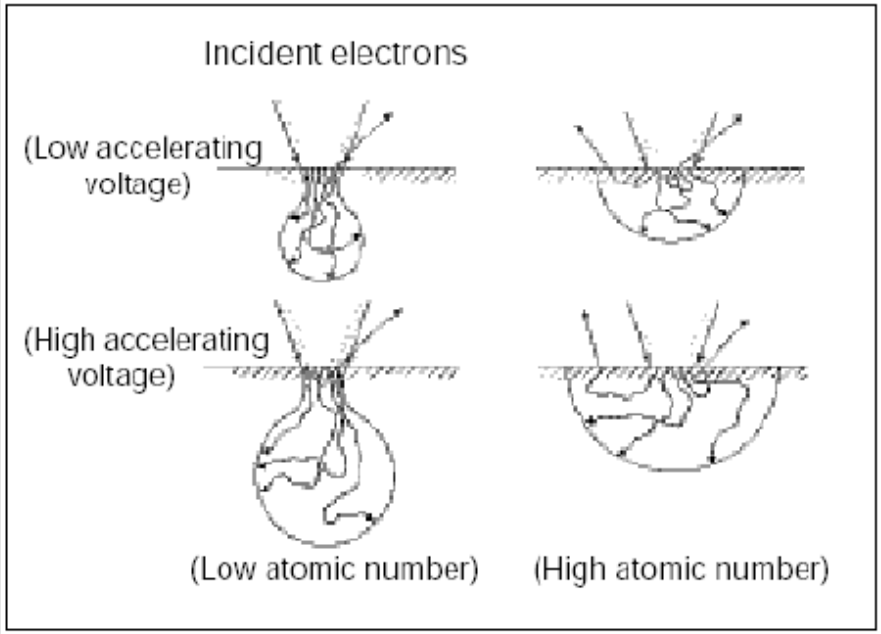


Fig.1 Diffusion of incident electrons (after Ducumb and Shields).

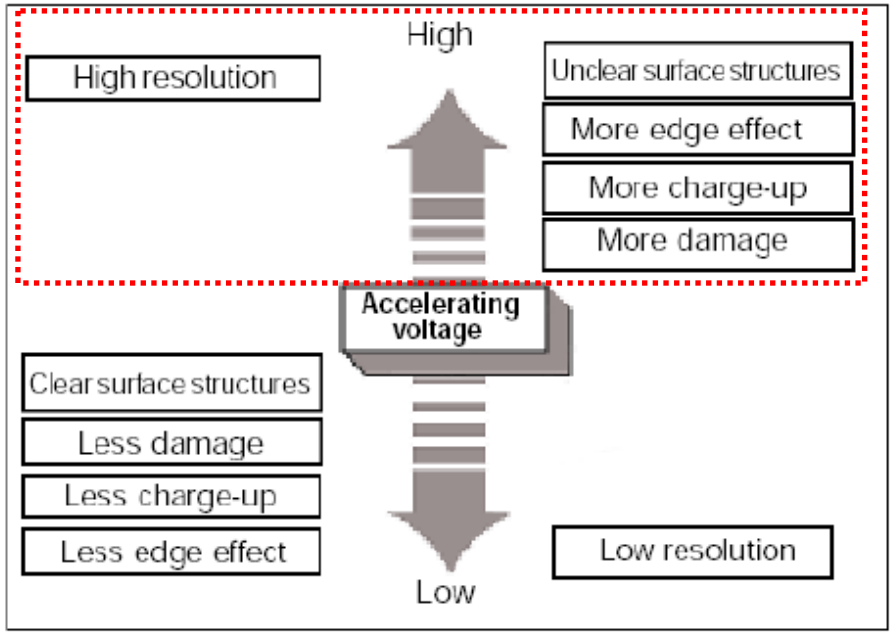
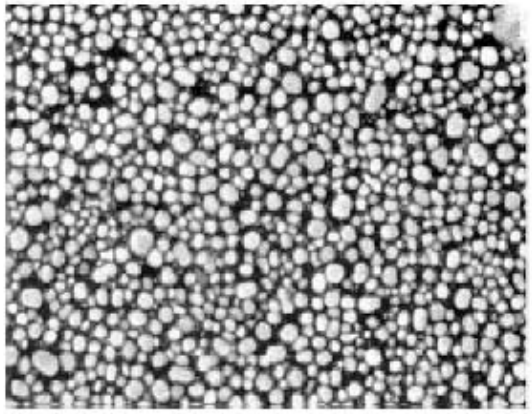
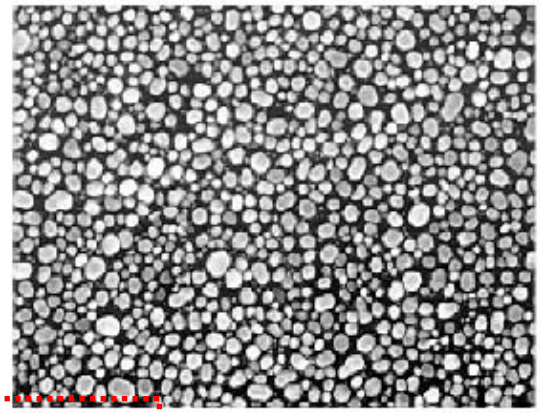


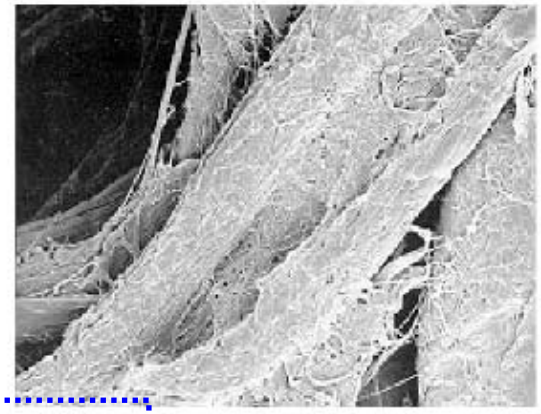
Fig. 2 Effect of accelerating voltage.

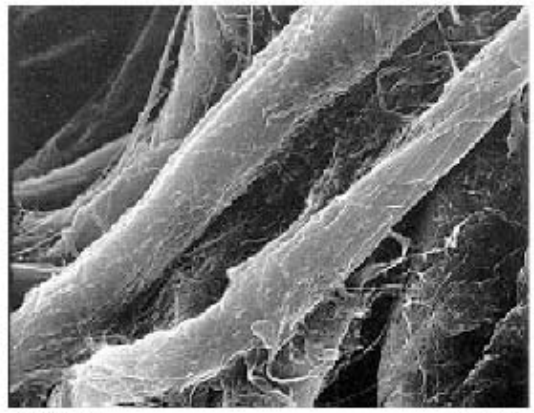
Gold particles





Toilet paper





- **LEFT: The image sharpness and resolution are better at the higher accelerating voltage, 25 kV.**
- **RIGHT: At 5 kV , the microstructures of the specimen surface are clearly seen as the penetration and diffusion area of incident electrons is shallow.**

Accelerating Voltage Influence on Image Quality

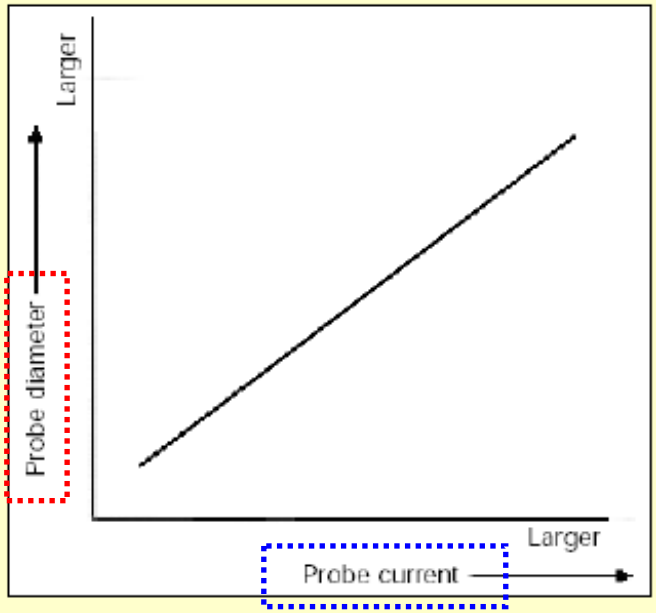
Toner

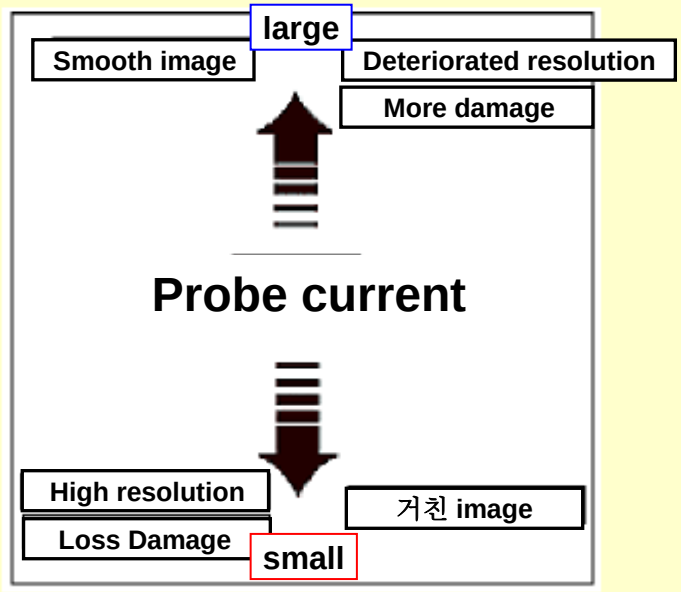
(a) 30 kV x 2,500

(a) 5 kV x 2,500

When high accelerating voltage is used as at (a), it is hard to obtain the contrast of the specimen surface structure. Besides, the specimen surface is easily charged up. The surface microstructures are easily seen at (b).

Probe Current, Probe Diameter, and Image Quality

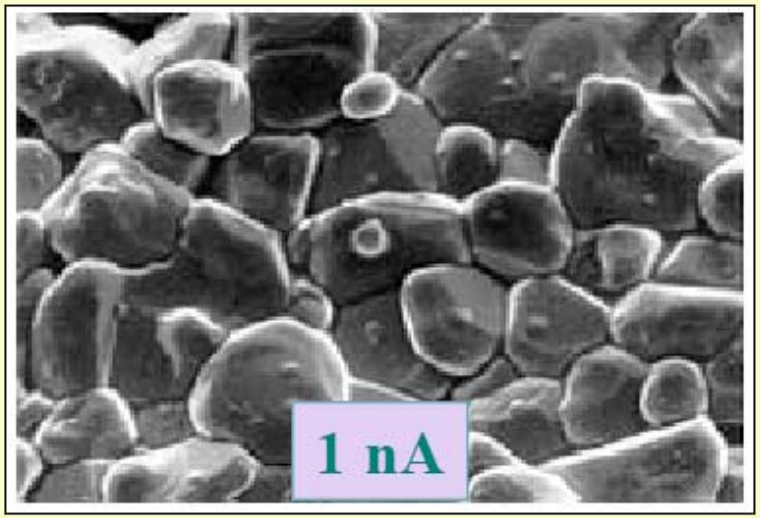




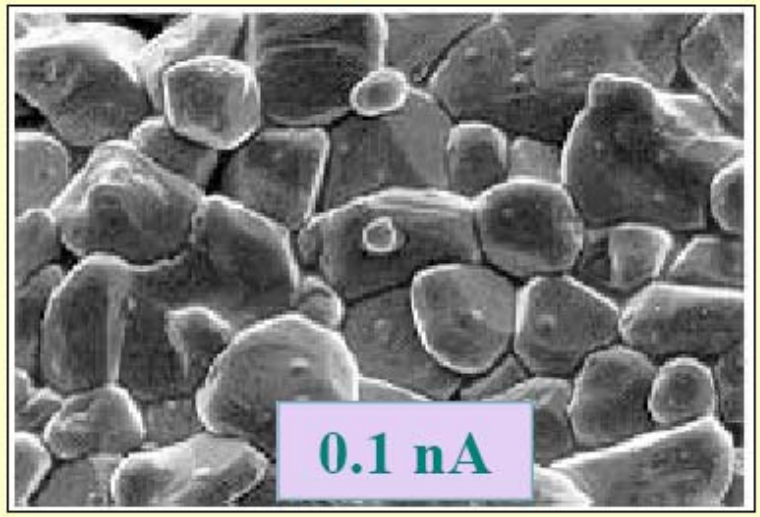
The smaller the electron probe diameter on the specimen, the higher the magnification and resolution. However, the image smoothness, namely, the S/N ratio depends on the probe current. Namely, as the probe diameter is reduced, the probe current is reduced.

It is therefore necessary to select a probe current suited for the magnification and observation conditions (accelerating voltage, specimen tilt, etc.) and the specimen.

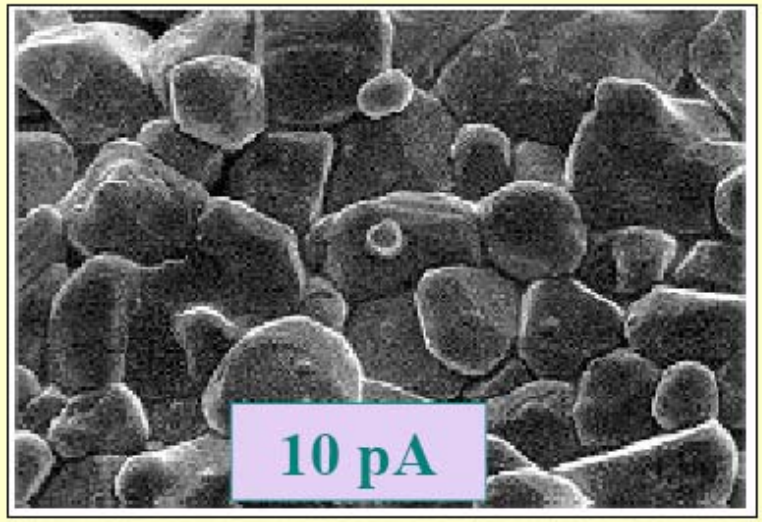
Ceramic, 10 kV, x5,400



$1/10^9$ A



$1/10^{10}$ A



$1/10^{11}$ A

- The smaller the probe current, the sharper is the image, but the surface smoothness is lost.

Edge effect Influence on Image Quality

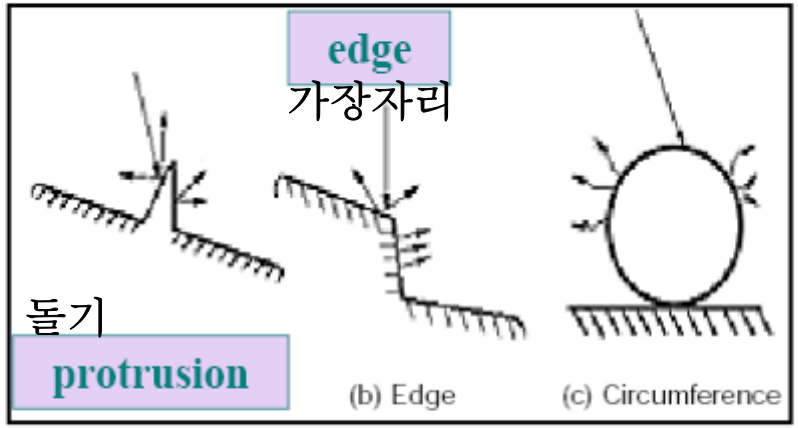
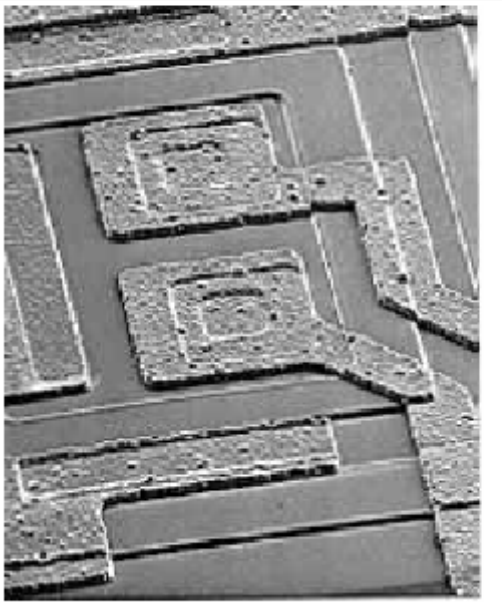


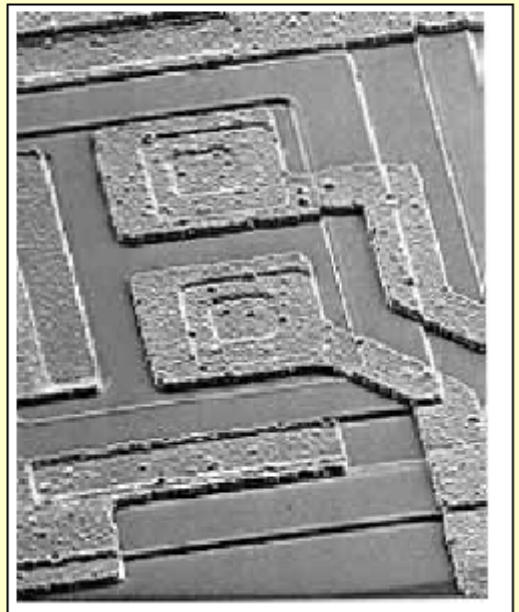
Fig. 11. Edge effect (secondary electron emission differing with surface condition).

circumference
원주



IC circuit

The higher the accelerating voltage, the greater is the edge effect, making the edges brighter.

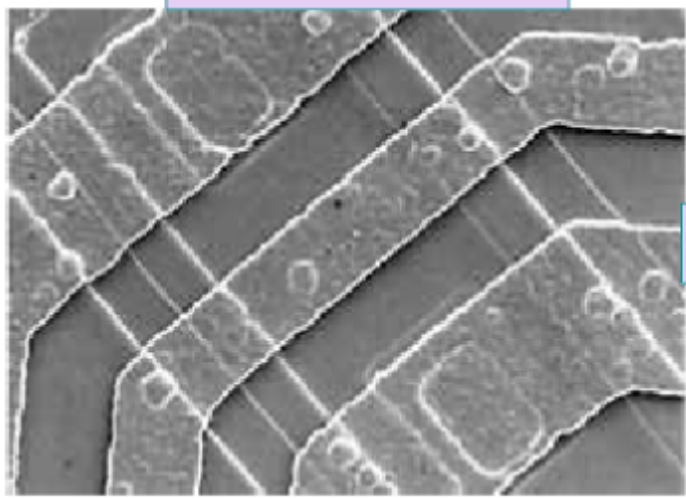


Specimen Tilt

Specimen tilt is aimed at:

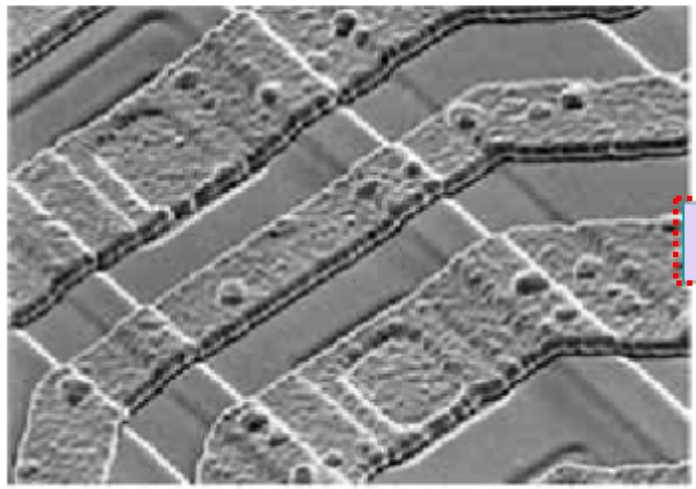
- **Improving the quality of secondary electron images**
- **Obtaining information different from that obtained when the specimen is not tilted, that is, observing topographic features and observing specimen sides.**
- **Obtaining stereo micrographs.**

IC circuit



(a) Tilt angle: 0°

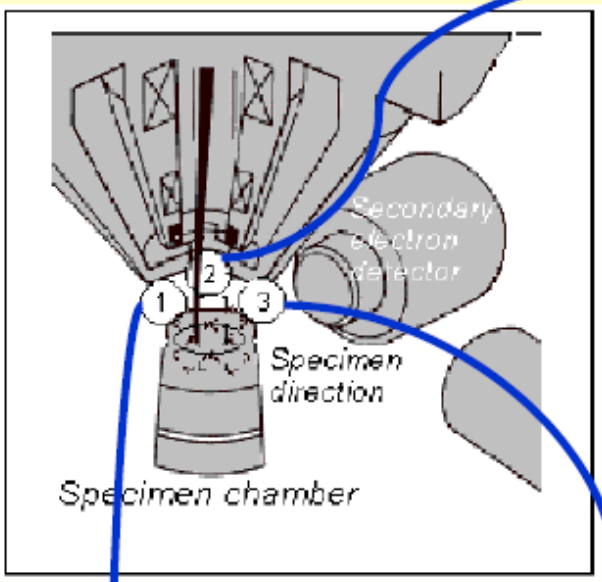
0°



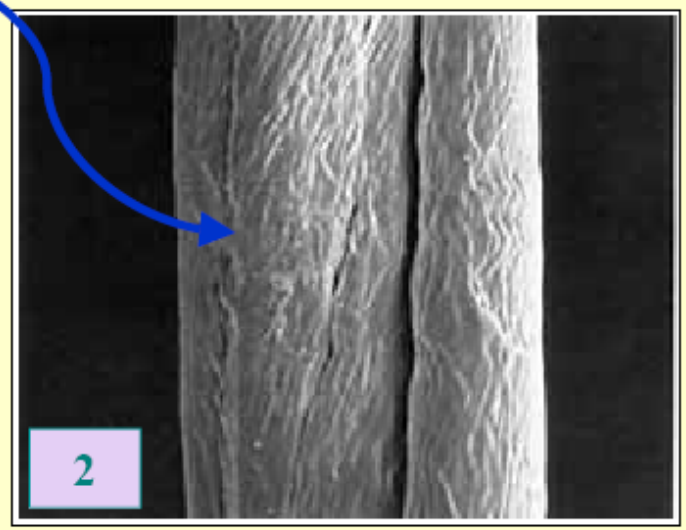
(b) Tilt angle: 45°

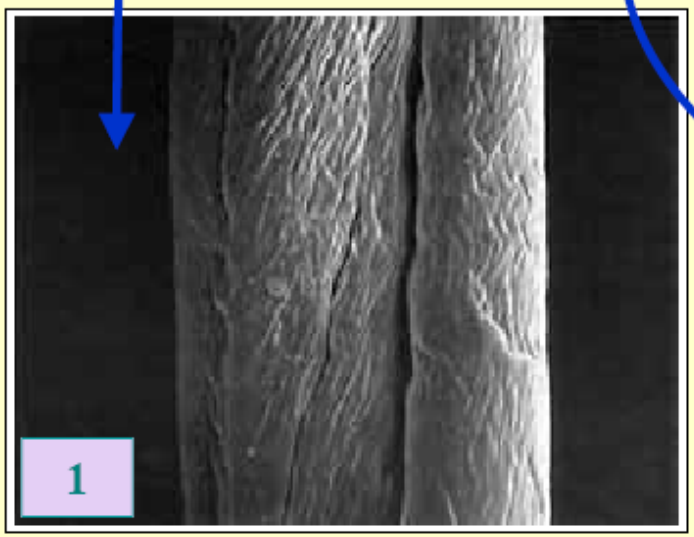
45°

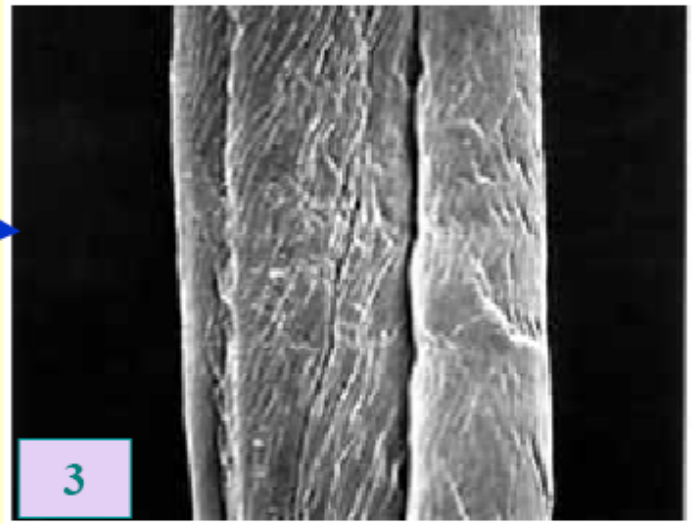
Detector position and specimen direction



Fiber







Composition of Signals

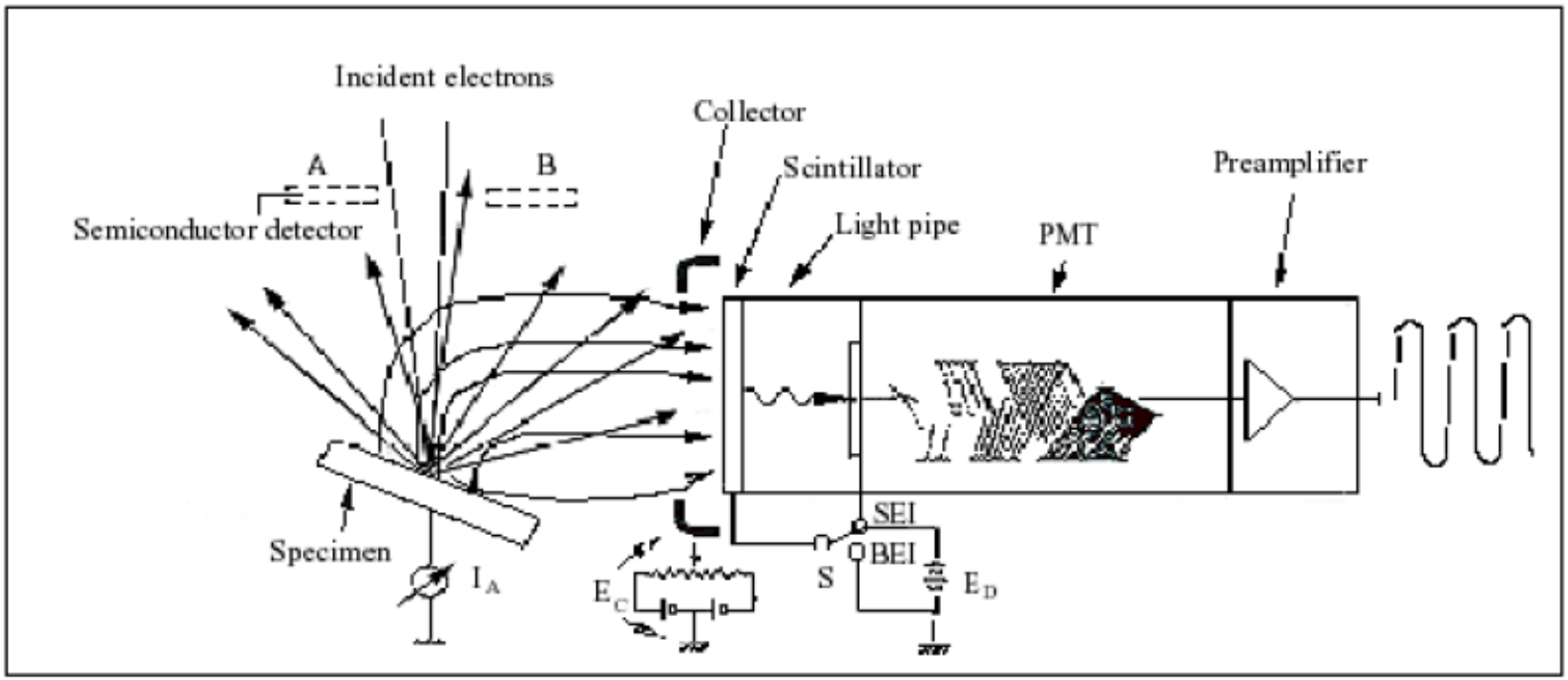


Fig. 17 Secondary electron detector

Composition of Signals

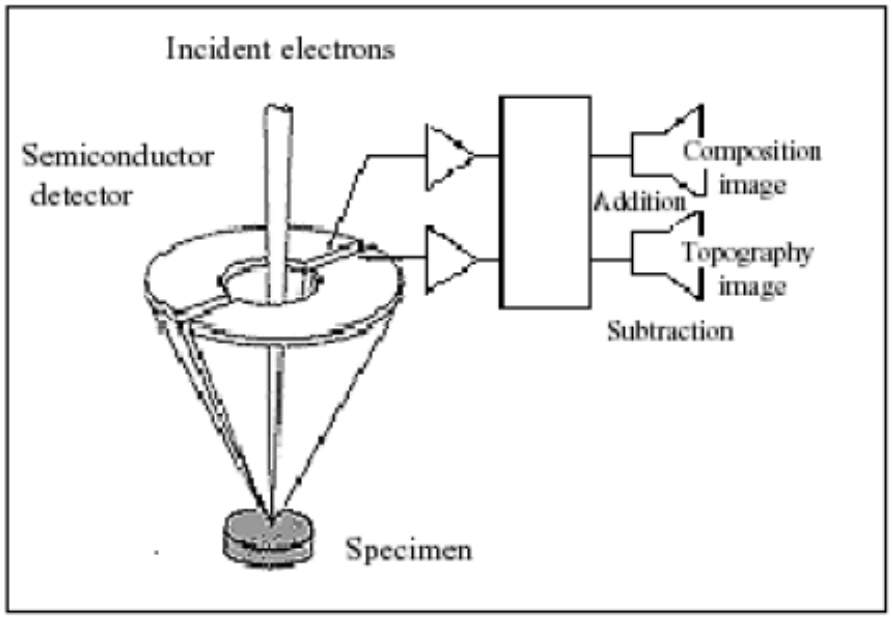


Fig. 18. Backscattered electron detector.

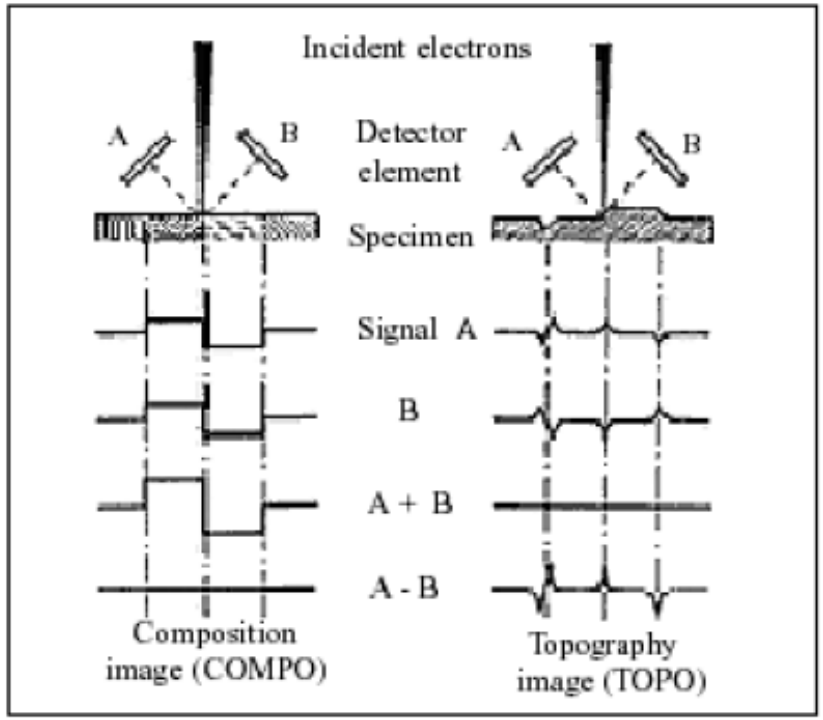


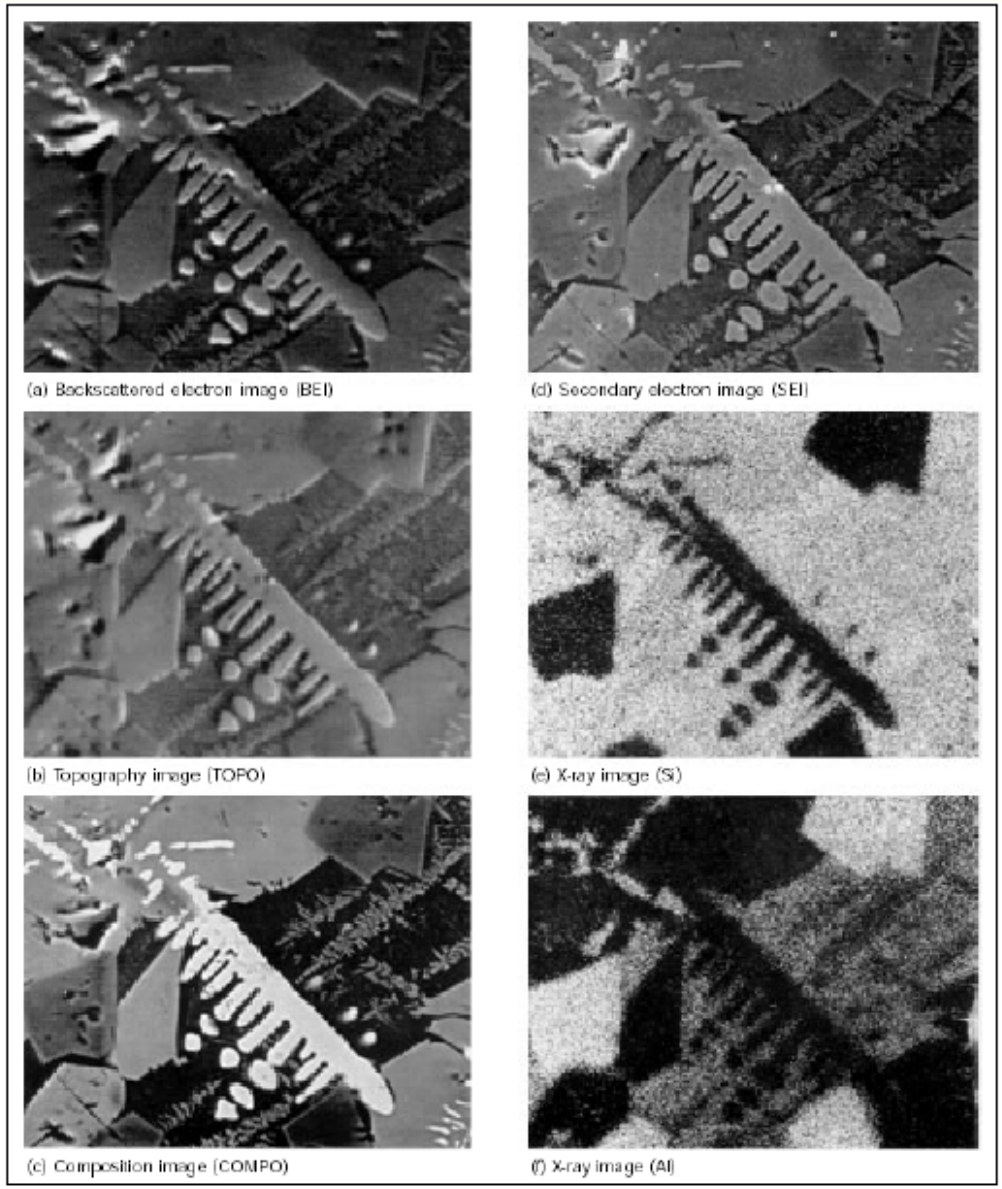
Fig. 19. Principles of composition image and topography image

Composition of Signals - Slug 20 kV, x1,100

BSE

Topography

Composition

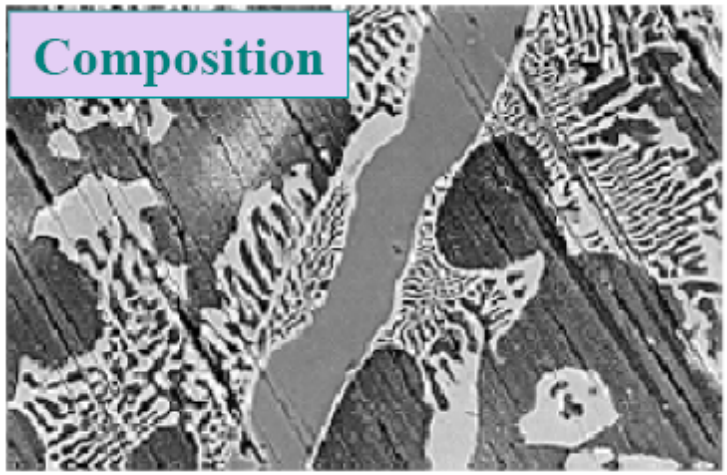


SE

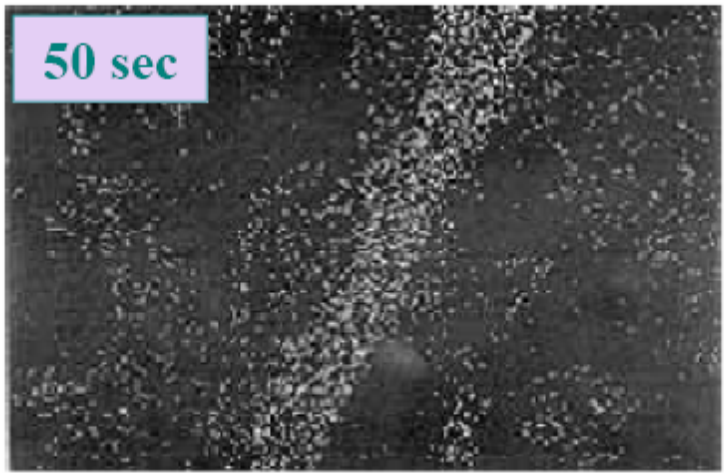
X-ray (Si)

X-ray (Al)

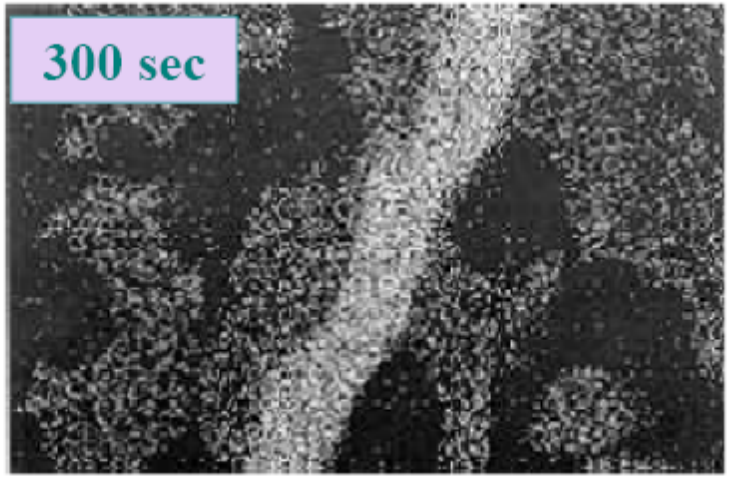
X-ray Exposure and Composition



(a) Composition image (COMPO)



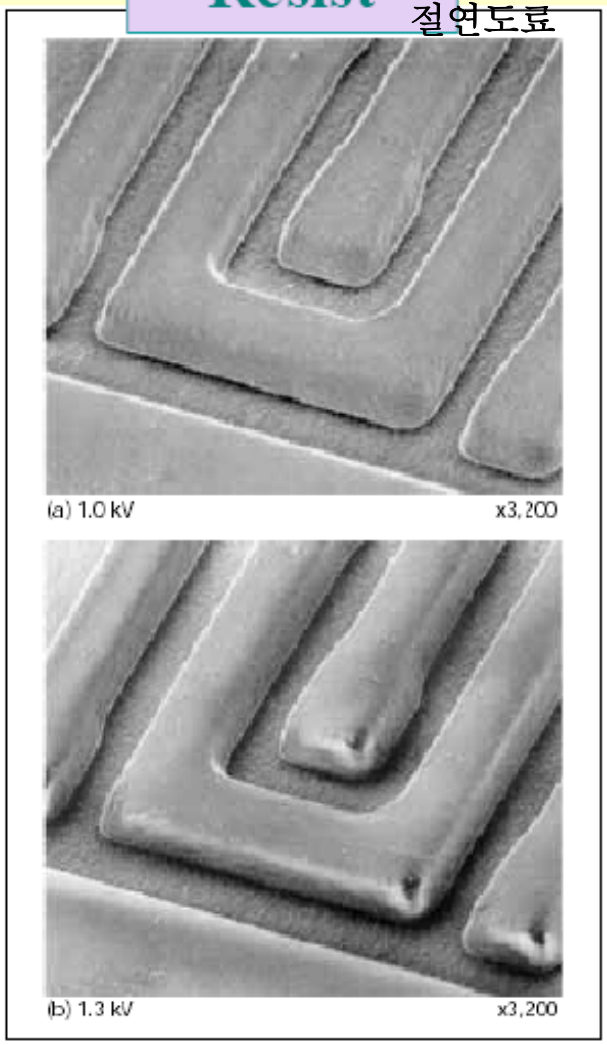
(b) X-ray image CA 50 seconds



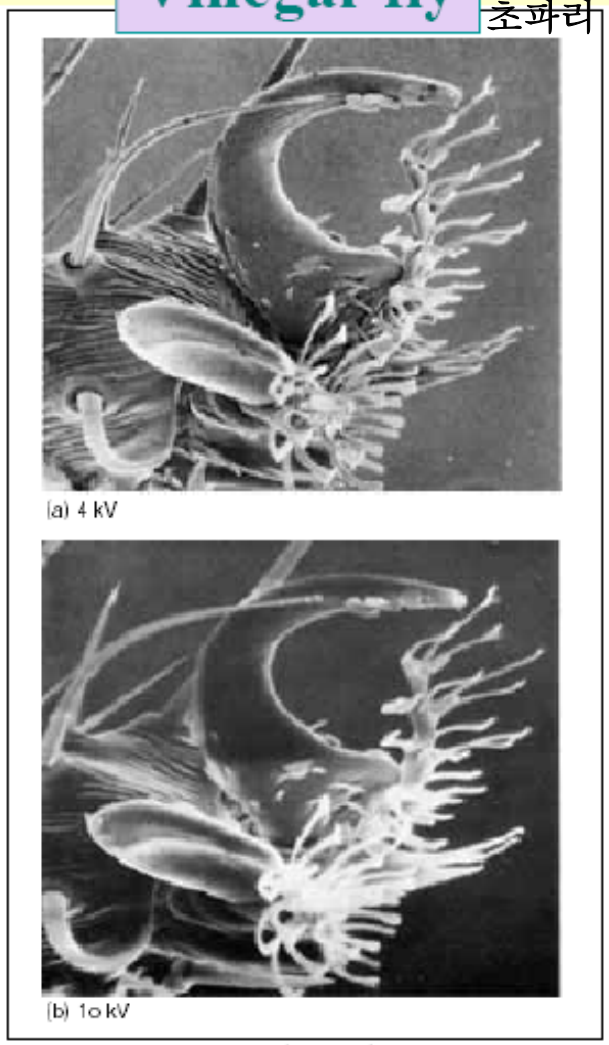
(c) X-ray image Ca 300 seconds

Influence of Charge-Up on Image Quality

Resist 절연도료



Vinegar fly 초파리



1.0 KV

1.3 KV

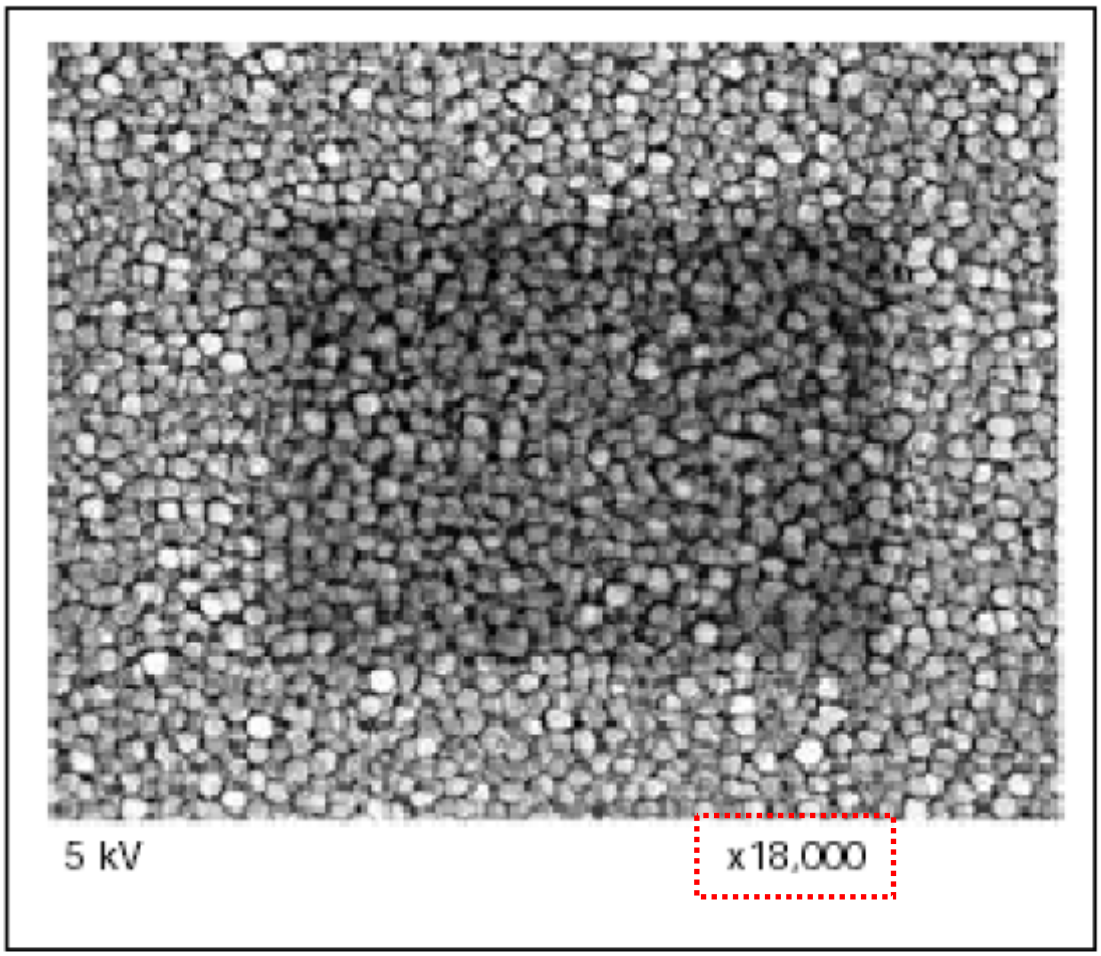
4 KV

10 KV

1) Probe current, 2) Accelerating voltage, 3) Tilting angle

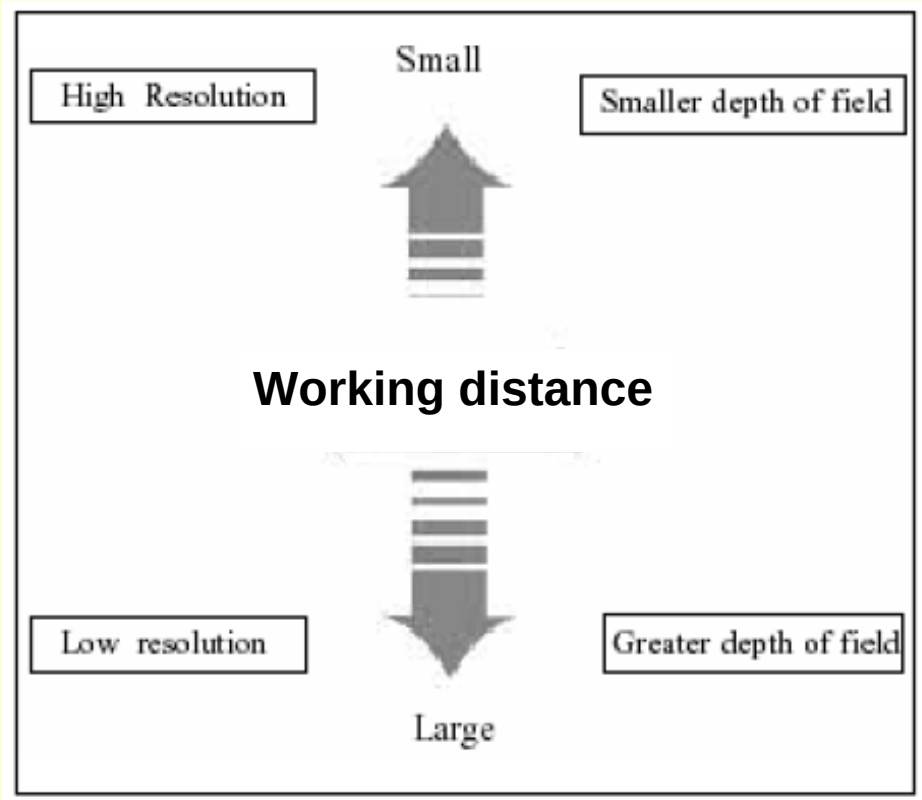
Contamination

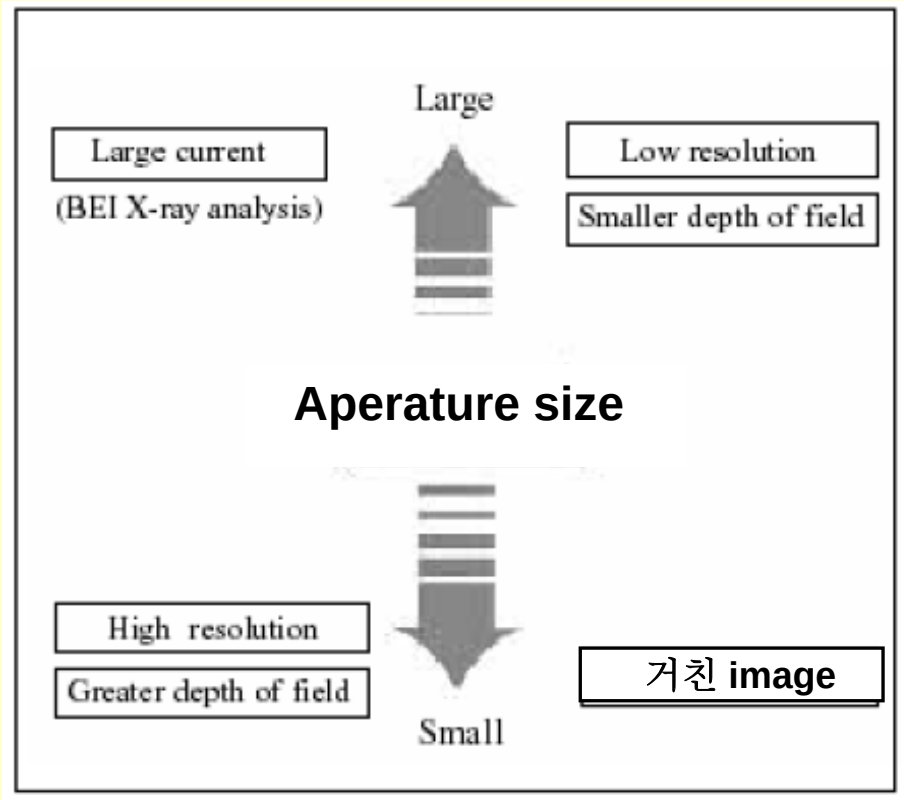
ITO



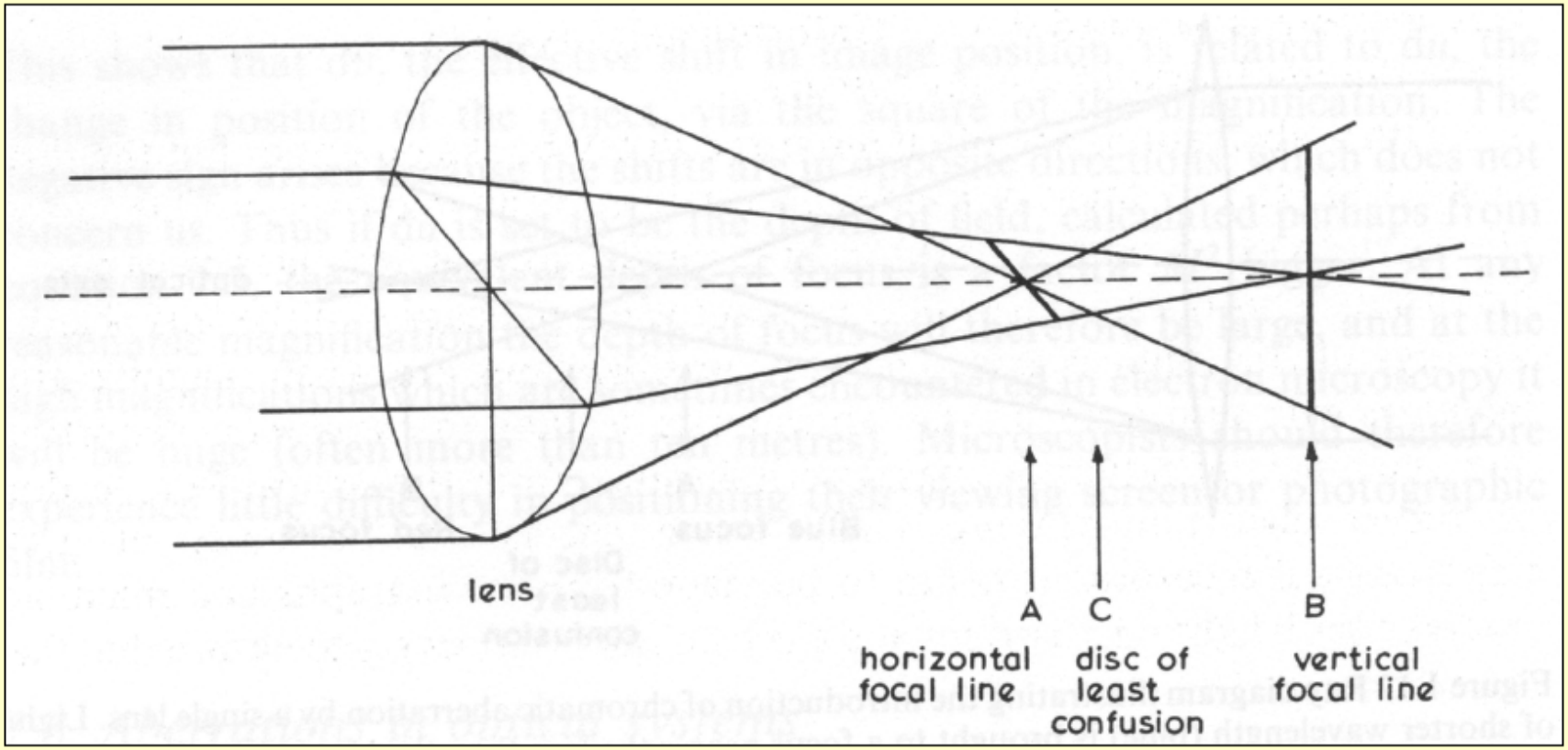
1) Dry and clean sample, 2) Low temp, 3) Small samples

Effect of Working Distance and Aperture Size





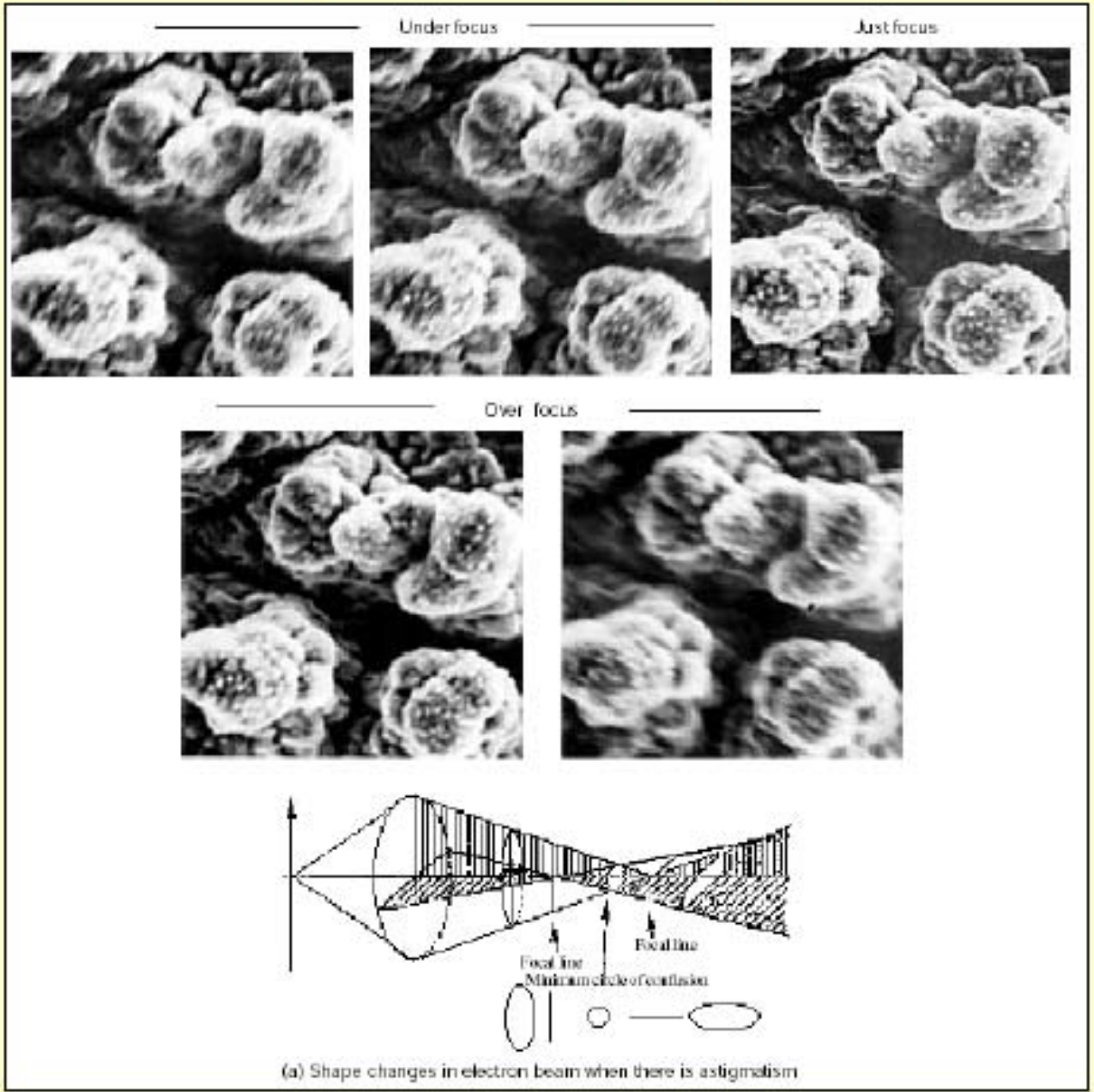
Astigmatism



Formation of astigmatism for a lens with slightly different optical properties in the horizontal and vertical directions

Astigmatism

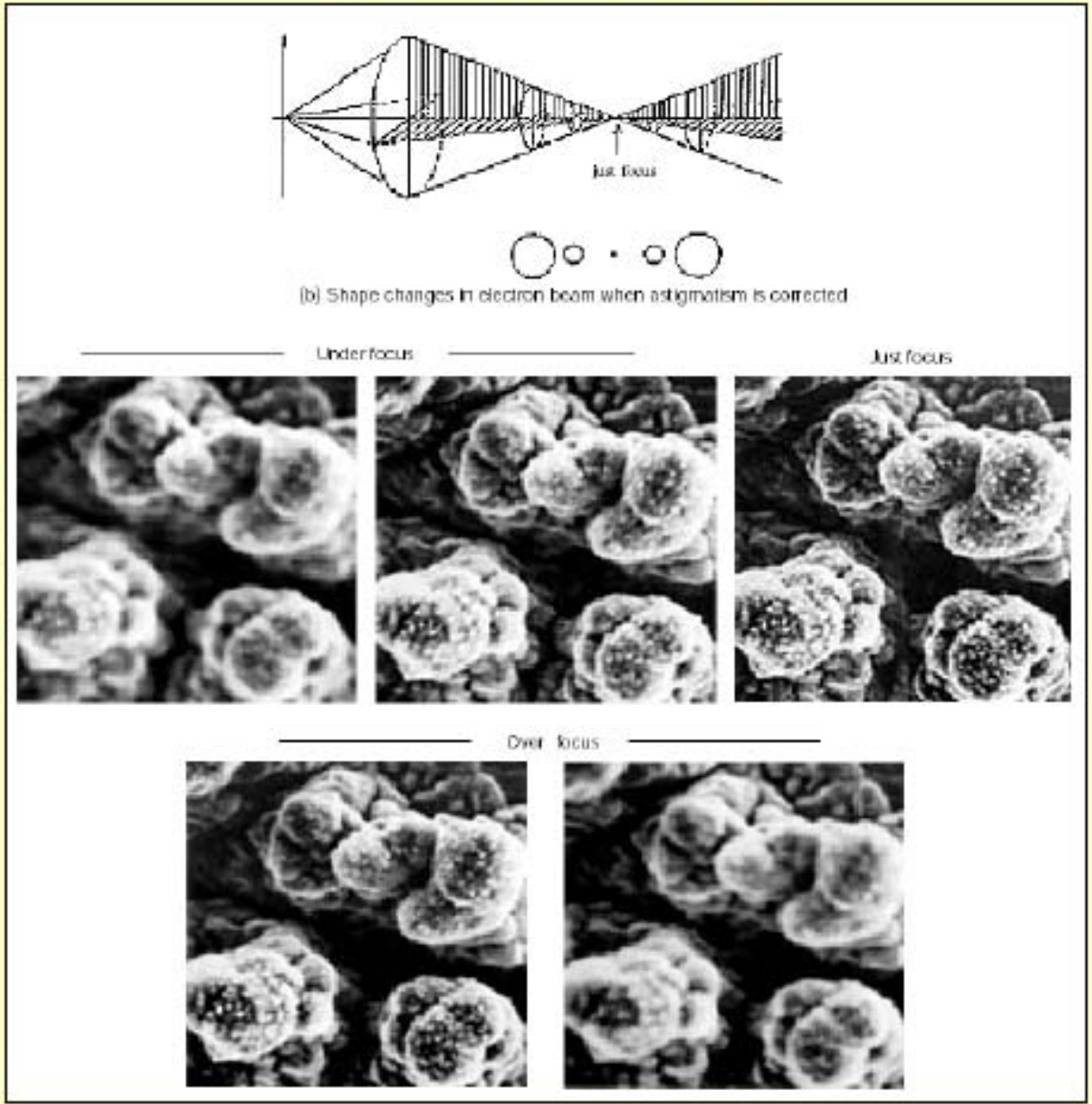
Wrong



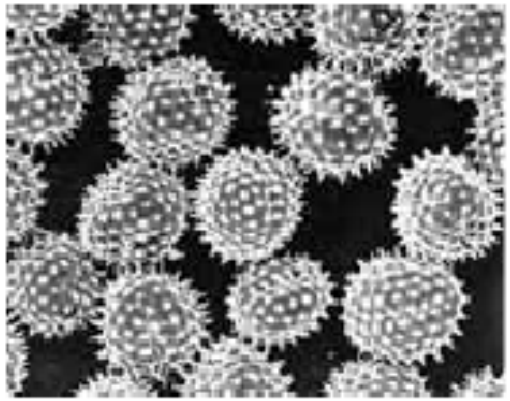
(a) Shape changes in electron beam when there is astigmatism

Astigmatism

Correct

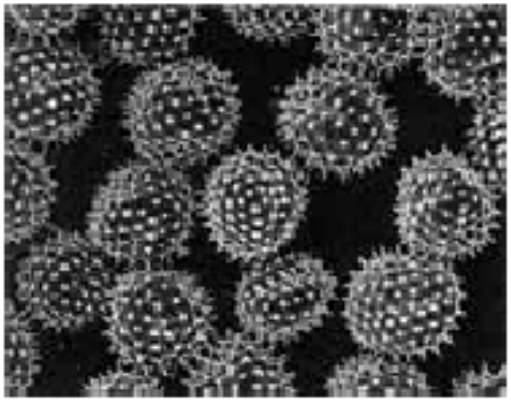


Contrast & Brightness

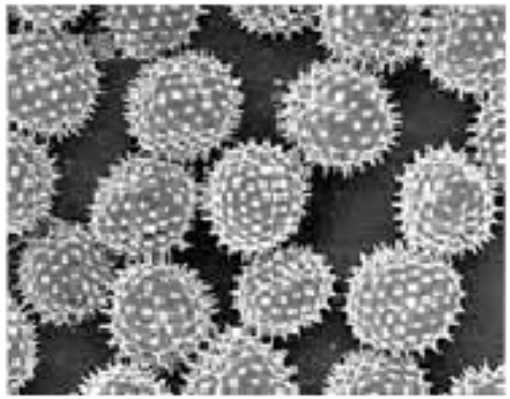


Excessive contrast

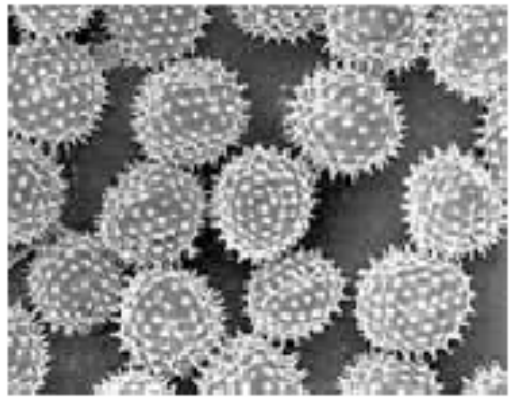
+



Insufficient brightness

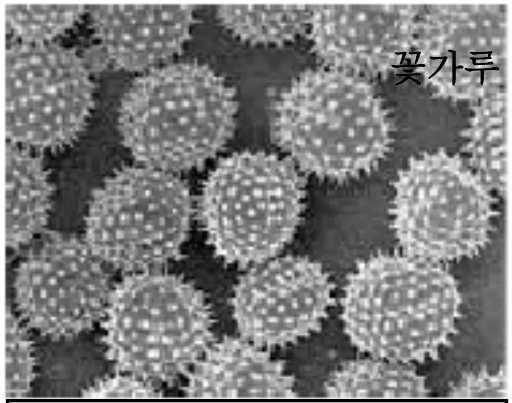


Optimum



Excessive brightness

-



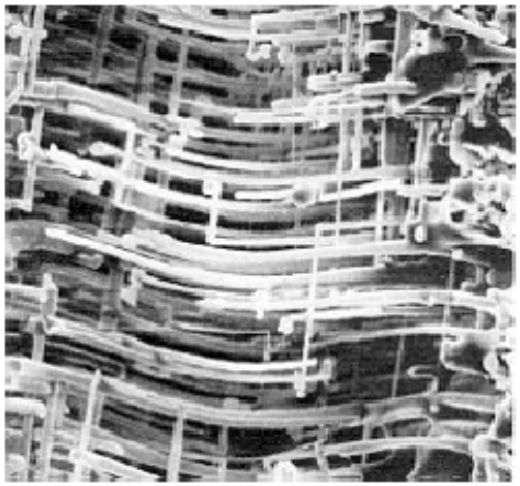
Insufficient contrast

Pollen of marigold

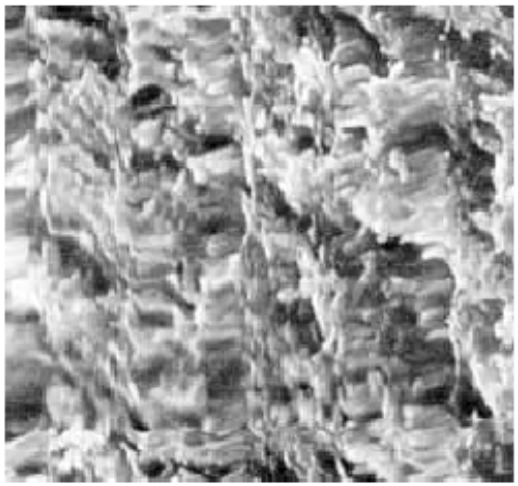
x360

External Disturbances

Magnetic Field



(a) Influenced by external magnetic field.

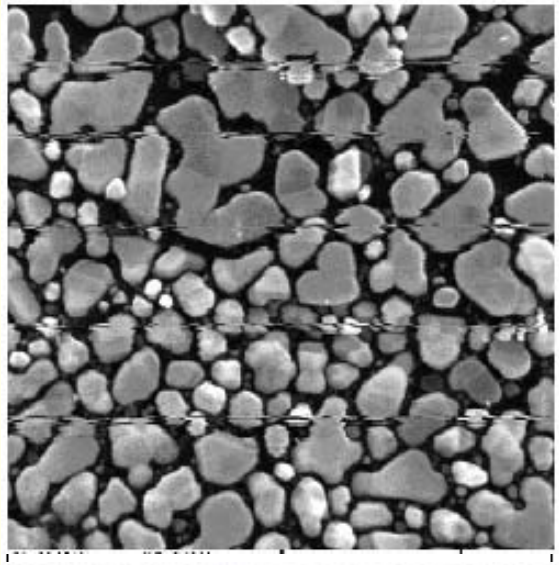


(b) Influenced by external magnetic field

Influenced

Uninfluenced

Mechanical vibration



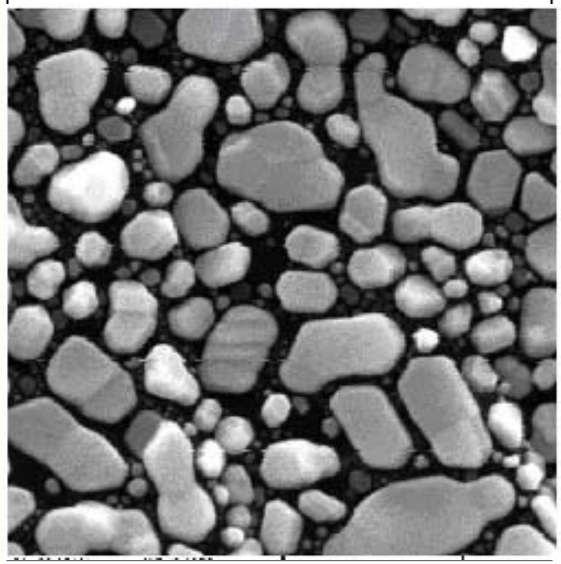
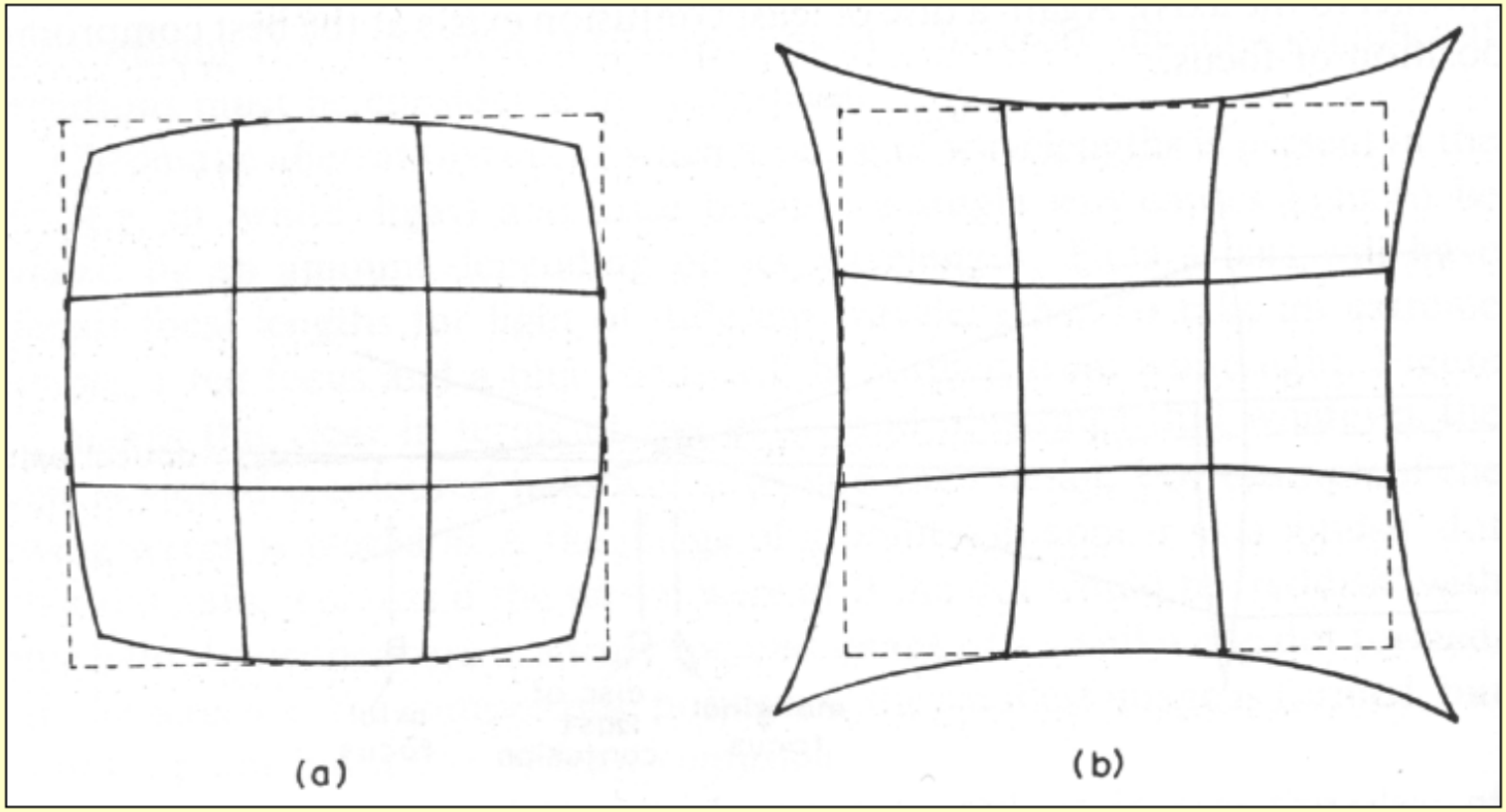


Image Distortion



Barrel

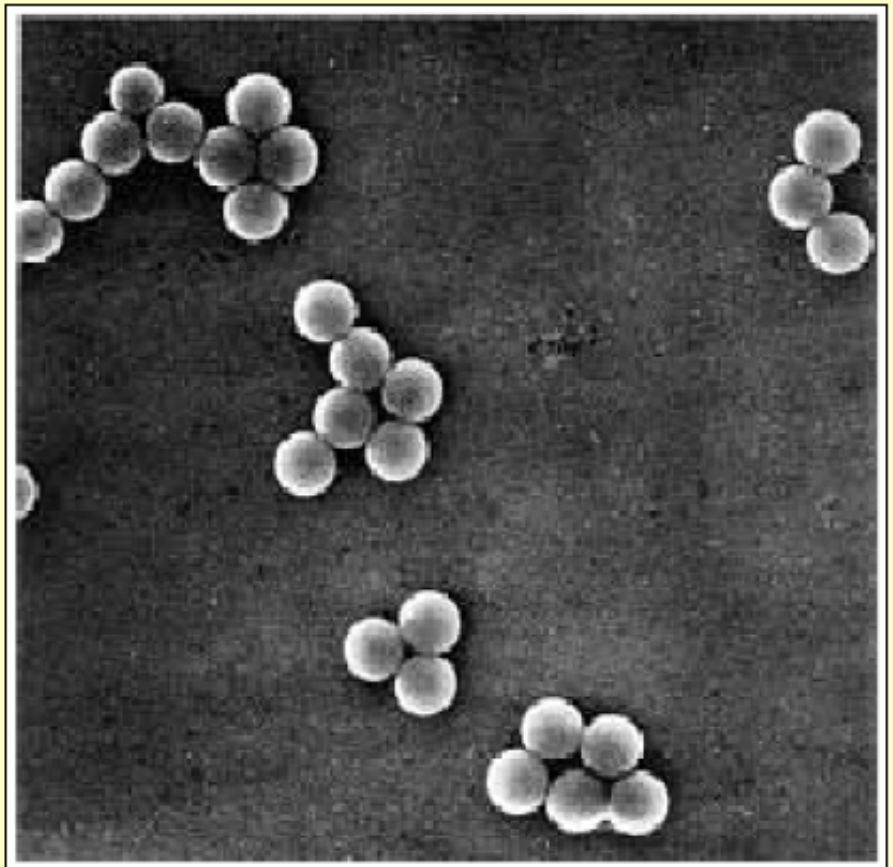
Pin-cushion

Barrel – Magnification decreases with distance from optical axis

Pin-cushion – Magnification increases with distance from optical axis

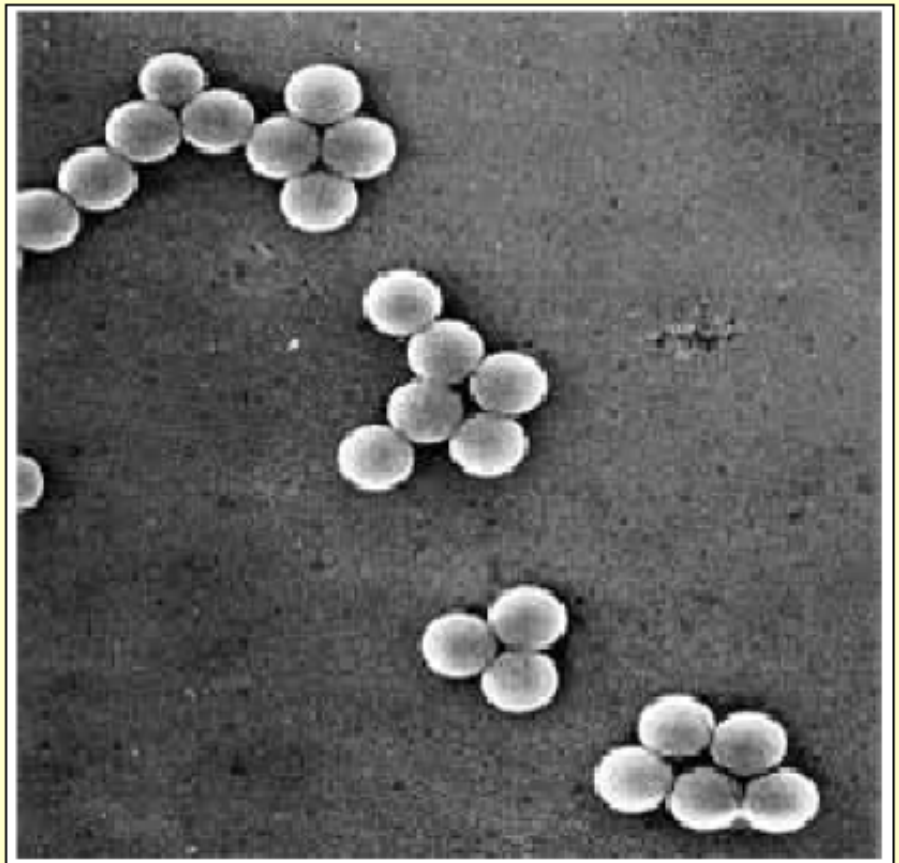
Image Distortion

Normal



(a) Normal Image

Horizontally Distorted



(b) Horizontally distorted image

Latex particles

고무나무 유액

Summary

What you see…..

Is not necessarily what you get!!!

Be careful with images…