The most versatile instrument for a materials scientist?

It is a microscope that produces an image by using an electron beam that scans the surface of a specimen inside a vacuum chamber.

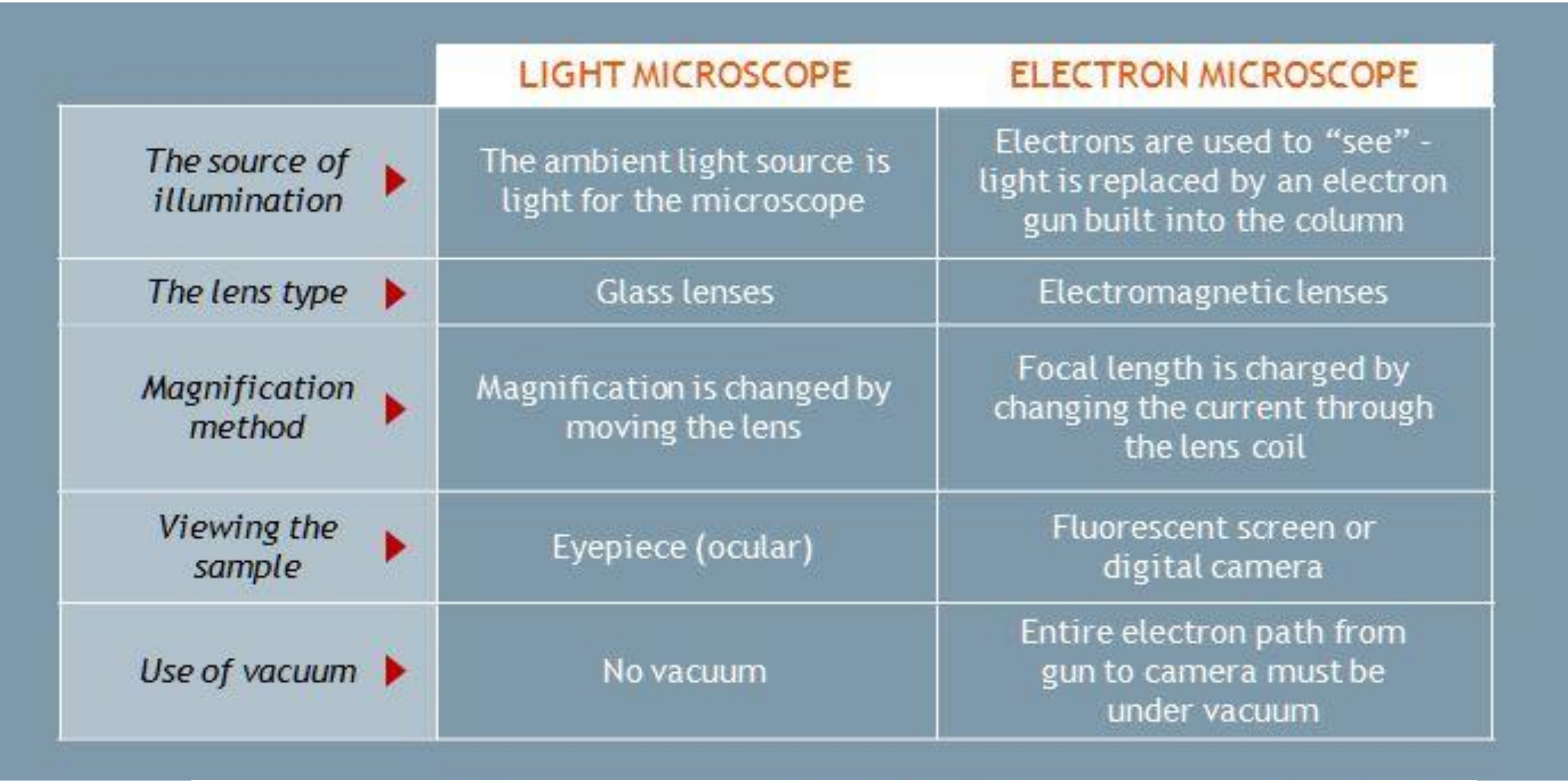
What can we study in a SEM?

- Topography and morphology
- Chemistry
- Crystallography
- Orientation of grains
- In-situ experiments:
  - Reactions with atmosphere
  - Effects of temperature

"Easy" sample preparation!!

"Big" samples!

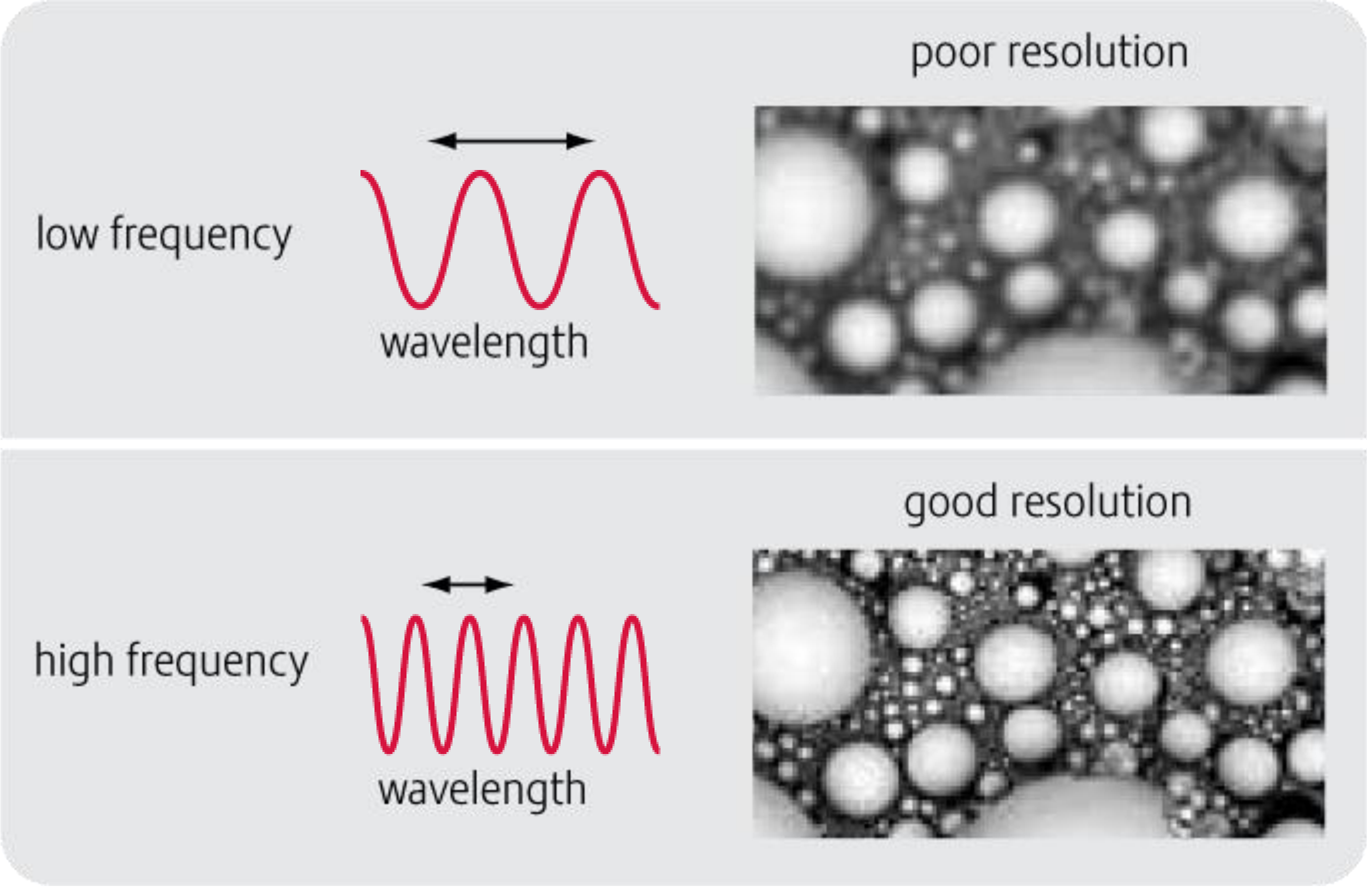
Optical Microscope versus Electron Microscope



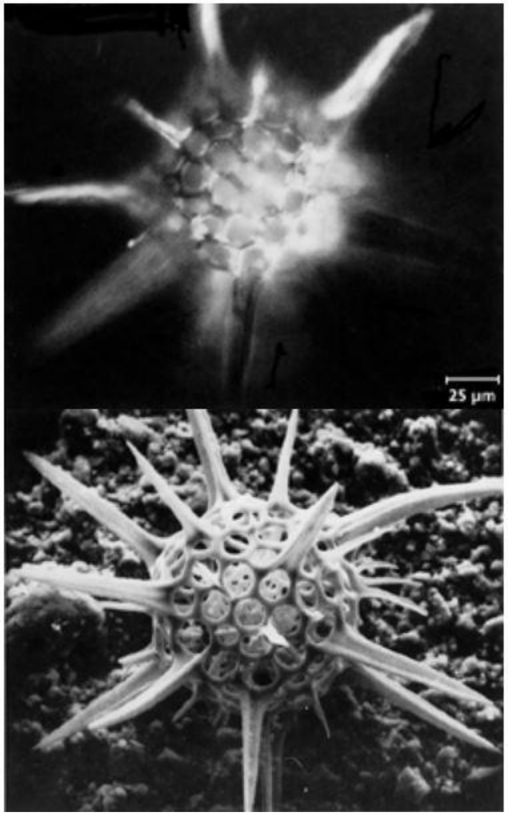
	LIGHT MICROSCOPE	ELECTRON MICROSCOPE
The source of illumination	The ambient light source is light for the microscope	Electrons are used to "see" - light is replaced by an electron gun built into the column
The lens type	Glass lenses	Electromagnetic lenses
Magnification method	Magnification is changed by moving the lens	Focal length is charged by changing the current through the lens coil
Viewing the sample	Eyepiece (ocular)	Fluorescent screen or digital camera
Use of vacuum	No vacuum	Entire electron path from gun to camera must be under vacuum

	Magnifications	Depth of Field	Resolution
OM:	4x – 1400x	0.5mm	~ 0.2mm
SEM:	10x – 500Kx	30mm	1.5nm

How is Resolution Affected by Wavelength?



Electronic Microscope for higher resolution



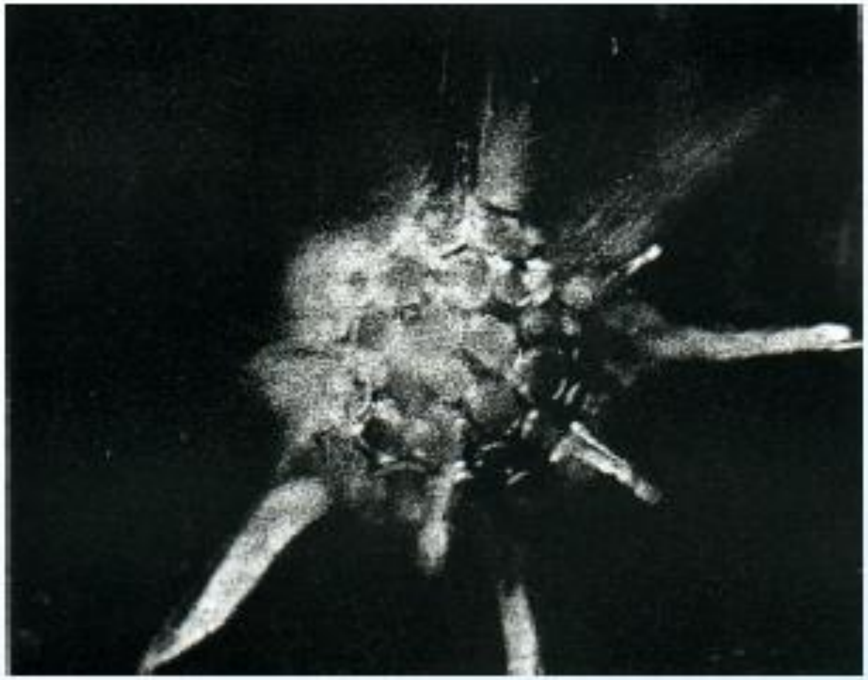
- Resolution limit of optical microscopes is due to the light diffraction; roughly optical resolution can be estimated as wavelength λ/2NA (NA is the numerical aperture of lens, usually ~ 1.0): for white light, average wavelength is around 500 nm, the best resolution is thus a few hundreds nm.

- Decreasing the wavelength is the way to improve the resolution.

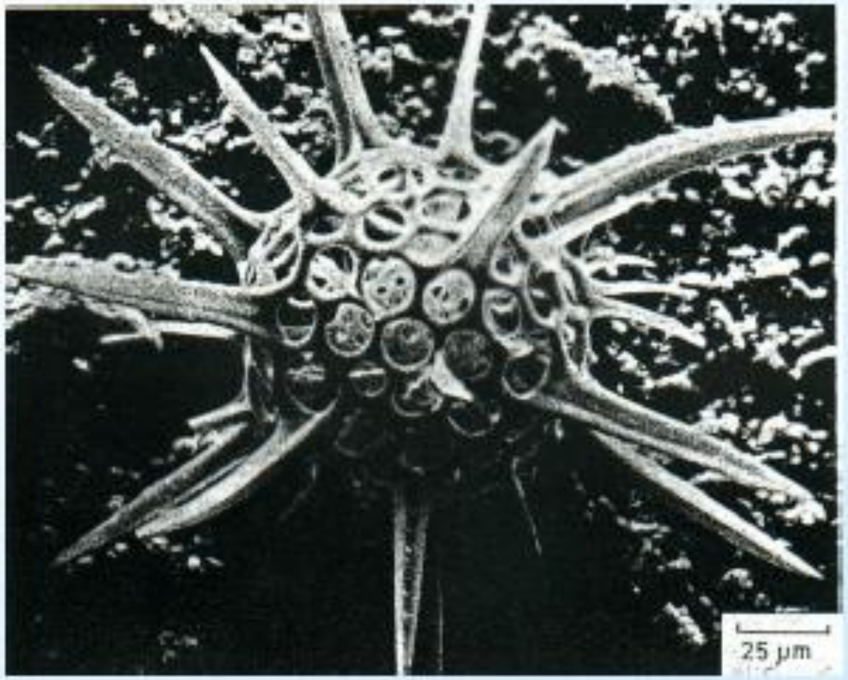
- Electron wave is a unique medium that can be used in imaging. By accelerating the electrons into high energy beam (via high voltage), the wavelength thus created is far shorter than white light. For example, for an electron beam produced from a 20 kV gun, the wavelength is only 1240.7/20,000 (eV) = 0.06 nm = 0.6 Å, corresponding to a resolution limit of λ/2 = 0.3 Å --- theoretically, it can be used to image a species as small as 0.3 Å. Most atoms are in size of 2-3 Å.

Scanning Electron Microscopy

Image Quality



Optical Microscope



SEM

SEM is very useful for examining objects at a wide range of magnifications, compared to optical microscope.

Electron Microscopy

SEM

FESEM

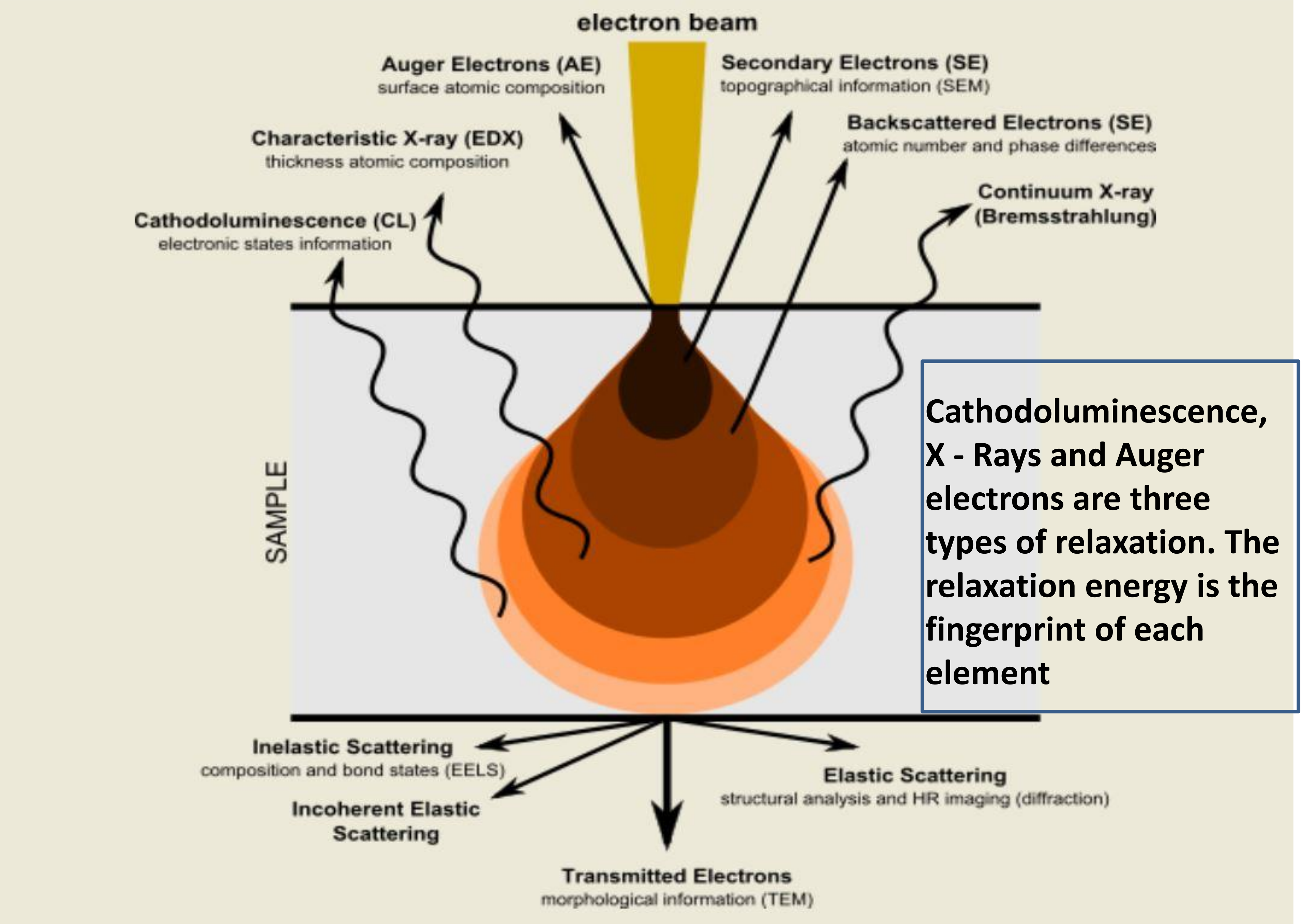
A standard SEM is typically used for low-to-medium magnification (10-150,000X) imaging of conductive samples, usually metals.

For non-conductive samples, a conductive coating of carbon, gold, chromium, etc. should be applied to avoid charging effects.

A variable pressure SEM (low vacuum or environmental) is used for non-conductive specimens like glass, polymers/paint, and biological materials.

A field-emission SEM (FESEM) is used for medium-high magnification (20,000- 800,000X) and high resolution at low electron beam accelerating voltages (0.5-2 kV) resulting in excellent surface texture images even on non-conductive materials.

Signals from the sample



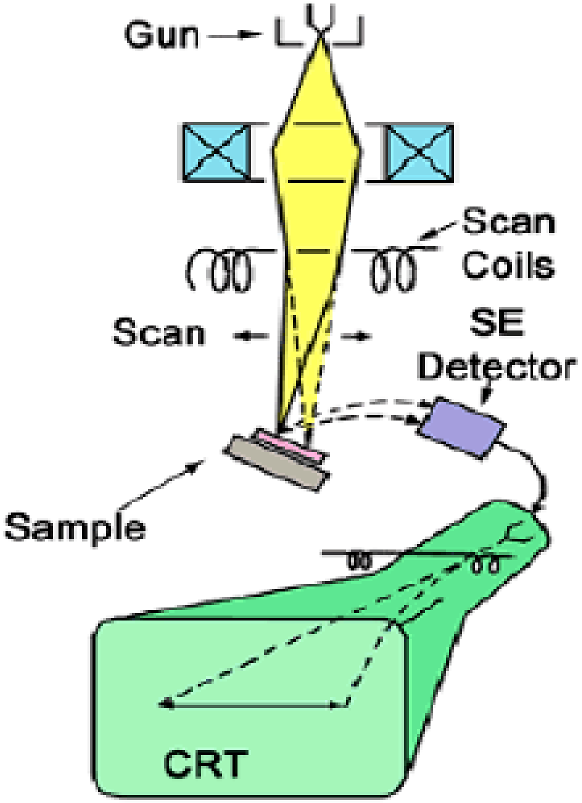
Cathodoluminescence, X - Rays and Auger electrons are three types of relaxation. The relaxation energy is the fingerprint of each element

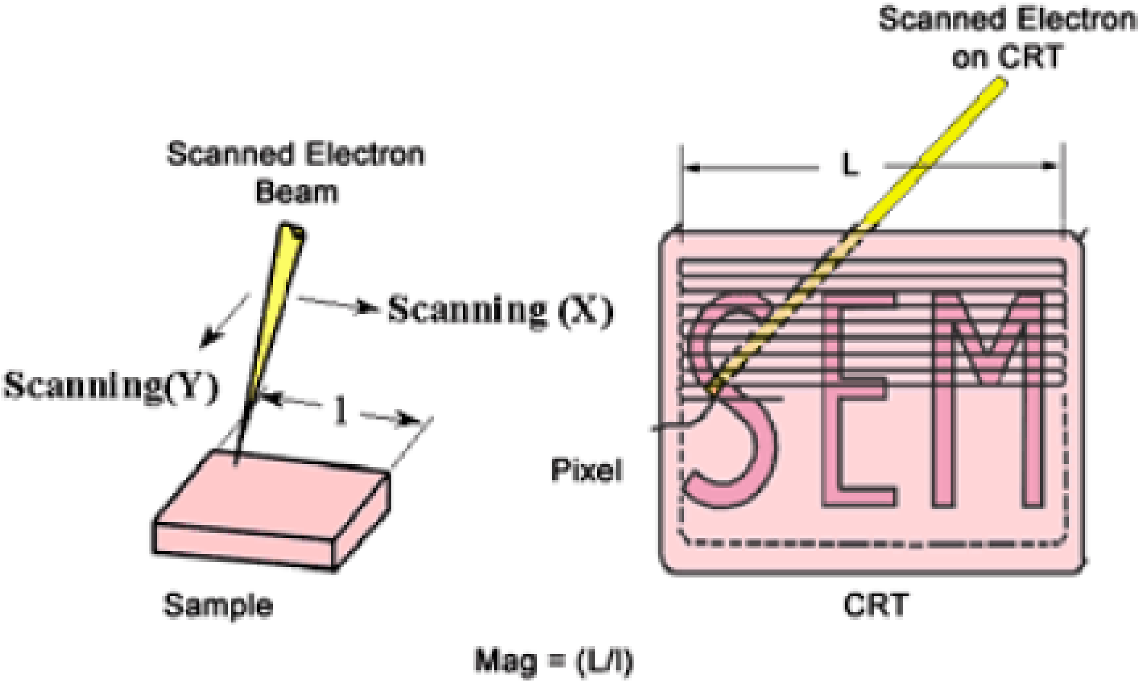
Scanning Electron Microscopy

The variety of electron induced signals that are produced in the SEM can provide

- Morphological (surface topography)
- Composition (chemical information)
- Physical Info (structural, electrical)

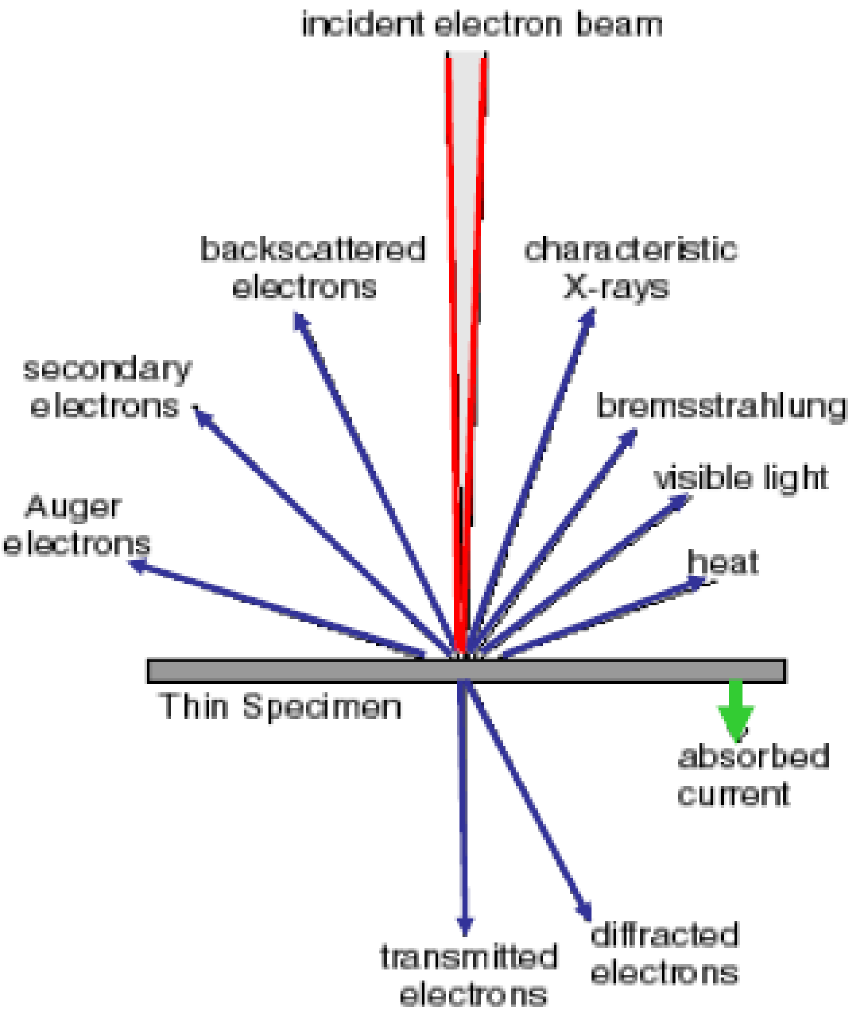
Scanning Electron Microscope

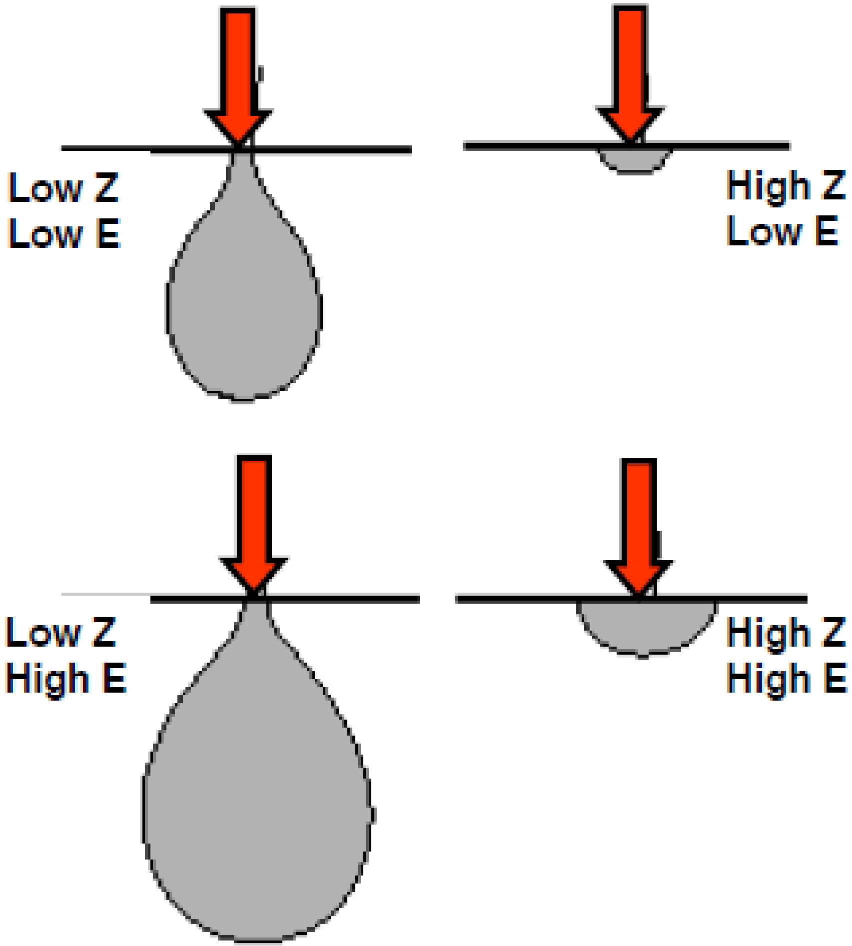




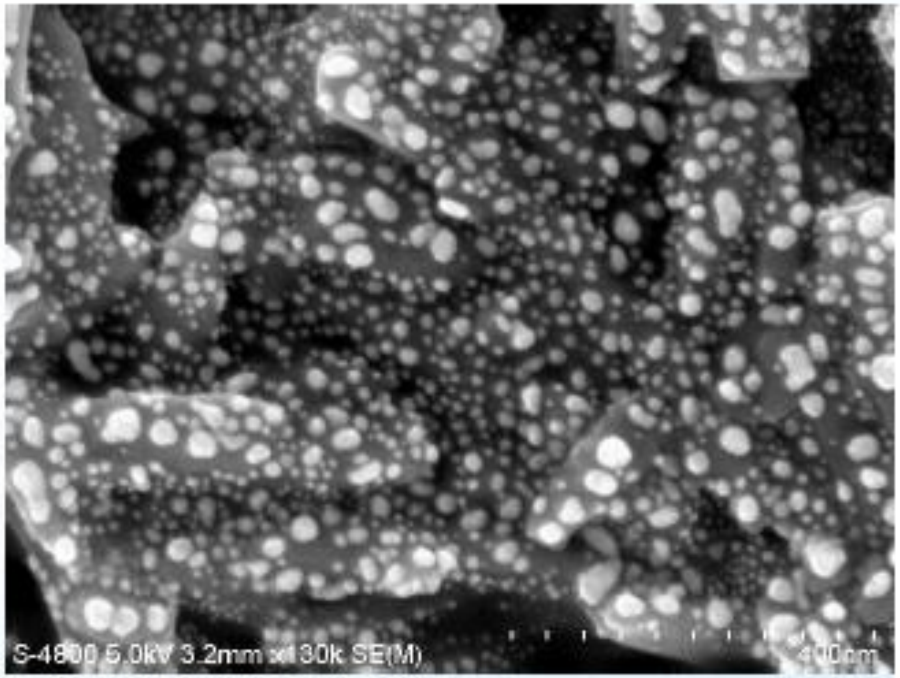
In SEM, an electron beam is focused to a fine probe which is scanned over the sample. As the beam is scanned, the beam interacts with the specimen to create several types of signals which can be detected. The detected signal is displayed on a CRT which is raster scanned in synch with the electron beam thus forming an image on the screen. The magnification of the image is a function of the ratio of the displayed image size over the scanned electron beam image size.

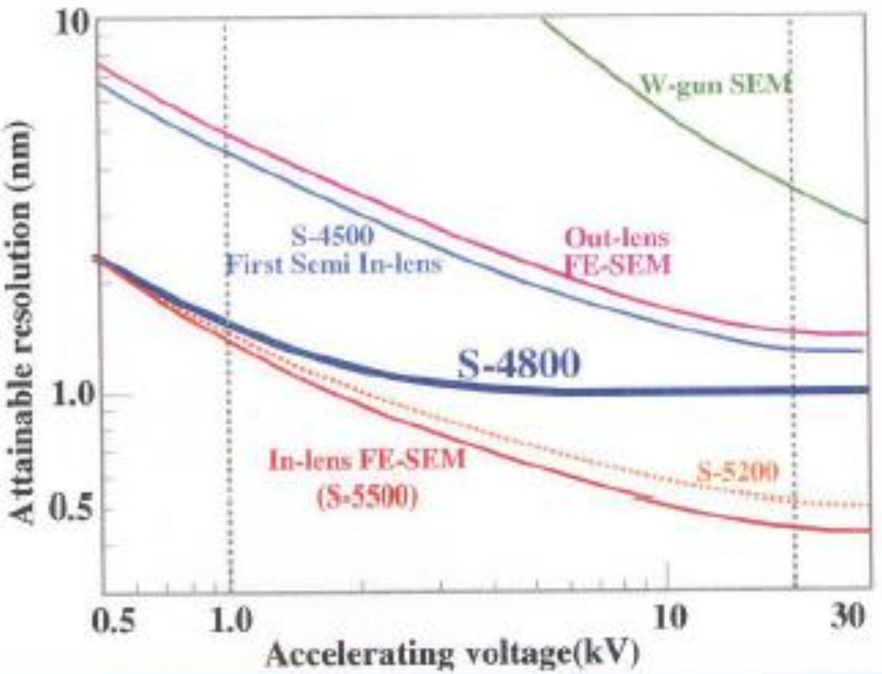
Electron Interaction with Solids





Resolution of SEMs



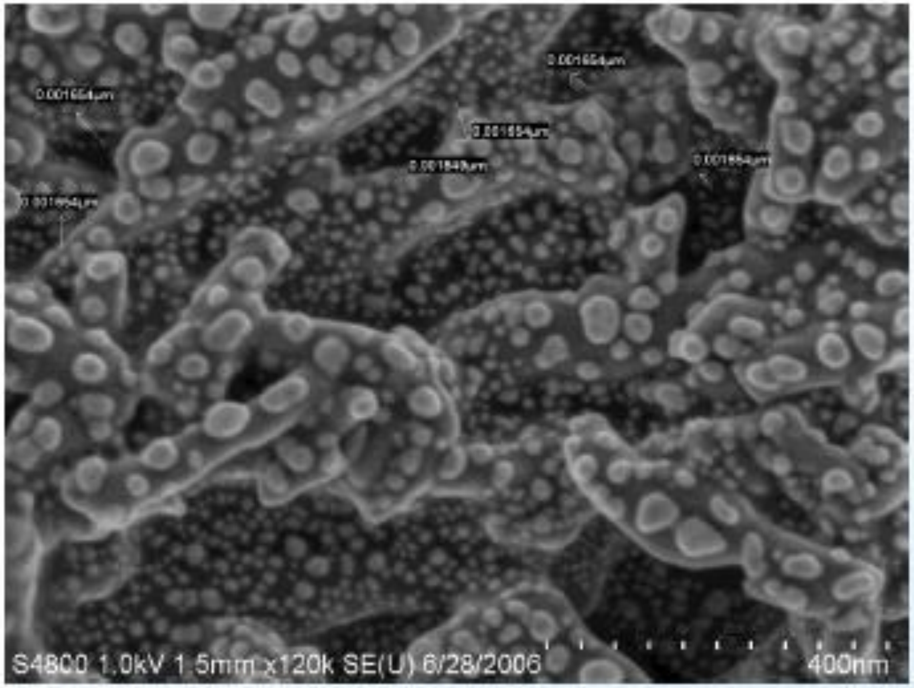


SEM provides high image magnification and resolution!

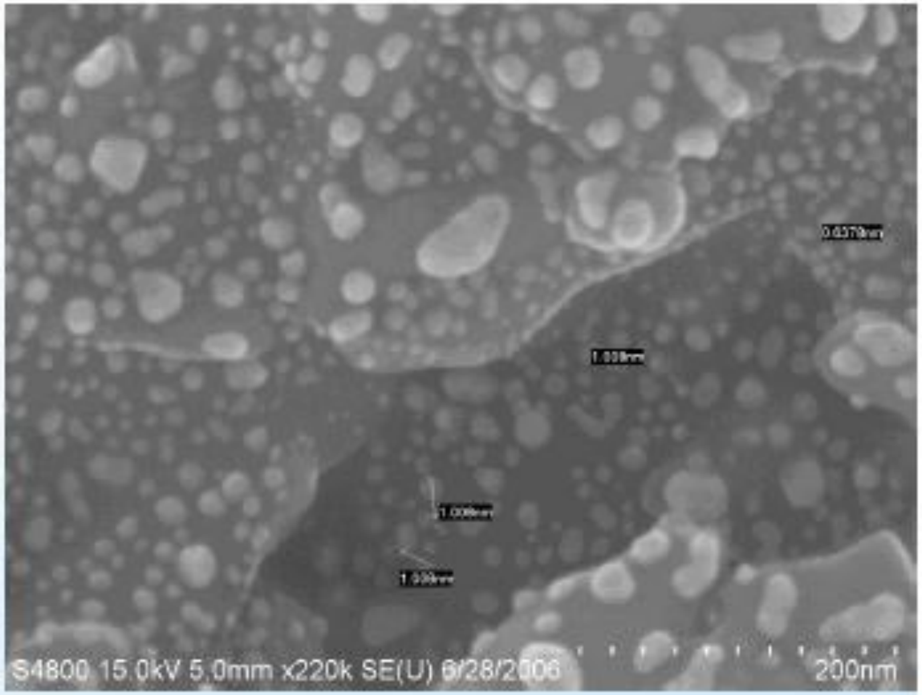
5.0 nm (50 Å) - common for commercial instruments

2.0 nm (20 Å) - for Advanced Research instruments

FESEM Resolution



1.0 KV

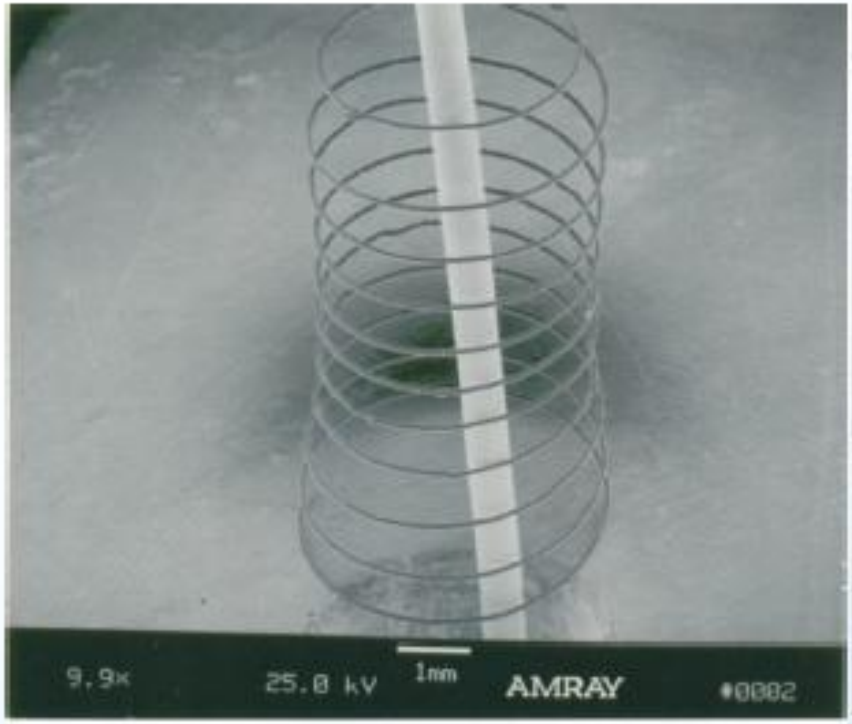


15.0 KV

Field-emission SEMs are superior!

Resolution of 1.0 nm can be easily attained.

3-D View of SEM Images

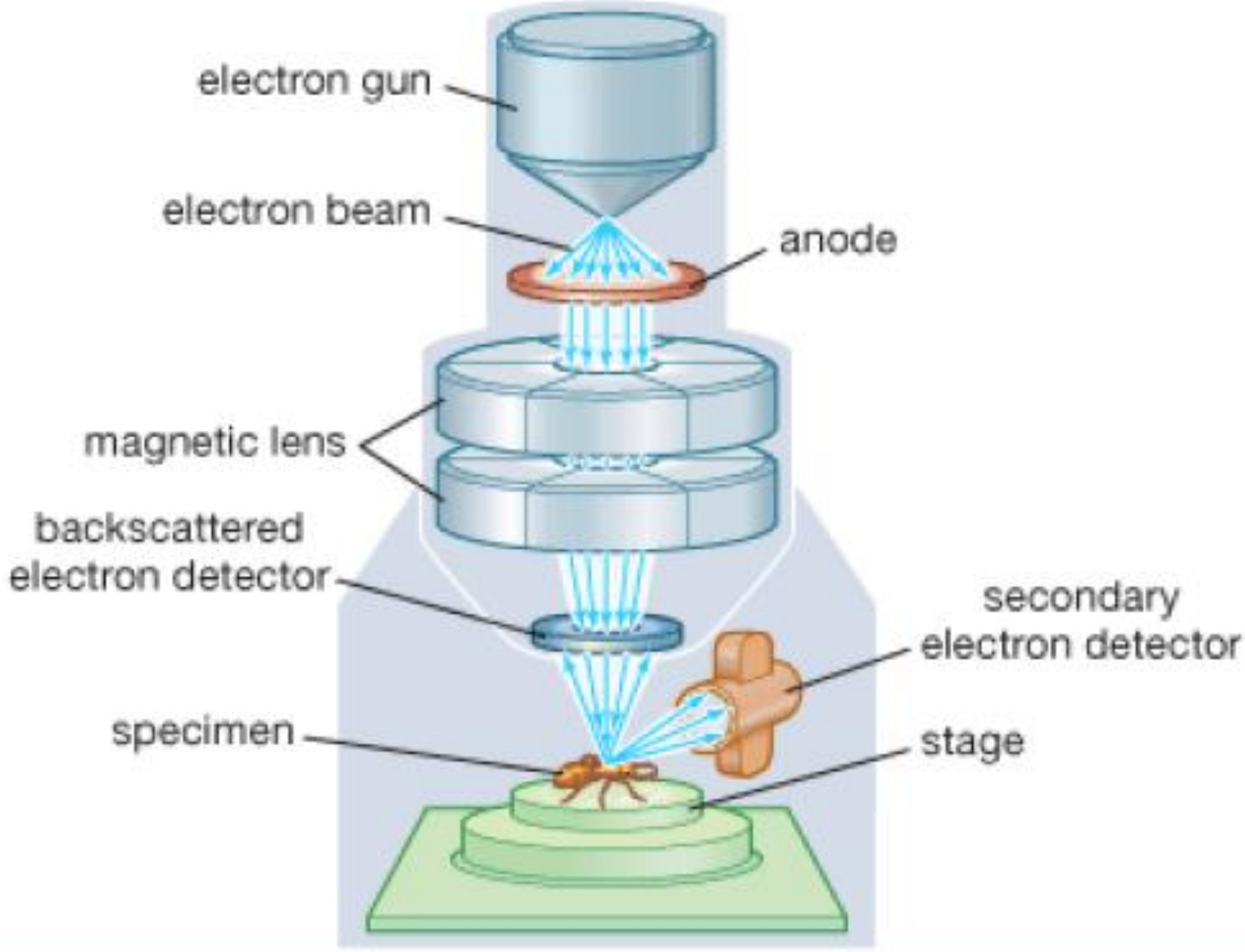


Large DOF provides more info about the specimen, and makes it easy to interpret the image.

Basic Components of SEM

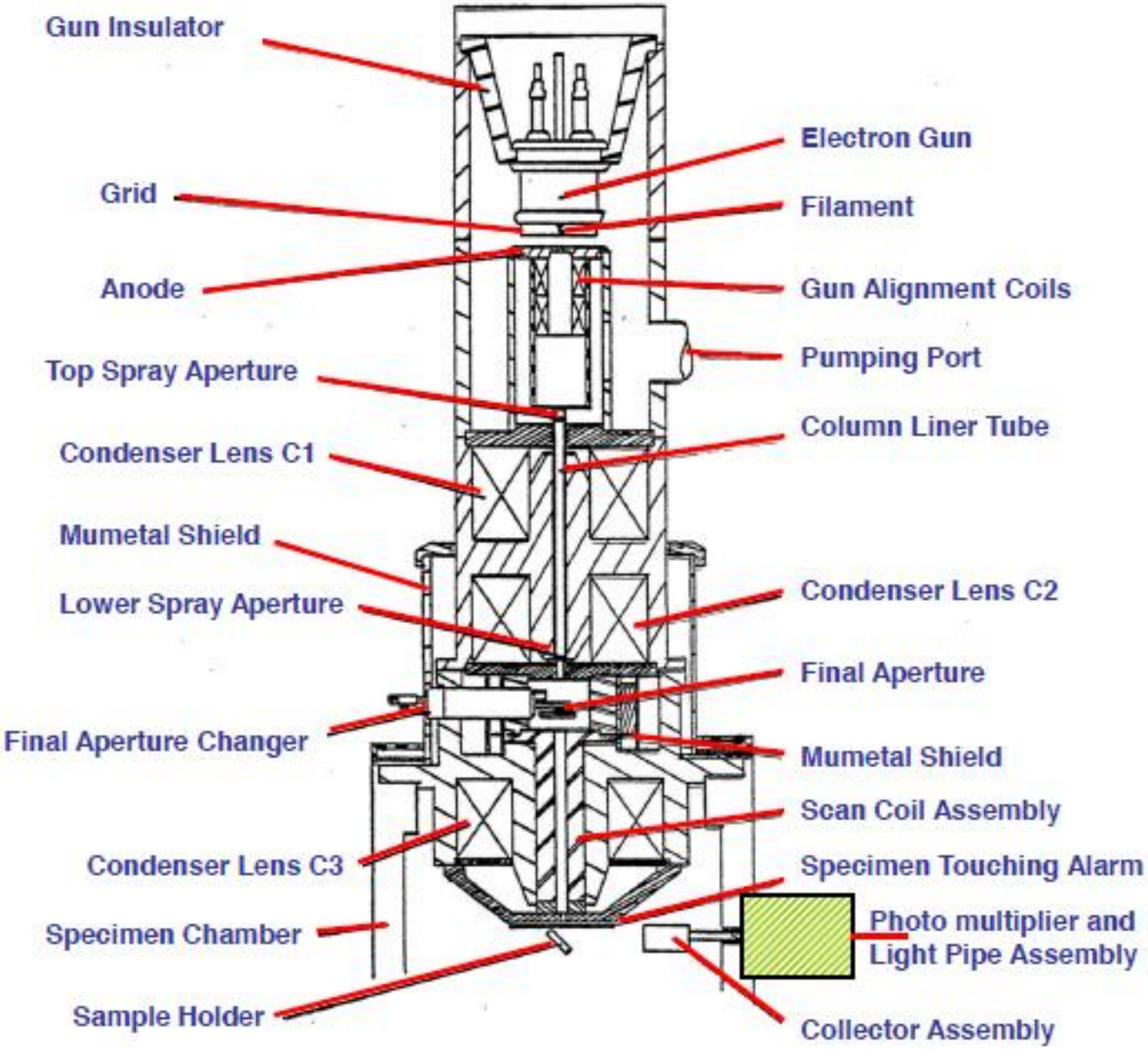
- Electron gun assembly -
  This produces a stable source of primary electron beam.
- Electromagnetic lenses and apertures -
  These focus the electron beam on the specimen.
- Vacuum system -
  This allows the passage of the electrons through the column without the interference of air molecules.
- Electron collector, signal detection and display components, and recording CRT – Imaging.
- Specimen Goniometer Stage -
  moving the sample under the electron beam.

The instrument in brief



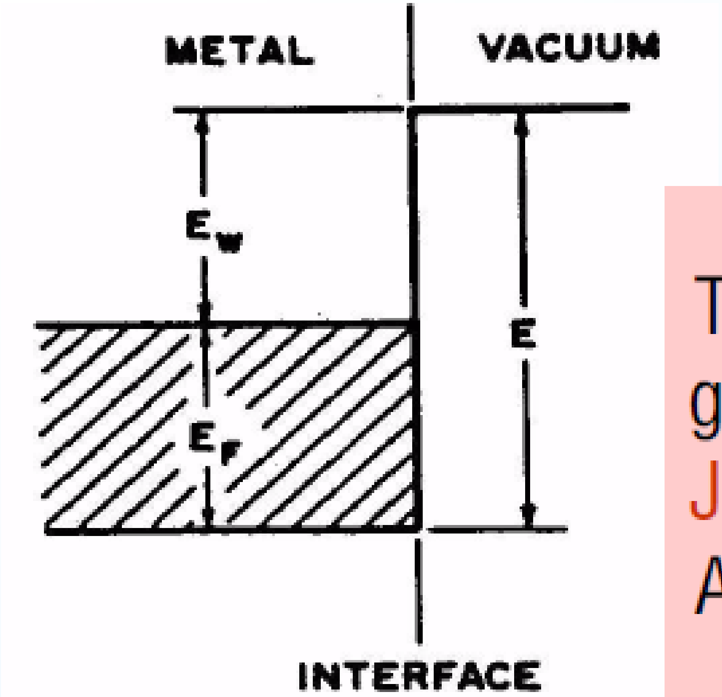
- Electron microscope follows the same ideas of optical microscope, but uses electrons instead of light;
- “Lens” here are not the optical materials (like glass), but electrical field.

Electron Optical Column



Concept of Work Function

Energy Model of Thermionic Emission



$E_W = E - E_F$

The emission current density is given by Richardson equation

$J_c = A_c T^2 \exp(-E_W/kT)$ A/cm^2

A_c - (A/cm^{2}K^2); T - (K)

At sufficiently high temperatures, a certain % of the electrons become sufficiently energetic to overcome the work function (E_W) of the cathode material and escape the source.

Electron Guns

- We want many electrons per time unit per area (high current density) and as small electron spot as possible
- Traditional guns: thermionic electron gun (electrons are emitted when a solid is heated)
  - W-wire, LaB_6-crystal
- Modern: field emission guns (FEG) (cold guns, a strong electric field is used to extract electrons)
  - Single crystal of W, etched to a thin tip

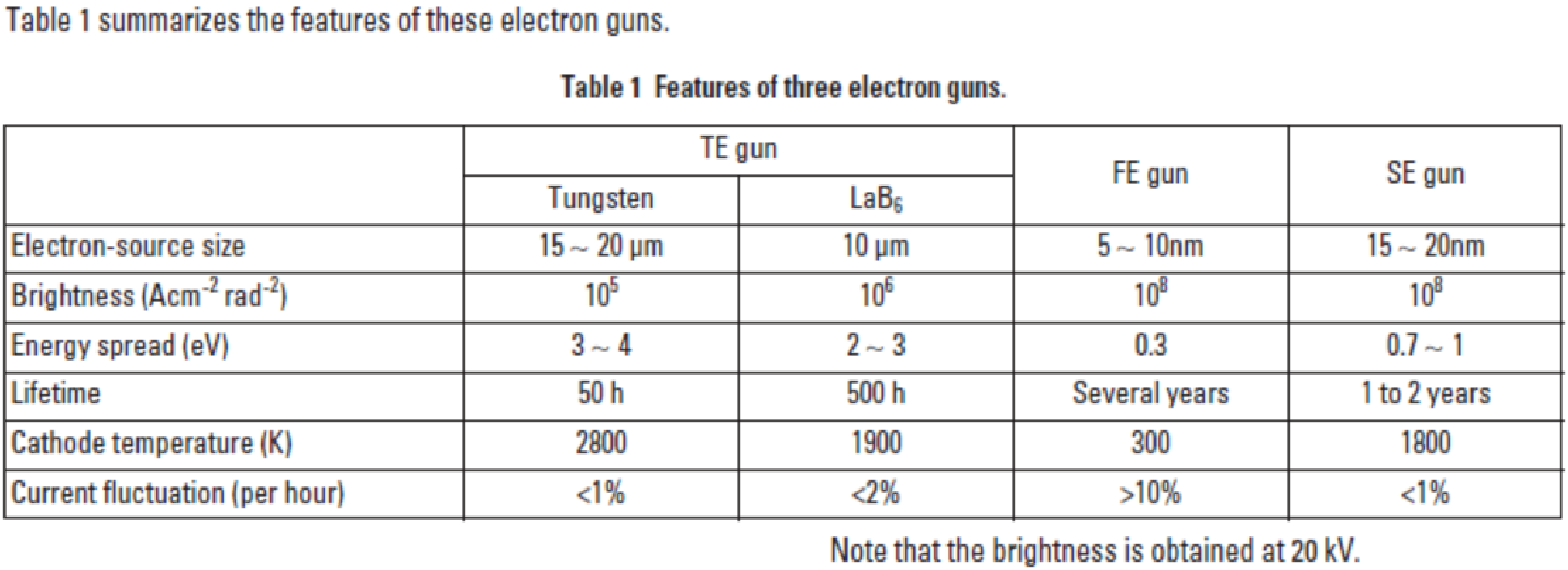


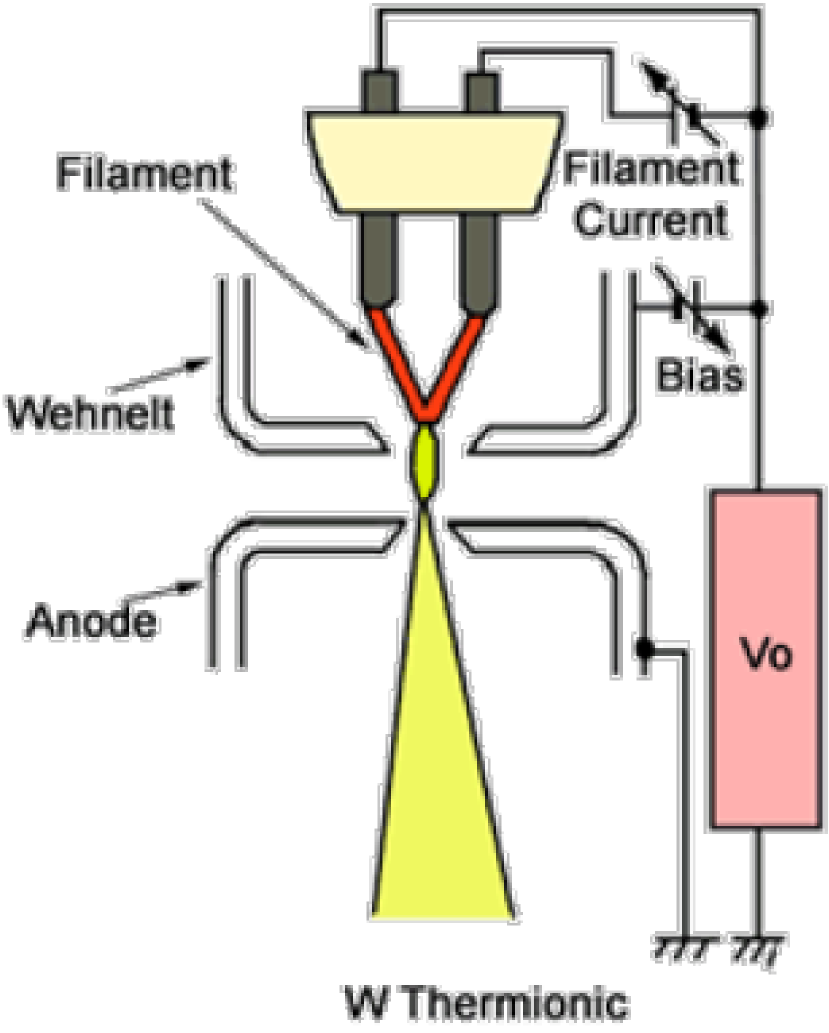
Table 1 summarizes the features of these electron guns.

Table 1 Features of three electron guns.

	TE gun		FE gun	SE gun
	Tungsten	LaB_6		
Electron-source size	15 ~ 20 µm	10 µm	5 ~ 10nm	15 ~ 20nm
Brightness ($Acm^{-2}rad^{-2}$)	10^5	10^6	10^8	10^8
Energy spread (eV)	3 ~ 4	2 ~ 3	0.3	0.7 ~ 1
Lifetime	50 h	500 h	Several years	1 to 2 years
Cathode temperature (K)	2800	1900	300	1800
Current fluctuation (per hour)	<1%	<2%	>10%	<1%

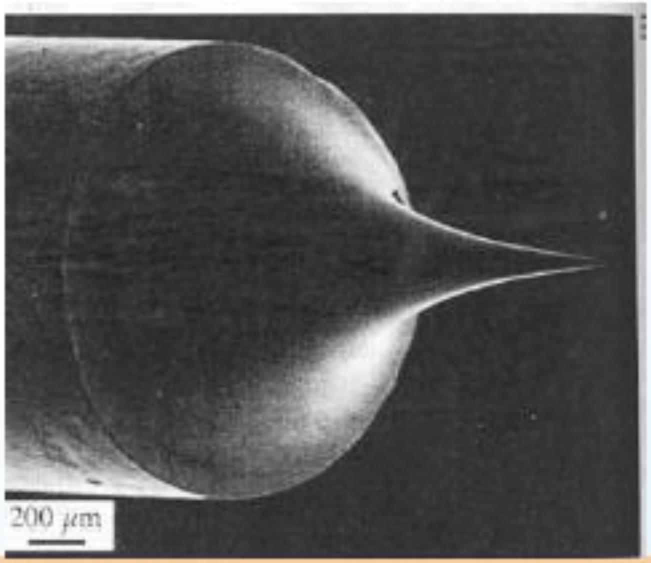
Note that the brightness is obtained at 20 kV.

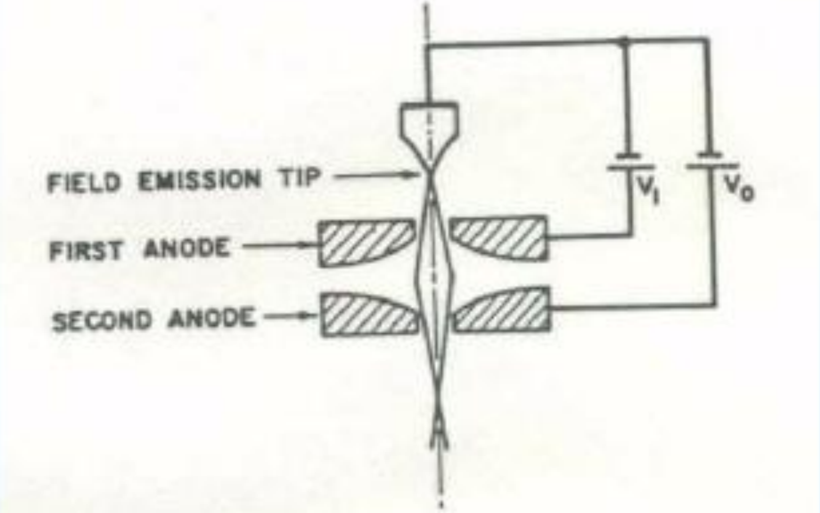
Thermionic Emission Electron Source



The most common emitter material for the electron beam is tungsten (W) or LaB6. The emitter is located at the top of the SEM column in the Gun assembly. The filament sits inside a housing called the Wehnelt cap. A potential is created between the sample and the gun and when the emitter is heated to a certain temperature electrons will begin streaming from the tip of the emitter and travel down the column. The electron beam is focused and defocused by the use of electromagnetic lenses.

Field Emission Gun



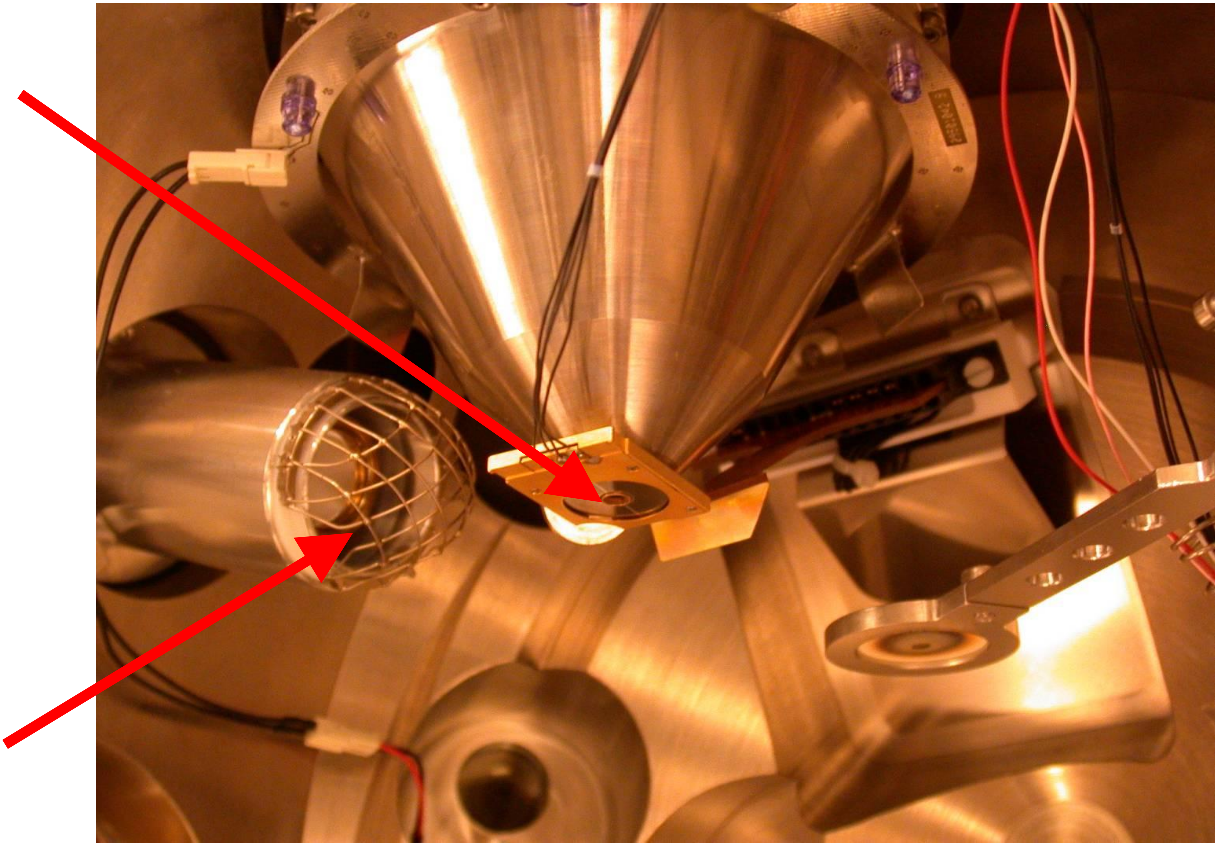


The effective source or crossover size (d_o) of a field emitter is approx. 10 nm, as compared to LaB_6 (10 μm), and W (50 μm).

No further demagnifying lenses are needed to produce an electron probe suitable for high resolution SEM.

Detectors

Our traditional detectors



Backscattered electron detector:
(Solid-State Detector)

Secondary electron detector:
(Everhart-Thornley)

- Secondary electrons: Everhart-Thornley Detector
- Backscattered electrons: Solid State Detector
- X-rays: Energy dispersive spectrometer (EDS)

Tuning Electron Current

In SEM, a focused beam of high energy electrons scans across the surface of the specimen.

The amount of current in the focused e-beam impinging on a specimen, determines the magnitude of the signals emitted.

The size of the final probe or beam determines the best possible resolution for many of the measured signals.

The electron optical system in SEM is designed so that maximum possible current is obtained in the smallest possible electron probe.

SE & BSE images

Secondary electron (SE) images

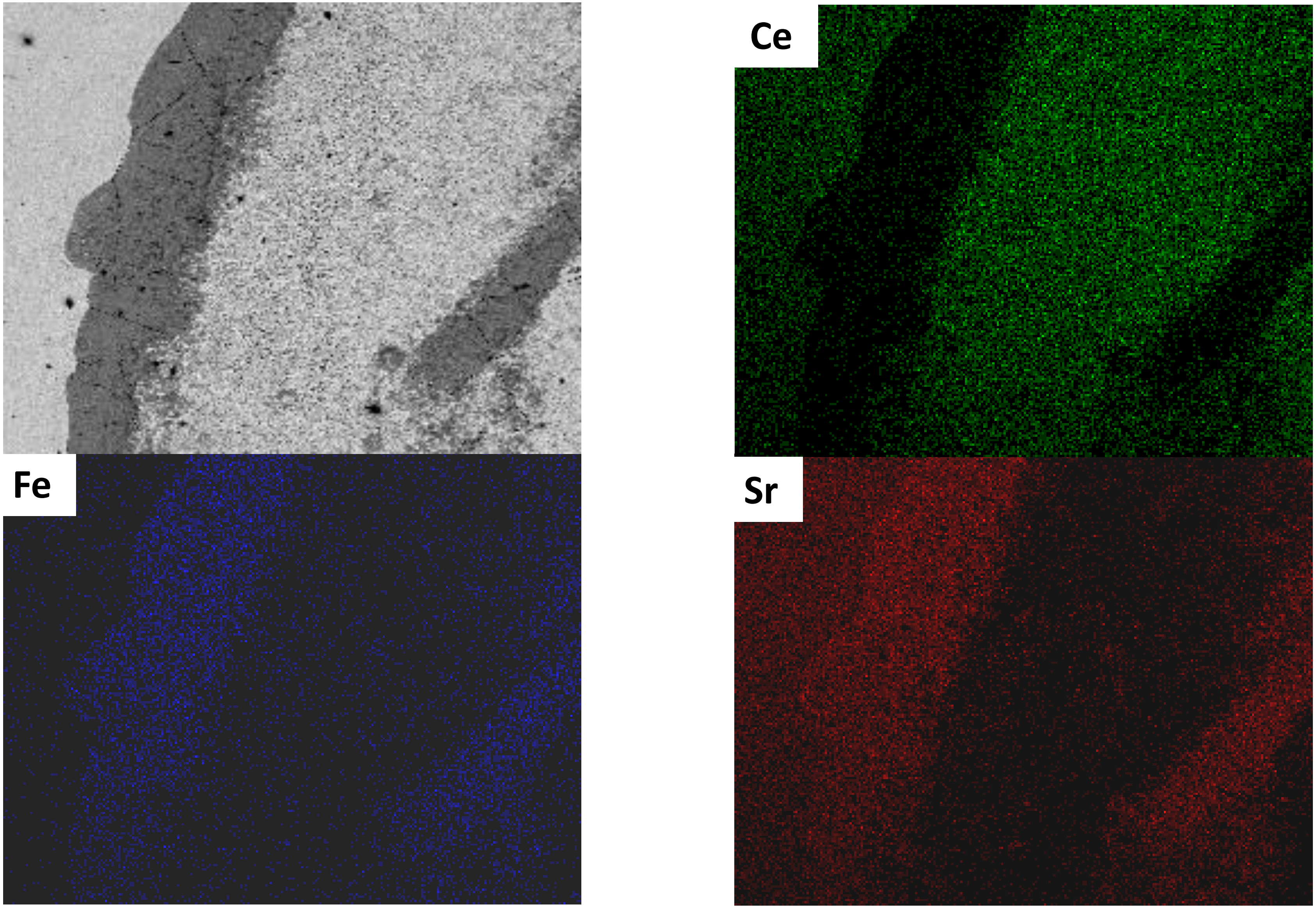
Secondary electrons are low energy electrons formed by inelastic scattering and have energy of less than 50eV. The low energy of these electrons allows them to be collected easily. This is achieved by placing a positively biased grill on the front of the SE detector, which is positioned off to one side of the specimen. The positive grill attracts the negative electrons and they go through it into the detector. This is the case for the Everhart-Thornley detector which is most commonly used but there is another kind of In-lens SE detector in some machines. The major influence on SE signal-generation is the shape (topography) of the specimen surface

Backscattered electron (BSE) images

Backscattered (BS) electrons are high-energy electrons (>50 eV) from the primary incident beam that are ejected back out from the sample. These BSE are used to produce a different kind of image. Such an image uses contrast to tell us about the average atomic number of the sample. For example, a grain of sand that is made up of a titanium mineral looks whiter than a grain made of a silicon material (Ti versus Si). There is a difference in contrast between the grains labelled Si and Ti whereas in images taken using secondary electrons, there is no difference in contrast between these grains. The sample is a mixture of mineral sand.

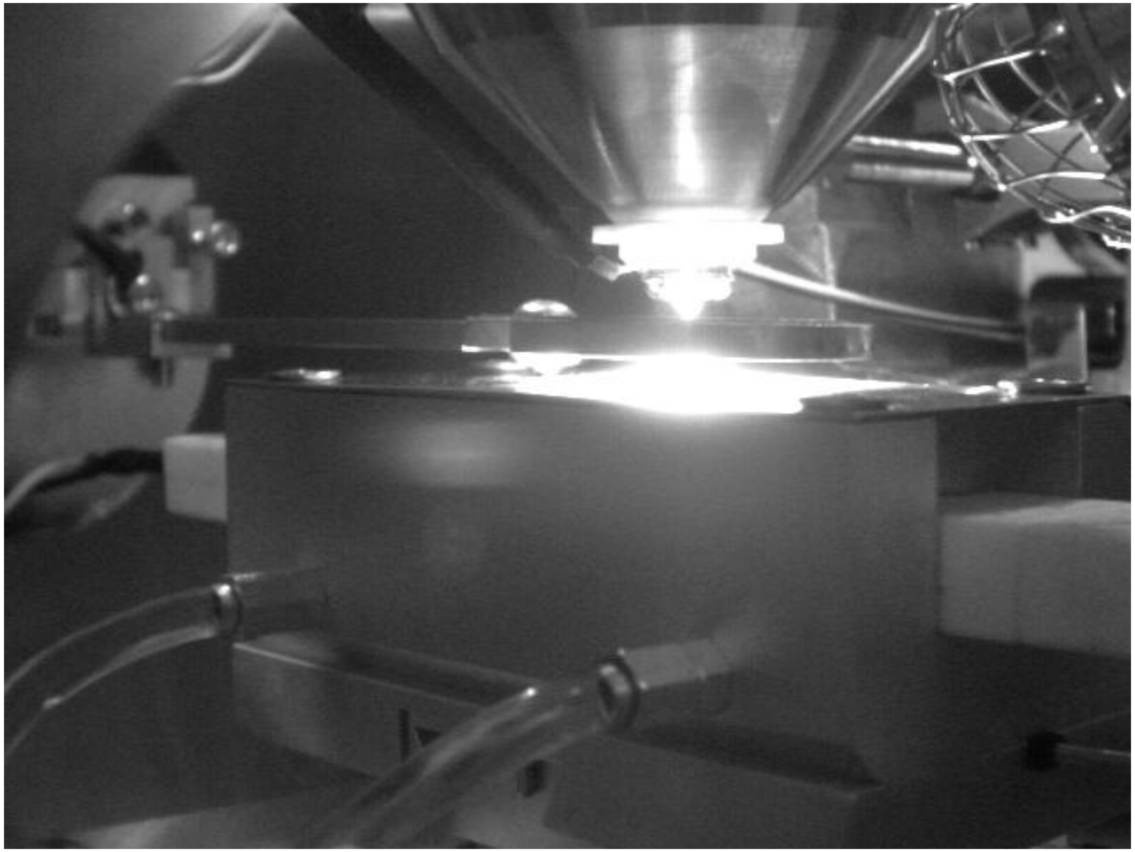
Chemistry

Images: Harald Fjeld, UiO



In-situ imaging

- A modern SEM can be equipped with various accessories, e.g. a hot stage



Operating parameters affecting signal quality

- Accelerating Voltage
- Probe Current
- Working Distance
- Specimen Tilt
- Aperture Size

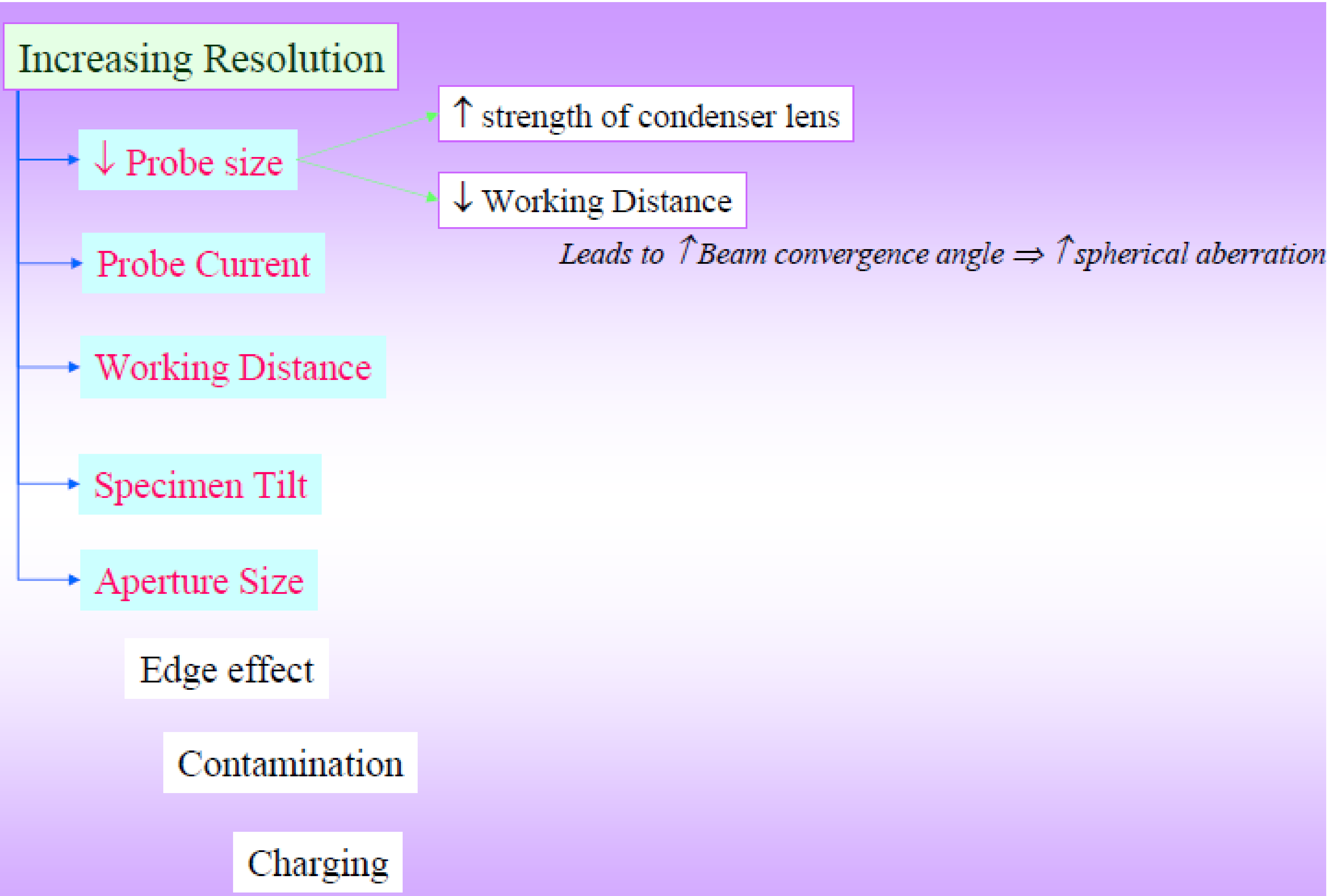
Edge effect

Contamination

Charging

Operating Parameter		Values
Gun voltage		~20 keV
Working distance		~26 mm
Probe size	W filament	~30 Å
	LaB_6	
	Field Emission	
Vacuum	W filament	10^{-5} Torr
	LaB_6	10^{-8} Torr
	Field Emission	10^{-10} Torr

- Probe current ↓ ➢ Probe diameter ↓ ➢ Resolution ↑
- This leads to decrease in image intensity ⇒ we have to use a brighter source *(W filament <* LaB_6 *< Field Emission gun)*



Increasing Resolution

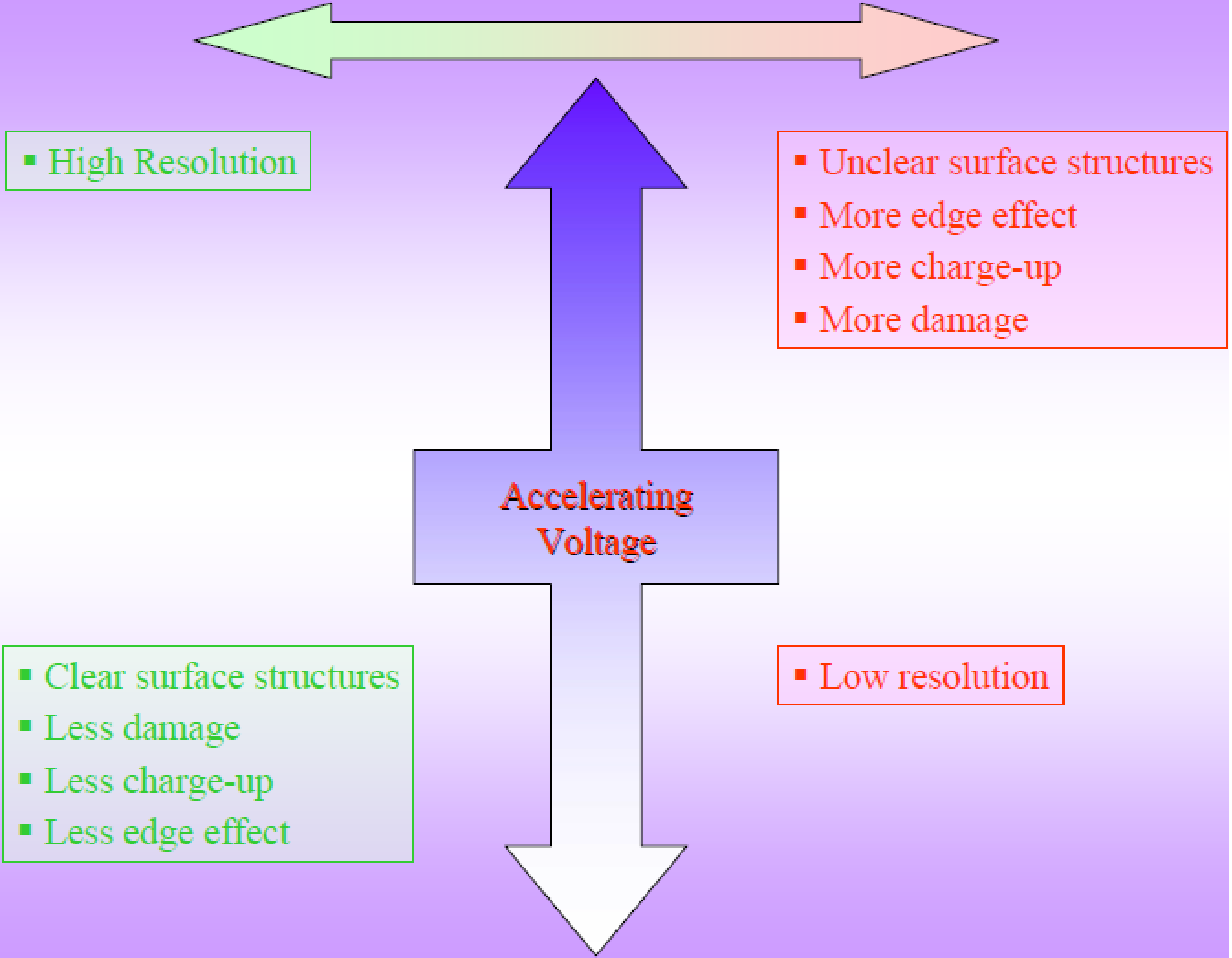
- ↓ Probe size
  - ↑ strength of condenser lens
  - ↓ Working Distance
- Probe Current
- Working Distance
- Specimen Tilt
- Aperture Size

Leads to ↑Beam convergence angle ⇒ ↑spherical aberration

Edge effect

Contamination

Charging



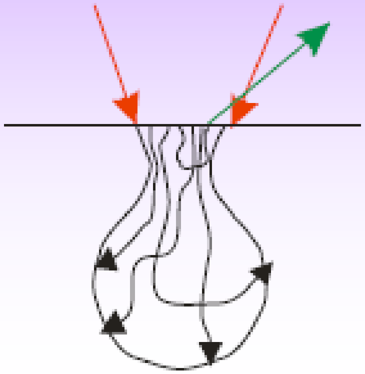
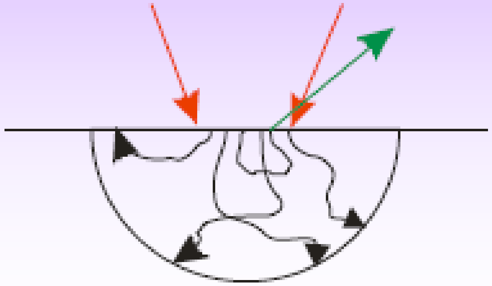
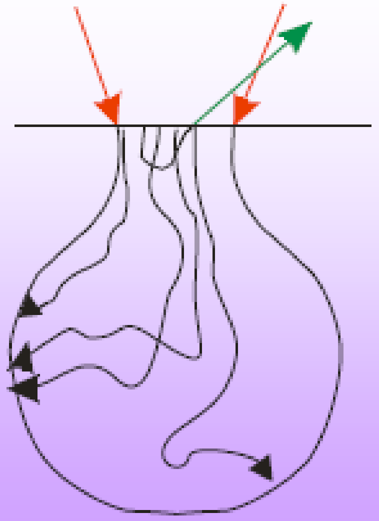
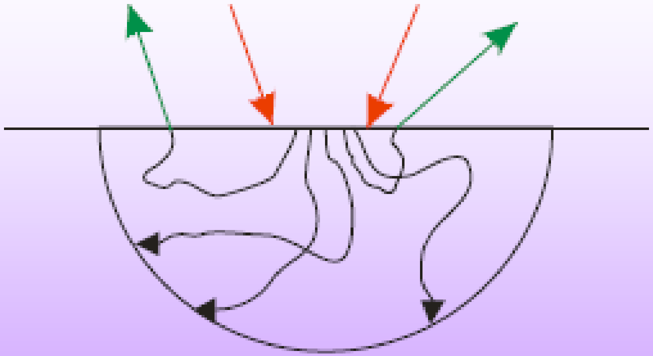
- High Resolution

- Unclear surface structures
- More edge effect
- More charge-up
- More damage

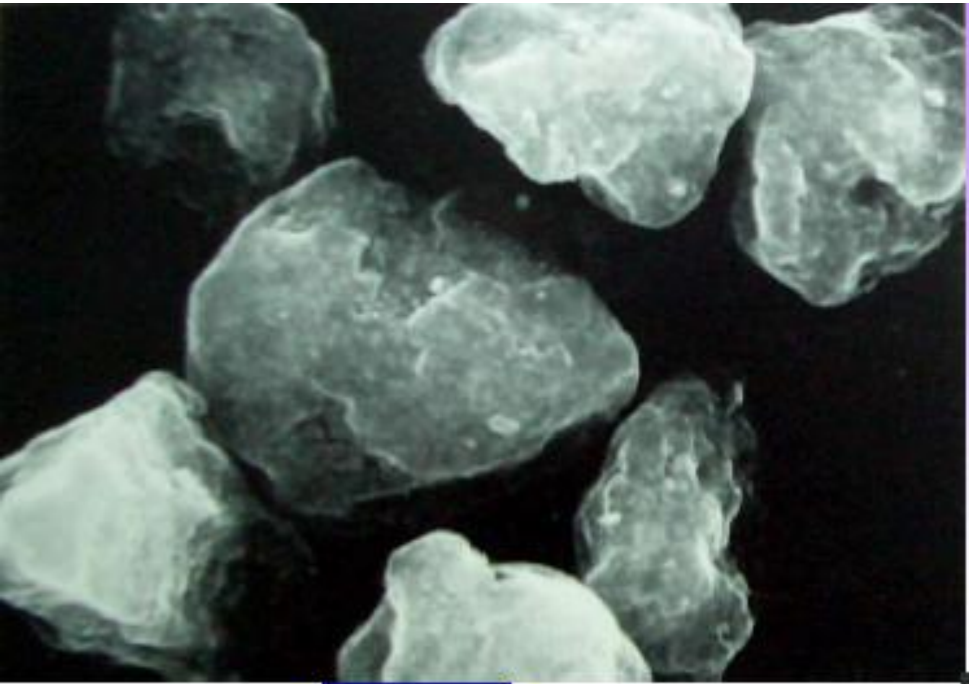
Accelerating Voltage

- Clear surface structures
- Less damage
- Less charge-up
- Less edge effect

- Low resolution

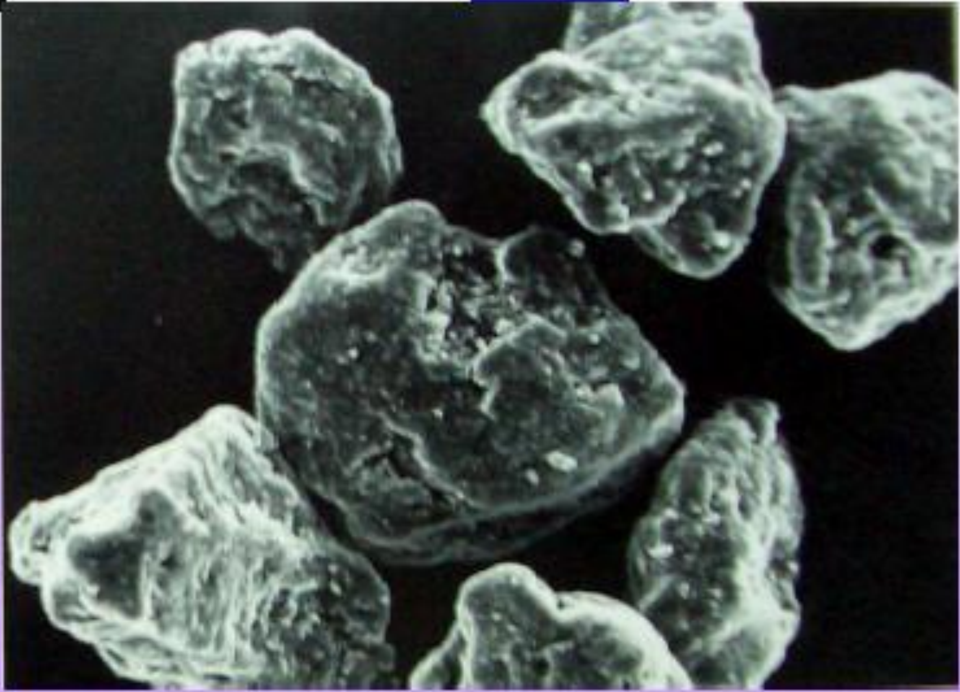
	Low atomic number	High atomic number
Low accelerating voltage		
High accelerating voltage		

2500 ×



30 kV

5 kV



Specimen: Toner

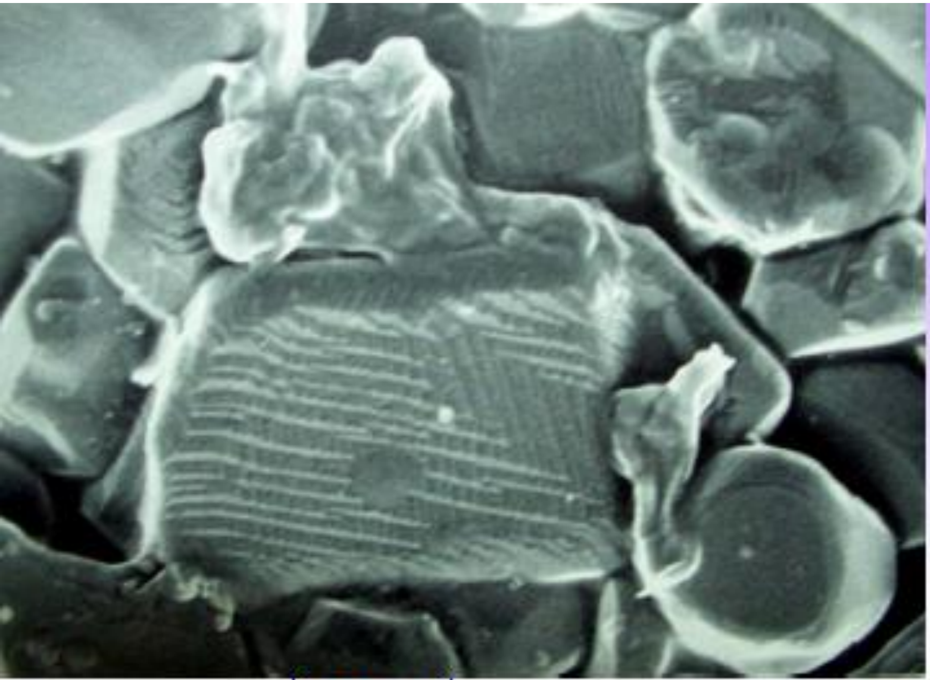
↑ Accelerating voltage

→ Increased contribution of BSE

- ➢ Low surface contrast
- ➢ Charging

Ref: SEM Manual, JEOL

7200 ×



5 kV

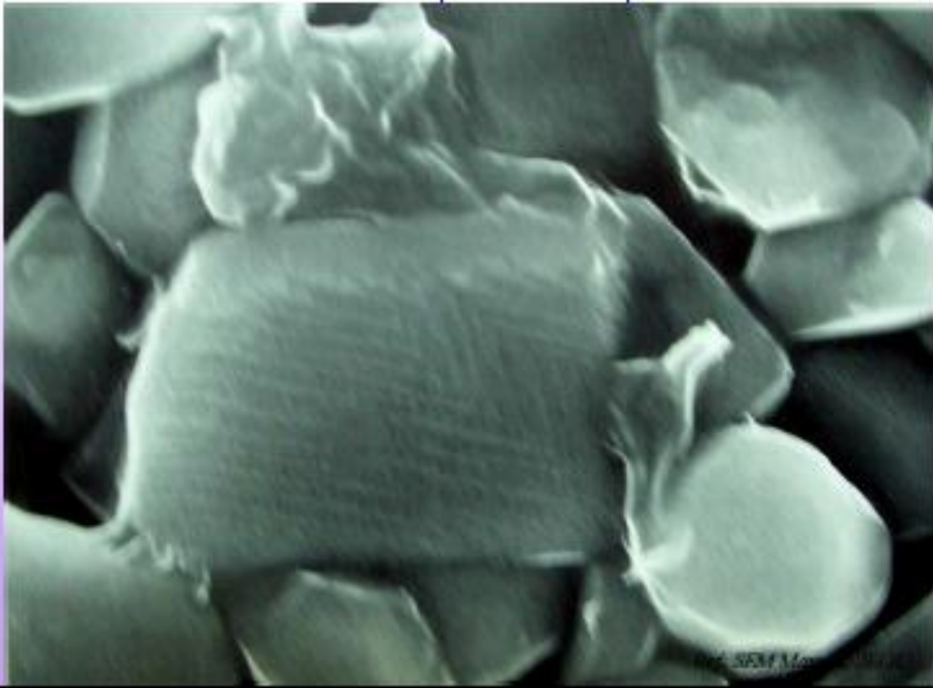
Specimen: Sintered powder

↓ Accelerating voltage

- Better surface contrast
- Not sharp at high magnifications

⇒ ↓ WD or ↓ probe diameter

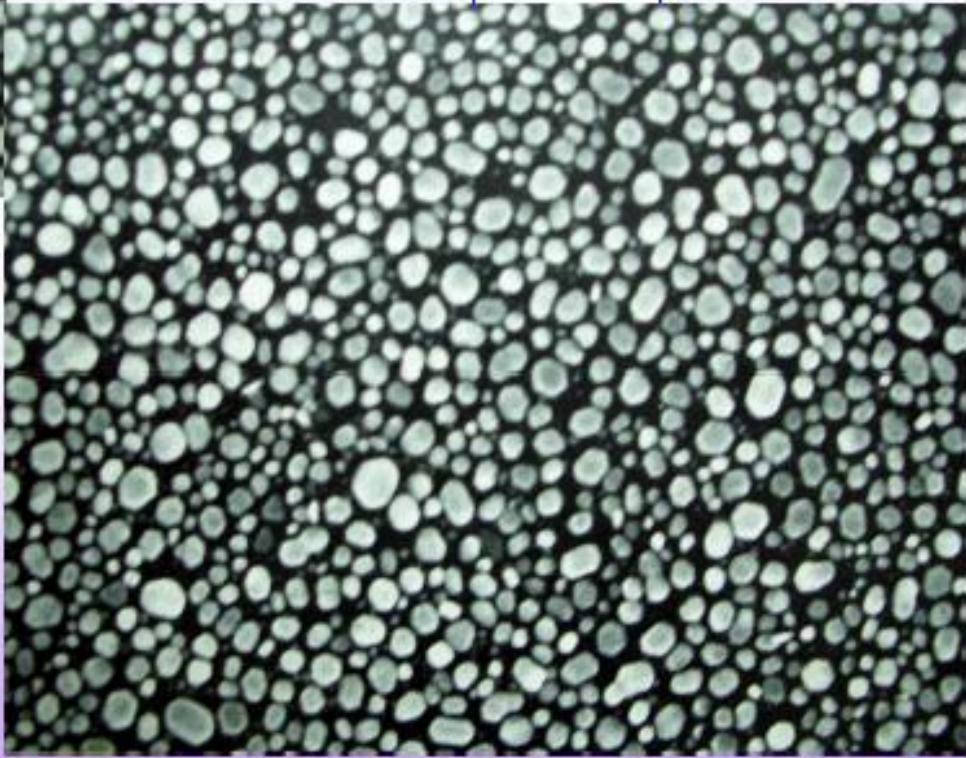
25 kV



36000 ×

5 kV

25 kV



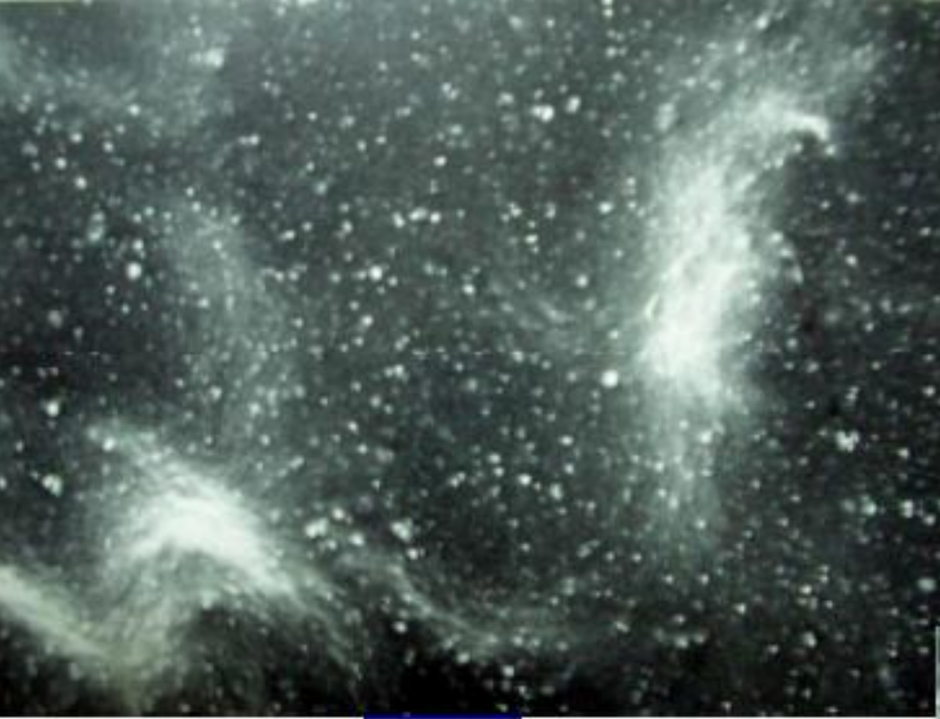
Specimen: Evaporated Au particles

↑ Accelerating voltage

- Better image sharpness
- Improved resolution

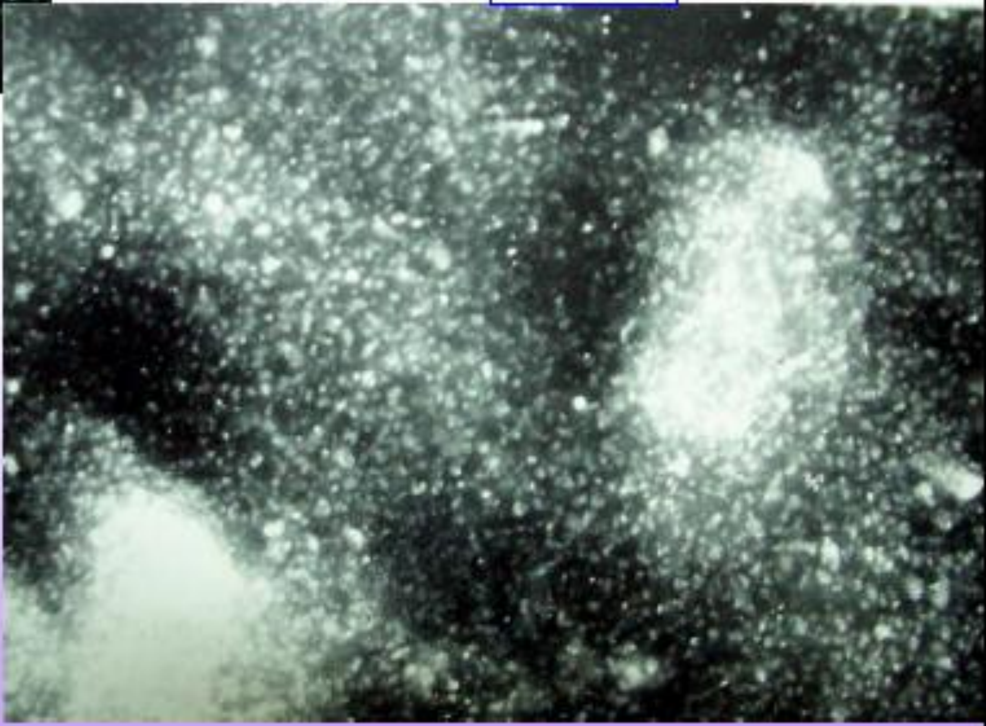
Ref: SEM Manual, JEOL

2500 ×



5 kV

25 kV



Specimen: Paint coat

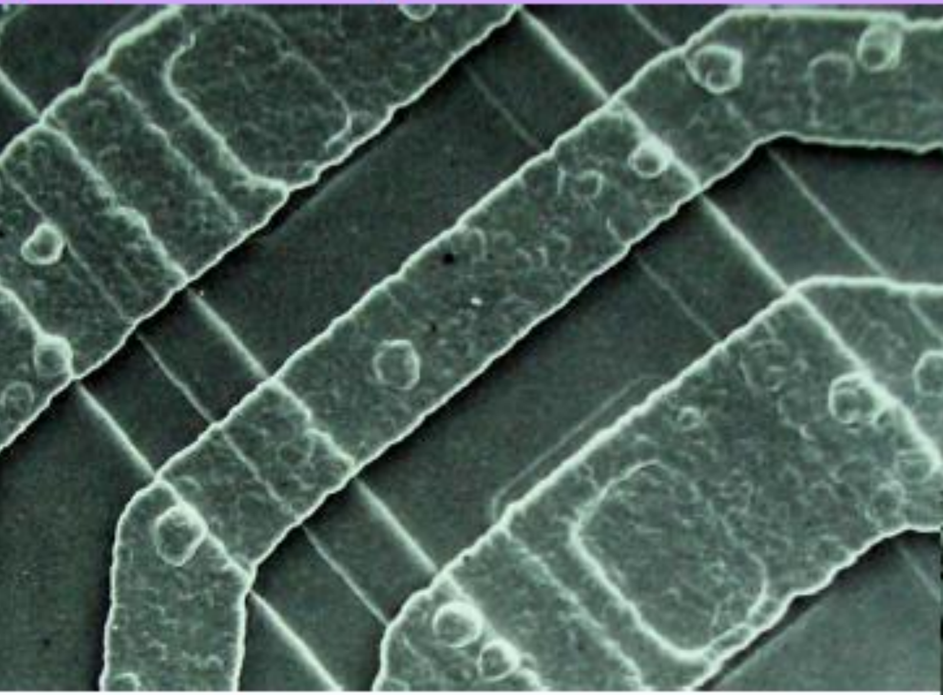
↑ Accelerating voltage

- ➢ Low surface contrast
- ➢ More BSE ⇒ contributions from within the specimen

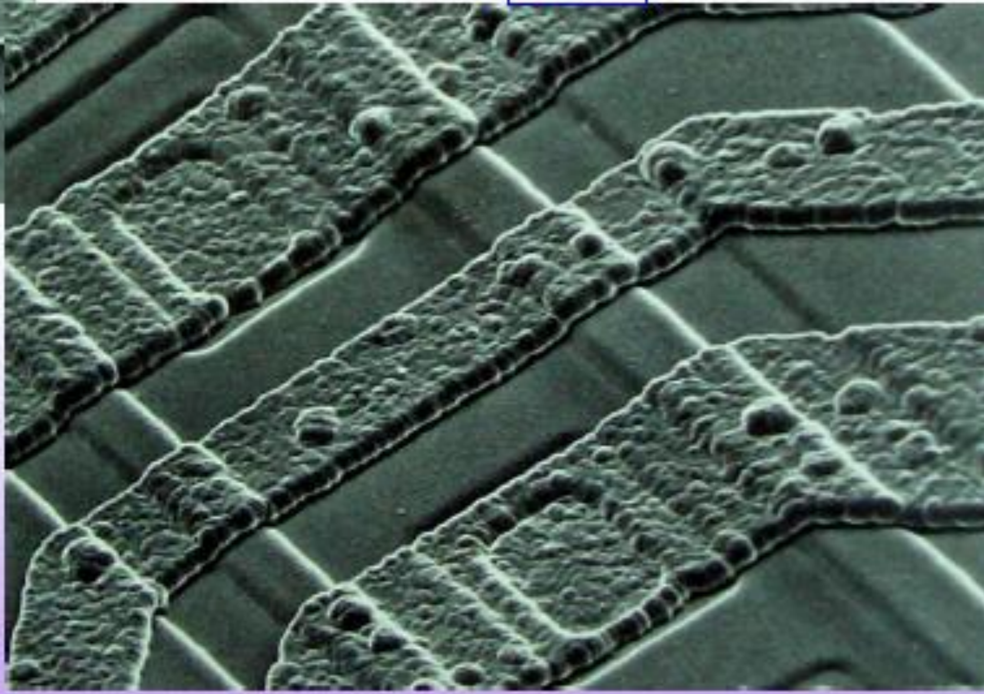
Ref: SEM Manual, JEOL

Specimen tilt

5kV, 1100 ×



0 °



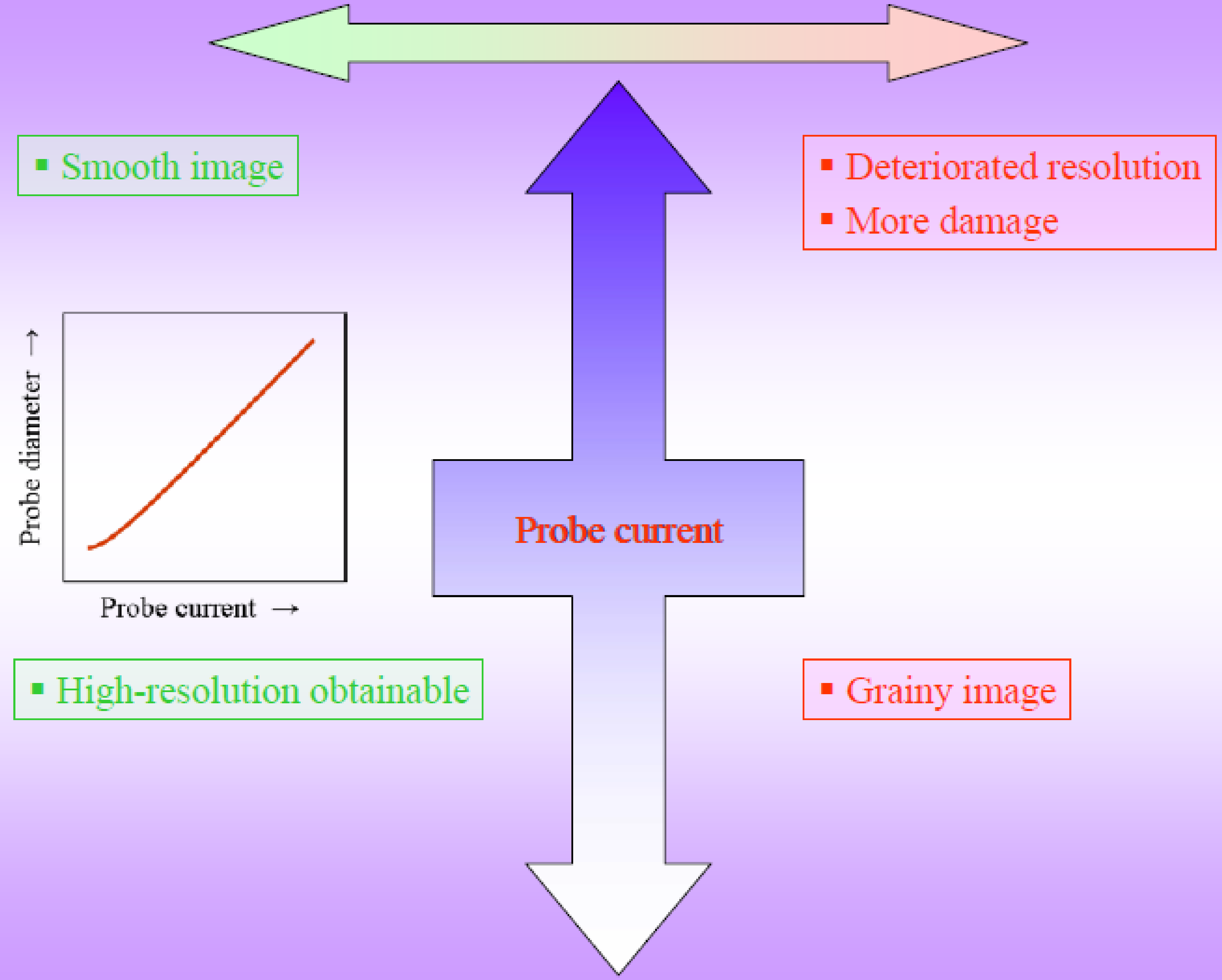
45 °

Specimen: IC chip

TILT

- Improve quality of SE images
- complete survey of topography
- Stereo images → images at 2 angles

Ref: SEM Manual, JEOL



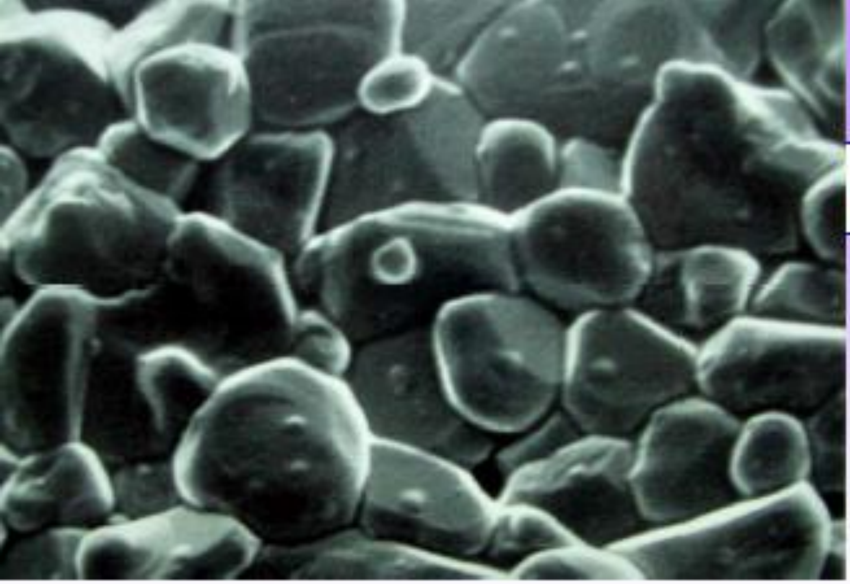
- Smooth image

- Deteriorated resolution
- More damage

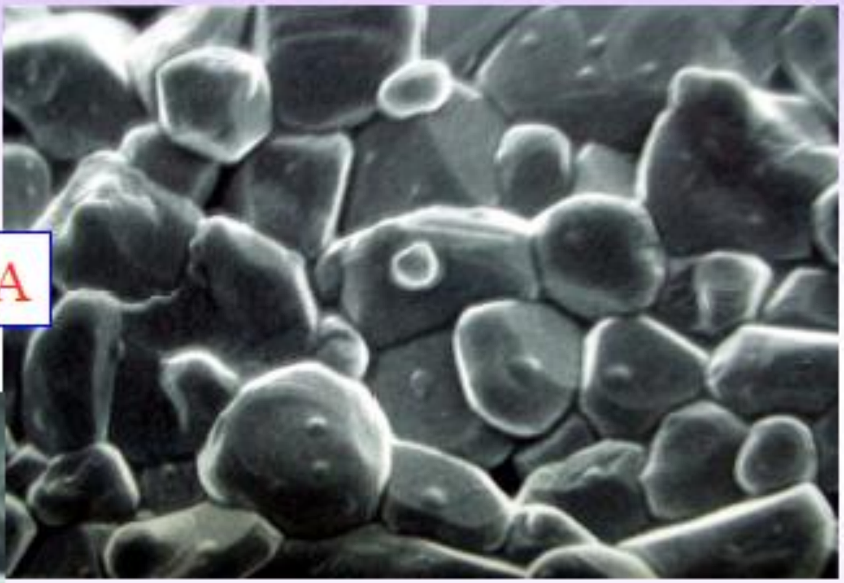
- High-resolution obtainable

- Grainy image

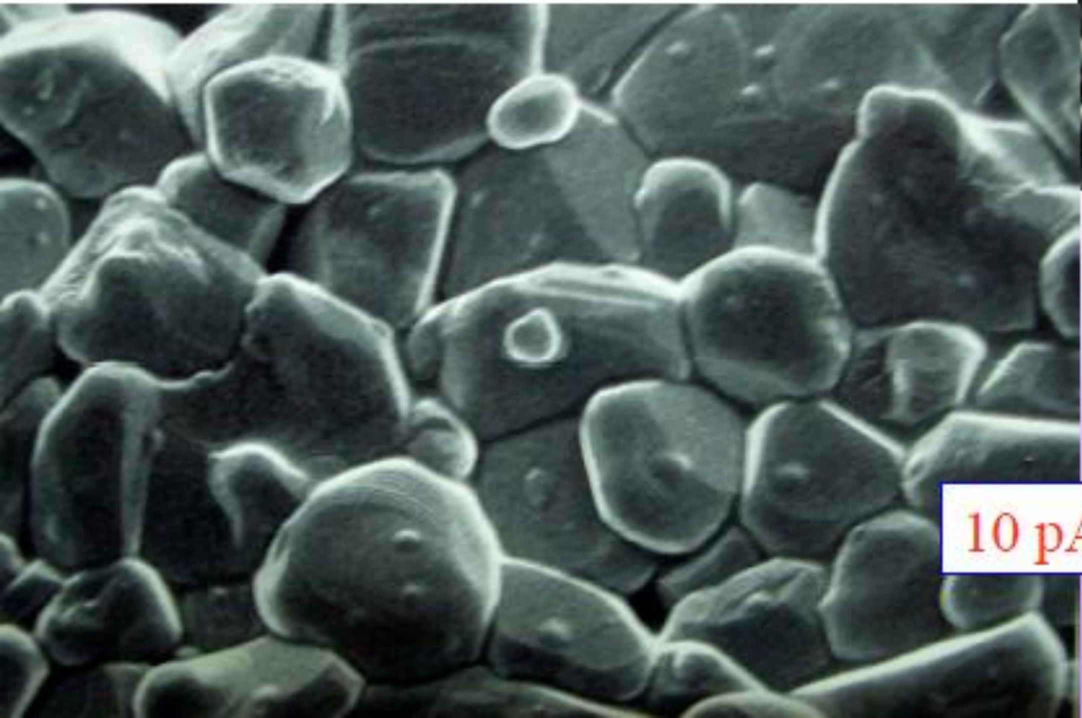
10 kV, 5400 ×



1 nA



0.1 nA



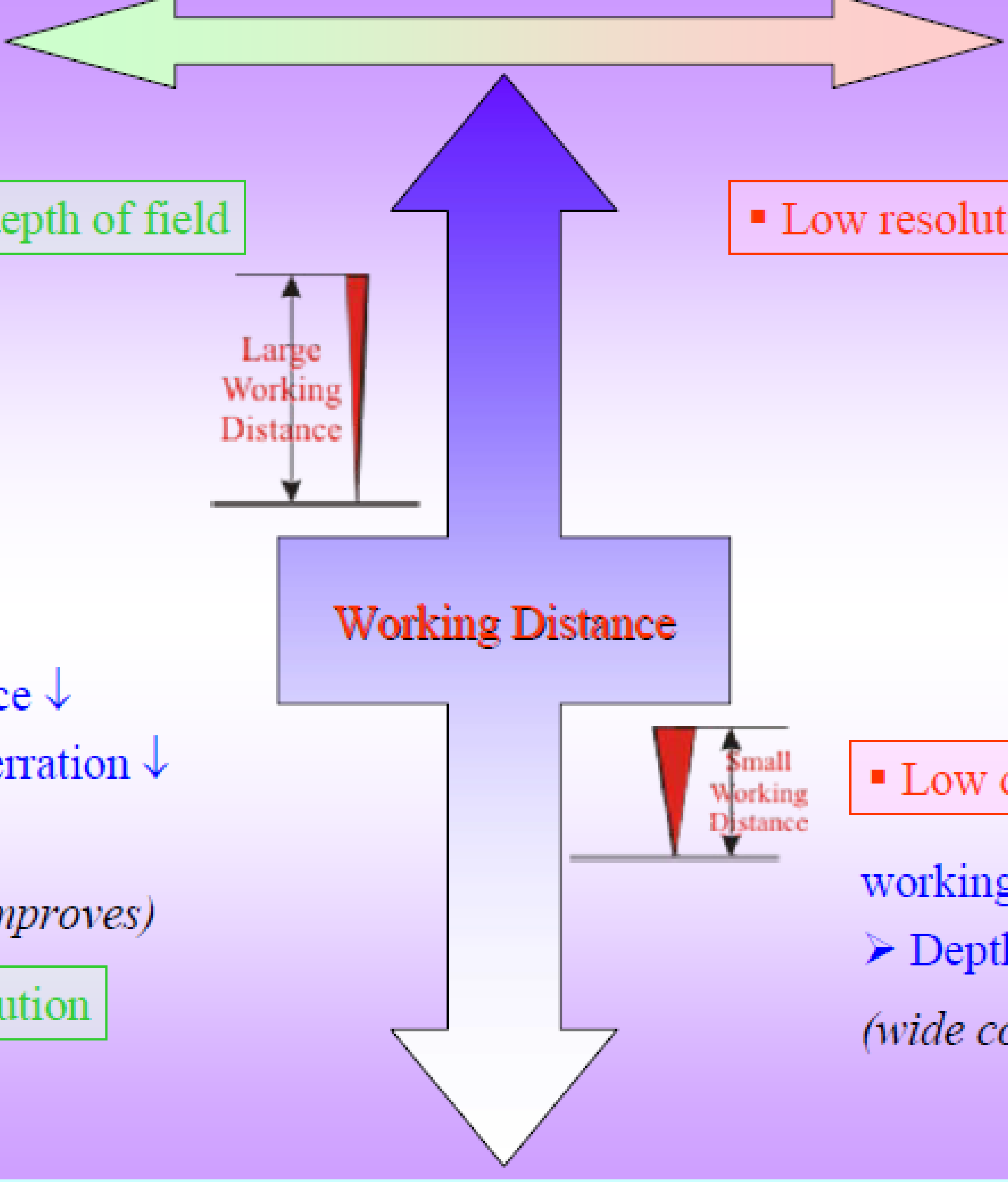
10 pA

Specimen: Ceramic

↓ Probe current

- ➢ ↑ image sharpness
- ➢ ↓ surface smoothness

Ref: SEM Manual, JEOL.



- Greater depth of field

- Low resolution

Working Distance

working distance ↓

➢ spherical aberration ↓

(spot size ↓

⇒ resolution improves)

- High resolution

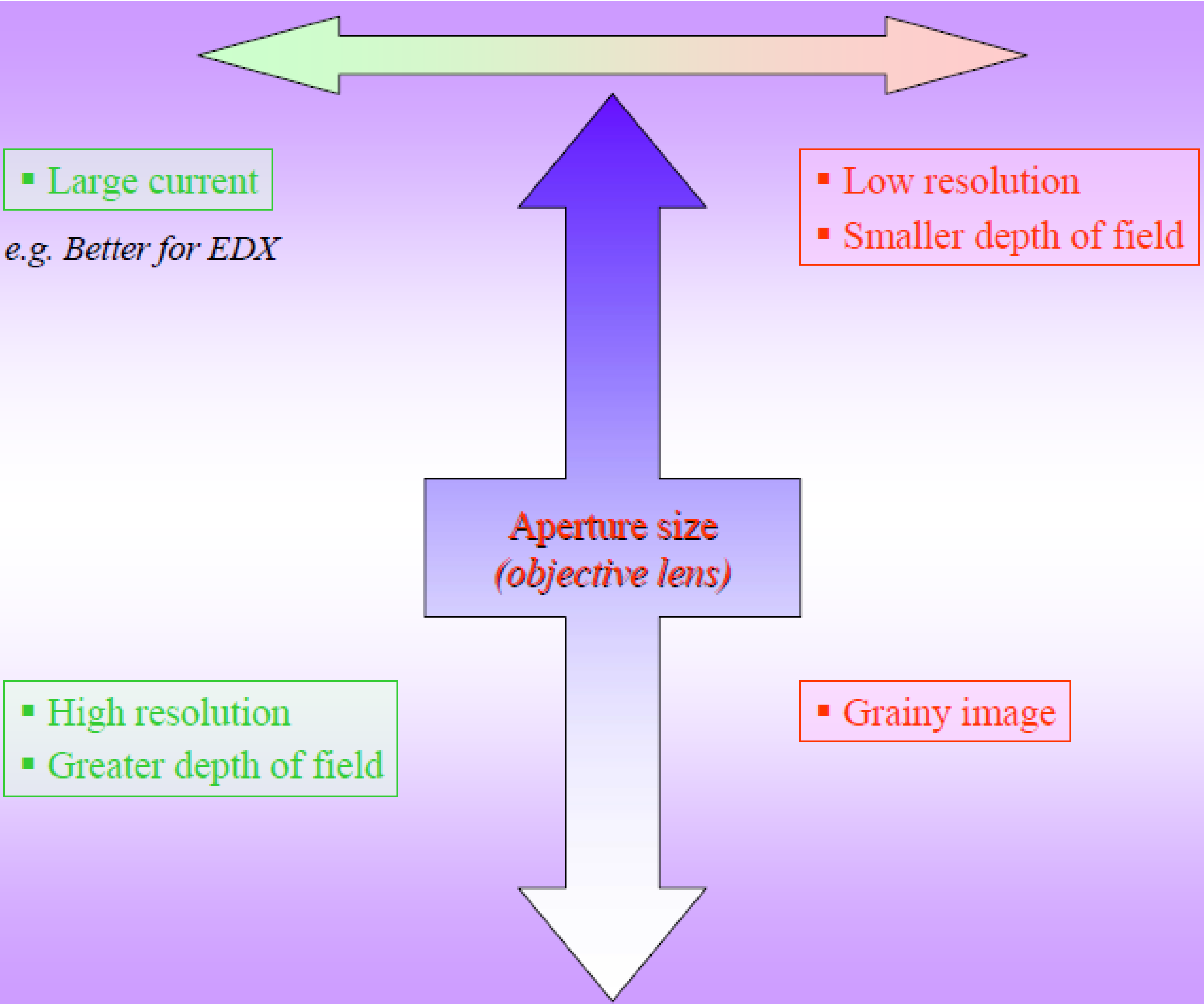
- Low depth of field

working distance ↓

➢ Depth of field ↓

(wide cone of electrons)

The working distance is the distance between the final condenser lens and the specimen



- Large current

e.g. Better for EDX

- Low resolution
- Smaller depth of field

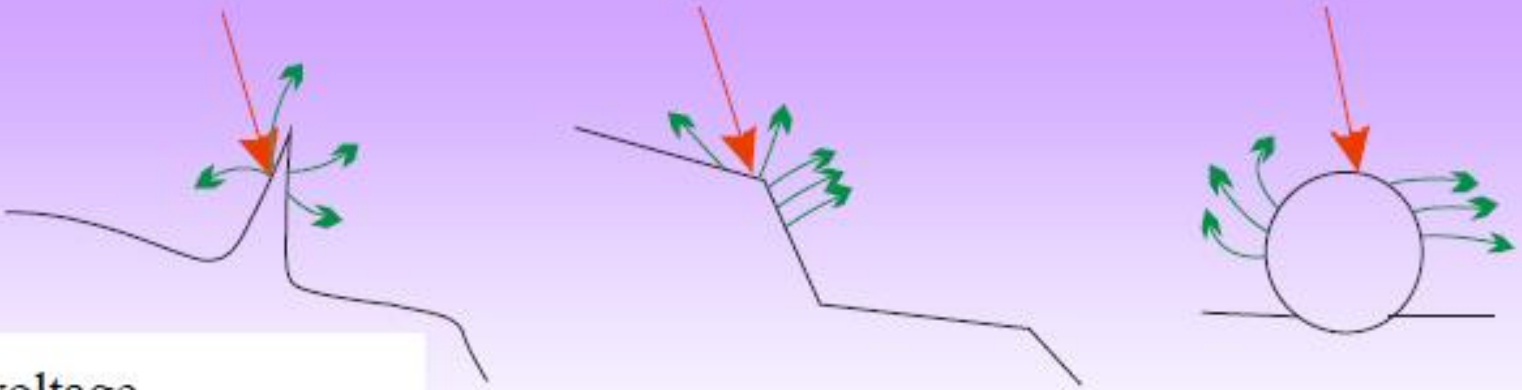
Aperture size
(objective lens)

- High resolution
- Greater depth of field

- Grainy image

Edge Effect

↑SE emission from protrusions and circumferences ⇒ appear bright

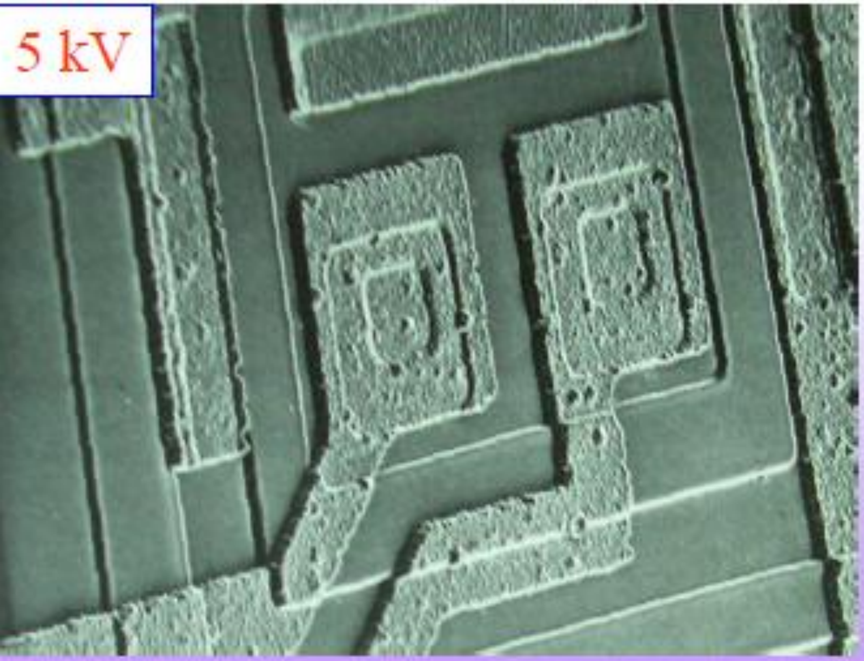


↑ Accelerating voltage

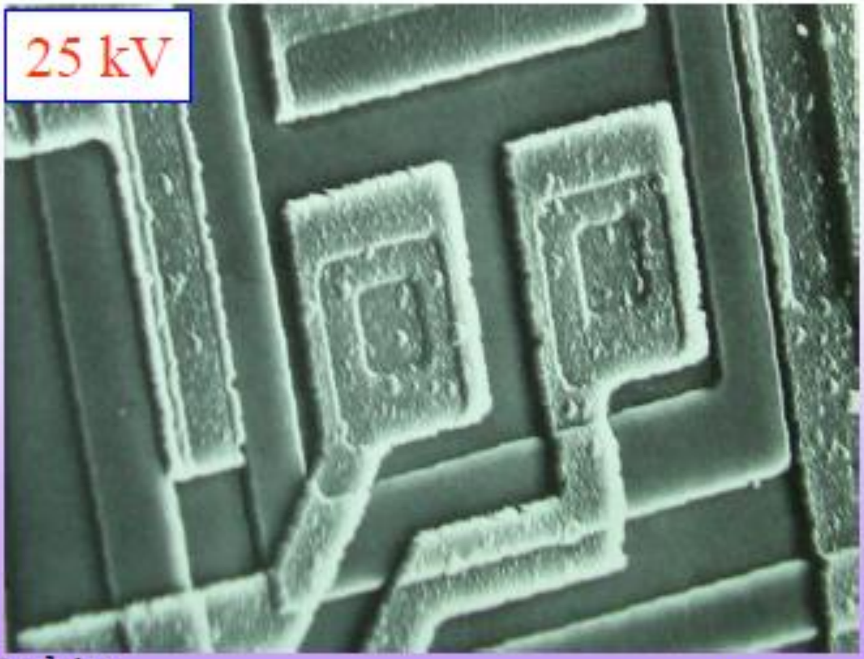
- ➢ Greater the edge effect

  (edges become brighter)

Tilt: 50°, 720 ×



5 kV



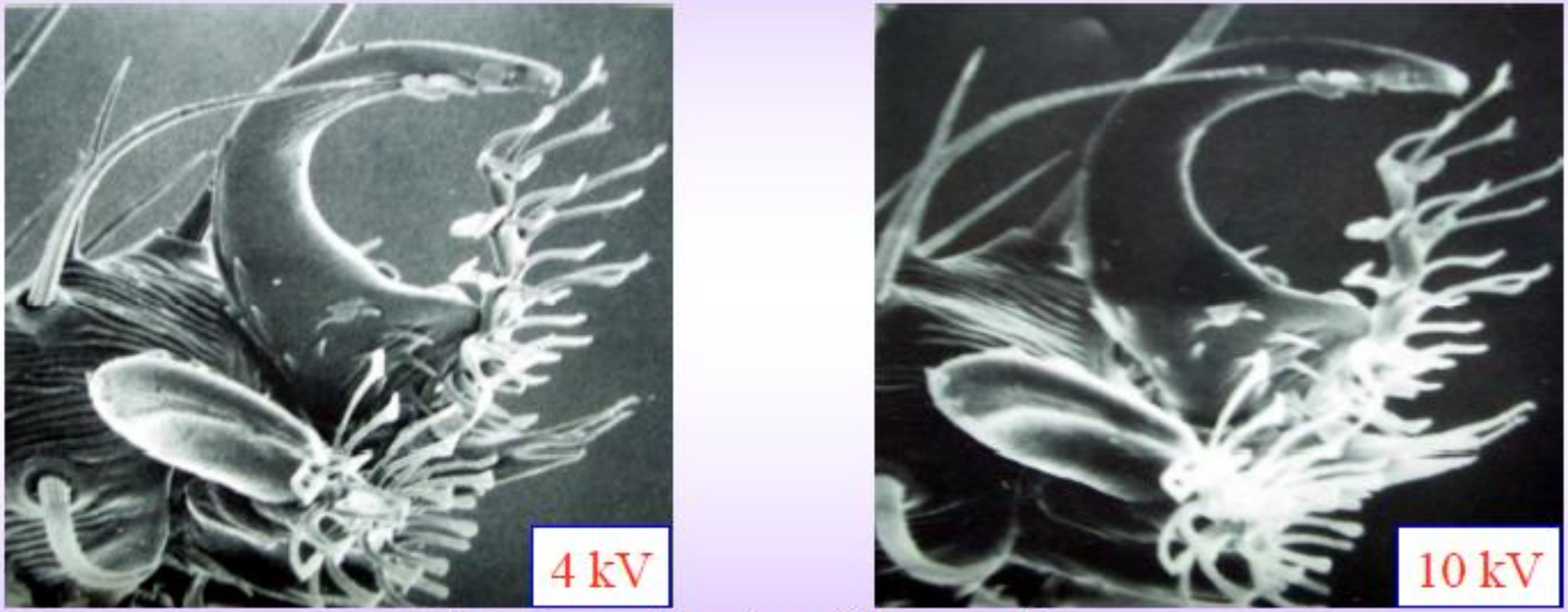
25 kV

Specimen: IC chip

Ref: SEM Manual, JEOL

Charging

- Due to low conductivity of sample
- Coating with a conducting material to avoid charging
- To ↓ charging → ↓ Voltage, ↓ probe current, tilt specimen



Specimen: Foreleg of vinegar fly

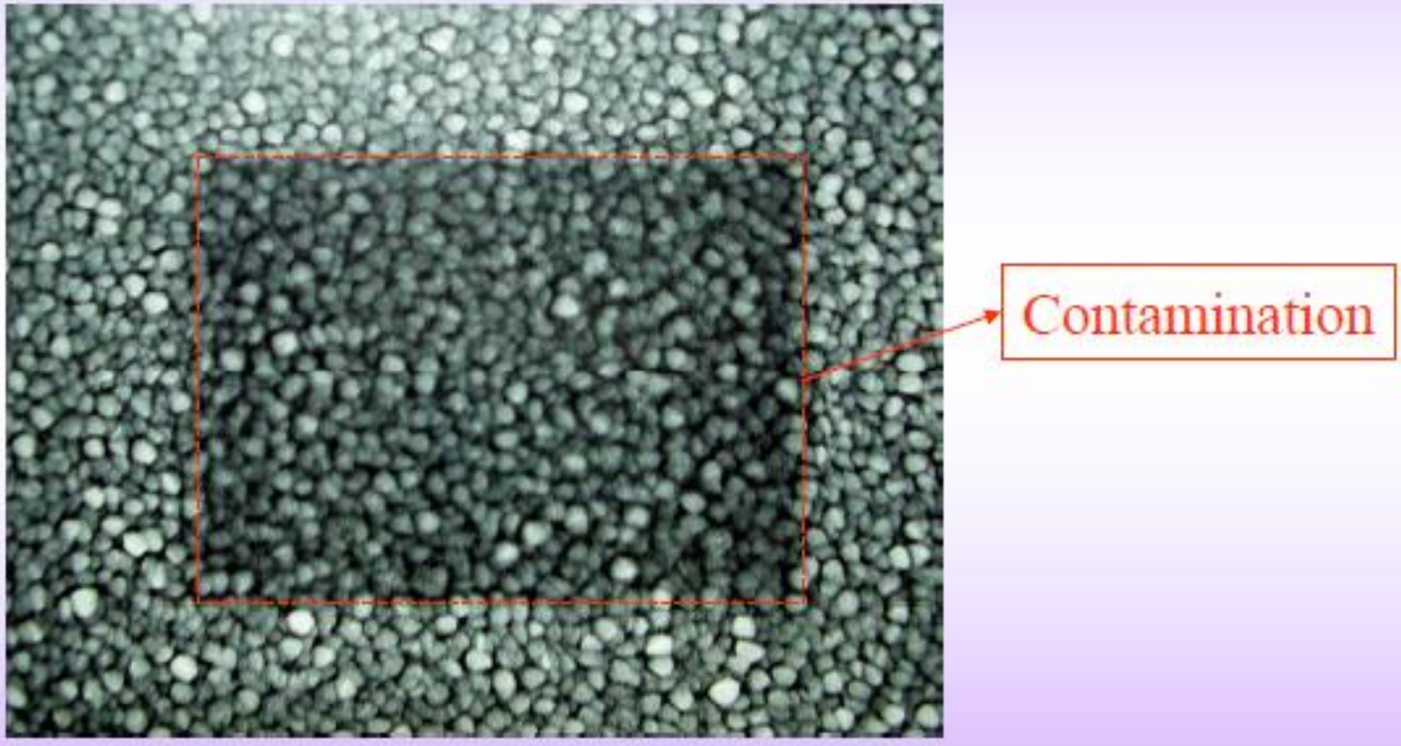
↑ Accelerating voltage

- ↑ Charging

Ref: SEM Manual, JEOL

Contamination

- Due to residual gas in the vicinity of the electron probe
- Leads to reduced contrast and loss in image sharpness
- Usually caused by scanning a small region for long time



5 kV

18000 ×

Specimen: ITO

Ref: SEM Manual, JEOL.