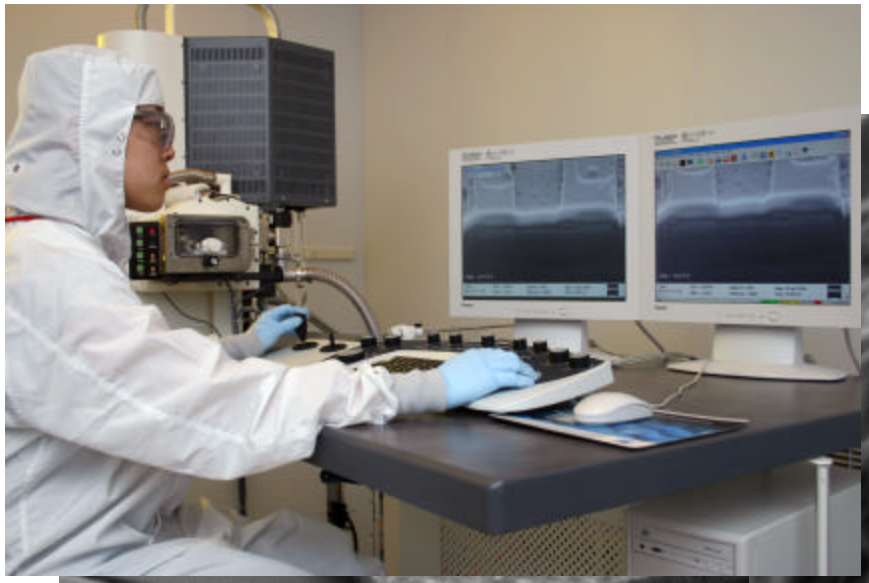
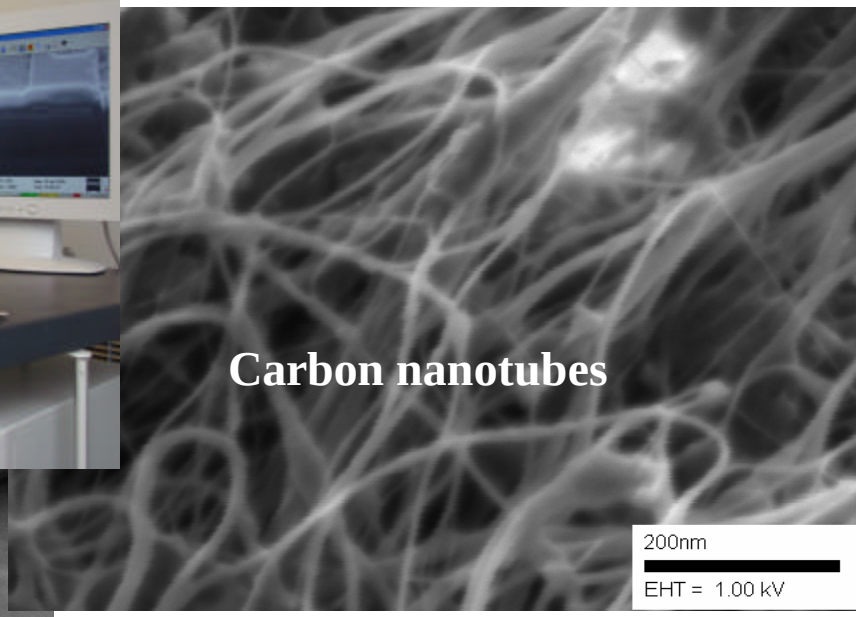
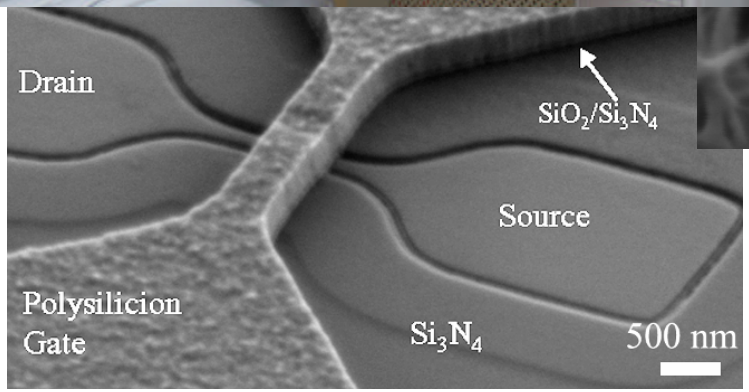
Basics of Scanning Electron Microscopy (SEM)





Above: ORNL



Above: Tiwari Group, Cornell

Outline

- Electron-Specimen Interactions
- SEM Equipment Overview
- Using an SEM
- Cheat Sheet
- References

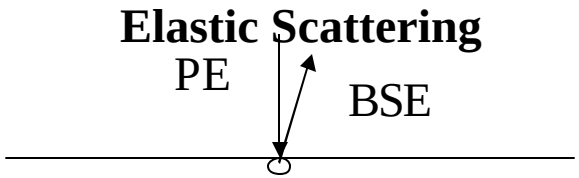
Electron Specimen Interactions

Electron Specimen Interactions

When a primary electron (PE) strikes a solid elastic and inelastic scattering can occur.

(a) Elastic Scattering

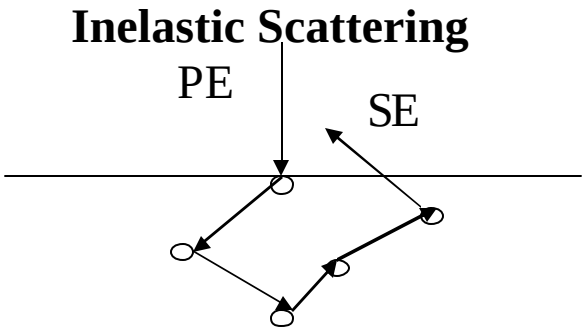
Electron scattered by interaction with atomic nucleus
Direction of beam electron changed, but velocity essentially the same
F_e = 0 to 180°
Causes emission of backscattered electrons (BSEs) or low loss electrons (LLEs)



Minimal energy loss occurs giving the BSE an energy close to the PE

(b) Inelastic Scattering

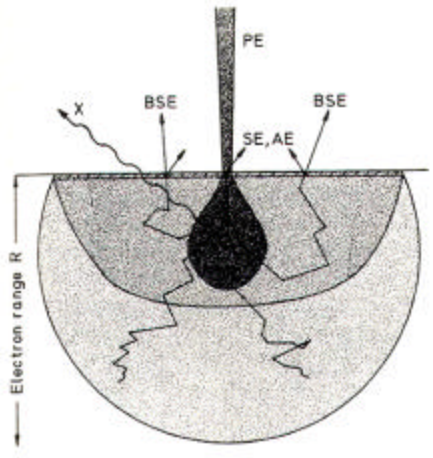
Energy transferred to tightly bound inner shell electrons or loosely bound outer-shell electrons
Kinetic energy of beam electron decreases
F_I £ 0.1°
This process generates more electrons referred to as secondary electrons (SEs)

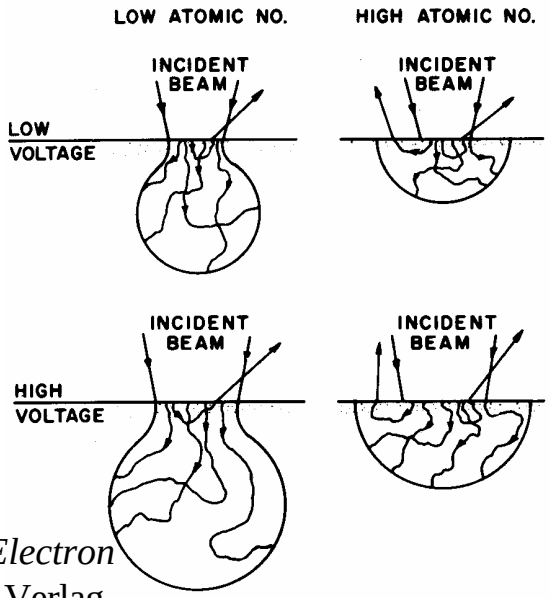


Energy loss occurs at each scattering site giving the emitted SE an energy lower than the PE

Interaction Volume

- For bulk samples, most secondary electrons generated by the primary electron beam do not make it out of the sample.
- The energy and distance of the electron from the surface are important
- The probability of emission goes up for:
  - Higher energy electrons
  - Electrons closer to the surface
- The electron signal is caused by the entire interaction volume, not by the diameter of the probe
- Volume is dependent on accelerating voltage and atomic number





Top taken from L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag.
Bottom taken from *Duncumb and Shields*

Emitted Electron Energy Spectra

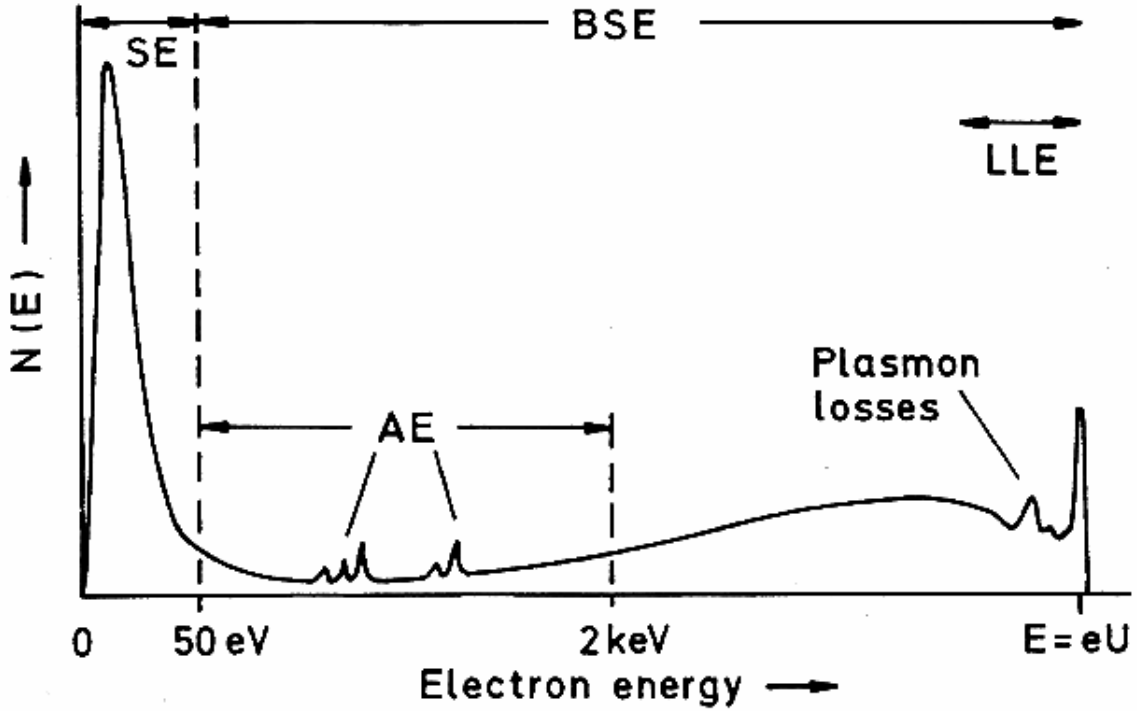


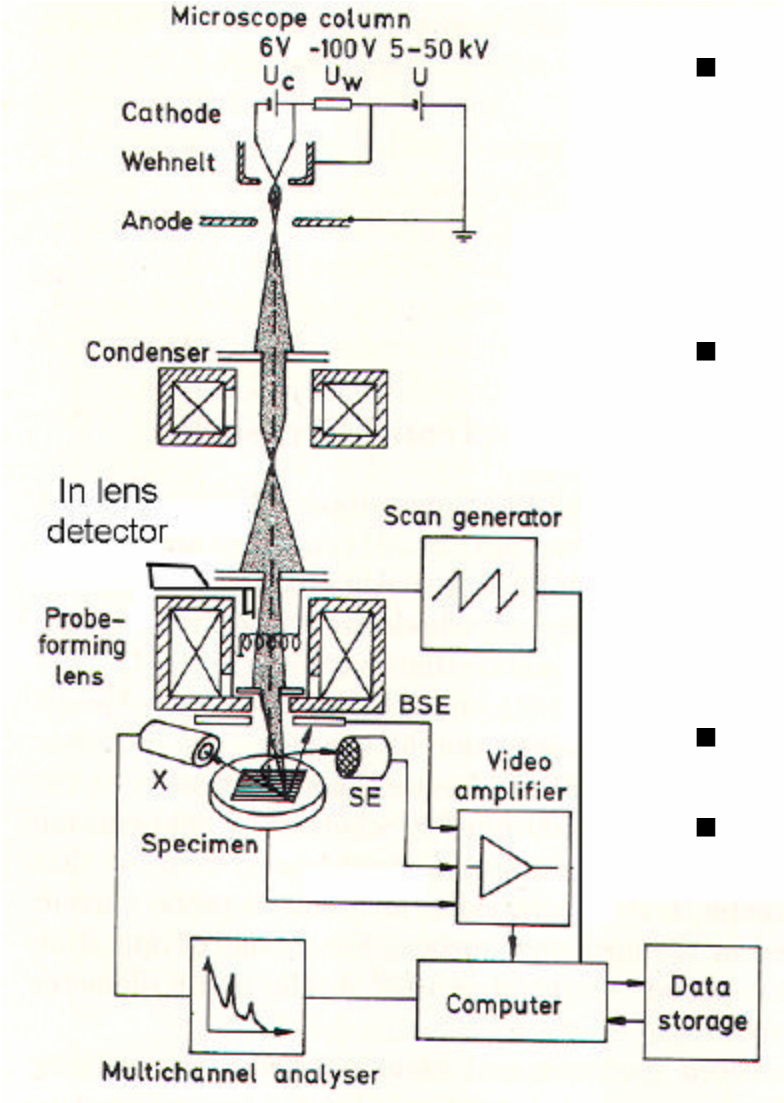
Fig. 1.5. Schematic energy spectrum of electrons emitted consisting of secondary electrons (SE) with $E_{SE} \leq 50$ eV, low-loss electrons (LLE) with energy losses of a few hundreds of eV, backscattered electrons (BSE) with $E_{BSE} > 50$ eV and peaks of Auger electrons (AE)

Most of the signal comes from low energy SEs generated by the primary beam

From L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag

Scanning Electron Microscope Equipment Overview

Scanning Electron Microscope (SEM)



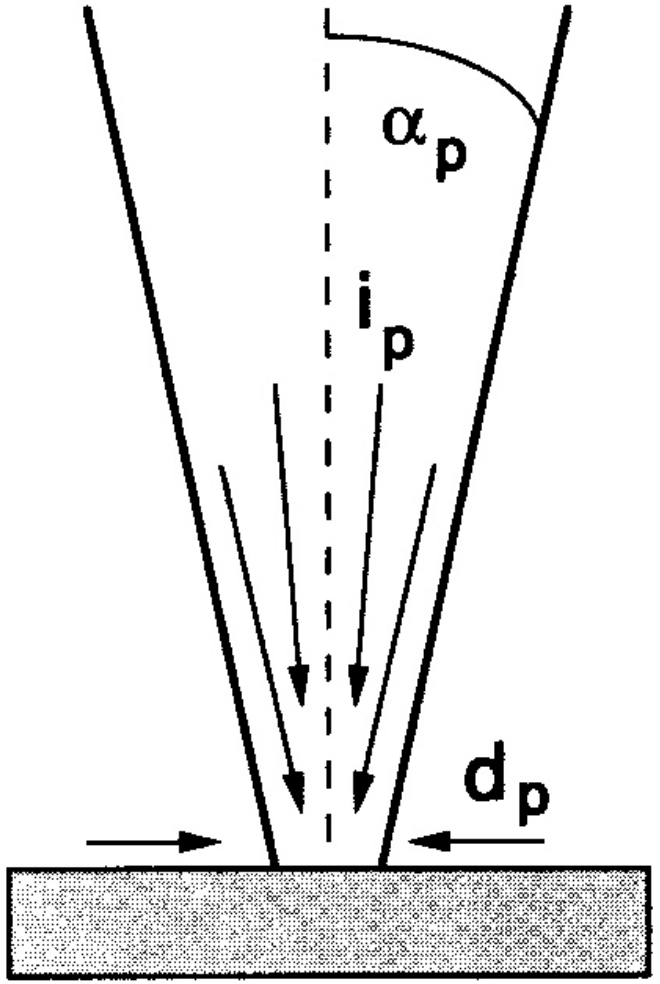
- The goal of the SEM is to scan a focused beam of primary electrons onto a sample, and to collect secondary electrons emitted from the sample to form an image
- Modern SEMs involve 5 main components
  - An electron source (a.k.a electron gun)
  - Focusing and deflection optics (referred to as the column)
  - A specimen stage
  - A detection system
  - An image acquisition and control system
- 1-4 are contained within a vacuum system
- 5 consists of a computer and a set of custom electronics

Figure based on L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag

Basic Electron Optics

- Three electron beam parameters determine sharpness, contrast, and depth of field of SEM images:
  - Probe diameter – d_p
  - Probe current – I_p
  - Probe convergence angle - α_p

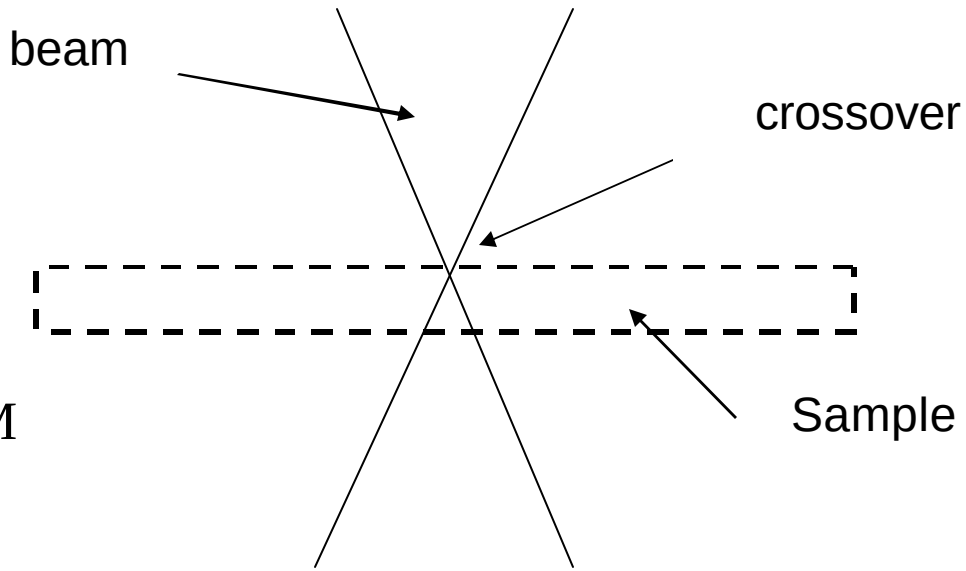
- You must balance these three depending on your goals:
  - High resolution
  - Best depth of field
  - Best image quality
  - Best analytical performance



From *Scanning Electron Microscopy and X-Ray Microanalysis,* Joseph I. Goldstein et al. Plenum Press

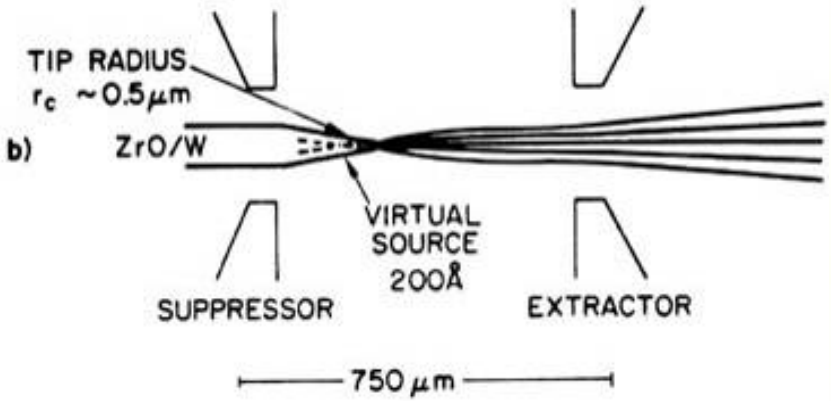
What Produces a Focused Image?

- d_p is the diameter of the beam measured at the point where it converges on itself. Note: if no sample was present it would diverge again
- The point at which the beam converges is referred to as the crossover
- The distance between the crossover and the end of the column is called the working distance (WD)
- A specimen is brought into focus by moving the specimen and the crossover to the same working distance
- The lenses and apertures in the column are what forms the crossover.
- Understanding their function is important for understanding micrographs produced by the SEM



Electron Sources

- Modern high resolution electron microscopes use two types of field emission (FE) electron sources
  - Cold cathode FE sources (referred to as CFE)
  - Thermally assisted FE or Schottky sources (referred to as TFE)
- CFE and TFE sources supply high brightness beams of electrons that can be focused into probe diameters around 1 nm
  - FE guns form a virtual source of electrons. This virtual source has a small diameter requiring less complicated focusing optics.
  - CFE sources have a smaller virtual source than TFE sources but are much less stable



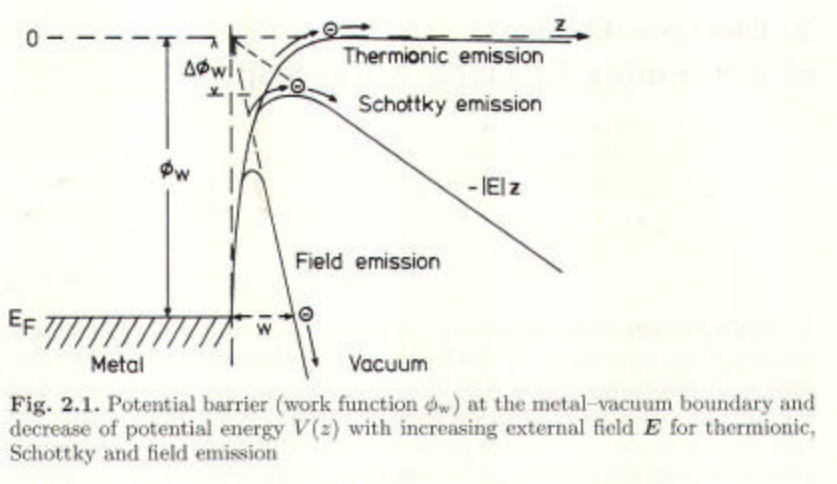


Fig. 2.1. Potential barrier (work function ϕ_w) at the metal–vacuum boundary and decrease of potential energy $V(z)$ with increasing external field E for thermionic, Schottky and field emission

Left taken from Rooks and McCord, SPIE Handbook of Microlithography
Right from L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag)

Basics of Electron Lenses

- Goal is to produce a small d_p
- Lenses produce a *demagnified* image of the virtual source at the specimen plane
- Without demagnification, the diameter of the virtual source, d_0, is too large to generate a sharp image
  - d_0 = 5 to 20 nm for FE sources
- Typical demagnification is on the order of 10 – 100
- Two basic types of electron lenses used in the SEM
  - Electrostatic: simple but have bad aberrations. Basically consist of biased electrodes
  - Electromagnetic: lower aberrations but more complex. Consist of coils wound in a high permeability material.

Aberrations of the Column

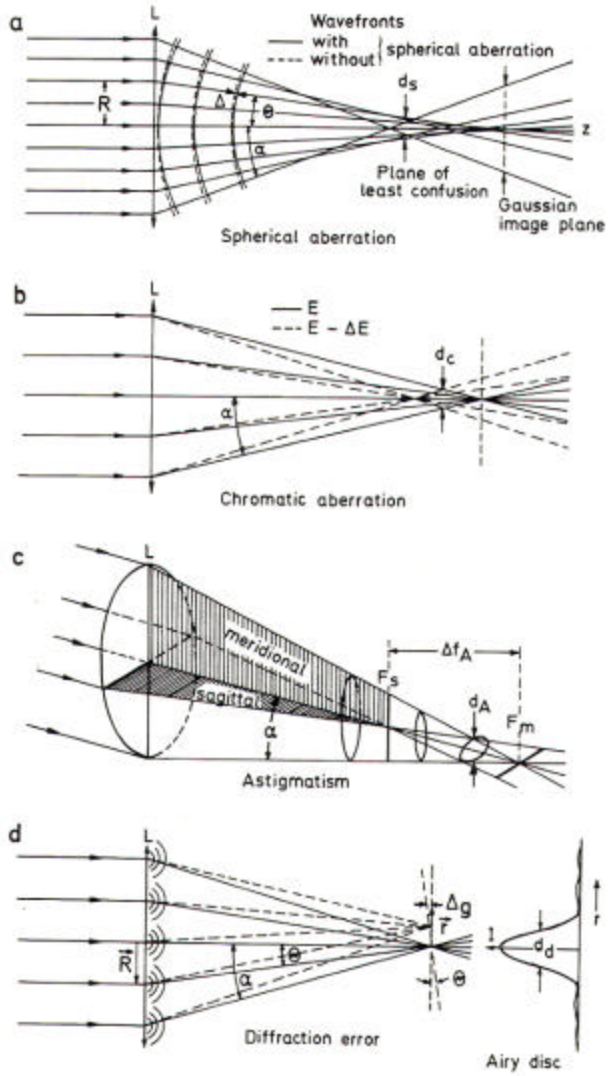


Fig. 2.9. Lens aberrations of an electron lens: (**a**) spherical and (**b**) chromatic aberration, (**c**) axial astigmatism and (**d**) diffraction error disc

- d_s, spherical aberrations result from the focusing properties of the lenses
  - Correcting this is a hot topic of research
- d_c, chromatic aberrations are caused by the energy spread of the source
  - Monochromatic sources exist but produce low I_p
- d_A, astigmatism is caused by imperfections in the lens
  - Can be corrected using electrodes called stigmators contained inside the objective lens
- d_d, diffraction causes a fundamental limit to the achievable probe size
  - $d_p = d_d$ is the ideal limit
- Aberrations of lenses add in quadrature to produce the final value of d_p
  - $d_p^2 = d_s^2 + d_c^2 + d_A^2 + d_d^2$

From L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag

Lenses Contained within the Column

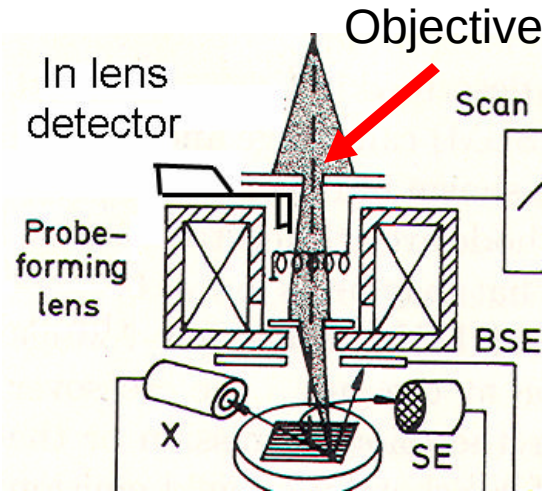
- Modern FE SEMs have two types of lenses:
  - A condenser lens (sometimes two)
    - Usually electromagnetic
    - Determines the I_p that impinges on the sample
    - Higher I_p – larger spot size – lower resolution – better S/N
    - Usually adjusted automatically by software
  - An objective lens
    - Either electromagnetic or combined with electrostatic (compound)
    - Focuses the beam by controlling the movement of the crossover along the optic-axis (Z-axis) of the column
    - Usually this is controlled by a knob labeled focus
    - The design of the lens incorporates space for the scanning coils, the stigmator, and the beam-limiting aperture
- All modern FE SEMS employ a semi-immersion lens design
  - Sample sits in an electromagnetic or electrostatic field generated by the lens
  - Significantly lowers spherical aberrations allowing better resolution
  - Can causes problems during imaging performance

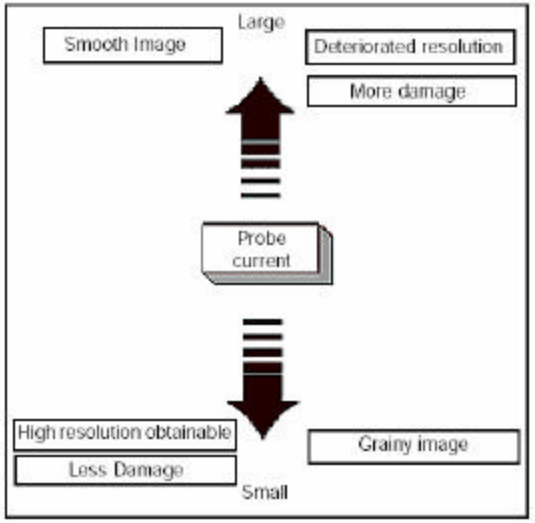
Objective Aperture

Affects all beam parameters:

- Large Aperture:
  - Large I_p - Good signal to noise (SNR), good analytical conditions
  - Large α_p - Poor depth of focus
  - Large d_p - Poor resolution

- Small Aperture:
  - Small I_p - Poor SNR, bad analytical conditions
  - Small α_p - Good depth of focus
  - Small d_p - High resolution





Upper taken from L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag, Lower taken from http://www.jeol.com/sem_/docs/sem_guide/guide.pdf

Basic SE Detector

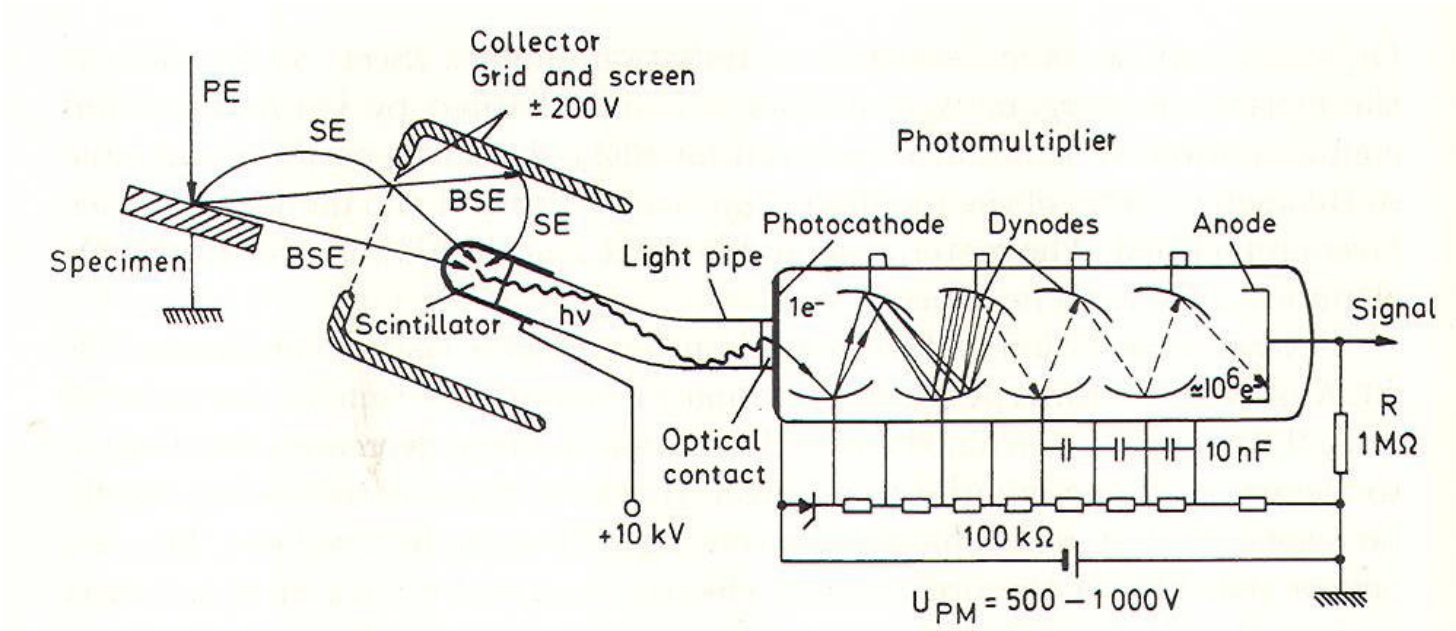


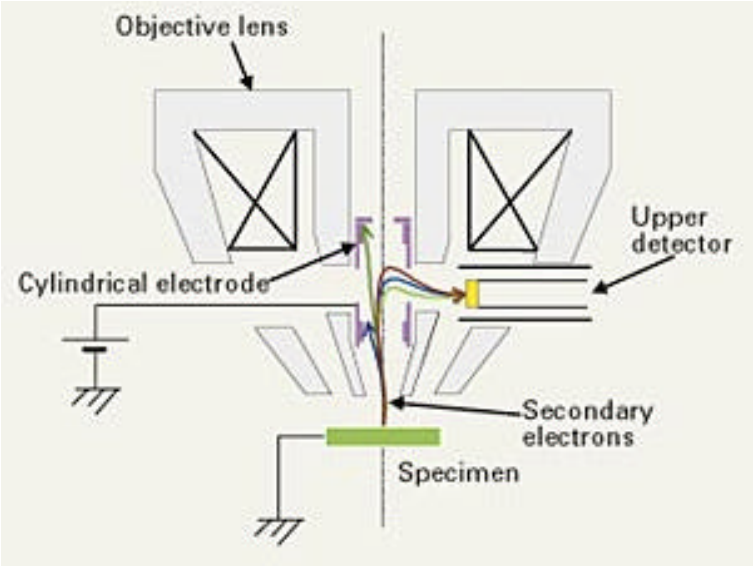
Fig. 5.1. Scintillator–photomultiplier combination (Everhart–Thornley detector) for recording secondary electrons

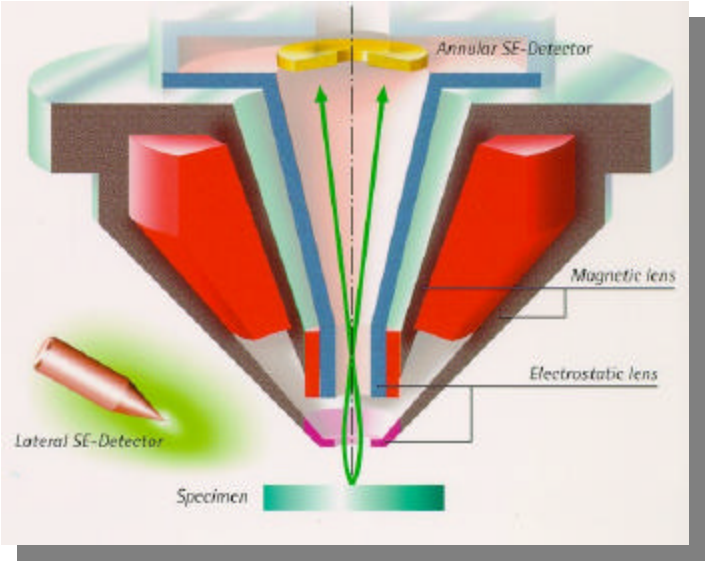
- Sometimes referred to as the lower, in chamber, SE2 or ET (Everhart-Thornley) detector
- Strongly dependent on sample orientation and topography
- Electrons travel several cm before they are collected
- High SNR

Taken from L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag

Through the Lens SE Detector

- Also referred to as a TTL, in lens or SE1 detector
- SEs travel back up the lens and are collected by a small detector similar to the ET detector
- Two different approaches
  - Upper: bias electrode forms a type of filter to push the electrons to the detector
  - Lower: detector mounted coaxially with the beam
- Shorter path length allows for more localized collection of SEs
  - Can give better resolution at short working distance

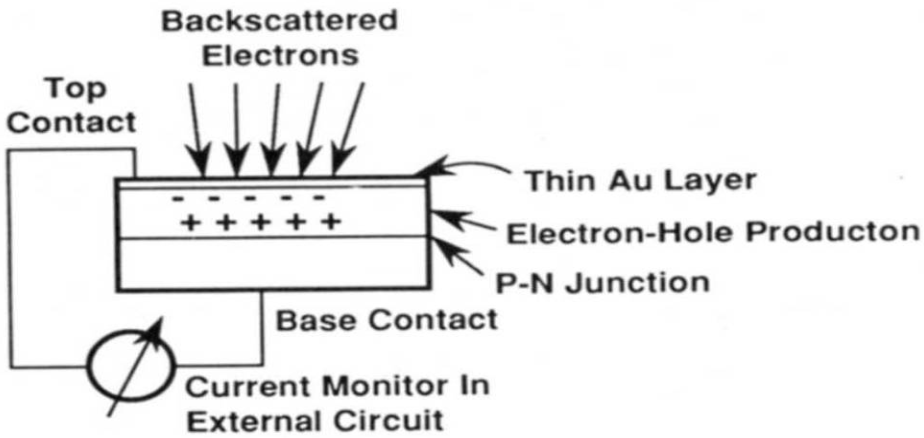


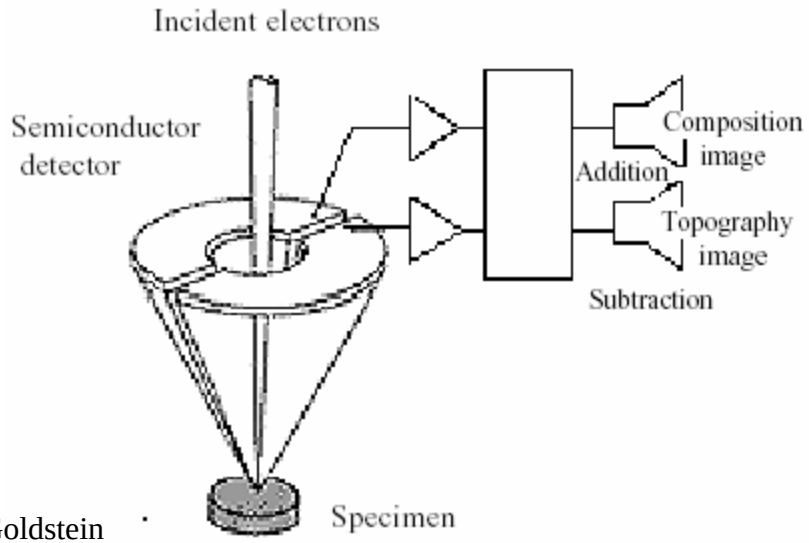


TOP: taken from
http://www.jeoleuro.com/news/news37E/htm/44/
BOTTOM: taken from www.smt.zeiss.com

Solid-State BSE Detector

- Single annular detector or smaller discrete detectors placed at the end of the objective lens
- Size permits close proximity to specimen – provides large solid angle for high geometric efficiency
- Sensitive to high energy BSEs only, not SEs





Left from *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press. Right: http://www.jeol.com/sem_/docs/sem_guide/guide.pdf

Contrast

- BSE Yield:
  - Atomic number dependent
    - Higher atomic number → Higher BSE yield → Brighter image
  - Contrast in BSE imaging is a combination of:
    - Surface topography
    - Composition - Z contrast

- SE Yield:
  - Less dependent on Z
  - More dependent on accelerating voltage
  - Contrast in SE imaging is dependent on:
    - Sample orientation (emission contrast)
    - Detector position (collector contrast)
    - Surface topography – sharp edges emit more SE's

Comparison of BSE and SE Yield

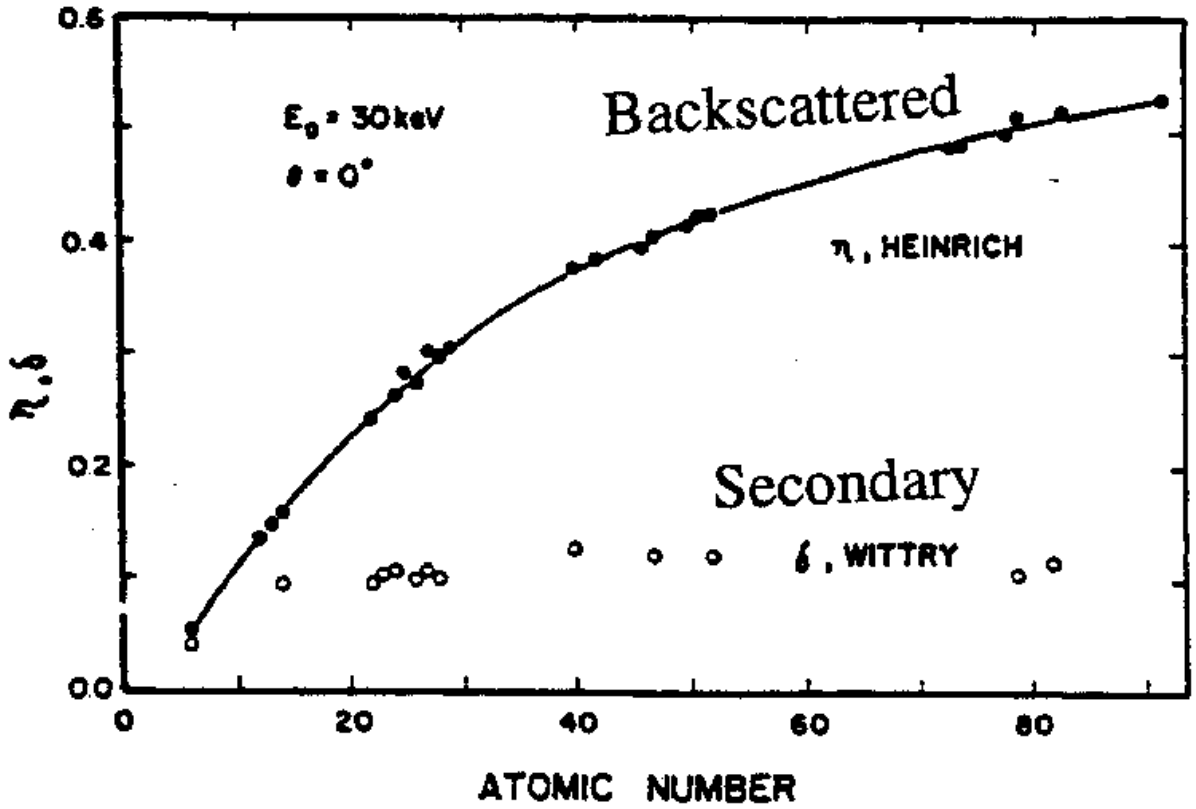
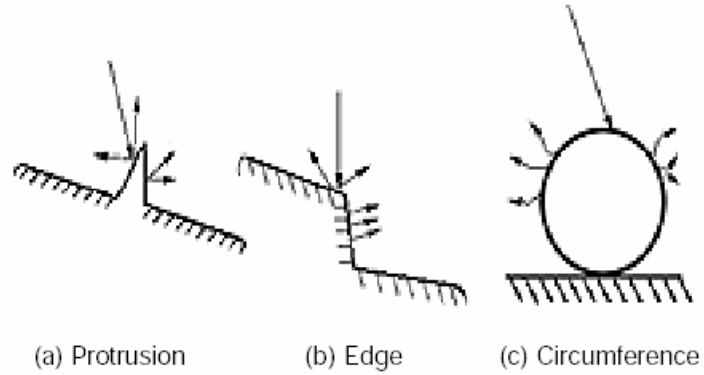
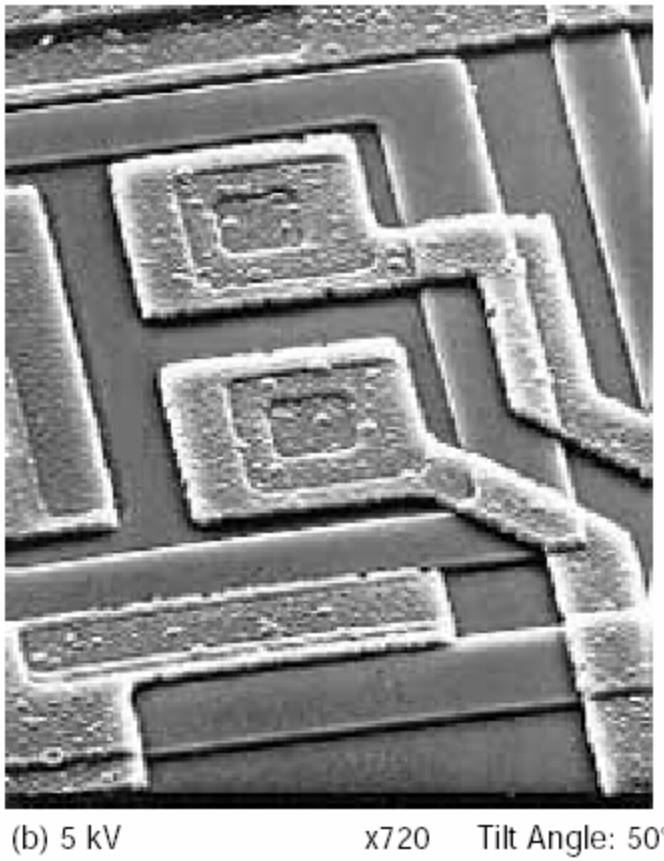


Figure 3.29. Comparison of backscattered electron coefficients and secondary-electron coefficients as a function of atomic number (Wittry, 1966; Heinrich, 1966).

The number of BSEs generated by the primary beam is dependent on the atomic number, Z, of the sample. This provides a mechanism for producing images with composition dependent contrast.

Contrast Due to Topography

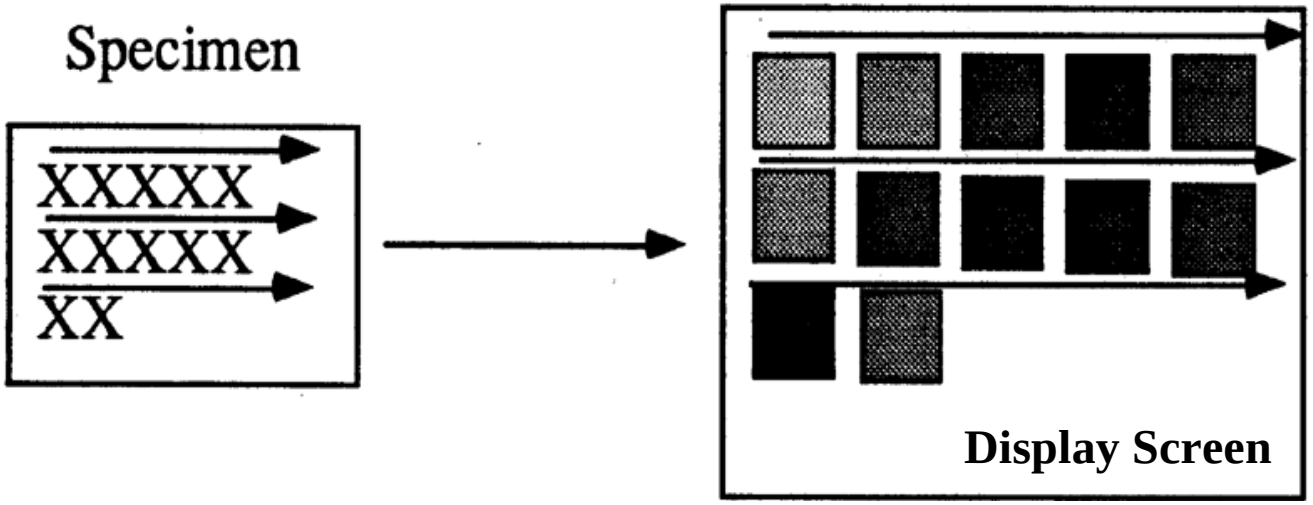




- Electron emission can be enhanced by topography
  - Easier to get out
- Creates topographic or edge contrast

Taken from http://www.jeol.com/sem_/docs/sem_guide/guide.pdf

Scan System: Image Formation



- As the beam rasters across the sample the intensity of the electron signal measured by the detector is recorded and displayed on the screen.
  - Bright means you are getting electrons
  - Dark means you are getting less or no electrons
- The number of electrons detected from the specimen determine the intensity changes
- Note: rastering over a smaller area has the effect of increasing the magnification.

From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

Understanding the Signals

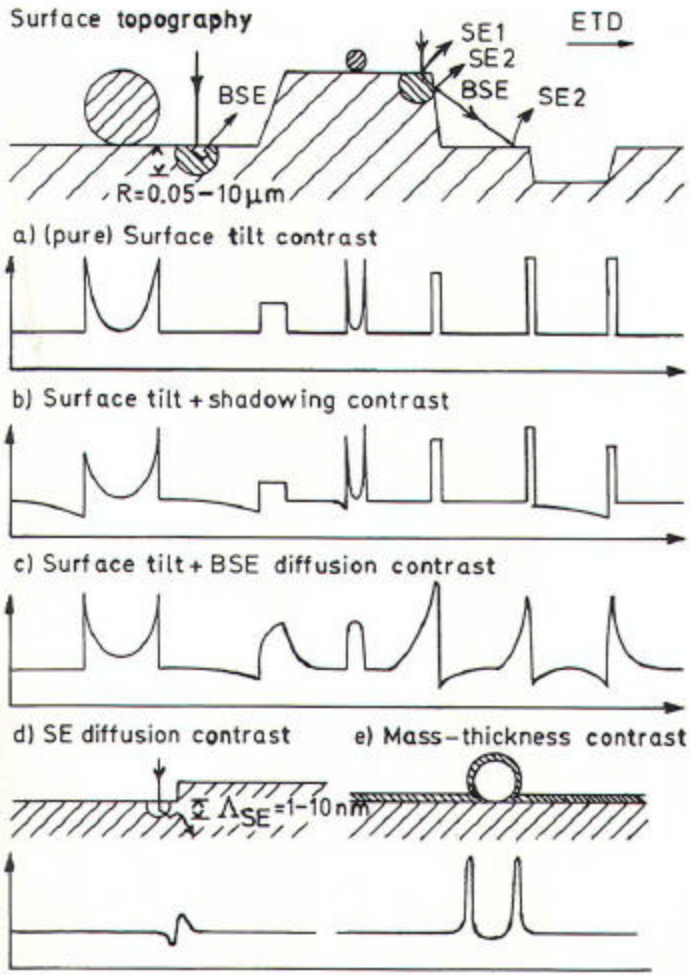


Fig. 6.1. Contributions to topographic contrast demonstrated schematically by surface contours (top) and linescans of SE signals; (a) surface tilt contrast, (b) shadowing contrast, (c) BSE diffusion contrast, (d) SE diffusion contrast and (e) mass-thickness contrast

- Different detectors "see" the sample in different ways
- Intensity is not always proportional to topography, composition, etc

Taken from L. Reimer,
Scanning Electron Microscopy,
2nd edition, Springer Verlag

Modern High Resolution FE SEM Systems

Zeiss Ultra

FEI Sirion

Hitachi S4800

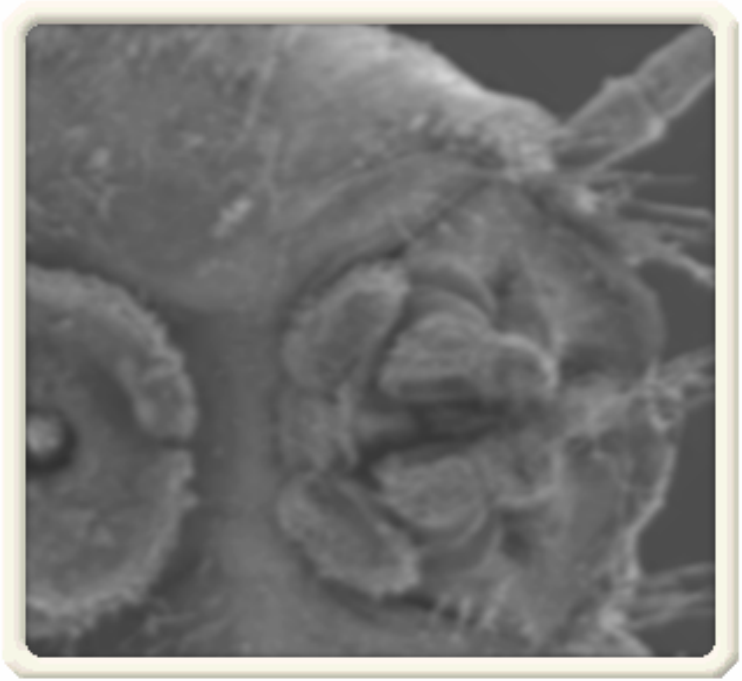
JEOL 7401F

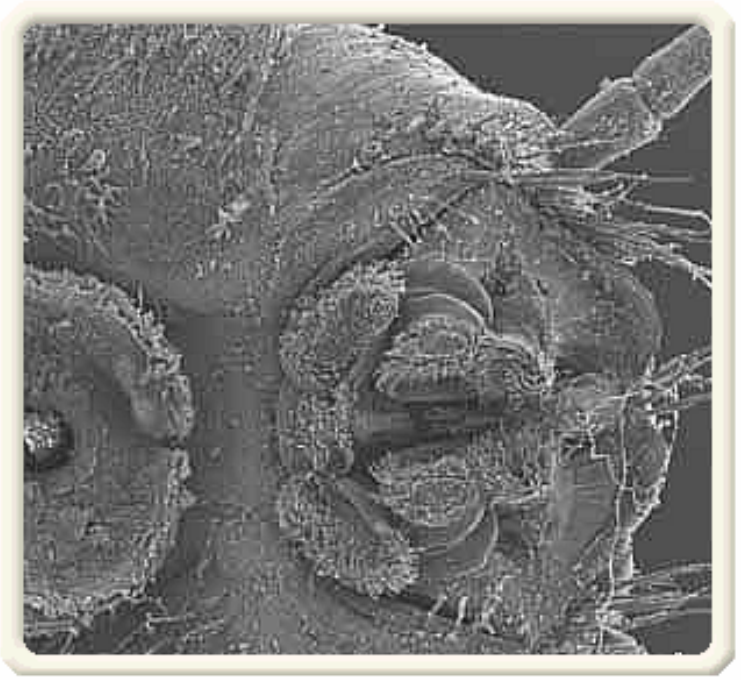
Instrument	Source	Objective lens	Resolution	Detectors	Other
Zeiss Ultra	TFE	Electrostatic-electromagnetic compound lens	1.7 nm @ 1kv 1.0 nm @ 15 KV	In Lens SE, In Lens BSE with E filter, E-T SE	Customizable software
FEI Sirion	TFE	Magnetic Semi immersion	unpublished	In Lens SE, E-T SE	
Hitachi 4800	Cold FE	Magnetic Semi immersion	2 nm @ 1kv 1.0 nm @ 15 KV	In Lens SE with E filter, E-T SE	Nice loadlock
JEOL 7401F	Cold FE	Magnetic Semi immersion	unpublished	In Lens SE with E filter, E-T SE	

Information and images taken from each manufacturers web site

Using an SEM

Getting Things in Focus



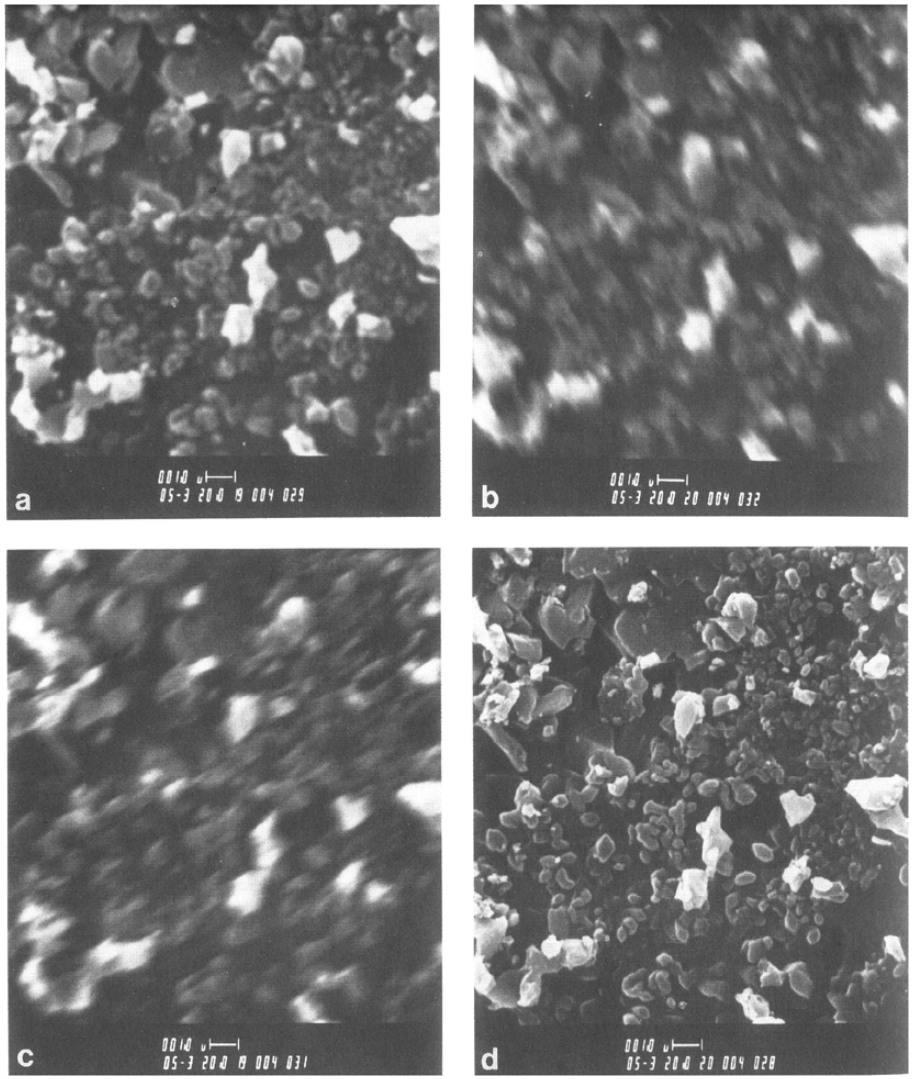


- Taken from the the SEM tutorial at:
  http://www.micro.magnet.fsu.edu/primer/java/electronmicroscopy/magnify1/index.html
- Give it a try!

Astigmatism: The Effect of a Stigmatic Beam

- Effect can be recognized by stretching of the image in two perpendicular directions, when the objective lens is underfocused and then overfocused

- At exact focus — stretching vanishes



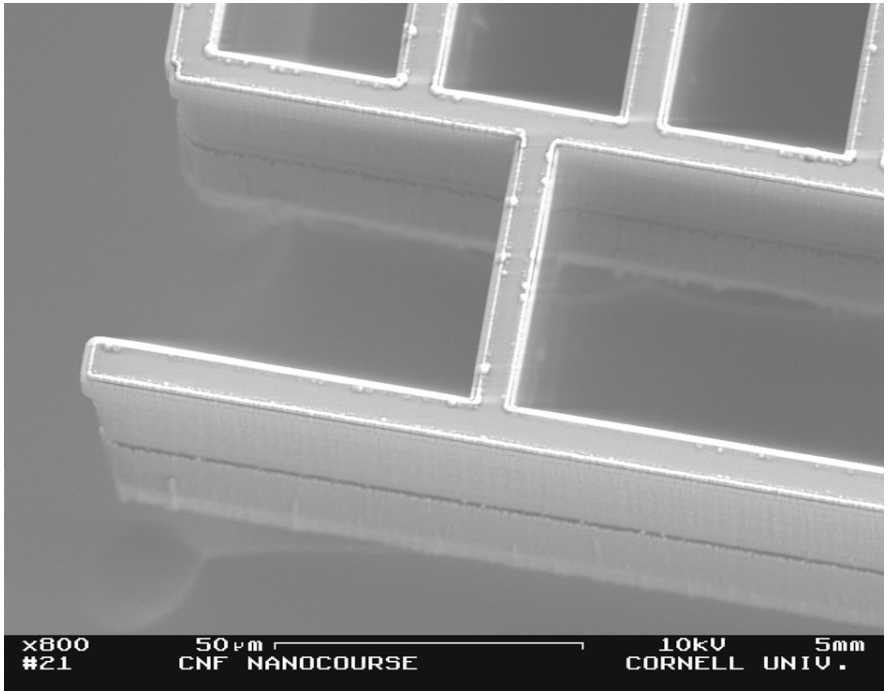
From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press

Aperture Alignment

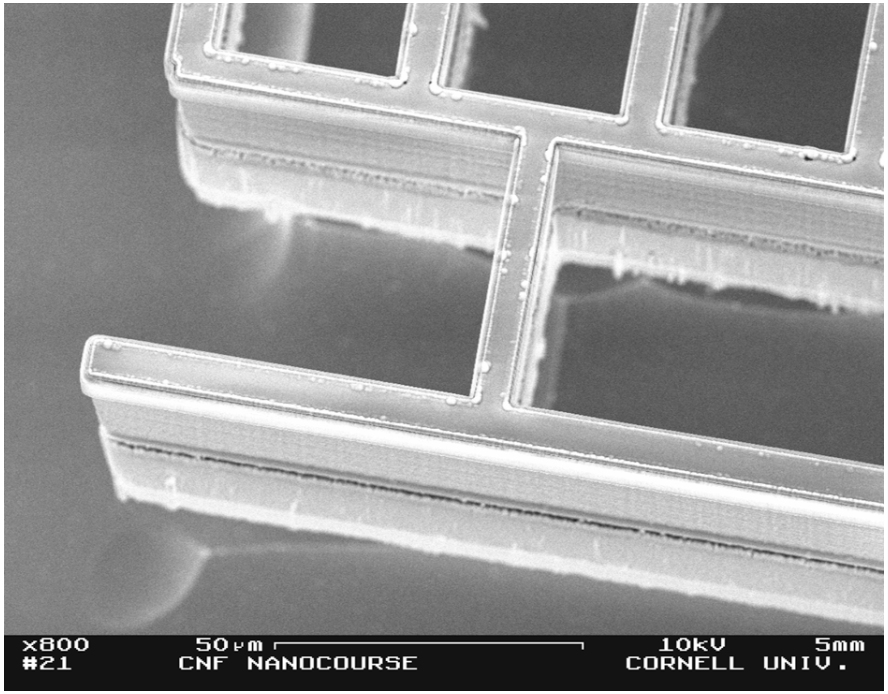
- For optimum resolution the beam limiting aperture must be centered on the electron optical axis
- If your image is shifting while focusing the aperture is not centered
- Most FE SEMS have a focus “wobble” to assist in the centering of the aperture –
  - When properly adjusted the image will show no side to side movement, It will only go in and out of focus
- Needs to be adjusted whenever the accelerating voltage or aperture is changed

Choosing an SE Detector

MEMs Comb Drive



ET SE-Detector (in chamber)



TTL SE-Detector (in lens)

Different information from each detector

Look at both signals before taking a picture

Picking an Accelerating Voltage

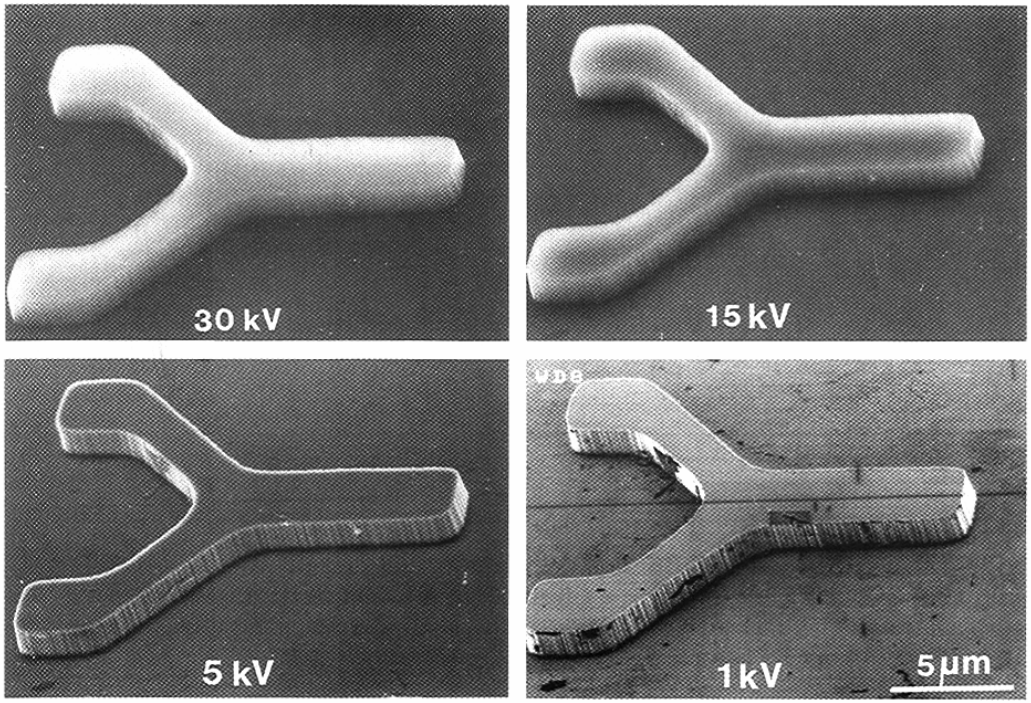


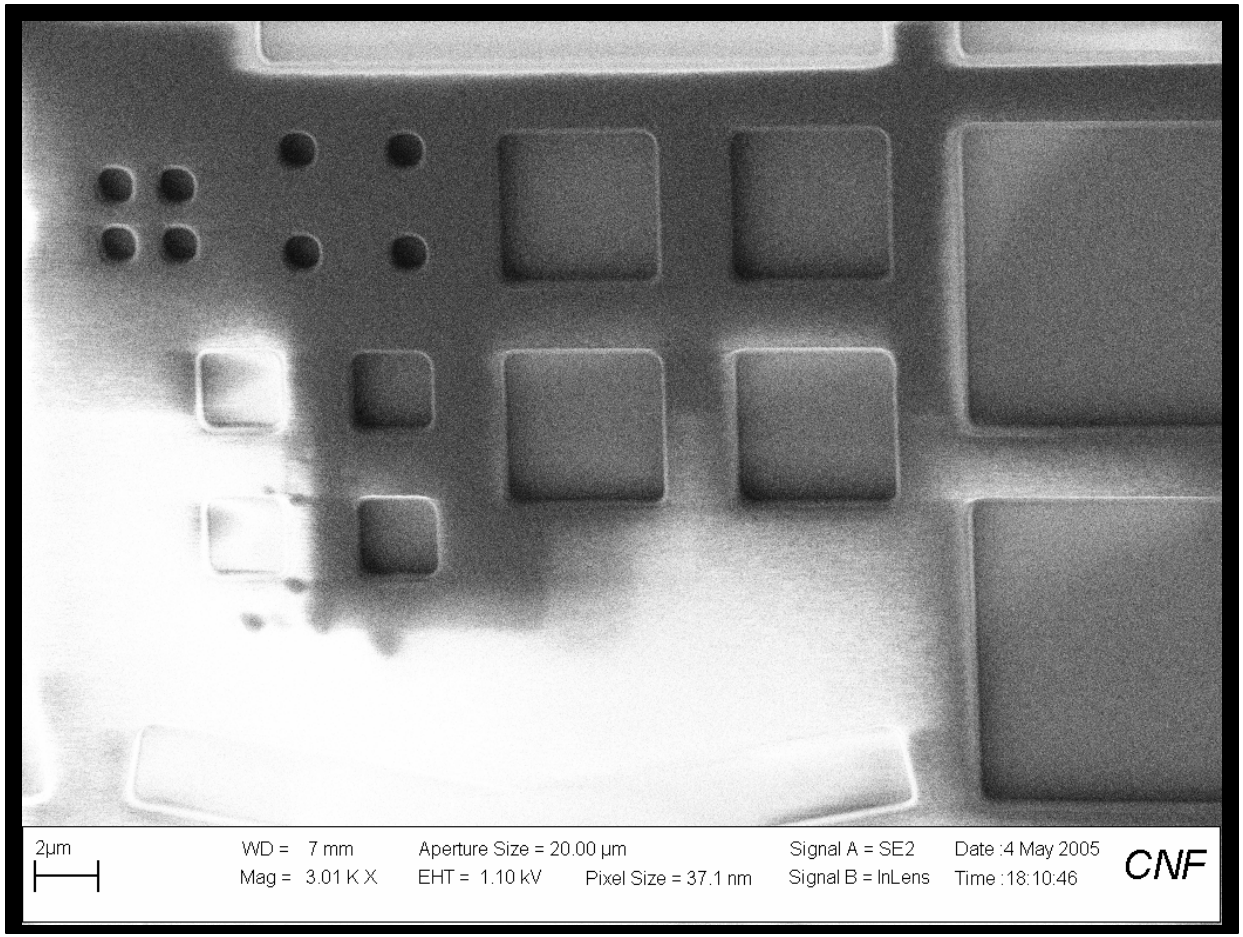
Fig. 6.8. SE image of a Y-shaped bar on silicon imaged with a primary electron energy of (**a**) 30 keV, (**b**) 15 keV, (**c**) 5 keV and (**d**) 1 keV (surface tilt $\phi = 45°$)

- For conductive samples you can use essentially any voltage
- More surface information at lower voltages
- Higher voltages penetrate deeply into the sample
  - Can see metal lines buried in dielectric

Unfortunately, many samples aren't conductive!
For these samples a means of mitigating excess charge must be found

Taken from L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag

Charging in Nonconductive Samples



Accelerating Voltage = 1.1 keV

Photoresist on SiO_2 on Si

Bright areas indicate negative charge accumulation

Low Voltage SEM: One Solution to Charging

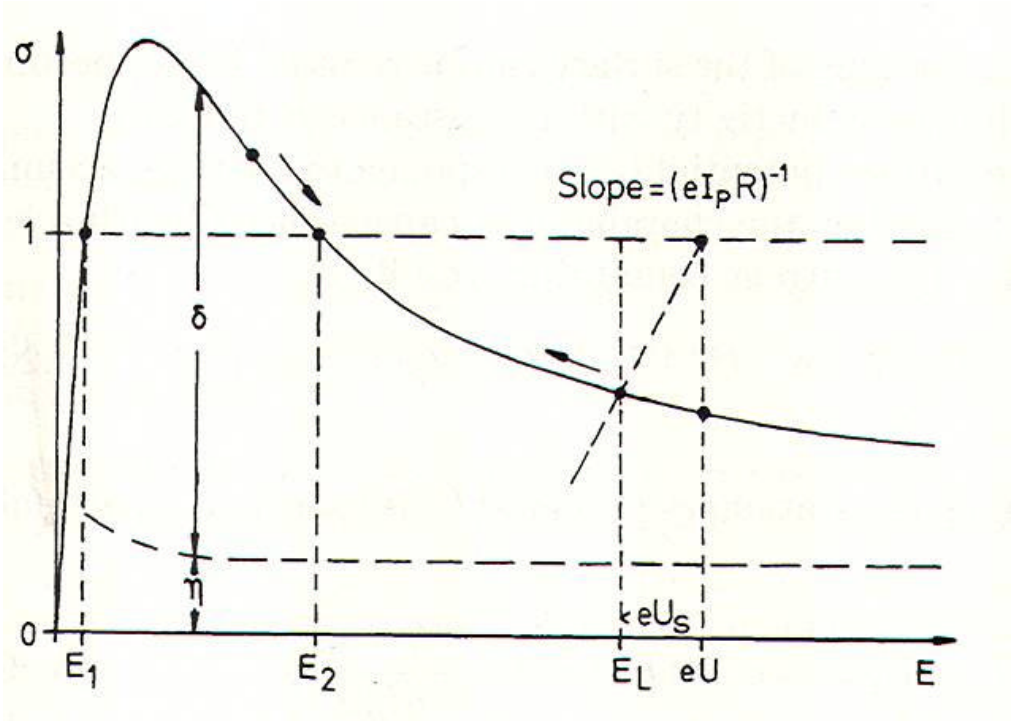
This plot shows the ratio of SE emitted from sample to the amount of incoming electrons from the primary beam.

In order to operate the SEM without charging the sample, you must operate at unity (1 electron in gets 1 electron out).

If more electrons come out than come in, the sample will **charge positively (image will look dark)**.

If more electrons come in than go out the sample will **charge negatively (image will look bright)**.

Due to the effect that this will have on the electron beam, **the most stable operating point is E_2.** E_2 is typically between 0.5 and 5 keV for most samples.



Taken from L. Reimer,
Scanning Electron Microscopy,
2nd edition, Springer Verlag

Determining the E_2 Stable Voltage

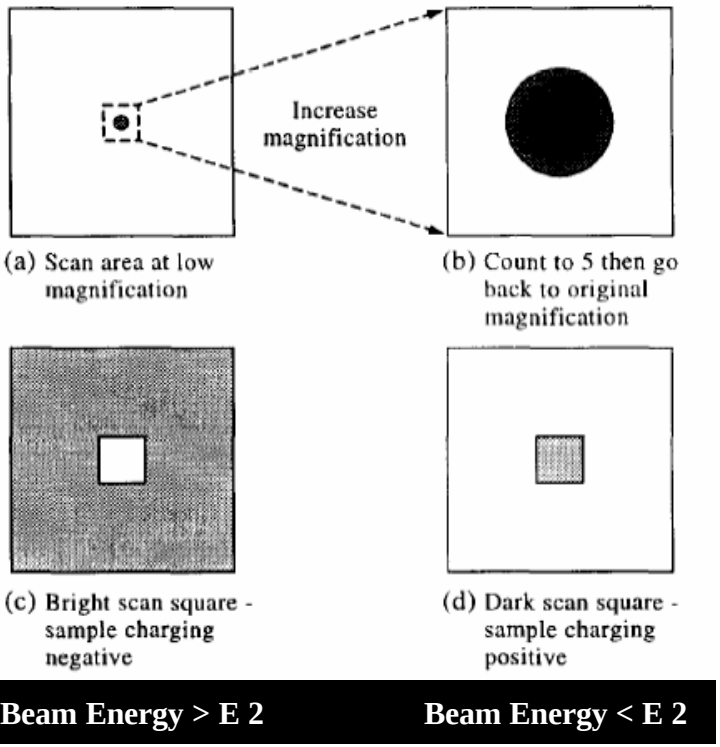


Fig. 14. Procedure for the determination of *E*2 using variable magnification scanning.

Table 1. *E*2 Data, semiconductors and other inorganics

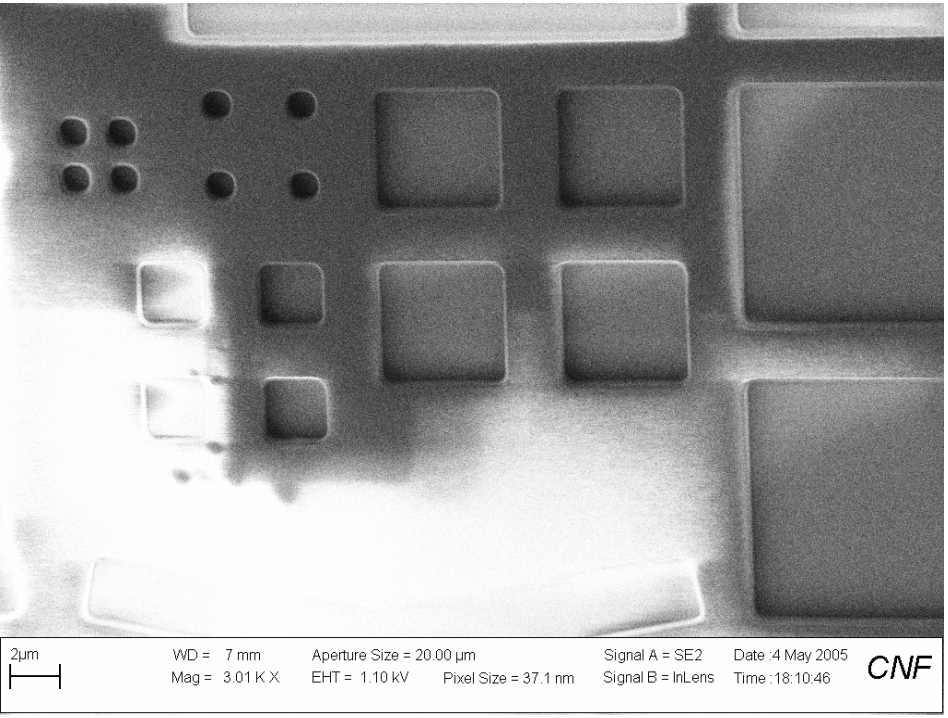
Semiconductors			
Compound	*E*2 (keV)	Compound	*E*2 (keV)
Low density resist	0.55	Cr on glass	2.0
Resist on Cr substrate	0.70	Glass passivation	2.0
Resist on oxide	0.90	SiO2 (quartz)	3.0
Resist on poly-Si	1.10	Alumina Al_2O_3	2.9
PMMA resist	1.6	High res. GaAs	2.6
Other inorganics			
Compound	*E*2 (keV)	Compound	*E*2 (keV)
NaCl	2.0	Pyrex glass	1.9
KCl	1.6	CaF2(fluorite)	1.9
LiF	1.9		

This protocol and table provide useful guidelines for finding the stable voltage, E_2

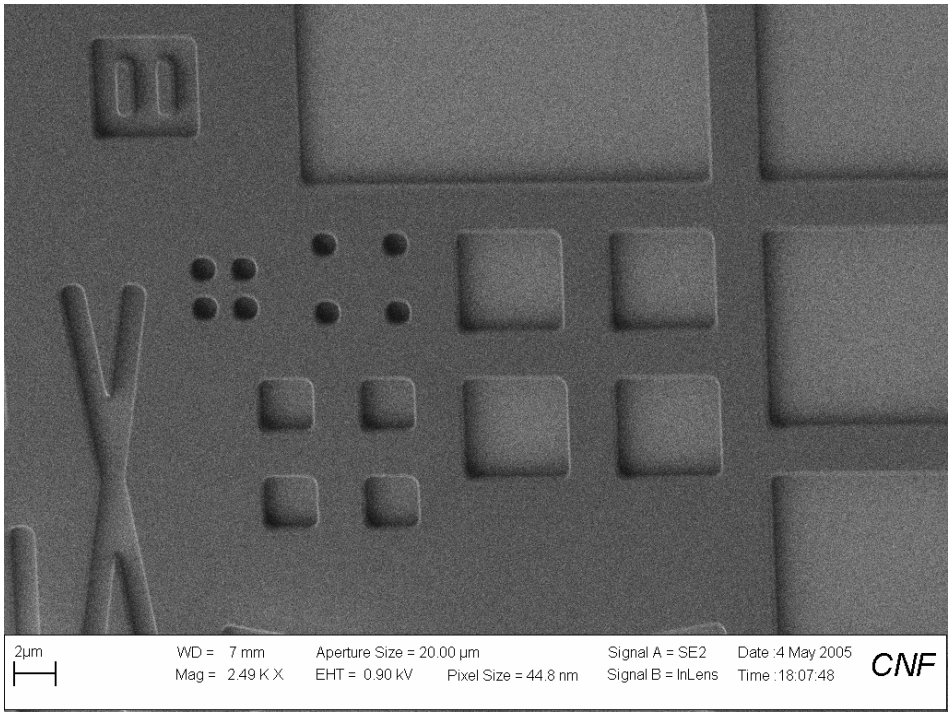
NOTE: these are only guidelines!

Taken from D. Joy, et al, Micron, 27, 247 (1996)

Example: Photoresist on SiO_2



1.1 kV

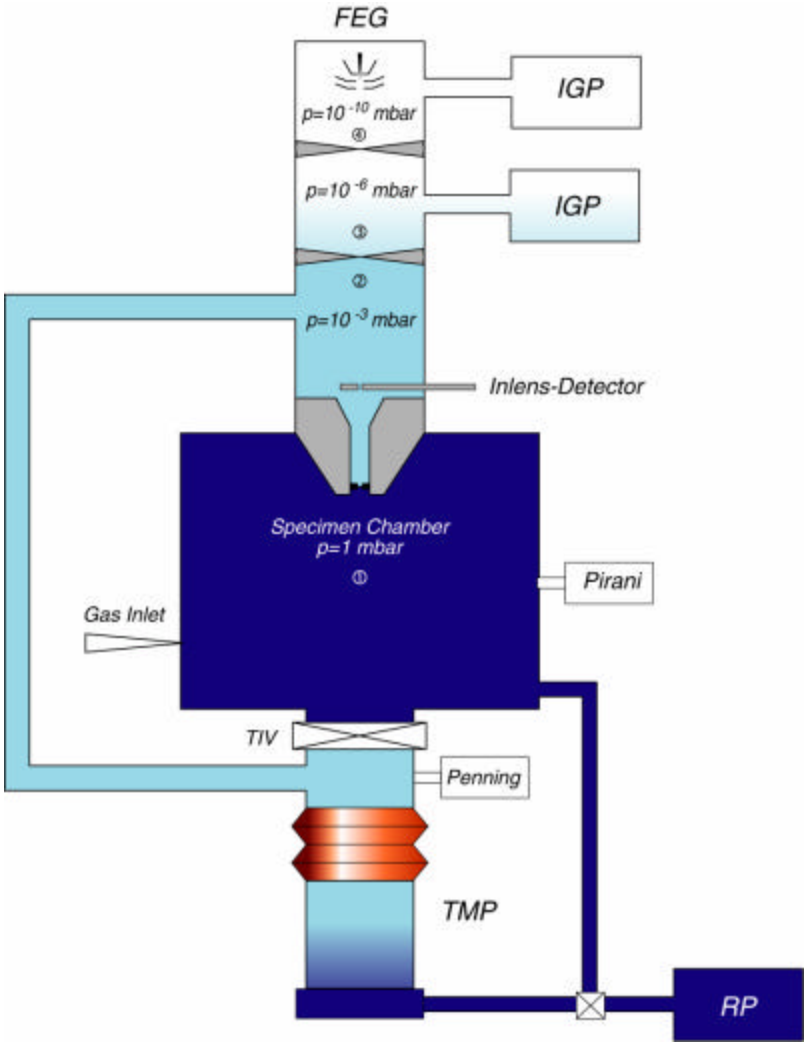


0.9 kV

Changing the beam energy by 200 V eliminates charging.
This suggests that 900 V is the stable beam energy (E_2) for this sample.

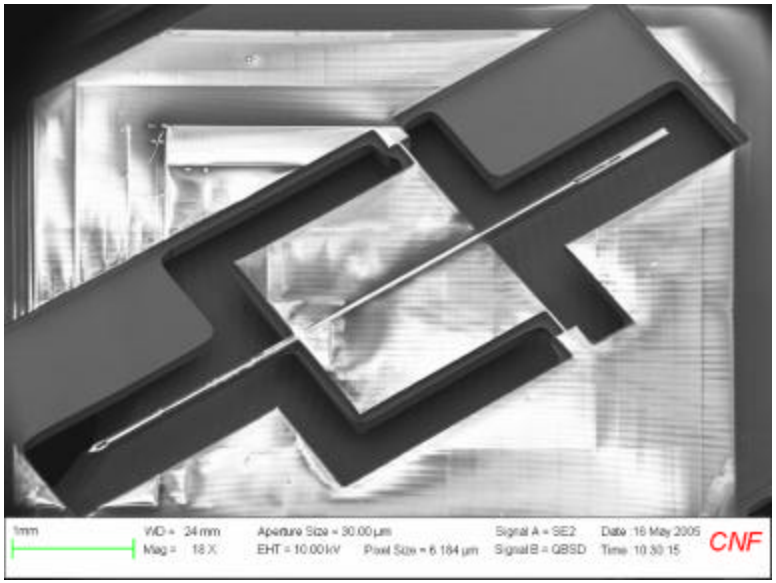
Variable Pressure (VP) SEM: Another Solution to Specimen Charging

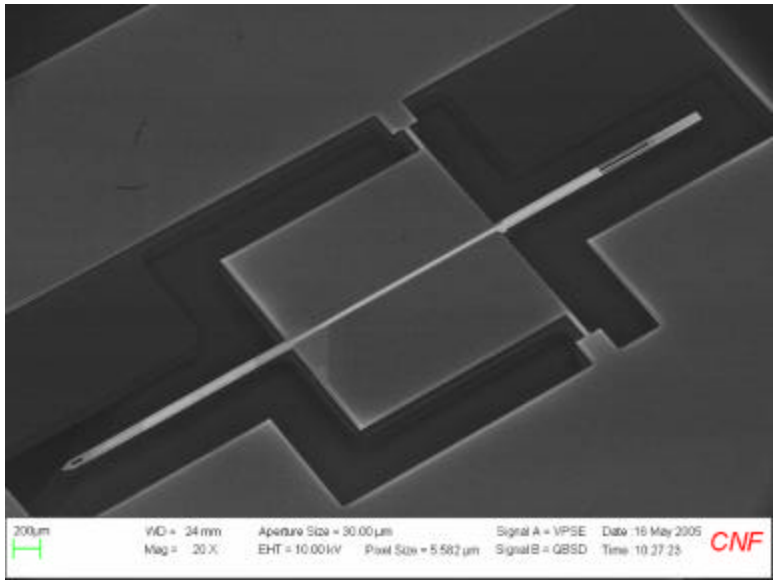
- Bleeds in gas to raise chamber pressure
- Gas molecules impinging on the surface provide a mechanism to relieve charge
- Modern VP FE SEMs have resolution close to regular FE SEMs
- Excellent tool for imaging insulating samples at higher voltage

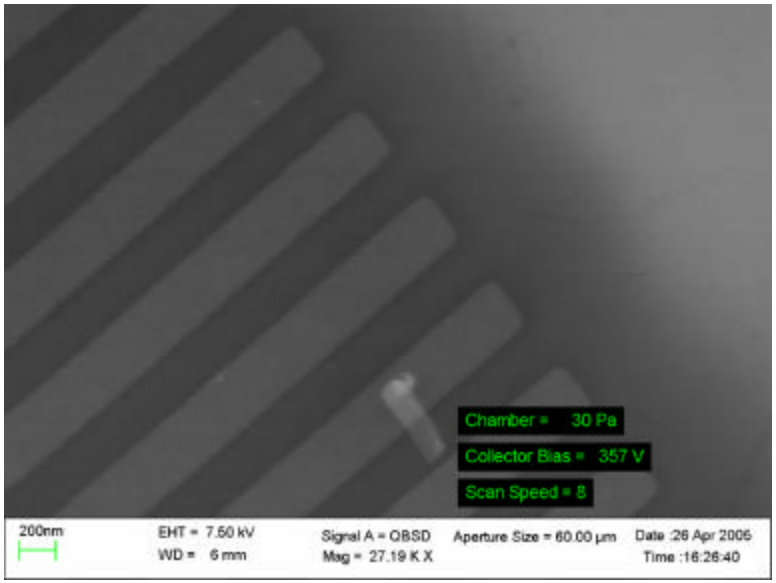


Taken from www.smt.zeiss.com

VP SEM Examples







- Upper left: Polyimide/parylene coated MEMS device imaged at 10 keV
- Upper right: Polyimide/parylene coated MEMS device imaged at 10 keV in VP mode
- Left 200 nm wide Pt lines on Pyrex imaged in VP mode

Depth of Field: Effect of Working Distance

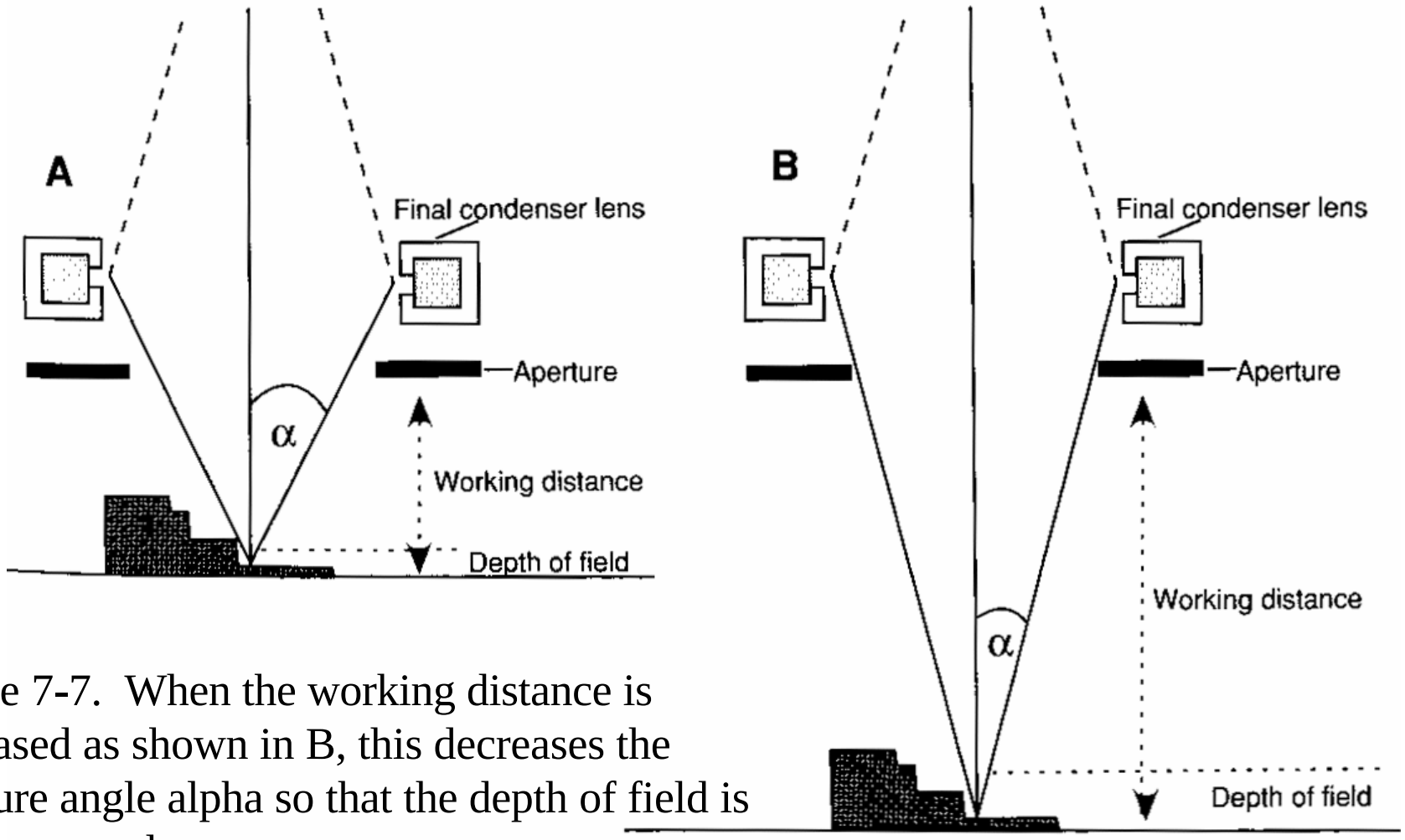
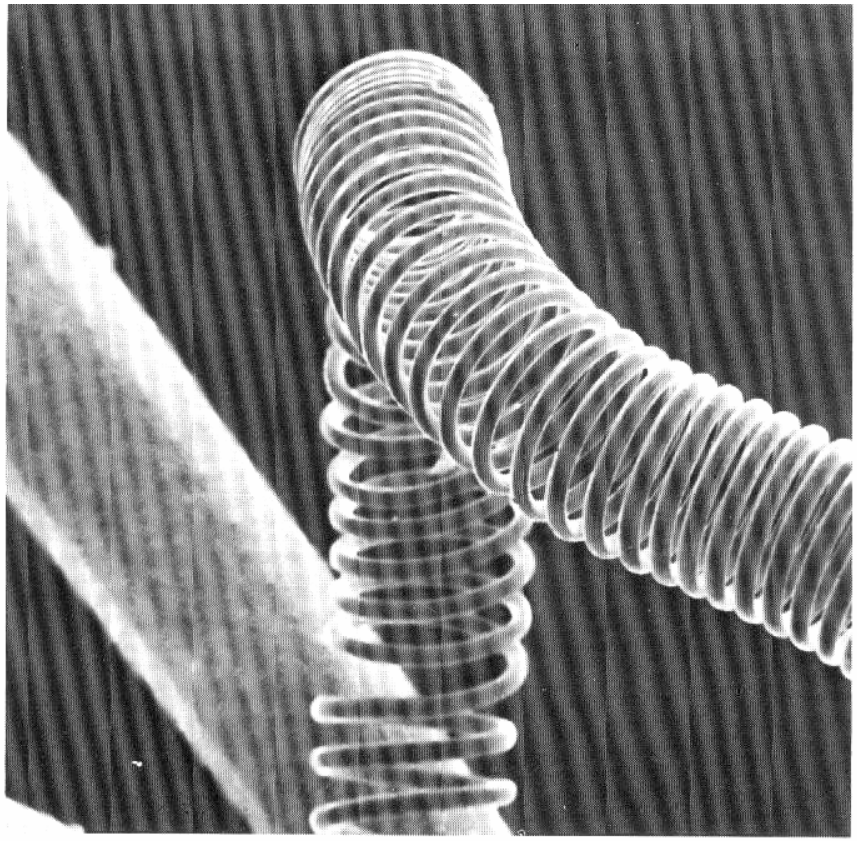


Figure 7-7. When the working distance is increased as shown in B, this decreases the aperture angle alpha so that the depth of field is also increased.

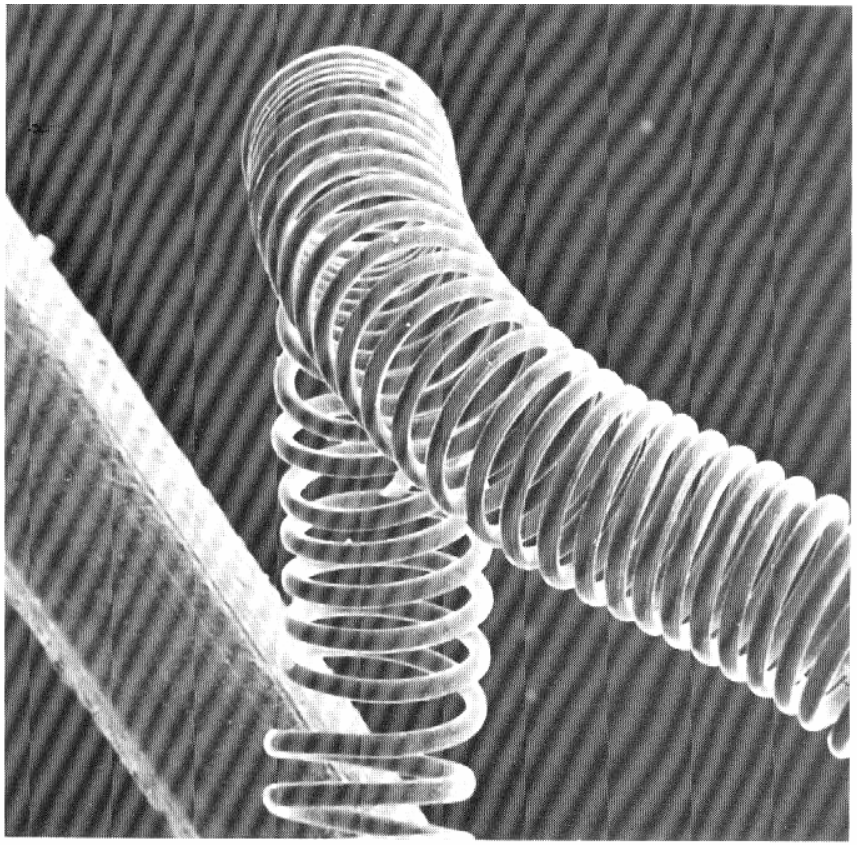
Redrawn from Postek et al., 1980

Depth of Field: Effect of Working Distance



(c) W. D. 13 mm Aperture size 100μm.

WD = 13 mm



(d) W. D. 37 mm Aperture size 100μm.

WD = 37 mm

Depth of Field: Effect of Aperture Size

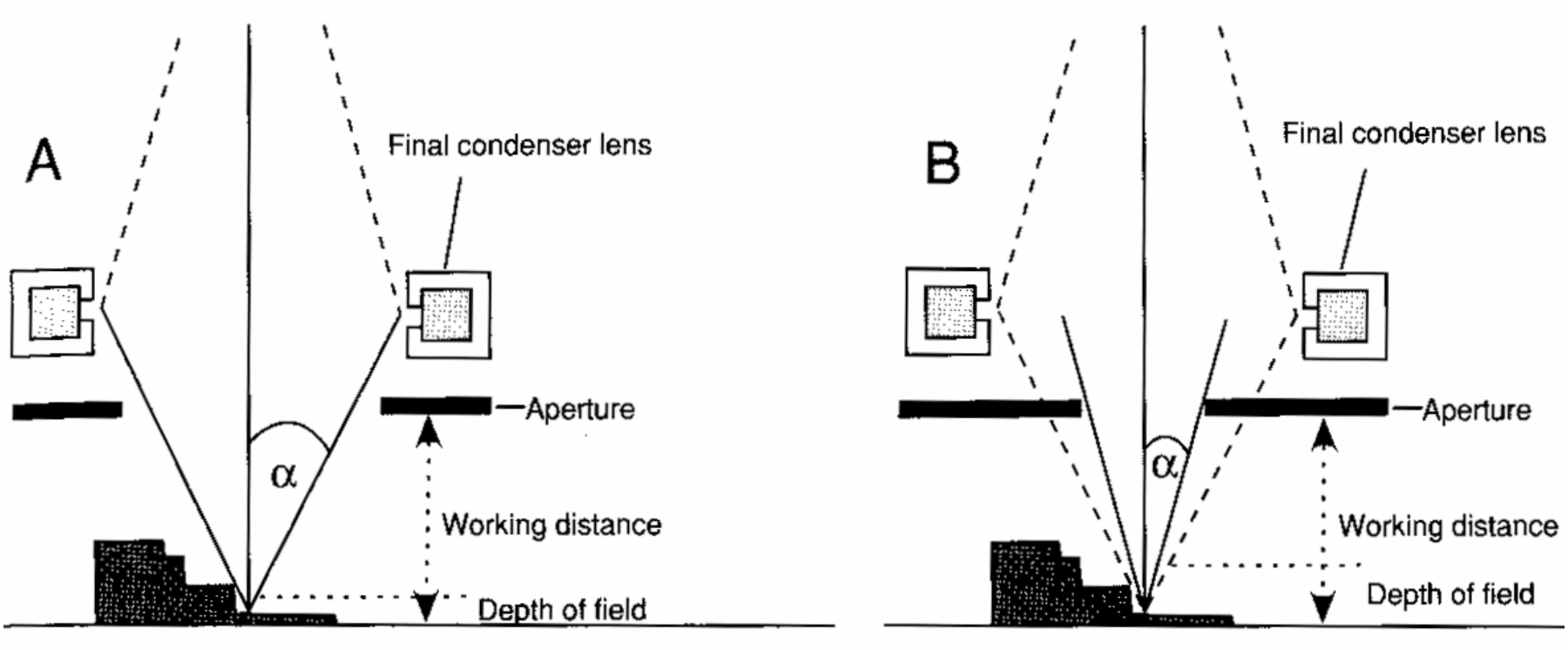
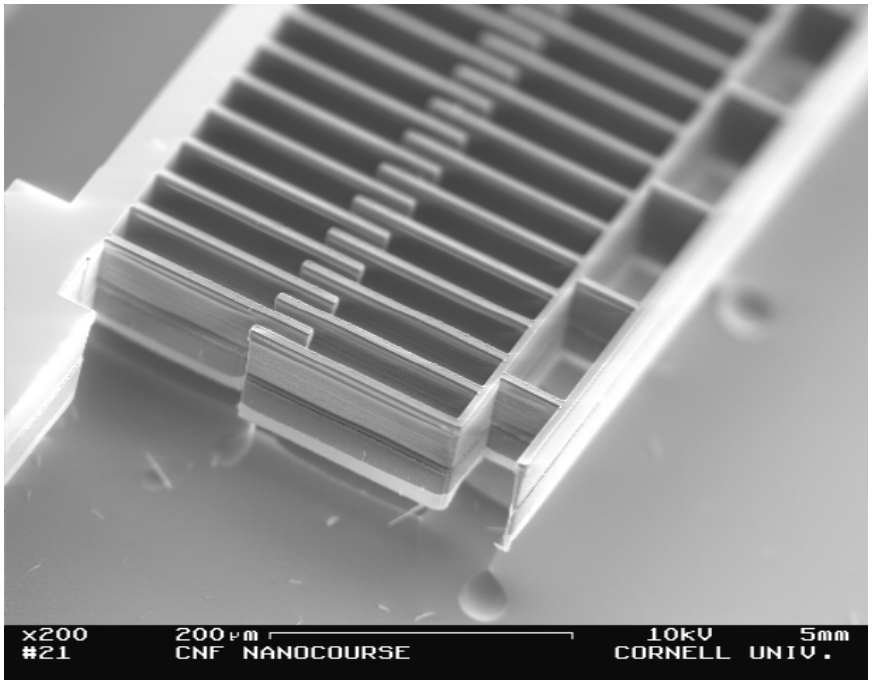


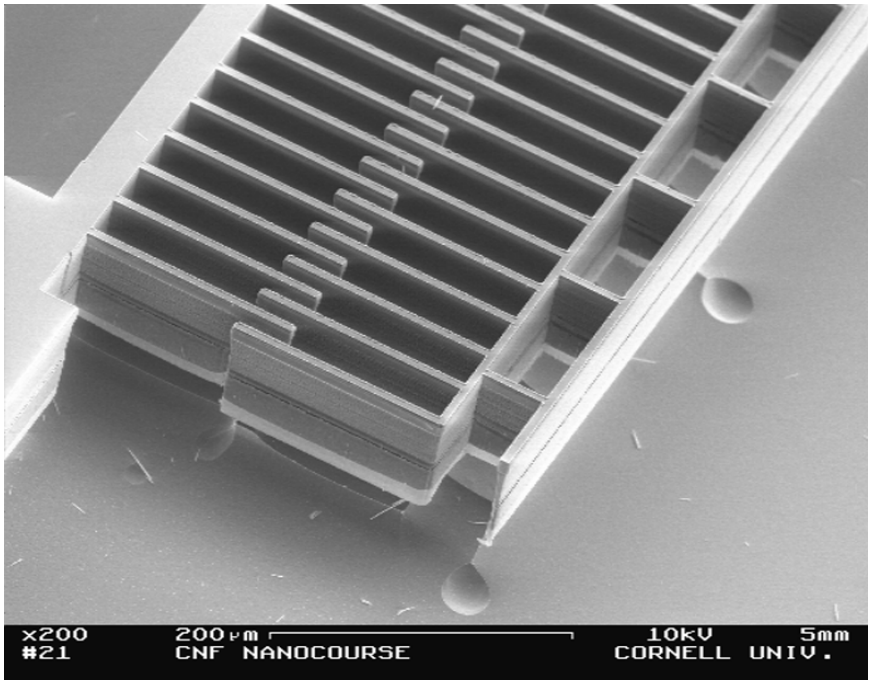
Figure 7-6. Depth of field (the depth that is in focus in the specimen) is increased by using smaller apertures as shown on right.

Redrawn from Postek et al., 1980

Depth of Field: Effect of Aperture Size



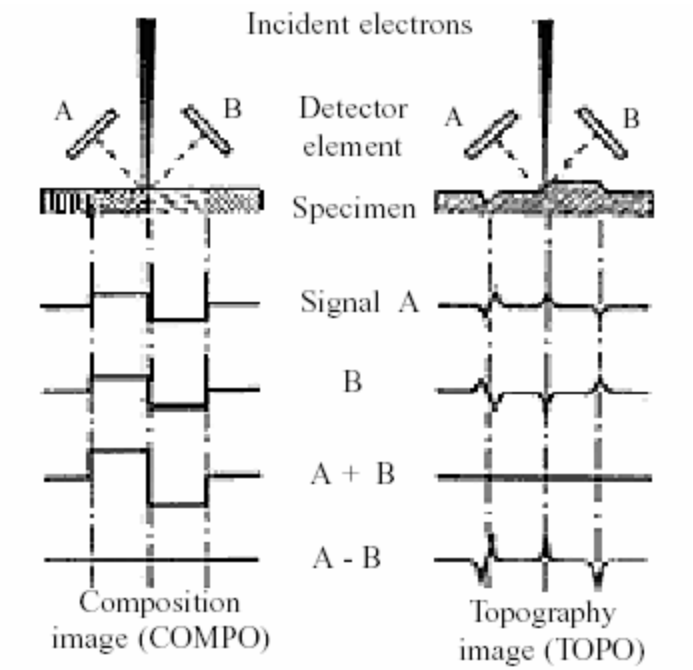
WD - 5 mm, Aperture size 120 µm

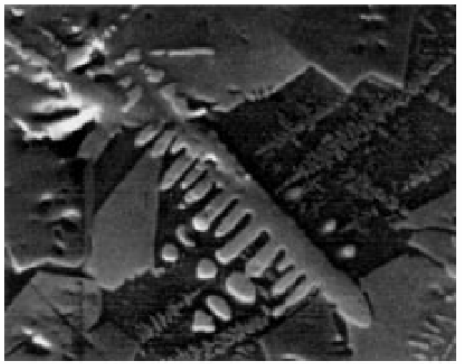


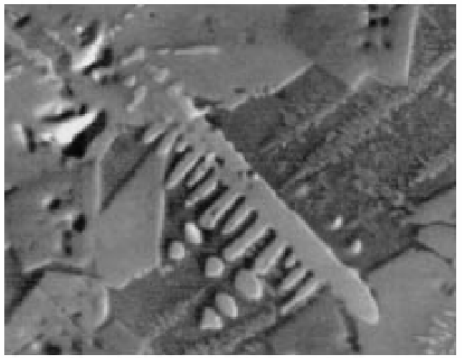
WD - 5 mm, Aperture size 20 µm

MEMS Comb Drive

BSE Imaging Modes




(a) Backscattered electron image (BEI)


(b) Topography image (TOPO)

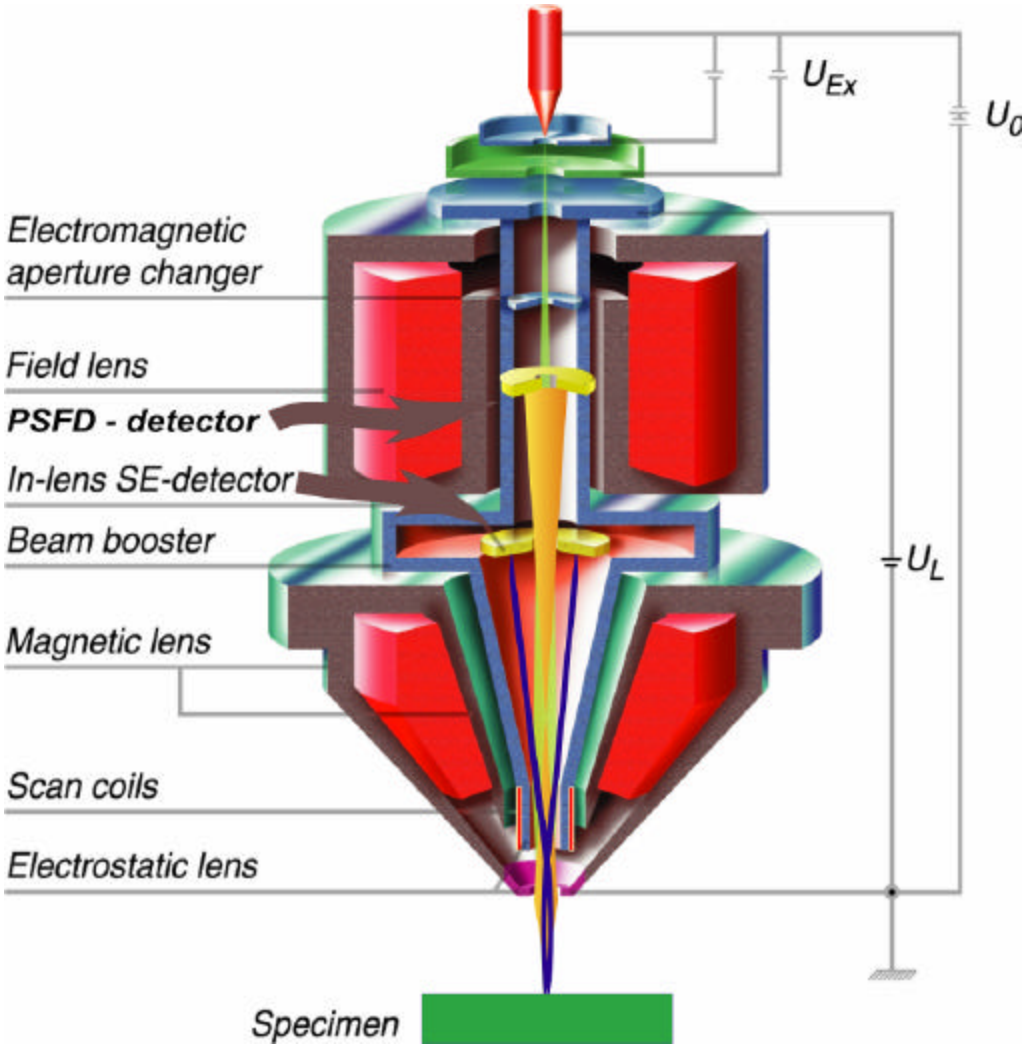

(c) Composition image (COMPO)

Signals from the split ring BSE detector can be combined in different was to produce different contrast

Taken from http://www.jeol.com/sem_/docs/sem_guide/guide.pdf

In Lens BSE Detector

- Uses energy filter to observe BSEs coming back into the lens
- Hitachi, FEI and Zeiss offer some variant of this technology
- Only Zeiss Ultra enables imaging of BSEs less than 2 keV



Taken from www.smt.zeiss.com

In Lens BSE Detector

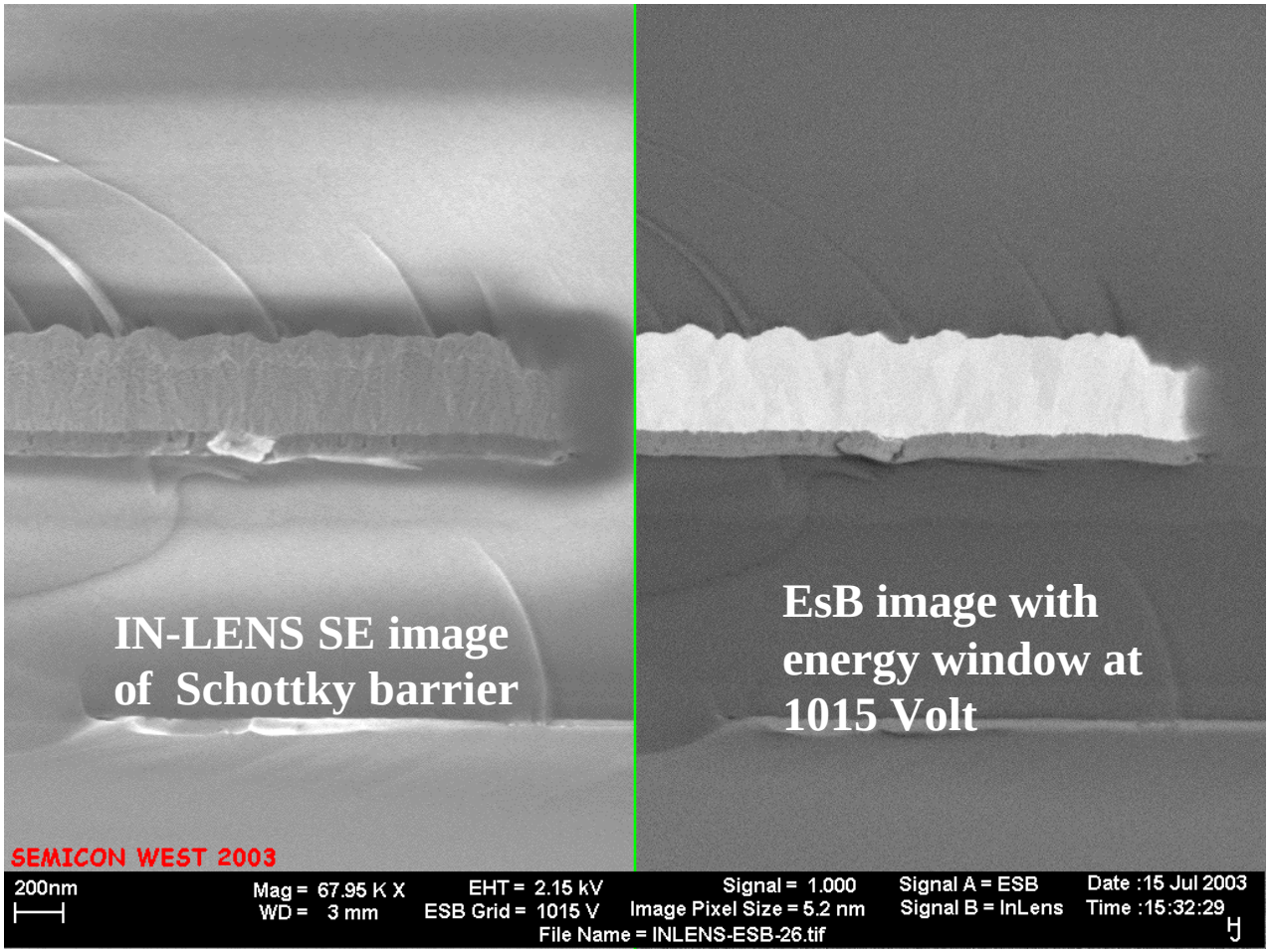
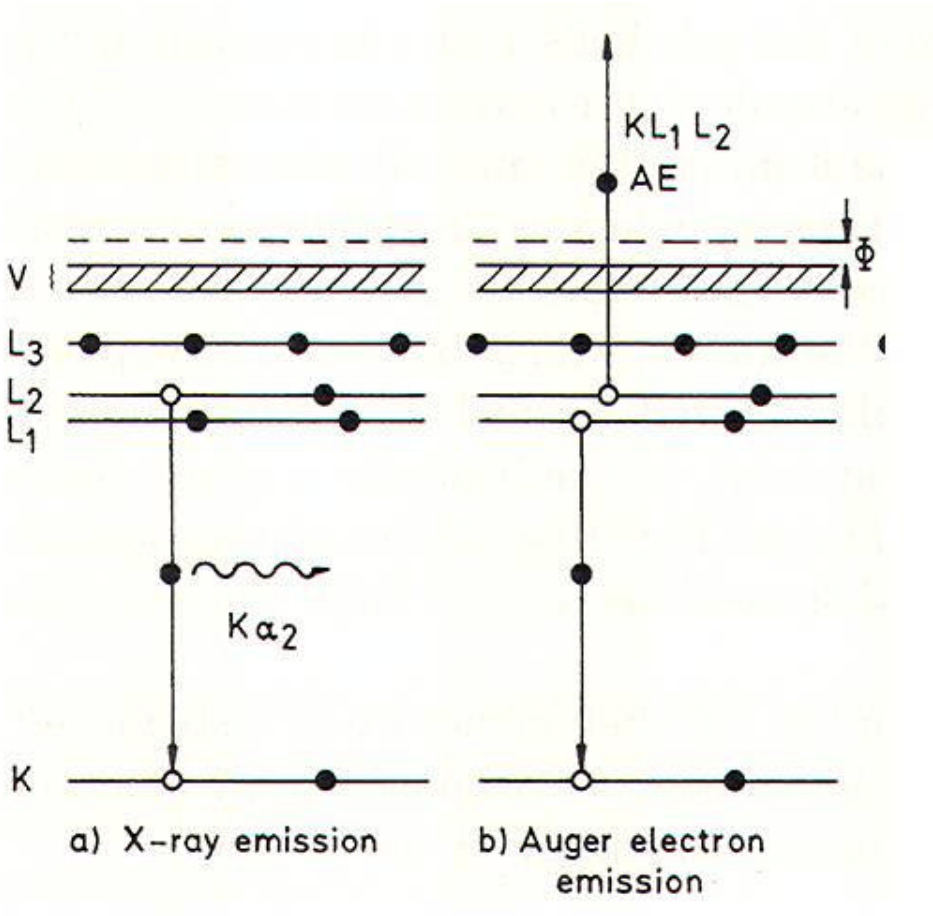


Image obtained using Zeiss in lens energy selective BSE detector

Taken from www.smt.zeiss.com

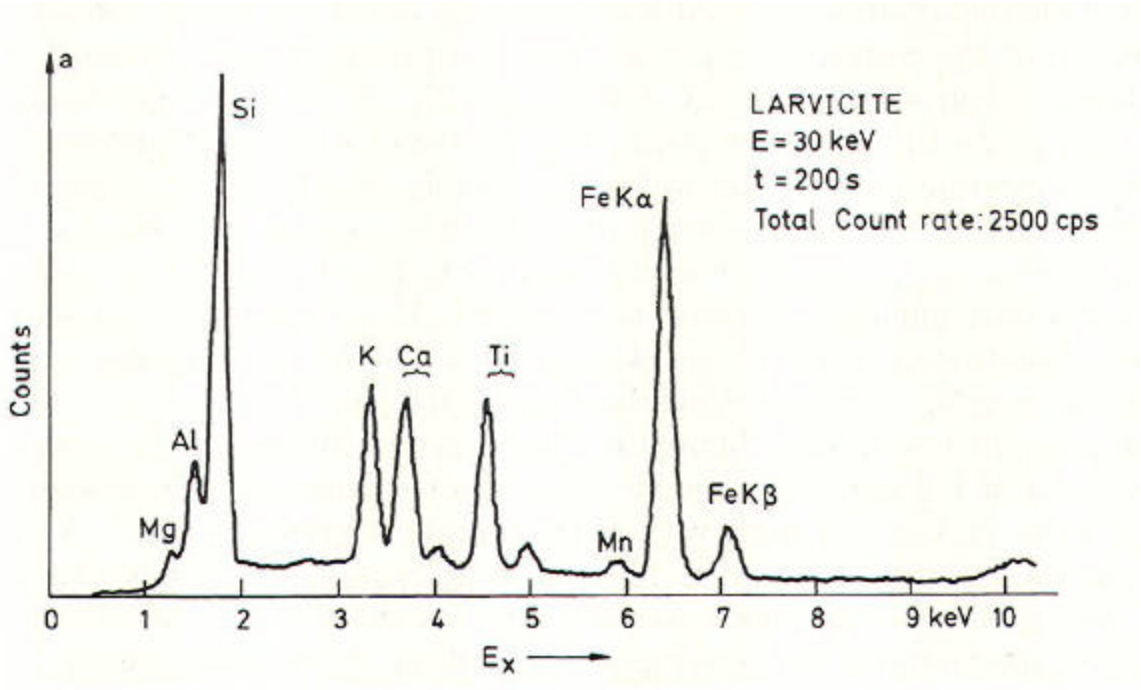
X-Ray and Auger Analysis

- Electrons changing in energy emit a characteristic x-ray photon
- This can be used for bulk compositional analysis
- Auger electron emission caused by radiationless energetic transition
- Emission is confined to surface region
  - Excellent for characterizing surfaces!



Taken from L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag

X-Ray Spectra

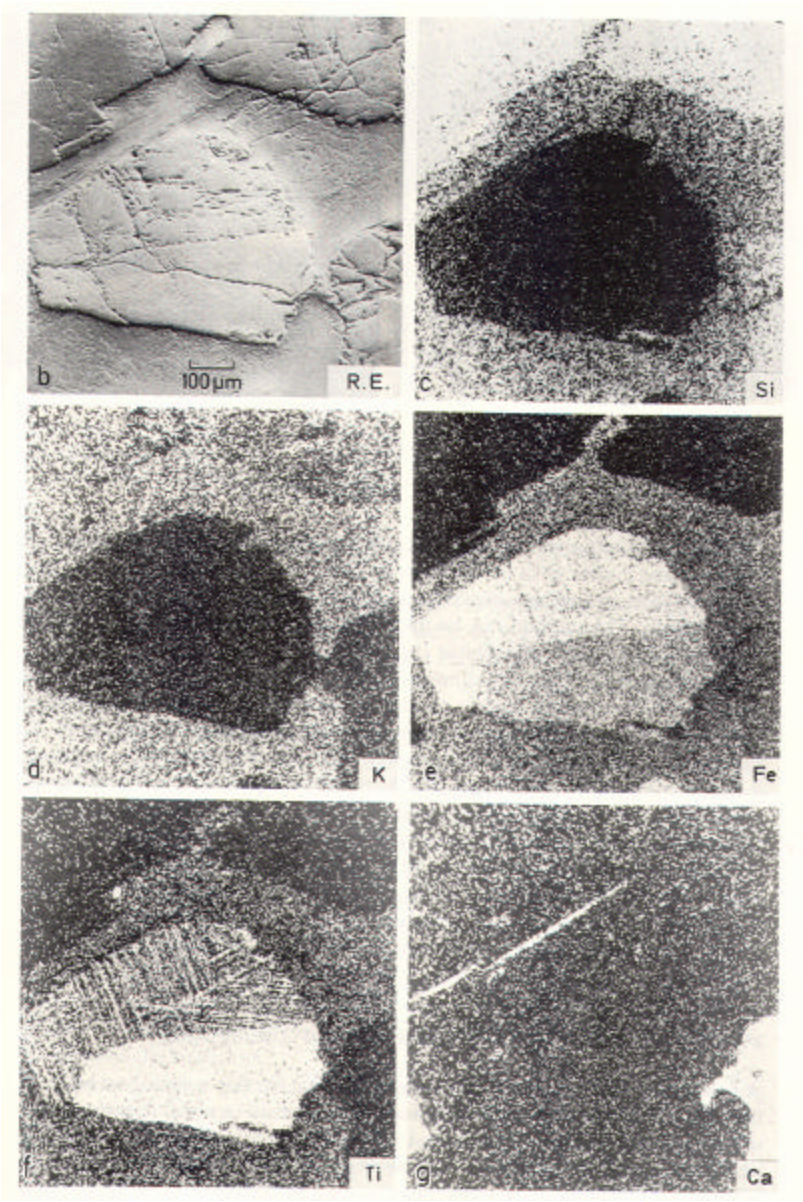


- Known as EDX or EDS (Energy Dispersive X-ray Spectra)
- Each peak corresponds to a different energetic transisition
- X-Ray emission peaks can by identified automatically using special software programs
- Bulk technique, not surface sensitive
- Prone to errors

Taken from L. Reimer,
Scanning Electron Microscopy,
2nd edition, Springer Verlag

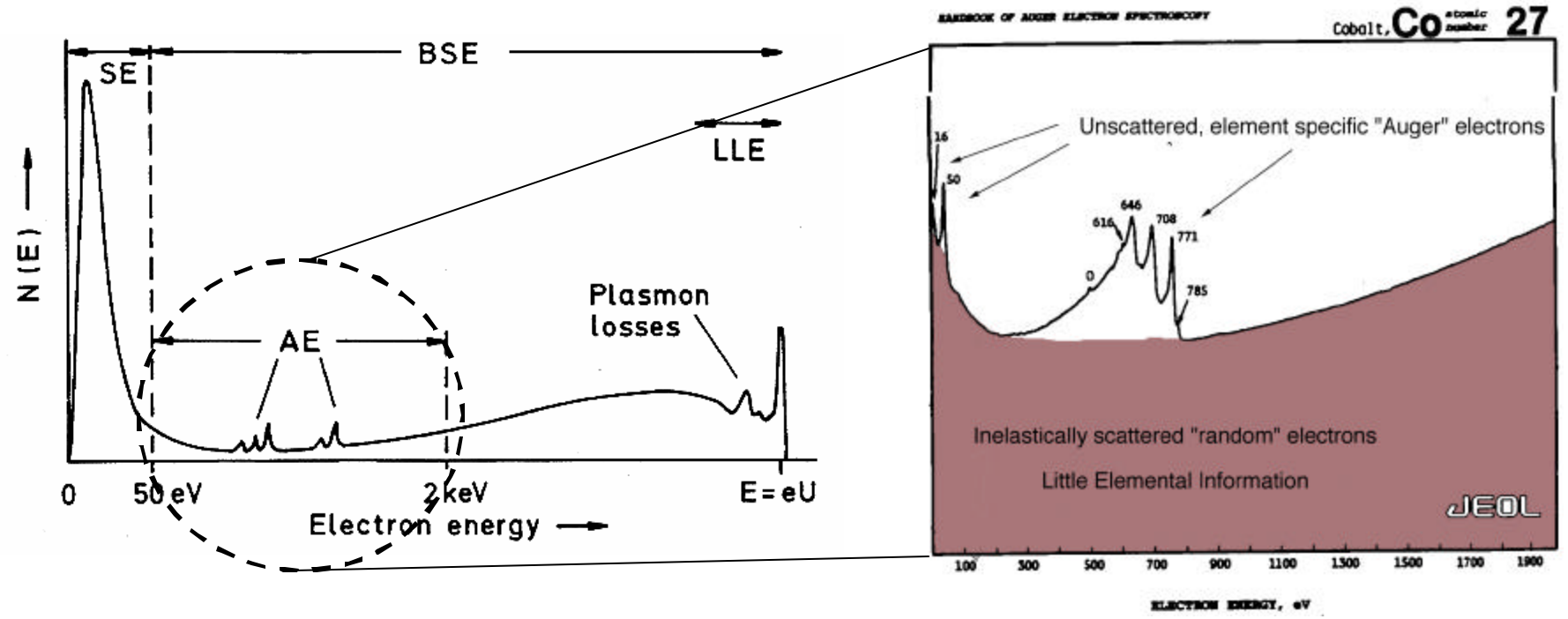
EDS Mapping

- Spectra can be acquired at each point in an image
- Different elements can be assigned different gray values or colors
- Very useful for gaining detailed compositional information
- Not high resolution due to scattering in substrate



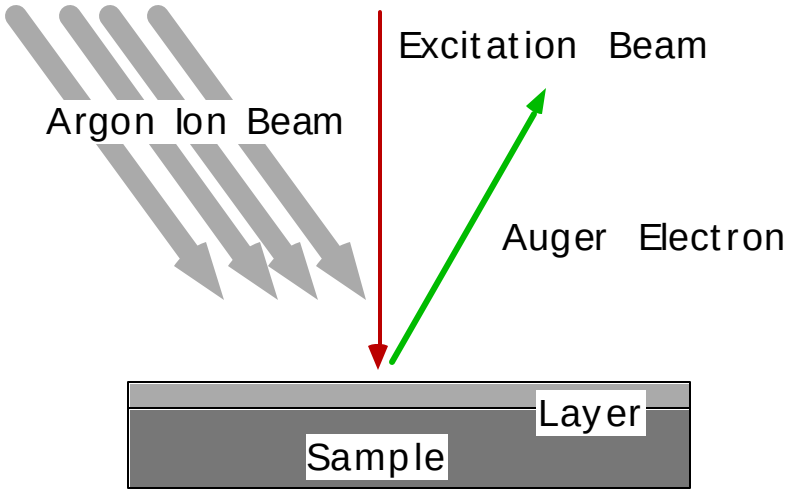
Taken from L. Reimer, *Scanning Electron Microscopy*, 2nd edition, Springer Verlag

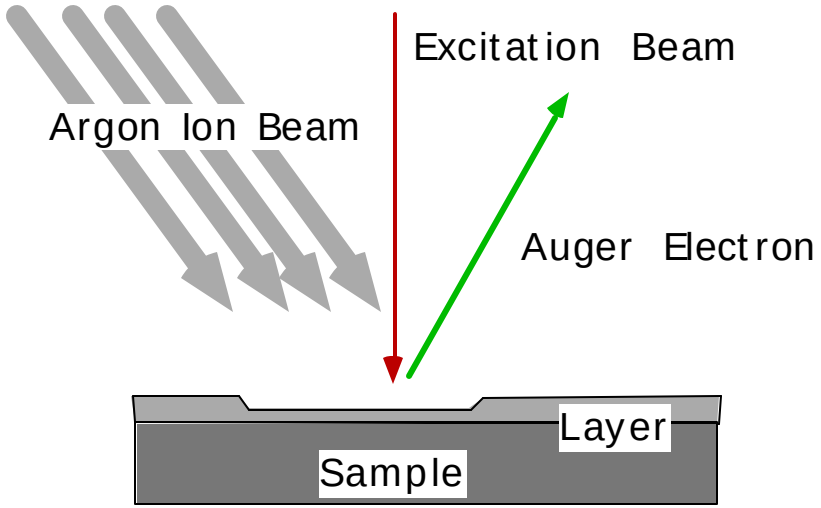
Auger Spectra



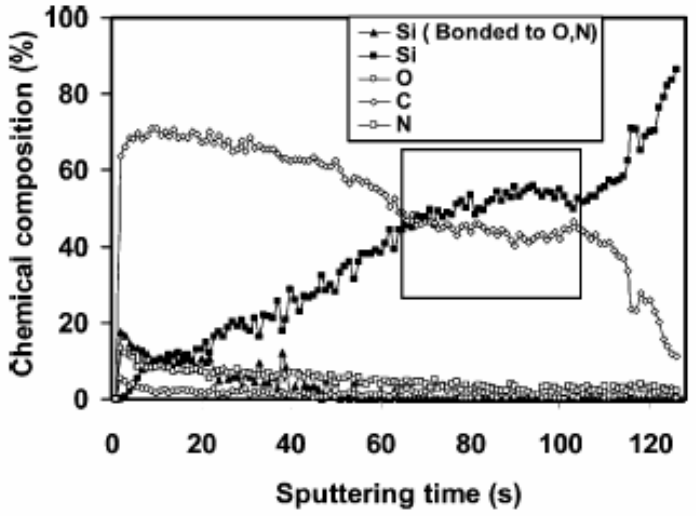
- Can be obtained using SEM-like instrument referred to as a scanning Auger microprobe
- Can take Auger spectra at specific locations
- Can be used to create detailed surface composition maps

Depth Resolved Auger





- Use Ar ion beam to sputter away material during analysis
- Creates composition Vs. time plot
- Below: Composition of Si substrate / carbon nanofiber interface



Yang, et al, Nano Lett., Vol. 3, No. 12, 2003

Cheat Sheet

To increase depth of field:

- Increase working distance
- Decrease aperture size
- Use collimated beam mode

Surface Sensitivity:

- Lower accelerating voltage
- Use the right detector!

High Resolution:

- Reduce spot size
  - smaller aperture
  - or condenser lens
- Short working distance
- Slower scan rate
- Use the right detector!

Analysis:

- Use as high of a beam current as your sample can take

Better S/N:

- Larger aperture
- Slower scan rate
- Use collimated beam mode

Charging or Insulating Sample:

- Lower accelerating voltage (find E2 voltage)
- Use a VP system
- Sputter coat with Au-Pd (last option)

Focus and Stigmation:

- Adjust focus and stigmation at higher mags than working mag
- Adjust to best focus
- Adjust 1 or both stigmator controls
- Iterate between focus and stigmation (roundish structures helpful in adjusting stigmation)
- Center apertures after changes in accelerating voltage or aperture

Sample Preparation

- Advantage of SEM - little preparation needed!
- Mount on appropriate stub making electrical contact
  - Use conductive Cu adhesive or clips to secure sample
- Various mounts:
  - Whole wafer mounts
  - Small piece mounts
- Cleave to look at cross-section
  - Can also polish or use focused ion beam
- If samples charge and low voltage or VP mode do not work, sputter coat with metal, e.g., Au-Pd
- Mark specimen with a small Cu tape arrow (cut the corner off of some Cu tape) and stick it close to your region of interest under an optical microscope

References

- **SEM:**
  - Scanning Electron Microscopy and X-Ray Microanalysis, Goldstein, Newbury, Echlin, Joy, Romig, Lyman, Fiori, and Lifshin
  - Scanning Electron Microscopy, L. Reimer, Springer Verlag, #45 in the optical sciences series
  - Free reference guide on using an SEM: http://www.jeol.com/sem_/docs/sem_guide/guide.pdf