Analytical Methods for Materials

Lesson 21

Electron Microscopy and X-ray Spectroscopy

Suggested Reading

►**Leng, Chapter 3, pp. 83-126; Chapter 4, pp. 127-160; Chapter 6, pp. 191-219**

- P.J. Goodhew, J. Humphreys and R. Beanland, Electron Microscopy and Analysis, 3rd Edition, (CRC Press, 2001) pp. 16-213.
- Brandon and Kaplan, Chapter 5, pp.261-331; Chapter 6, pp. 332-390

About these notes

- This is a very long module.
- This particular set of lecture notes represents an abbreviated introduction to electron microscopy.
- For the student, this set of notes only scratches the surface. You are still responsible for the majority of chapters 3, 4, and 6 in your text.

What are electron microscopes?

Scientific instruments that use a focused beam of electrons to examine objects with much higher magnification and resolution.

What is electron microscopy?

- Electron microscopy is the science and technology of using an electron beam to form a magnified image.

- Advantages:
  - The use of electrons rather than light provides a ~1000× increase in resolving power (i.e., ability to focus fine details) over light.

- Disadvantages:
  - High cost
  - Time commitment
  - Small areas of analysis

Magnification and Resolution

- **Magnification** = how large an object can be made (and still resolved).
  - $$\text{magnification} = \frac{\text{image size}}{\text{object size}}$$

- **Resolution** = the closest distance between two points that can clearly be resolved as separate entities through the microscope.
  - $$r_o = \frac{d_1}{2} = \frac{0.61\lambda}{\mu \sin\alpha} = \frac{0.61\lambda}{NA}$$

λ = wavelength of illuminant
α = semi-angle
μ = index of refraction
NA = numerical aperture

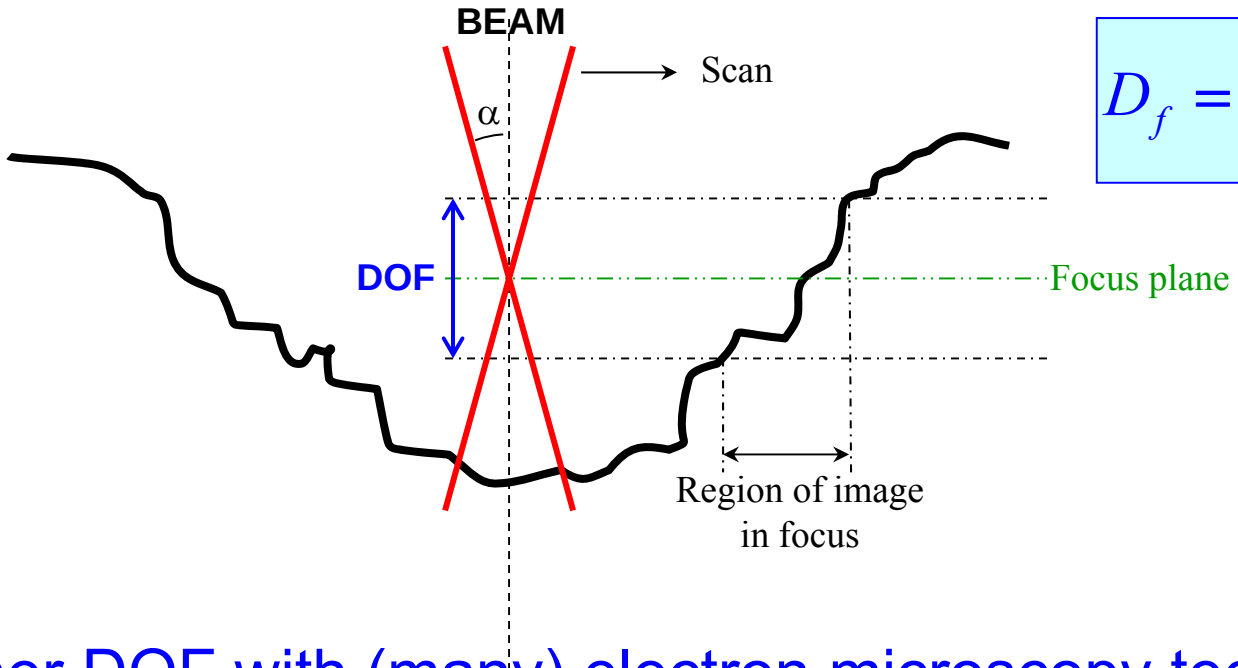
Advantages of electron microscopy over optical microscopy

- Higher magnifications in electron microscopes than you can in light microscopes.

- Smaller wavelengths of radiation leads to higher resolving power.

Depth of Field

- How much of the object that we are looking at remains in focus at the same time.
- DOF is a function of magnification, α, and probe size



$$D_f = \frac{1.22\lambda}{\mu \sin\alpha \tan\alpha}$$

- Higher DOF with (many) electron microscopy techniques than light.

What information can we obtain from electron microscopes?

- Topography
  - Surface features of an object. "How it looks."
- Morphology
  - Size and shape of particles making up object.
- Composition
  - Relative amount of elements and compounds making up the object.
- Structure
  - Crystallography. How atoms are arranged in the object
  - Substructure. Defect type and content.

Primary types of electron microscopes

Transmission electron microscope (TEM)

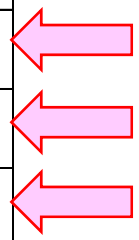
Scanning electron microscope (SEM)

History of electron microscopes

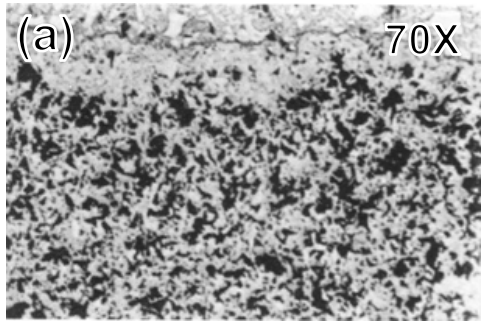
- Developed due to limitations of light microscopes
  - **LOM**: ~1000x magnification; 0.2 μm (200 nm) resolution
- TEM was developed first.
  - M. Knoll and E. Ruska, 1931
  - Patterned “exactly” like a LOM. Uses electrons rather than light.
- SEM came later.
  - 1942

How the major types of electron microscopes compare

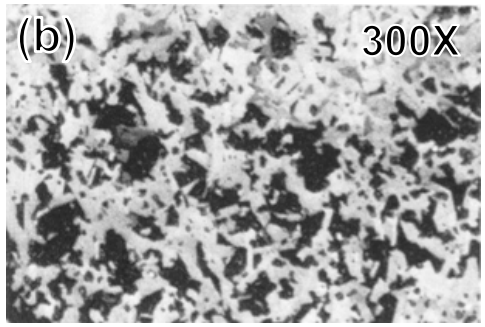
FEATURE	Optical Microscope	SEM	TEM
Uses	Surface morphology and sections (1-40 μm)	Surface morphology	Sections (40-150 nm) or small particles on thin membranes
Source of Illumination	Visible light	High-speed electrons	High-speed electrons
Best resolution	~200 nm	3 – 6 nm	0.2 nm
Magnification range	2 – 2,000×	20 – 150,000×	500 – 1,000,000×
Depth of field	0.002-0.05 nm (NA=1.5)	0.003-1 mm	0.004-0.006 mm ($NA=10^{-3}$)
Lens type	Glass	Electromagnetic	Electromagnetic
Image ray-formation spot	On eye by lens	On CRT by scanning device	On phosphorescent screen by lens
Information generated	Phases Reflectivity	Topography Composition Crystal orientation	Crystal structure Crystal orientation Defects Composition
Limiting Factors	Wavelength of light	Brightness, signal/noise ratio, emission volume	Lens quality

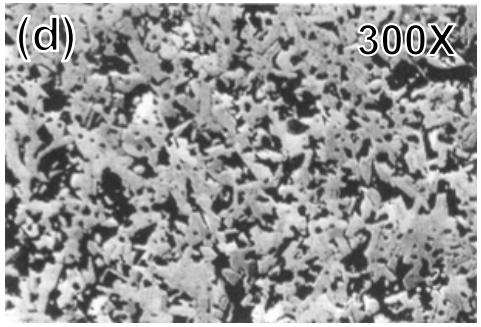


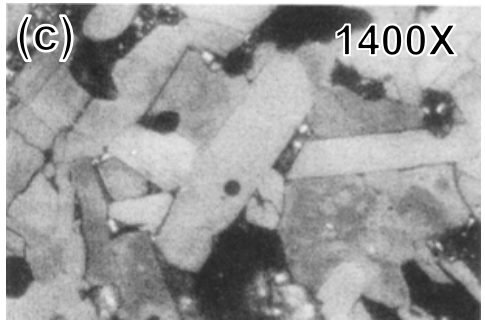
Electron Microscopes versus Optical Microscopes

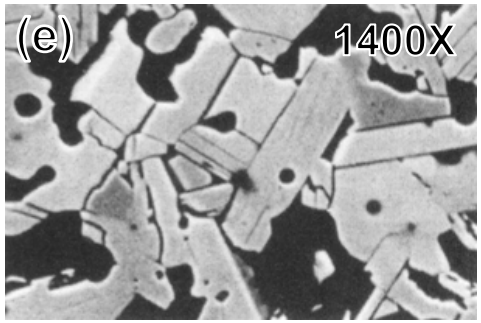


SEM micrographs









LOM micrographs

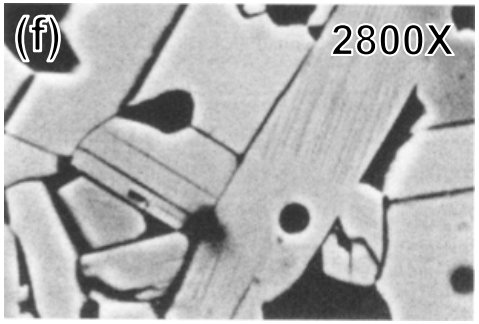
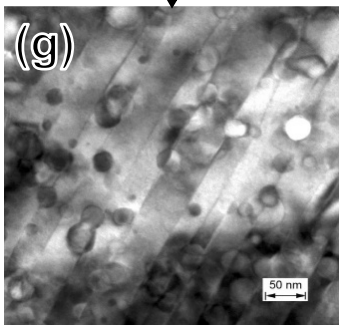


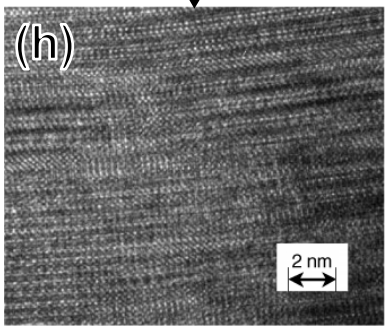
Figure A series of optical, SEM and TEM micrographs of the high temperature superconductor $YBa_2Cu_3O_7$ at increasing magnification. Original magnifications: (a) 70×; (b) and (d) 300×; (c) and (e) 1400×; and (f) 2800×. (g) TEM image and (h) HR-TEM image.

Images (a) – (f) reproduced from Goodhew, Humphreys & Beanland, Electron Microscopy and Microanalysis, 3rd Edition, Taylor & Francis, London, 2001. Images (g) and (h) reproduced from V. F. Solovyov et al., *Superconductor Science and Technology* 20 (2007) pp. L20 – L23.

TEM micrograph

HRTEM micrograph





How do electron microscopes work?

- Form a stream of electrons and accelerate them towards a specimen using a positive electrical potential.

- Use apertures and magnetic lenses to focus the stream onto the sample.

- Interactions occur inside the irradiated area of the sample that we *collect in a suitable detector*.

Illumination sources
(aka, electron guns)

- Thermionic
  - Tungsten
  - LaB_6
- Field Emission
  - Cold FEG
  - Schottky FEG

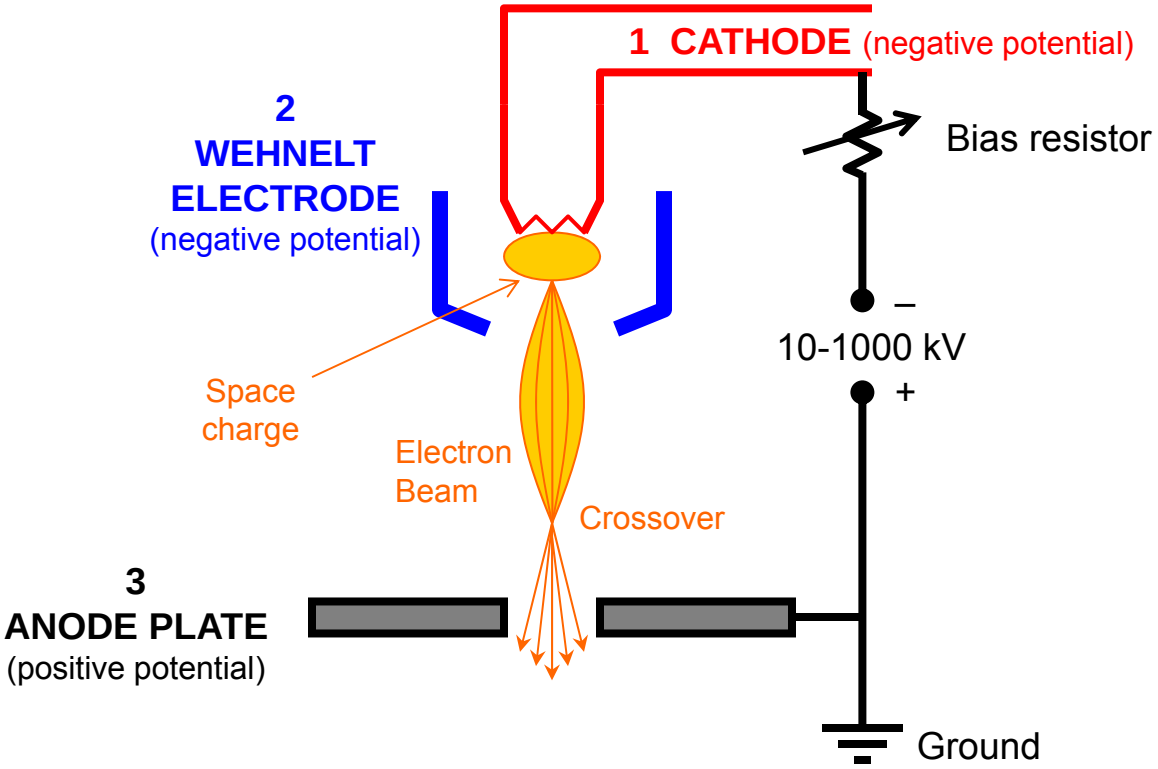
Table 3.3 Comparison of Electron Guns (From Leng, p. 82)

	Tungsten filament	LaB_6	Field emission	
			Thermal	Cold
Operation temperature (K)	~2800	~1800	1600 ~ 1800	~300
Brightness[a] At 200 kV (A cm^{-2} sr)	$\sim 5 \times 10^5$	$\sim 5 \times 10^6$	$\sim 5 \times 10^8$	$\sim 5 \times 10^8$
Requirement to vacuum (Torr[b])	10^{-4}	10^{-6}–10^{-7}	10^{-9}	10^{-9}–10^{-10}

[a] Intensity emitted per unit cathode surface in unit solid angle.

[b] 1 torr = 133 Pa.

Anatomy of an electron source
(*i.e.*, electron gun)



Electron Lenses

- Use electrostatic or electromagnetic fields to focus beams of charged electrons.

- ELECTROMAGNETIC – Most lenses are of this type. Consist of Cu wire coils around soft Fe cores. Sometimes an Fe pole-piece is used to “shape” the field.

- ELECTROSTATIC – Unusual. Only common example is the Wehnelt aperture in the electron gun.

Electromagnetic Lenses

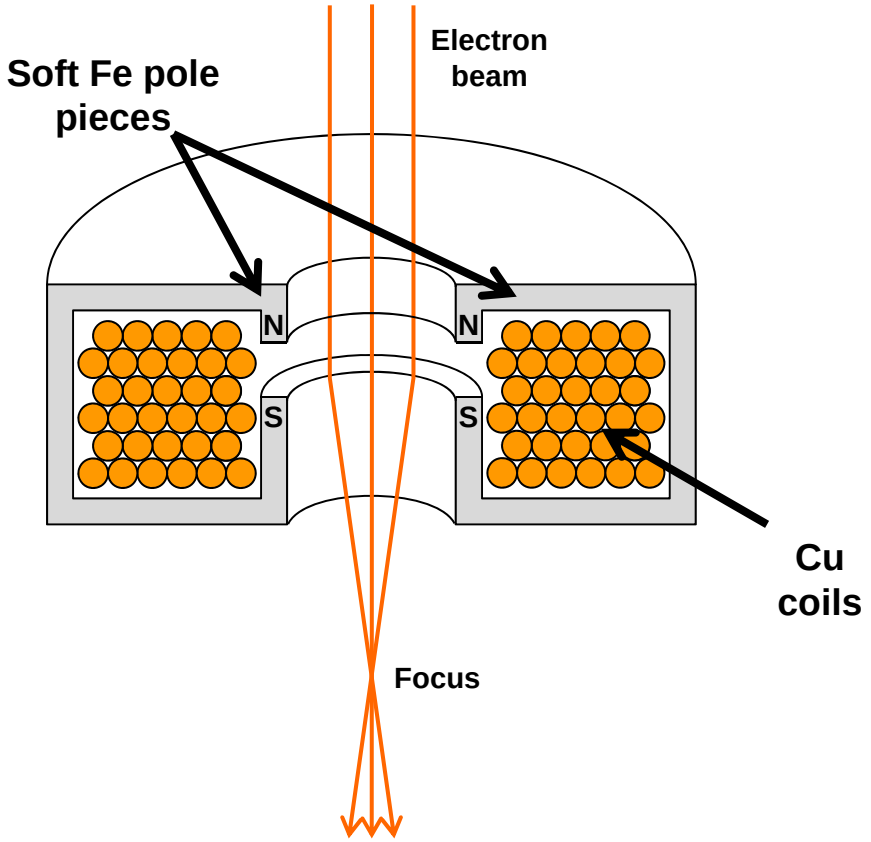


Figure 3.3 Structure of an electromagnetic lens. The magnetic field concentrated between the N and S poles deflects the electron beam. Adapted from Leng, p. 83.

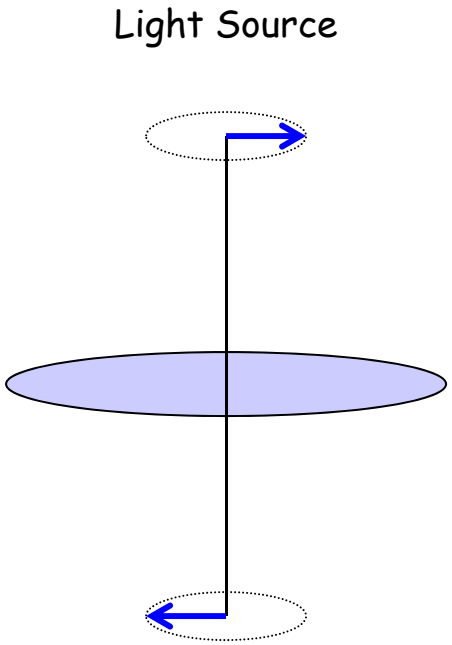
Consists of a soft magnetic core (case) that encloses a solenoid.

Poles located at annular opening in case.

Concentrates magnetic field between poles.

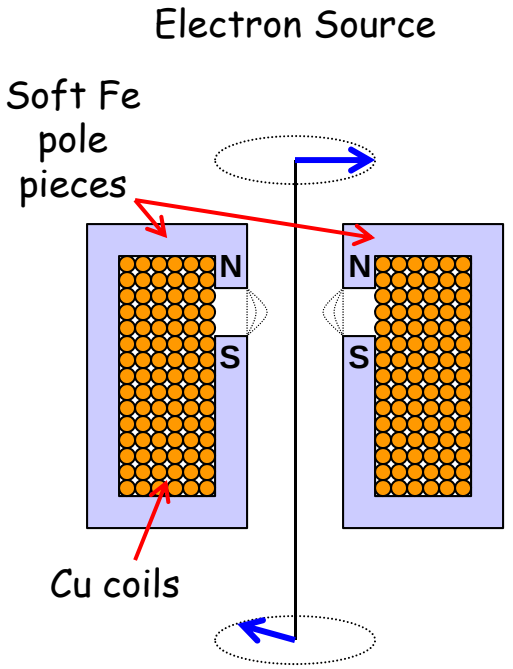
Electron Lenses

OPTICAL LENS



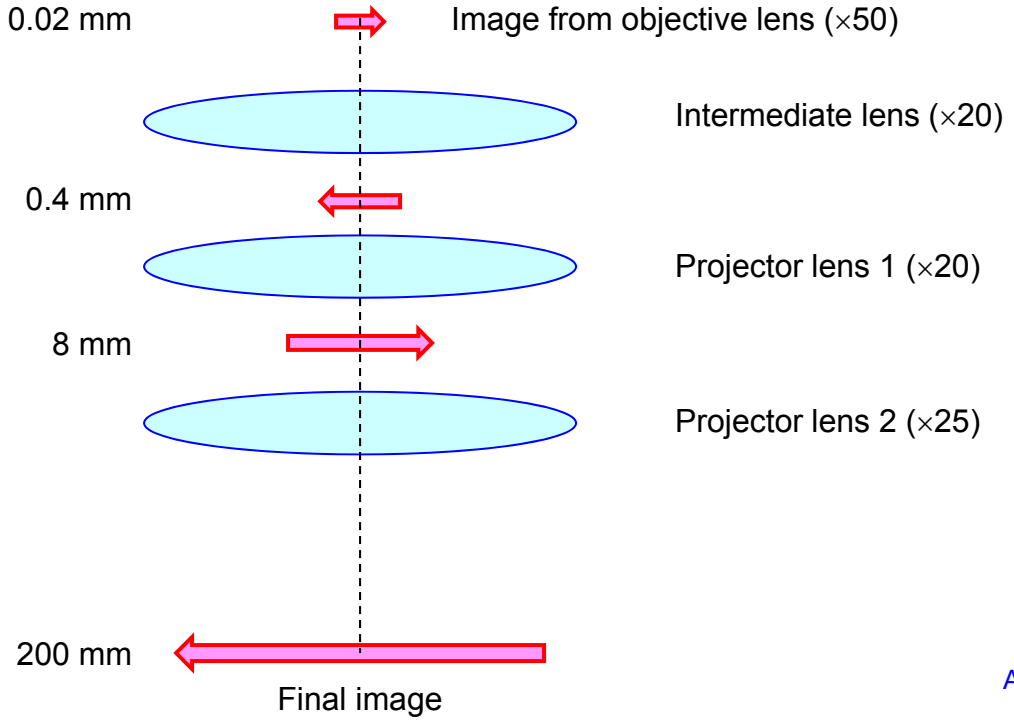
ELECTROMAGNETIC lenses focus the electron beam to as small a spot as is possible. They are equivalent to convex lenses in optical lens systems.

MAGNETIC LENS

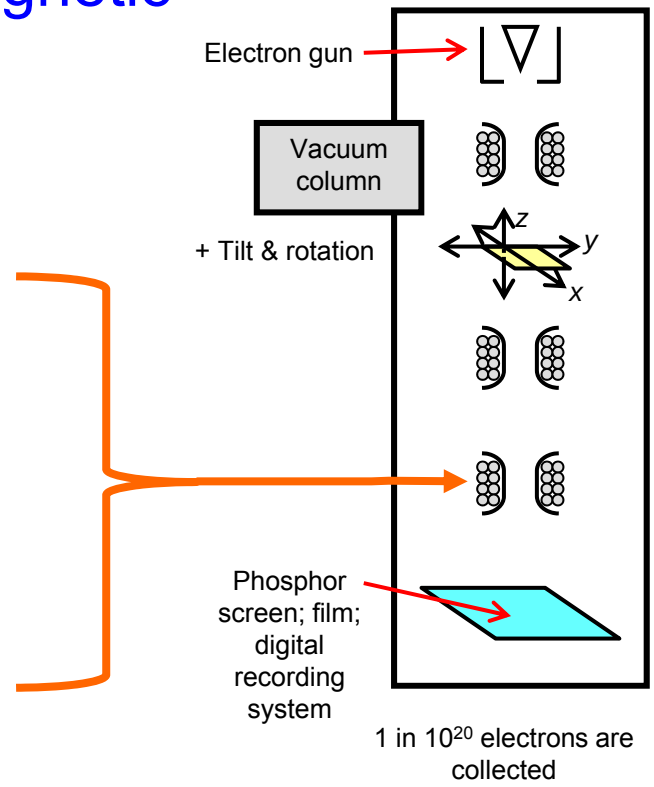


Imaging System (lenses)

- We use combinations of electromagnetic lenses to increase magnification.



Final magnification = 50 × 20 × 20 × 25 = ×500,000



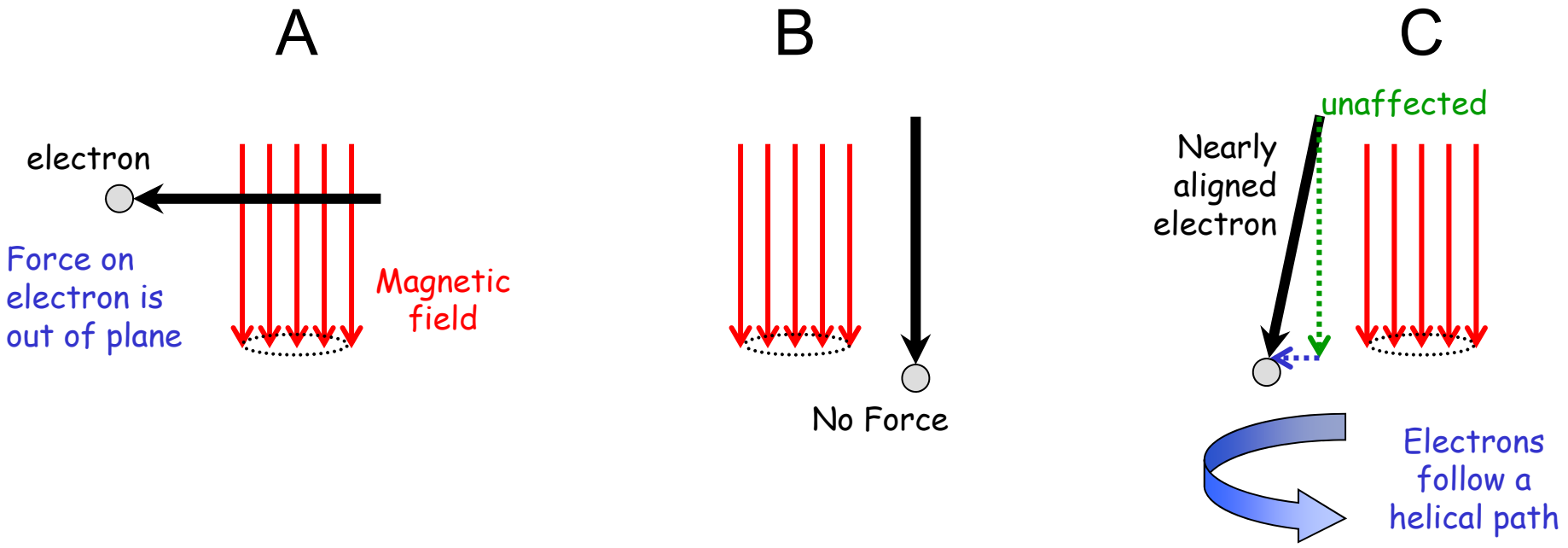
Adapted from B.D. Huey, MMAT322 Lecture Notes, University of Connecticut (2005)

AND

★ http://www.matter.org.uk/tem/lenses/projector_lens.htm

Electrons in Magnetic Fields

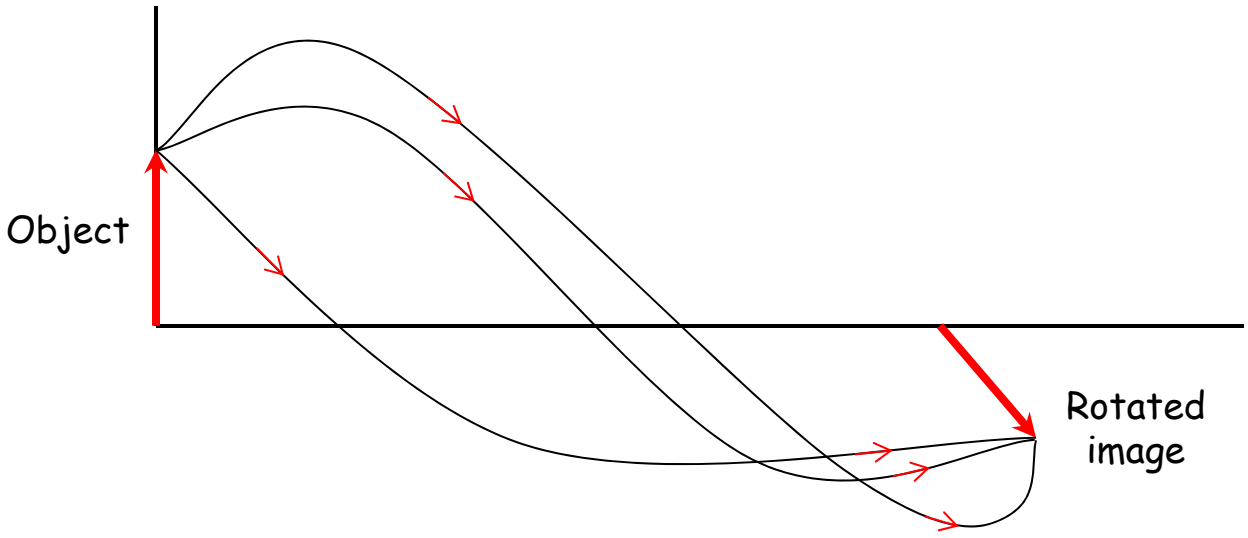
A. Electrons moving through a perpendicular magnetic field experience perpendicular forces.
B. Electrons moving parallel to a magnetic field are unaffected.
C. Electrons moving nearly parallel to a magnetic field adopt a helical path around the direction of the magnetic field.

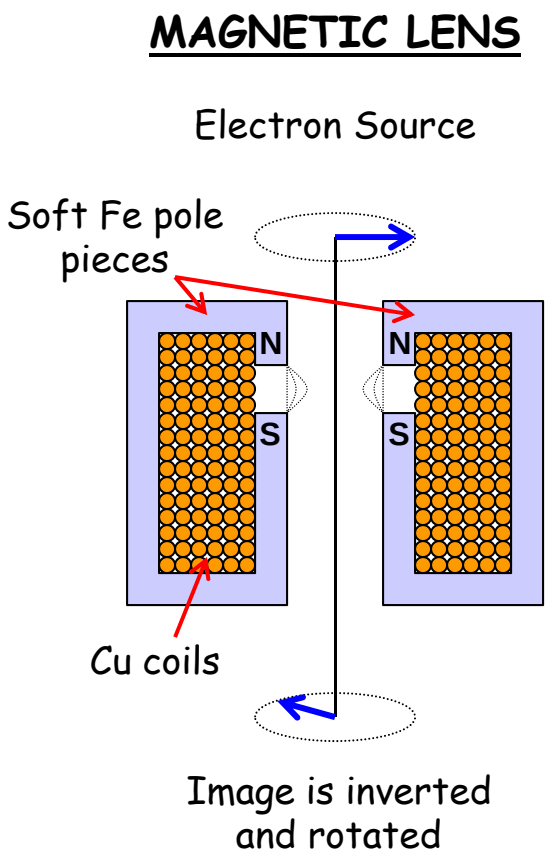


Adapted from
B.D. Huey, MMAT322 Lecture Notes, University of Connecticut (2005)

Trajectories in Electromagnetic Lenses

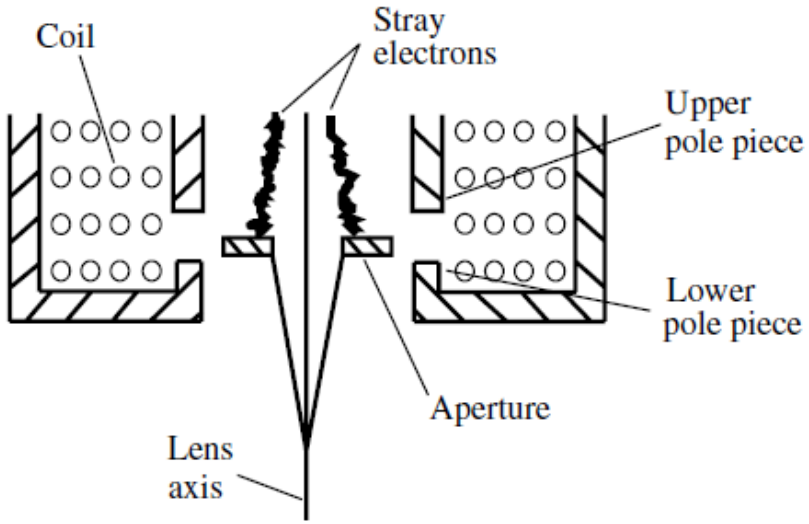
- When you adjust the magnification (and the focal length), you modify the lens strength by adjusting the current in the electromagnetic lens coils.
- Since the magnetic field is changed, so are the helical trajectories.
- Leads to image rotation in TEM (which must be corrected for or calibrated on older microscopes).





Apertures

- Similar to light microscopes
- Essentially a piece of metal with a hole in it.
- Used to limit scattering and/or to select the diffracted or non-diffracted beams.



(From Leng)

Figure 3.5 The objective aperture located in the electromagnetic lens.

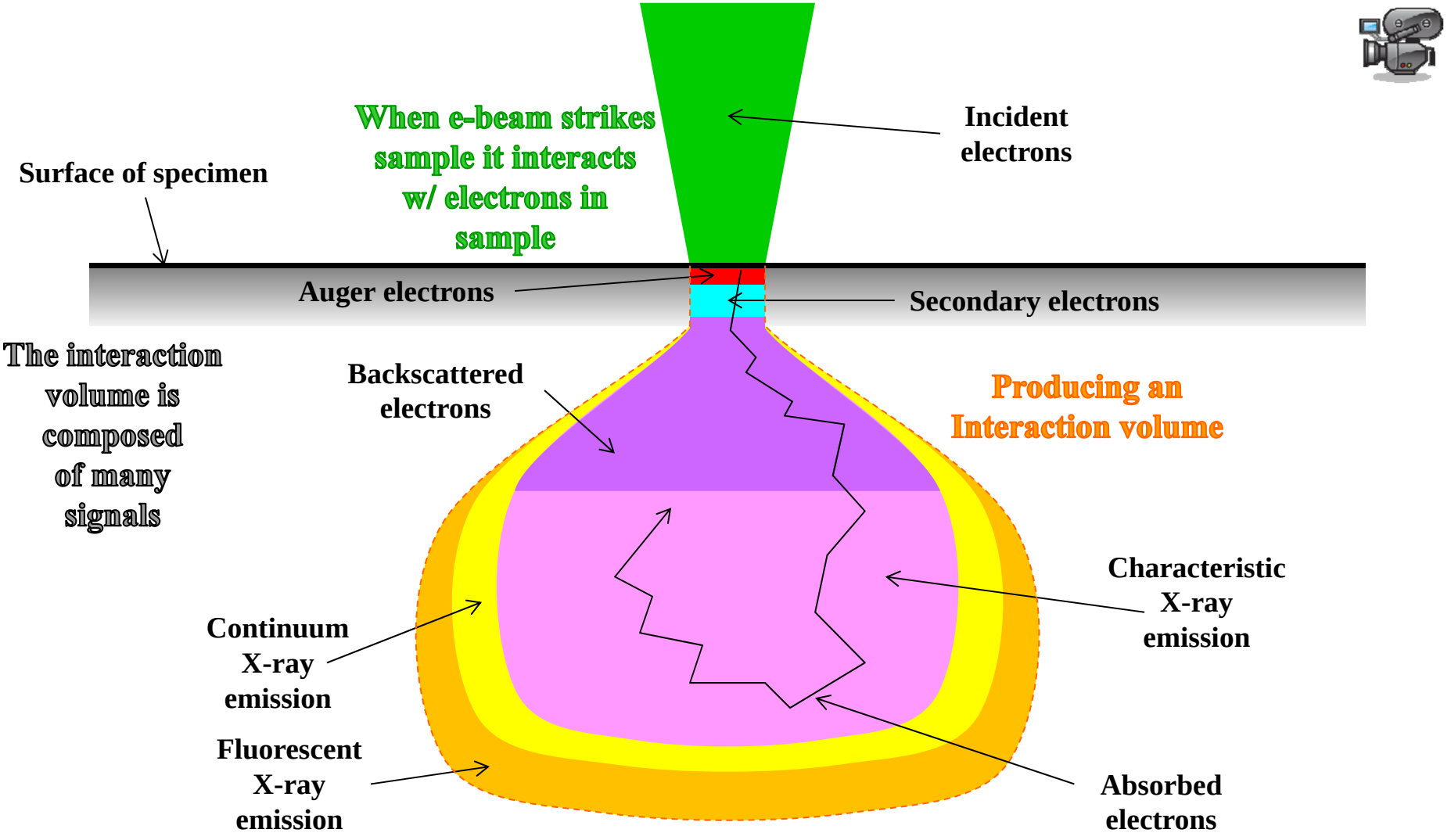
Types of Apertures

- Objective
- Condenser
- Selected area
- Each has a specific function

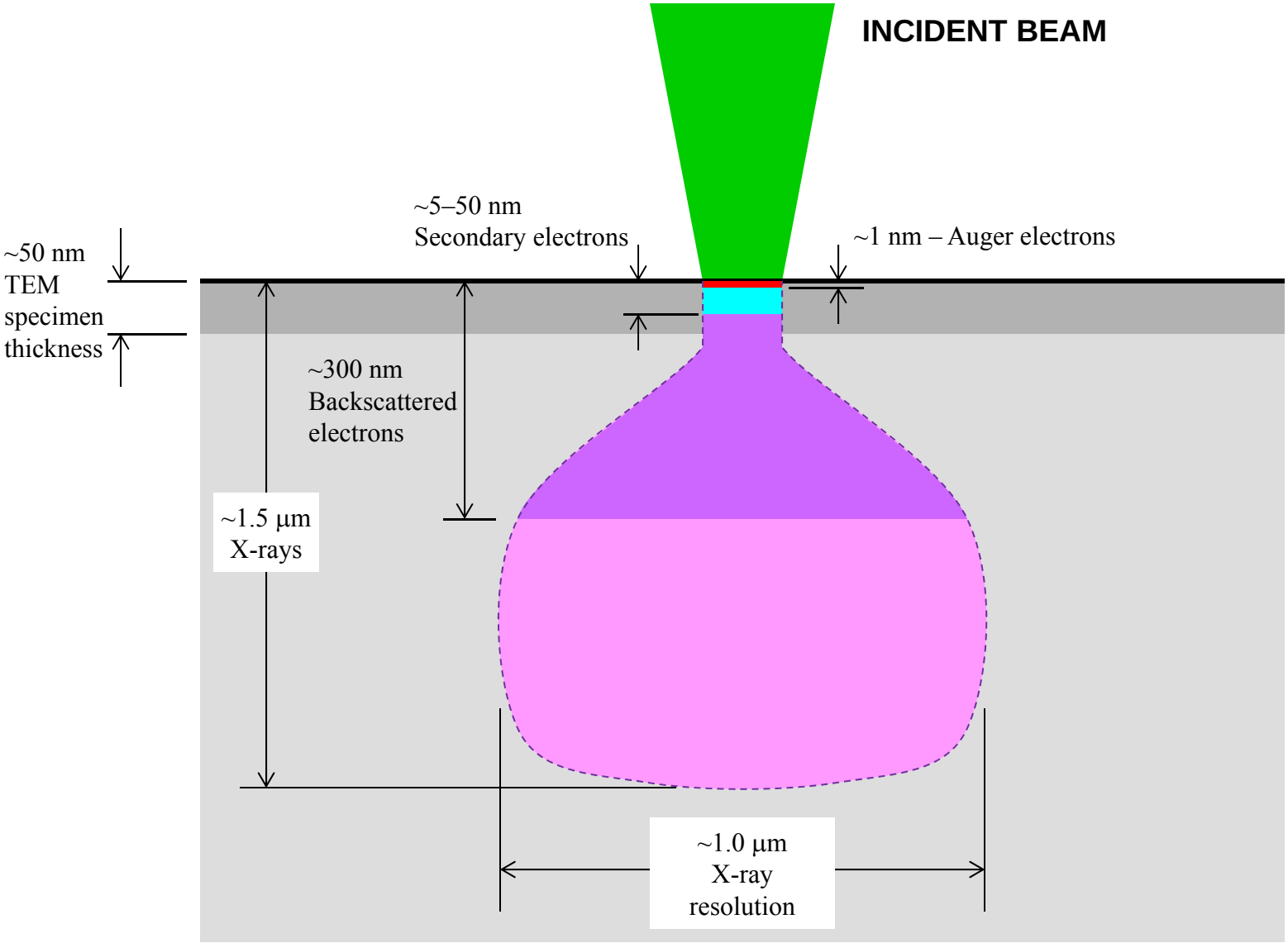
WHAT HAPPENS WHEN ELECTRONS INTERACT WITH A MATERIAL?

Interaction Volume

- Represents the region penetrated by electrons.

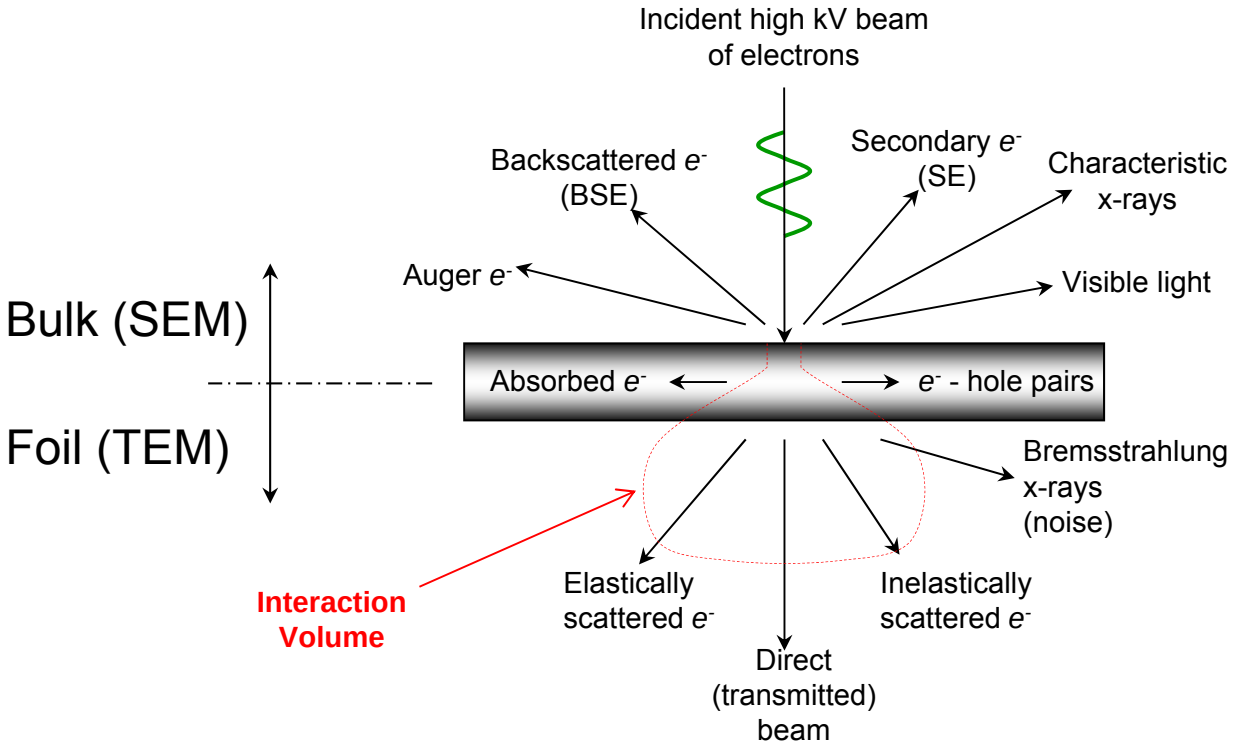


Signals originate from different depths inside of the sample
Signals must escape the sample to be detected



Z = 29 (Cu), Accelerating voltage = 20kV

Signals and Electron Microscopy



Important signals in analytical electron microscopy

Backscattered electrons (BSE)

- **Formation**
  - Caused when incident electrons collide with an atom in a specimen that is nearly normal to the path of the incident beam.
  - Incident electron is scattered backward ("reflected").

- **Use**
  - Imaging and diffraction analysis in the SEM.
  - Production varies with atomic number (Z).
  - Higher Z elements appear brighter than lower Z elements.
  - Differentiate parts of specimen having different atomic number

Backscattered electrons are not as numerous as others. However, they generally carry higher energies than other types of electrons.

Secondary Electrons (SE)

- **Formation**
  - Caused when an incident electron "knocks" and inner shell electron (e.g., *k*-shell) out of its site.
  - This causes a slight energy loss and path change in the incident electron and ionization of the electron in the specimen.
  - The ionized electron leaves the atom with a small kinetic energy (~5 eV)
- **Use**
  - IMAGING!
  - Production is related to topography. Due to low energy, only SE near the surface can exit the sample.
  - Any change in topography that is larger than the sampling depth will change the yield of SE.

More abundant than other types of electrons. They are electrons that escape the specimen with energies below ~50eV

X-rays

- **Formation**
  - Same as AE. Difference is that the electron that fills the inner shell emits energy to balance the total energy of the atom.

- **Use**
  - X-rays will have characteristic energies that are unique to the element(s) from which it originated.
  - Collect and sort signals according to energy or wavelength to yield compositional information.
    - Energy Dispersive X-ray Spectroscopy (EDS)
    - Wavelength Dispersive X-ray Spectroscopy (WDS)

Also foundation of XPS (X-ray photoelectron spectroscopy). XPS can be used to determine the "state" of an atom and to identify chemical compounds.

Transmitted electrons

- Used in Transmission Electron Microscopy (TEM)

- Can be used to determine:
  - thickness
  - crystallographic orientation
  - atomic arrangements
  - phases present
  - etc.

Auger Electrons (AE)

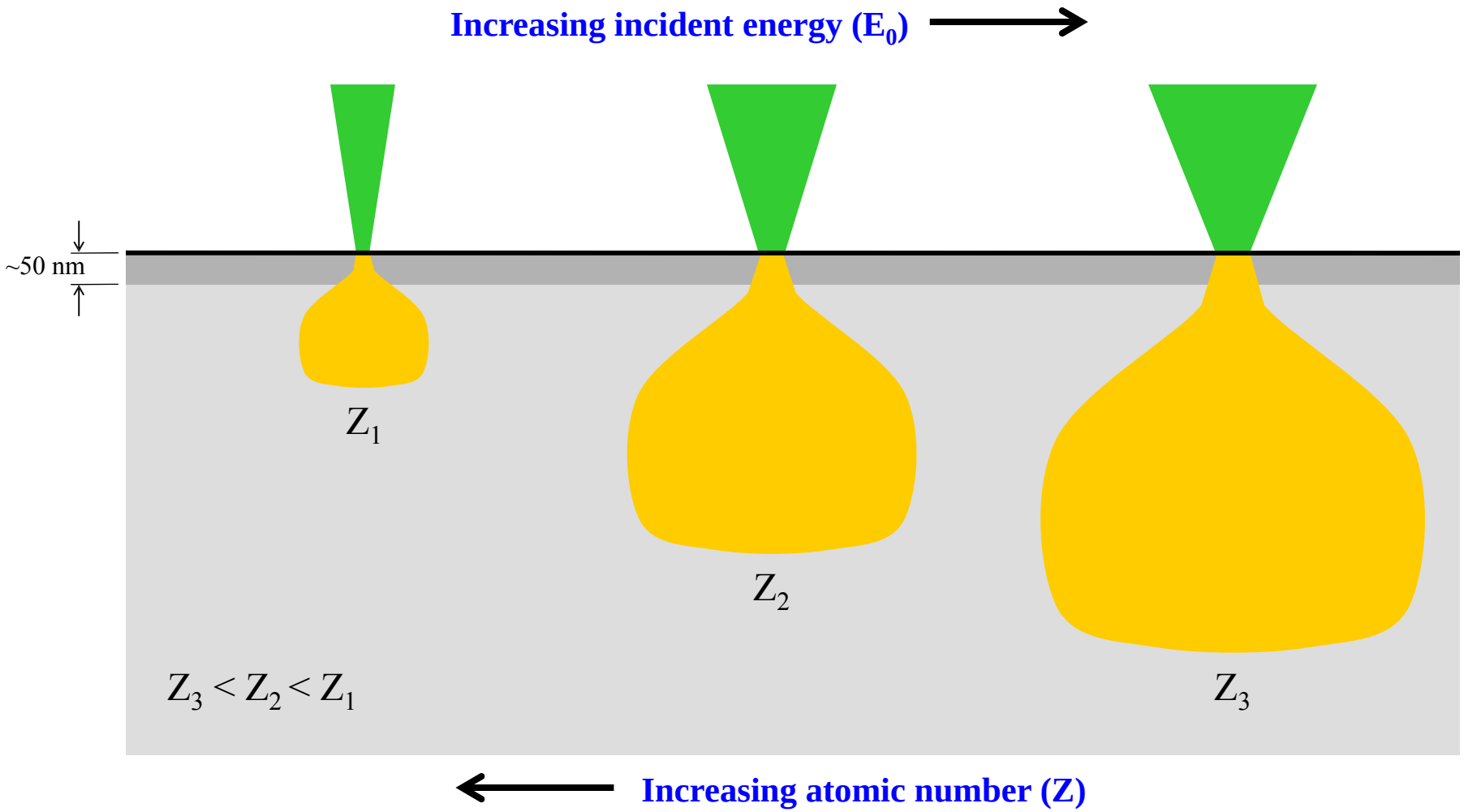
- **Formation**
  - De-energizing of the atom after a secondary electron is produced.
  - During SE production, an inner shell electron is emitted from the atom leaving a vacancy.
  - Higher energy electrons from the same atom can fall into the lower energy hole. This creates an energy surplus in the atom which is corrected by emission of an outer shell (low energy) electron.

- **Use**
  - AE have characteristic energies that are unique to each element from which they are emitted.
  - Collect and sort AE according to energy to determine composition.
  - AE have very low energy and are emitted from near surface regions.
  - SURFACE SCIENCE!

Size of specimen Interaction Volume

- Depends upon:
  - Z of material being examined
    - higher Z materials absorb more electrons and have smaller interaction volume
  - Accelerating Voltage
    - higher voltages penetrate further into the specimen and generate larger interaction volumes
  - Angle of incidence of electron beam
    - larger angle leads to a smaller interaction volume

Effects of accelerating voltage and Z on interaction volume



- Interaction volume is larger for materials that have lower atomic numbers and for higher incident beam energies!

“Instruments of the trade”

- Primary Instruments
  - Transmission Electron Microscope (TEM)
  - Scanning Electron Microscope (SEM)

- Variants
  - Electron Probe Microanalyzer (EPMA); i.e., “microprobe”
  - Scanning Transmission Electron Microscope (STEM)
  - Environmental SEM (ESEM) {aka variable pressure SEM}
  - High Resolution TEM (HRTEM)
  - High Voltage TEM (HVTEM)
  - DualBeam™ FIB
  - etc...

- There are others as well. All have specific purposes.

SEM

TEM

EPMA

Tabletop
SEM

XPS / ESCA

DB-FIB

He-Ion Microscope

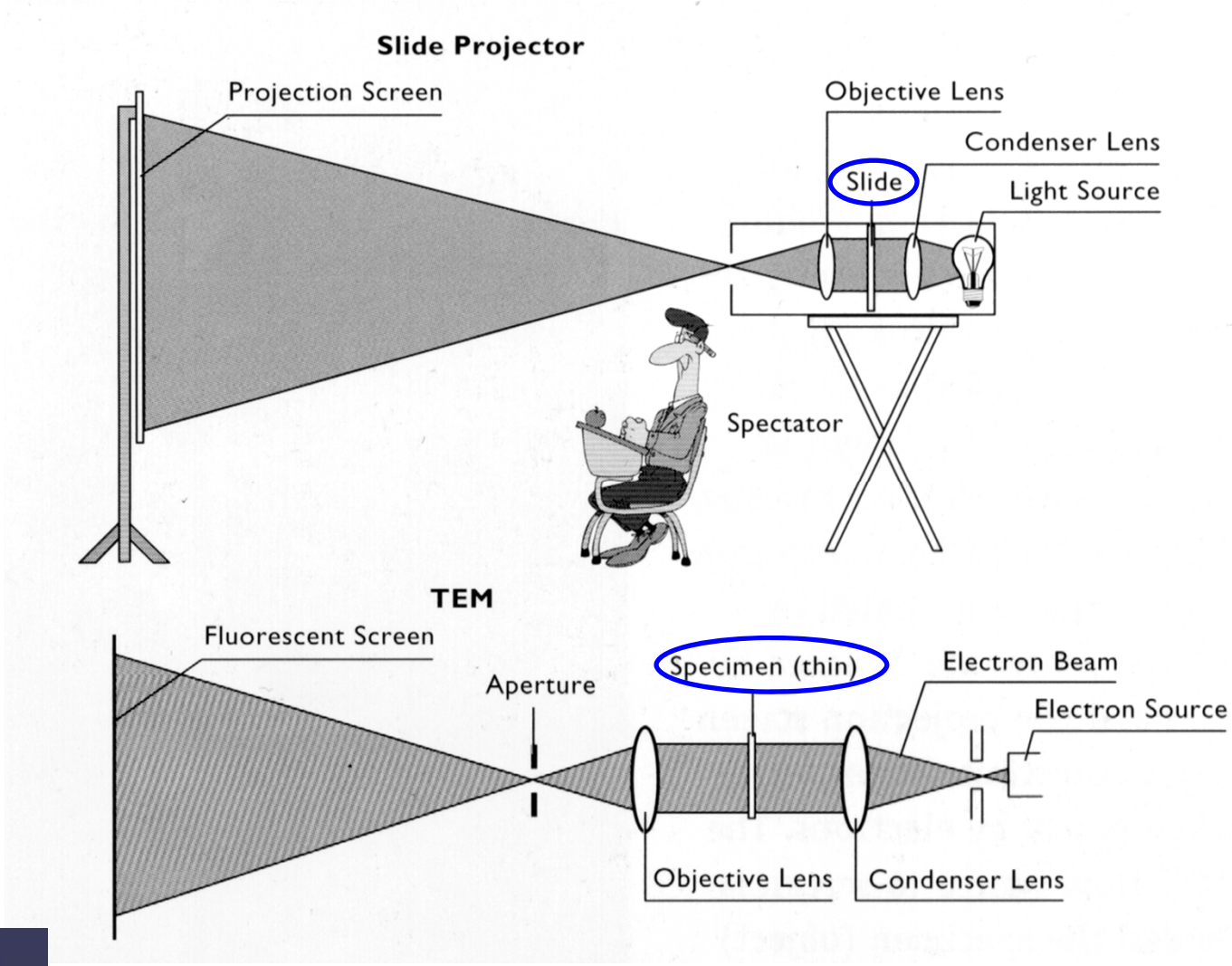
LET'S CONSIDER THE TEM FOLLOWED BY THE SEM

TEM

- Patterned after transmission optical microscopes

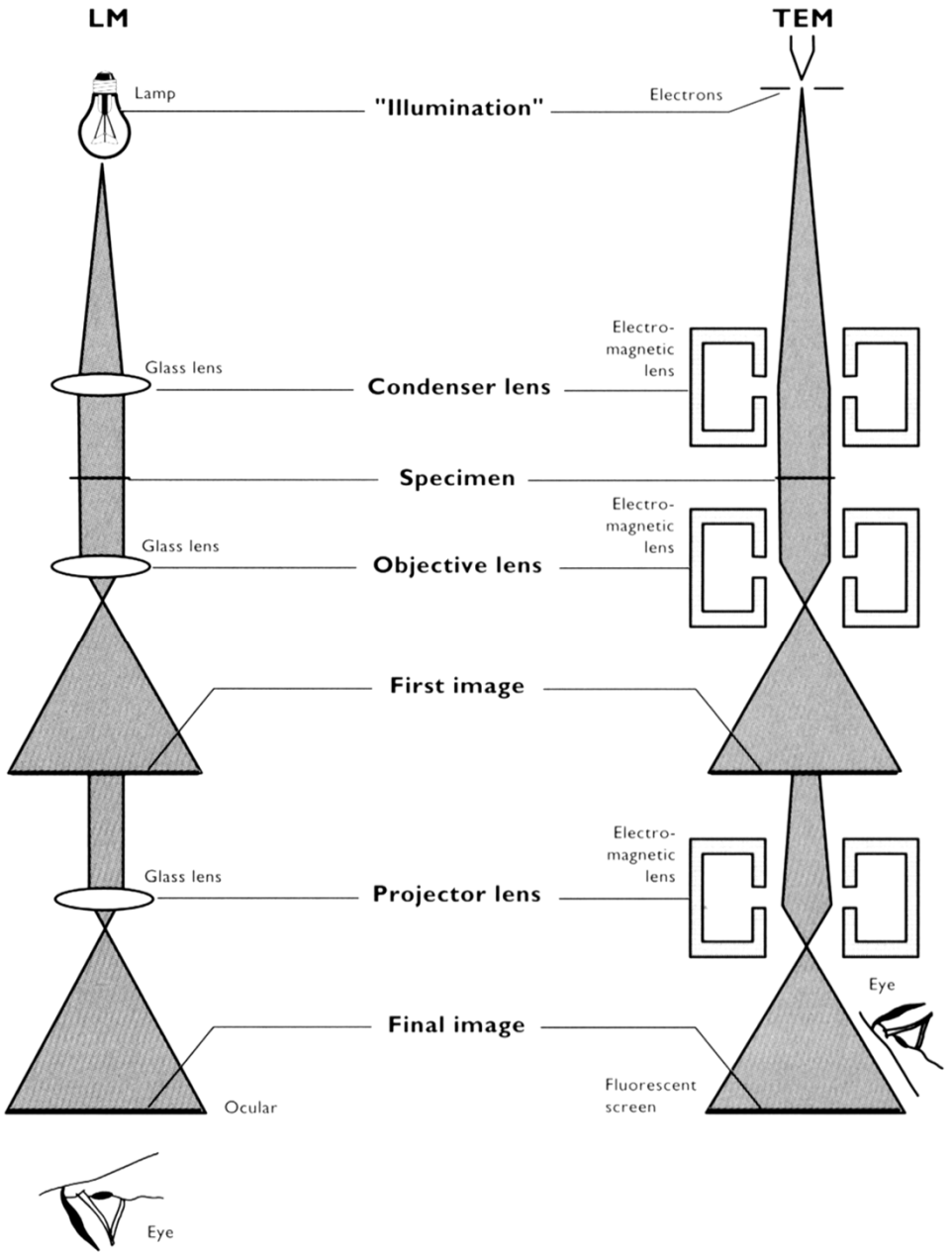
- Yield Following Information:
  - Morphology
    - Size shape and arrangement of particles, precipitates, etc.
  - Crystallographic information
    - Atomic arrangement
  - Compositional Information
    - If proper detector is present

TEM is a projection device



From the lecture notes of Hendrik O. Colijn, OSU Campus Electron Optics Facility,
www.ceof.ohio-state.edu/classes/MSE605.ppt

Similarity of LM and TEM



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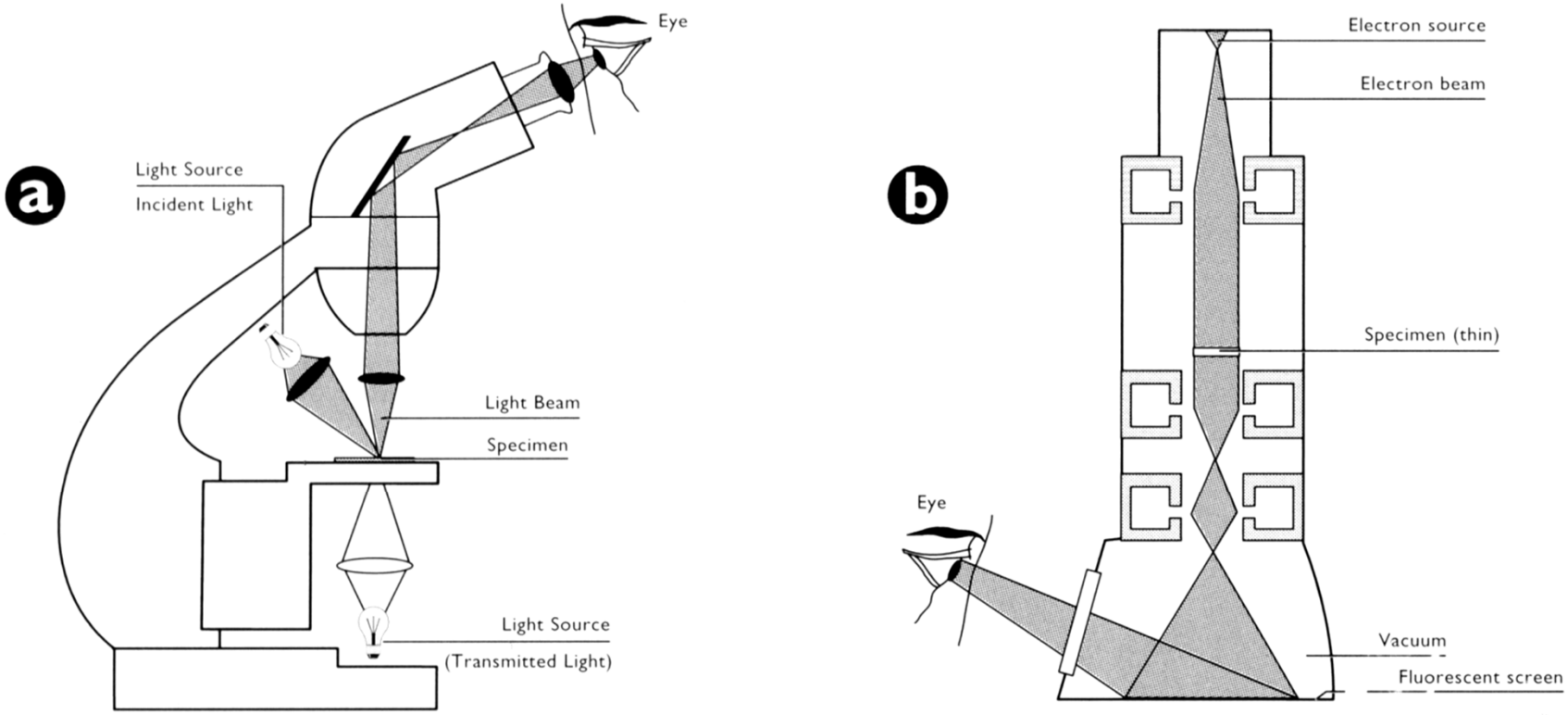

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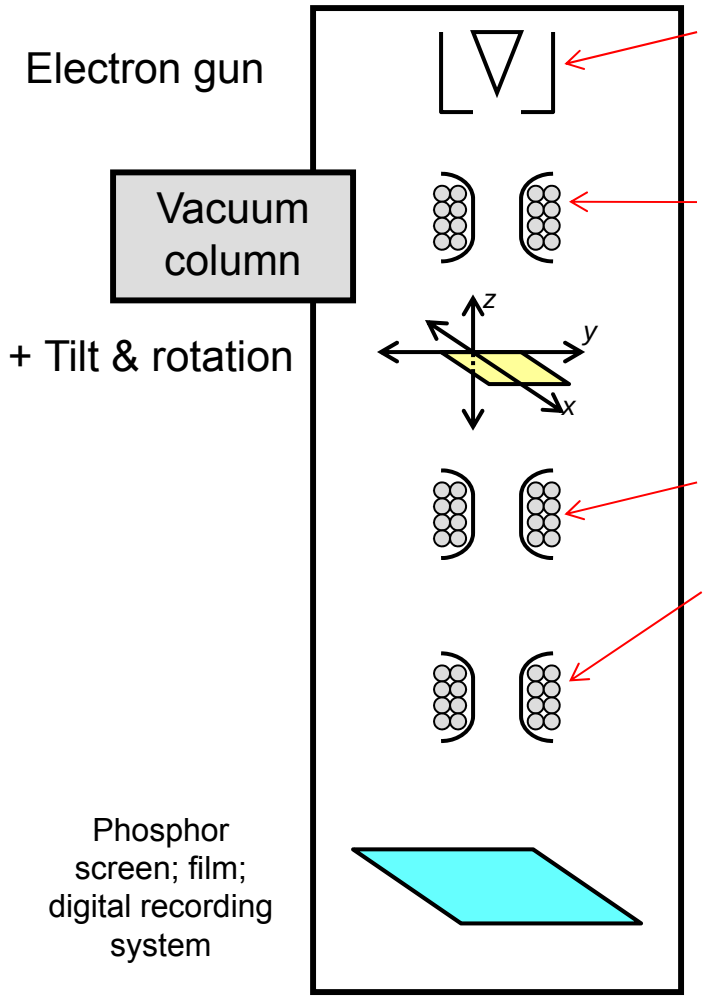
Components of the TEM



1 in 10^{20} electrons are collected

- **Source** – filament plus anode plates with applied accelerating voltage.
- **Condenser Lenses** – electromagnetic lenses adjusted by lens currents not position.
- **Specimen Stage** – allows translations and tilts.
- **Objective Lens** – usually <50X.
- **Imaging System** – multiple electromagnetic lenses below the objective: set magnification, focal plane (image vs. diffraction pattern).
- **Detector/Imaging** – fluorescent screen, plate film, CCD camera.

Instruments often have attachments such as X-ray detectors

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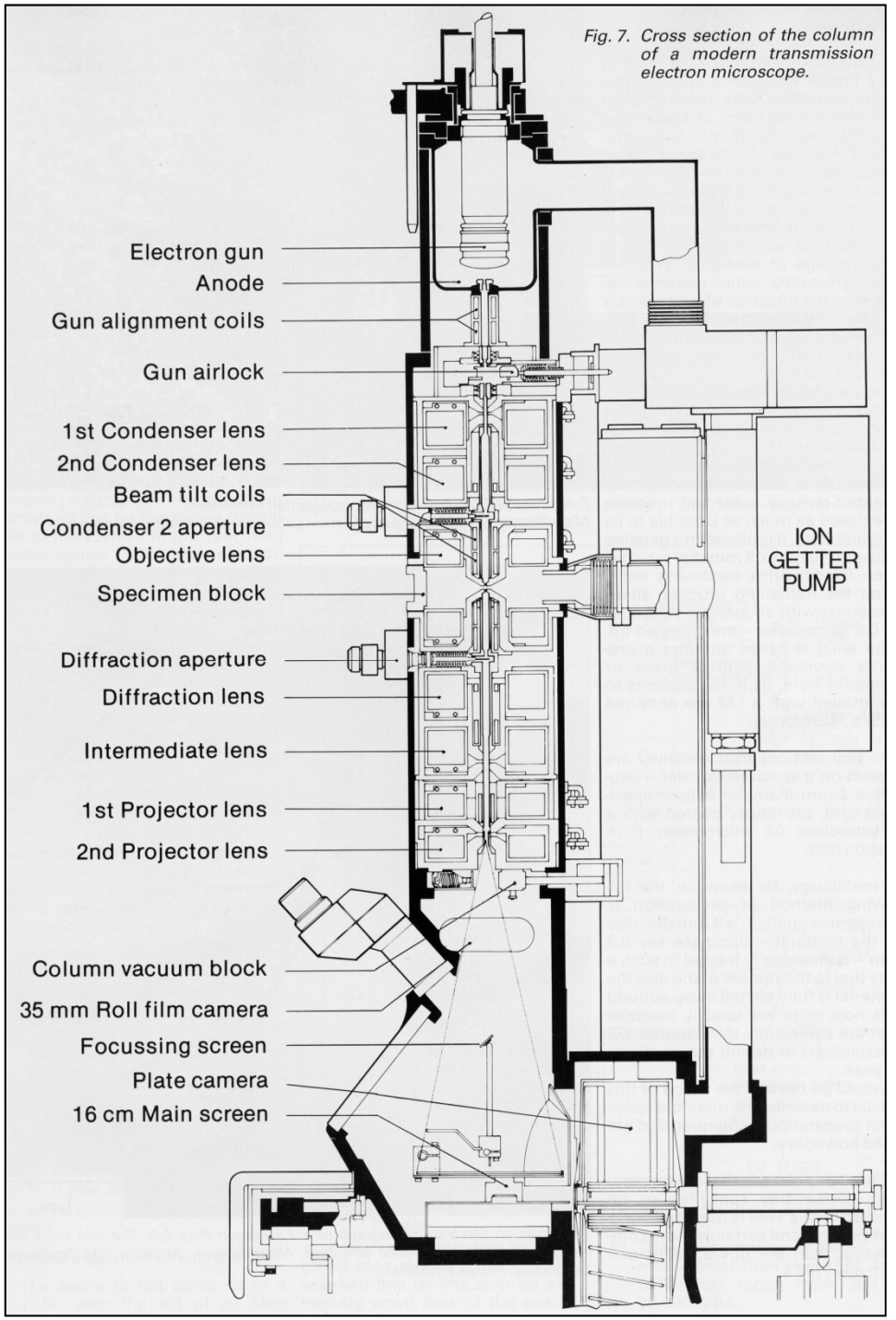
TEM Schematic



How does a TEM work?

- Pass a focused beam of electrons through a thin foil
- As beam passes through sample, it is scattered
- Project the transmitted (scattered) beam onto a phosphor screen to form an enlarged image.
- Imaging Modes:
  - **Bright Field / Dark Field** modes for visualization of structure and defects
  - **Selected Area Diffraction / Convergent Beam Diffraction** for crystallographic information

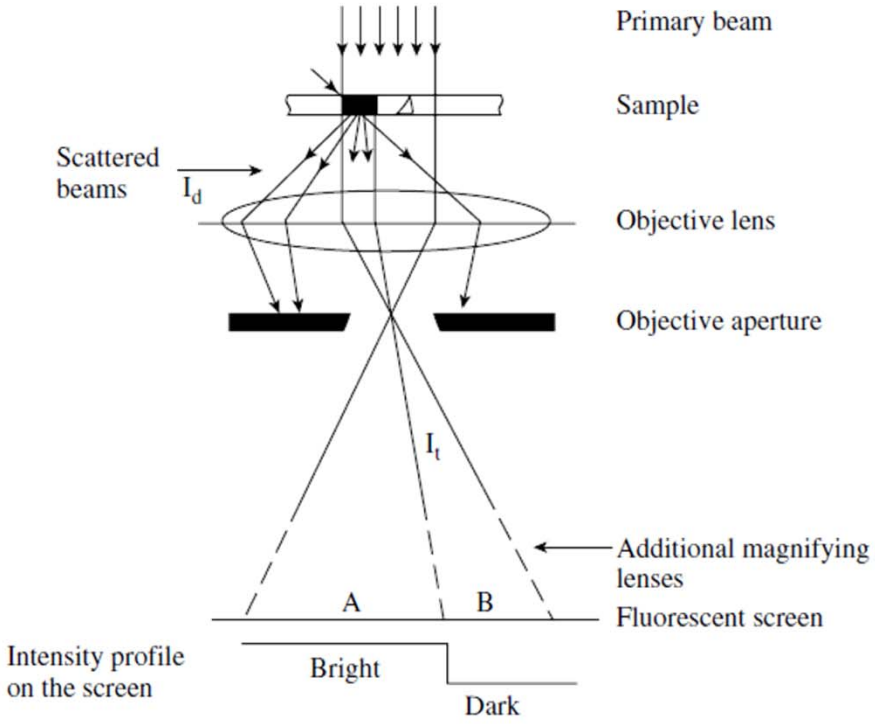
Contrast in TEM

- Generated when there is a difference in the number of electrons being scattered away from the transmitted beam.

- Mechanisms:
  - Mass-density contrast
  - Diffraction contrast

Mass-density Contrast

- Difference in thickness and density in specimen generates variation in electron intensity.



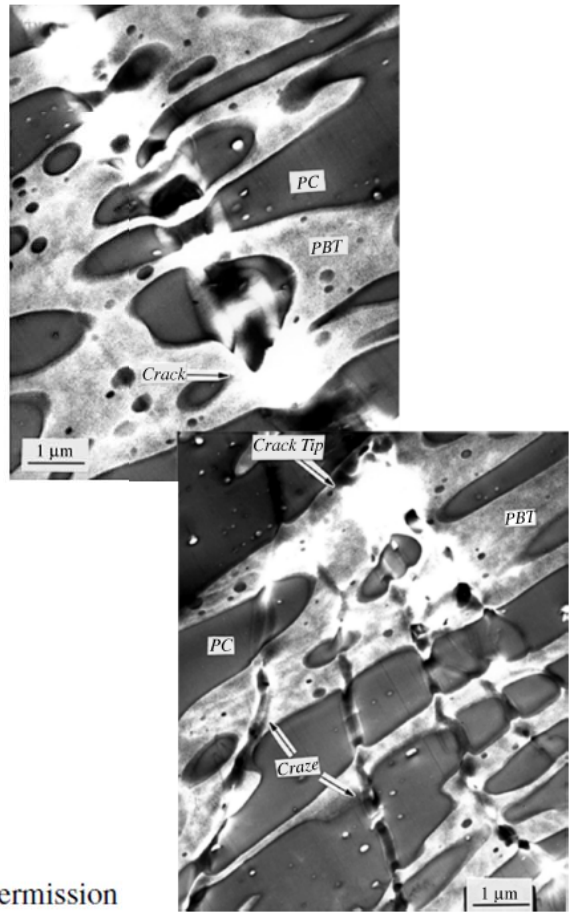


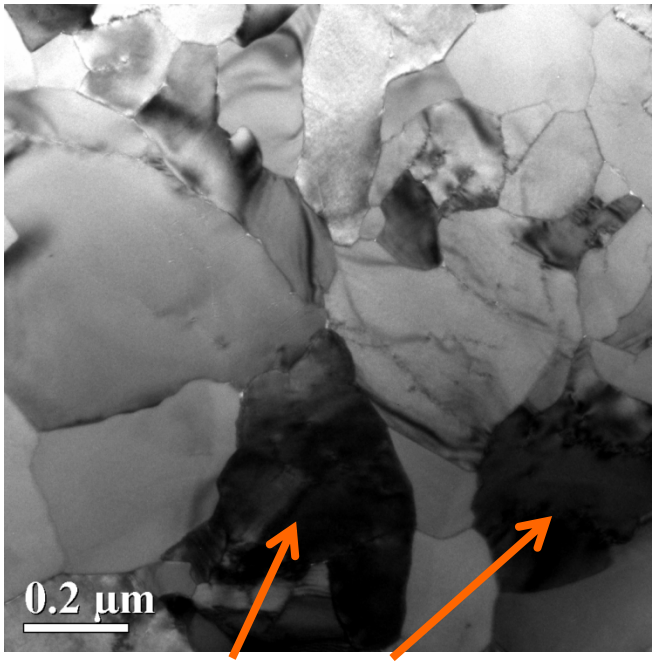
Figure 3.13 Optical arrangement of mass-density contrast in the TEM. (Reproduced with permission from M. von Heimandahl, *Electron Microscopy of Materials*, Academic Press, New York. © 1980 Elsevier B. V.)

(Figures from Leng)

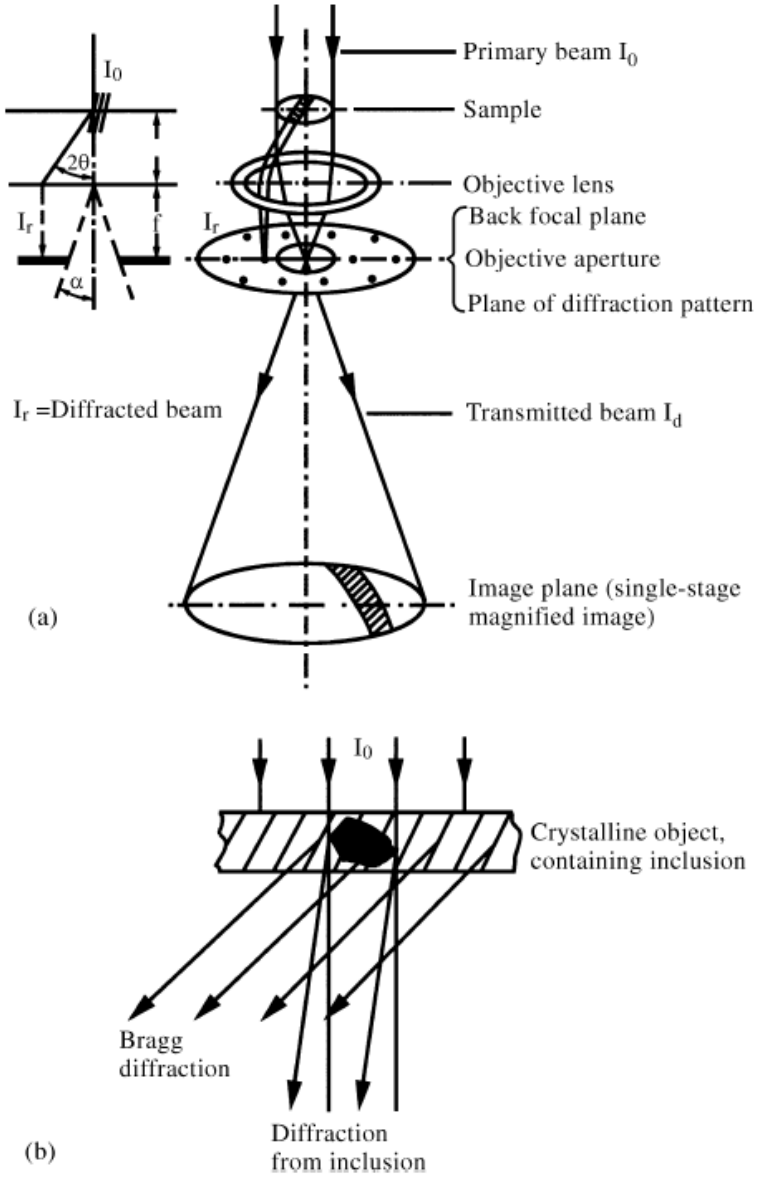
Use aperture to reduce intensity of heavily scattered beam

Diffraction Contrast

- When Bragg's law is satisfied, constructive diffraction occurs resulting in reduced intensity of the transmitted beam.



Bragg's law satisfied for these areas



(Figures (a) and (b) from Leng)

TEM Imaging

- In most cases, you are using amplitude contrast rather than phase contrast.
- Like light microscopy, you can do BF and DF imaging.
- You can also do diffraction from sub-micron areas to examine crystal structure.

Adapted from the lecture notes of Hendrik O. Colijn,
OSU Campus Electron Optics Facility,
www.ceof.ohio-state.edu/classes/MSE605.ppt

Brightfield vs. Darkfield

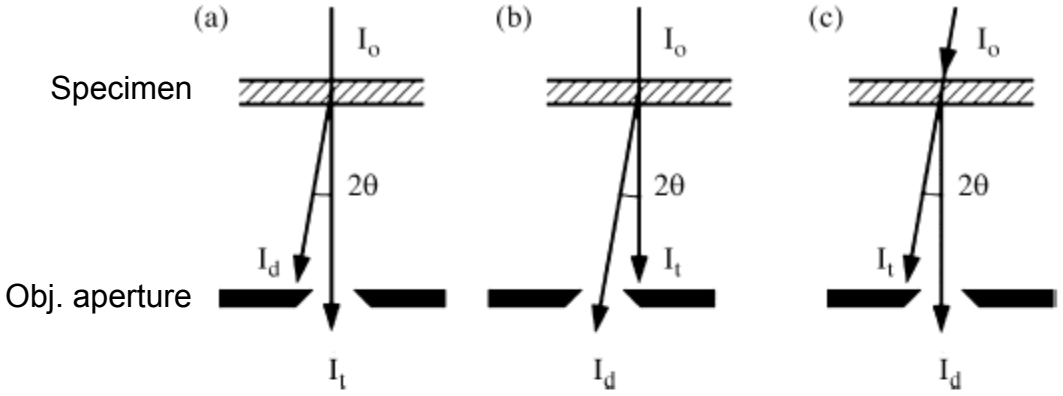


Figure 3.17 Arrangement for: (a) bright-field image; and (b, c) dark-field image.

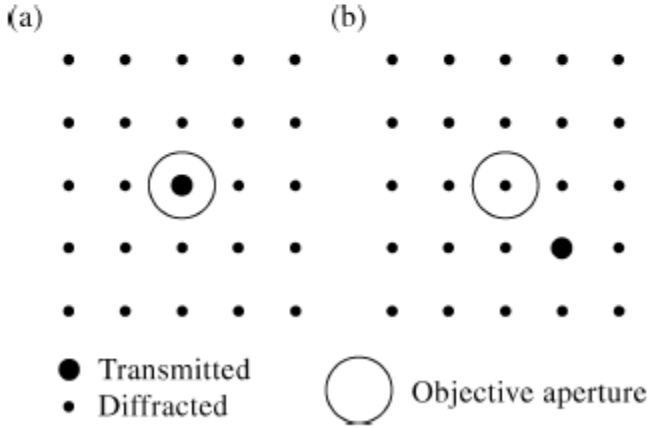
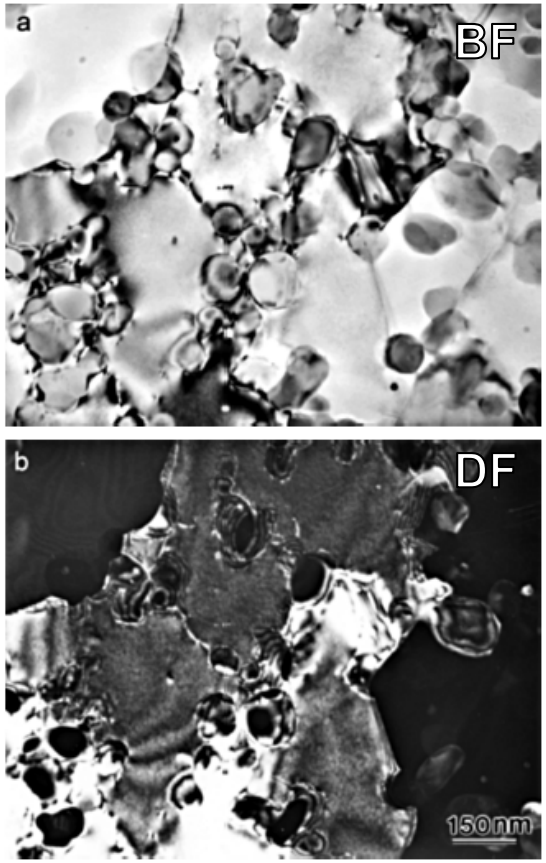


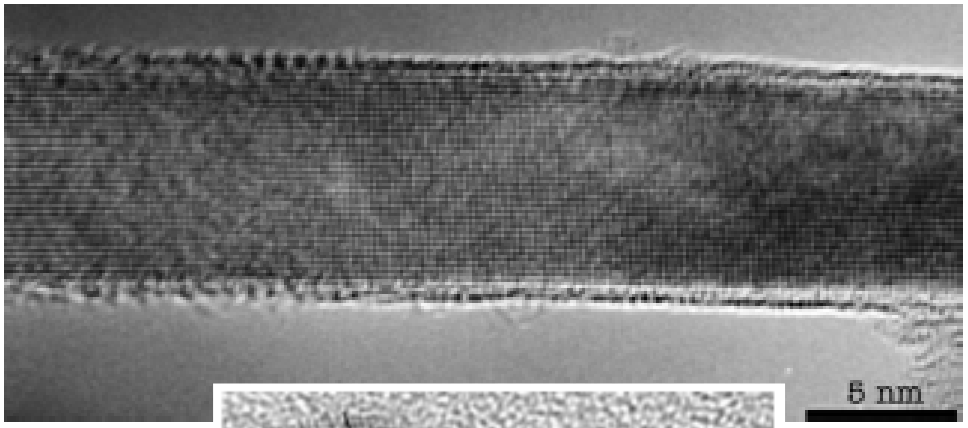
Figure 3.18 Selection of a diffraction spot with an objective aperture for dark-field imaging.

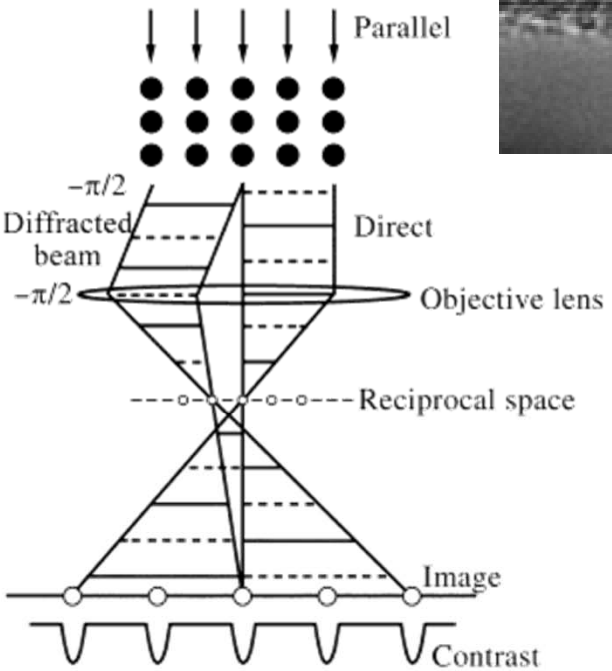


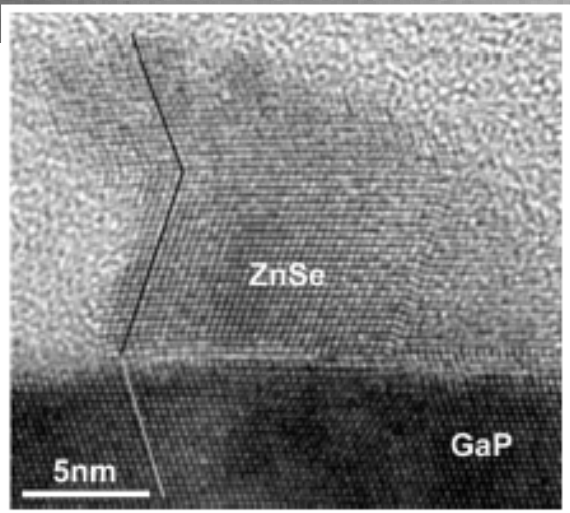
(Figures from Leng)

Phase Contrast

- Provides highest resolution of lattice.
- Used primarily in HRTEM.





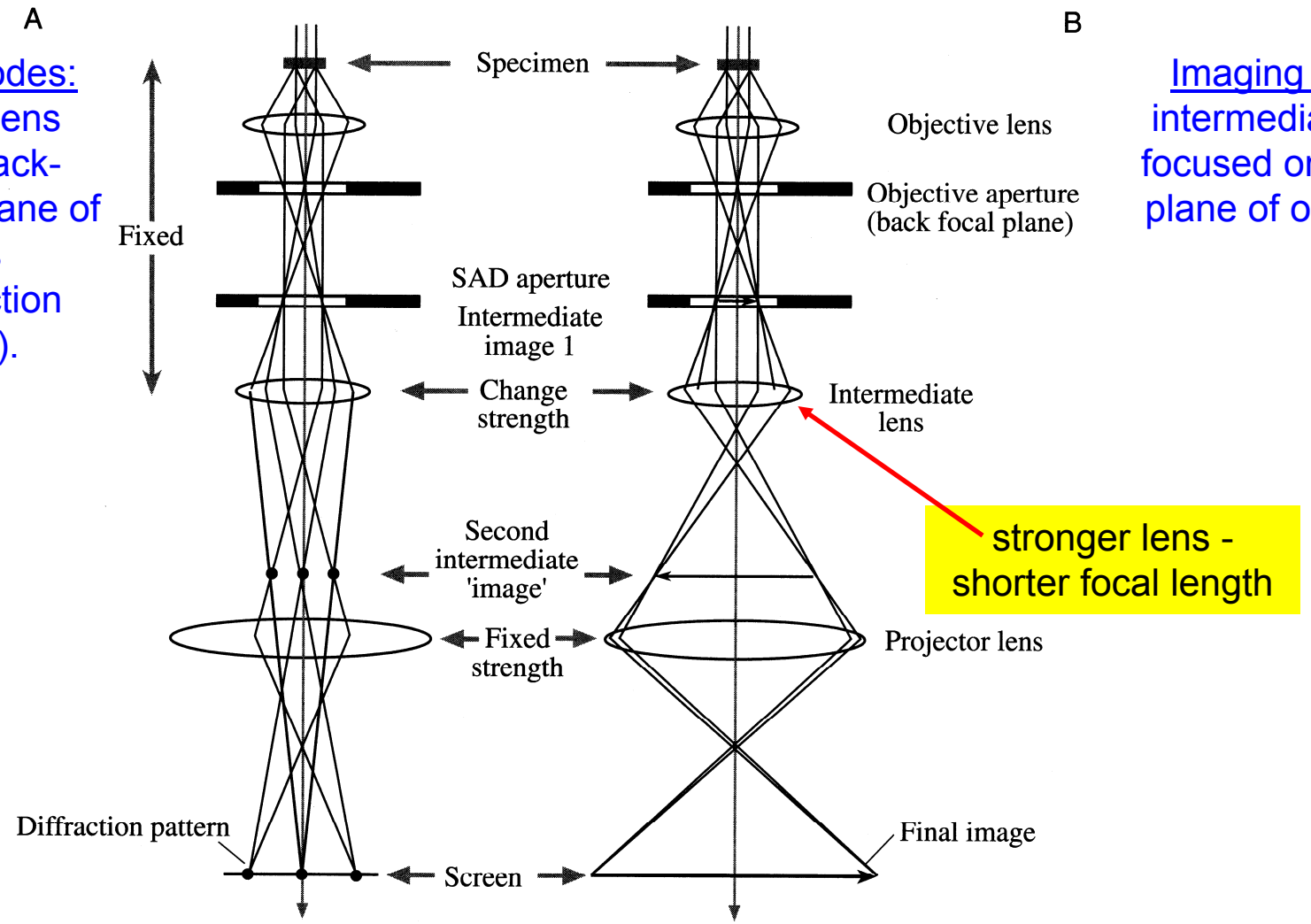


(Figures from Leng)

Figure 3.22 Phase contrast formation from a crystalline specimen. Phase difference is generated by crystal diffraction and an objective lens. This results in a phase difference of $-\pi$ between the direct and transmitted beams.

Diffraction vs. Imaging

Diffraction Modes: intermediate lens focused on back-focal plane plane of objective lens (where diffraction pattern forms).



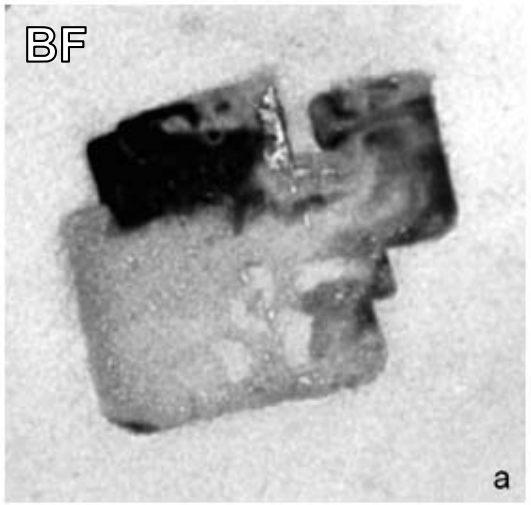
Imaging Modes: intermediate lens focused on image plane of objective lens.

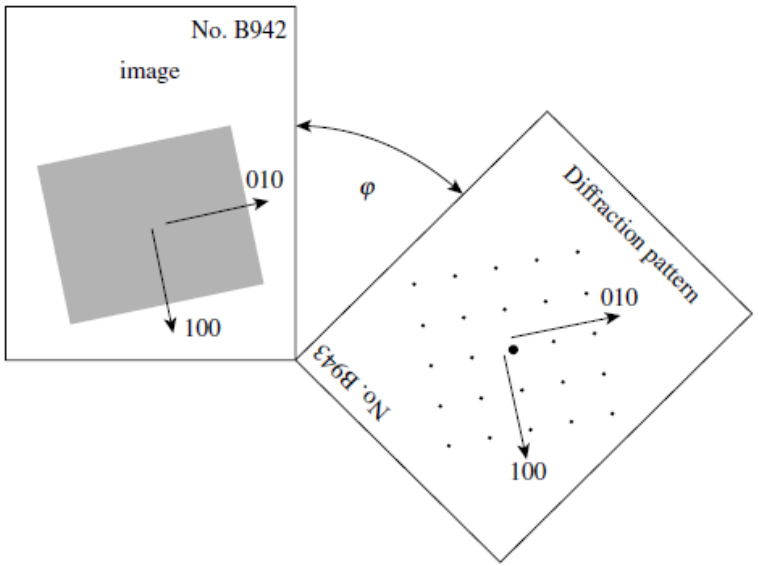
Adapted from the lecture notes of Hendrik O. Colijn,
OSU Campus Electron Optics Facility,
www.ceof.ohio-state.edu/classes/MSE605.ppt

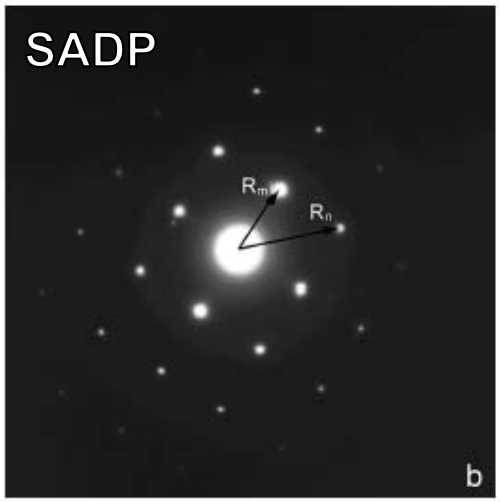
Selected Area Diffraction

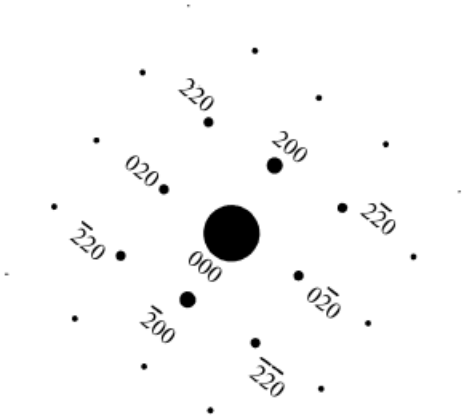
Specimen

Obj. aperture









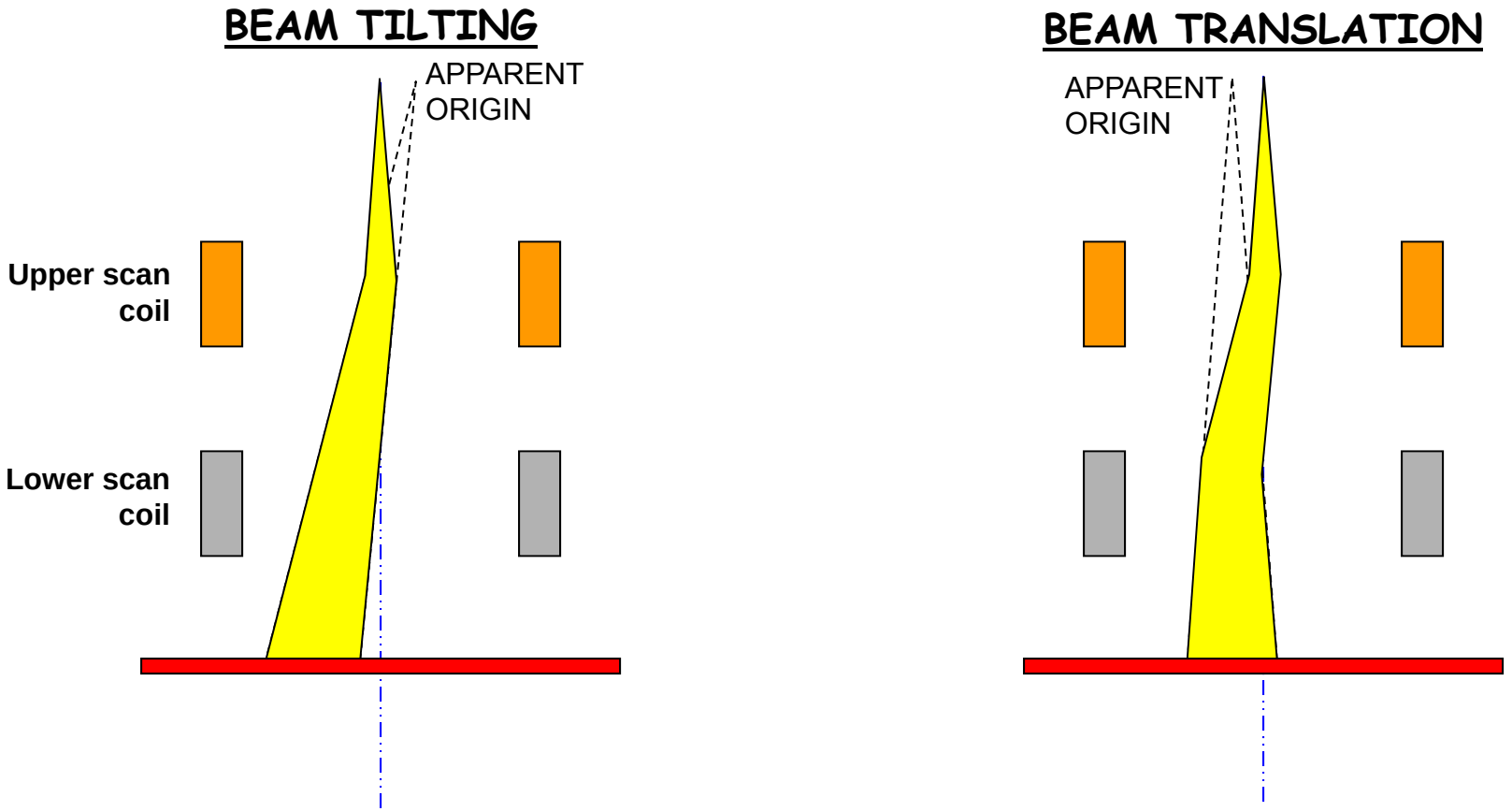
Phase ID
Crystal orientation

(Figures from Leng)

Figure 3.30 Single crystal of NaCl: (a) bright-field image; and (b) selected area diffraction pattern. R_m, R_n are the radii of spots m and n, respectively. The transmitted beam direction is parallel to [001].

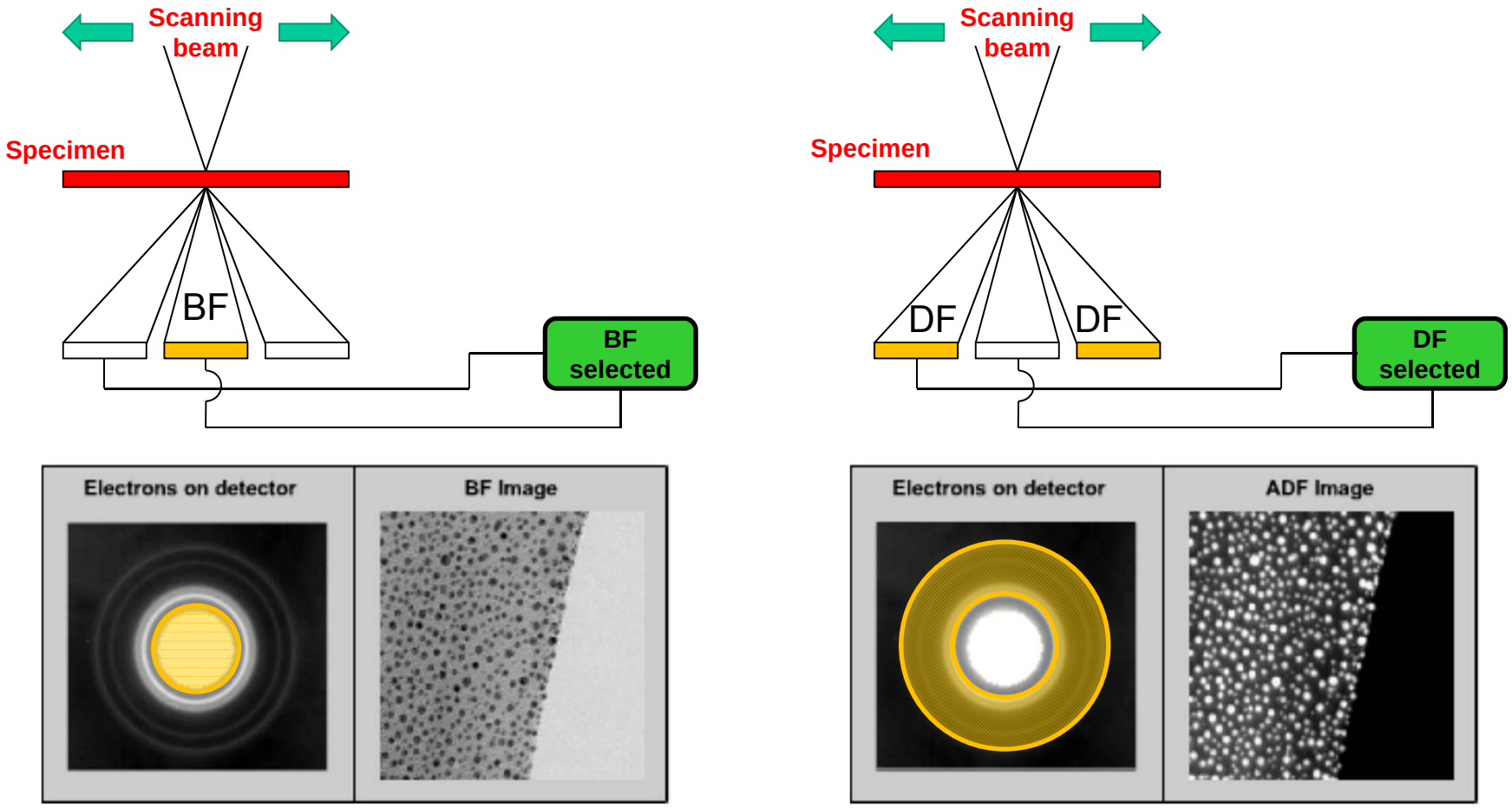
Beam Tilting and Translation

- The electron beam can be positioned for fine measurements (spot modes) or scanning (SEM, STEM)



Scanning TEM (STEM)

- Selection of bright field or dark field electrons results in contrast variations that provide crystallographic information.



Adapted from MATTER website (http://www.matter.org.uk/tem/stem_images.htm)

More on TEM

- There are plenty of things that we can do with a TEM that go far beyond the scope of this introductory course.
  - Phase identification
  - Defect identification and analysis
  - Etc…
- Some of them are described in Ch. 3 of the text.
- You can learn about these things in MTE 655 (Transmission Electron Microscopy).

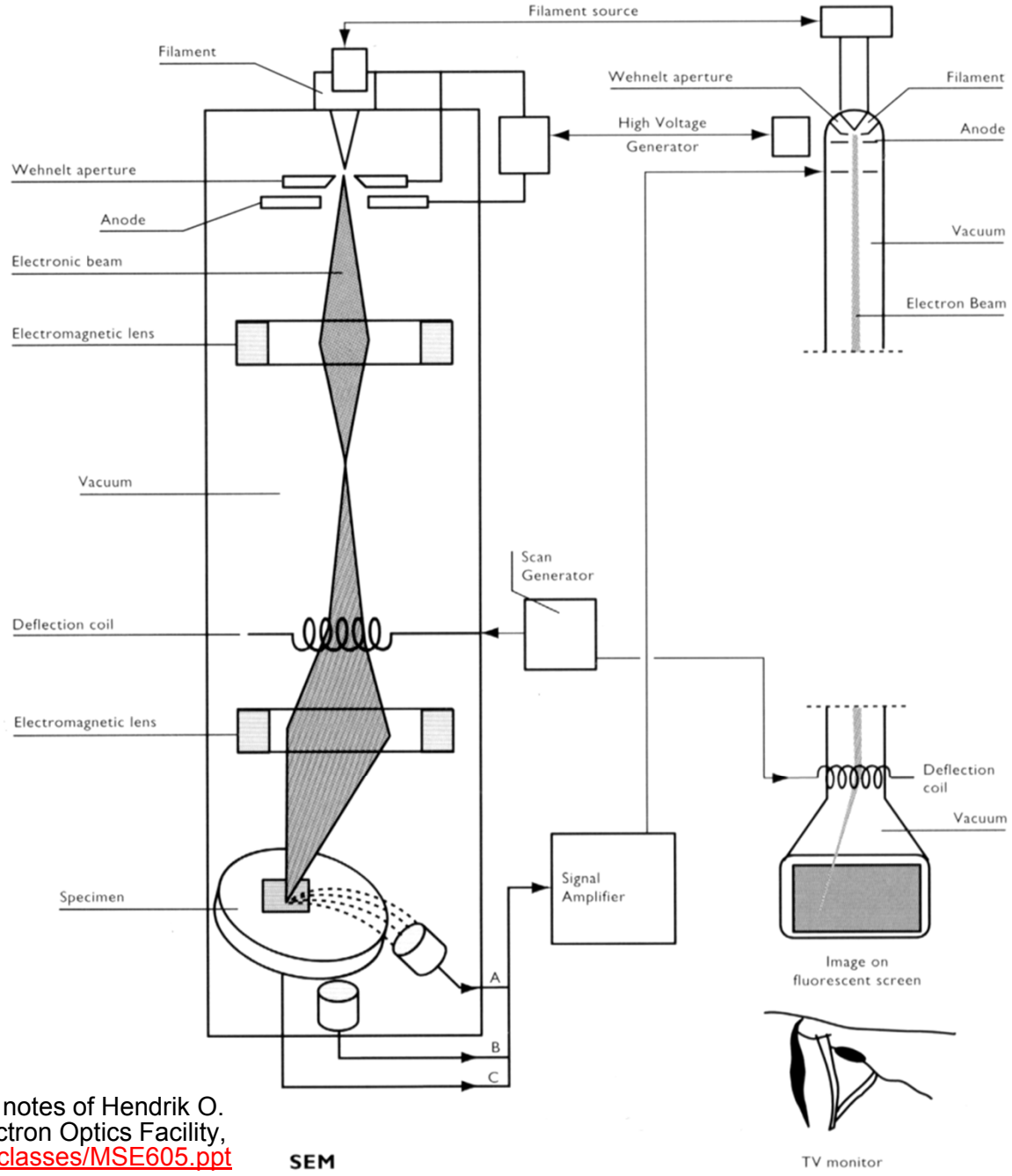
Some Technical Details

- Produce a stream of monochromatic electrons in the electron gun
- Focus the stream into a small coherent beam using C1 and C2
  - C1 determines the “spot size” (*i.e.*, size of electron probe)
  - C2 changes intensity or brightness
- Use condenser aperture to restrict the beam
- Part of the beam is transmitted through the sample
- Focus transmitted portion using the objective lens to form an image
- Objective and selected area apertures are used to restrict the beam further
  - allows examination of diffraction from specific atoms, crystals, features…
  - SAD, CBD
- Enlarge image with intermediate and projector lenses

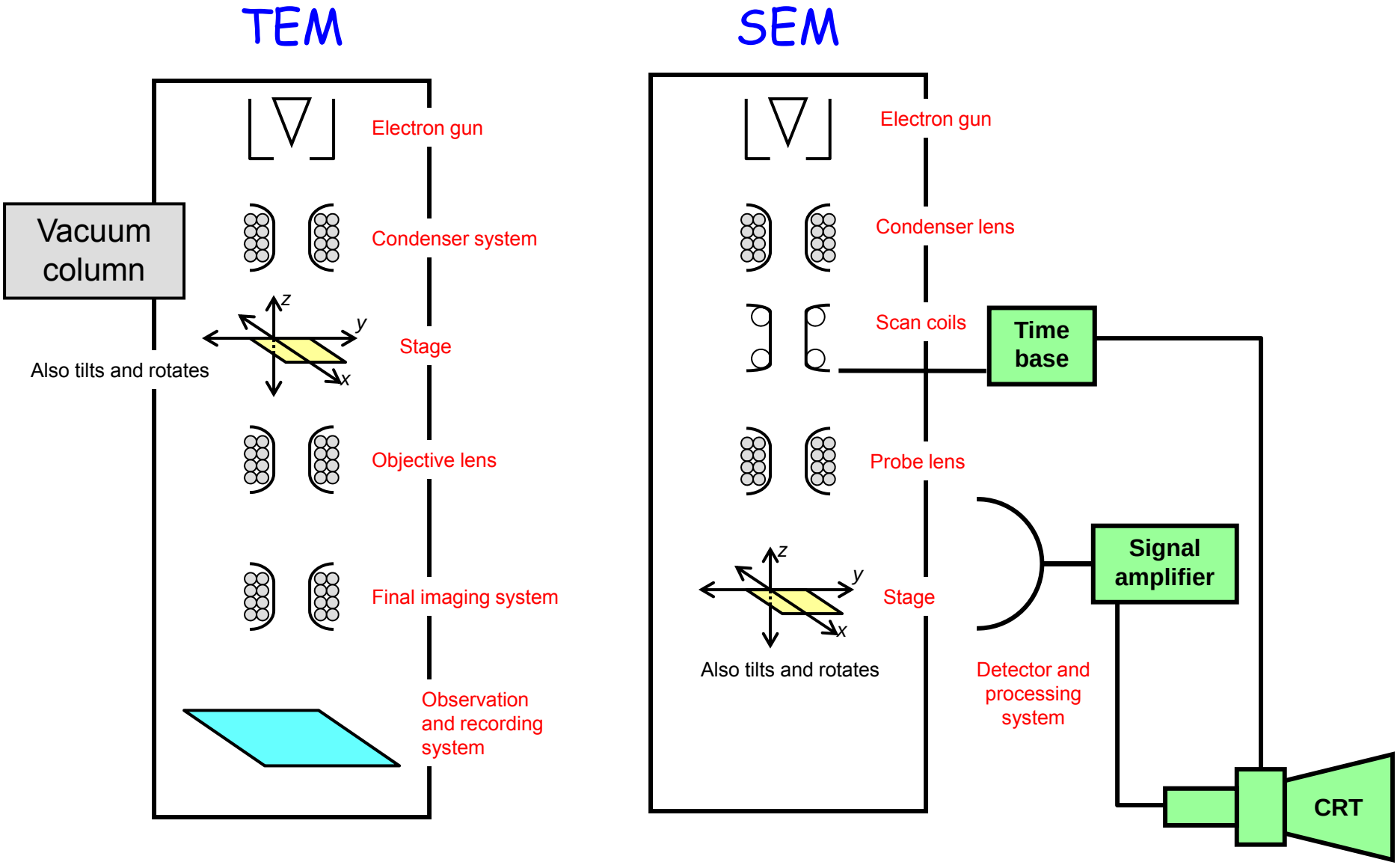
SEM

- Patterned after reflecting light optical microscopes
  - Forms an image by scanning a focused beam of electrons over the surface of a sample
- Yield Following Information:
  - Topography
    - Surface features of an object. Detectable features limited to a few nanometers depth.
  - Morphology
    - Size shape and arrangement of particles, precipitates, etc
  - Compositional Information
    - Elements and compounds the sample is composed of
  - Crystallographic information
    - Possible using new techniques (OIM/BKD)a

SEM



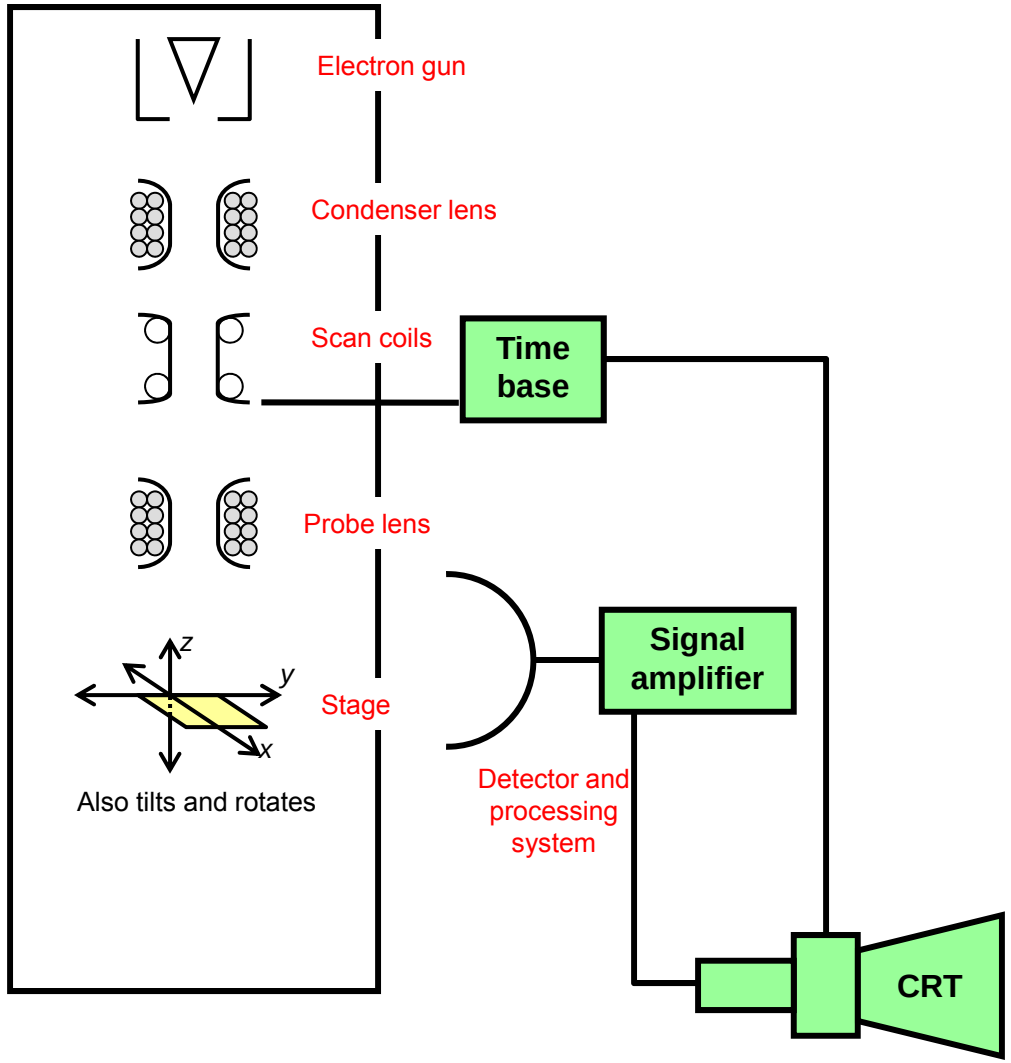
Adapted from the lecture notes of Hendrik O. Colijn, OSU Campus Electron Optics Facility, www.ceof.ohio-state.edu/classes/MSE605.ppt



In the SEM you use secondary signals to acquire images.

Components of an SEM

- **Source**:
  - same as TEM but lower V
- **Condenser**:
  - same as TEM
- **Scan Coils**:
  - raster the probe
- **Probe Lens**:
  - lens that forms a spot at the specimen surface
- **Detector & Processing System**:
  - collects signals such as X-rays and electrons as a function of time and position.
  - Provides digital images for real-time viewing, processing, and storage.



Structure of SEM

Two condensers used to reduce crossover diameter of e-beam

Objective focuses e-beam

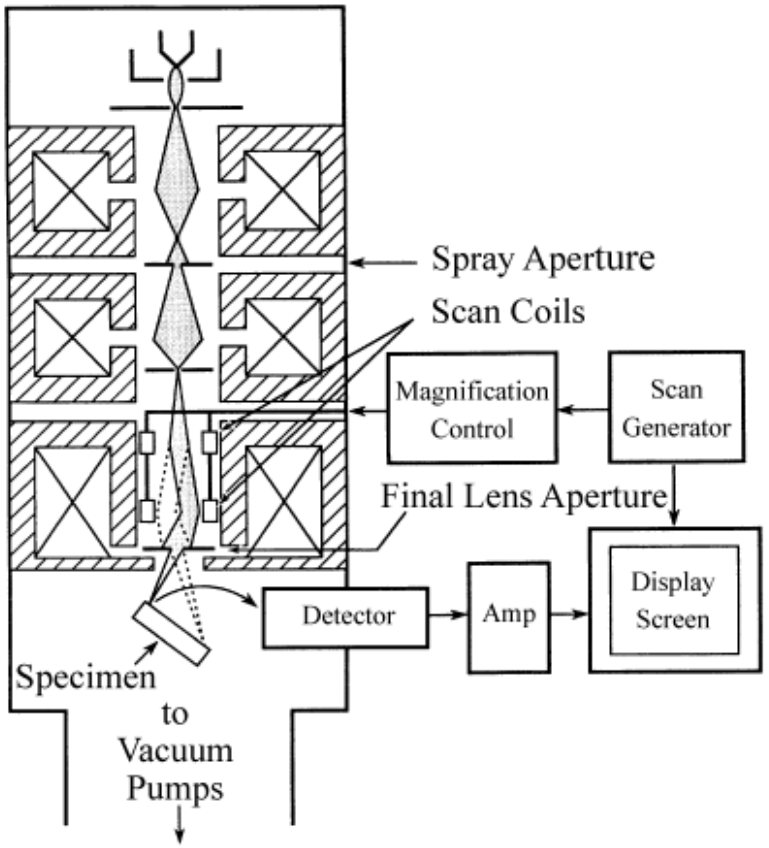
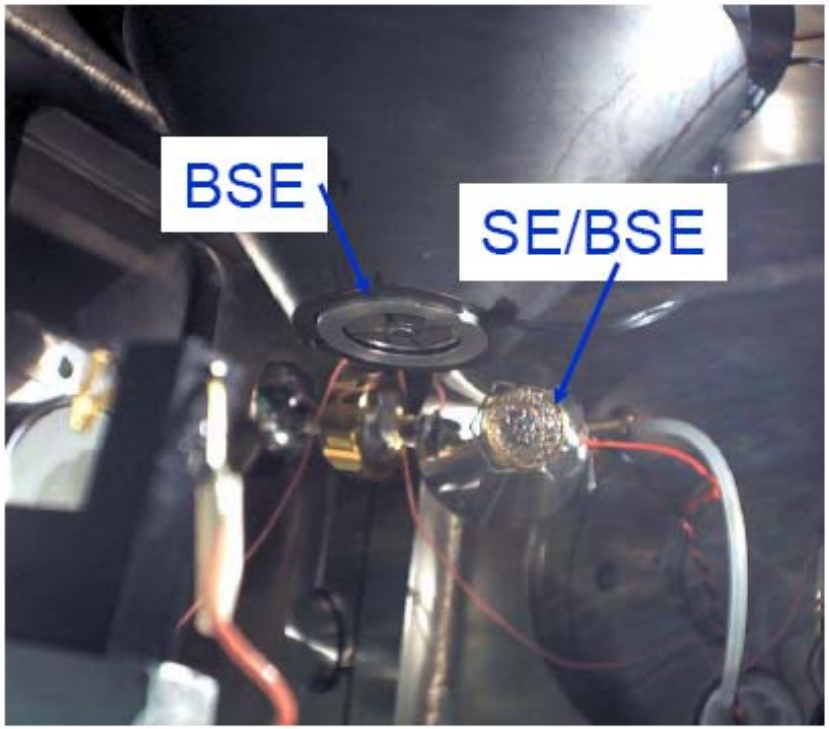
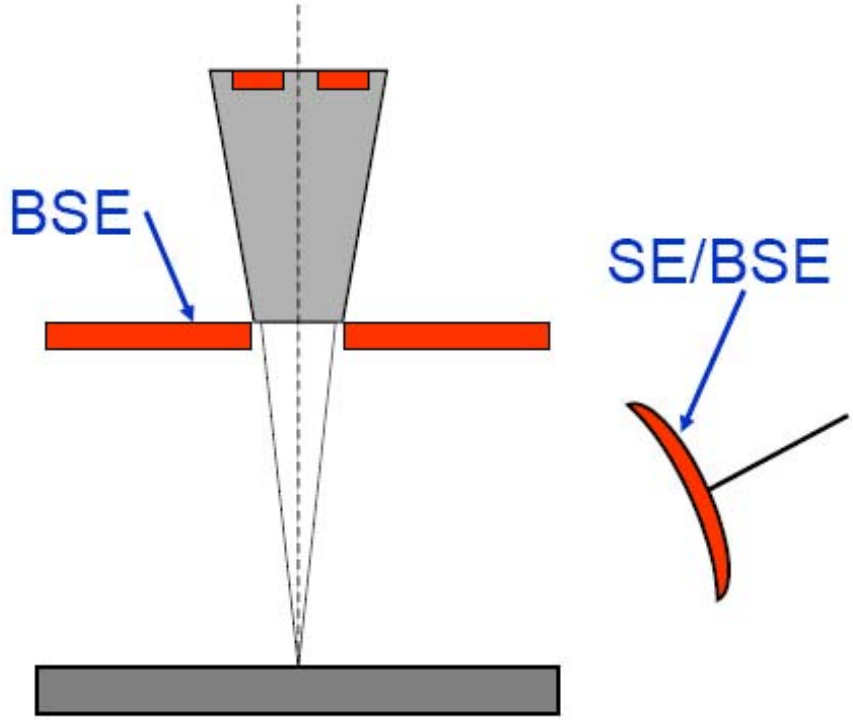


Figure 4.1 Structure of a scanning electron microscope (SEM). (Reproduced with kind permission of Springer Science and Business Media from J.I. Goldstein et al, *Scanning Electron Microscopy and X-ray Microanalysis*, 2nd ed., Plenum Press, New York. © 1992 Springer Science.)

(Figures from Leng)

Detectors for Imaging with Electrons



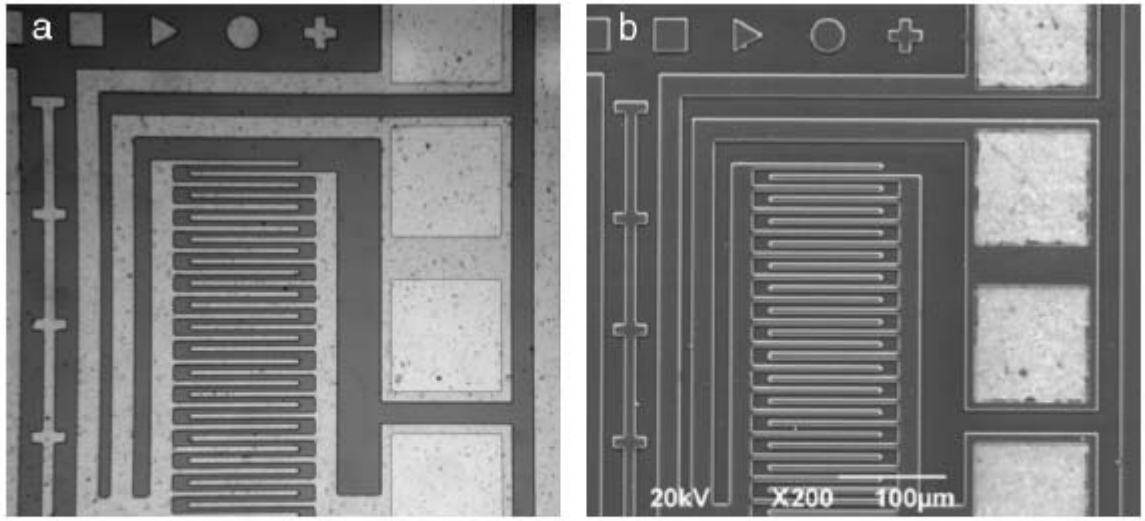


- Everhardt-Thornley: Scintillator/photomultiplier pair for SE or BSE depending on grid bias.
- Solid-state: segmented for Z or orientation in BSE.

B.D. Huey, MMAT322 Lecture Notes, University of Connecticut (2005)

Comparison of LM and SEM images

LOM **SEM**



Increased DOF!
Z contrast!

Figure 4.2 Comparison of images taken in: (a) a light microscope; and (b) an SEM. An SEM image (b) is able to provide the 3-D appearance of an integrated circuit while revealing the same in-plane details as the light microscopic image (a).

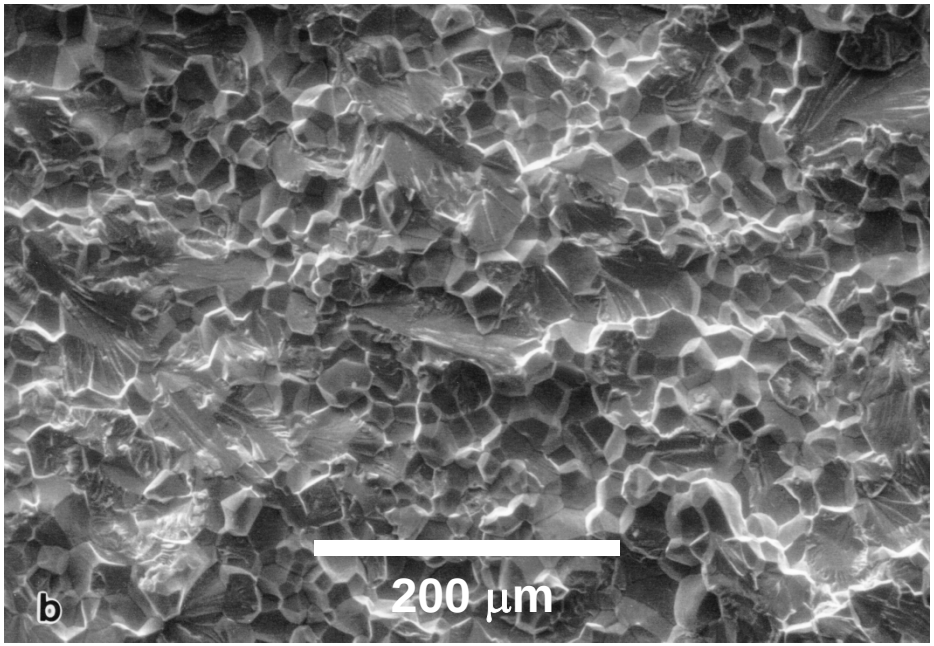
(Figures from Leng)

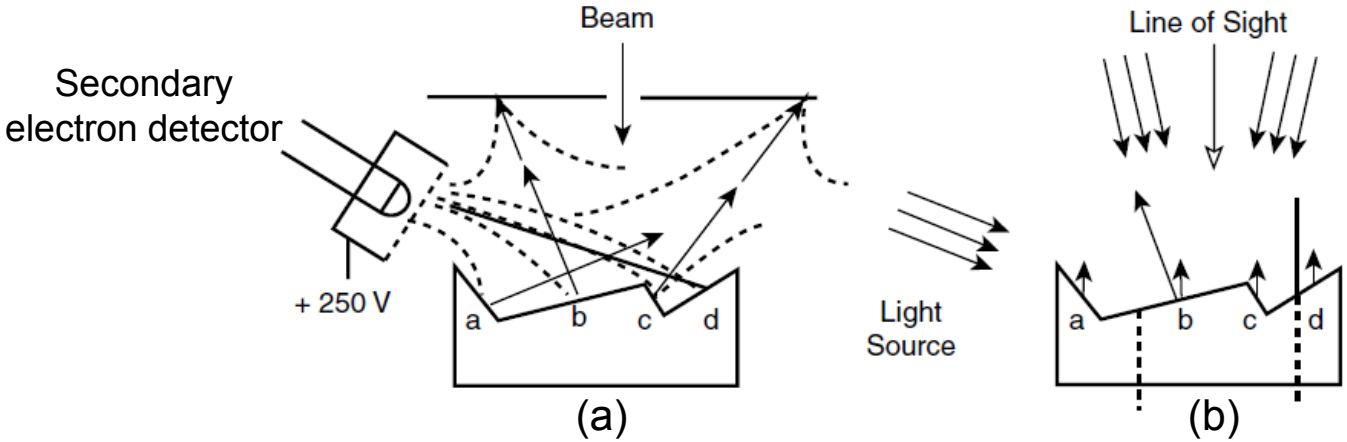
SEM: Technical Details

- Produce a stream of monochromatic electrons in the electron gun
- Focus the stream using the first condenser lens
  - Coarse probe current knob
- The beam is constricted by the condenser aperture (eliminates high-angle electrons)
- Second condenser lens is used to form electrons into a thin, tight, coherent beam.
  - Use fine probe current knob
- Use objective aperture to limit beam (*i.e.,* eliminate high-angle electrons)
- Scan coils raster the beam across the sample, dwelling on the points for a predetermined period of time (selected using scan speed)
- Final objective lens focuses beam on desired region.
- When beam strikes the sample, interactions occur. We detect what comes out of the sample.

Secondary electron mode

Topographic contrast

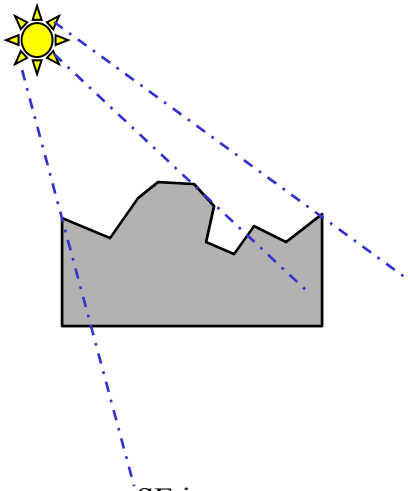




(Figures from Leng)

Figure 4.11 Generation of topographic contrast: (a) the trajectory effect, which arises from the orientation of surface with respect to the detector in an SEM, is similar to: (b) reflected light effects from the orientation of surface with respect to the light source in a light microscope. (Reproduced with kind permission of Springer Science and Business Media from J.I. Goldstein et al, *Scanning Electron Microscopy and X-ray Microanalysis*, 2nd ed., Plenum Press, New York. © 1992 Springer Science.)

You can use BSE signals in conjunction with SE signals to yield **enhanced topographical information**



SE image

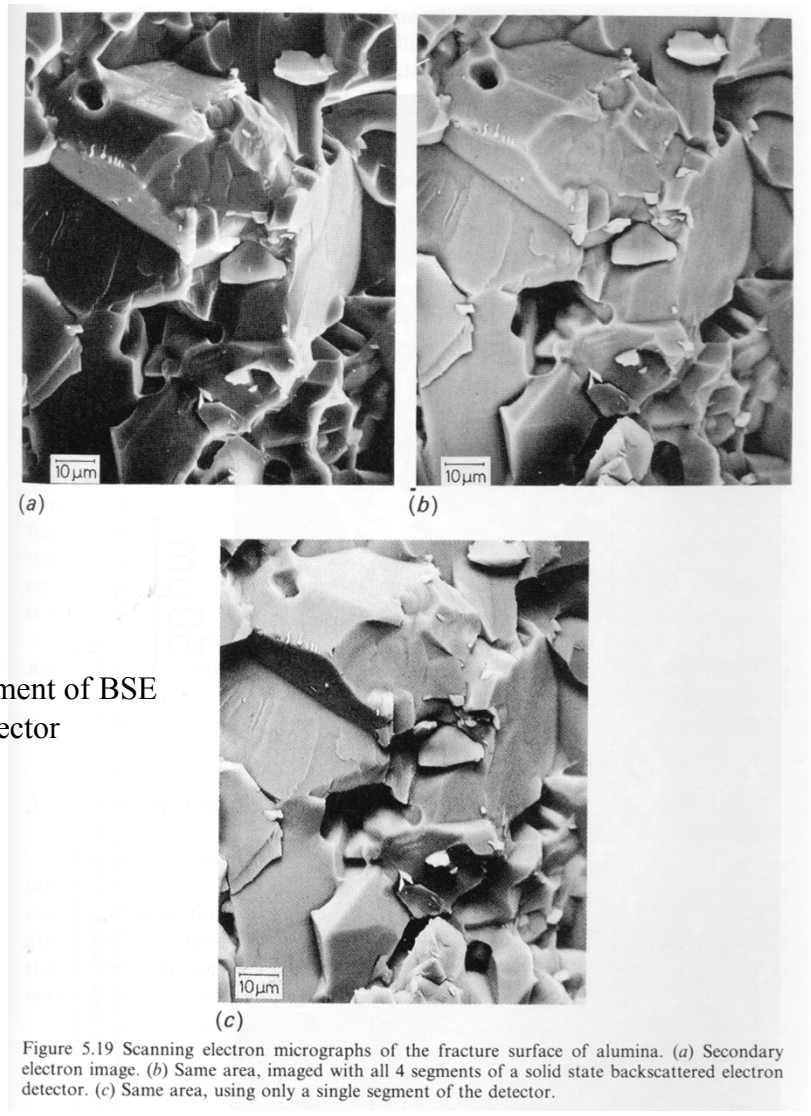
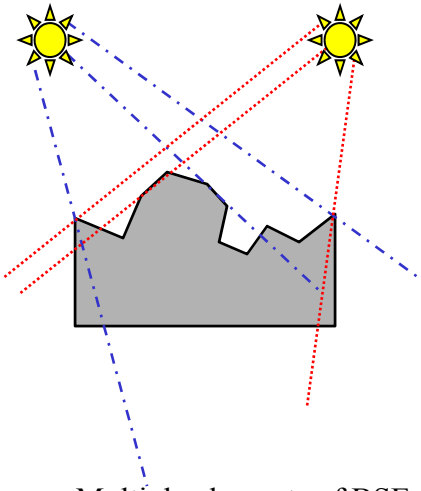


Figure 5.19 Scanning electron micrographs of the fracture surface of alumina. (*a*) Secondary electron image. (*b*) Same area, imaged with all 4 segments of a solid state backscattered electron detector. (*c*) Same area, using only a single segment of the detector.

Single segment of BSE detector



Multiple elements of BSE detector
"Overlapping shadows"
Reduced resolution

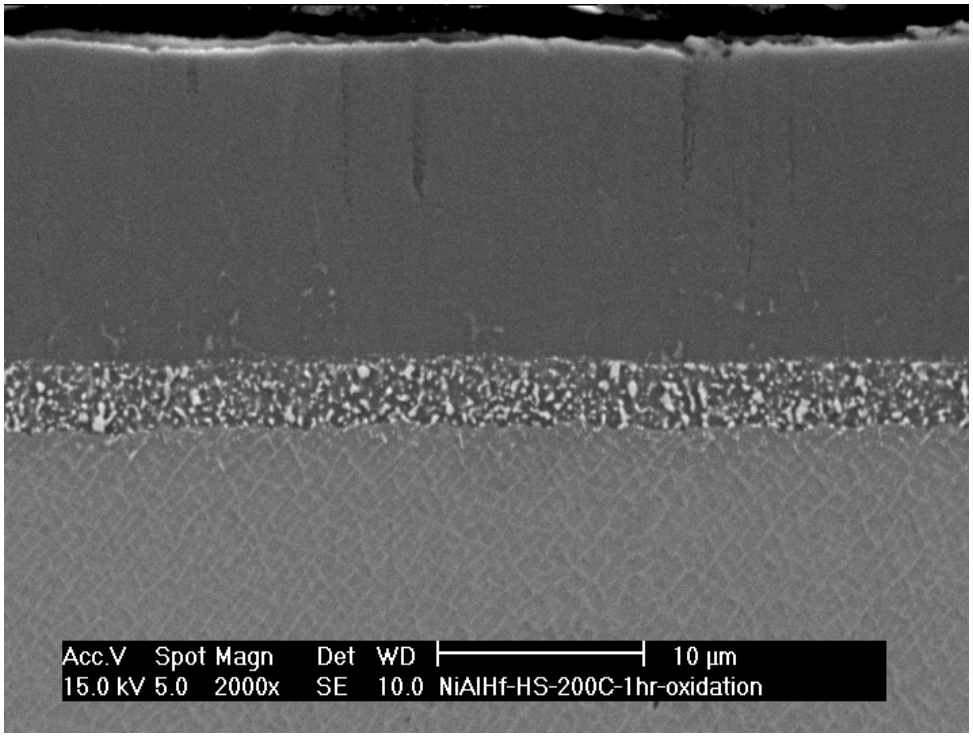
P.J. Goodhew, J. Humphreys, and R. Beanland, *Electron Microscopy and Analysis, 3rd edition*; (Taylor Francis, London, 2001) p. 144

The BSE sampling volume is large which limits resolution

Secondary electron mode

Topographic contrast

Some atomic number contrast



Secondary vs. Backscattered

Higher resolution
No Z contrast

Lots of Z contrast
Lower resolution

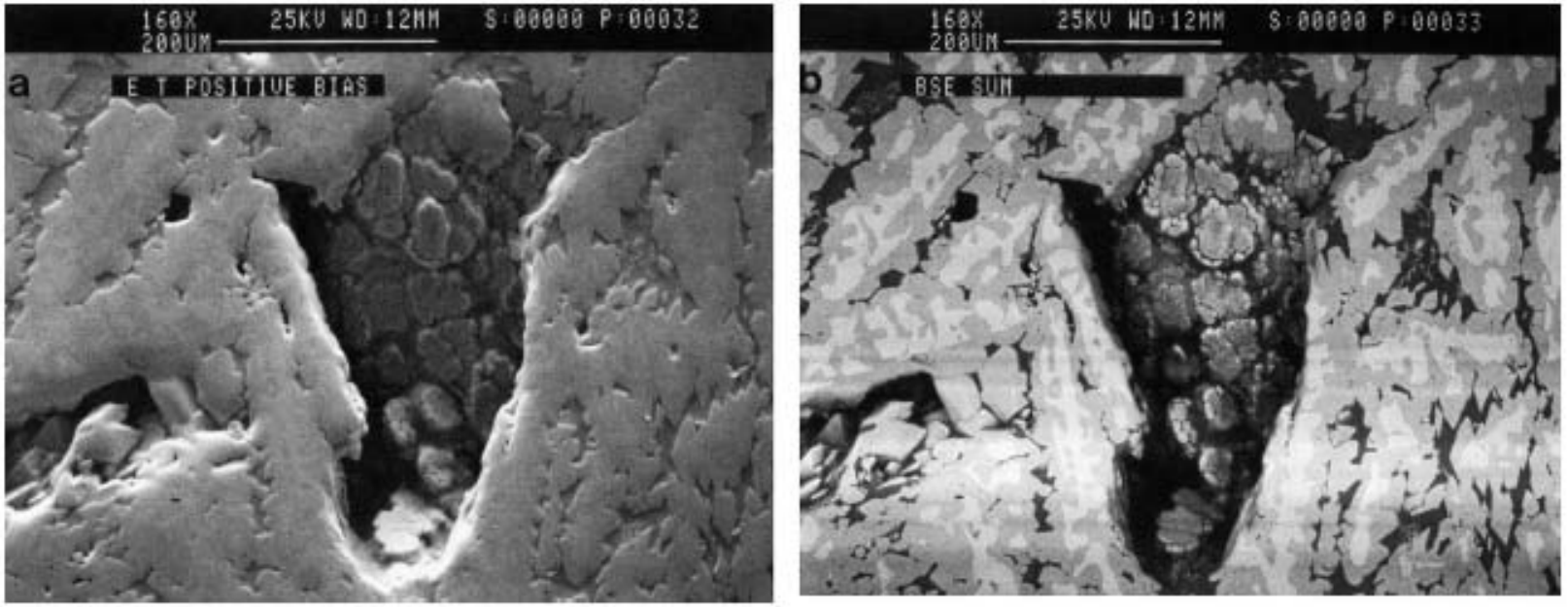
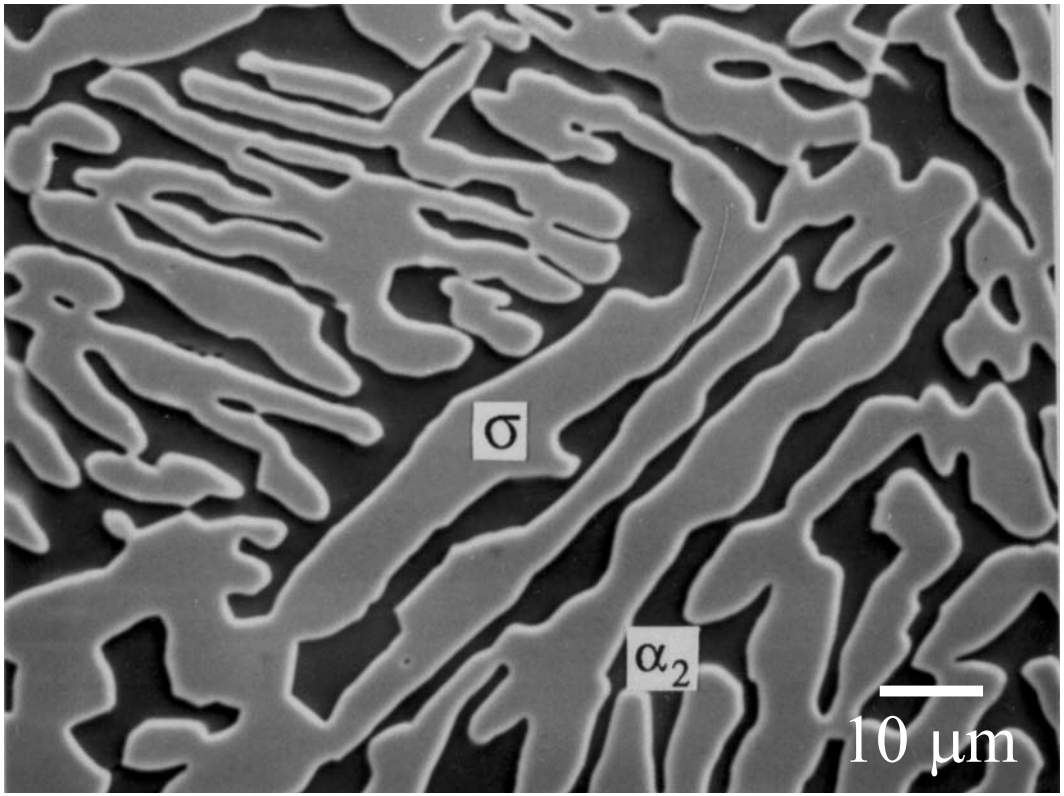


Figure 4.16 Comparison between: (a) a secondary electron image; and (b) a backscattered electron image for the same area of nickel alloy. Additional compositional information is obtained from the backscattered image. (Reproduced with kind permission of Springer Science and Business Media from J.I. Goldstein et al, *Scanning Electron Microscopy and X-ray Microanalysis*, 2nd ed., Plenum Press, New York. © 1992 Springer Science.)

(Figures reproduced from Leng)

Imaging with BSE's

- More backscattering observed for high Z (qualitative chemical sensitivity).
- Local contrast is higher when sample is normal to the beam.
- Local contrast is lower when the sample is tilted away from the beam.

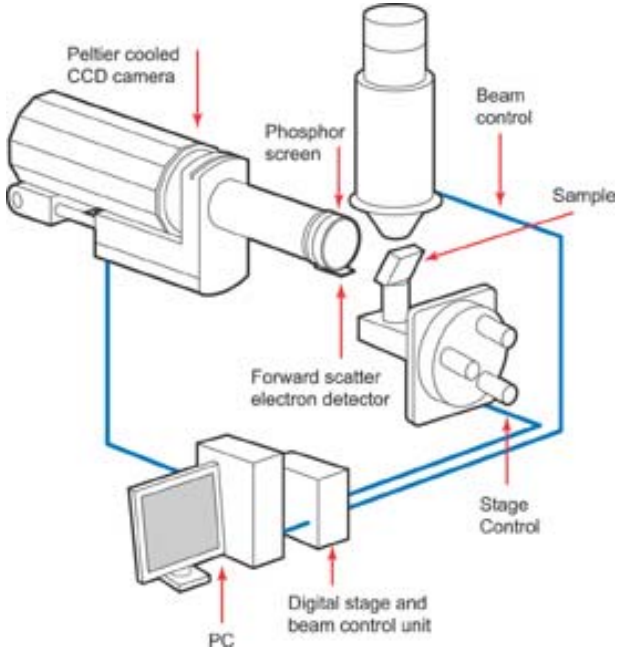


Al-Ta-Ti ternary alloy

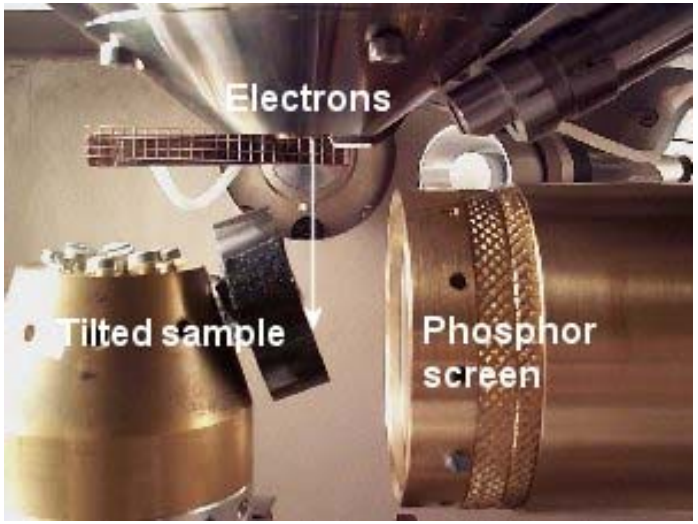
$\alpha_2 = (Ti,Ta)_3Al$

$\sigma = Al_2(Ta,Ti)$

Electron Backscattered Diffraction

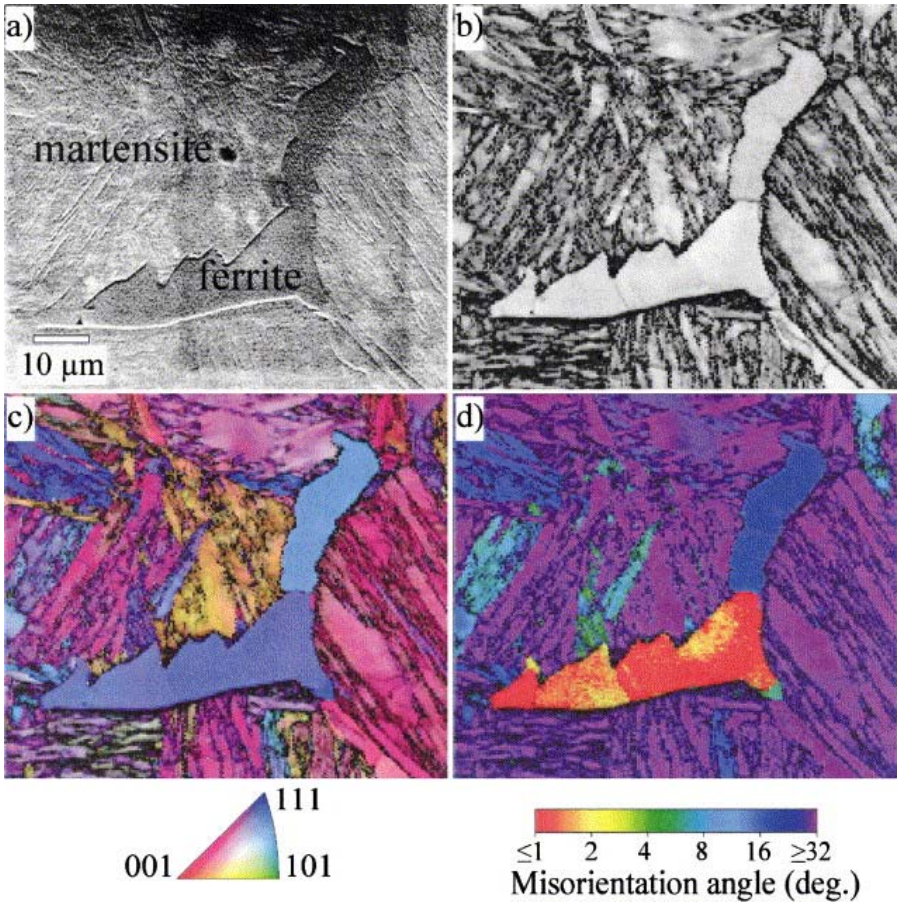


Principal components of an EBSD system



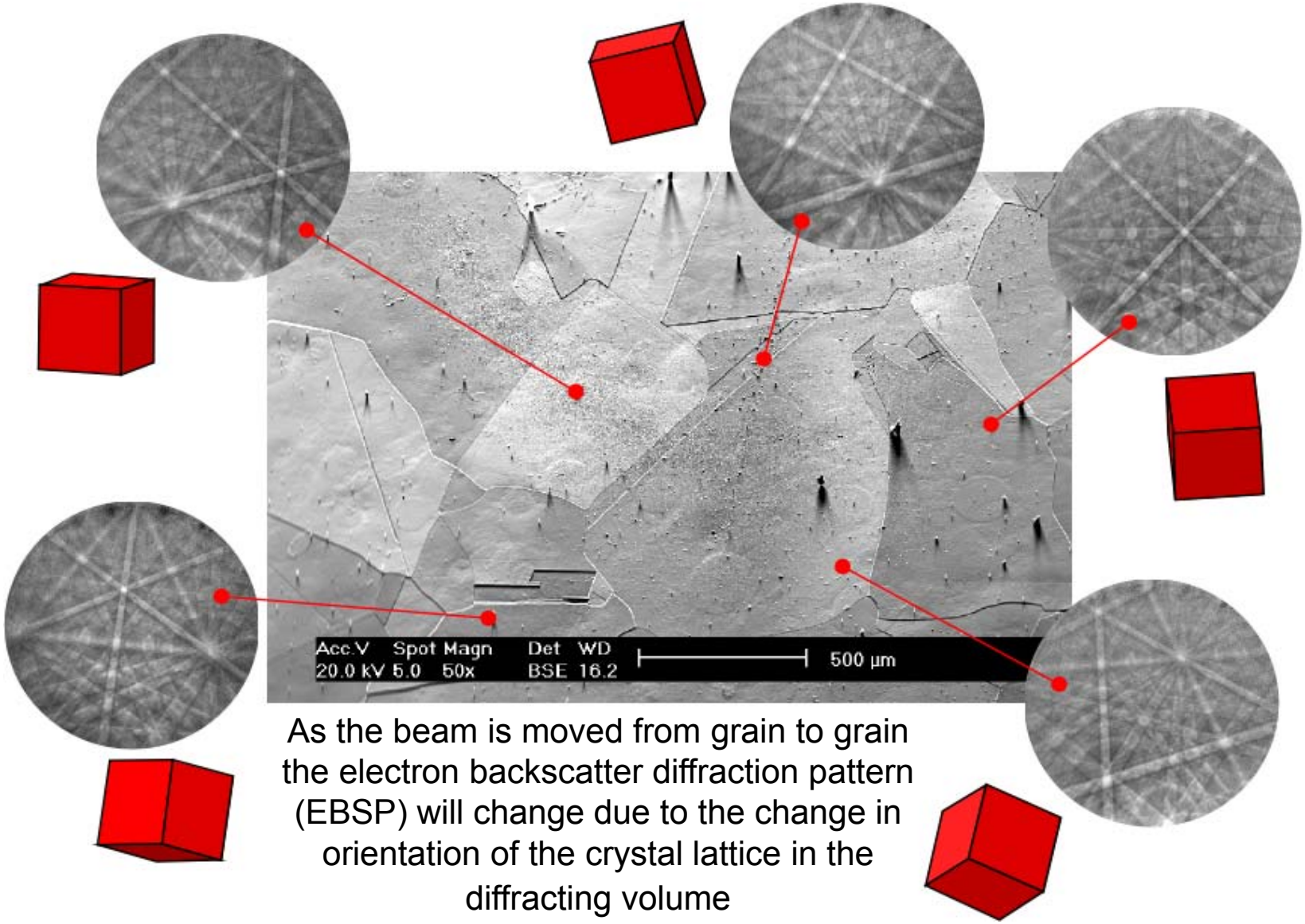
Photograph of an SEM chamber showing typical arrangement for EBSD

Yields crystallographic contrast



A.W. Wilson and G. Spanos, *Materials Characterization*, 46 (2001) 407-418

Figure 1

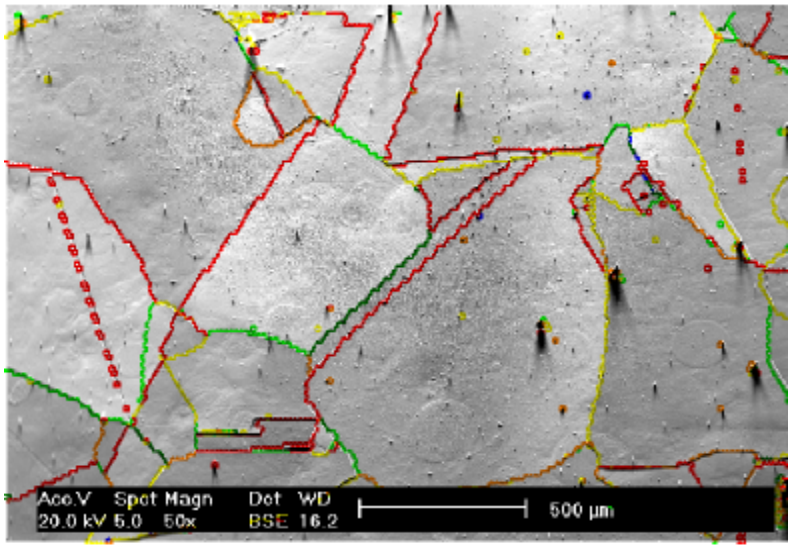


As the beam is moved from grain to grain the electron backscatter diffraction pattern (EBSP) will change due to the change in orientation of the crystal lattice in the diffracting volume

Adapted from http://www.coe.drexel.edu/ret/personalsites/2003/Mosessonm/

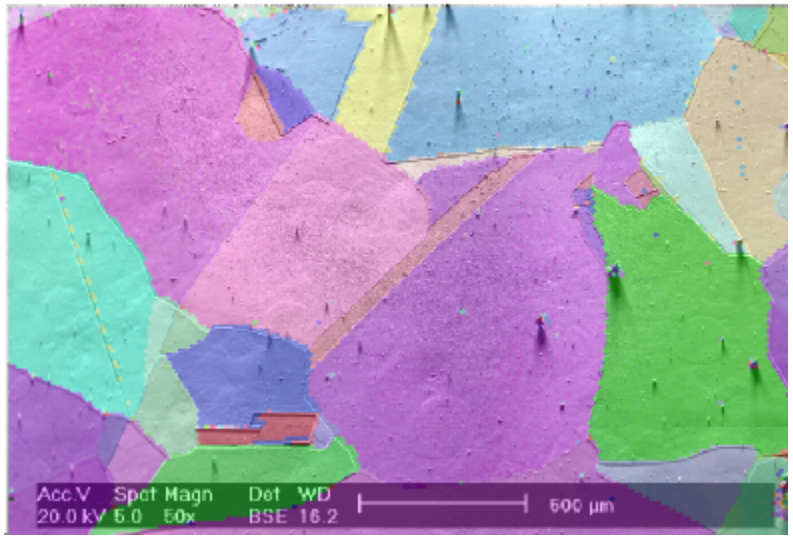
EDAX

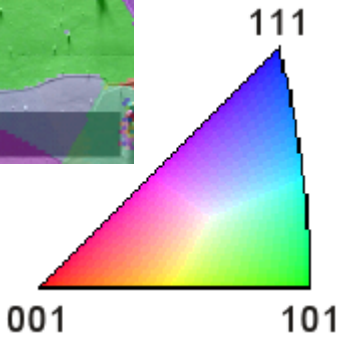
Grain Boundary Map



5-15°
15-25°
25-35°
35-45°
45-55°
55-65°

Orientation Map





A Grain Boundary Map can be generated by comparing the orientation between each pair of neighboring points in an OIM scan. A line is drawn separating a pair of points if the difference in orientation between the points exceeds a given tolerance angle. An Orientation Map is generated by shading each point in the OIM scan according to some parameter reflecting the orientation at each point. Both of these maps are shown overlaid on the digital micrograph from the SEM.

Adapted from http://www.coe.drexel.edu/ret/personalsites/2003/Mosessonm/

SEM vs. TEM

- Image formation
  - TEM – parallel optics and lenses;
  - SEM – focused optics and detectors.
- Depth of field
  - Small apertures yield high magnifications (i.e., diameter of object >> δ) for both; SEM up to 20 μm thickness and TEM up to 200 nm thickness.
- Specimens
  - TEM – lens and holder geometry limit samples to 3 mm dia. and 150 μm thick.
  - SEM – limited by size of chamber.
- Vacuum system
  - Vacuum required for both. Eliminates scattering of electron beam, contamination of specimen and/or microscope components, and gun instabilities.
  - Better vacuum systems are required for TEM than SEM.

CHEMICAL ANALYSIS

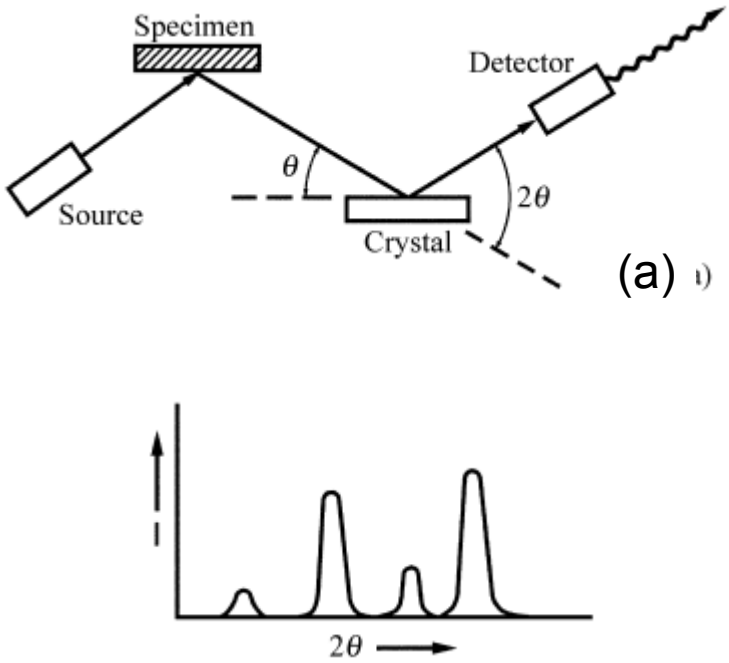
(aka Microanalysis)

Microanalysis in Electron Microscopy

- Characteristic X-rays are always generated by interactions between the incident electron beam and the sample.
- They constitute a ***fingerprint*** of the local chemistry.
- Collect X-ray signal to determine local chemistry.
- Common spectroscopic techniques:
  - Wavelength-Dispersive Spectrometry/Spectroscopy (**WDS**)
  - Energy-Dispersive Spectrometry/Spectroscopy (**EDS**)
  - Micro X-ray Fluorescence (micro XRF)

EDS vs. WDS

WDS



EDS

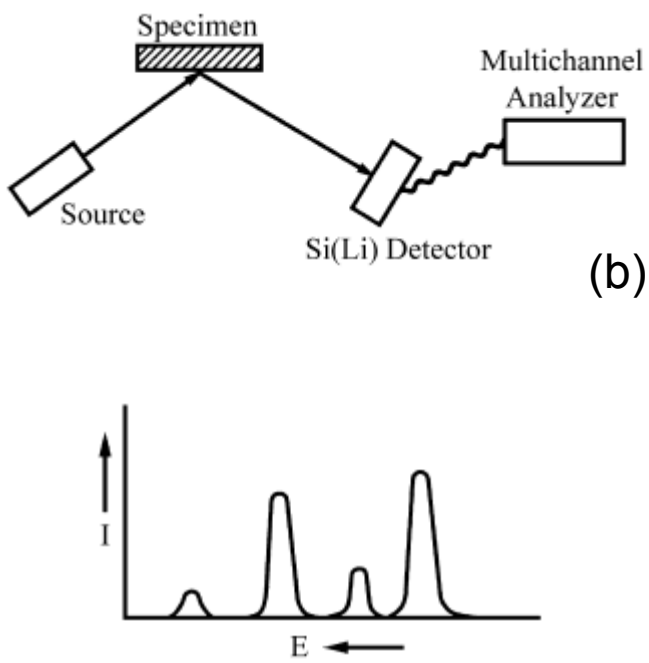
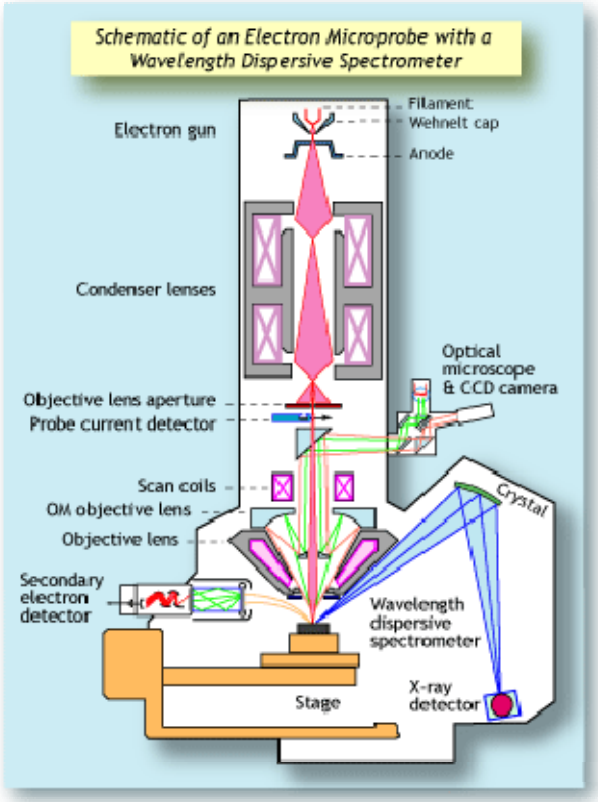


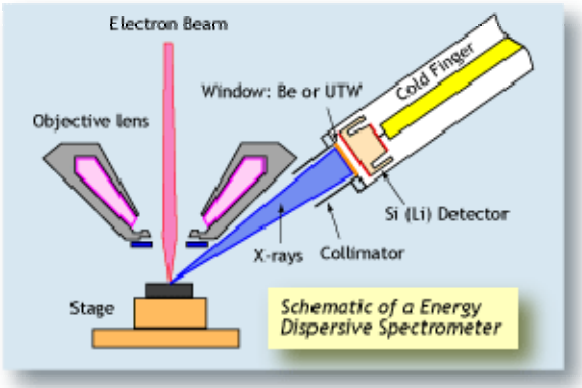
Figure 6.6 Main components and dispersive spectra of : (a) WDS; and (b) EDS. (Figures reproduced from Leng)

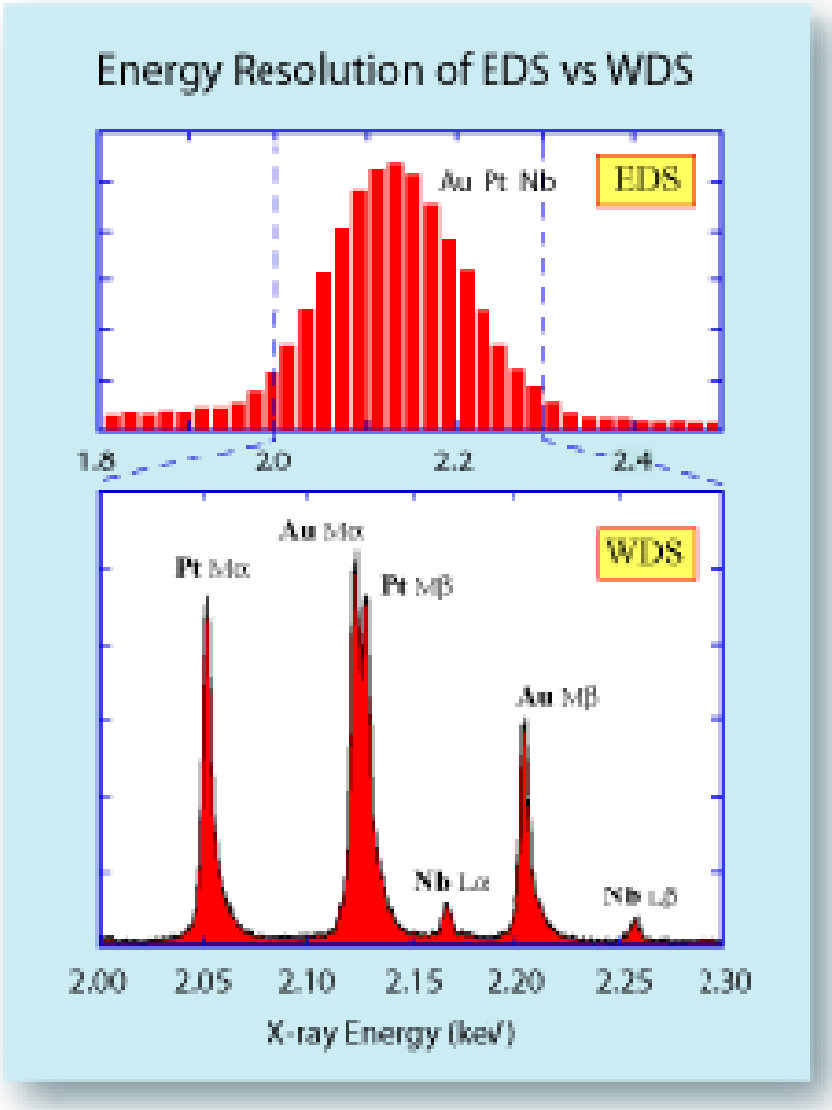
WDS uses single crystal diffraction to detect characteristic wavelengths emitted by specimen

EDS uses photon detector to separate characteristic x-ray photons according to energy

EDS vs. WDS



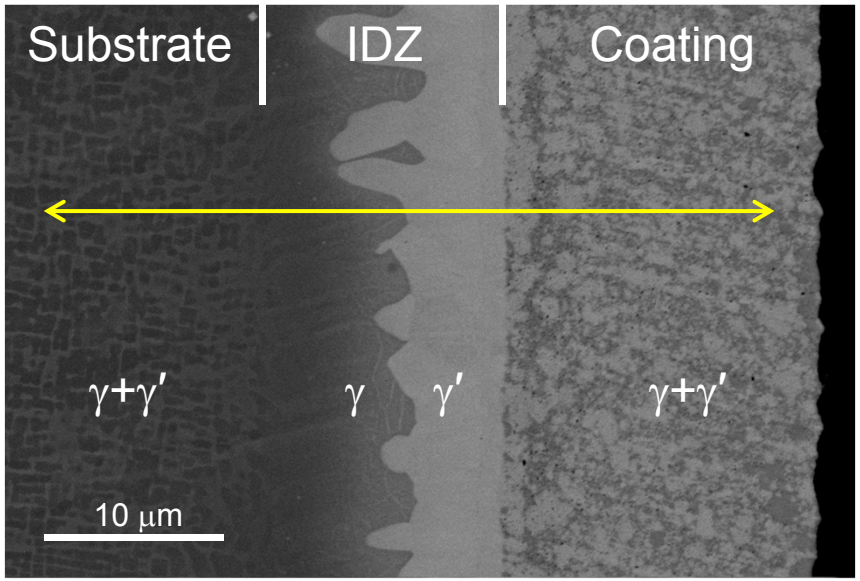




http://www.mcswiggen.com/TechNotes/WDSvsEDS.htm

Backscattered electron mode and WDS analysis

Atomic number contrast



X-ray detectors are common
Allow chemical analysis
EDS, WDS, XRF

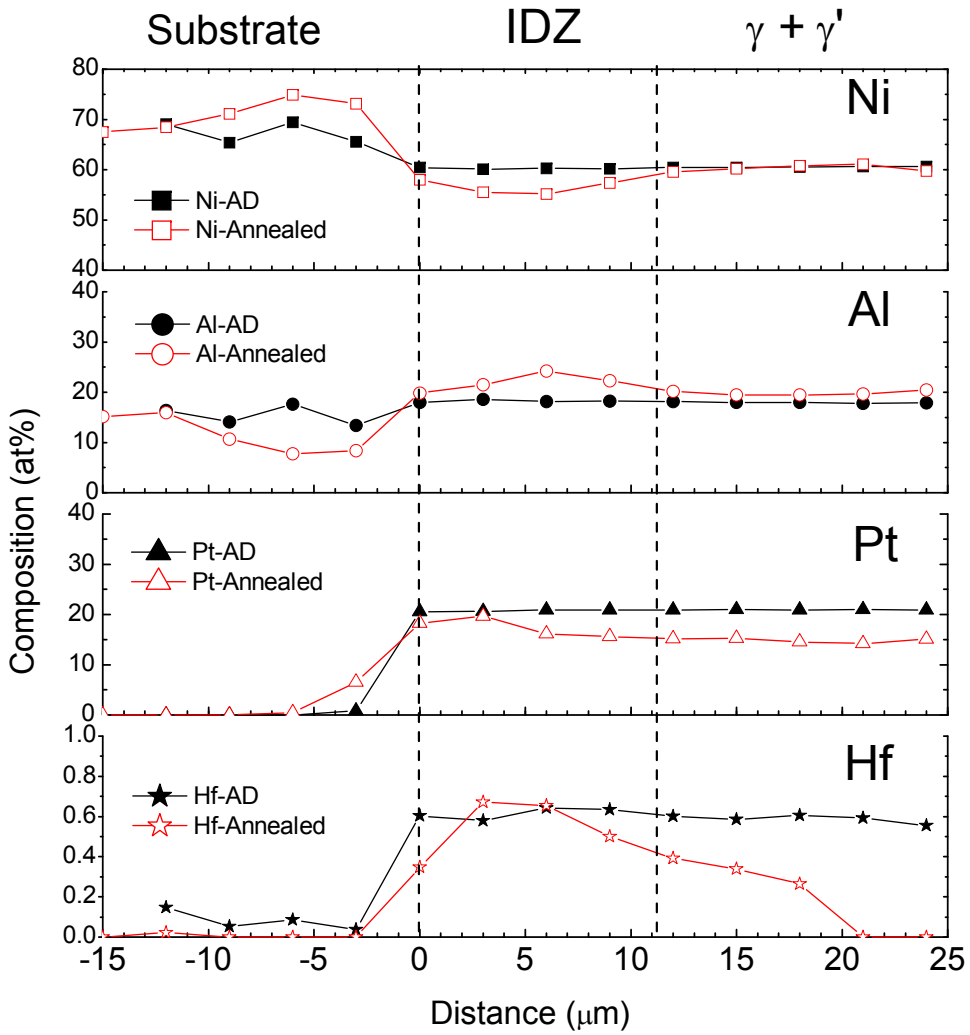


Figure 4.8. Composition profiles of the major elements measured by WDS in an EPMA.

EDS mapping

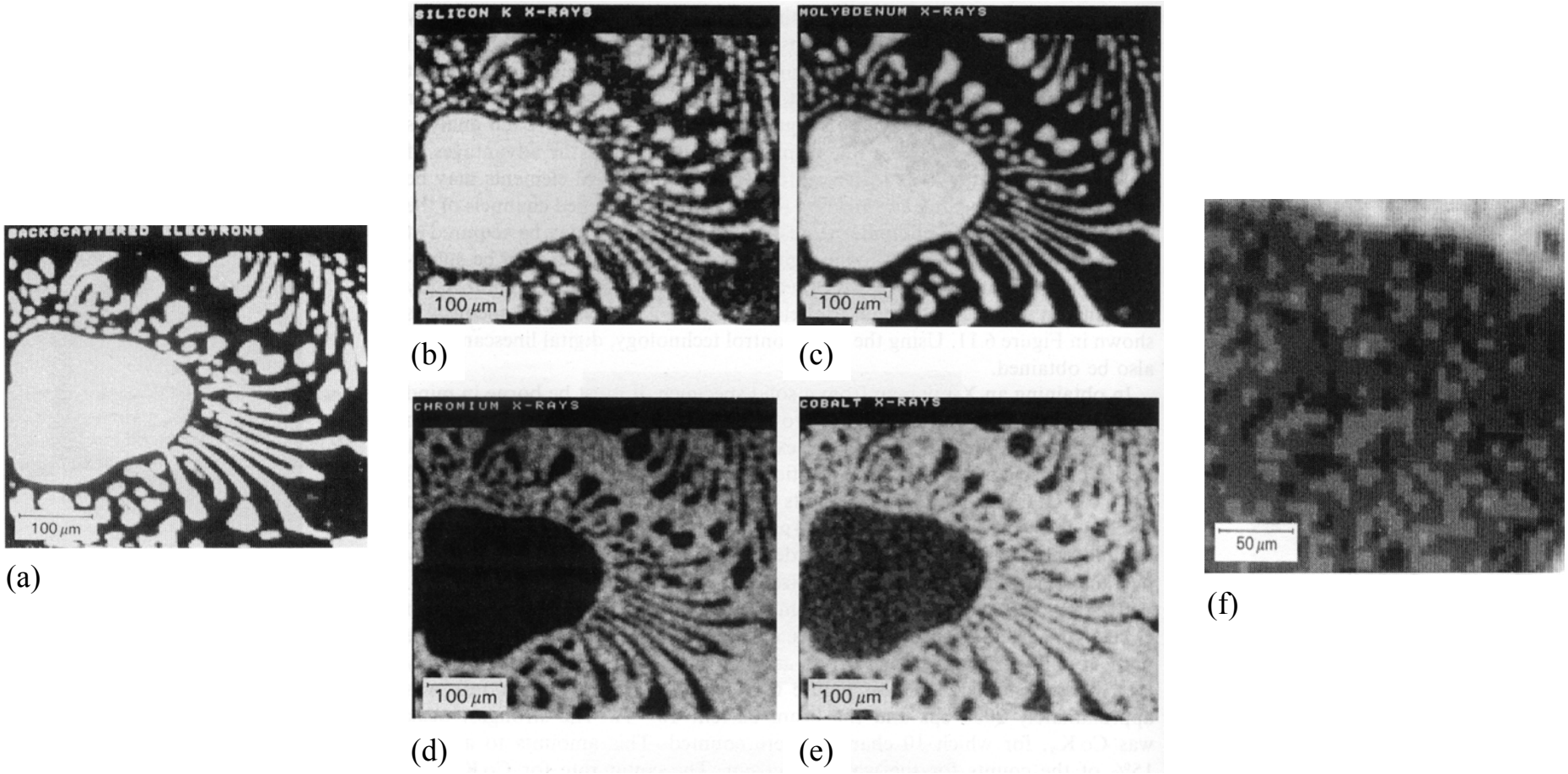
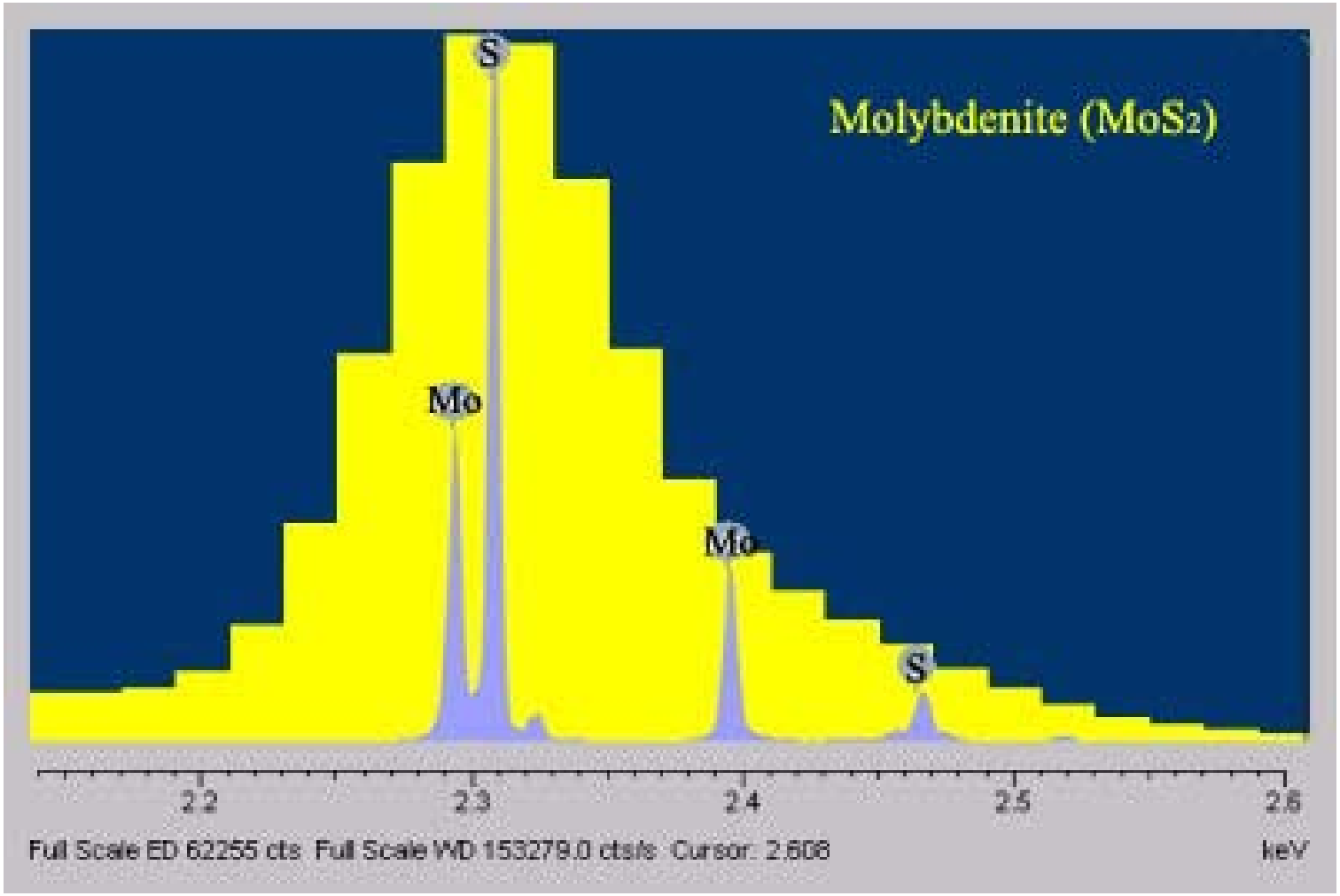


Figure 6.11 EDS maps of a polished specimen of a hard-facing alloy. (a) Backscattered image, (b) Si Lα elemental map, (c) Mo Kα, (d) Cr Kα, (e) Co Kα, and (f) detail of (e) showing the individual pixels. (Figure adapted from P.J. Goodhew and F.J. Humphreys, *Electron Microscopy and Analysis, 3rd ed.*, (Taylor & Francis, London, 1988) pp. 190-191)

WDS

- Yields best discrimination of emitted X-ray signal
- Use a series of bent crystals to cover the range of wavelengths of interest
- Scan wavelengths within each range by rotating the crystal and moving the detector while keeping the position of the crystal fixed.
- X-rays are collected from the sample at a fixed angle. The angle at the collecting crystal will vary with 2θ and the diameter of the focusing circle will change



Comparison of resolution of Mo and S spectral lines in EDS (yellow) vs. WDS (blue). In the EDS spectrum the molybdenum and sulfur lines are overlapped, but can be resolved in the WDS spectrum. Image from Oxford Instruments.

EDS vs. WDS

- Pulse height is recorded by the detector. It is related to the energy of the photon responsible for the pulse.
- Solid-state detectors are generally used.
- EDS is faster than WDS
- Problems with EDS:
  - Poor discrimination or energy resolution. WDS systems are much better, particularly when characteristic lines from different elements overlap.
  - Need a windowless or thin window detector to pick up light elements.
  - WDS is more quantitative