What are Electron Microscopes?

Electron Microscopes are scientific instruments that use a beam of highly energetic electrons to examine objects on a very fine scale. This examination can yield the following information:

Topography

The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties (hardness, reflectivity...etc.)

Morphology

The shape and size of the particles making up the object; direct relation between these structures and materials properties (ductility, strength, reactivity...etc.)

Composition

The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties (melting point, reactivity, hardness...etc.)

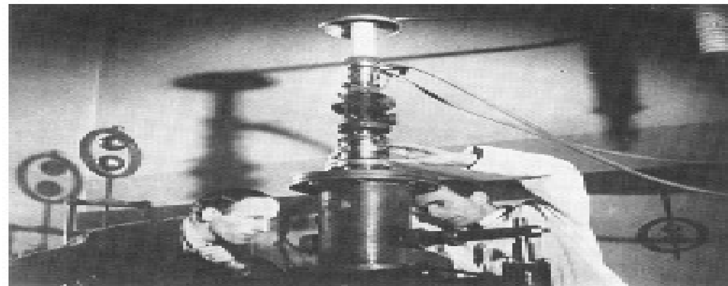
Crystallographic Information

How the atoms are arranged in the object; direct relation between these arrangements and materials properties (conductivity, electrical properties, strength...etc.)

Invention and Evolution of the Modern TEM

- In 1932, invented by E. Ruska *et al.*
- In 1986, Ruska received the Nobel Prize

Ruska & Knoll

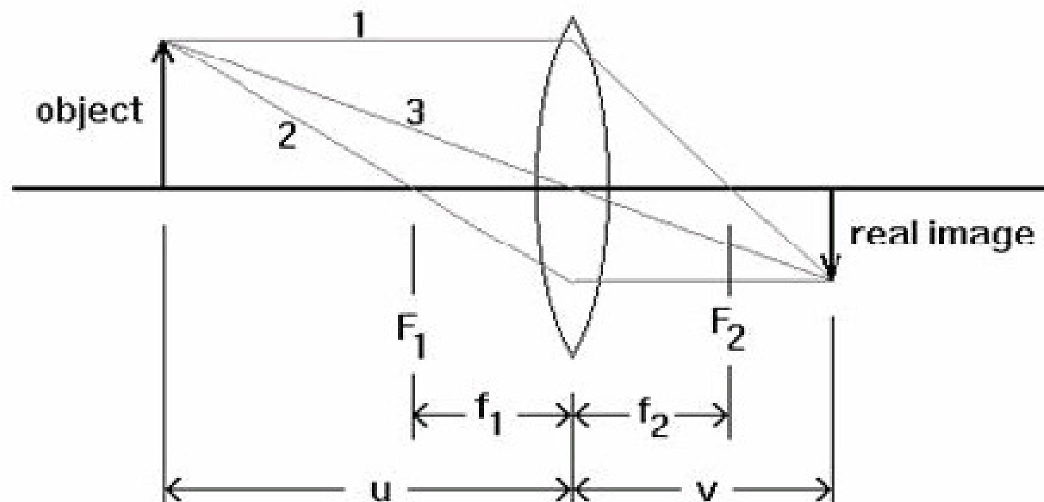


Difference between optical and electron microscopes

- Electron Microscopy bridges the 1 nm – 1 μm gap between x-ray diffraction and optical microscopy

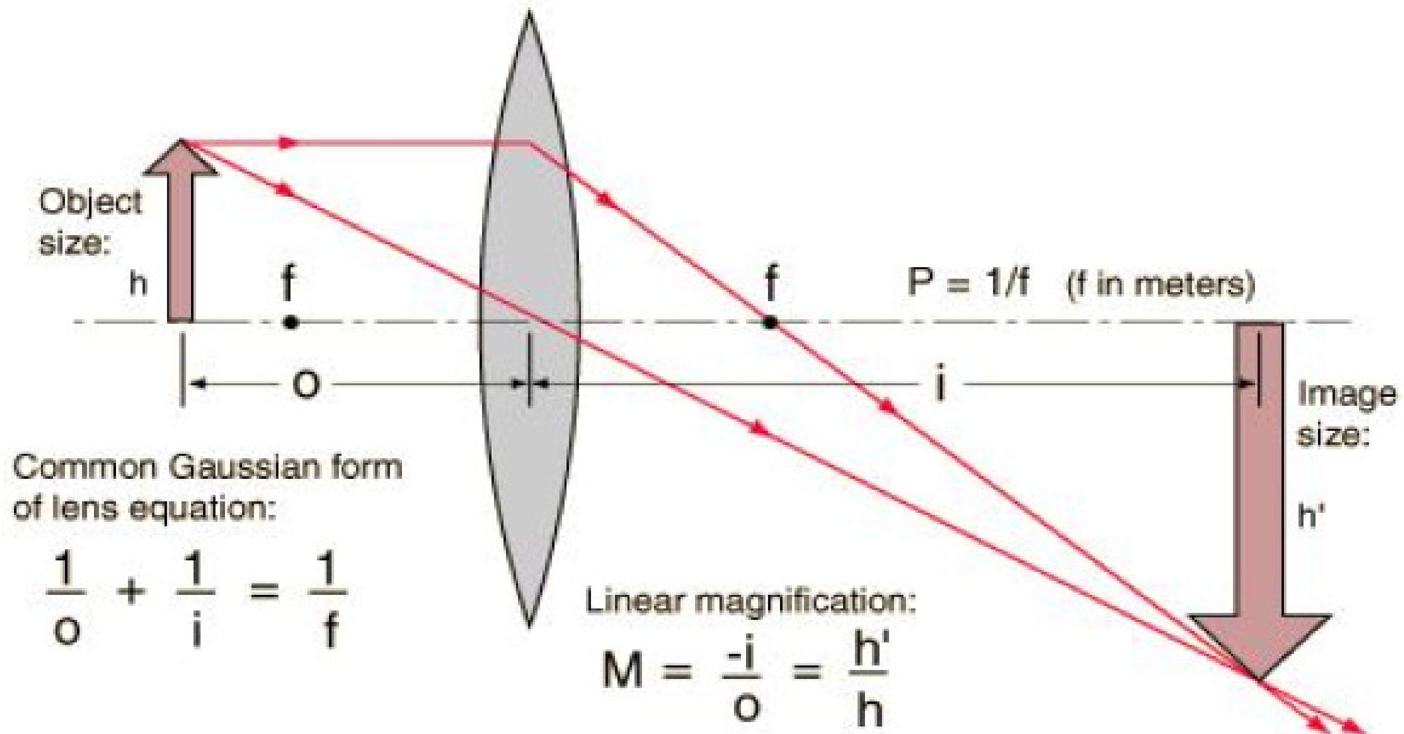
Image formation

- Light rays coming out of an illuminated object diverge from each point on the object
- A lens can be used to refract the rays and converge them at a different location
- This is the basic mechanism of image formation



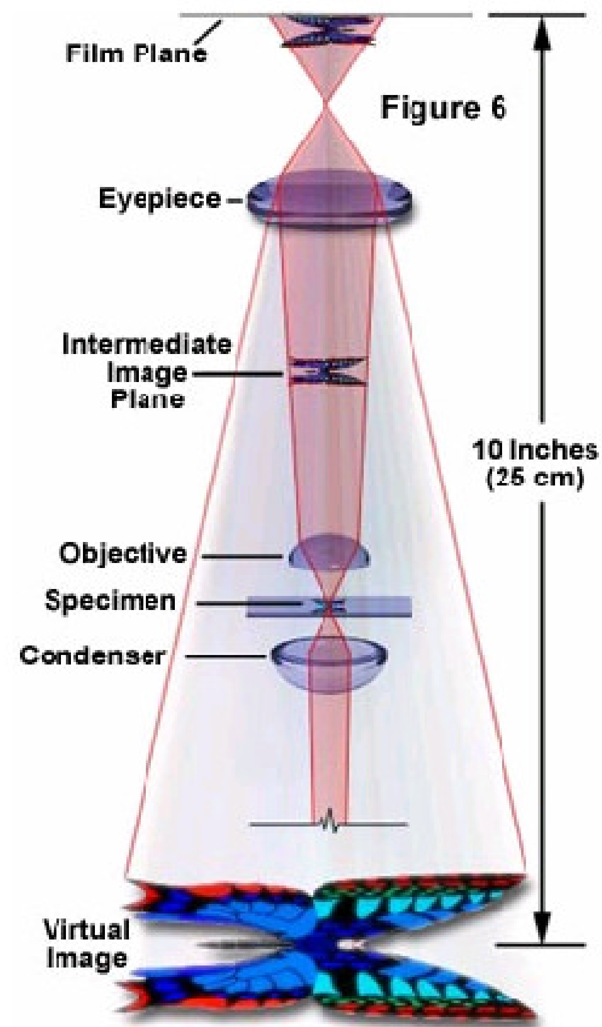
A lens changes the angle of a beam depending on its incidence angle and location of entrance on the lens

De/Magnification



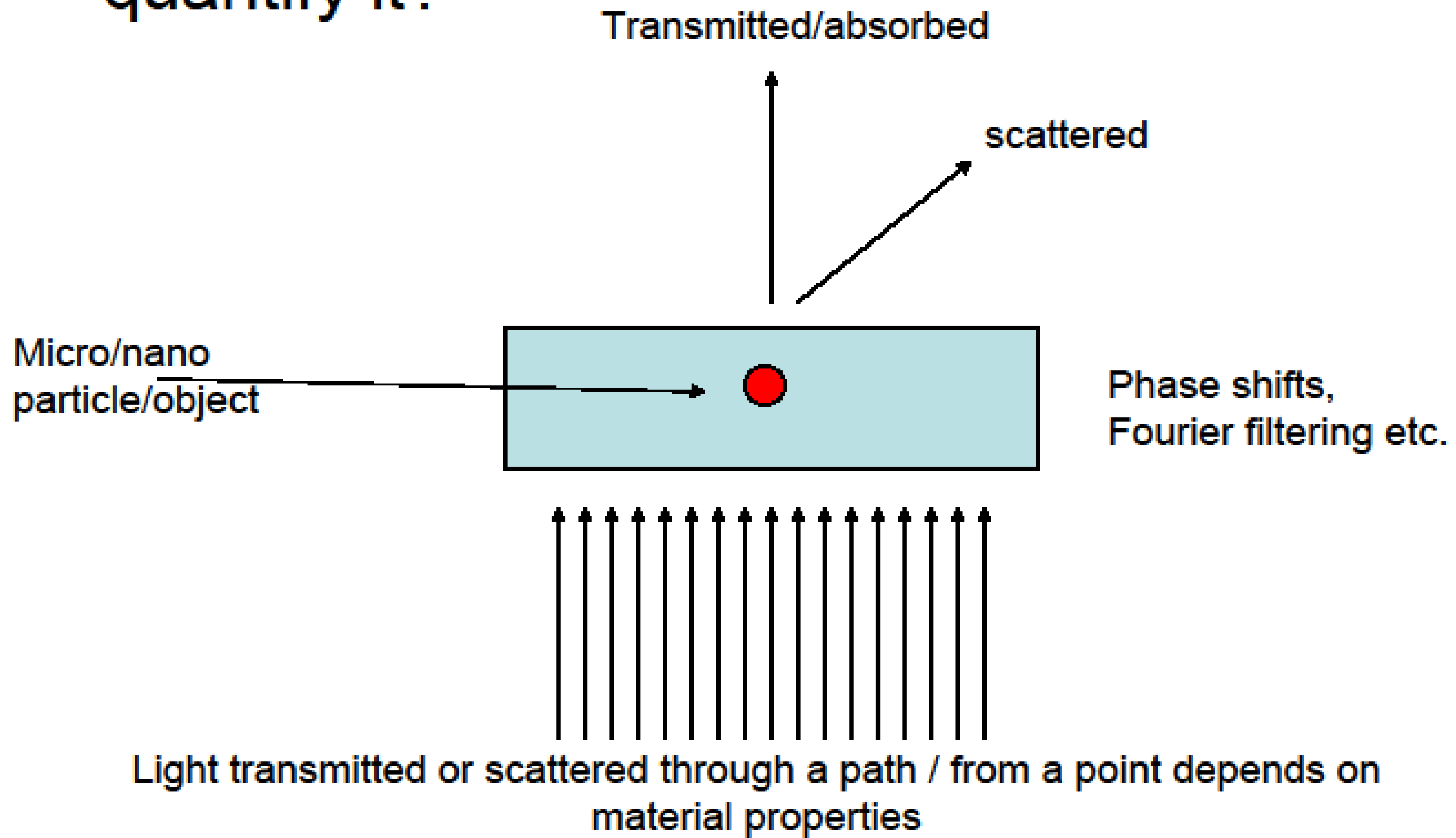
Anatomy of a Light Microscope

- Illumination
  - An even illumination is important for imaging
- Objective Lens
  - Collects light from the sample and nearly collimates it
- Eyepiece
  - Refocuses the light from the objective to form the image



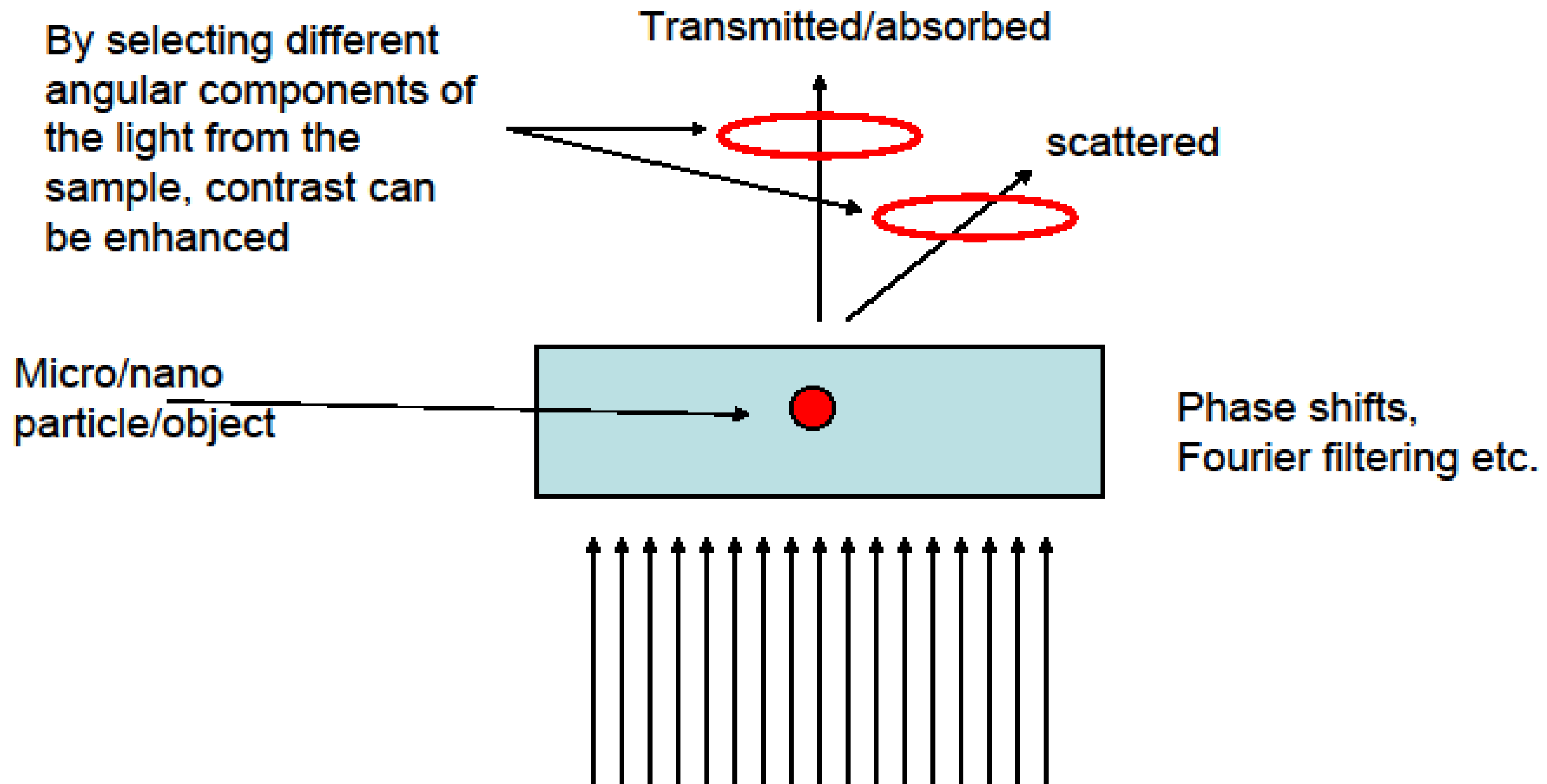
Contrast

- What causes contrast and how can we quantify it?



Contrast Enhancement

By Placing optical components in the beam path, selective imaging is possible



Modulation Transfer Function

- Is a measure of how much of the constrast is imaged

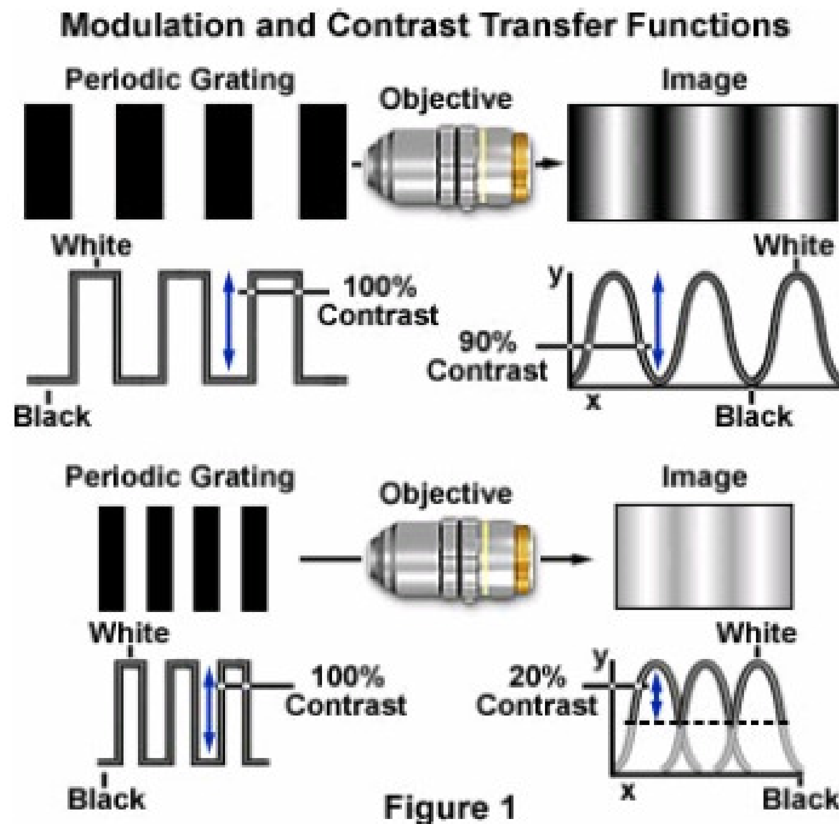


Figure 1

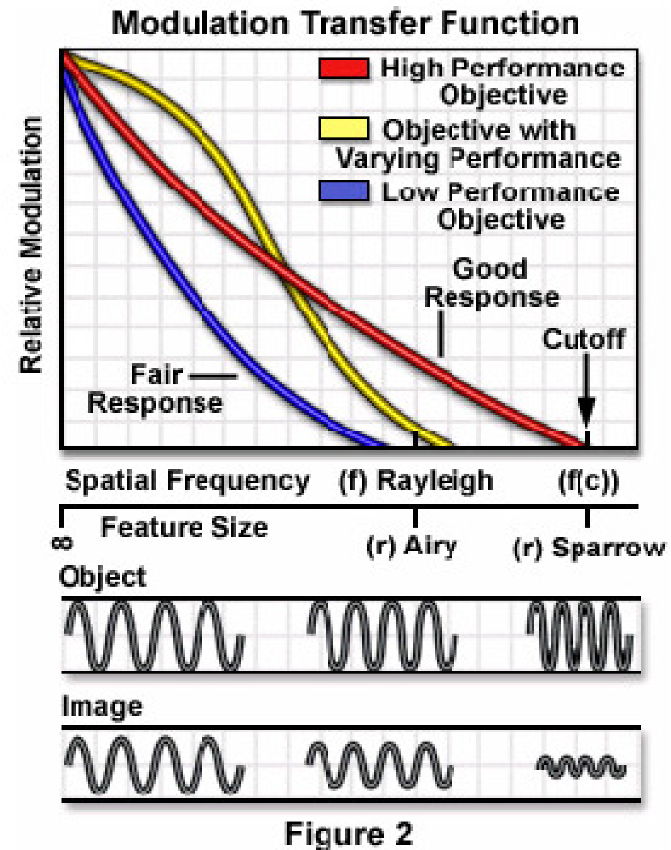
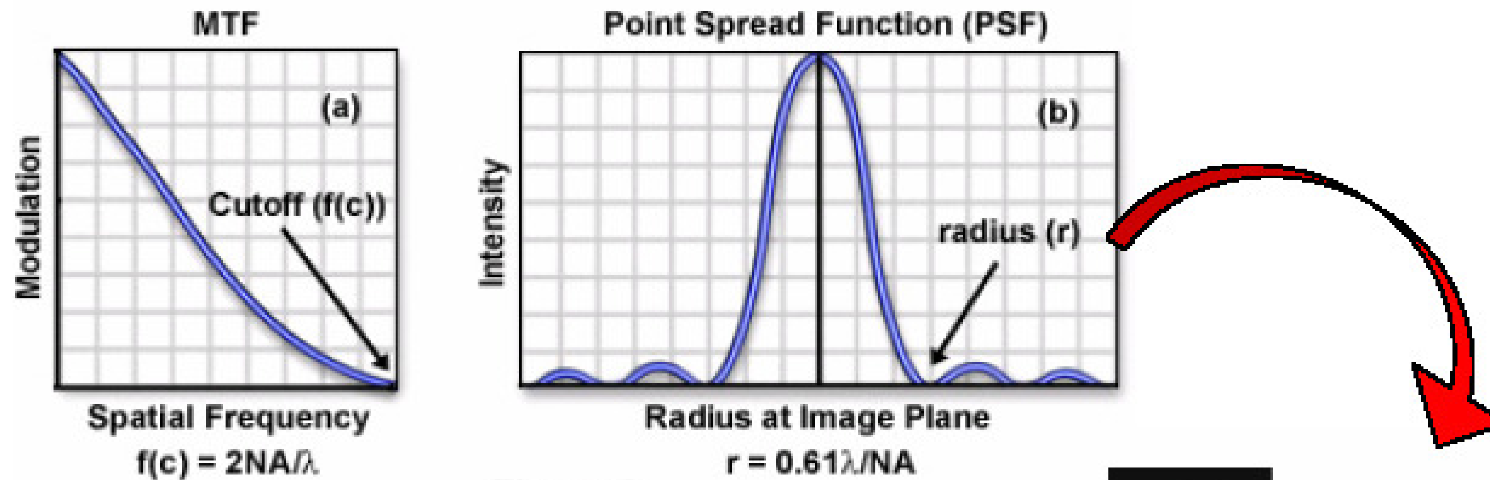
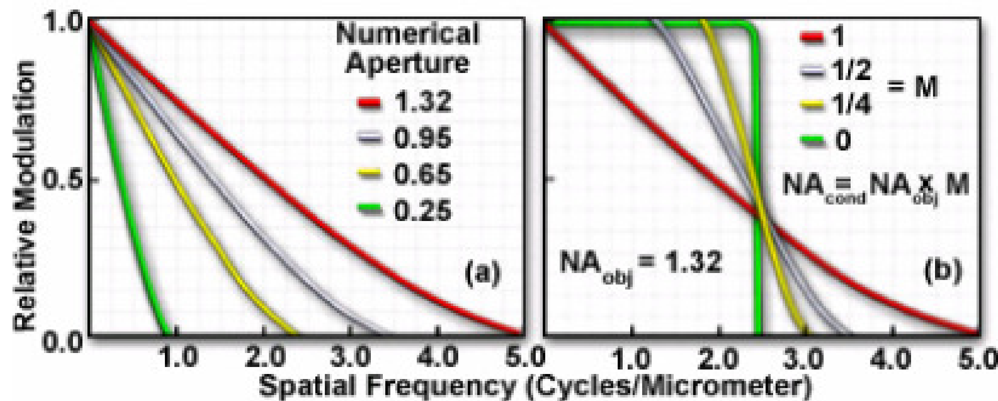


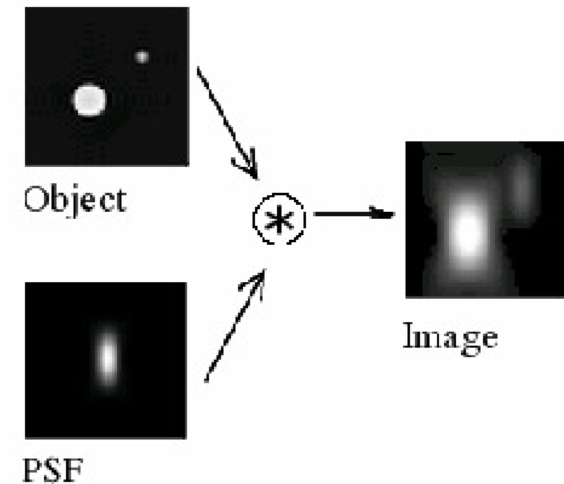
Figure 2

Modulation Transfer Function

- Related to the Point-Spread-Function







Contrast enhancement and MTF

- Contrast enhancement can significantly alter MTF

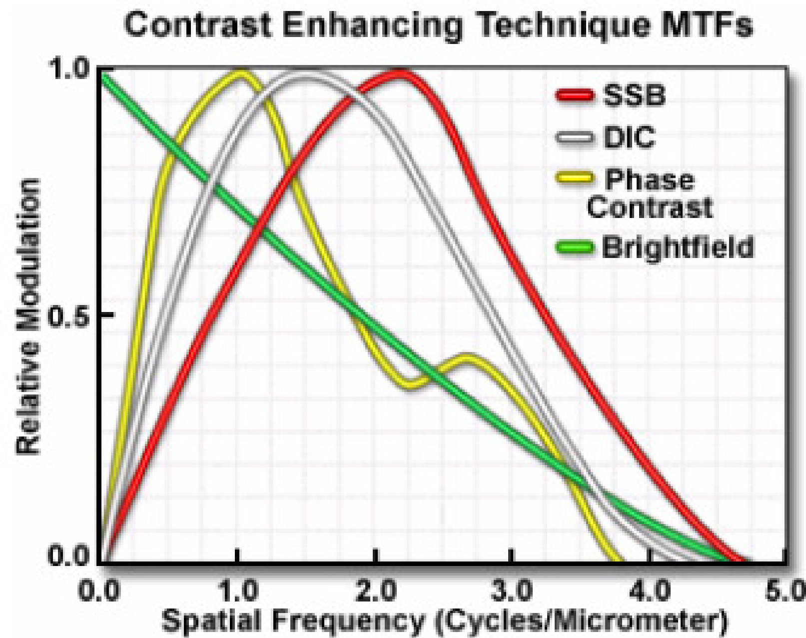


Figure 5

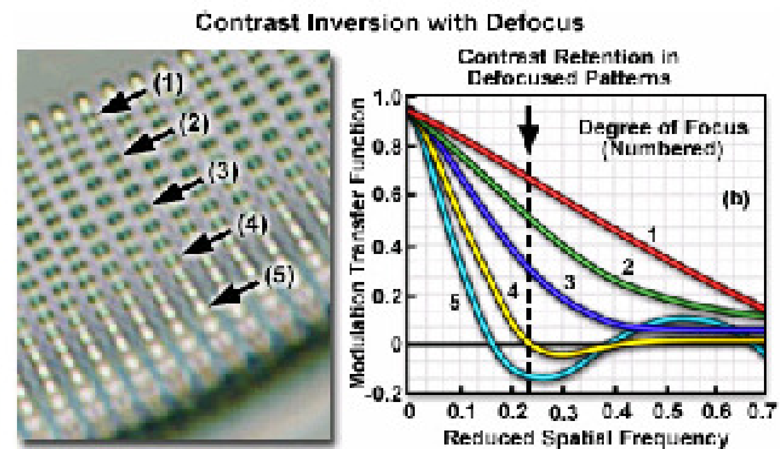
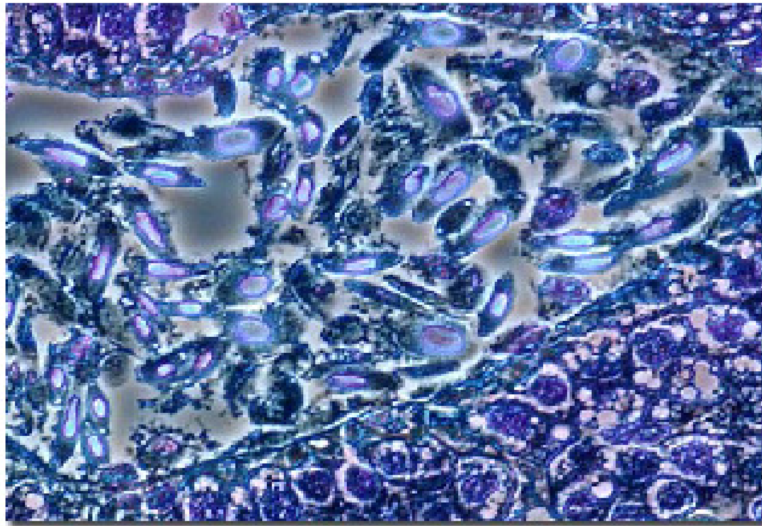
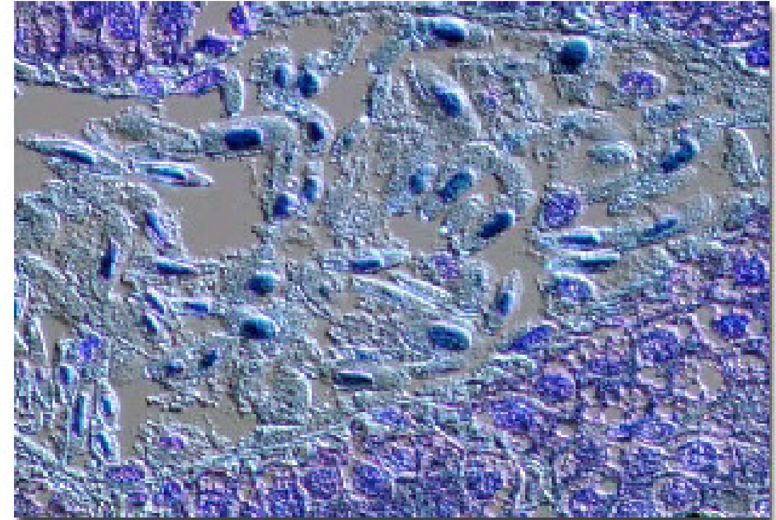


Figure 8

Focus series can be used to get more information

Examples of Contrast enhancement



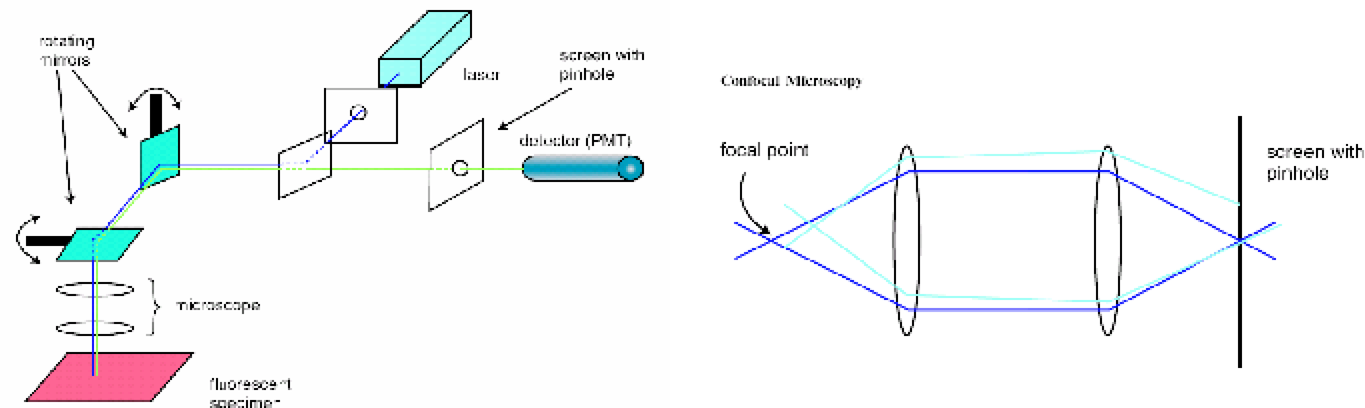


Phase contrast

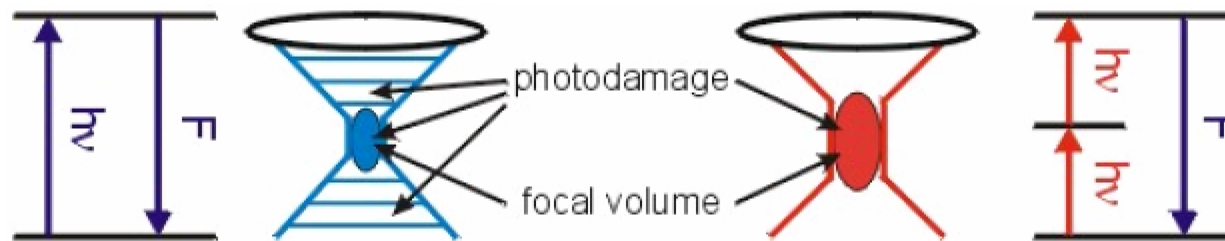
Differential Interference Contrast

Confocal Microscopy

- A laser beam (or sample) is scanned and fluorescence is recorded
- Light is collected from the focused laser spot only
- diffraction limited spot of submicron size



Multiphoton Microscopy



Principle of fluorescence induced by one-photon absorption (left) and two-photon absorption (right). While the resolution in two-photon fluorescence mciroscopy (2PFM) is less good, photodamage is lower and penetration depth is higher compared to single-photon (confocal) fluorescence microscopy (1PFM)

Due to nonlinear nature of two-photon absoprtion, signal comes not from the focal cone but from a smaller focal sphere

Why electron microscopy

- Primary reason: Spot size

$$\lambda_B = \frac{h}{p}$$

$$\lambda = \frac{h}{p} = \frac{h}{mv}\sqrt{1 - \frac{v^2}{c^2}}$$

DeBroglie wavelength of a particle

If speeds are large or total acceleration voltage is close to rest mass of particle
You should better use relativistic formulas for energy, momenta etc.

For an electron with KE = 1 eV and rest mass energy 0.511 MeV, the associated DeBroglie wavelength is 1.23 nm, about a thousand times smaller than a 1 eV photon.

Electron Optical Elements and Attachments

- Electron Sources
- Lenses
- Deflection Coils
- Stigmators
- Electron Detectors
- Attachments for photons or X-rays

Electron Source

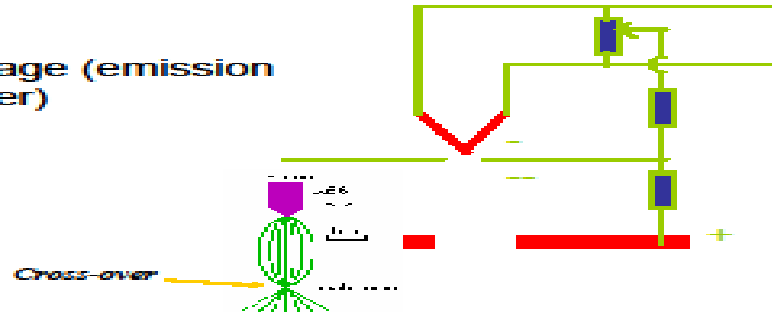
- Generation of electrons that can be accelerated by high tension to obtain the illuminating electron beam

Electron Source

- Thermionic Gun
  - triode or self-biasing gun
  - W, LaB_6, CeB_6
- Field Emission Gun
  - single crystal W

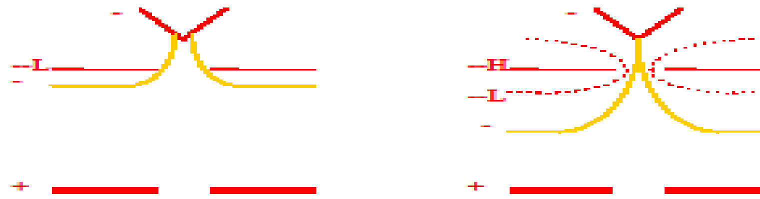
Electron Source
Thermionic Gun

- Filament
- Wehnelt
  - bias voltage (emission parameter)
- Anode



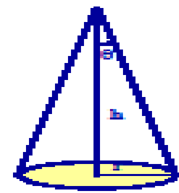
Electron Source
Thermionic Gun

- Increasing bias voltage restricts emission, thereby reducing the total emitted current

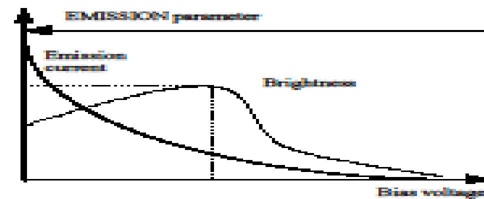


Electron Source
Thermionic Gun

- Brightness = electron current by a source with unit area and unit solid angle



$\Omega = \pi\theta^2$

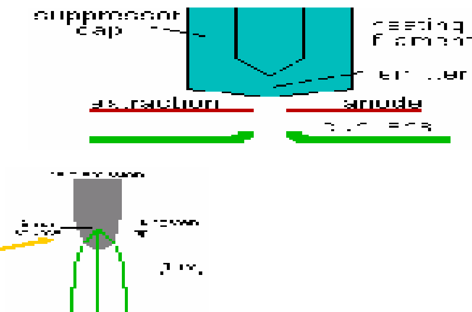


Electron Source
Thermionic Gun

- Energy Spread
  - imperfections of filament
  - instability of high tension
  - surface temperature
  - Boersch effect (mutual interaction)
- Source Spotsize
  - 30 µm for W
  - 5 µm for LaB_6

Electron Source
Field Emission Gun (FEG)

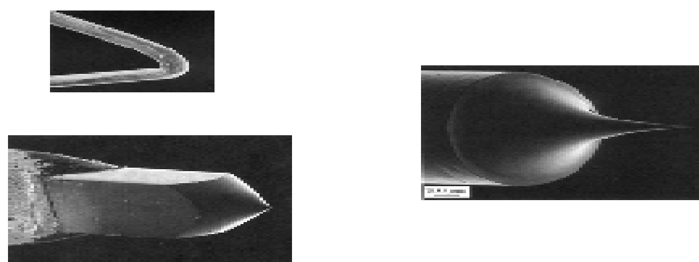
- Heating Filament
- Single Crystal Emitter
- Suppressor Cap
- Extraction Anode
- Electrostatic lens



Electron seemingly originating from tip itself

Comparison of Electron Sources

	W	LaB_6	FEG (Schottky)
Maximum Current (nA)	1000	500	300
Normalised Brightness (-)	1	10-30	2500
Energy spread (eV)	3-4	1.5-3	0.6-1.2
Source spotsize	30-100 μm	5-50 μm	15-30 nm
Required Vacuum (Pa)	10^{-3}	10^{-5}	10^{-7}
Temperature (K)	2700	2000	1800
Life time (hr)	60-200	1000	>2000
Normalised Price (-)	1	10	100



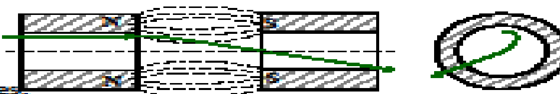
Lenses

- Provide means to (de)focus the electron beam on the specimen, to focus the image, to change the magnification, and to switch between image and diffraction

Round Lenses

Magnetic lenses

- change the direction of electrons
- magnifying (diverging)
- diminishing (converging)
- condenser lenses, objective lenses,
- intermediate lenses, projection lenses

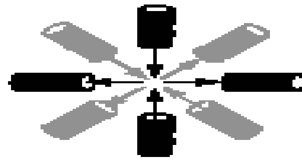


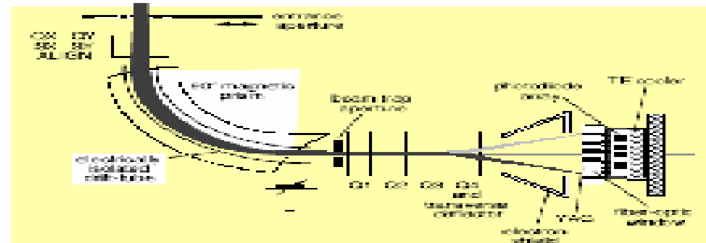
Electrostatic lenses: the Wehnelt cap

- Advantage
  - rotation free
- Disadvantage
  - high precision in construction
  - high precision in alignment
  - extreme cleanliness

Pole Lenses

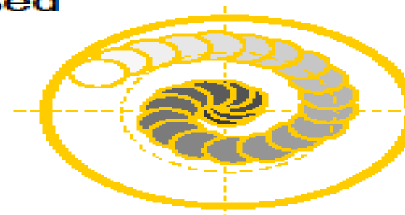
- Pole lenses are all electromagnetic, no electrostatic
- Different magnifying power in X, Y direction is possible
- The construction is just like the stigmators
- Usually seen in Cs correctors and EELS
- Qudrapole, Hexapole, Octupole lenses are common.





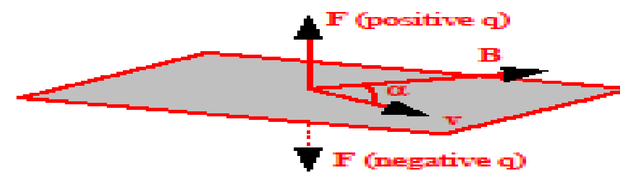
Lenses

- Electromagnetic lenses are based on the fact the moving electrons are forced into a spiral trajectory, i.e. focused into one point



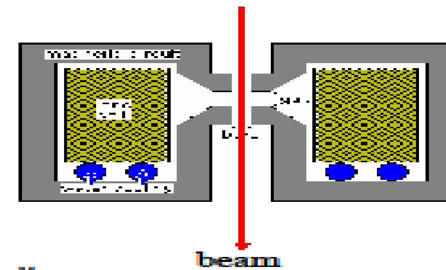
Lenses

- Working Principle: Lorenz Force
  - electrons are only *deflected* by magnetic fields



Lenses

- the focal length is given by:



$$f = \frac{K \cdot U}{(N \cdot I)^2}$$

K : constant
U : accelerating voltage
N : windings
I : lens current

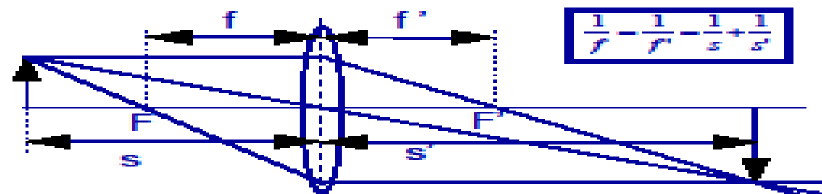
Electromagnetic Lenses for Electrons

- Focus
- Magnification and demagnification
- Electron trajectory changed by magnetic field
- F = - e v x B
- F = evB sinθ
- If v // B, F = 0

$$R = \frac{m_0 v}{eB}$$

Lenses

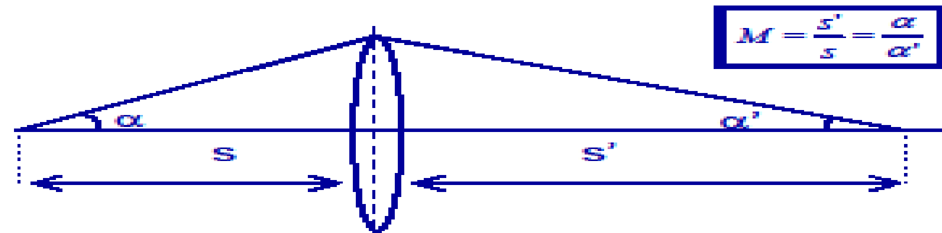
- Gaussian Law



$$\frac{1}{f} = \frac{1}{f'} = \frac{1}{s} + \frac{1}{s'}$$

Lenses

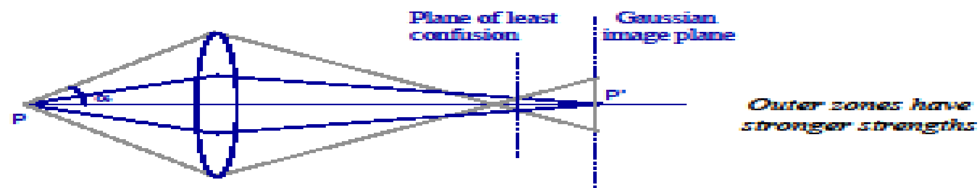
- Gaussian Law



Lenses
Spherical Aberration

$$\delta_s = C_s \alpha^3$$

- Lens imperfections lead to different focal lengths in centre and at edges of lens



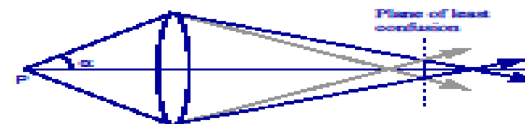
Lenses
Spherical Aberration

- C_s can be reduced by:
  - increasing the lens strength
  - decreasing the lens gap

Product	C_s objective	Lens Gap	Focal Length	Tilt Angle	Point Resolution
Tecnai 12-BioTWIN	6.3 mm	20 mm	6.1 mm	±80°	0.49 nm
Tecnai 12-TWIN	2.0 mm	9 mm	2.7 mm	±70°	0.34 nm

Lenses
Chromatic Aberration

- Blurring due to energy spread in electron beam and lens current fluctuations

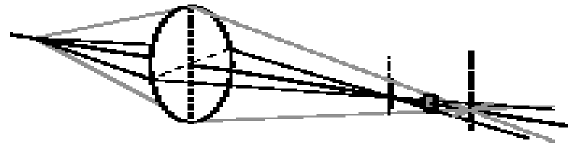


$$\delta_c = C_c \alpha \left(\frac{\Delta E}{E} + \frac{2\Delta I}{I} \right)$$

Lenses

Astigmatism

- Lens defect caused by magnetic field asymmetry



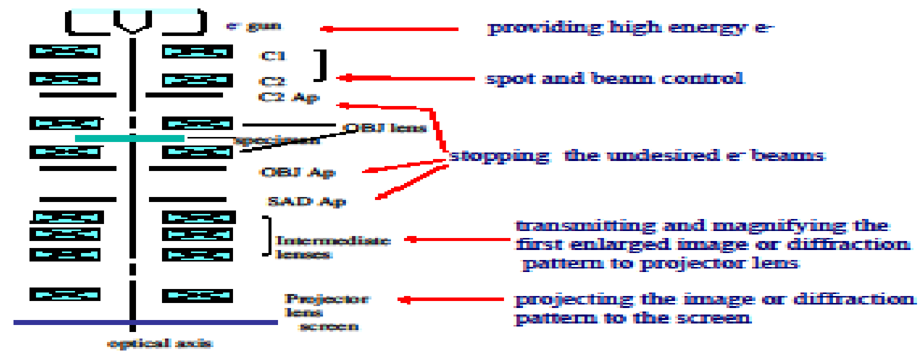
- can be corrected using stigmators!

Lens System

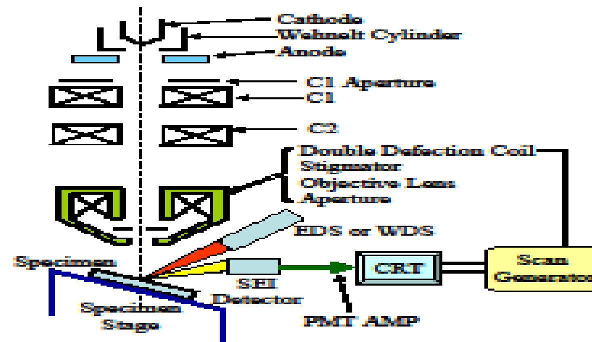
- Condenser C1Lens
- Condenser C2 Lens
- Objective Lens

- Imaging Lenses (TEM)
  - diffraction (1st intermediate lens)
  - intermediate
  - projector

Lens System of TEM



Lens System of SEM



Lens System
Condenser C1 and C2

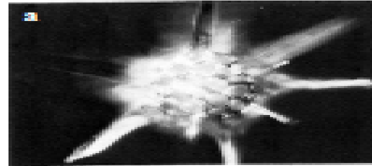
- C1
  - strong demagnifying lens
  - spotsize setting
- C2
  - weak lens
  - intensity control

Lens System &
Microscope Resolution

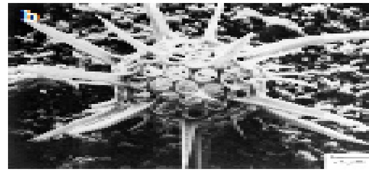
- Microscope resolution is governed by: (for TEM)
  - wavelength of electrons
  - C_s of objective lens
  - other lenses are less crucial (α/M)

$$\delta = 0.66 \times C_s^{1/4} \lambda^{3/4}$$

Depth of Field or Depth of Focus



OM image

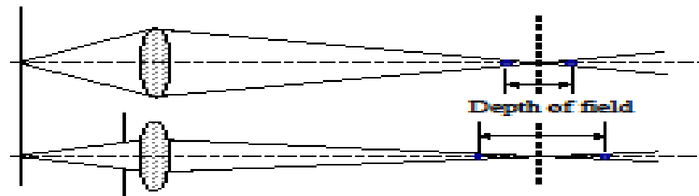


SEM image

How to increase the depth of focus of SEM image

Smaller α

(1) use smaller OBJ aperture
(2) increase Working Distance

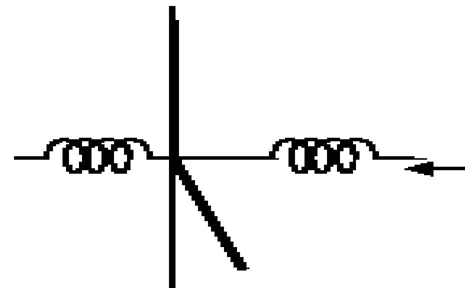


Deflection Coils

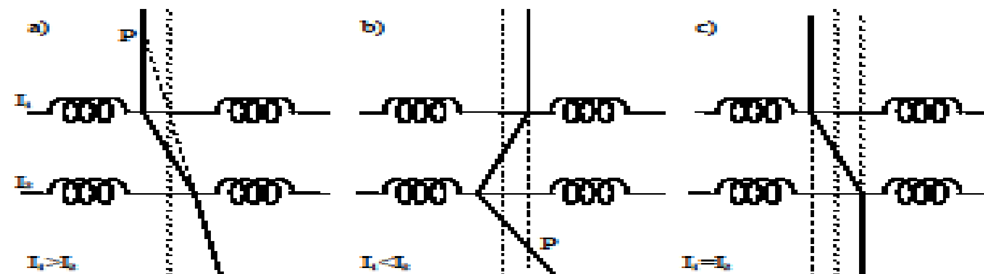
- Provide means to shift or to tilt the electron beam, to correct for mechanical misalignments of the optical system, and to obtain specific imaging effects

Deflection Coils

- Basic Principle
  - Gun coils
  - Beam coils
  - Image coils
  - Scanning coil
  - ....
  - ......



Deflection Coils

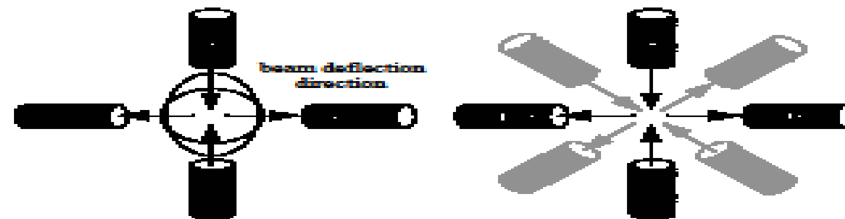


Stigmators

- Provide means to correct for deficiencies in the magnetic lenses
- EM stigmators:
  - At condenser, objective and diffraction lens (TEM)
  - At condenser, objective (SEM)
  - closely positioned to the lenses

Stigmators

- Working Principle



Electron Detectors

- TEM
  - phosphor screen, Film, CCD, Image Plate…
- SEM
  - SE detector, BE detector….
- STEM
  - BF detector, DF detector, ………

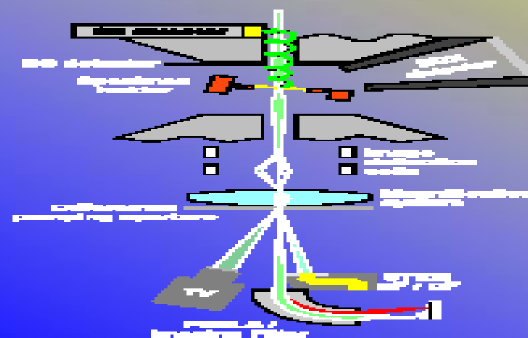
Attachments for photons or X-rays

WDS:
- Crystal Spectrometers
- detecting the wave-length of characteristic X-rays
- Gas proportional counter is used as the X-ray detector
- Single-Channel Analyzer (SCA)
- Long acquisition time (~ 30 min.)
- High energy resolution (~ 5 eV)

EDS:
- Solid State X-ray Detectors
- detecting the energy of characteristic X-rays
- Si(Li) detector is used as the X-ray detector
- Multi-Channel Analyzer (MCA)
- Short acquisition time (100 ~ 200 s)
- Low energy resolution (133 eV for Mo K_α)

•CL:
- detecting the photons

Signals and Detectors



- In TEM
  - Energy Filter
  - TV / CCD camera
  - Plate camera
- In STEM
  - BF / DF
  - HAADF
  - BS & SE (SEM)
- In STEM and TEM
  - EDX and PEELS

The instruments and techniques

- Stationary Electron Beam
-- TEM: CTEM SAD/BF/CDF/WBDF, HRTEM
-- AEM: CBED, NBD, EDS, EELS, and EFTEM

- Scanning Electron Beam
-- STEM (BF, DF, and HAADF)
-- SEM (SEI, BEI)
-- SEM + WDS = EPMA

- Modern TEMs are all capable of HR works, but for some analytic works, attachments such as EDS and EELS must be added.

AEM vs. Conventional TEM

(Differences in aimed signals)

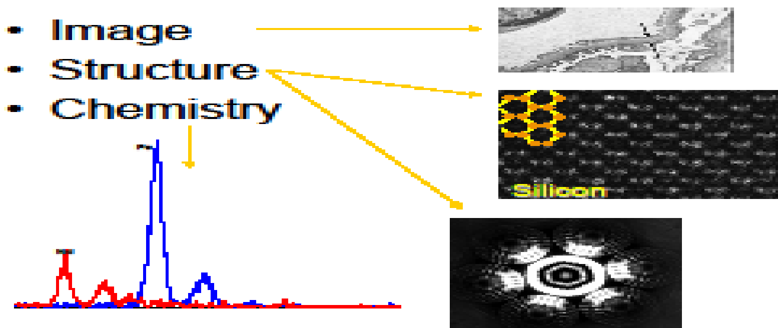
- CTEM and HREM deal mainly with the **elastically scattered** electrons.
- AEM deals mainly with the **in-elastically scattered** electrons and their resulting X-rays (by EELS or EDS) for the composition determination. But **elastically scattered** electrons are also collected to obtain structural information (by STEM).

AEM vs. Conventional TEM

(Differences in Instrumentation)

- Different illumination requirements: parallel illumination for CTEM (and HRTEM) but conical illumination for AEM
- Different designs for the objective lens to match the illumination system
- With analytical attachments: EDS for characteristic X-rays, EELS for in-elastic scattered electrons, and annular detectors for incoherent elastic electrons.
- Scanning function

Types of Information from AEM

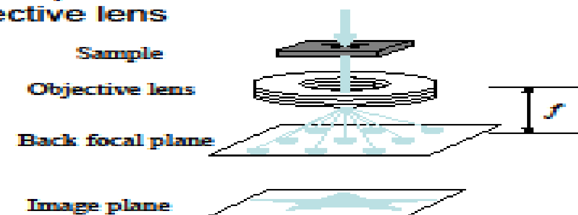


Examples of AEM Applications to the Characterization of Materialss

- Morphology (imaging): CTEM (BF,DF), HRETM, and STEM (BF,DF, and HAADF)
- Crystal Structure (diffraction): SAED, NBED, and CBED
- Chemistry: composition (EDS,EELS, and STEM HAADF), chemical state (EELS)

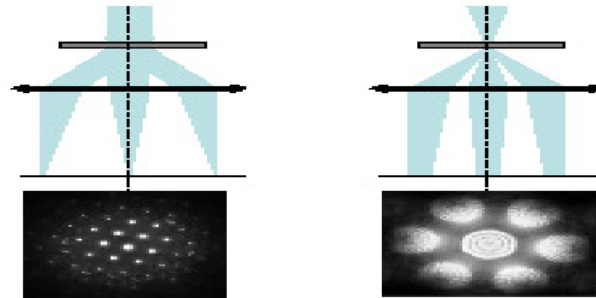
Electron diffraction

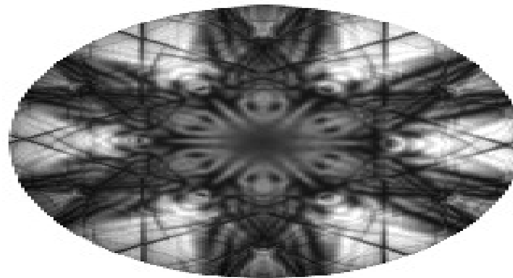
- Diffraction pattern locates at the back focal plane of the objective lens



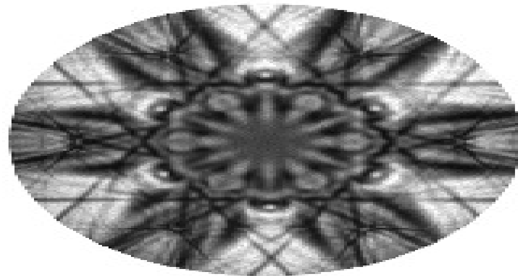
Diffraction with parallel illumination and conical illumination

- Parallel beams are focused at the back focal plane
- Parallel illumination results sharp spots at the plane
- Conical illumination results discs at the plane





LACBED pattern along [111] of GaAs with buried InAs quantum dots



LACBED pattern along [111] of Ge

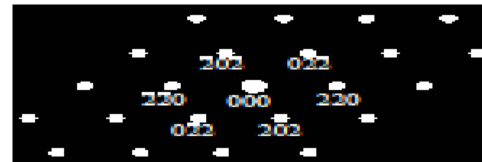
Spot pattern

- Single crystal within the illumination area
- The regular arrangement of spots
- Spot brightness relates to the structure factor
- Spot position relates to the d-spacing

Standard spot pattern

- Example 1: f.c.c

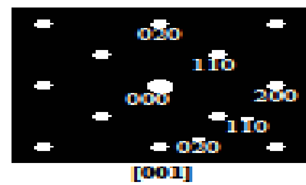
[001]



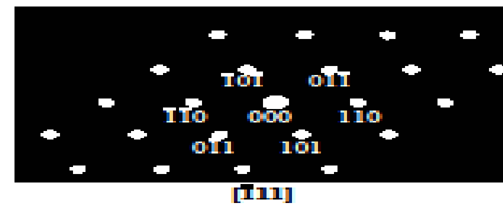
$[\bar{1}11]$

Standard spot pattern

- Example 2: b.c.c

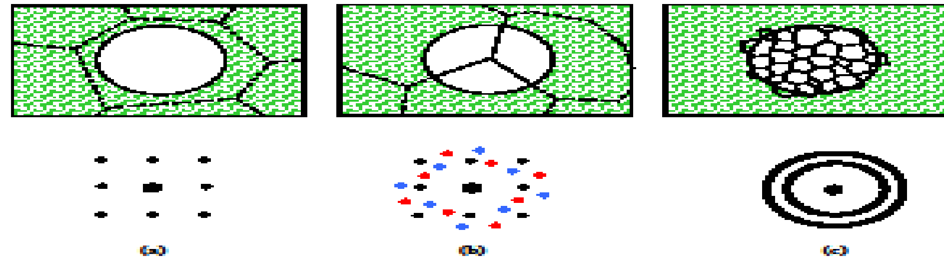


[001]

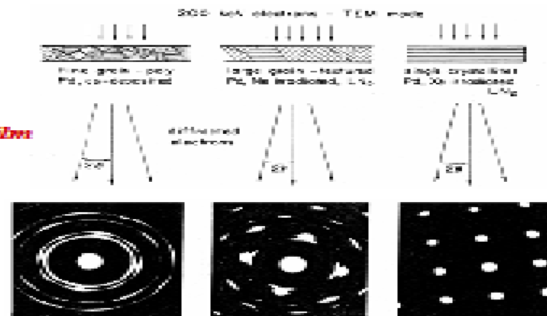


$[\bar{1}11]$

Electron Diffraction Pattern--Spot to Ring

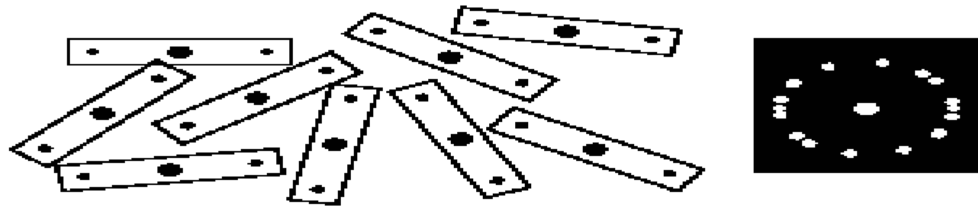


Electron Beam Diffraction of a Pd film



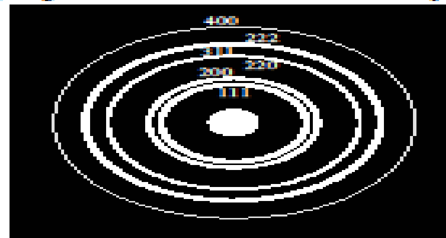
Ring pattern

- Many fine particles in the illumination area, each of them is a single crystal and orientated randomly



Ring pattern

- Typical polycrystalline Au diffraction pattern

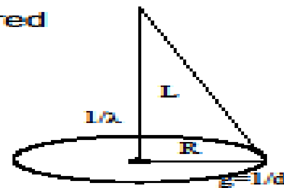


Ring pattern: what can we obtain

- d-spacing

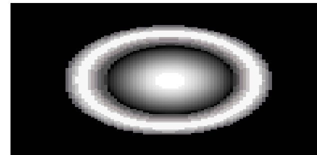
  $$Rd_{hkl} = L\lambda$$

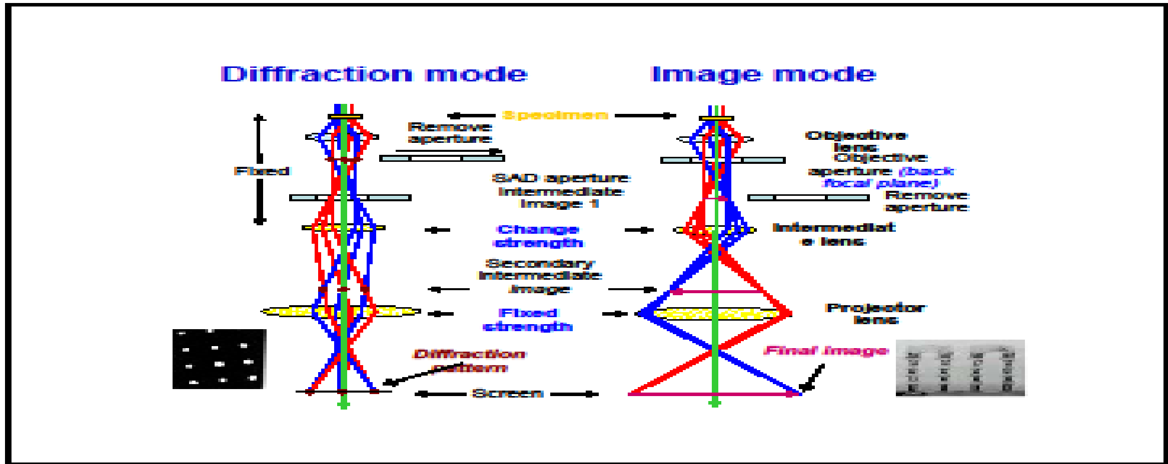
  R: the measured ring radius
  d_{hkl}: the d-spacing being measured
  L: camera length
  λ: wave length of electron beam
- Camera length calibration
- Crystalline / particle fineness

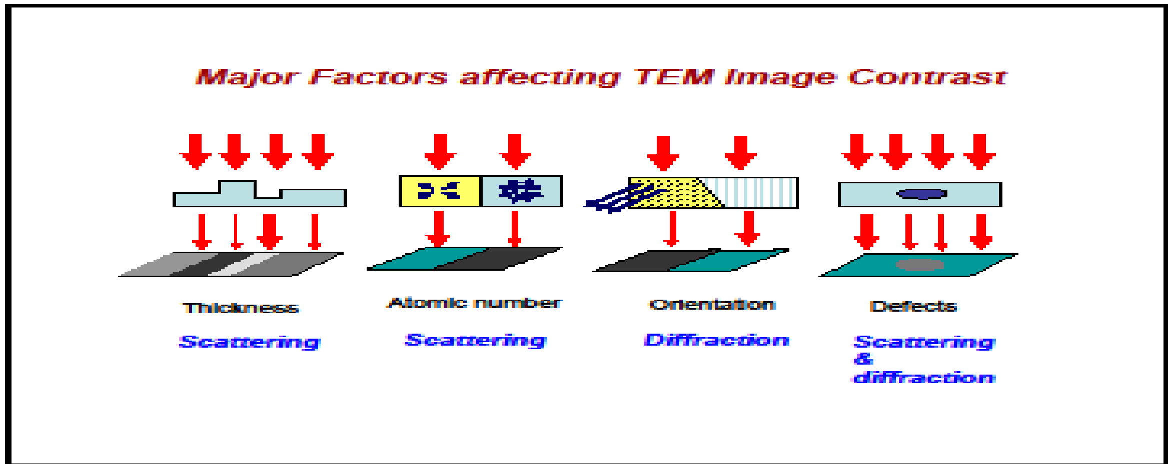


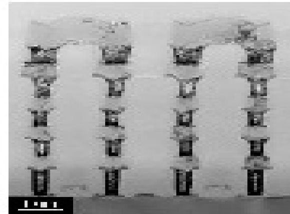
Amorphous materials

- Diffused ring pattern
- Reflecting the short range ordered structure
- Often seen at contamination layer or on carbon support film

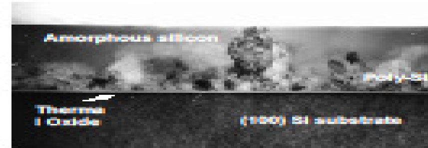


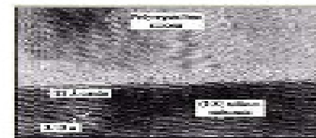

Diffraction mode
Image mode
Specimen
Remove aperture
Fixed
Objective lens
Objective aperture (back focal plane)
SAD aperture
Intermediate image 1
Remove aperture
Change strength
Intermediate lens
Secondary intermediate image
Fixed strength
Projector lens
Diffraction pattern
Final image
Screen


Major Factors affecting TEM Image Contrast
Thickness
Scattering
Atomic number
Scattering
Orientation
Diffraction
Defects
Scattering & diffraction

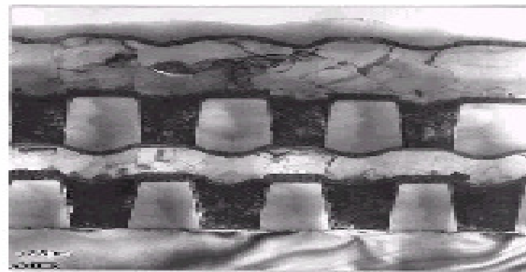


IC connectors in (five) stages. Pillars made of tungsten (hollow, dark) are connected by pieces of Al (lighter). Thin layers of TiN prevent the tungsten and Al from moving around.



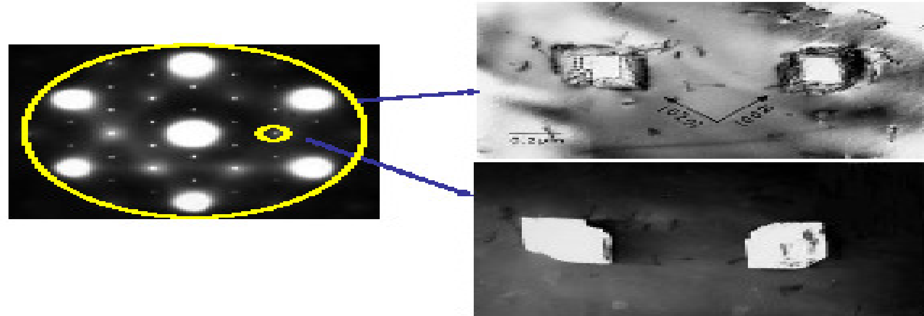


IC Cross section (CTEM BF)

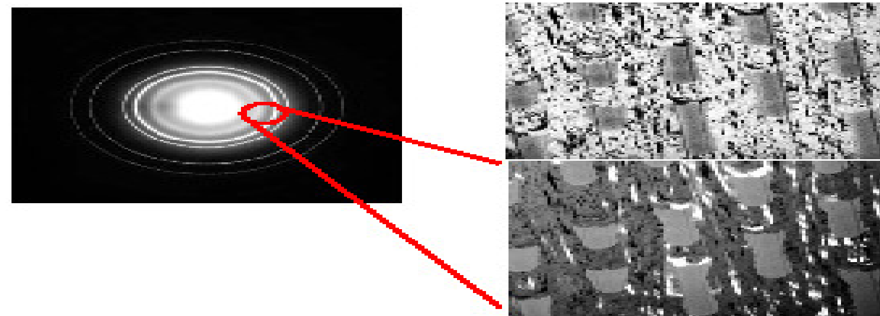


Images Courtesy of [illegible]

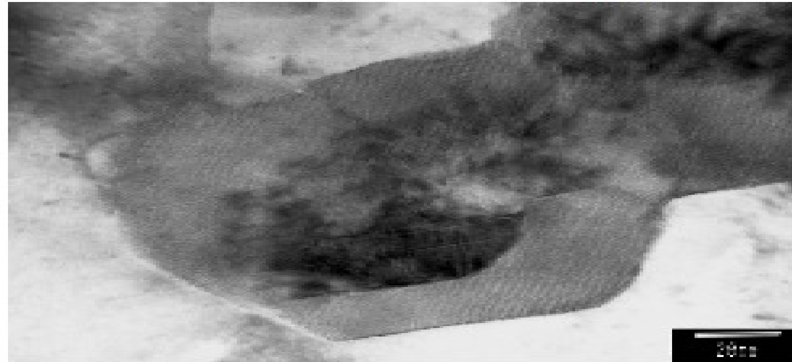
BF vs. CDF (1)



BF vs. CDF (2)

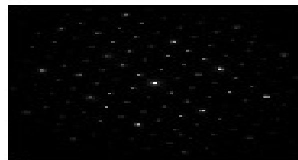


Precipitates in metal Alloys, I

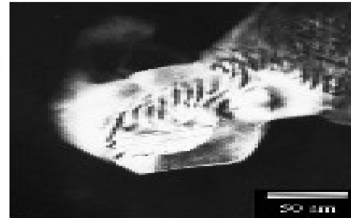


HRTEM of $Cr_{23}C_6$ in 403 Martentistic Stainless Steel

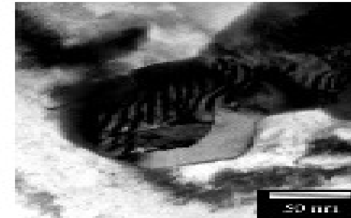
Precipitates in metal Alloys, I (cont.)



SAD



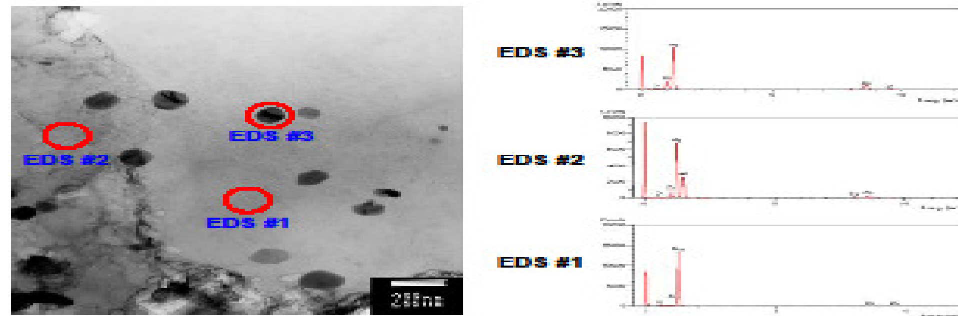
DF



BF

$Cr_{23}C_6$ in 403 Martentistic Stainless Steel

Precipitates in metal Alloys, II



MgZn in Li-Zn-Al-Mg Alloy

Precipitates in metal Alloys, II (cont.)

SAD DF BF

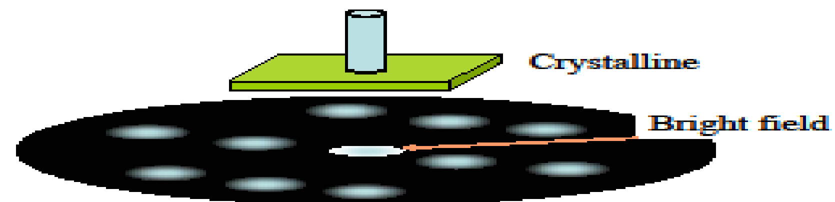
MgZn in Li-Zn-Al-Mg Alloy

What is HREM?

- It is NOT defined by its direct resolution (1nm or 0.3nm?)
- It is NOT defined by directly seeing atomic structure (in most cases it does not directly show crystal structure!)
- It displays many-beam (2D) interference fringes
- It is phase contrast image

Many-beam

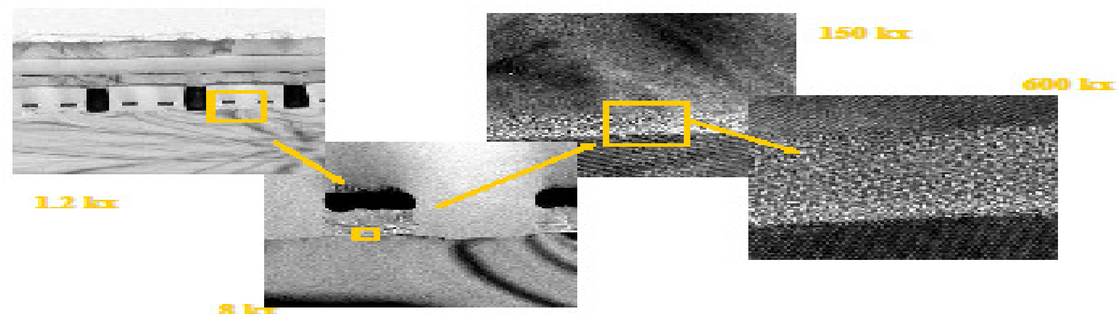
- Referred to the scattering effect
- Comparing to diffraction contrast, 'one-beam' technique

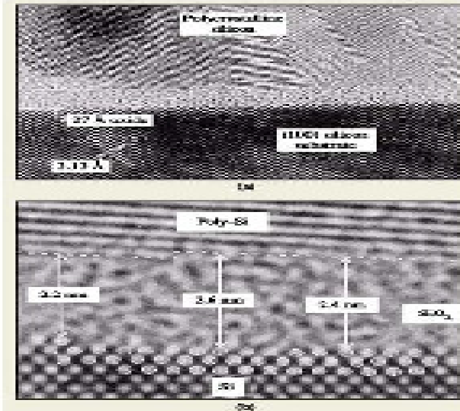


HREM image formation

- Scattering is a strong interaction
  - excellent statistics and useful signal
  - no simple relationship between an image and the specimen structure
- Imaging system is imperfect:
  - Generally no direct correspondence between image & structure
- Image interpretation is absolutely needed

CTEM BF and HRTEM

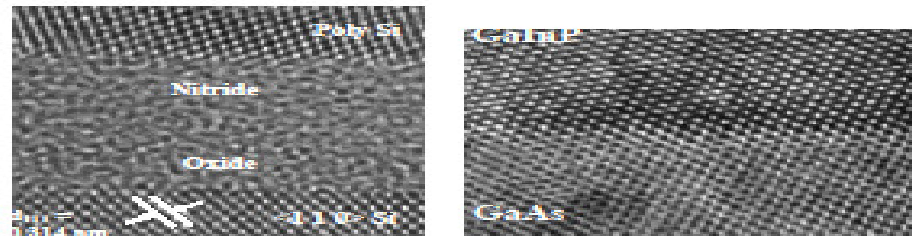




HRTEM for oxide thickness Measurement in MOS structure

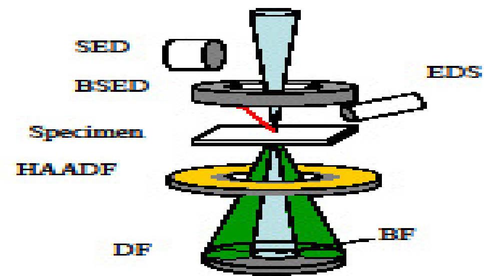
Cross-sectional high-resolution transmission electron microscope (HRTEM) images for MOS structure with (a)~2.7 nm and (b) ~2.4 nm image. The poly-Si grains are easily noticeable in (a); the Si/SiO_2 and poly-Si/SiO_2 interface are shown in (b). On a local, atomic scale, thickness variation of ~2-3 Å are found which are a direct result of atomic silicon steps at both interfaces.

HREM Image — Interface

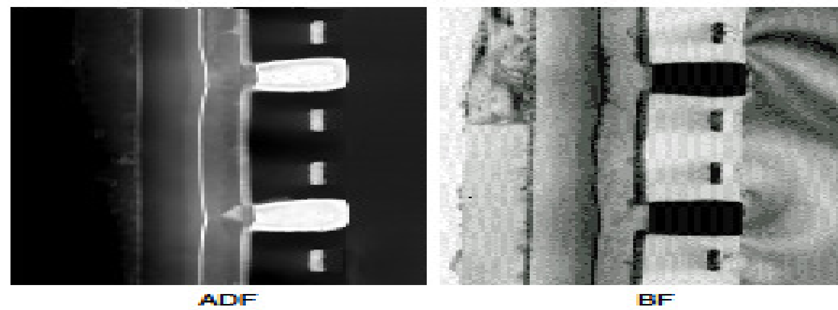


Fundamentals of STEM

- More detectors than a SEM below the specimen, which collect beam transmitted, or diffracted, from the specimen
- The beam intensity variation contains the useful information about the location where beam is currently situated

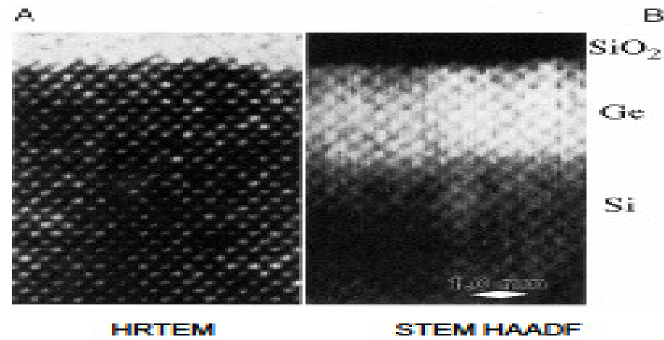


STEM BF and ADF images from a semiconductor device



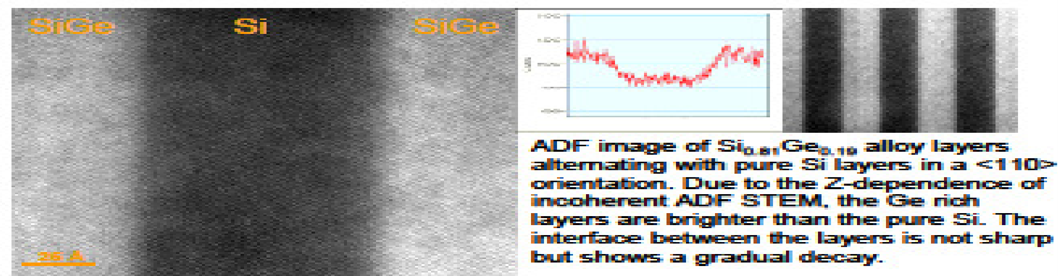
ADF BF

HREM vs. STEM HAADF Image — Interface



HRTEM STEM HAADF

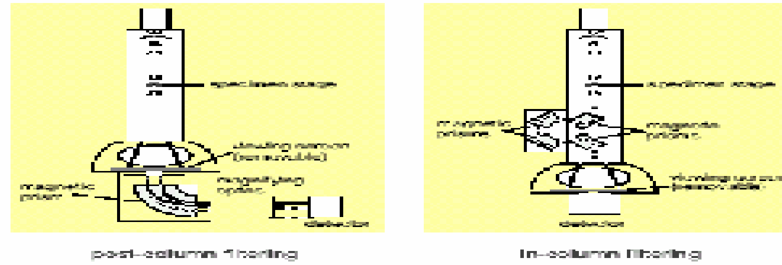
HAADF image of SiGe alloy layers



ADF image of $Si_{0.81}Ge_{0.19}$ alloy layers alternating with pure Si layers in a <110> orientation. Due to the Z-dependence of incoherent ADF STEM, the Ge rich layers are brighter than the pure Si. The interface between the layers is not sharp but shows a gradual decay.

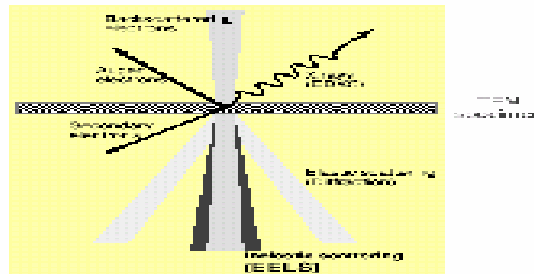
EELS configurations in TEM

Experimental setups for measuring EELS data

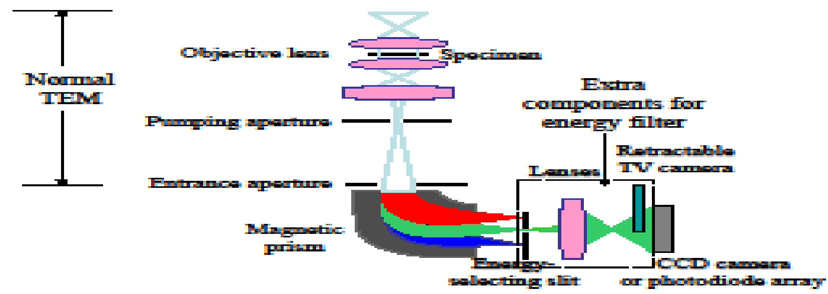


Signals for EELS

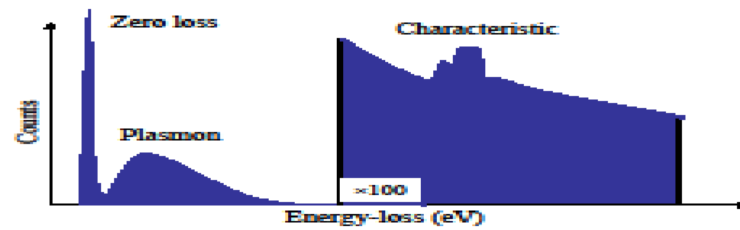
TEM beam-specimen interactions and signals



Post-column EELS

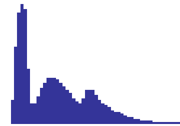


A typical EELS spectrum



Plasmon peak

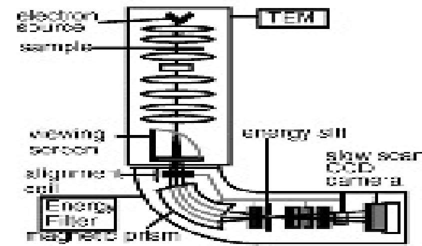
- Caused by the collective response to the incident beam by all the valence electrons
- If the sample is thicker, the plasmon peak is also higher and the second peak may appear
- The ratio of plasmon peak intensity to zero-loss peak intensity may estimate the sample thickness



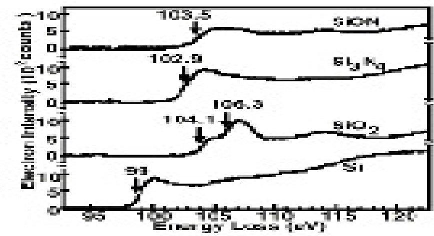
EELS vs. EDS

- More efficient signal collection
  - the first order phenomenon
  - most of the transmitted electrons enter the prism, comparing to 1% X-rays being detected
- Better signal to noise ratio
- Spectrum is electronic structure sensitive, e.g. O peaks in MnO and NiO are different in shape
- Slightly better spatial resolution
- Very high background and worse peak to background ratio, leading to the large error in quantification
- Complex peak structure makes identification difficult, it is worst when there is peak overlap
- Thin sample needed
- Operation and interpretation are more difficult

EELS for light elements



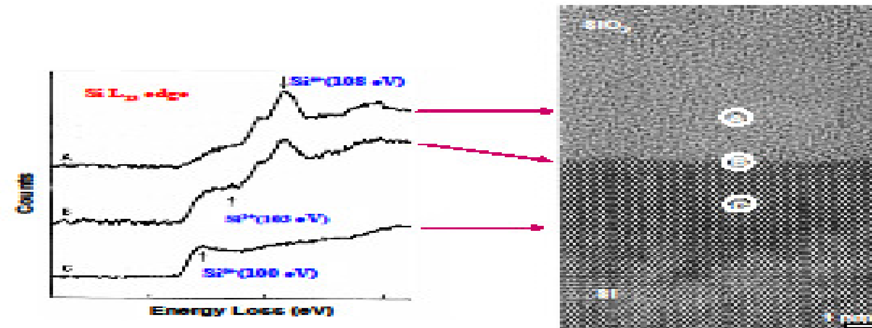
Schematic diagram of AEM-EELS



Chemical shift of core-loss edge energy in EELS spectra of some Si compounds

Y. Mitani et al., IEDM'98

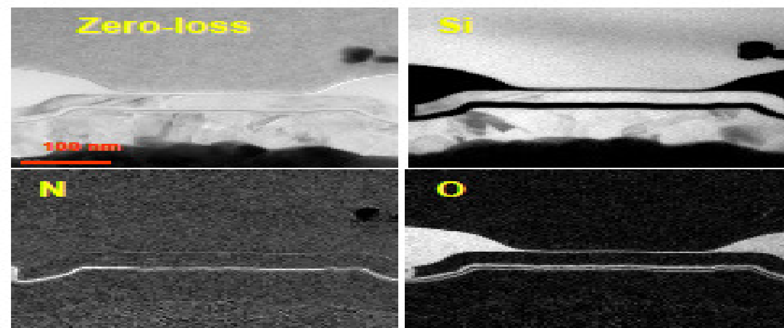
TEM and HREELS for the SiO2 / Si Interface

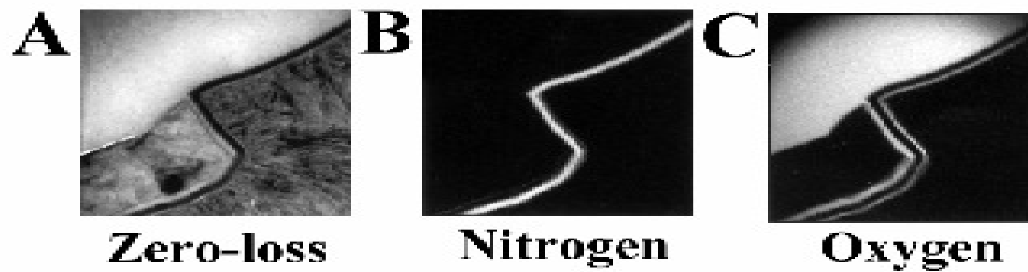


Energy filter

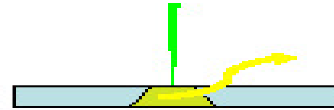
- An energy selective slit as small as 10eV is used
- Signal within the slit is collected and displayed, representing the element map
- For better mapping, background must be properly removed, normally by setting up windows before and after the slit

Energy filter


EFTEM mapping of a DRAM
Zero-loss
Si
N
O

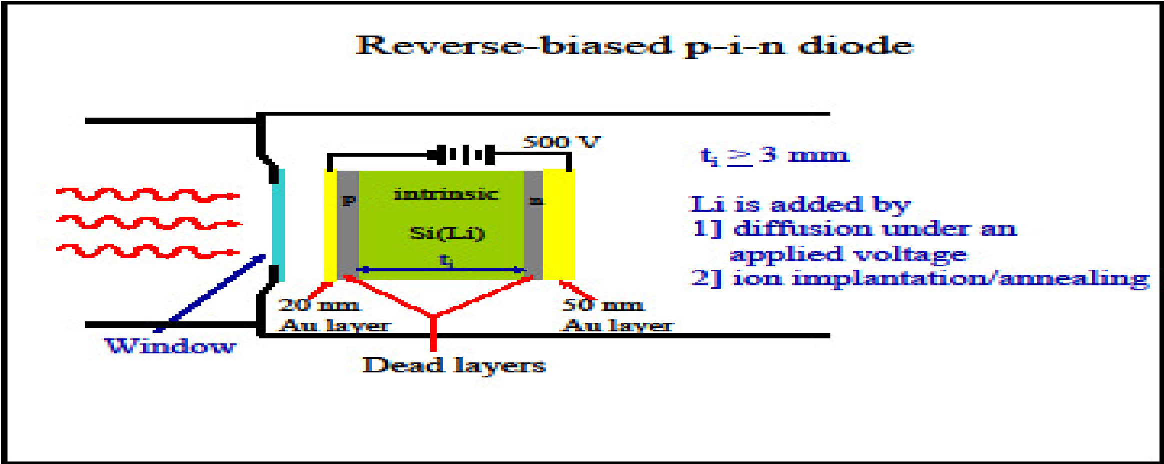

EFTEM mapping of the ONO layer in a DRAM
A
B
C
Zero-loss
Nitrogen
Oxygen

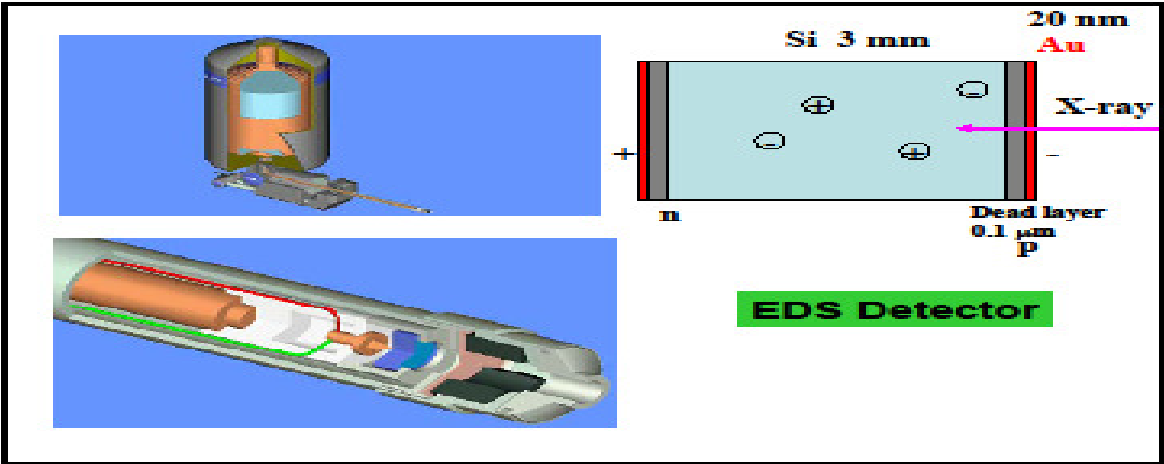
EDS



- Elements analysis
  - Qualitative, or quantitative [Z ≥ 5(B)]
- Elemental mapping
- Spatial resolution (volume of X-ray generation) ≥ probe size

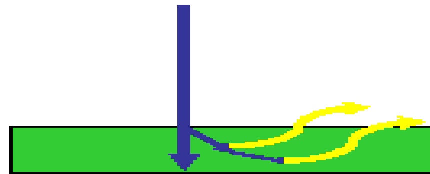
EDS system on TEM


Reverse-biased p-i-n diode
500 V
$t_i \geq 3$ mm
p
intrinsic
Si(Li)
n
t_i
Li is added by
1] diffusion under an applied voltage
2] ion implantation/annealing
20 nm
Au layer
50 nm
Au layer
Window
Dead layers


Si 3 mm
20 nm
Au
X-ray
+
-
n
Dead layer
0.1 μm
p
EDS Detector

Spatial Resolution

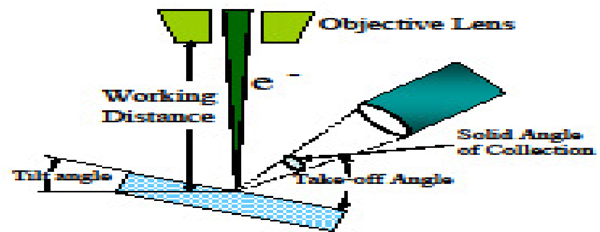
- Beam broadening size $b_{TEM} < b_{SEM}$
- Beam broadening size $b_{EELS} < b_{EDS}$



Factors on Spatial Resolution

- Probe size
- Interaction volume (SEM)
- Specimen thickness (TEM)
- Specimen drift
- Contamination

Parameters of EDS Collection

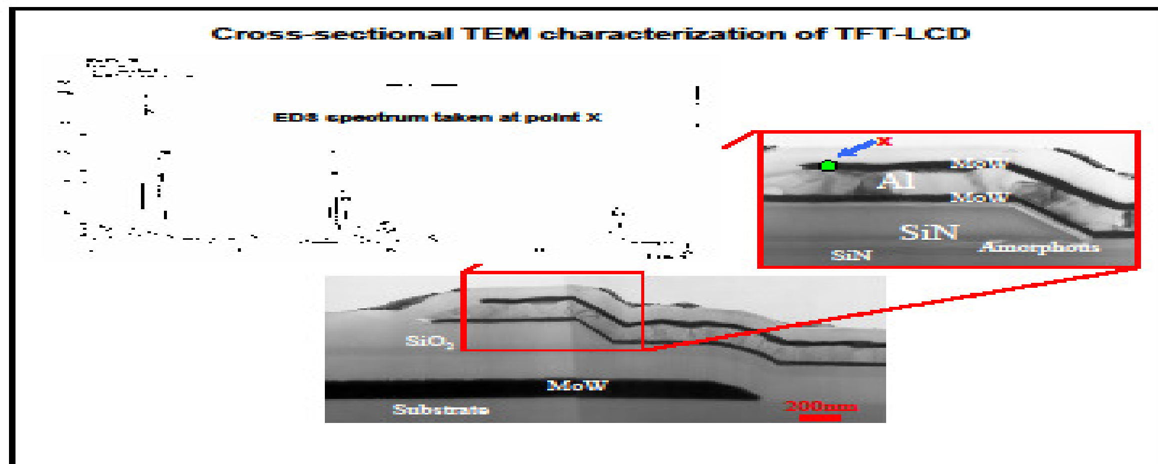


Contamination

Strengths and Weaknesses of EDS

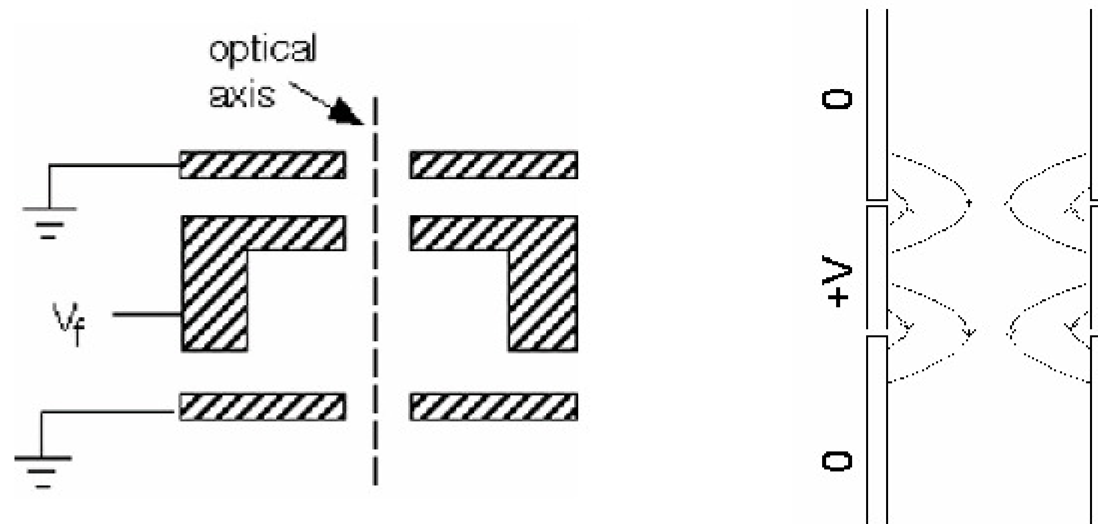
- **Strengths**
  - Quick, 'first look' analysis
  - Versatile & inexpensive
  - Quantitative for some samples (flat, polished, homogeneous)
- **Weaknesses**
  - Quantification
  - Size restrictions
  - May spoil subsequent analysis

Cross-sectional TEM characterization of TFT-LCD



Ion and Electron Optics

- We need something that changes the direction of electrons or ions in a beam, depending on initial direction and radial location within the beam



An electrostatic *lens*

Ion and Electron Optics

- Magnetic Lens

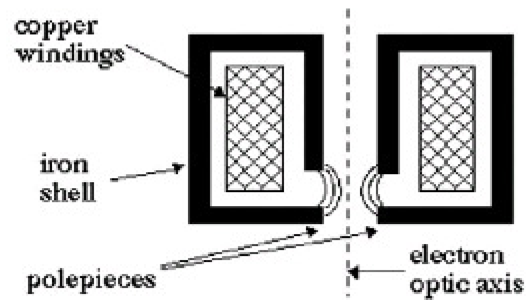
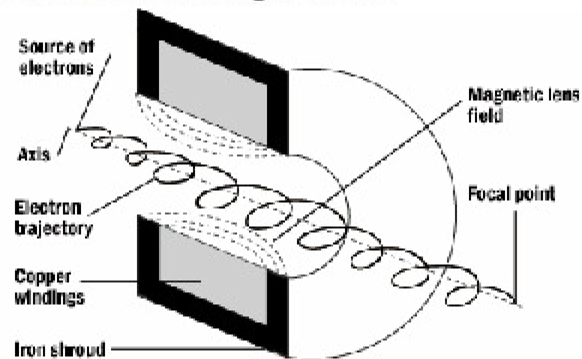
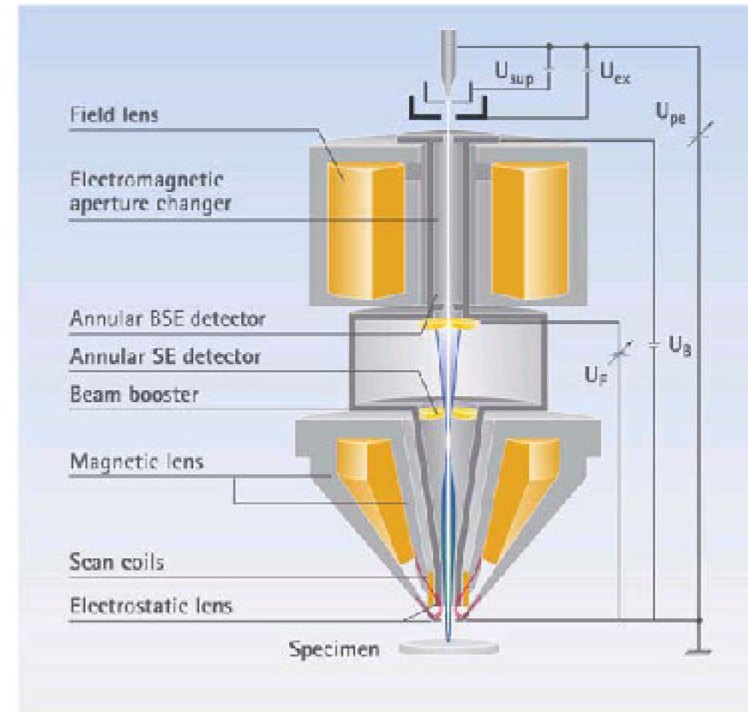


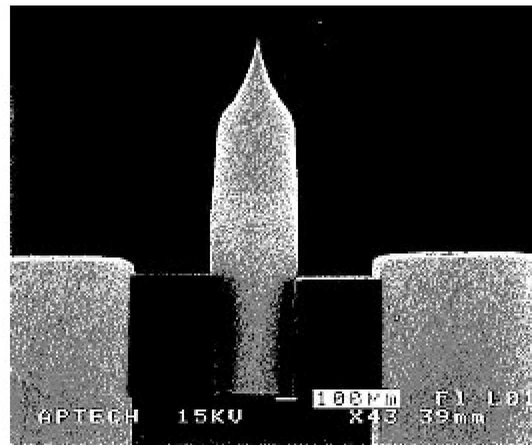
FIGURE 2.6. Cross-section through a magnetic lens with lines showing the magnetic field distribution.

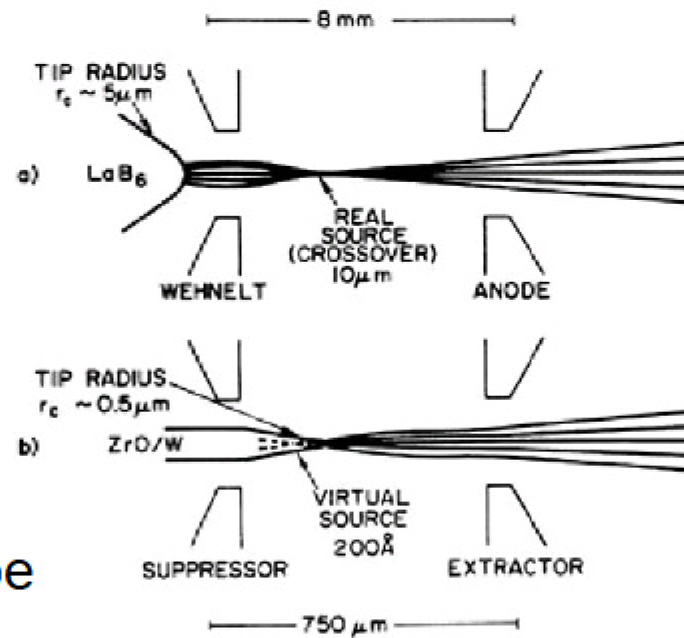
Cylindrically symmetric magnetic Field with radial gradients





Sources

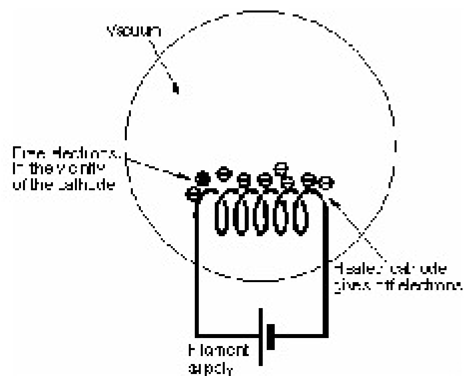


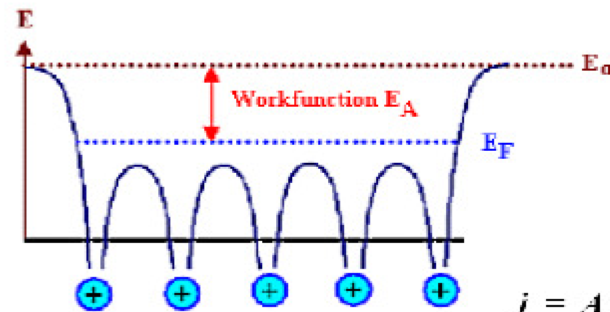


Electron emission can be achieved by different physical mechanisms

Emission

- Thermal emission



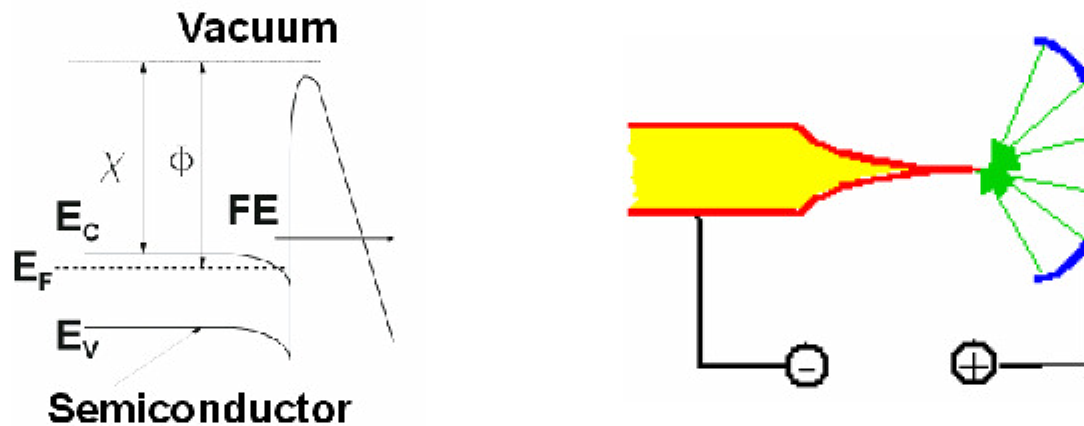


$$j = A \cdot T^2 \cdot \exp - \frac{E_A}{kT}$$

Material	Fe	Ni	Pt	Ta	W	Cs	LaB_6
A [$Acm^{-2}K^{-2}$]	26	30	32	55	60	162	25
E_A [eV]	4,5 - 4,8	5,15 - 5,35	5,65	4,15 - 4,8	4,2	1,8 - 2,14	2,6
T_m [^oC]	1 535	1 452	1 755	2 850	3 410	28,4	2 210

Emission

- Field emission



Field emission starts for $E > 10^7$ V/cm
High current density: $J(E) = A \cdot E^2 \varphi \exp(-B \varphi^{1.5} / E)$

Strong nonlinear current-voltage characteristic
Very short switching time ($t < ns$)

Small spot size due to field enhancement at the tip apex

Ion and Electron Optics

- Electron beam sources

TABLE 2.1 Properties of the electron sources commonly used in electron beam lithography tools.

source type	brightness ($A/cm^2/sr$)	source size	energy spread (eV)	vacuum requirement (Torr)
tungsten thermionic	$\sim 10^5$	25 um	2-3	10^{-6}
LaB_6	$\sim 10^6$	10 um	2-3	10^{-8}
thermal (Schottky) field emitter	$\sim 10^8$	20 nm	0.9	10^{-9}
cold field emitter	$\sim 10^9$	5 nm	0.22	10^{-10}

Source Size and Spot diameter

- The source size can be large (micrometers) and, if so, must be *DEMAGNIFIED* to achieve small (nanometer) spot at the sample plane

Source Stability

- E-beam current must be stable and low noise for clear imaging and stable electron beam manipulation processes

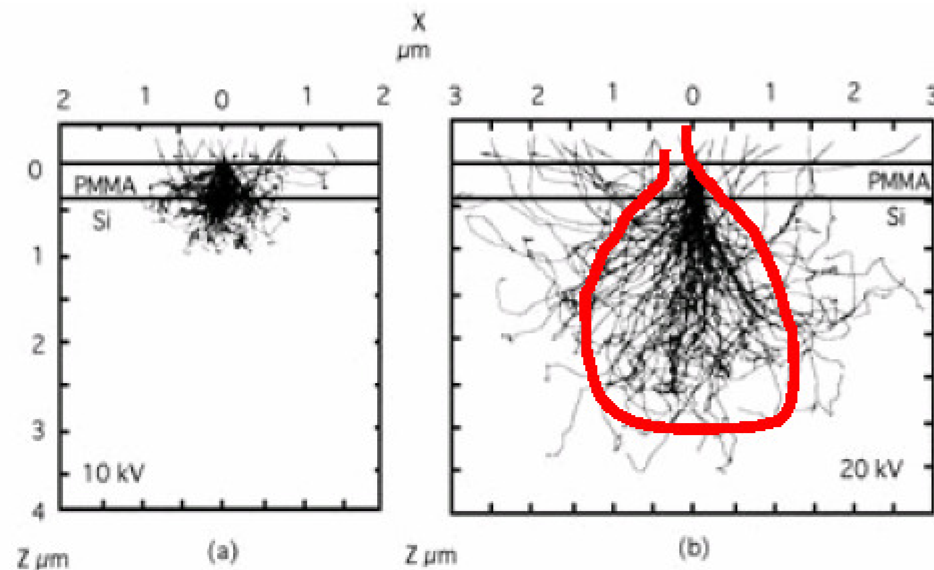
Monochromatic beam is also important

Scanning Electron Microscope

- Sequential imaging similar to the optical scanning confocal microscope
- Can be used in reflection or transmission modes (STEM)

Electron Beam and Sample Interaction

- Depends on energy of beam, material of the sample. The beam penetrates the sample
- Beam Spot size isn't everything



Electron microscopy and microanalysis: aims and means

• Microscopies: morphologies in small scales (micrometer or nanometer)
Optical microscopy, Electron microscopy, Ion microscopy, Scanning probe microscopy….., offer images only.
• Microanalyses: composition and/or structures in small scales (micrometer or nanometer)
Energy Dispersive Spectroscopy, Wave-length Dispersive Spectroscopy, Electron Energy Loss Spectroscopy, Auger Electron Spectroscopy, Convergent Beam Electron Diffraction,
Select Area Diffraction….., offer spectra and/or diffraction pattern

Why electrons?

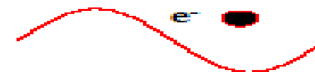
- Wave Behaviour
- – images and diffraction patterns
- – wavelength can be tuned by energies
- Charged Particle Behaviour
- – strong electron-specimen interactions
- – chemical analysis is possible

Electron microscopy and microanalysis: aims and means

- Microscopies: morphologies in small scales (micrometer or nanometer)
  Optical microscopy, Electron microscopy, Ion microscopy, Scanning probe microscopy….., offer images only.

- Microanalyses: composition and/or structures in small scales (micrometer or nanometer)
  Energy Dispersive Spectroscopy, Wave-length Dispersive Spectroscopy, Electron Energy Loss Spectroscopy, Auger Electron Spectroscopy, Convergent Beam Electron Diffraction, Select Area Diffraction….., offer spectra and/or diffraction patterns

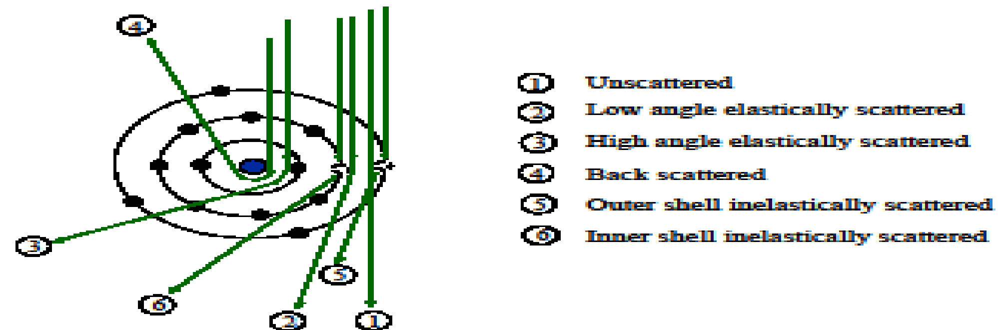
Why electrons?



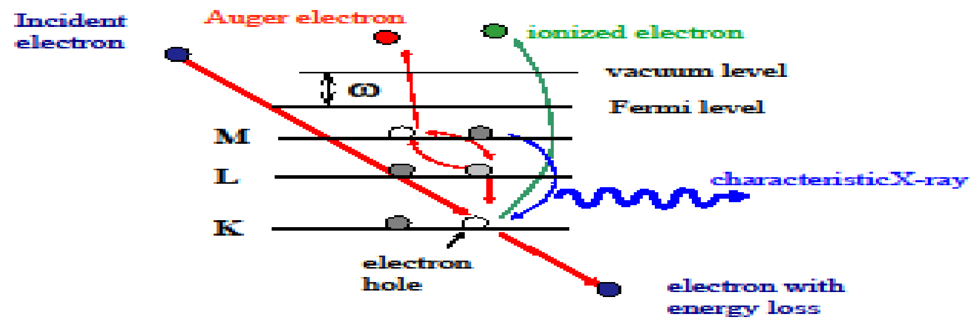
- Wave Behaviours
  - images and diffraction patterns
  - wavelength can be tuned by energies
- Charged Particle Behaviours
  - strong electron-specimen interactions
  - chemical analysis is possible

Interaction of high energy (~kV) electrons with (solid) materials-I

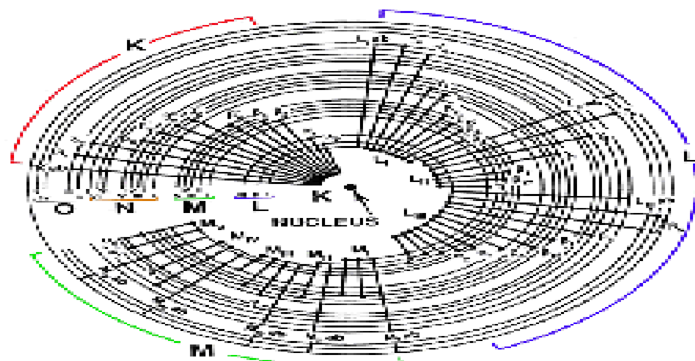
Interaction with an Atom



Interaction of high energy (~kV) electrons with (solid) materials-I, cont.



Interaction of high energy (~kV) electrons with (solid) materials-I, cont.



K lines

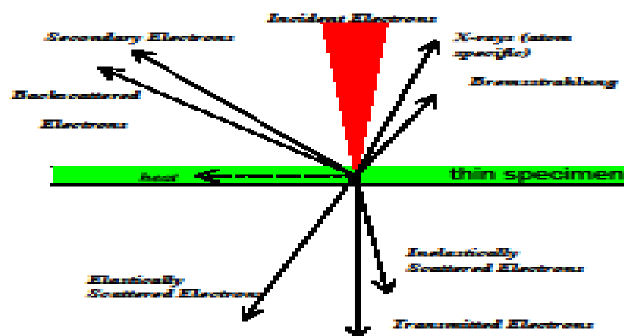
Kα, L → K
Kβ, M → K

L lines

Lα, M→L,
Lβ, N→L,
Lγ, O→L

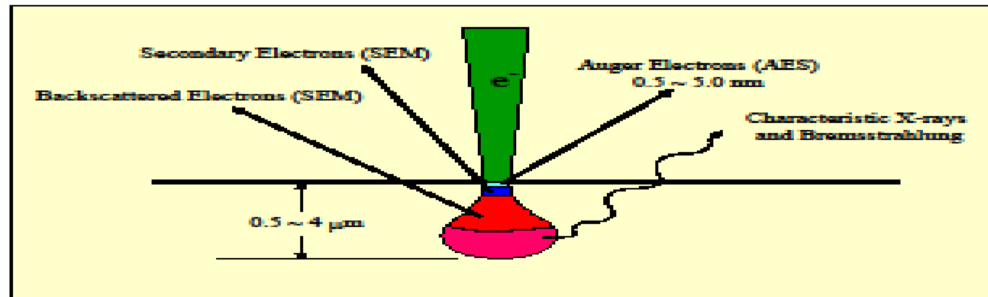
Interaction of high energy (~kV) electrons with (solid) materials-II

Interaction with a thin specimen (TEM & STEM)



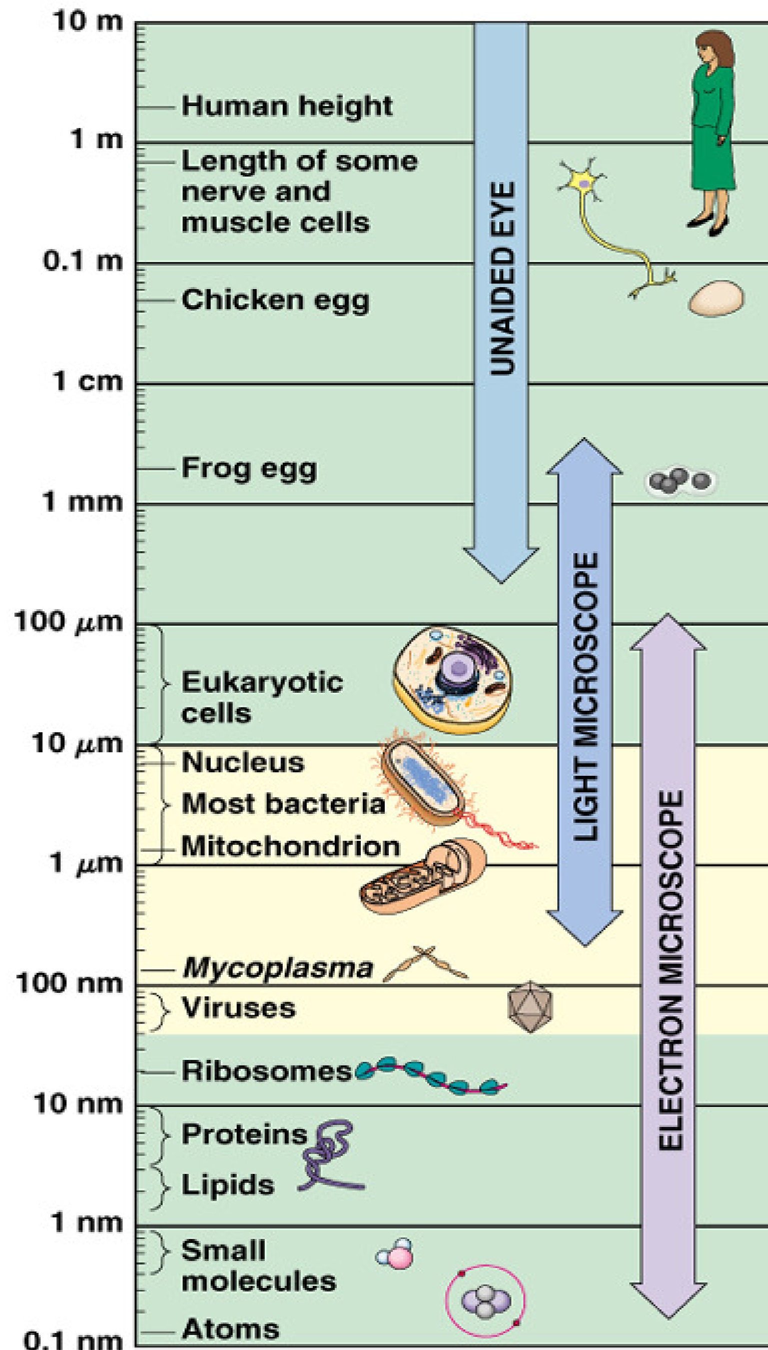
Interaction of high energy (~kV) electrons with (solid) materials-III

Interaction with a thick specimen (SEM)



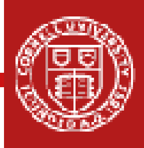
Basic electron optics

- Electrons and ions are charged particles, they can be accelerated in a E field.
- The trajectory of an accelerated charged particle can be deflected by E and/or B field.
- According to *de Broglie*, the accelerated (high-energy) particles also behave like waves.



©Addison Wesley Longman, Inc.

Biological and Electronic Component Dimensions



David Muller 2008

Electron microscopes are operated in vacuum because the mean free path of electrons is air is short – this mean biological samples should not degas – they can either be dehydrated or frozen – pathology, not *in-vivo.*

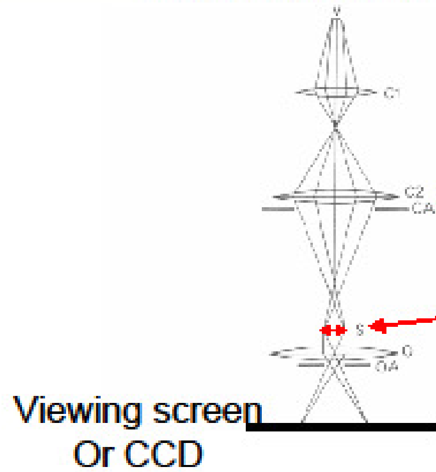
- Electron microscopes have higher resolution than optical microscopes – atomic resolution is possible.
- Chemical imaging and spectroscopy – mapping π and σ bonds at 1nm resolution can be done.
- Radiation damage is severe and limits the image quality and resolution (not as bad as x-rays or neutrons though! – see R. Henderson, *Quarterly Reviews of Biophysics* **28** (1995) 171-193.)

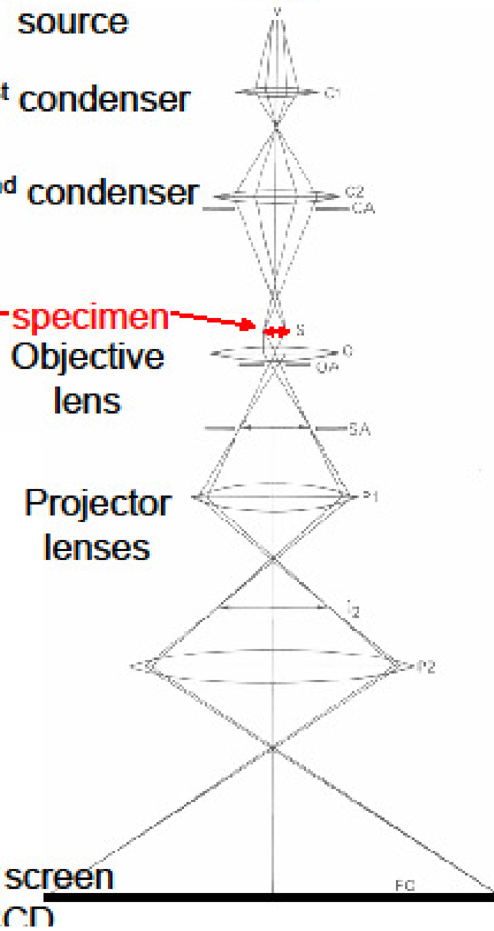
Comparison of Optical and Electron Microscopes

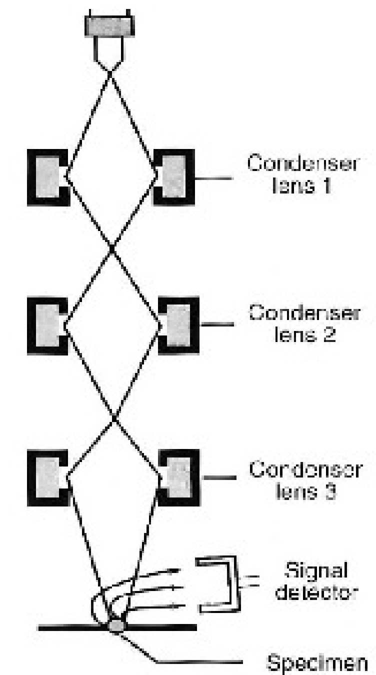
Light Microscope

TEM

SEM or STEM







CA condenser aperture

OA objective aperture

SA selected area aperture

Image formed by scanning a small spot

David Muller 2008

Where did Electron Microscopes Come From?

Electron Microscopes were developed due to the limitations of Light Microscopes which are limited by the physics of light to 500x or 1000x magnification and a resolution of 0.2 micrometers. In the early 1930's this theoretical limit had been reached and there was a scientific desire to see the fine details of the interior structures of organic cells (nucleus, mitochondria...etc.). This required 10,000x plus magnification which was just not possible using Light Microscopes.

The Transmission Electron Microscope (TEM) was the first type of Electron Microscope to be developed and is patterned exactly on the Light Transmission Microscope except that a focused beam of electrons is used instead of light to "see through" the specimen. It was developed by Max Knoll and Ernst Ruska in Germany in 1931.

The first Scanning Electron Microscope (SEM) debuted in 1942 with the first commercial instruments around 1965. Its late development was due to the electronics involved in "scanning" the beam of electrons across the sample

How do Electron Microscopes Work?

Electron Microscopes(EMs) function exactly as their optical counterparts except that they use a focused beam of electrons instead of light to "image" the specimen and gain information as to its structure and composition.

The basic steps involved in all EMs:

A stream of electrons is formed (by the Electron source) and accelerated toward the specimen using a positive electrical potential

This stream is confined and focused using metal apertures and magnetic lenses into a thin, focused, moochromatic beam.

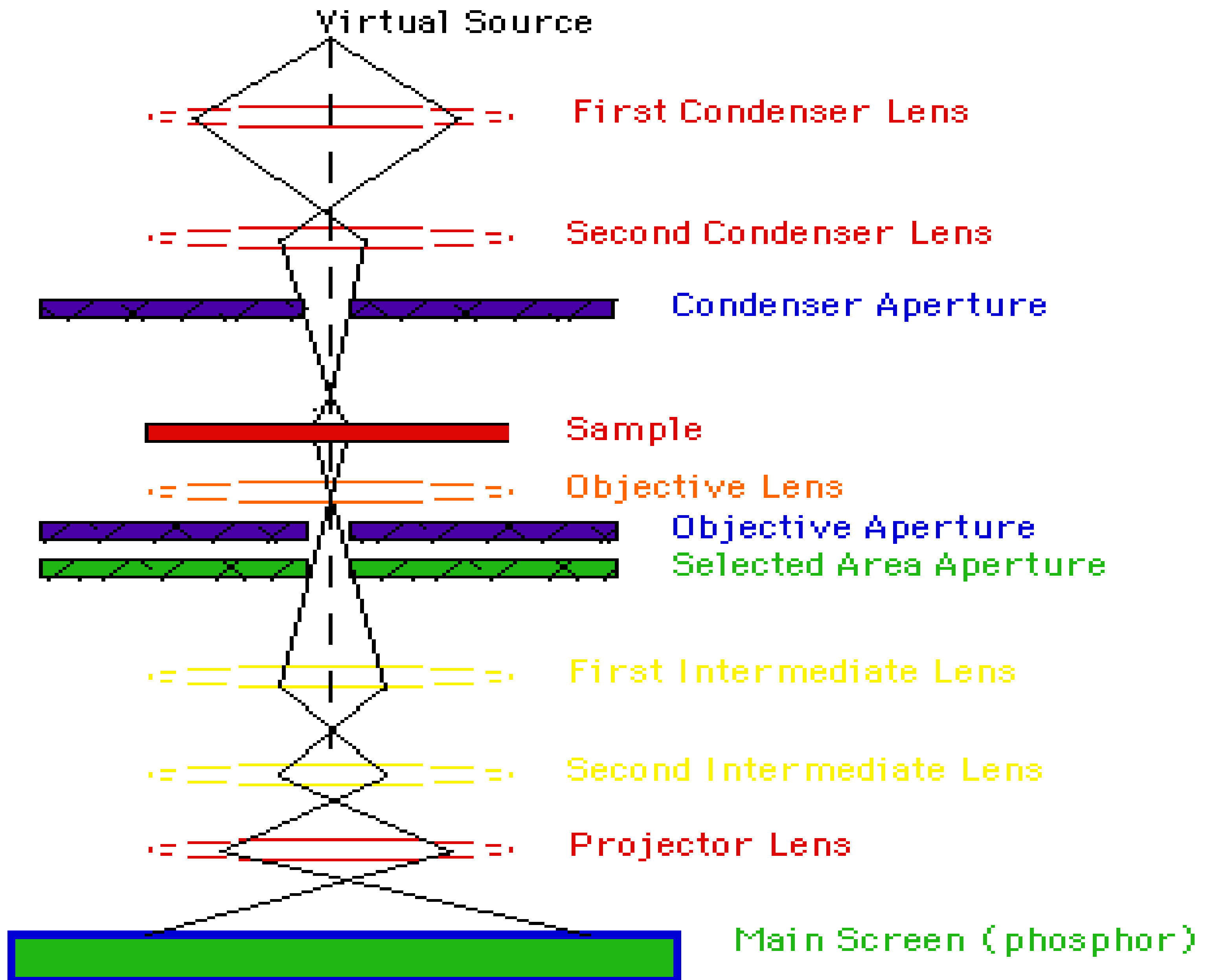
This beam is focused onto the sample using a magnetic lens

interactions occur inside the irradiated sample, affecting the electron beam

These interactions and effects are detected and transformed into an image

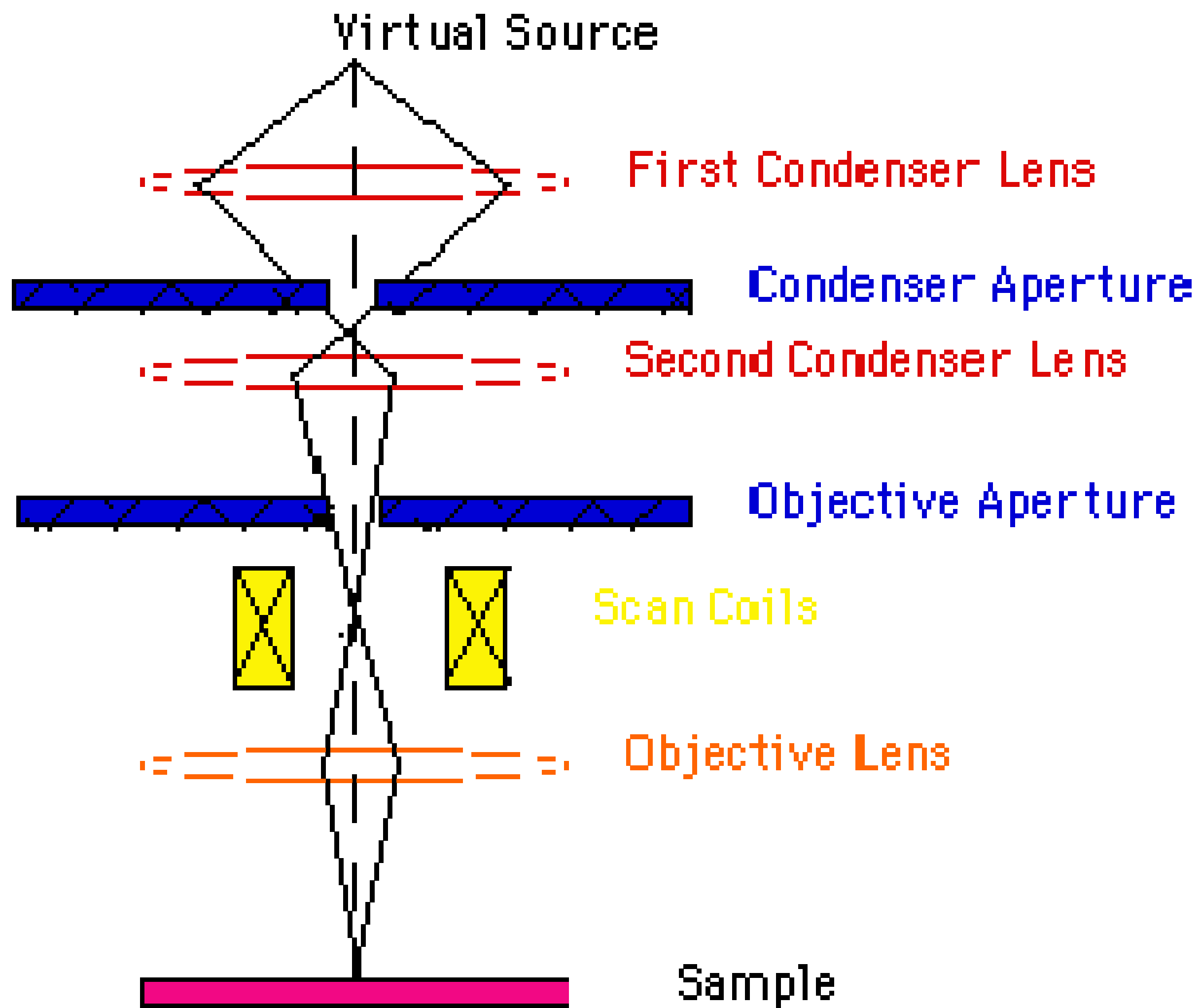
The above steps are carried out in all EMs regardless of type

1.The "Virtual Source" at the top represents the electron gun, producing a stream of monochromatic electrons.
2.This stream is focused to a small, thin, coherent beam by the use of condenser lenses 1 and 2. The first lens(usually controlled by the "spot size knob") largely determines the "spot size"; the general size range of the final spot that strikes the sample. The second lens(usually controlled by the "intensity or brightness knob" actually changes the size of the spot on the sample; changing it from a wide dispersed spot to a pinpoint beam.
3.The beam is restricted by the condenser aperature (usually user selectable), knocking out high angle electrons (those far from the optic axis, the dotted line down the center)
4.The bean strikes the specimen and parts of it are transmitted.
5.This transmitted portion is focused by the objective lens into an image
6. Optional Objective and Selected Area metal aperatures can restrict the beam; the Objective aperture enhancing contrast by blocking out high-angle diffracted electrons, the Selected Area aperture enabling the user to examine the periodic diffraction of electrons by ordered arrangements of atoms in the sample
7.The image is passed down the column through the intermediate and projector lenses, being enlarged all the way
8.The image strikes the phosphor image screen and light is generated, allowing the user to see the image. The darker areas of the image represent those areas of the sample that fewer electrons were transmitted through (they are thicker or denser). The lighter areas of the image represent those areas of the sample that more electrons were transmitted through (they are thinner or less dense)



Virtual Source
First Condenser Lens
Second Condenser Lens
Condenser Aperture
Sample
Objective Lens
Objective Aperture
Selected Area Aperture
First Intermediate Lens
Second Intermediate Lens
Projector Lens
Main Screen (phosphor)

typical SEM :



1.The "Virtual Source" at the top represents the electron gun, producing a stream of monochromatic electrons.

2.The stream is condensed by the first condenser lens (usually controlled by the "<u>coarse</u> probe current knob"). This lens is used to both form the beam and limit the amount of current in the beam. It works in conjunction with the condenser aperture to eliminate the high-angle electrons from the beam

3.The beam is then constricted by the condenser aperture (usually not user selectable), eliminating some high-angle electrons

4.The second condenser lens forms the electrons into a thin, tight, coherent beam and is usually controlled by the "<u>fine</u> probe current knob"

5.A user selectable objective aperture further eliminates high-angle electrons from the beam

6.A set of coils then "scan" or "sweep" the beam in a grid fashion (like a television), dwelling on points for a period of time determined by the scan speed (usually in the microsecond range)

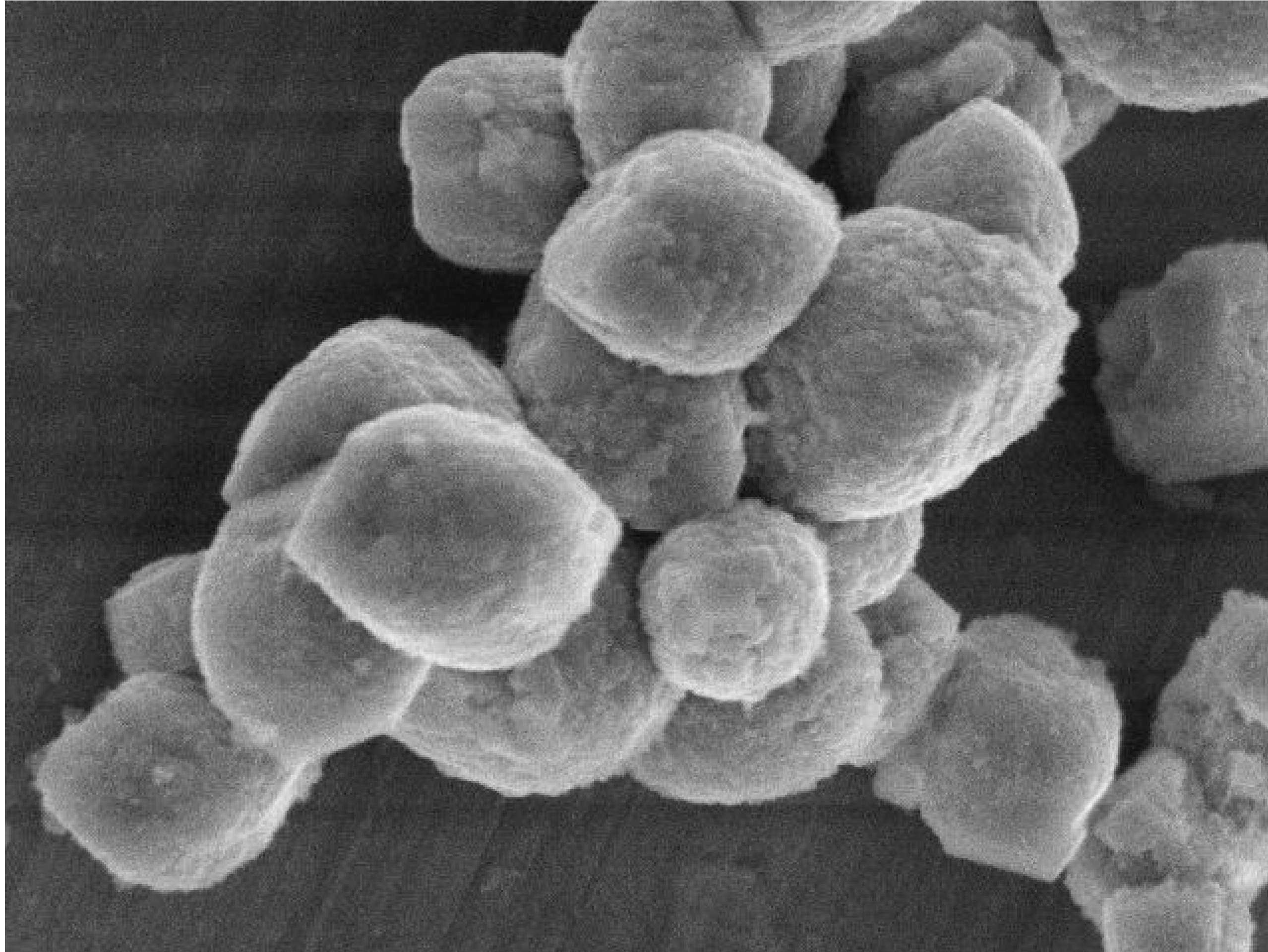
7.The final lens, the Objective, focuses the scanning beam onto the part of the specimen desired.

8. When the beam strikes the sample (and dwells for a few microseconds) interactions occur inside the sample and are detected with various instruments

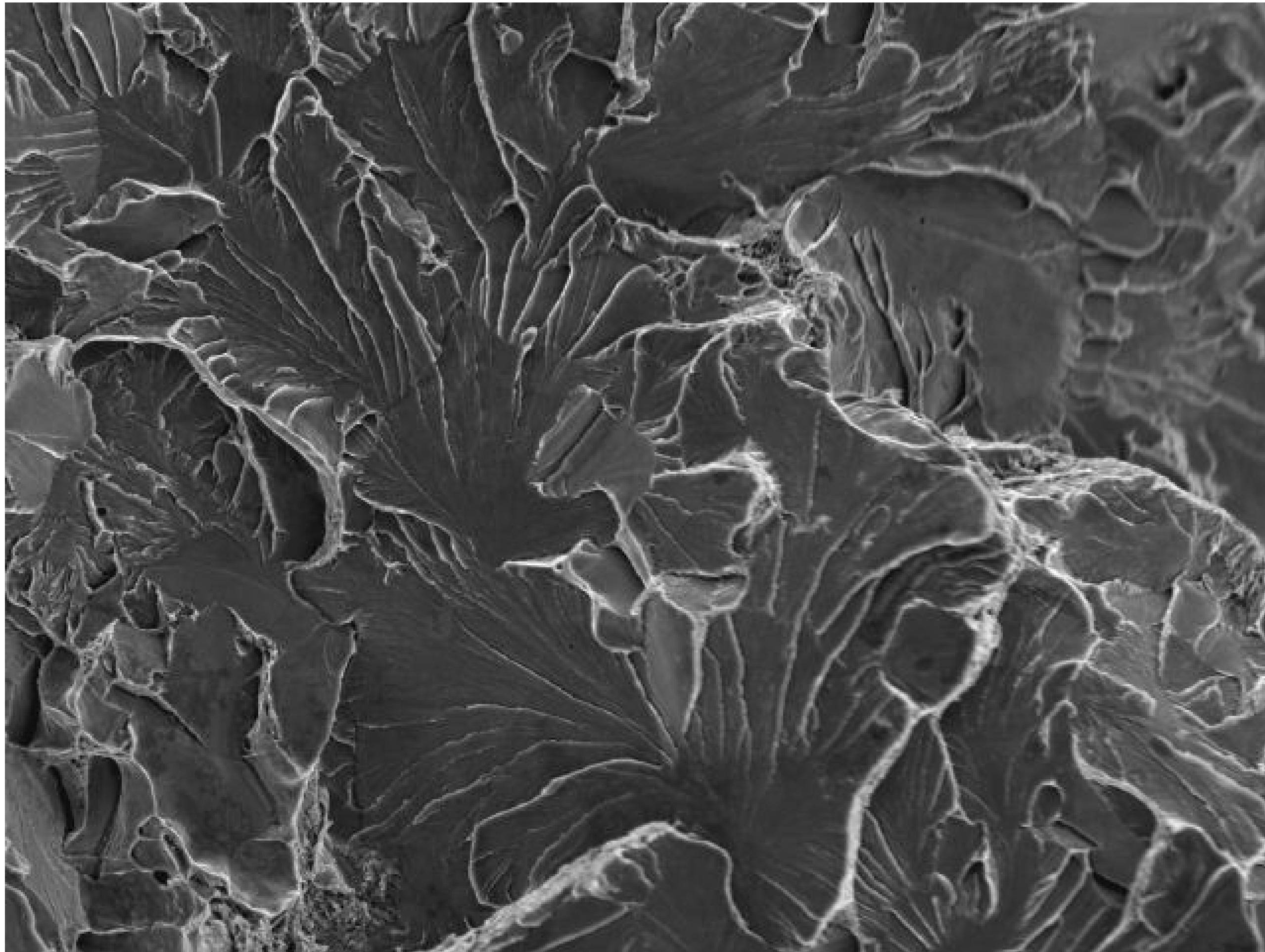
9.Before the beam moves to its next dwell point these instruments count the number of interactions and display a pixel on a CRT whose intensity is determined by this number (the more reactions the brighter the pixel).

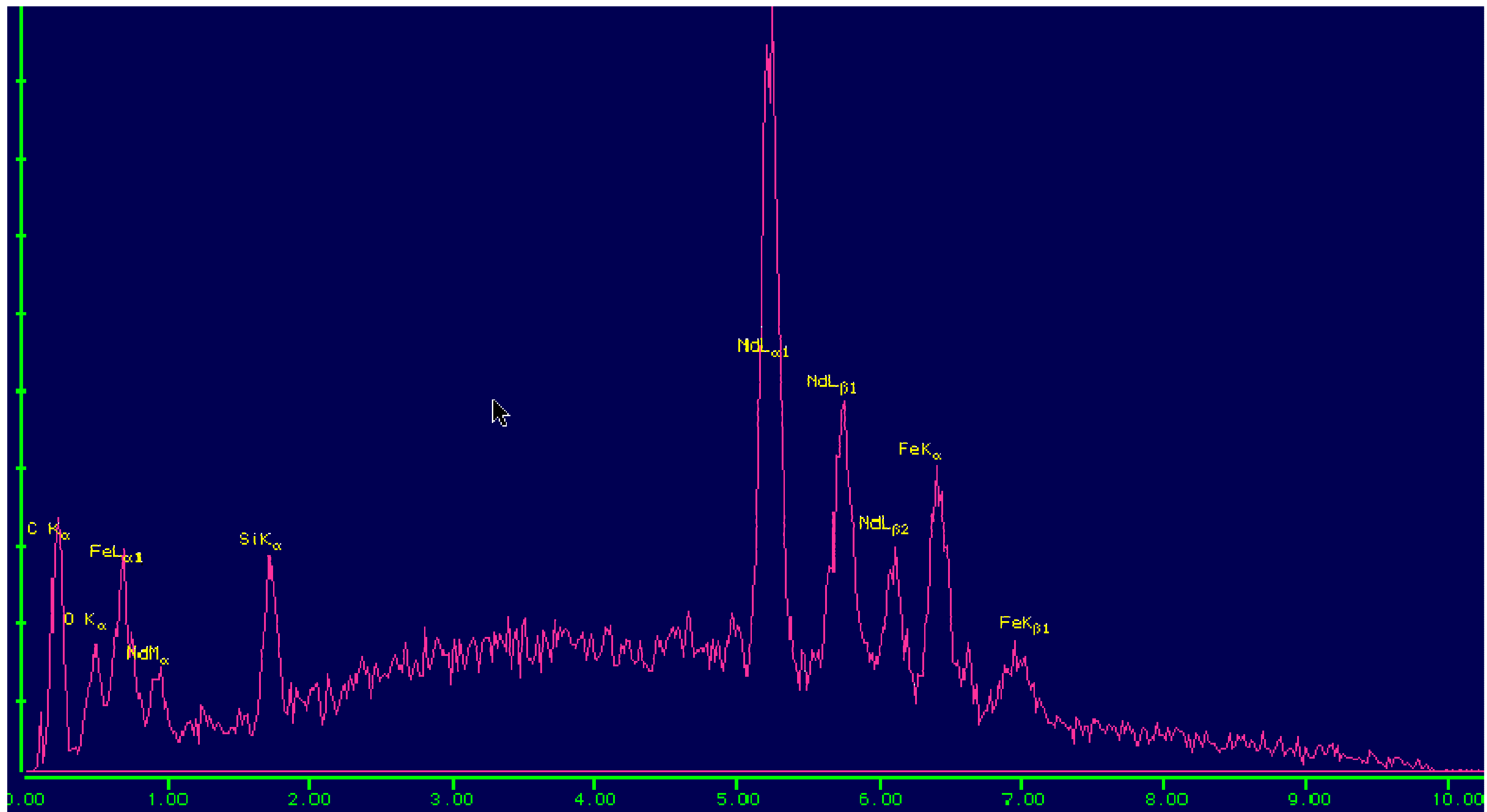
10. This process is repeated until the grid scan is finished and then repeated, the entire pattern can be scanned 30 times per second

Iron oxide



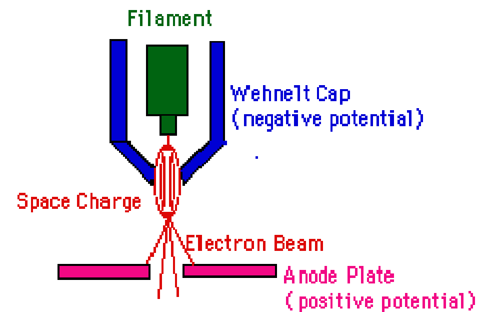
Brittle fractured steel





C Kα
O Kα
FeLα1
NdMα
SiKα
NdLα1
NdLβ1
NdLβ2
FeKα
FeKβ1
0.00
1.00
2.00
3.00
4.00
5.00
6.00
7.00
8.00
9.00
10.00

All Electron Microscopes utilize an electron source of some kind with the majority using a Themionic Gun as shown below:



A Thermionic Electron Gun functions in the following manner

1. An positive electrical potential is applied to the anode
2. The filament (cathode) is heated until a stream of electrons is produced
3. The electrons are then accelerated by the positive potential down the column
4. A negative electrical potential (~500 V) is applied to the Whenelt Cap
5. As the electrons move toward the anode any ones emitted from the filament's side are repelled by the Whenelt Cap toward the optic axis (horizontal center)
6. A collection of electrons occurs in the space between the filament tip and Whenelt Cap. This collection is called a space charge
7. Those electrons at the bottom of the space charge (nearest to the anode) can exit the gun area through the small (<1 mm) hole in the Whenelt Cap
8. These electrons then move down the column to be later used in imaging

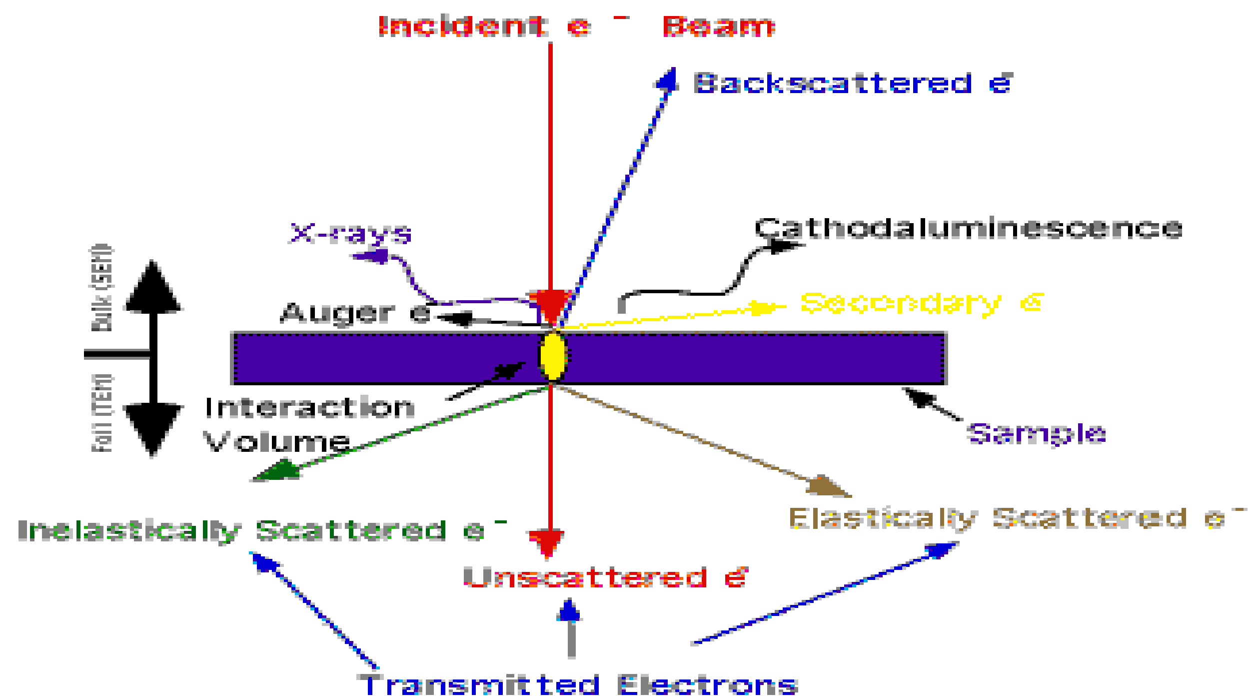
This process insures several things:

That the electrons later used for imaging will be emitted from a nearly perfect point source (the space charge)

The electrons later used for imaging will all have similar energies (monochromatic)

Only electrons nearly parallel to the optic axis will be allowed out of the gun area

The energetic electrons in the microscope strike the sample and various reactions can occur as shown below. The reactions noted on the top side of the diagram are utilized when examining thick or bulk specimens(SEM) while the reactions on the bottom side are those examined in thin or foil specimens (TEM). A diagram showing the generation depths of the interactions is also available



Bulk Specimen Interactions

Backscattered Electrons:

Formation

Caused by an incident electron colliding with an atom in the specimen which is nearly normal to the incident's path. The incident electron is then scattered "backward" 180 degrees.

Utilization

The production of backscattered electrons varies directly with the specimen's atomic number. This differing production rates causes higher atomic number elements to appear brighter than lower atomic number elements. This interaction is utilized to differentiate parts of the specimen that have different average atomic number. An example is shown in the SEM output section, specifically the mechanically alloyed specimen micrograph.

Secondary Electrons:

Source

Caused by an incident electron passing "near" an atom in the specimen, near enough to impart some of its energy to a lower energy electron (usually in the K-shell). This causes a slight energy loss and path change in the incident electron and the ionization of the electron in the specimen atom. This ionized electron then leaves the atom with a very small kinetic energy (5eV) and is then termed a "secondary electron". Each incident electron can produce several secondary electrons.

Utilization

Production of secondary electrons is very topography related. Due to their low energy, 5eV, only secondaries that are very near the surface (<10nm,) can exit the sample and be examined. Any changes in topography in the sample that are larger than this sampling depth will change the yield of secondaries due to collection efficiencies. Collection of these electrons is aided by using a "collector" in conjunction with the secondary electron detector. The collector is a grid or mesh with a +100V potential applied to it which is placed in front of the detector, attracting the negatively charged secondary electrons to it which then pass through the grid-holes and into the detector to be counted.

Auger Electrons

Source

Caused by the de-energization of the specimen atom after a secondary electron is produced. Since a lower (usually K-shell) electron was emitted from the atom during the secondary electron process an inner (lower energy) shell now has a vacancy. A higher energy electron from the same atom can "fall" to a lower energy, filling the vacancy. This creates and energy surplus in the atom which can be corrected by emitting an outer (lower energy) electron; an Auger Electron.

Utilization

Auger Electrons have a characteristic energy, unique to each element from which it was emitted from. These electrons are collected and sorted according to energy to give compositional information about the specimen. Since Auger Electrons have relatively low energy they are only emitted from the bulk specimen from a depth of <3).

X-rays

Source

Caused by the de-energization of the specimen atom after a secondary electron is produced. Since a lower (usually K-shell) electron was emitted from the atom during the secondary electron process an inner (lower energy) shell now has a vacancy. A higher energy electron can "fall" into the lower energy shell, filling the vacancy. As the electron "falls" it emits energy, usually X-rays to balance the total energy of the atom so it .

Utilization

X-rays or Light emitted from the atom will have a characteristic energy which is unique to the element from which it originated. These signals are collected and sorted according to energy to yield micrometer diameter) of bulk specimens limiting the point-to-point comparisons available

Thin Specimen Interactions

Unscattered Electrons

Source

Incident electrons which are transmitted through the thin specimen without any interaction occurring inside the specimen.

Utilization

The transmission of unscattered electrons is inversely proportional to the specimen thickness. Areas of the specimen that are thicker will have fewer transmitted unscattered electrons and so will appear darker, conversely the thinner areas will have more transmitted and thus will appear lighter.

Elasticity Scattered electrons

Source

Incident electrons that are scattered (deflected from their original path) by atoms in the specimen in an elastic fashion (no loss of energy). These scattered electrons are then transmitted through the remaining portions of the specimen.

Utilization

All electrons follow Bragg's Law and thus are scattered according to Wavelength=2*Space between the atoms in the specimen*sin(angle of scattering). All incident electrons have the same energy(thus wavelength) and enter the specimen normal to its surface. All incidents that are scattered by the same atomic spacing will be scattered by the same angle. These "similar angle" scattered electrons can be collated using magnetic lenses to form a pattern of spots; each spot corresponding to a specific atomic spacing (a plane). This pattern can then yield information about the orientation, atomic arrangements and phases present in the area being examined

Inelastically Scattered Electrons

Source

Incident electrons that interact with specimen atoms in a inelastic fashion, loosing energy during the interaction. These electrons are then transmitted trough the rest of the specimen

Utilization

Inelasticaly scattered electrons can be utilized two ways

Electron Energy Loss Spectroscopy: The inelastic loss of energy by the incident electrons is characteristic of the elements that were interacted with. These energies are unique to each bonding state of each element and thus can be used to extract both compositional and bonding (i.e. oxidation state) information on the specimen region being examined.

Kakuchi Bands: Bands of alternating light and dark lines that are formed by inelastic scattering interactions that are related to atomic spacings in the specimen. These bands can be either measured (their width is inversely proportional to atomic spacing) or "followed" like a roadmap to the "real" elasticity scattered electron pattern.

The volume inside the specimen in which interactions occur while being struck with an electron beam. This volume depends on the following factors:

•Atomic number of the material being examined; higher atomic number materials absorb or stop more electrons and so have a smaller interaction volume.

•Accelerating voltage being used; higher voltages penetrate farther into the sample and generate larger interaction volumes

•Angle of incidence for the electron beam; the greater the angle (further from normal) the smaller the volume

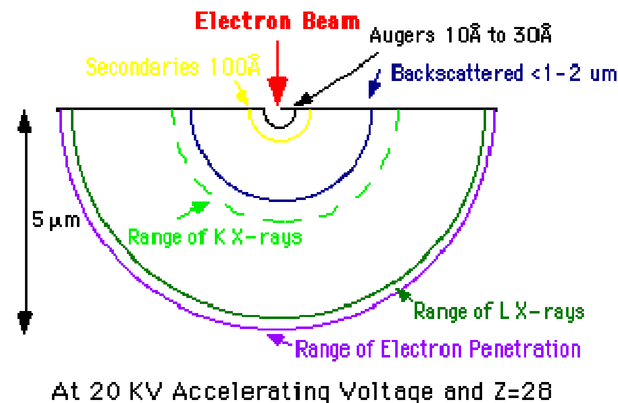
Below is an example of a typical Interaction Volume for

•Specimen is predominately Atomic number 28

•Accelerating Voltage is 20 kV

•0 degrees tilt, incident beam is normal to specimen surface

noting the approximate maximum sampling depths for the various interactions. See specimen interactions for details on specific interactions listed.



This technique is used in conjunction with SEM and is not a surface science technique. An electron beam strikes the surface of a conducting sample. The energy of the beam is typically in the range 10-20keV. This causes X-rays to be emitted from the point the material. The energy of the X-rays emitted depend on the material under examination. The X-rays are generated in a region about 2 microns in depth, and thus EDX is not a surface science technique. By moving the electron beam across the material an image of each element in the sample can be acquired in a manner similar to SAM. Due to the low X-ray intensity, images usually take a number of hours to acquire. Elements of low atomic number are difficult to detect by EDX. The SiLi detector (see below) is often protected by a Beryllium window. The absorbtion of the soft X-rays by the Be precludes the detection of elements below an atomic number of 11 (Na). In windowless systems, elements with as low atomic number as 4 (Be) have been detected, but the problems involved get progressively worse as the atomic number is reduced.

Summary

- The goal of this short course is to provide you with a better understanding of some common techniques or tools of electron microscopy & microanalysis for materials characterization.
- No single analytical technique can solve all of your problems. Each technique has its particular advantage.
- Good specimen will give excellent results.