Introduction to Electron Microscopy and Microanalysis

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Outlines

- **Electron microscopy and microanalysis: aims and means**
- **Interaction of high energy (~kV) electrons with (solid) materials**
- **Basic electron optics**
- **The instruments and techniques**
- **Summary**

Electron microscopy and microanalysis: aims and means

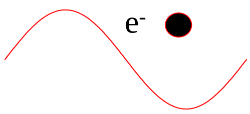
- Microscopies: morphologies in small scales (micrometer or nanometer)

  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

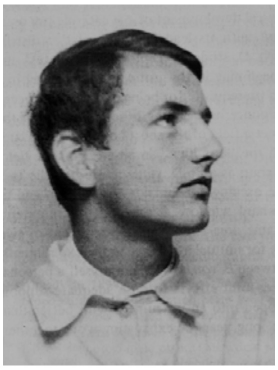
Why electrons?



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

Invention and Evolution of the Modern TEM

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

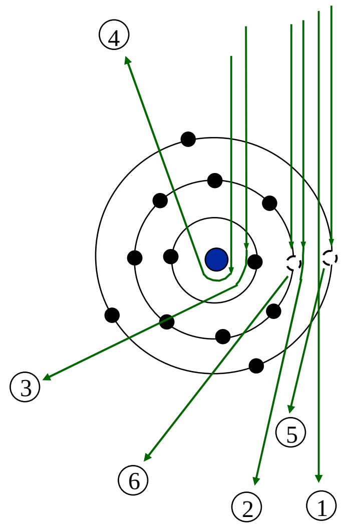


Ruska & Knoll

Fig. 6. The first electron microscope. The publication (48) contained only one cross-sectional drawing corresponding to Figure 5. This photograph, reproduced here for the first time, with M. Knoll and the author – was actually taken several years later, on 8 February 1944.

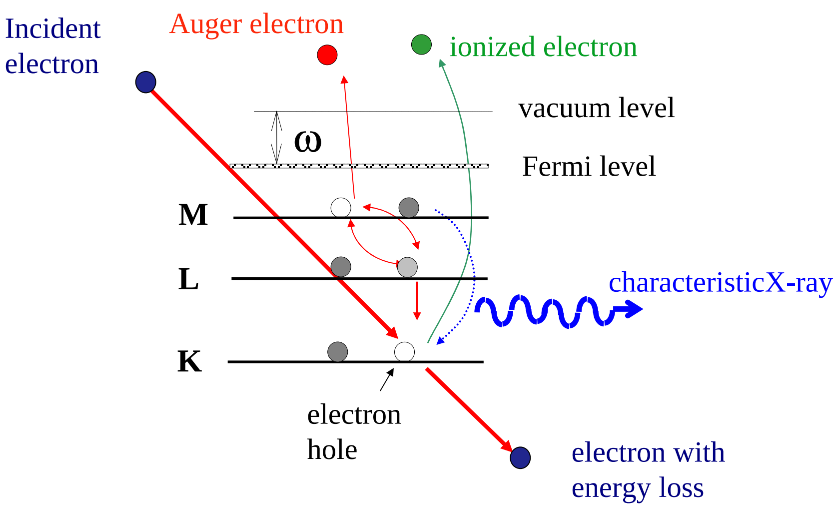
Interaction of high energy (~kV) electrons with (solid) materials-I

Interaction with an Atom

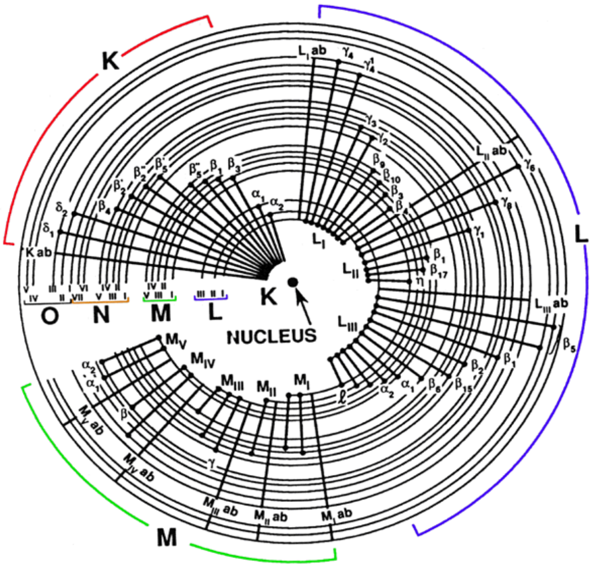


1. Unscattered
2. Low angle elastically scattered
3. High angle elastically scattered
4. Back scattered
5. Outer shell inelastically scattered
6. Inner shell inelastically scattered

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



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



K lines

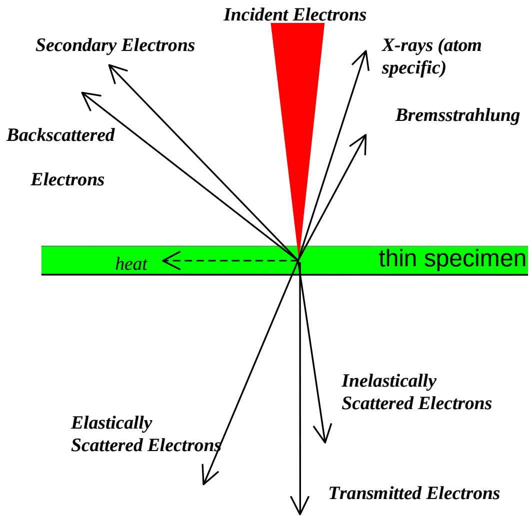
$K\alpha$, $L \rightarrow K$
$K\beta$, $M \rightarrow K$

L lines

$L\alpha$, $M \rightarrow L$,
$L\beta$, $N \rightarrow L$,
$L\gamma$, $O \rightarrow L$

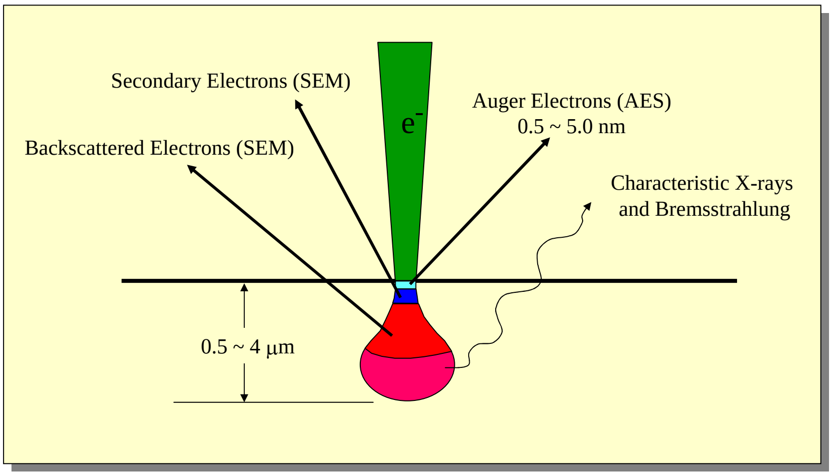
Interaction of high energy (~kV) electrons with (solid) materials-II

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.

Electron Optical Elements and Attachments

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

Electron Source

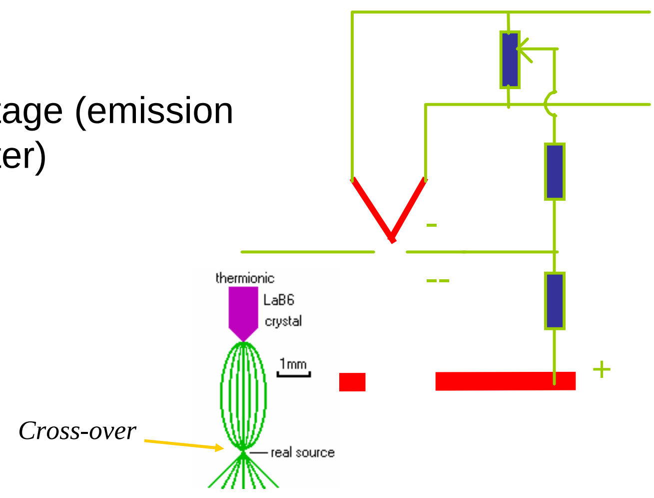
- Generation of electrons that can be accelerated by high tension to obtain the illuminating electron beam

Electron Source

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

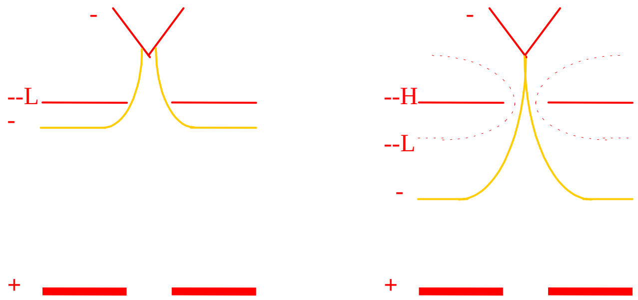
Electron Source Thermionic Gun

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



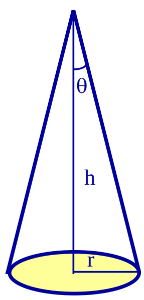
Electron Source
Thermionic Gun

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

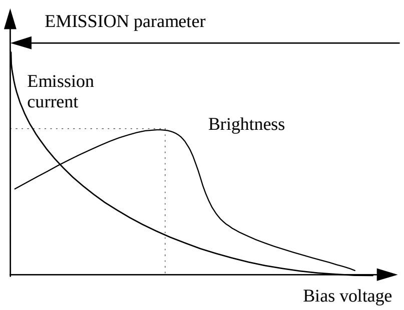


Electron Source
Thermionic Gun

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



$$\Omega = \pi\theta^2$$

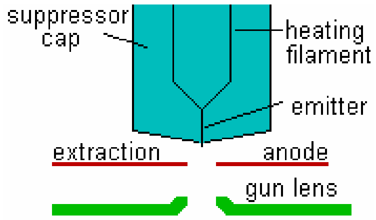


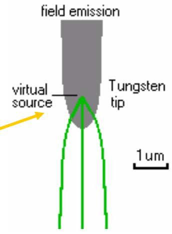
Electron Source
Thermionic Gun

- 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

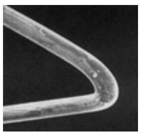


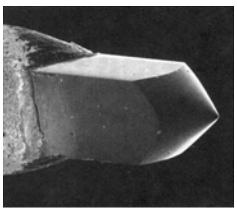


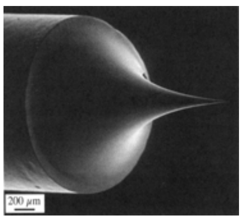
Electron seemingly originating from tip itself

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







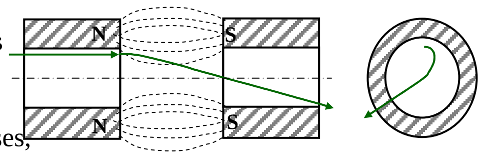
Lenses

- 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

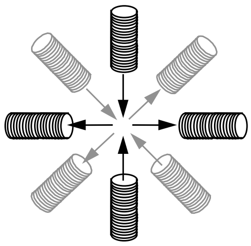


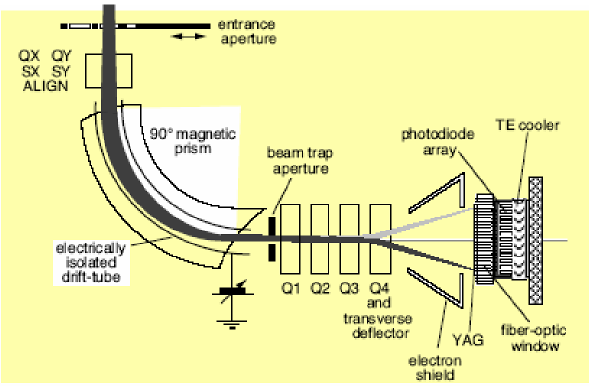
Electrostatic lenses: the Wehnelt cap

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

Pole Lenses

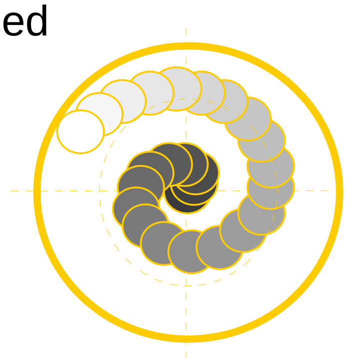
- Pole lenses are all electromagnetic, no electrostatic
- Different magnifying power in X, Y direction is possible
- The construction is just like the stigmators
- Usually seen in Cs correctors and EELS
- Qudrapole, Hexapole, Octupole lenses are common.





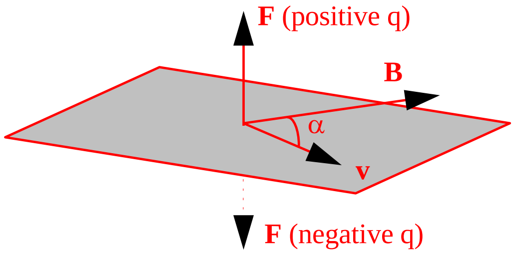
Lenses

- 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

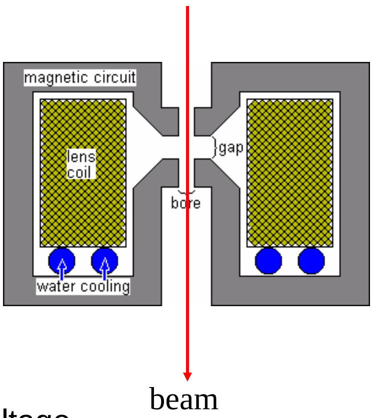


Lenses

- 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



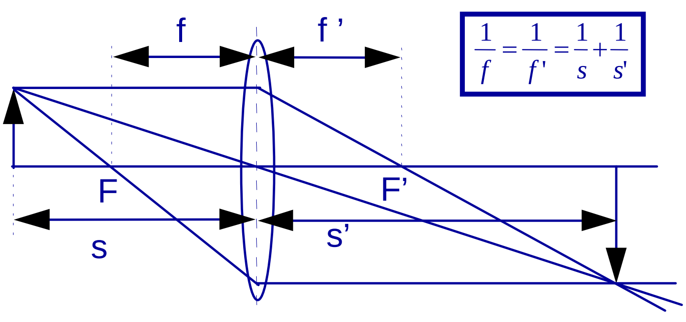
Electromagnetic Lenses for Electrons

- 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

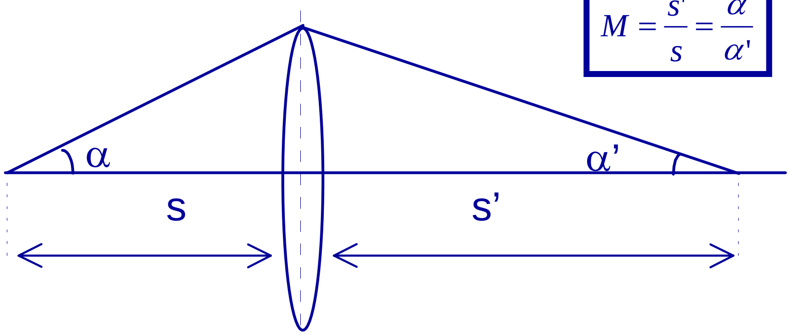
- Gaussian Law



Lenses

- Gaussian Law

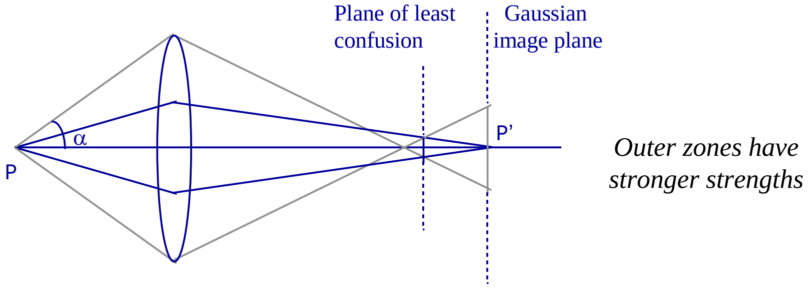
$$M = \frac{s'}{s} = \frac{\alpha}{\alpha'}$$



Lenses
Spherical Aberration

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

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



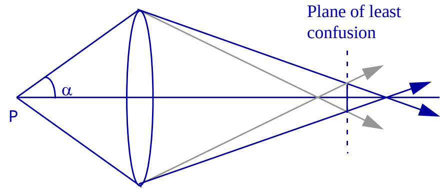
Lenses
Spherical Aberration

- 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

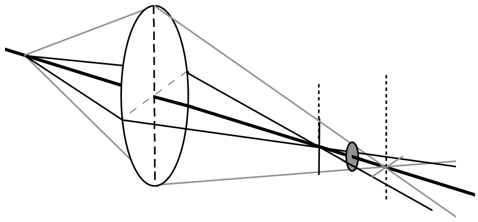


$$\delta_c = C_c \alpha \left(\frac{\Delta E}{E} + \frac{2\Delta I}{I} \right)$$

Lenses

Astigmatism

- Lens defect caused by magnetic field asymmetry



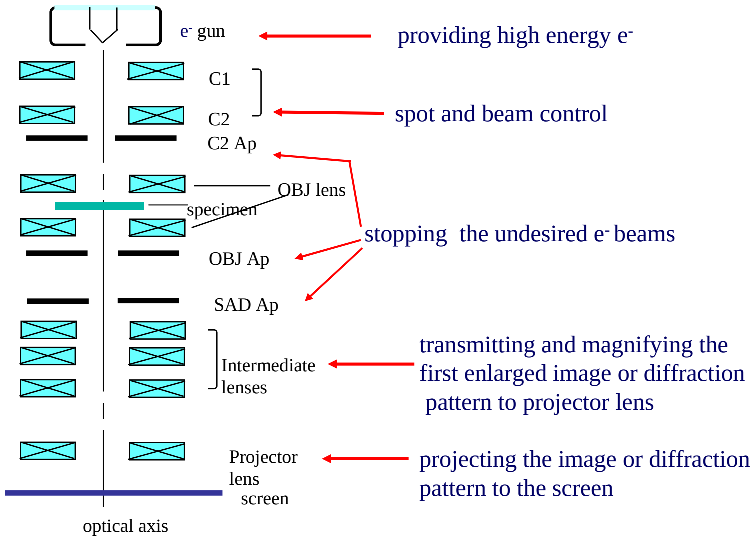
- can be corrected using stigmators!

Lens System

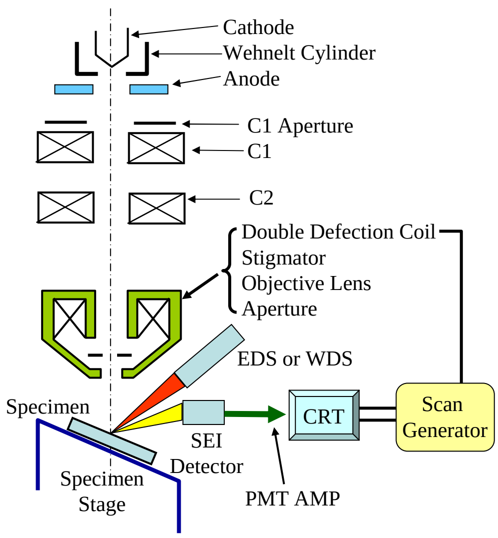
- Condenser C1Lens
- Condenser C2 Lens
- Objective Lens

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

Lens System of TEM



Lens System of SEM



Lens System
Condenser C1 and C2

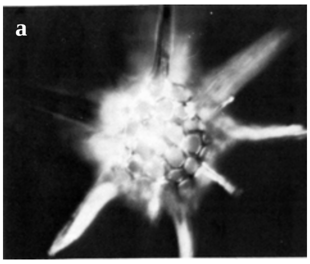
- C1
  - strong demagnifying lens
  - spotsize setting
- C2
  - weak lens
  - intensity control

Lens System &
Microscope Resolution

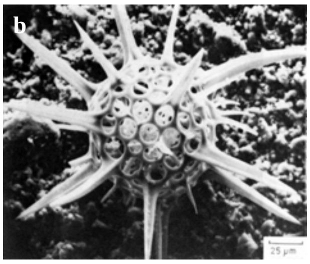
- Microscope resolution is governed by: (for TEM)
  - wavelength of electrons
  - C_s of objective lens
  - other lenses are less crucial (α/M)

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

Depth of Field or Depth of Focus



OM image

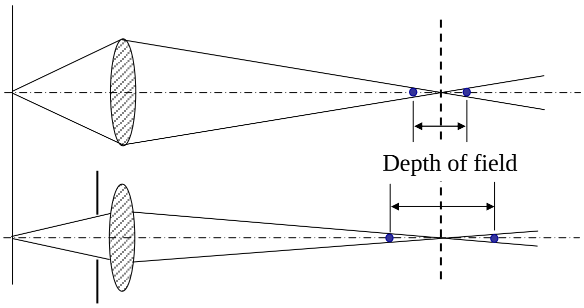


SEM image

How to increase the depth of focus of SEM image

Smaller α

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

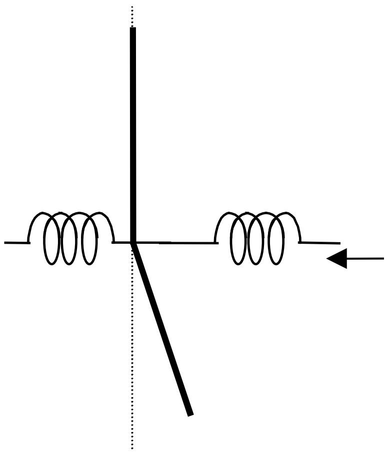


Deflection Coils

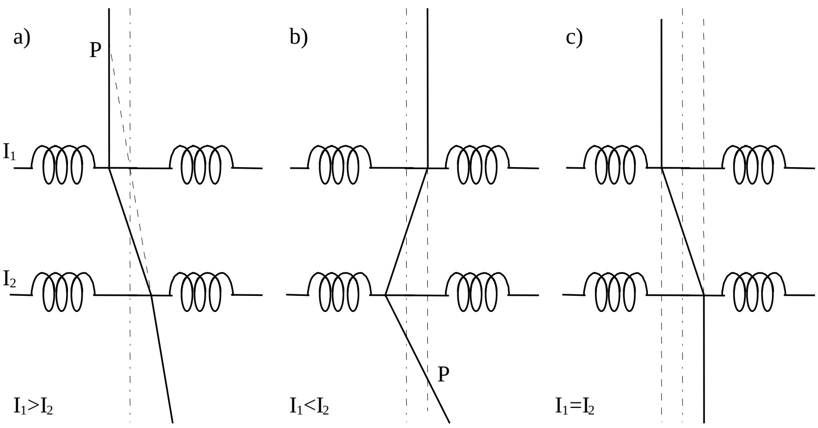
- Provide means to shift or to tilt the electron beam, to correct for mechanical misalignments of the optical system, and to obtain specific imaging effects

Deflection Coils

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



Deflection Coils

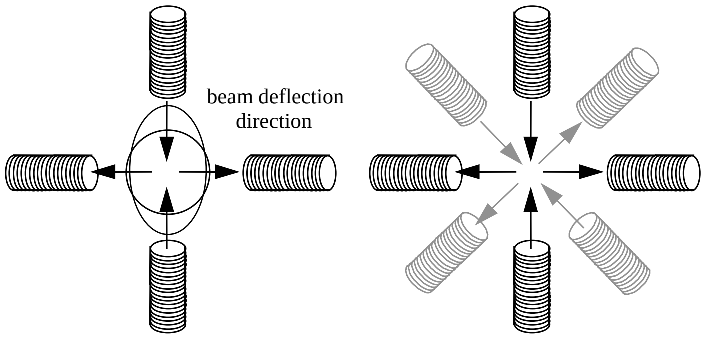


Stigmators

- 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, ………

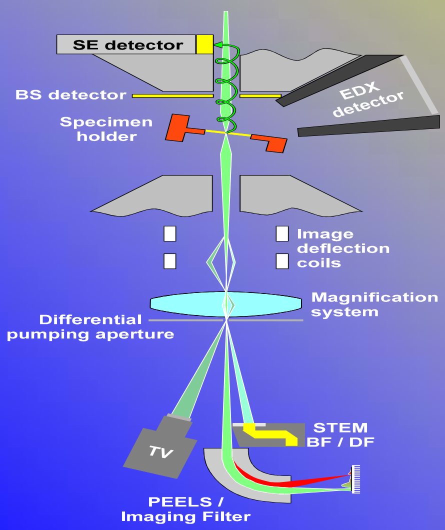
Attachments for photons or X-rays

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_a)

•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).

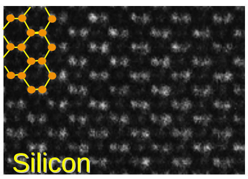
AEM vs. Conventional TEM

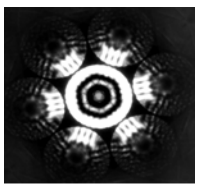
(Differences in Instrumentation)

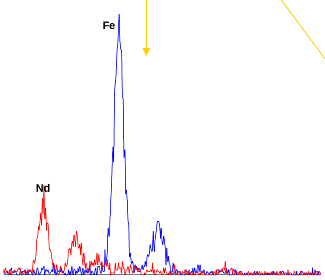
- Different illumination requirements: parallel illumination for CTEM (and HRTEM) but conical illumination for AEM
- Different designs for the objective lens to match the illumination system
- With analytical attachments: EDS for characteristic X-rays, EELS for in-elastic scattered electrons, and annular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

Types of Information from AEM

- Image
- Structure
- Chemistry





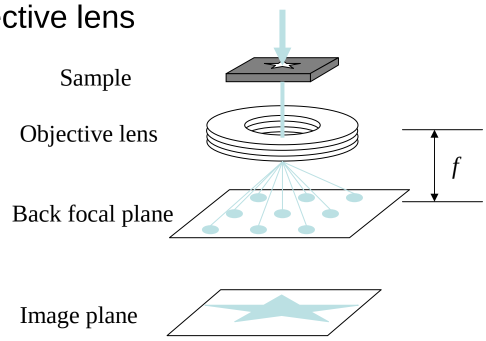


Examples of AEM Applications to the Characterization of Materialss

- 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)

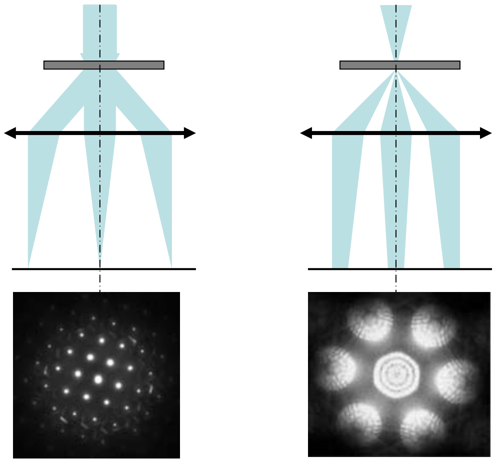
Electron diffraction

- 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

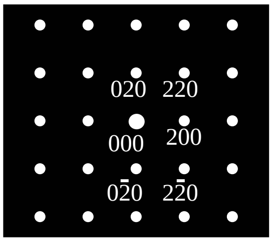
LACBED pattern along [111] of Ge

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



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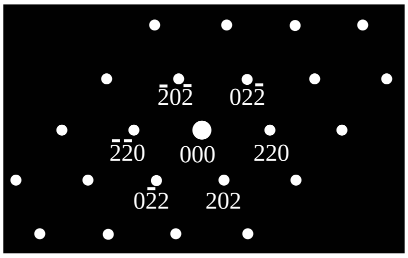
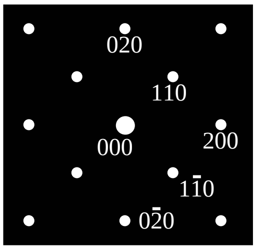


[1̄11]

Standard spot pattern

- Example 2: b.c.c



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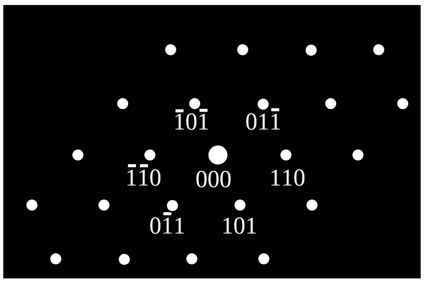
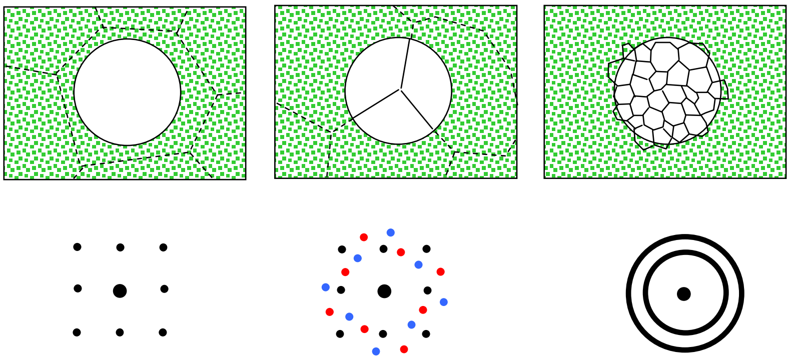


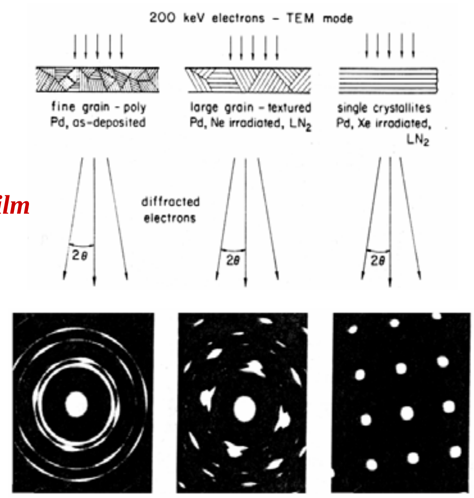
[1̄11]

Electron Diffraction Pattern--Spot to Ring



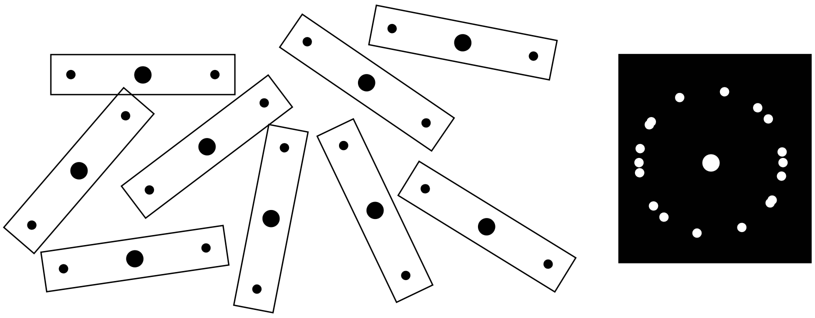
(a) (b) (c)

Electron Beam Diffraction of a Pd film



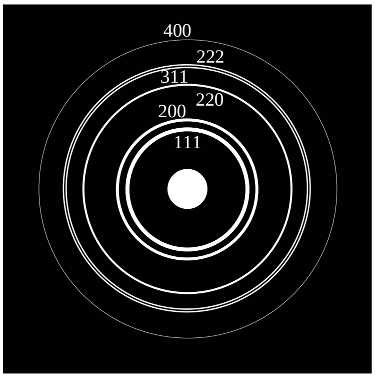
Ring pattern

- 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

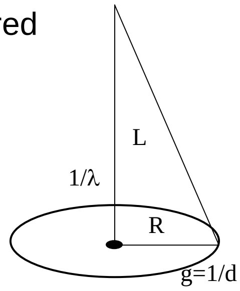
  $$Rd_{hkl} = L\lambda$$

  R: the measured ring radius

  d_{hkl}: the d-spacing being measured

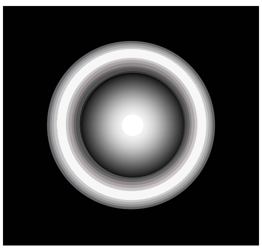
  L: camera length

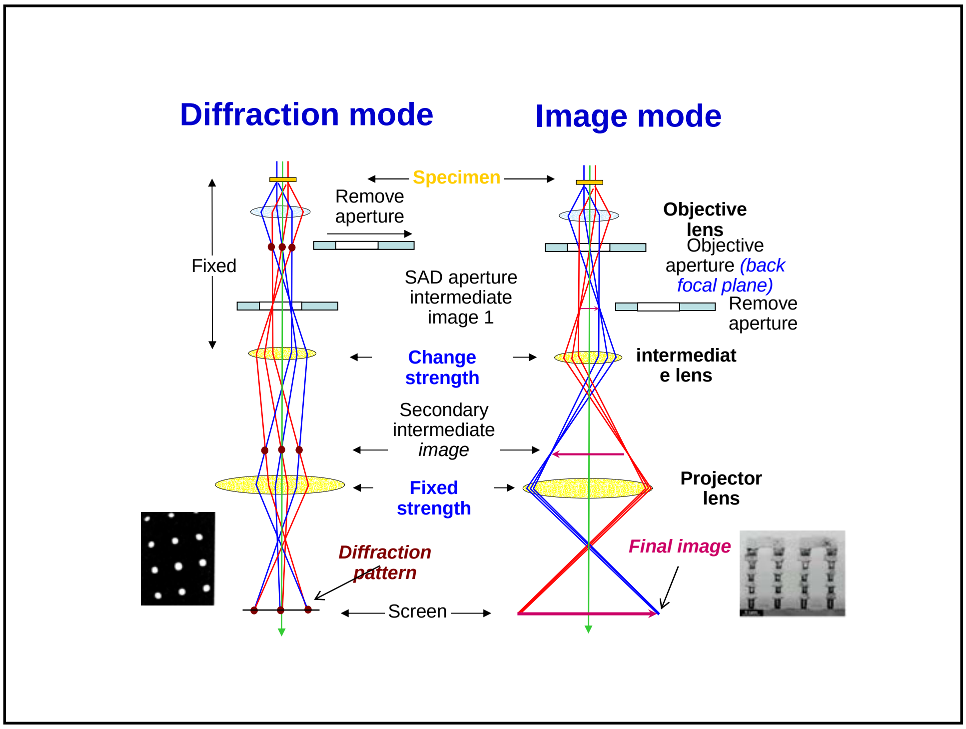
  λ: wave length of electron beam
- Camera length calibration
- Crystalline / particle fineness



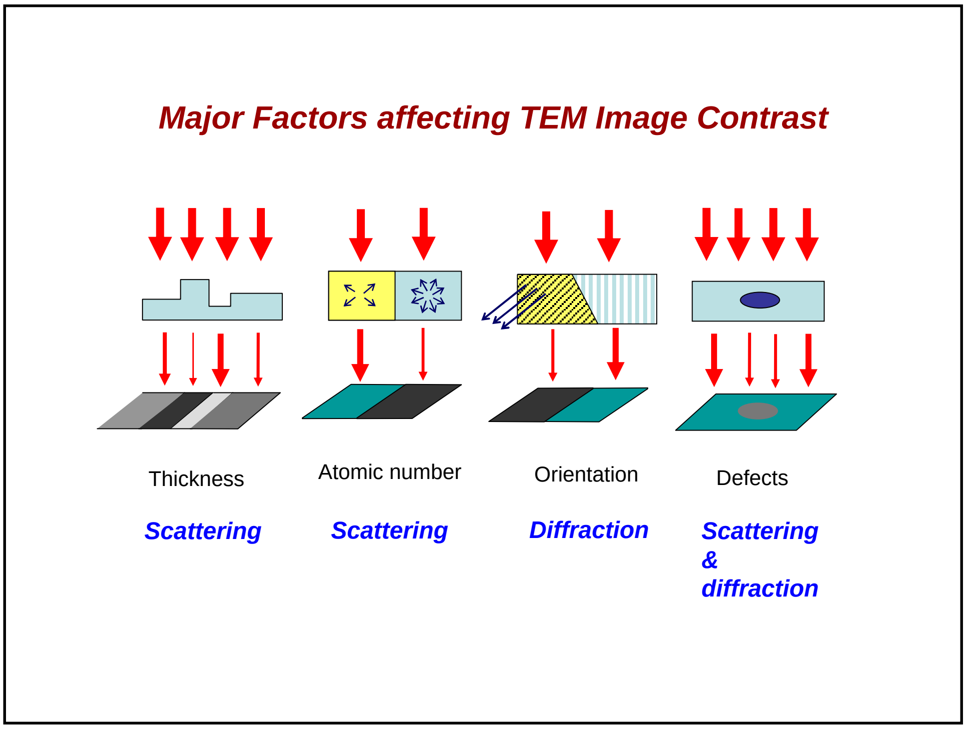
Amorphous materials

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

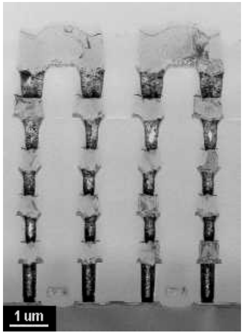




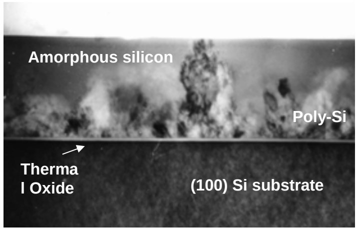
Diffraction mode
Image mode
Specimen
Remove aperture
Fixed
SAD aperture intermediate image 1
Objective lens
Objective aperture (back focal plane)
Remove aperture
Change strength
intermediat e lens
Secondary intermediate image
Fixed strength
Projector lens
Diffraction pattern
Final image
Screen

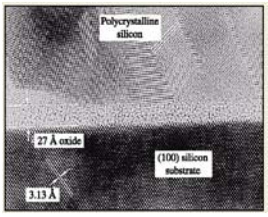


Major Factors affecting TEM Image Contrast
Thickness
Atomic number
Orientation
Defects
Scattering
Scattering
Diffraction
Scattering & diffraction

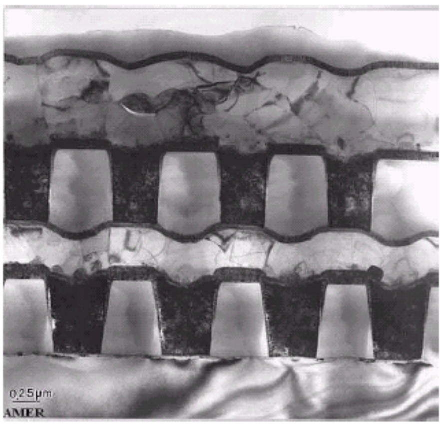


IC connectors in (five) stages. Pillars made of tungsten (hollow, dark) are connected by pieces of Al (lighter). Thin layers of TiN prevent the tungsten and Al from moving around.



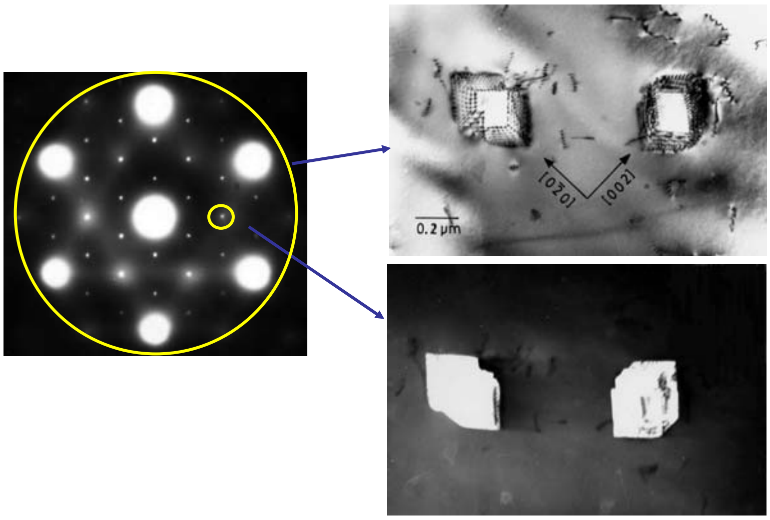


IC Cross section (CTEM BF)

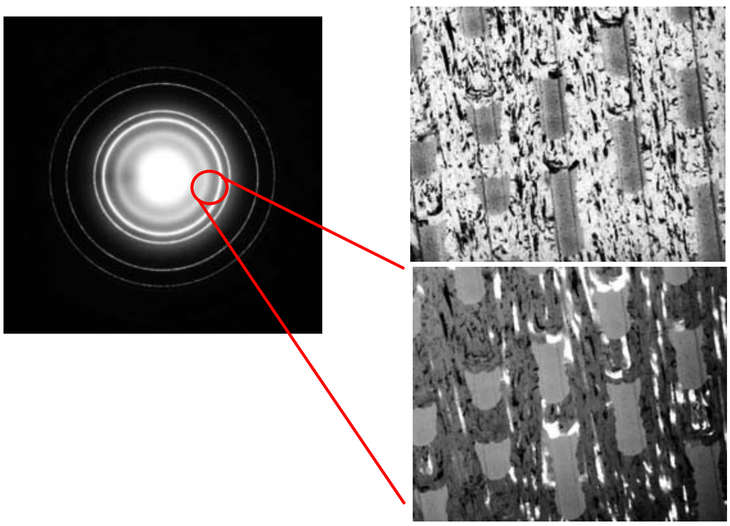


Images Courtesy of AMER

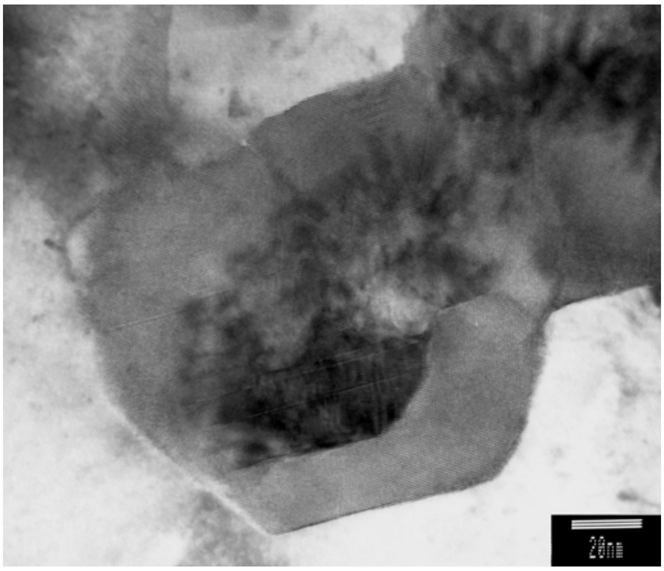
BF vs. CDF (1)



BF vs. CDF (2)

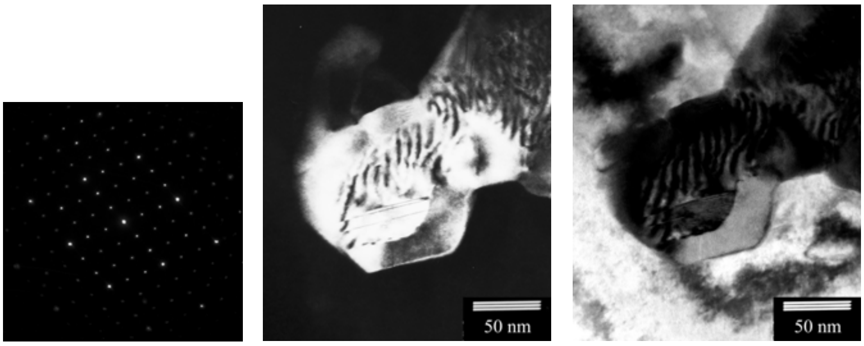


Precipitates in metal Alloys, I



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

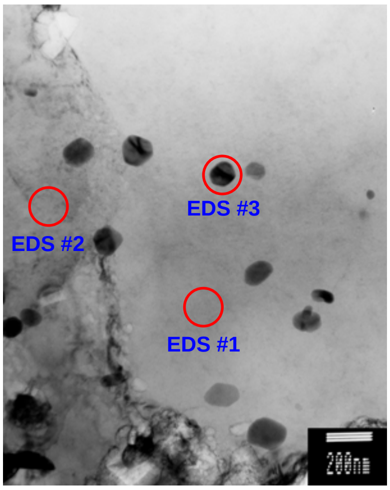
Precipitates in metal Alloys, I (cont.)



SAD DF BF

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

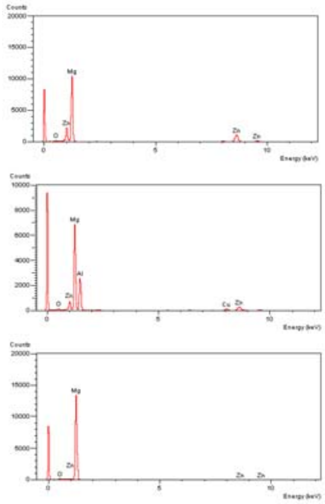
Precipitates in metal Alloys, II



EDS #3

EDS #2

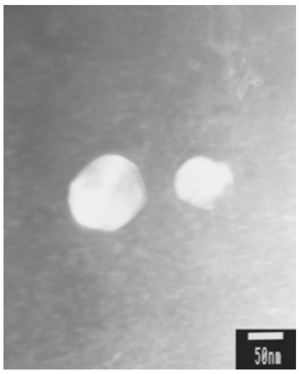
EDS #1



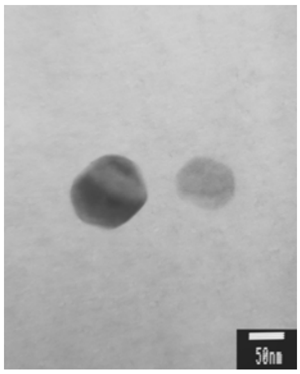
MgZn in Li-Zn-Al-Mg Alloy

Precipitates in metal Alloys, II (cont.)

SAD



DF



BF

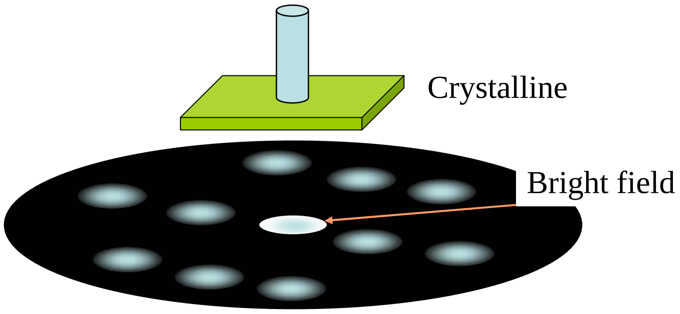
MgZn in Li-Zn-Al-Mg Alloy

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

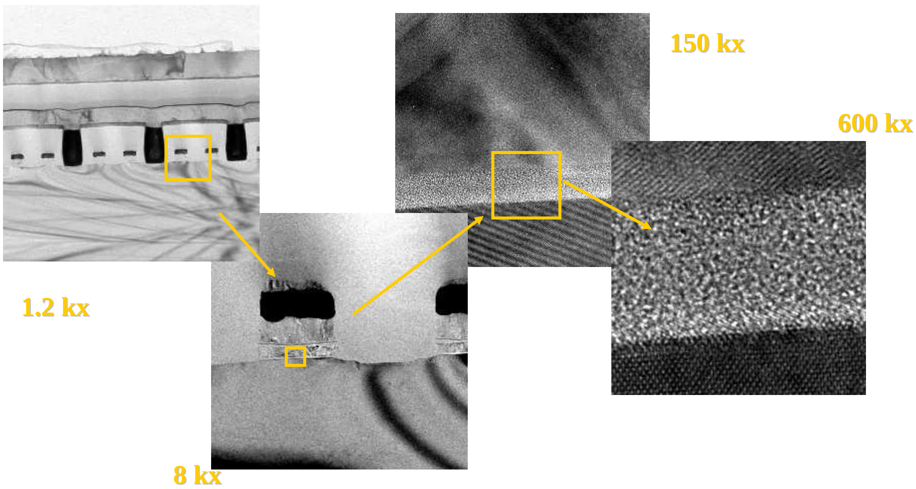
- Referred to the scattering effect
- Comparing to diffraction contrast, 'one-beam' technique

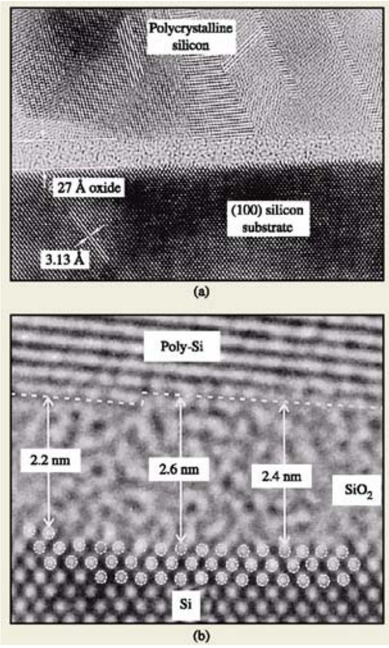


HREM image formation

- 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

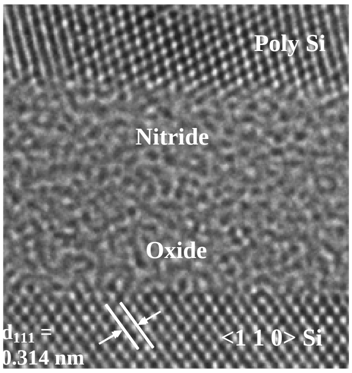


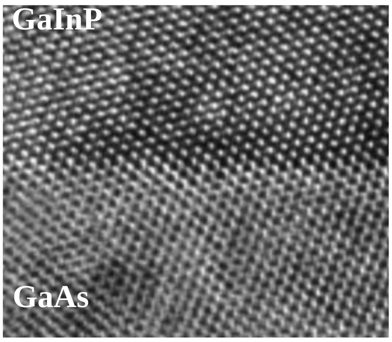


HRTEM for oxide thickness Measurement in MOS structure

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.

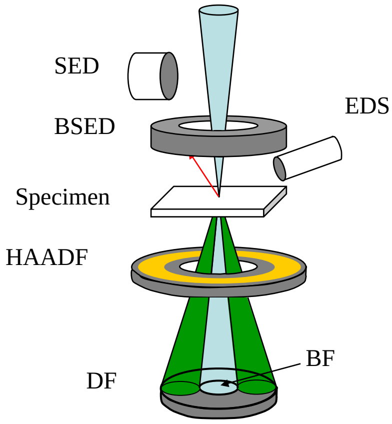
HREM Image — Interface



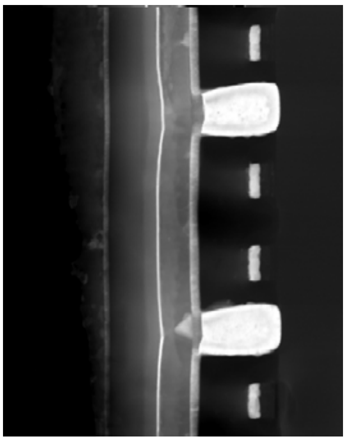


Fundamentals of STEM

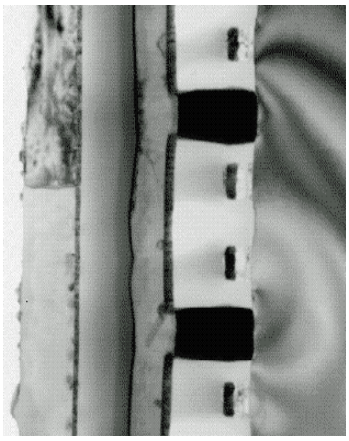
- More detectors than a SEM below the specimen, which collect beam transmitted, or diffracted, from the specimen
- The beam intensity variation contains the useful information about the location where beam is currently situated



STEM BF and ADF images from a semiconductor device

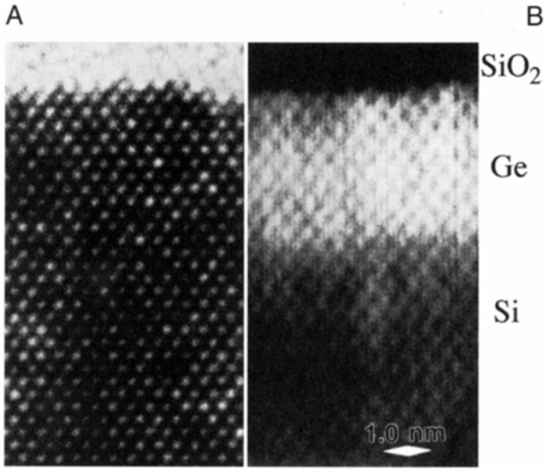


ADF



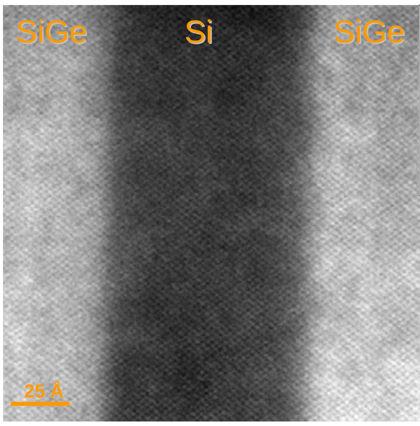
BF

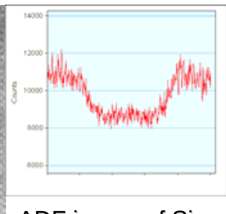
HREM vs. STEM HAADF Image — Interface

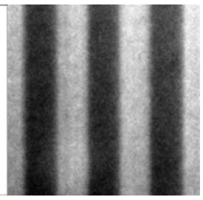


HRTEM STEM HAADF

HAADF image of SiGe alloy layers





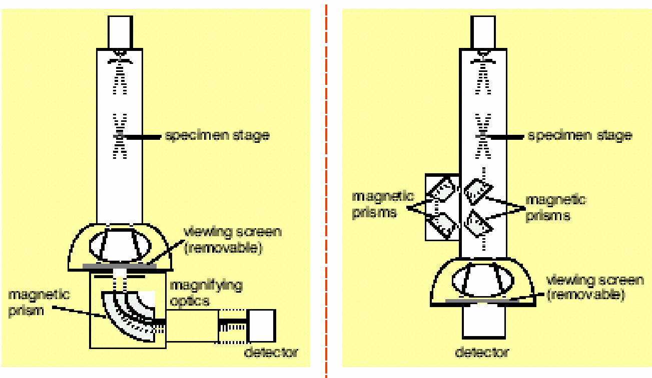


ADF image of $Si_{0.81}Ge_{0.19}$ alloy layers alternating with pure Si layers in a <110> orientation. Due to the Z-dependence of incoherent ADF STEM, the Ge rich layers are brighter than the pure Si. The interface between the layers is not sharp but shows a gradual decay.

Tecnai F20 S-Twin

EELS configurations in TEM

Experimental setups for measuring EELS data

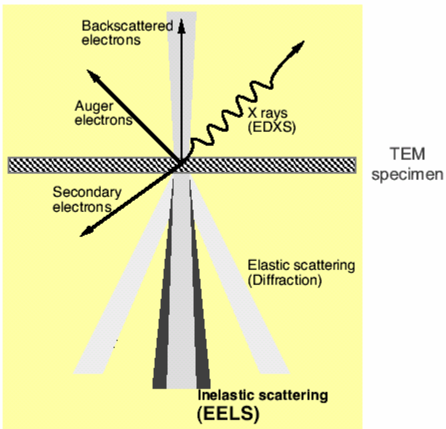


post-column filtering

in-column filtering

Signals for EELS

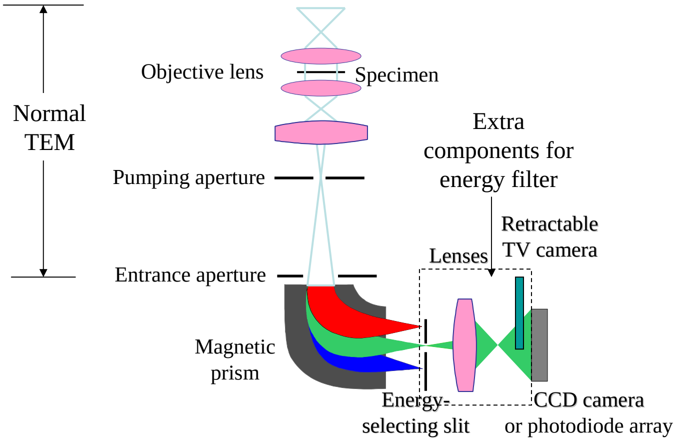
TEM beam-specimen interactions and signals



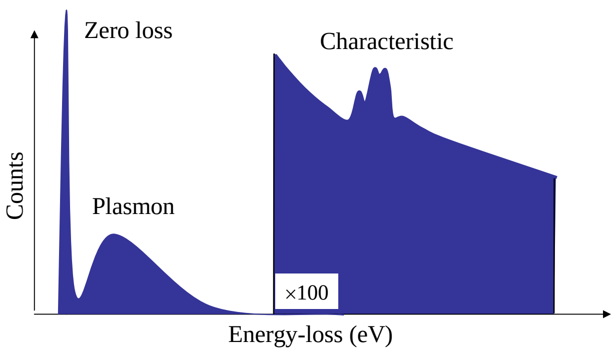
M.K. Kundmann: *Introduction to EELS in TEM*

Gatan EELS Imaging and Analysis School - May 2001 2

Post-column EELS

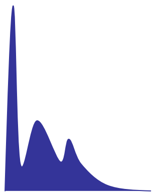


A typical EELS spectrum



Plasmon peak

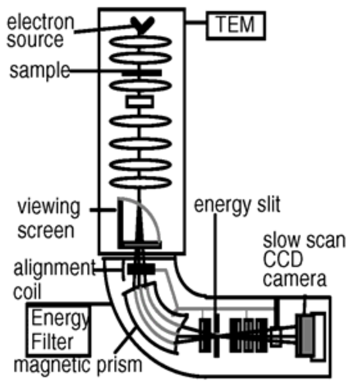
- Caused by the collective response to the incident beam by all the valence electrons
- If the sample is thicker, the plasmon peak is also higher and the second peak may appear
- The ratio of plasmon peak intensity to zero-loss peak intensity may estimate the sample thickness



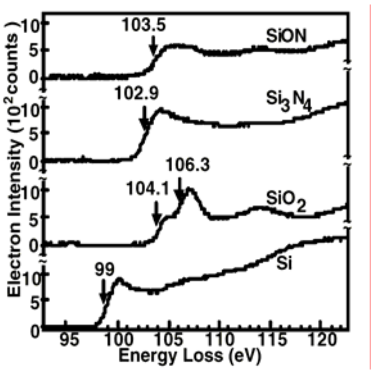
EELS vs. EDS

- 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



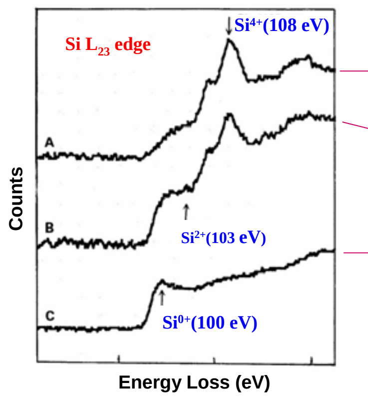
Schematic diagram of AEM-EELS

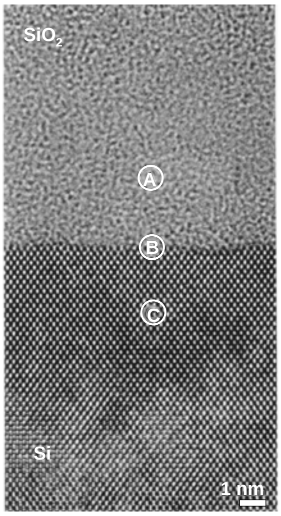


Chemical shift of core-loss edge energy in EELS spectra of some Si compounds

Y. Mitsui et al., IEDM'98

TEM and HREELS for the SiO2 / Si Interface

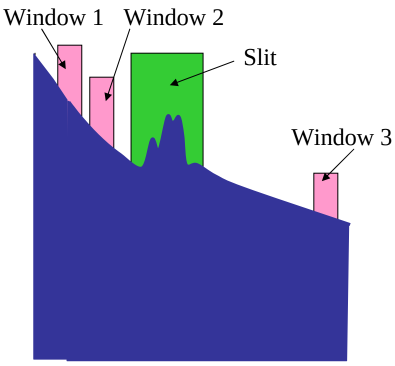




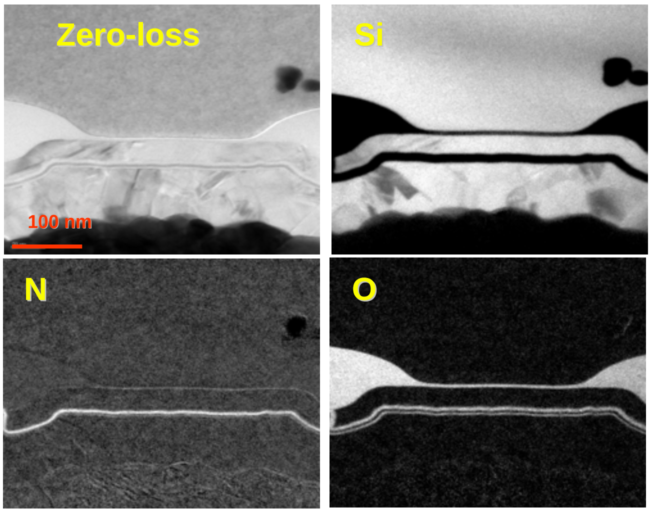
Energy filter

- 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

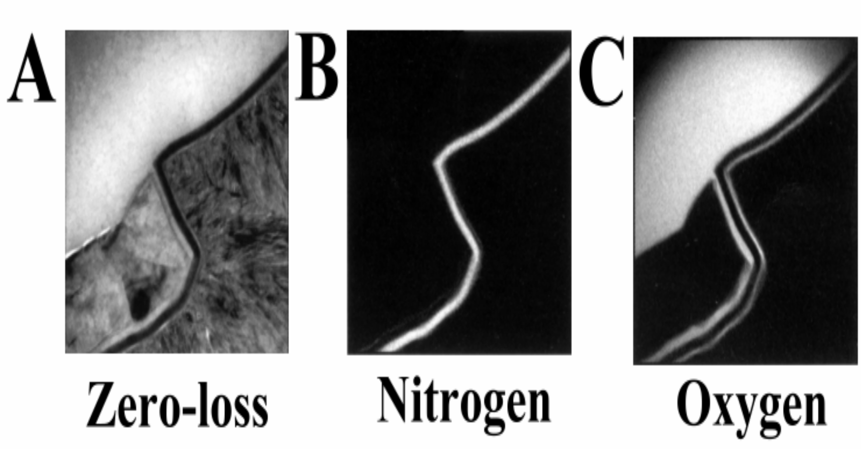


EFTEM mapping of a DRAM

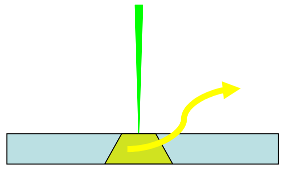


Tecnai F20 S-Twin

EFTEM mapping of the ONO layer in a DRAM



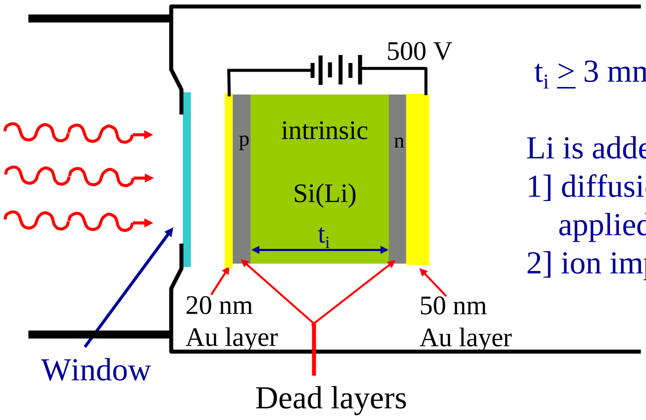
EDS



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

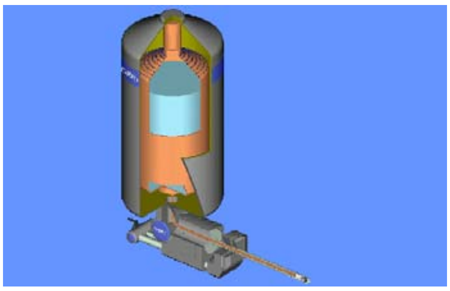
EDS system on TEM

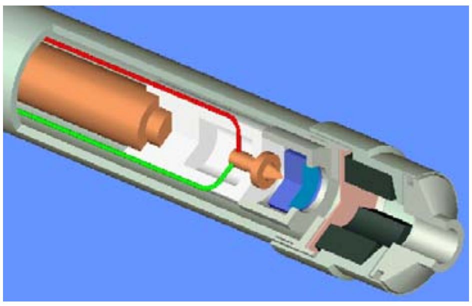
Reverse-biased p-i-n diode

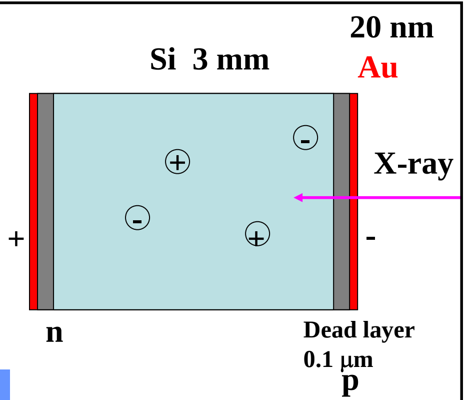


$t_i \geq 3$ mm

Li is added by
1] diffusion under an applied voltage
2] ion implantation/annealing



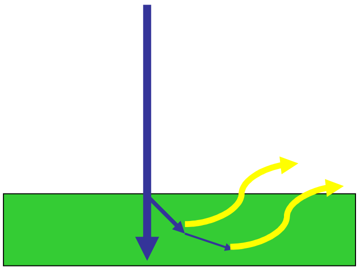




EDS Detector

Spatial Resolution

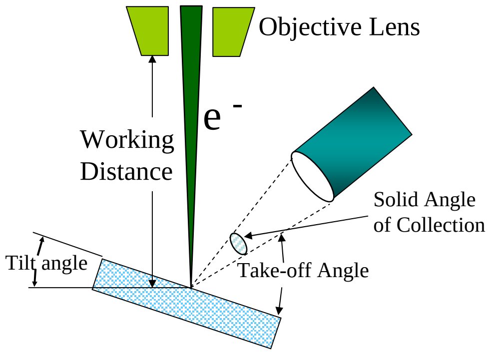
- Beam broadening size $b_{TEM} < b_{SEM}$
- Beam broadening size $b_{EELS} < b_{EDS}$



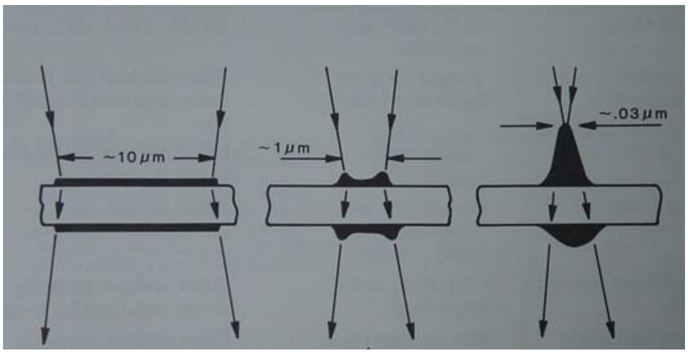
Factors on Spatial Resolution

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

Parameters of EDS Collection



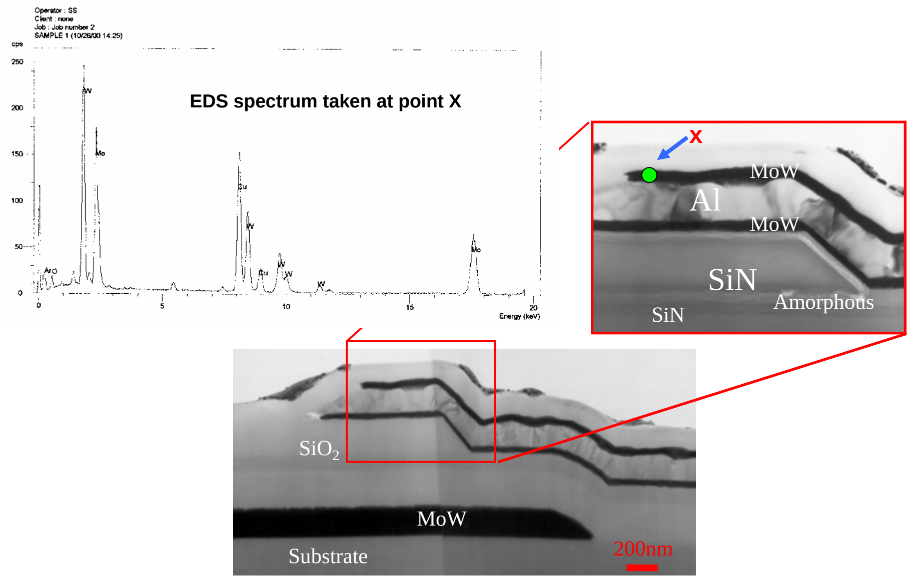
Contamination



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



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.