Fundamentals of Scanning Electron Microscopy and Energy Dispersive X-ray Analysis in SEM and TEM

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Outline

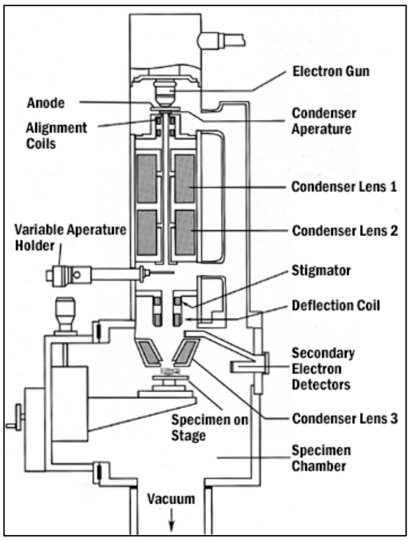
- SEM
  - Microscope features
  - BSE
  - SE
- X-ray EDS
  - X-rays - origin & characteristics
  - EDS system
  - Qualitative & quantitative analysis

SEM: Basic Principles

The scanned image is formed point by point in a "serial fashion".

"microscope"

control console (electronics)



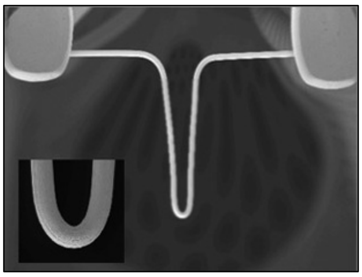
- Electron source - gun
- Electron column
  - lenses
  - apertures
  - deflection coils
- Specimen chamber with detectors

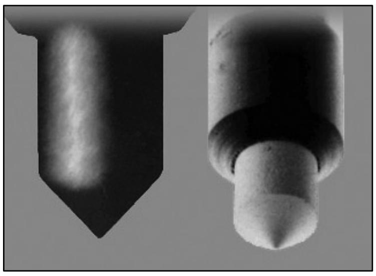
SEM: Electron Gun

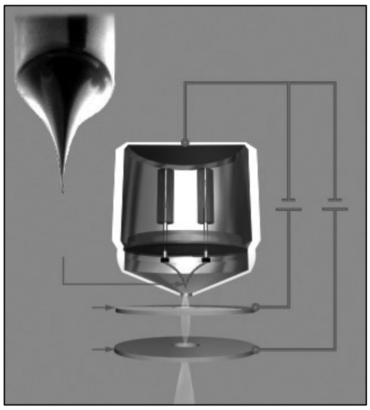
- Generates & accelerates electrons to an energy range 0.1-30 keV.
- Characteristics:
  - Emission current, i_e
  - Brightness,

    β= current/area·solid angle$=4i_p/\pi^2 d_p^2 \alpha_p^2$
  - Source size
  - Energy spread
  - Beam stability
  - Lifetime & cost

SEM: Electron Emitters

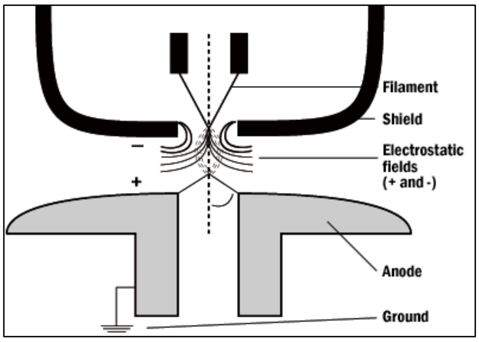


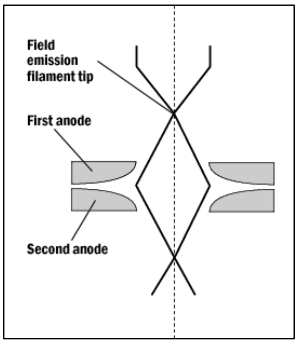




W- hairpin
$\beta=10^5$ A/cm^{2}sr
d_s= 30-100 μm

LaB_6
$\beta=10^6$ A/cm^{2}sr
d_s= 5-50 μm

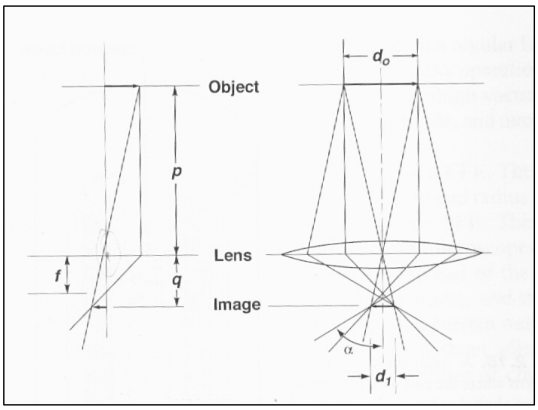


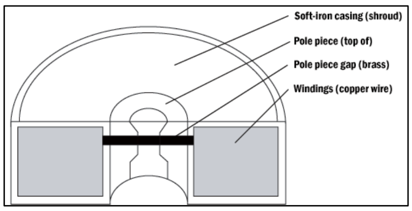


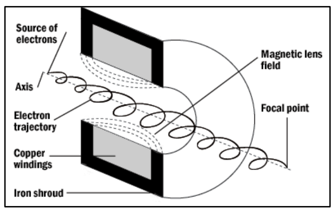
Field emission
- Cold
- Thermal
- Schottky

$\beta=10^8$ A/cm^{2}sr
d_s<5nm

SEM: Electron Lenses





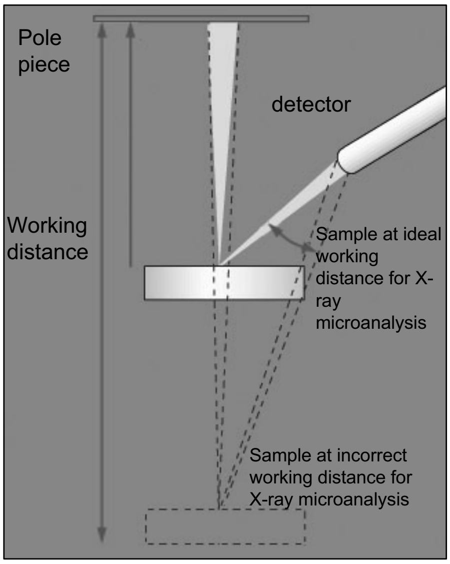


$1/f=1/p+1/q$

Magnification $=M=q/p$
Demagnification$=m=p/q$

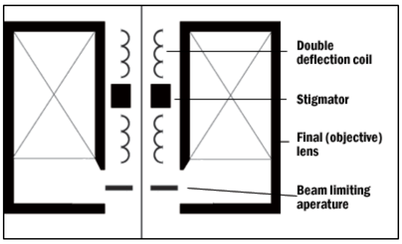
Electromagnetic lens defects:
- Spherical aberration: $d_s=1/2C_s\alpha^3$
- Chromatic aberration: $d_c= C_c\alpha(\Delta E/E_0)$
- Diffraction: $d_d=0.61\lambda/\alpha$
- Astigmatism

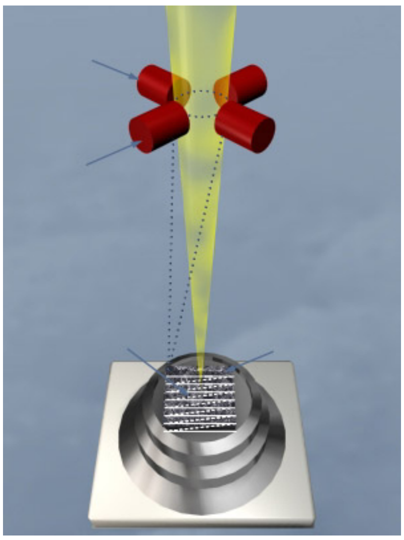
SEM : working distance

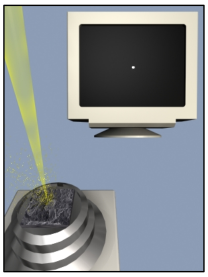


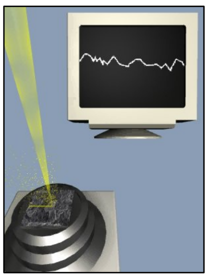
- The working distance is defined as the distance between the lower pole piece of the objective lens and the plane at which the probe is focused.

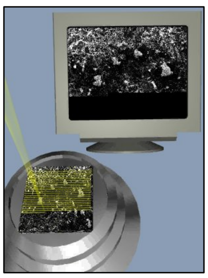
SEM: Deflector (scan) Coils



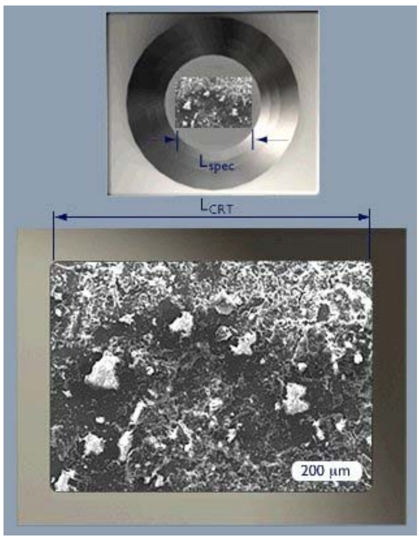




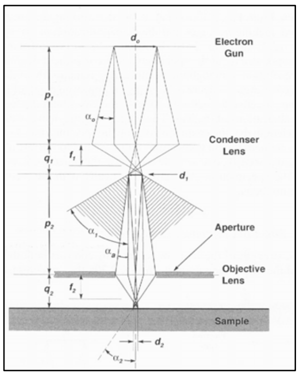




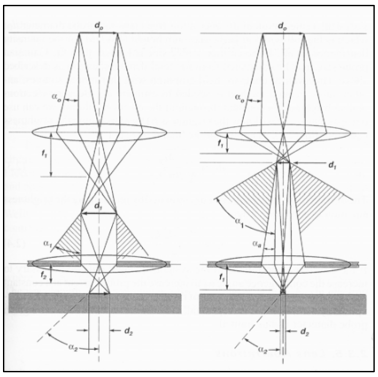
- **Magnification= $L_{specimen}/L_{CRT}$**



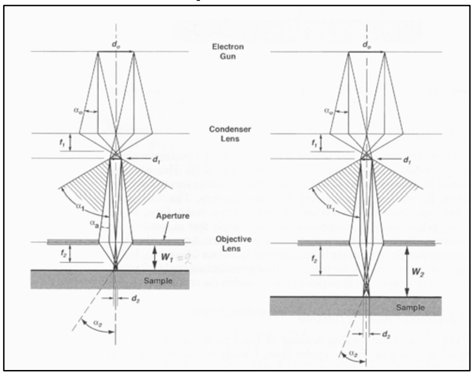
Operator control of SEM Lenses



Effect of aperture size



Effect of condenser lens strength

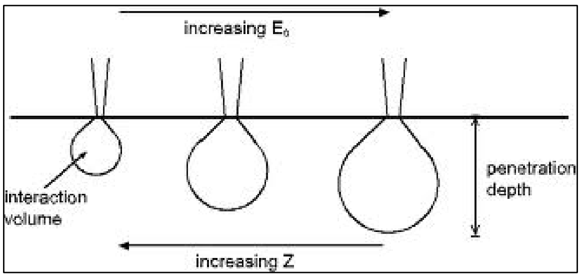


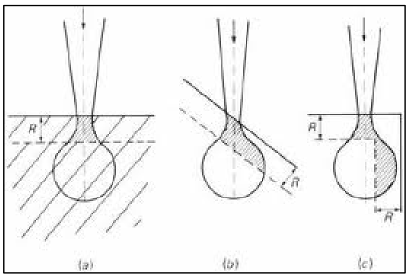
Effect of working distance

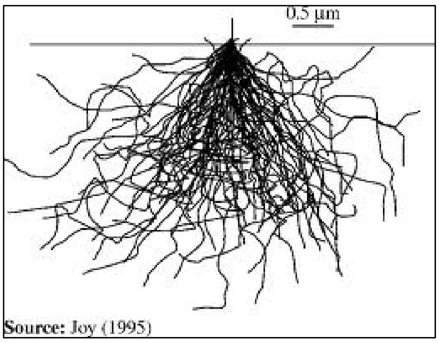
$$d_p=(d_G{}^2+d_S{}^2+d_d{}^2+d_C{}^2)^{1/2}$$

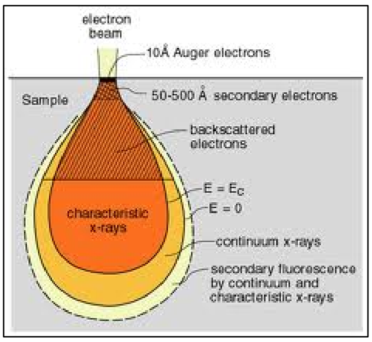
$$d_{min}= KC_s{}^{1/4}I^{3/4}(i_p/\beta\lambda^2 +1)^{3/8}$$

SEM: Interaction Volume





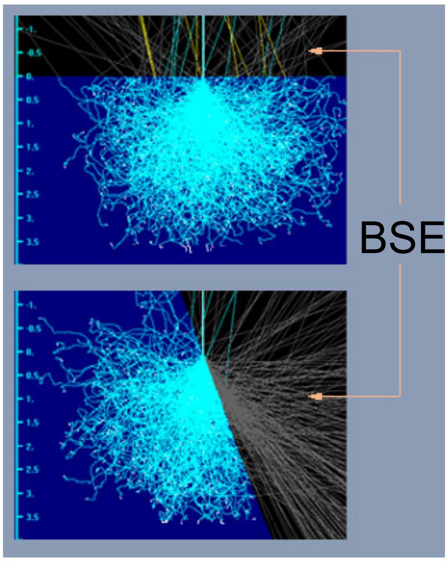


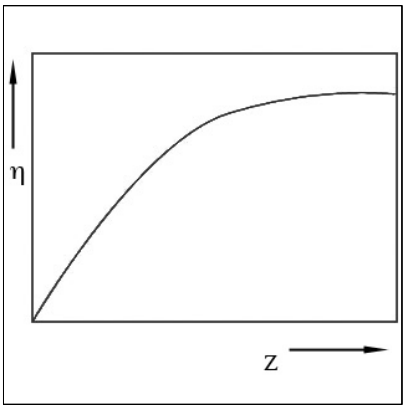


- Monte Carlo simulation

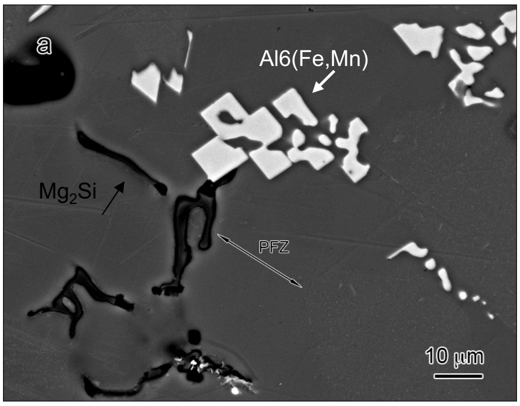
- Penetration depth: 1-5 µm

SEM: Backscattered electrons

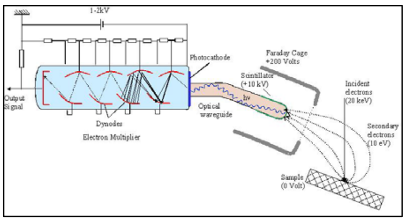


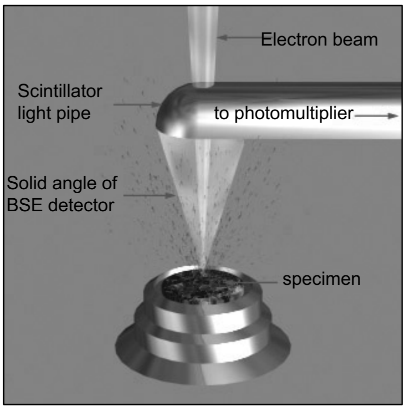


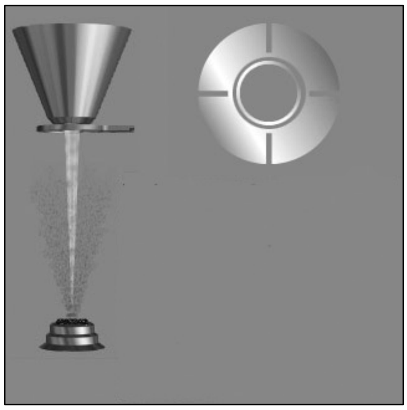
- $\eta = n_{bse}/n_b = i_{bse}/i_b$
- $\eta(\phi) = \eta_n \cos\phi$



SEM: BSE Detectors

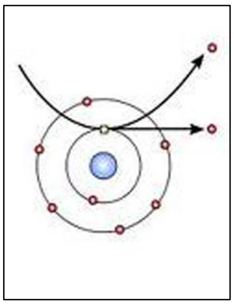




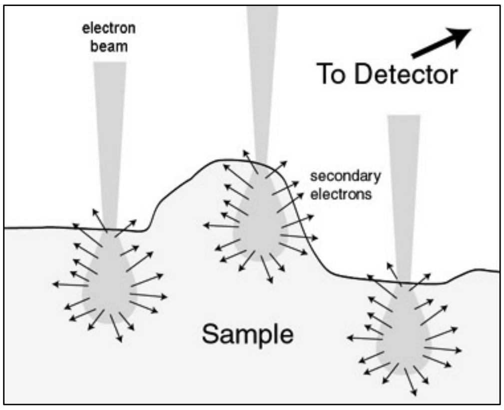


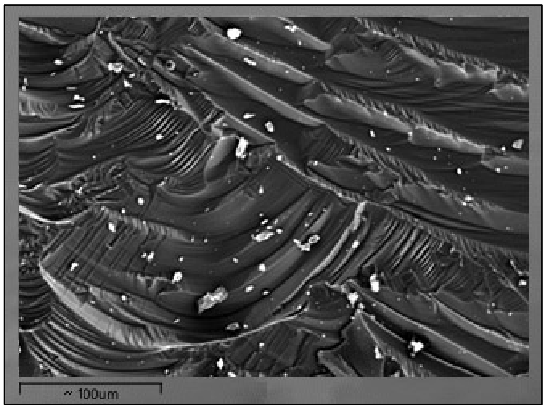
- Everhart-Thornley detector (with negative bias)
- Scintillator detector
- The solid state detector

SEM: Secondary electrons

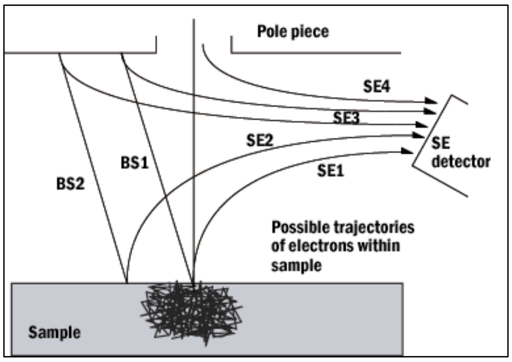


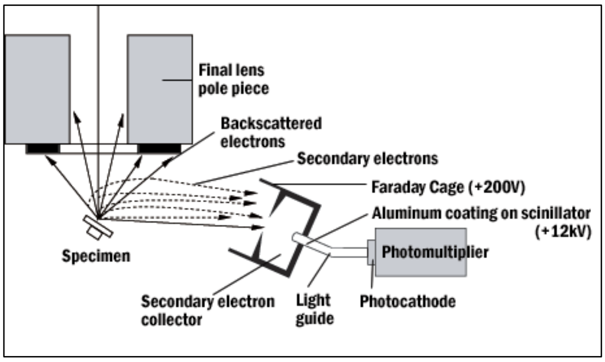
- SE I - surface
- SE II - surface + volume
- SE III & IV - noise





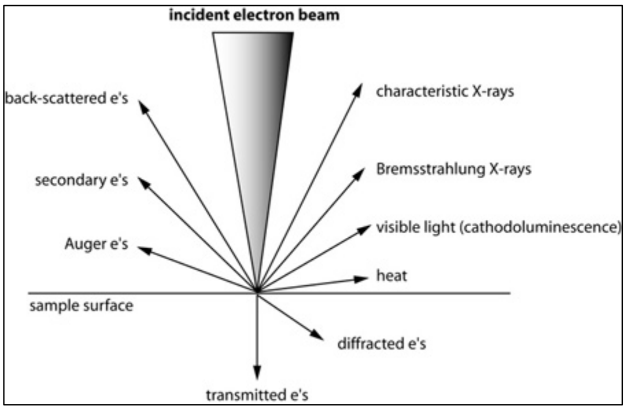
SEM: SE detector

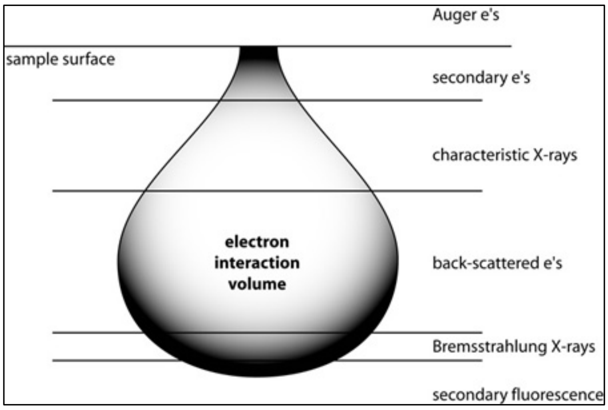




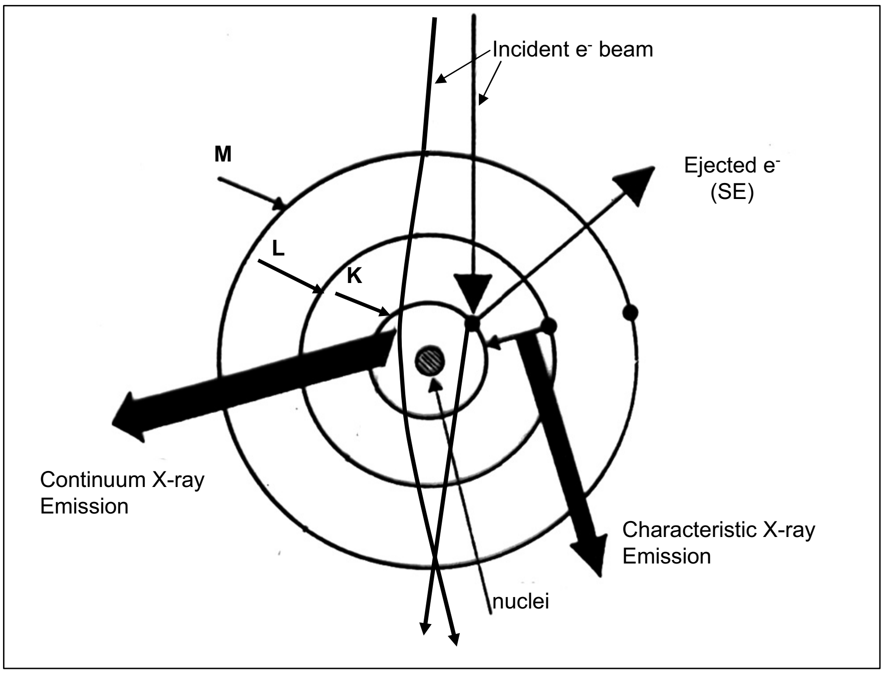
- Everhart-Thornley detector (with positive bias)

SEM: Other signals





Generation of X-rays

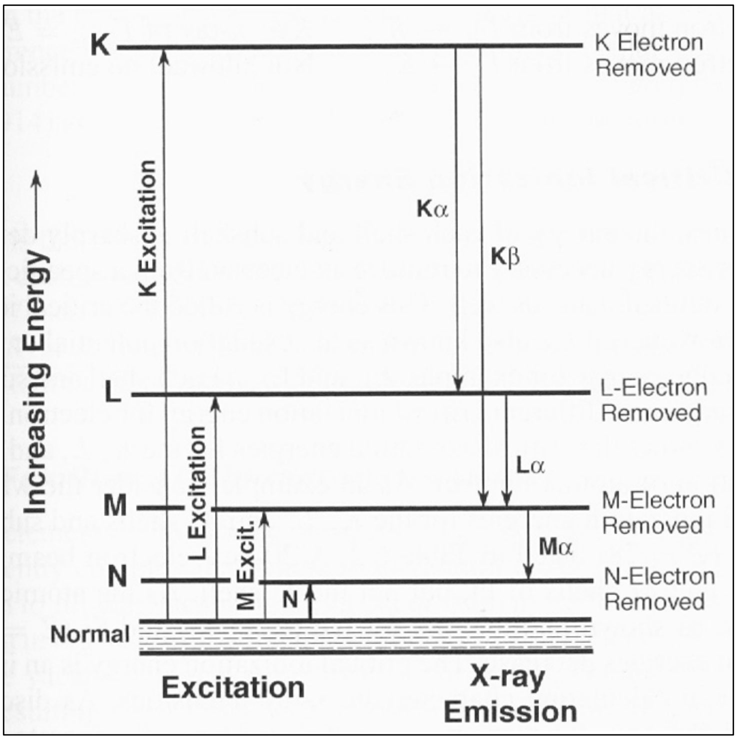


Characteristic X-rays photons

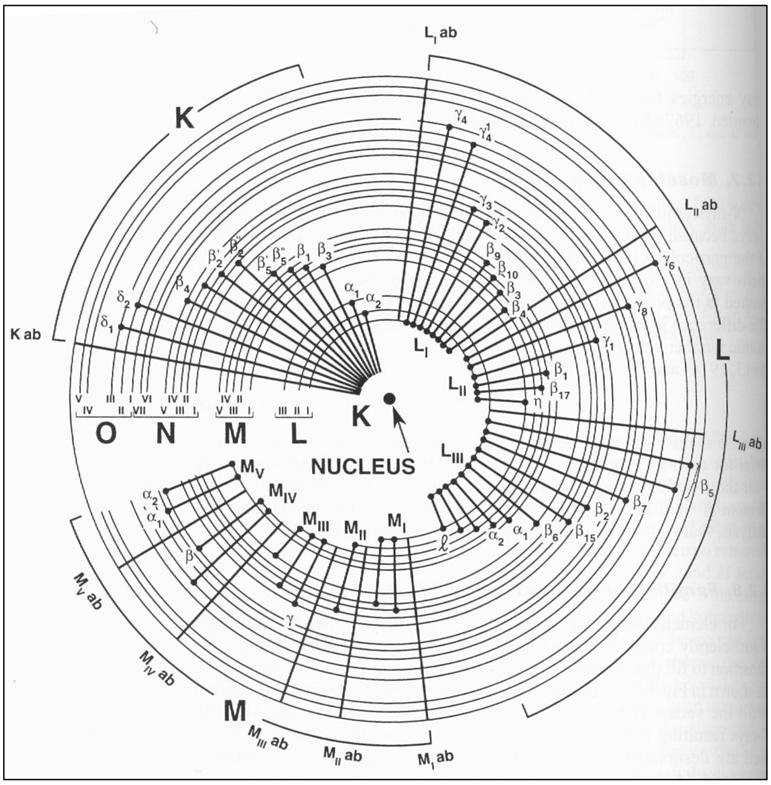
Auger electrons

Continuum X-rays or bremsstrahlung radiation

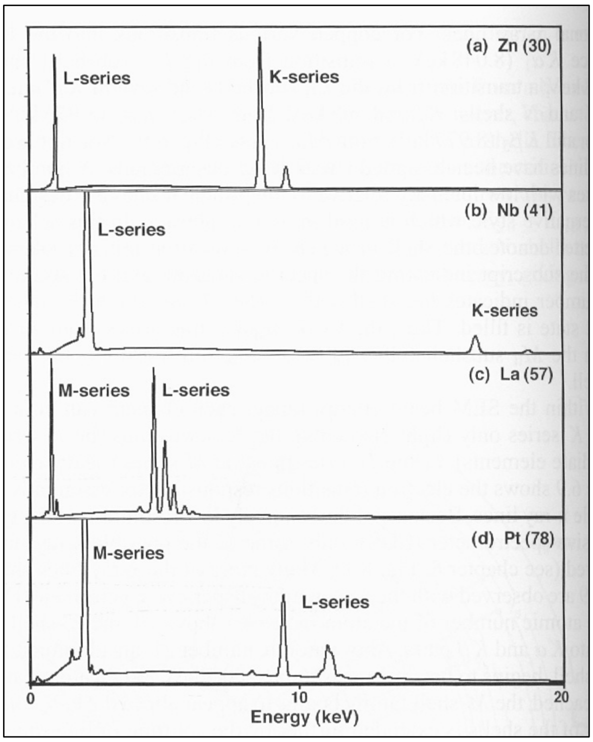
Electron Transitions



Families of Characteristic lines

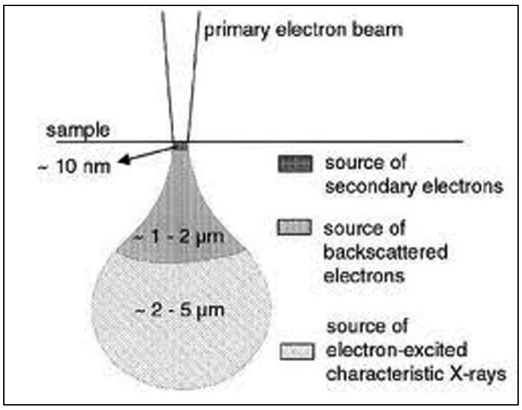


EDS Spectra for Families of X-ray lines



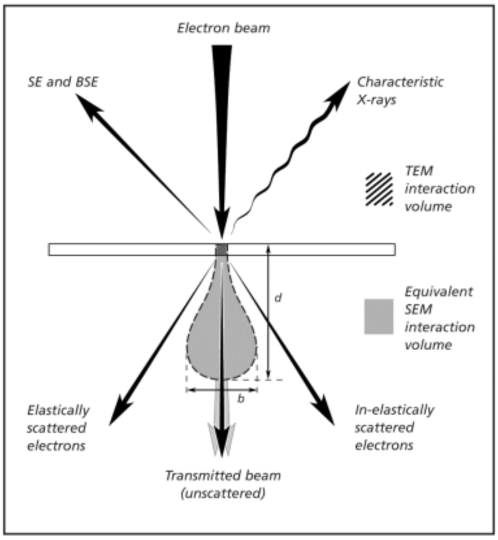
EDS: SEM - vs - STEM (TEM)

SEM



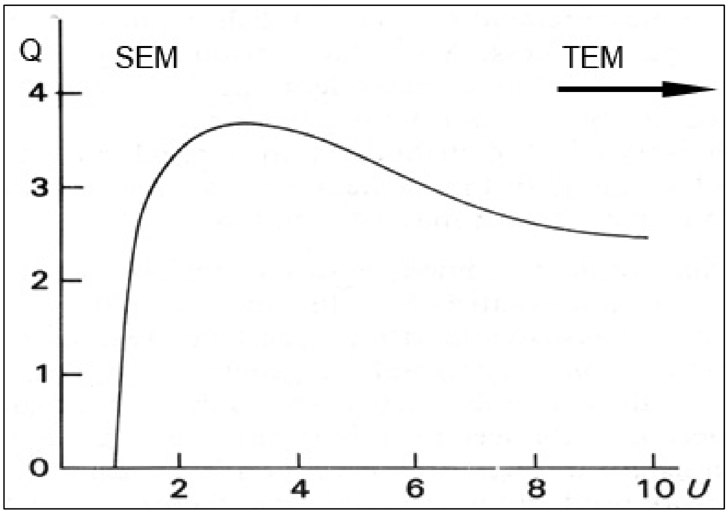
- e beam energy 0.1-**20** keV
- min probe size > 1nm
- interaction volume ~ µm

STEM (TEM)



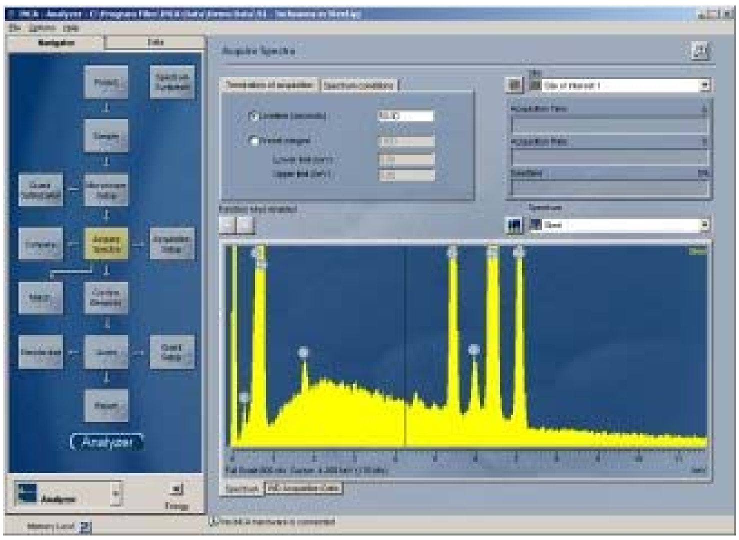
- e beam energy 100-400keV
- min probe size < 1nm
- specimen thickness <100 nm

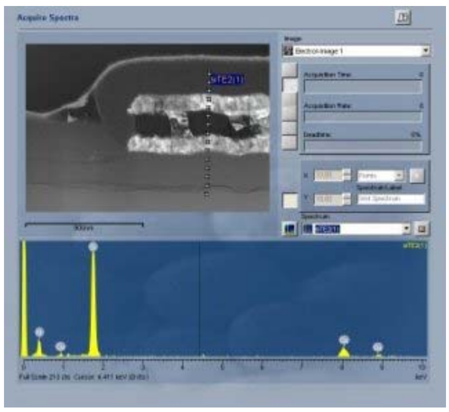
EDS: SEM - vs - STEM (TEM)

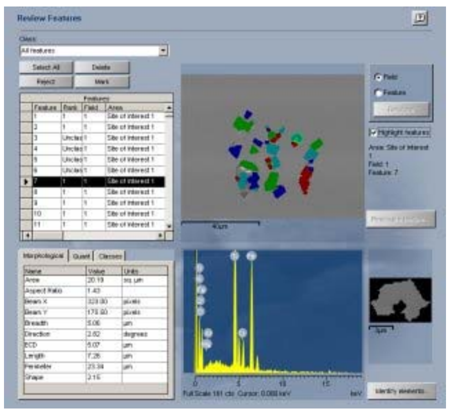


- The energy of the incident electron must be larger than ionization energy for a given shell.
- U is overvoltage, $U=E/E_J$
- Optimal value of U≈2

EDS User Interface



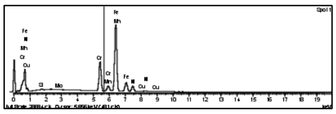




EDS System

X- ray Detector:
Detects and converts X-rays into electronic signals

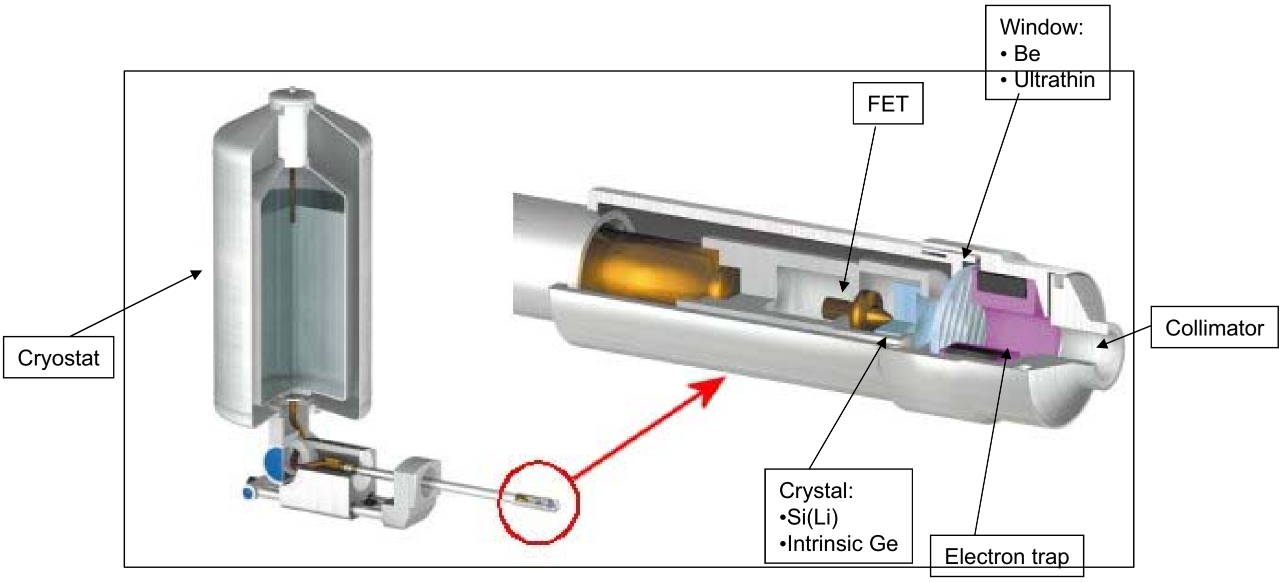
Pulse processor:
Measures the electronic signals of each X-ray measured.



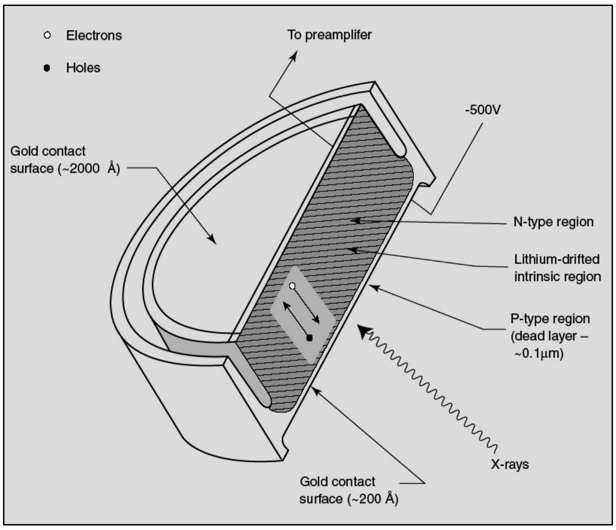
Element	Weight%	Atomic%
Si	0.32	0.68
Cr	18.84	20.00
Mn	2 1.39	1.42
Fe	70.25	69.31
Ni	8.76	8.24
Cu	0.21	0.20
Mo	0.23	0.15
Totals	100.00	100.00

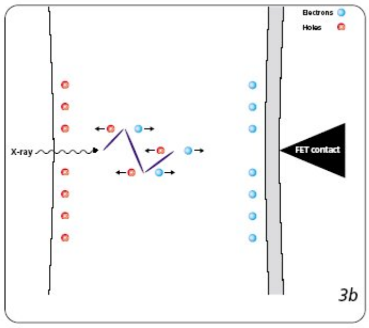
Analyzer:
Displays & analyses data.

EDS: Detector components

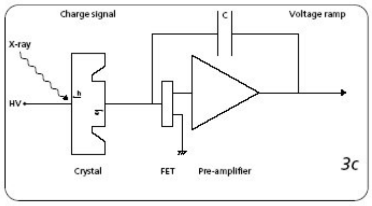


EDS: How the detector works?





- creation of electron/hole pair 3.8 eV in average
- electronic noise & incoplete charge collection reduce energy resolution.



EDS: Pulse detection and analysis

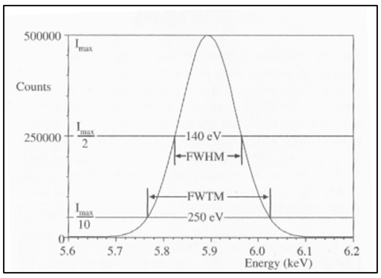
- Time constant, τ

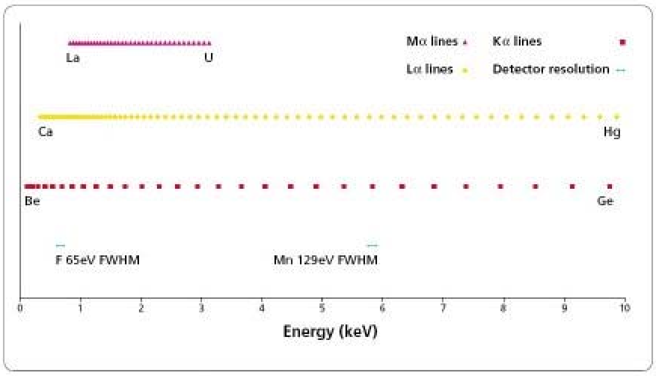
  - shorter τ: higher count rate, but peak broadening (lower energy resolution)

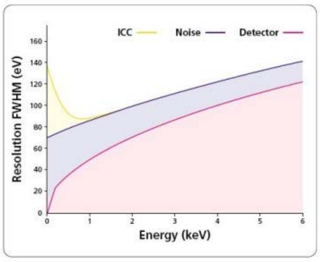
  - longer τ: better resolution, but increase in dead time

- Dead time
  - Dead time , %= $(1-R_{out}/R_{in})$x100%

EDS: Energy resolution



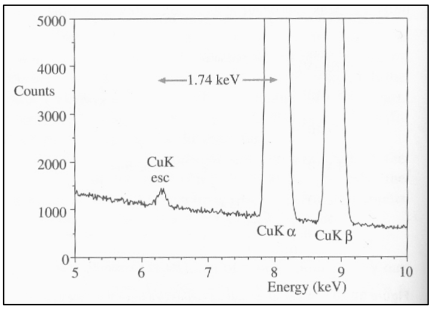


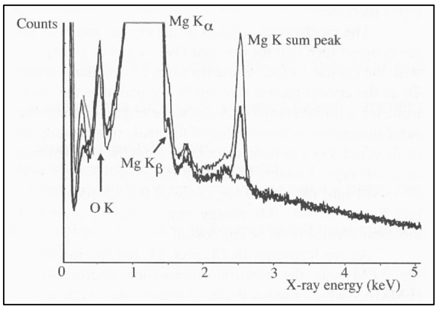


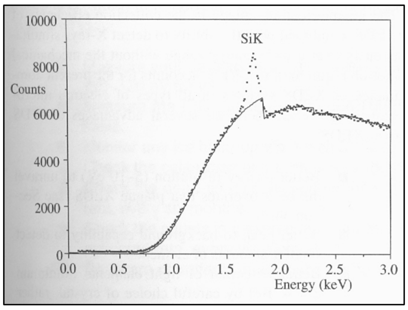
$$FWHM^2 = kE + FWHM_{noise}^2$$

- FWHM of Mn Kα, about 130eV
- Low energy resolution: FWHM of F Kα
- Low energy resolution: FWHM of C Kα

XEDS: Artifacts







Si "escape peak"

- 1.74keV bellow true characteristic peak position

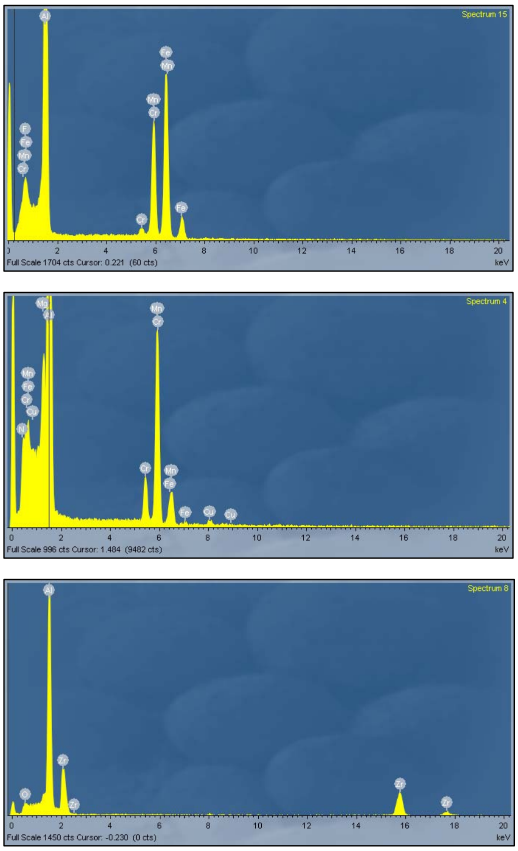
The sum peak

- too high count rate

The internal fluorescence peak

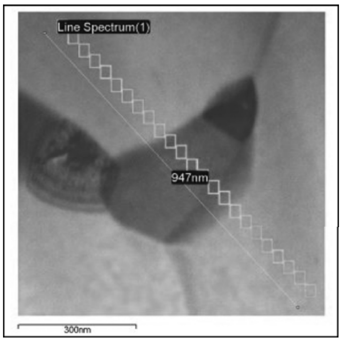
- A small Si Kα peak at 1.74keV

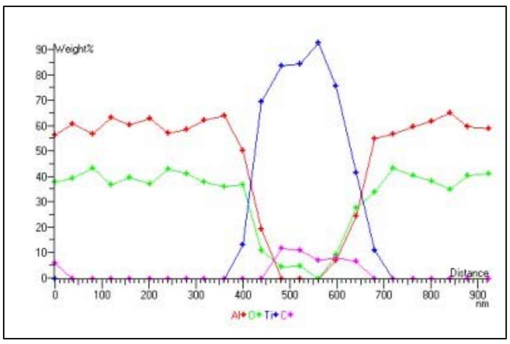
EDS: Qualitative Analysis

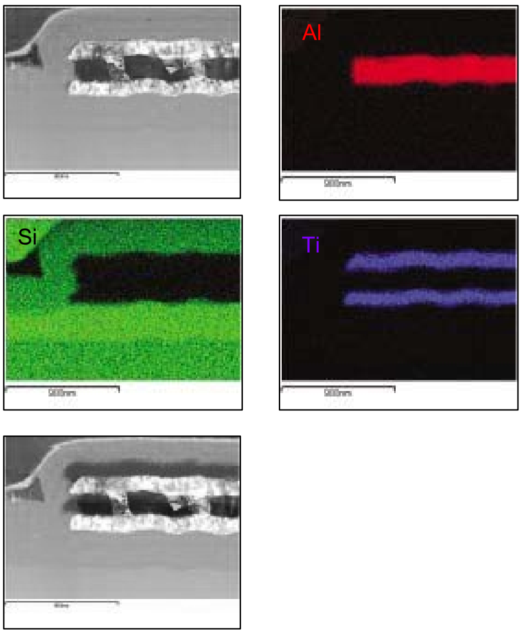


- Spectrum : peak identification
  - Families of peaks
  - Peak overlaps / deconvolution
  - Artifacts & spurious peaks
  - Background subtraction
- Count rate & collection time
- Limits:
  - Probe size
  - Spatial resolution
  - Energy resolution
  - Detectability limit

EDS: Qualitative Analysis







- Profile (line spectrum)
- Elemental mapping

EDS: Quantitative Analysis for SEM

- Acqusition under best conditions
  - Flat specimen, avoid coating, homogeneous specimen (at list in the analysis/interaction volume)
  - High count rate, but optimum dead time
  - Optimum overvoltage
- Standards : ci/cstd=Ii/Istd=ki
- Matrix correction ZAF
  - Z : effect of R & S
  - A : absorption
  - F: secondary fluorosence
- Recalculate concentrations

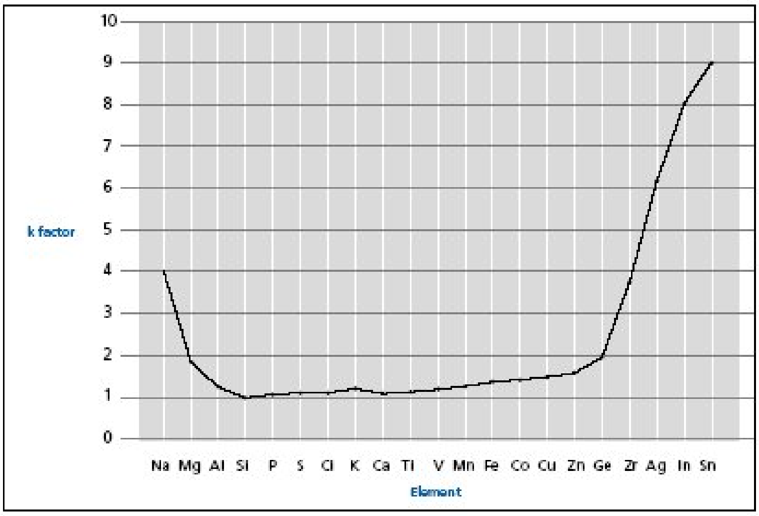
- Other corrections: PROZA, PaP, XPP
- Standardless analysis

EDS: Quantitative Analysis for TEM

$$C_A/C_B = K_{AB}.I_A / I_B$$

$C_{A,B}$ - concentration in wt%
$I_{A,B}$ - peak intensity
K_{AB} - Cliff-Lorimer factor

$$k_{AB} = k_{AR} / k_{BR}$$

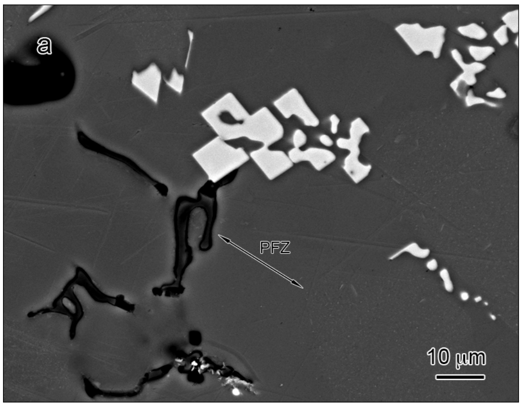


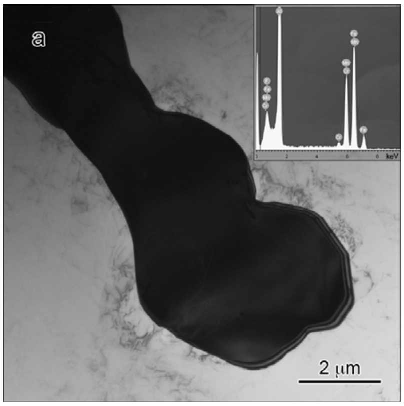
Typical k ASi curve for Kα lines for a Be window detector (after P. J. Sheridan [1989])

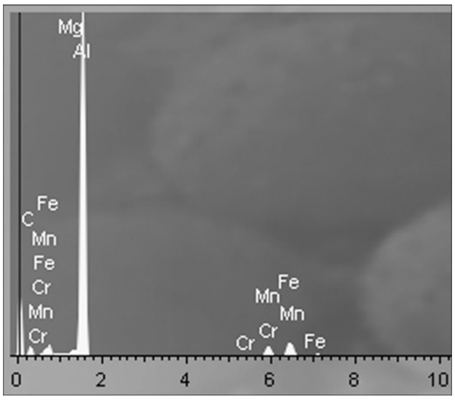
$$k_{AR} = A_A\, w_R\, Q_B\, a_R\, e_R / A_R\, w_A\, Q_A\, a_A\, e_A$$

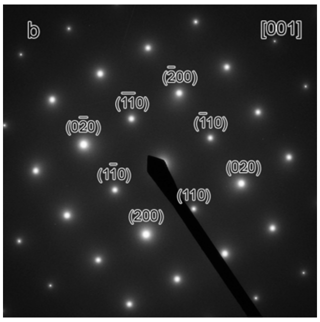
A = atomic weight
w = fluorescent yield
Q = ionisation cross section
a = the fraction of the total line, e.g. Kα / (Kα +Kβ) for a Ka line
e = the absorption due to the detector window at that line energy.

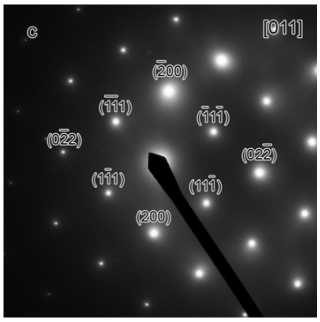
SEM - EDS - TEM











NFMC Spring School on Electron Microscopy, April 2011

SEM - EDS - TEM

STATE	Grain size, µm	$Al_6(Fe,Mn)$ area fraction, %	Al-Mg-Si based particles, area fraction, %	Precipitates free zoneÕs width, µm
As-cast	384±24	3.41±0.9	1.78±0.66	15.47±6.68
1-stage homogenization				
2-stage homogenization	549±50	1.95±0.64	0.33±0.25	17.84±8.85
3-stage homogenization	566±50	3.44±1.24	0.93±0.48	15.91±7.36

TEM Specimen Preparation in Materials Science

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Why is the specimen preparation that important?

No good specimen - No good TEM!

- Microscopic - vs - macroscopic
- No simple rules for various materials & purposes
- Artifacts
- Arts -vs- Science ?

Basic requirements

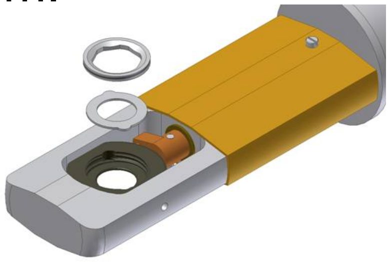
- Representative of the material under investigation
- Artifact free
- Clean, without contamination
- Mechanically rigid and stable
- Resistant to the electron beam irradiation
- Electrically conductive
- Large area transparent to the electron beam

Principals for a TEM specimen preparation

- **Safety**
- Time-effective
- Cost-effective
- Repeatable

Size of a TEM specimen

- Diameter: 3mm, 2.3mm or even 1mm!
  - Reduce size of a bulk specimen.
  - Use grid support for small specimens.



- Thickness: 10-200nm
  - Material (chemical composition)
  - Kind of the experiment (HREM-vs-CTEM, EELS-vs-EDS)

Choice of a specimen preparation technique

Specimen
-self-supported (bulk)
-supported by a grid (thin films, nano-particles ect)

Material:
-ductile (metals)
-hard & brittle (semiconductors & ceramics)

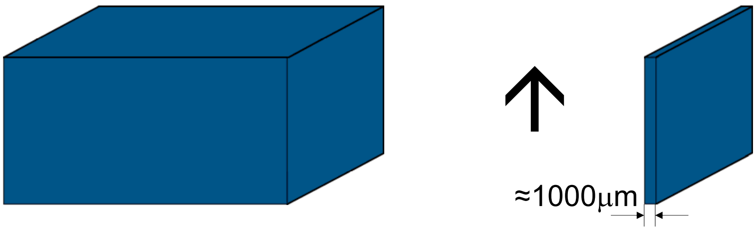
Geometry:
-plan view
-cross-section

Electrical properties:
-conductive
-insulating

Types of specimen preparation techniques

Mechanical	• Mechanical polishing to the electron transparency (Tripod) • Cleavage • Ultramicrotomy • Crushing
Mechanical+ionic	• Grinding, dimpling+ion milling
Ionic	• FIB
Chemical	• Electro-chemical polishing • Chemical polishing or etching
Physical	• Replica • Thin film deposition

Step 1: Slicing the specimen



<u>Ductile materials</u> (metals)

➢Cut slice 500-1000 μm thick:
- chemical wire/string saw
- **wafering saw (not diamond!!!)**
- spark erosion

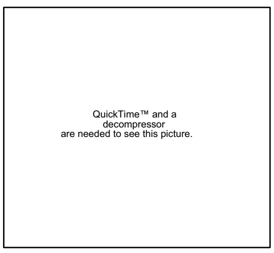
<u>Brittle materials</u> (semiconductors, ceramics…)

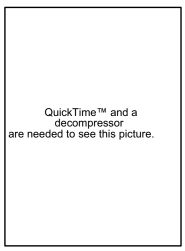
➢Cut slice ≈1000 μm thick:
- **diamond wafering saw**

➢Cleave (NaCl, MgO…)

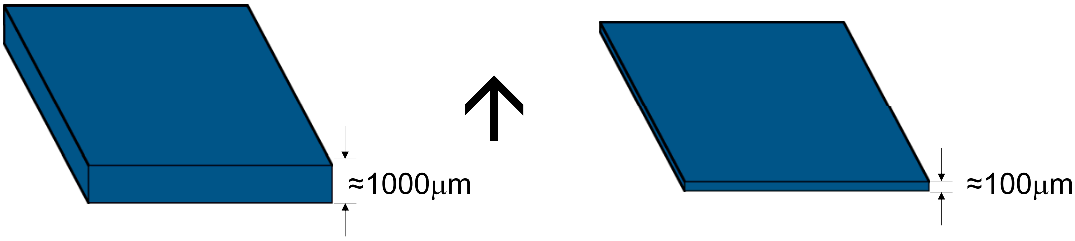
➢Ultramicrotome







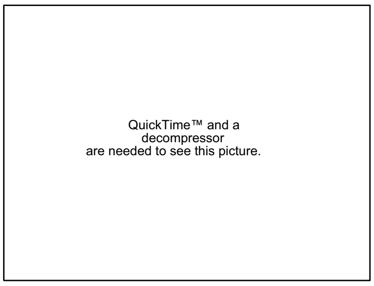
Step 2: Grinding

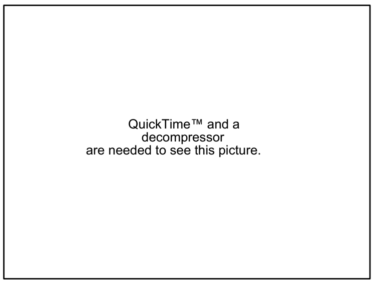


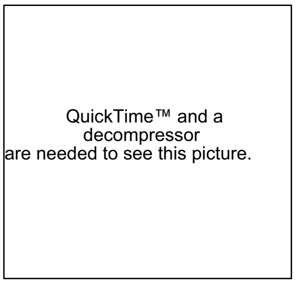
- Mechanically grind down the specimen to about 100μm in thickness.

  Ductile materials: manually

  Hard materials: grinding/polishing machines







Step 3: Cutting 3mm disk

Ductile materials (metals)

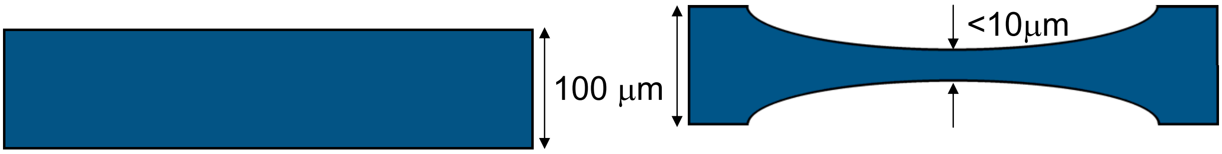
- Mechanical disc punch

Brittle materials (semiconductors, ceramics…)

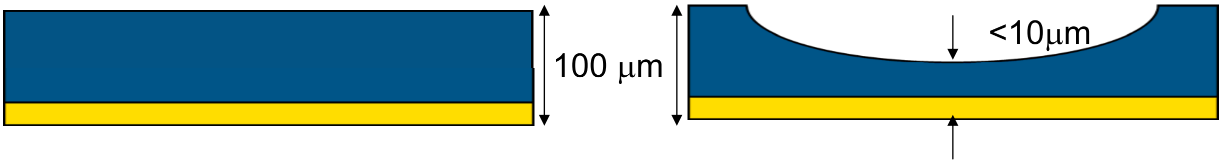
- Ultrasonic drill
- Grinding (slurry) drill
- Spark erosion (for conducting specimens)

Step 4: Prethinning the disk - dimpling

- Plan view & cross-section specimens

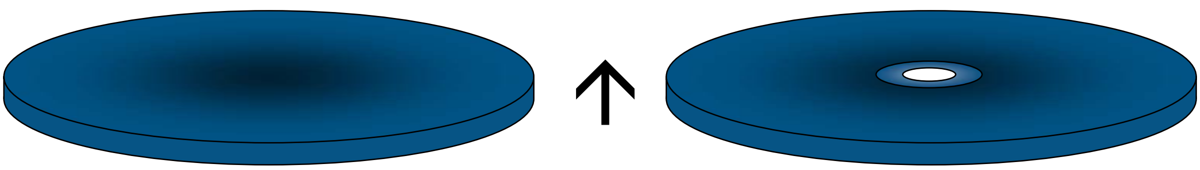


- Plan view: thin film on the substrate



QuickTime™ and a decompressor are needed to see this picture.

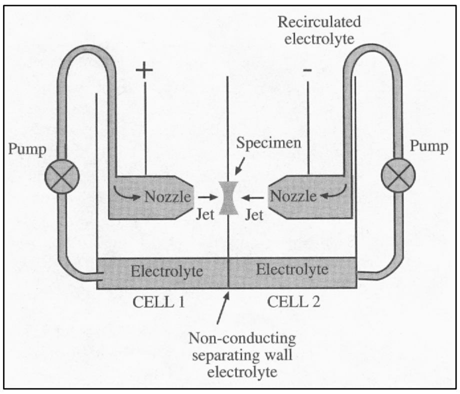
Step 5: Final thinning of the disk



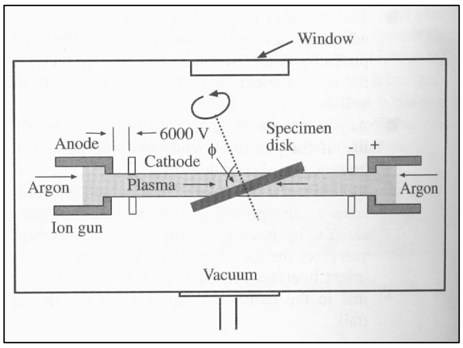
- Electropolishing
- Ion Milling

Electropolishing

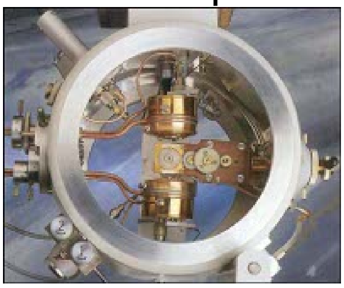
- Only for electrically conducting specimens



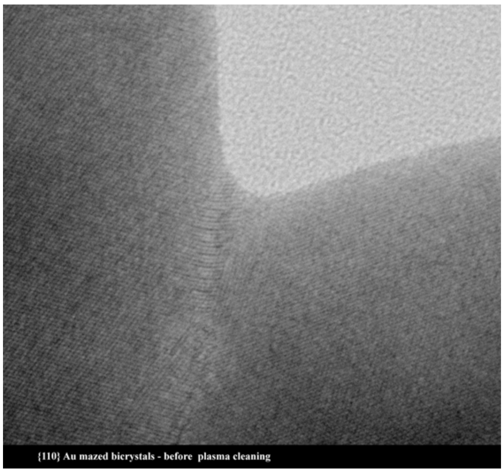
Ion Milling



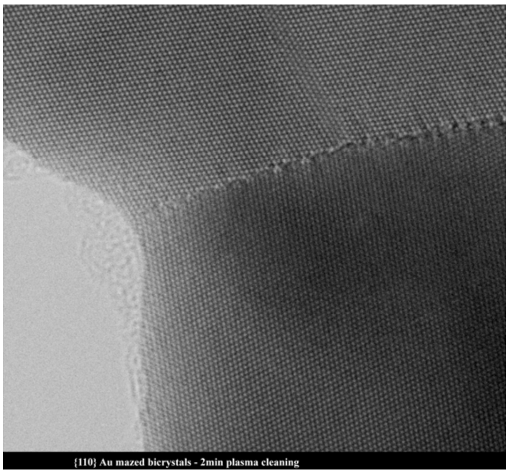
- Ion milling involves bombarding a thin TEM specimen with energetic ions (Ar^+) and sputtering material from the specimen until it is thin enough to be electron transparent.



Plasma Cleaning



before



2min cleaning

Preparation of a cross-section specimen

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General tools & equipment

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