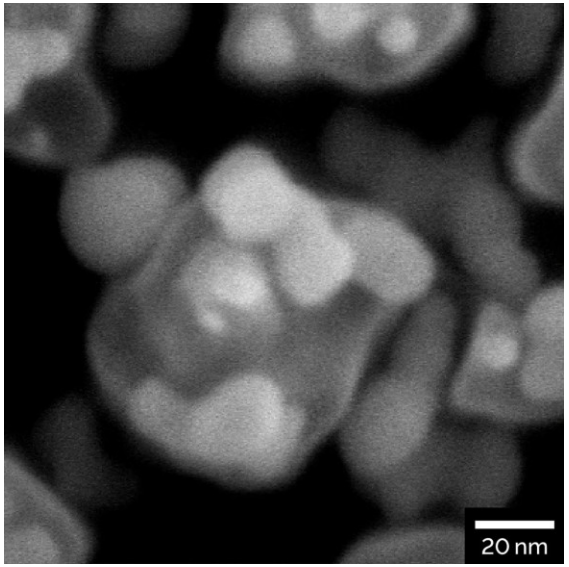
Electron microscopy and microanalysis

Radek Škoda
Institute of Geological Sciences, Faculty of Science
Masaryk University

Why an electron microscope?

- Optical microscope uses light and optical lenses
  - Magnification limited by wavelength of light 400-600 nm
  - Image observed directly by eyes

- Electron microscope uses a beam of accelerated electrons and electromagnetic lenses
  - Wavelength of accelerated electrons is down to 6 pm
  - Scanning electron microscope - magnification up to 300 000 x
  - Transmission electron microscope magnification up to 1 500 000 x
  - image not observed directly, but using detectors, electronic and screens
  - Interaction of accelerated electrons with a sample causes a production of different kinds of radiation and particles
    - their detection is used for better characterization of the sample



Basic terms

- Electron microscope
  - Transmission electron microscope (TEM, HRTEM)
    - Electron beam penetrates through the sample, the signal is detected below the sample
    - Magnification up to 1 500 000 x.
    - Individual atoms can be seen (HRTEM)
  - Scanning electron microscope (SEM, REM)
    - Electron beam is moving on the sample surface
    - Magnification 3x-300 000x
- Electron microanalysis
  - Method of chemical analysis based on detection of characteristic X-ray generated by interaction of accelerated electrons with a sample
  - Requires very small sample area
- Electron microprobe
  - Electron microscope specially designed to microanalysis.

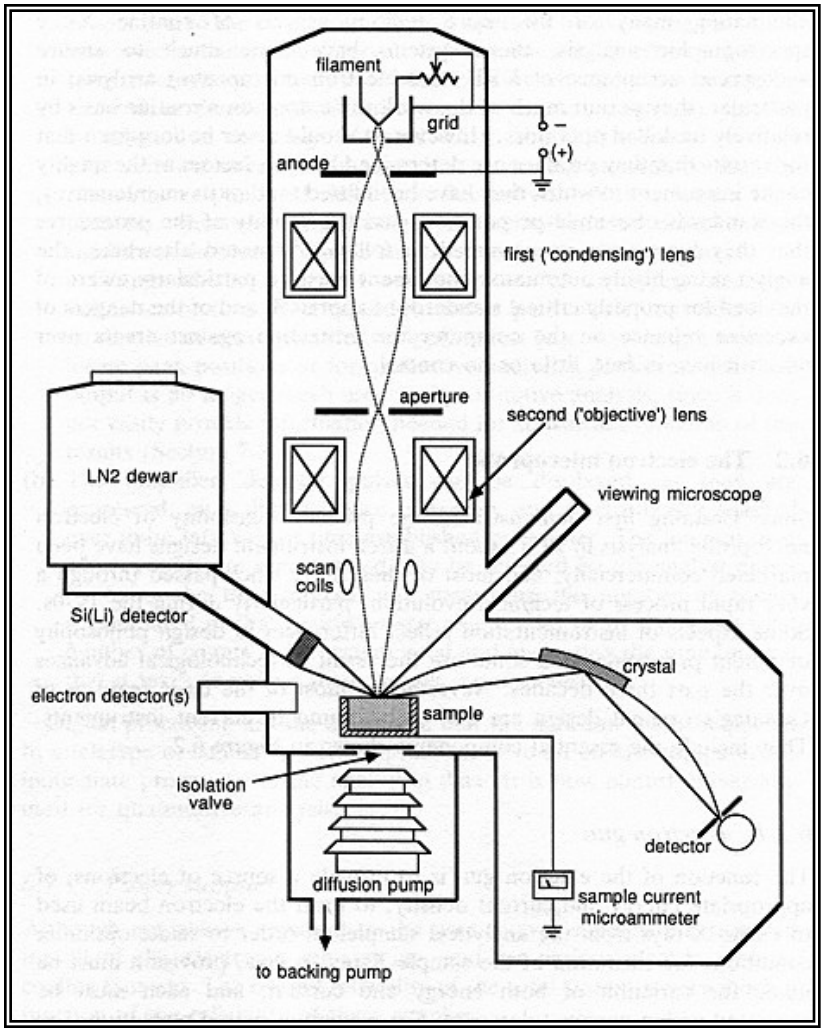
SEM –Tescan Magna

TEM Jeol JEM-2100

Electron microprobe CAMECA SXFive

Scanning electron microscope

- Vacuum system
- Electron gun
- Electron optic
- Chambre for samples
  - Motorized stage X,Y,Z,R,T
- detectors
  - BSE, SE, CL, EBSD, EDS, WDS, …

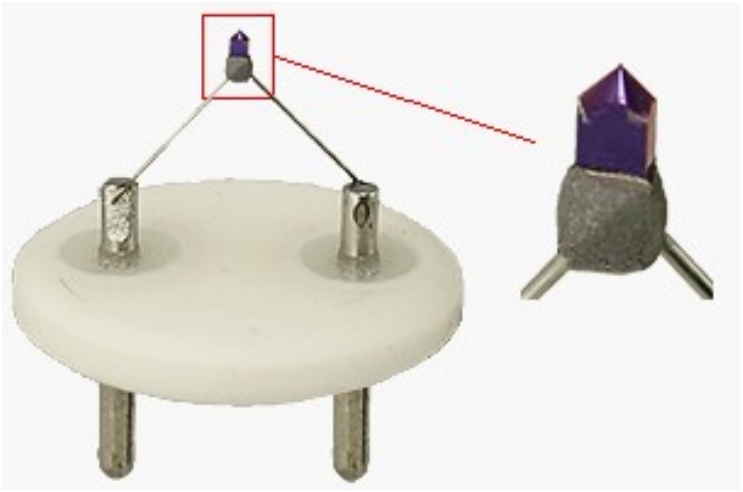


Electron gun

- Produces electrons in a form of electron beam
- Electrons escapes from filament when gained a cerain energy
- 3 types of electron sources
  - Thermionic sources
  - „field emission“ sources
  - „thermal-field“ sources

termionic sources

- Thermoemission – energy given in a form of a heat
  - Tungsten filament
  - LaB_6 crystal



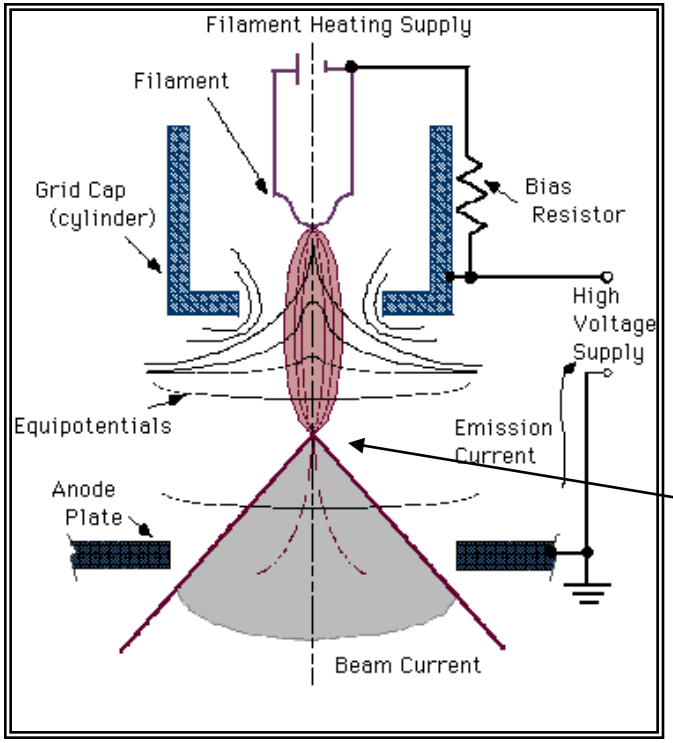
work function for individual elements

Element	eV	Element	eV	Element	eV	Element	eV	Element	eV
Ag:	4.52 – 4.74	Al:	4.06 – 4.26	As:	3.75	Au:	5.1 – 5.47	B:	~4.45
Ba:	2.52 – 2.7	Be:	4.98	Bi:	4.31	C:	~5	Ca:	2.87
Cd:	4.08	Ce:	2.9	Co:	5	Cr:	4.5	Cs:	2.14
Cu:	4.53 – 5.10	Eu:	2.5	Fe:	4.67 – 4.81	Ga:	4.32	Gd:	2.9
Hf:	3.9	Hg:	4.475	In:	4.09	Ir:	5.00 – 5.67	K:	2.29
La:	3.5	Li:	2.93	Lu:	~3.3	Mg:	3.66	Mn:	4.1
Mo:	4.36 – 4.95	Na:	2.36	Nb:	3.95 – 4.87	Nd:	3.2	Ni:	5.04 – 5.35
Os:	5.93	Pb:	4.25	Pd:	5.22 – 5.6	Pt:	5.12 – 5.93	Rb:	2.261
Re:	4.72	Rh:	4.98	Ru:	4.71	Sb:	4.55 – 4.7	Sc:	3.5
Se:	5.9	Si:	4.60 – 4.85	Sm:	2.7	Sn:	4.42	Sr:	~2.59
Ta:	4.00 – 4.80	Tb:	3	Te:	4.95	Th:	3.4	Ti:	4.33
Tl:	~3.84	U:	3.63 – 3.90	V:	4.3	**W:**	**4.32 – 5.22**	Y:	3.1
Yb:	2.6	Zn:	3.63 – 4.9	Zr:	4.05	**LaB6**	**2.5**	CeB6	2.5

http://en.wikipedia.org/wiki/Work_function

tungsten filament

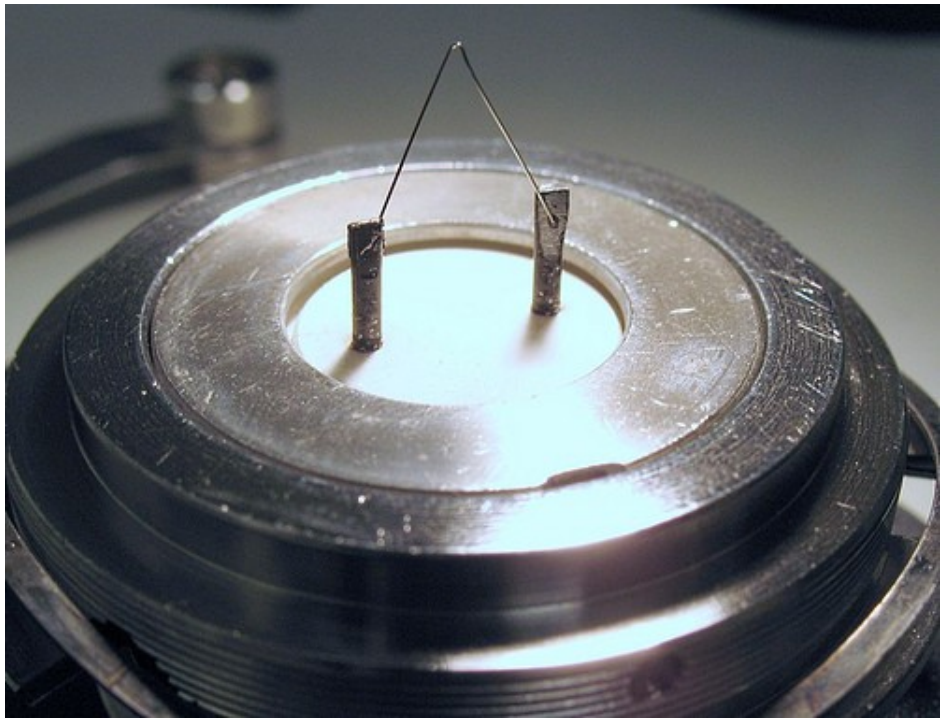
- bended W wire 100-150 um in diameter
- Heated up to 2700 K
- lifetime 100-1000 hours



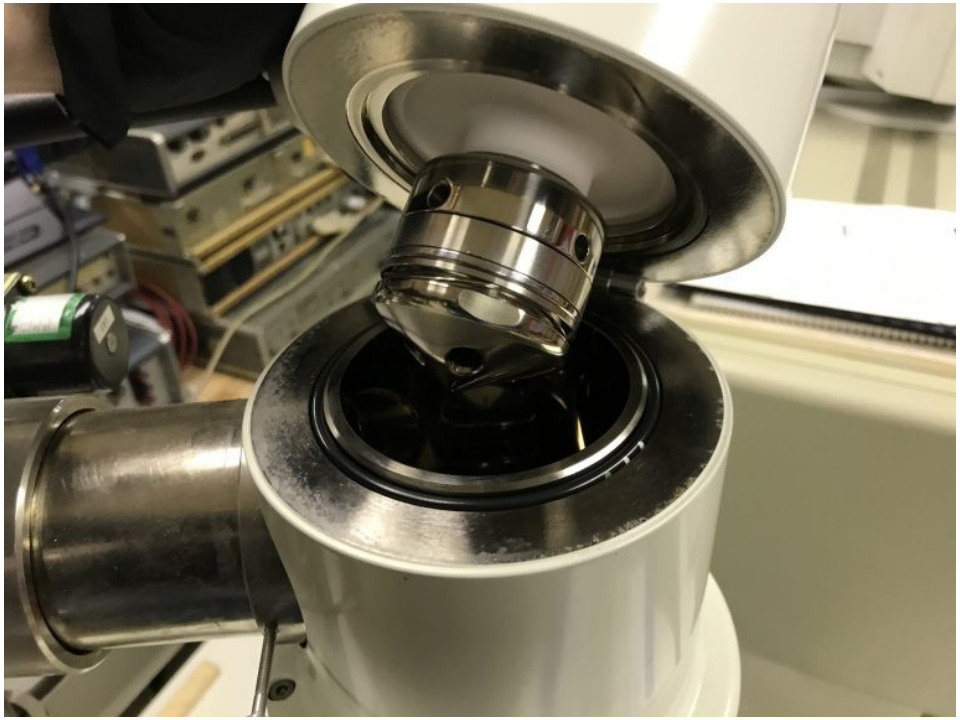
- Heating – production of slow electrons
- wehnelt cylinder – bias between filament and cylinder – X00 V
  - shaping of a thermal electrons flow - electrostatic lens
  - Bias influences the emission current
- Accelerating volage between cathode and anode is in the range 0.2-40 KV, usually 10 to 30 kV

focal point (10-100 um) – „effective source“ of electrons

Anode has a hole inside to allow electrons passing through

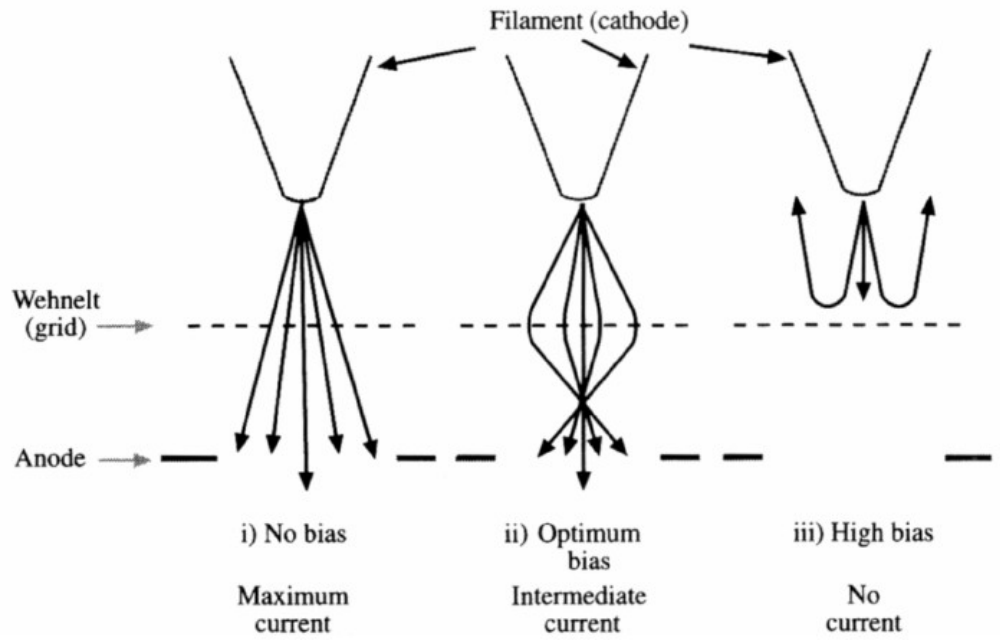


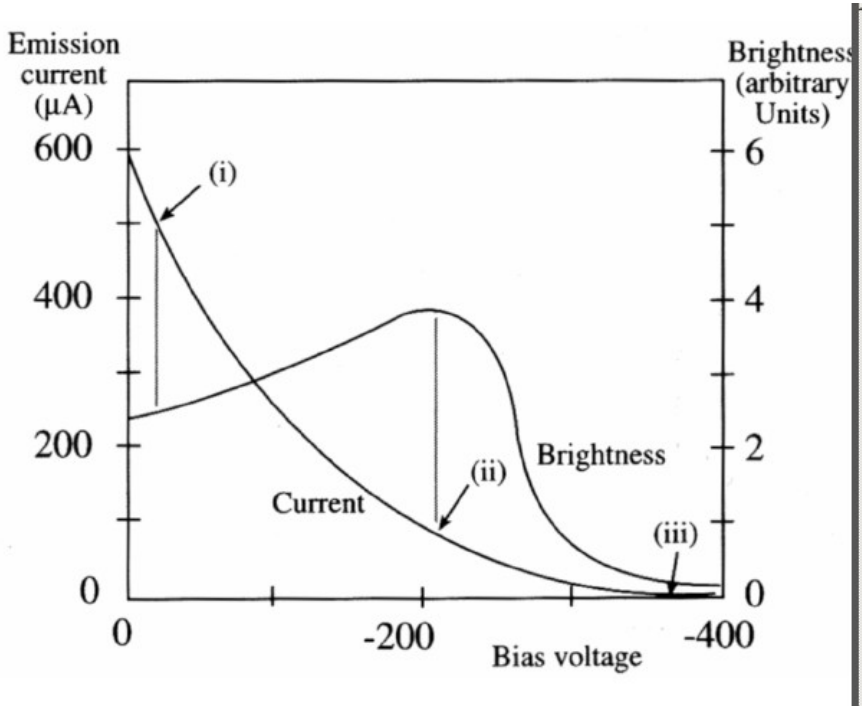
JEOL K-type filament



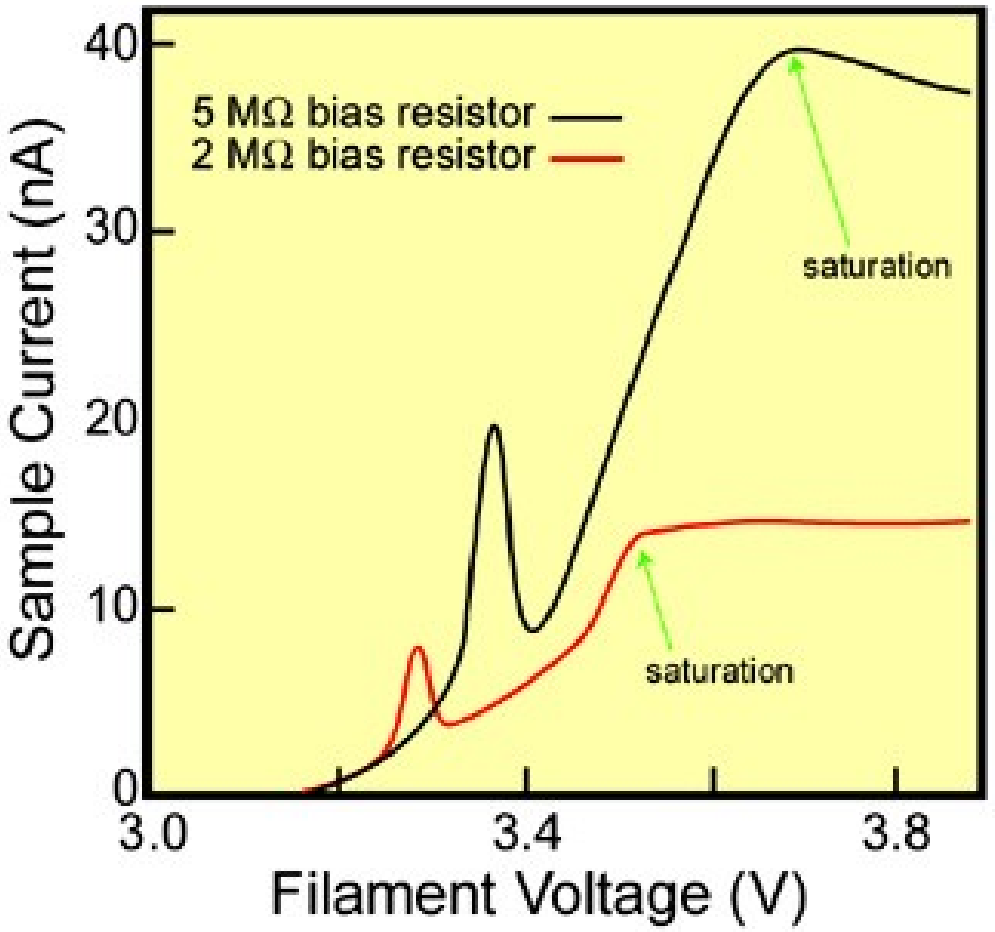
wehnelt cylinder in the gun

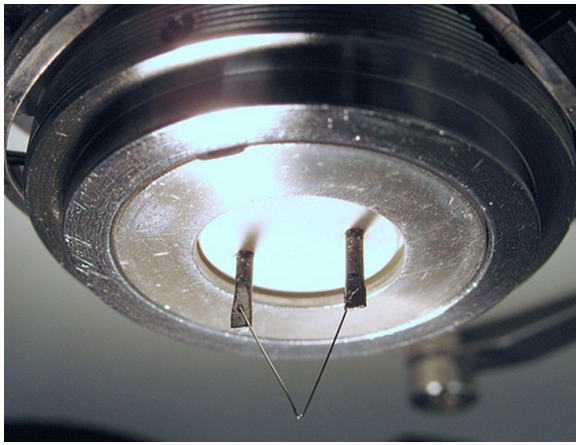
Wenhelt principle and function





Effect of filament heating to production of electrons





LaB_6 source

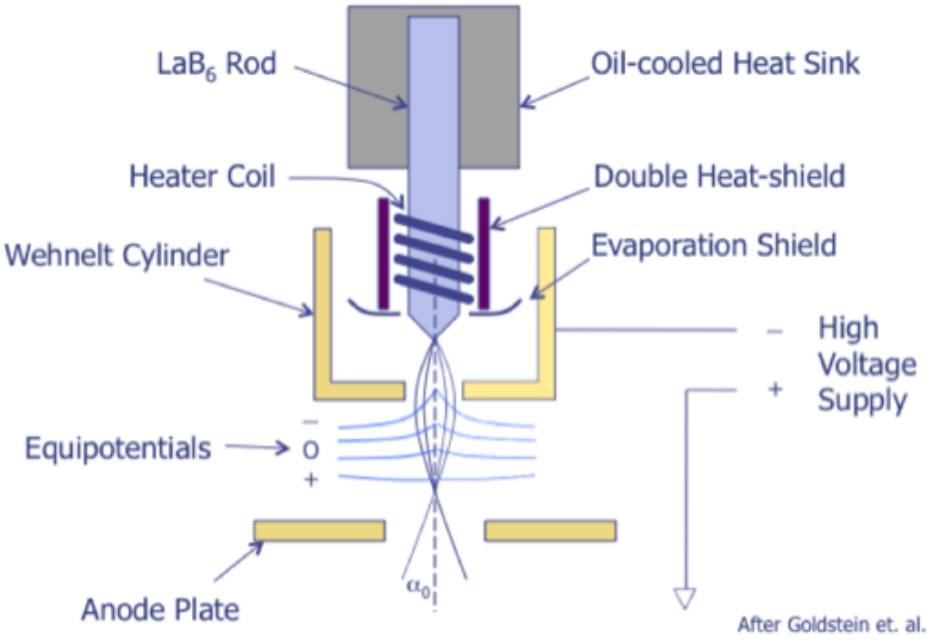
Cathode – lanthanum hexaboride

Ground off to a tip

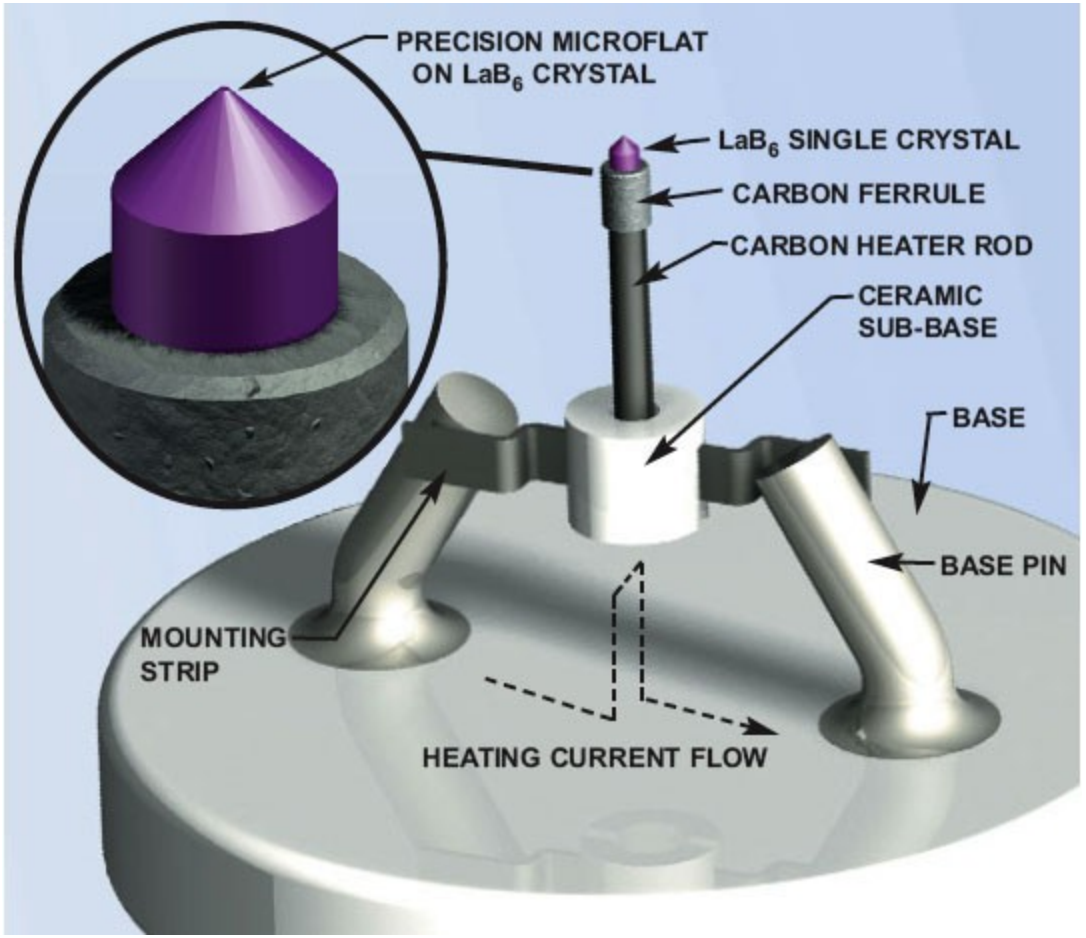
Low „work function“ 2.5 eV

Higher electron flux at lower heating temperature (compare to W filament) = brighter electron image

Longer lifetime (compare to W filament), X months



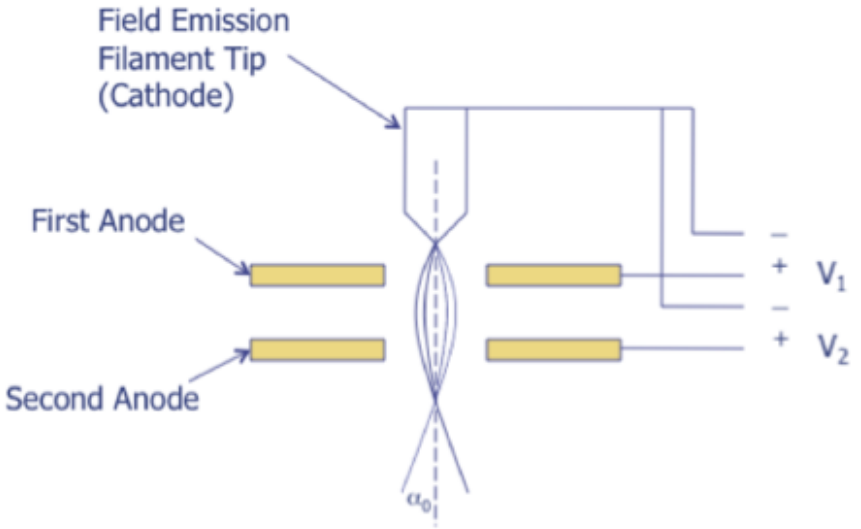
http://www.semitracks.com/index.php/blog/archive-blog-posts



http://www.tedpella.com/apertures-and-filaments_html/Kimball-lab6-cathodes.htm

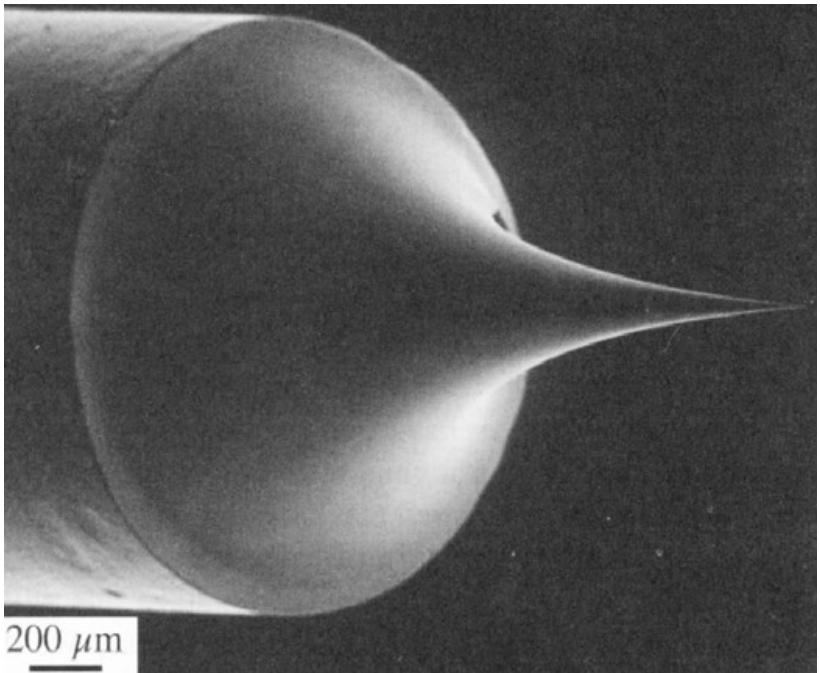
cold Field Emission source

- Emission of electrons by strong electric field > 1 GV/m
- Cathode monocrystal of W, ended by a tiny tip
  - Electrostatic potential depends on E_f – work function of the cathode
  - Requires high vacuum 1.5 10^{-7} Pa or better



First anode – extraction of electrons
Second anode – acceleration/deceleration of electrons

After Goldstein et. al.



Thermal Field Emission source

Field emission from a heated cathode

Shottky-type source

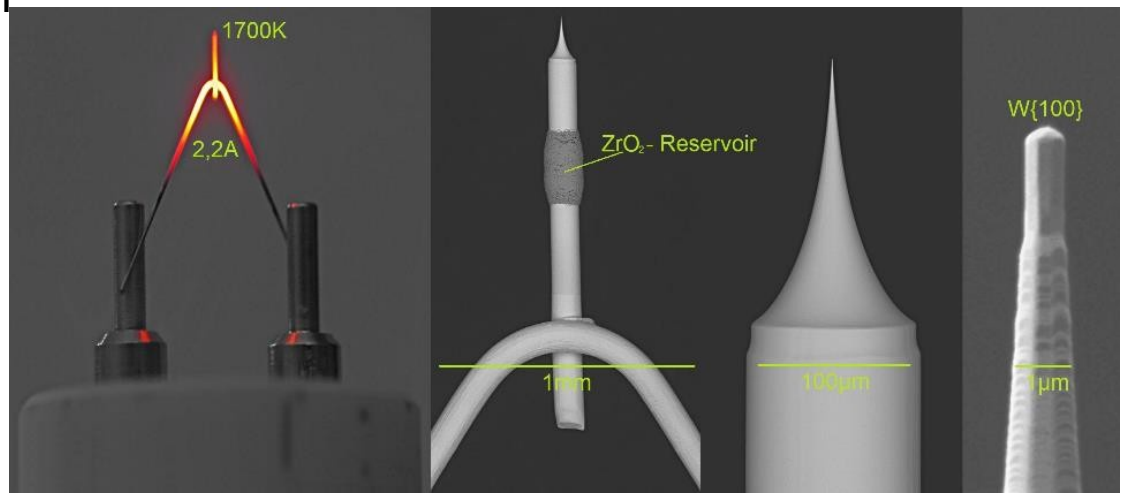
Layer of ZrO_2 on the surface lowers work function

Does not require such high vacuum

Lower electrostatic fields comparing to cold FE

circa 10 x higher electron current flux in comparison to cold FE

Heating to 1000-1800 K lowers the needs of very high electrostatic field

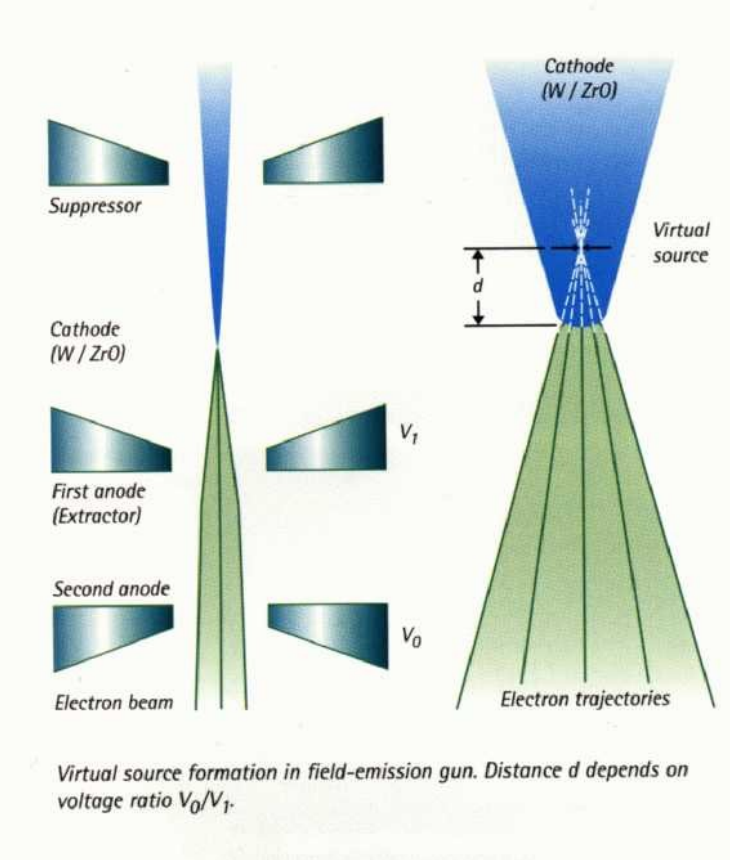


Suppressor – filters out the thermal electrons

First anode – electron extraction

Second anode – electron acceleration

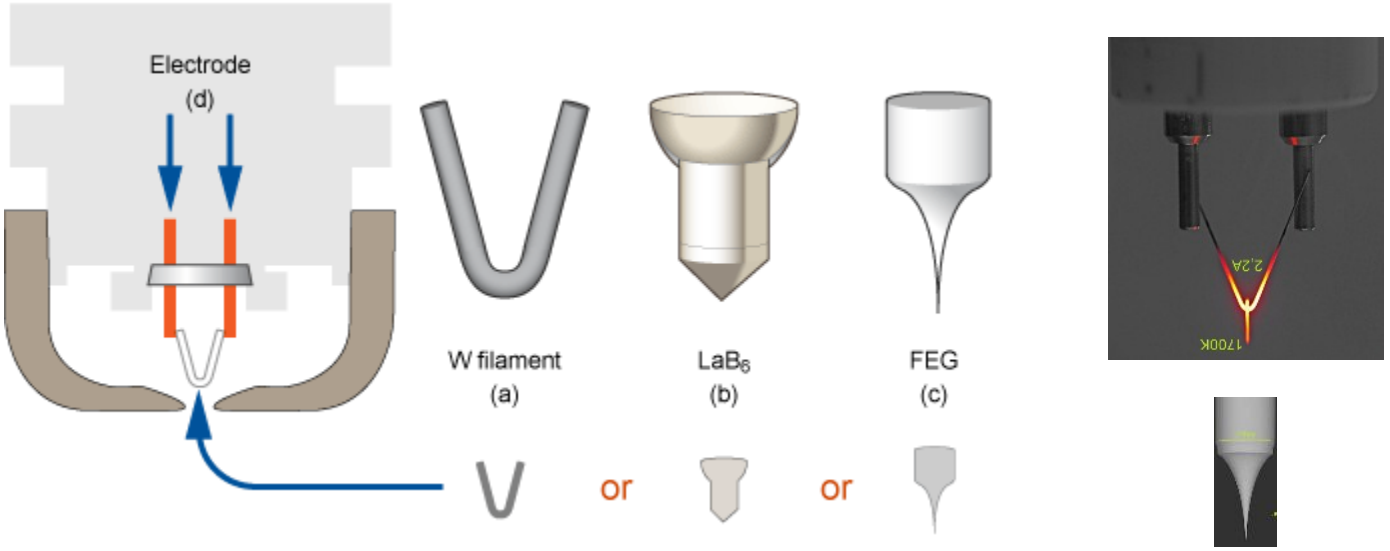
Emission current up to 200 uA



Virtual source formation in field-emission gun. Distance d depends on voltage ratio V_0/V_1.

http://www.nanophys.kth.se/nanophys/facilities/nfl/manual/sem-adjust/semadj2.html

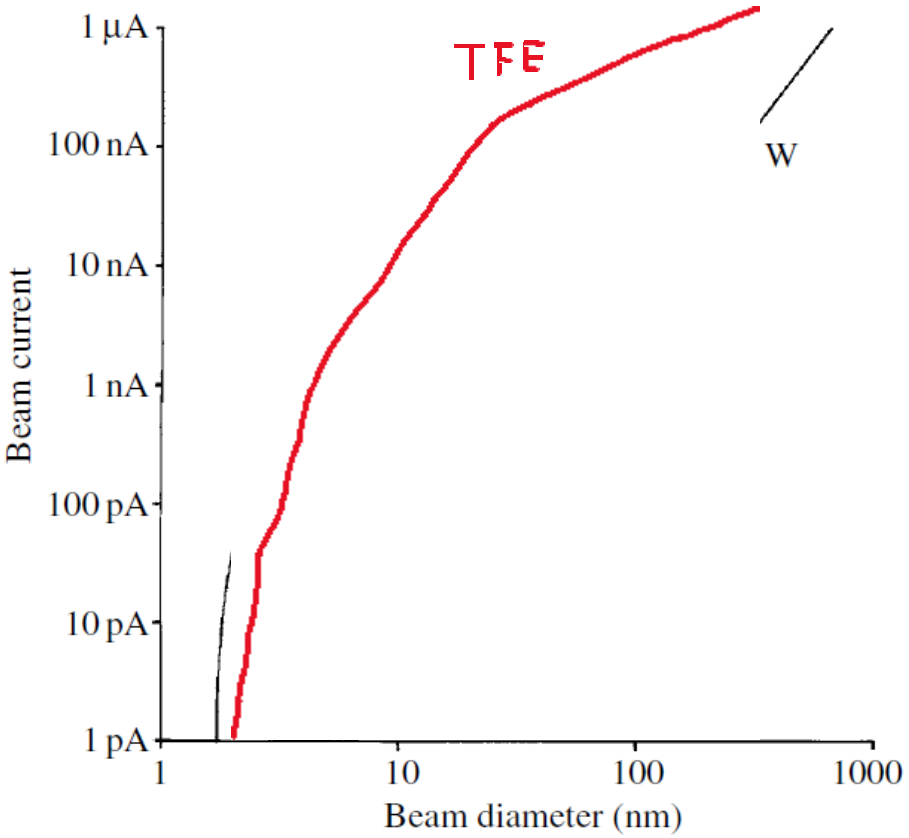
Comparison od W, LaB_6, cold and thermal FEG

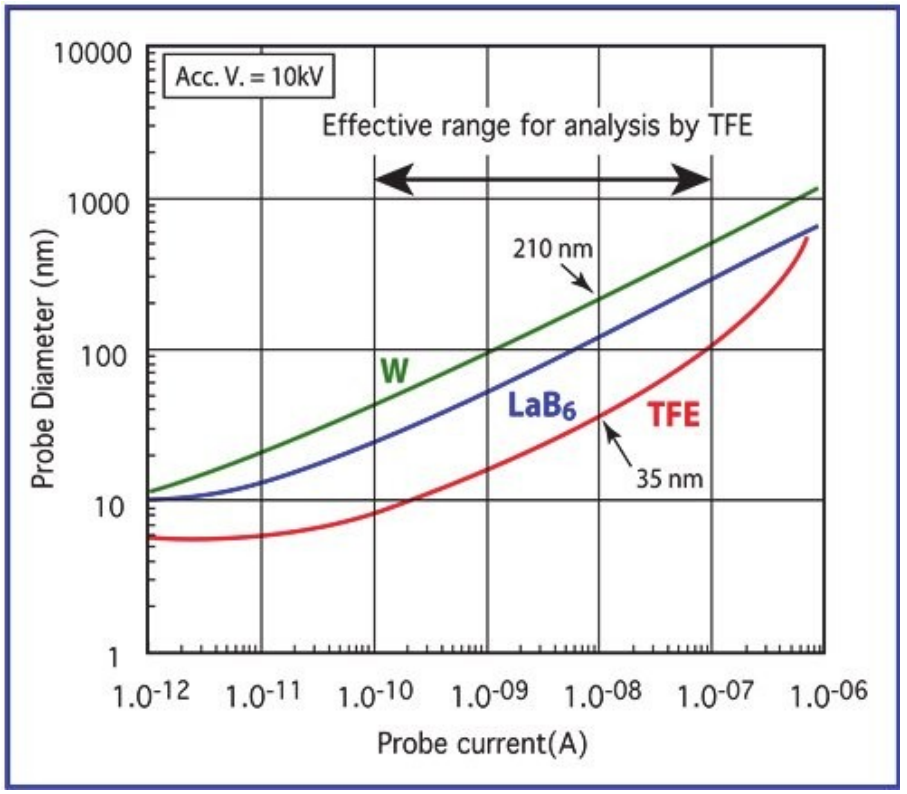


http://www.ammrf.org.au/myscope/sem/practice/principles/gun.php

http://www.ammrf.org.au/myscope/

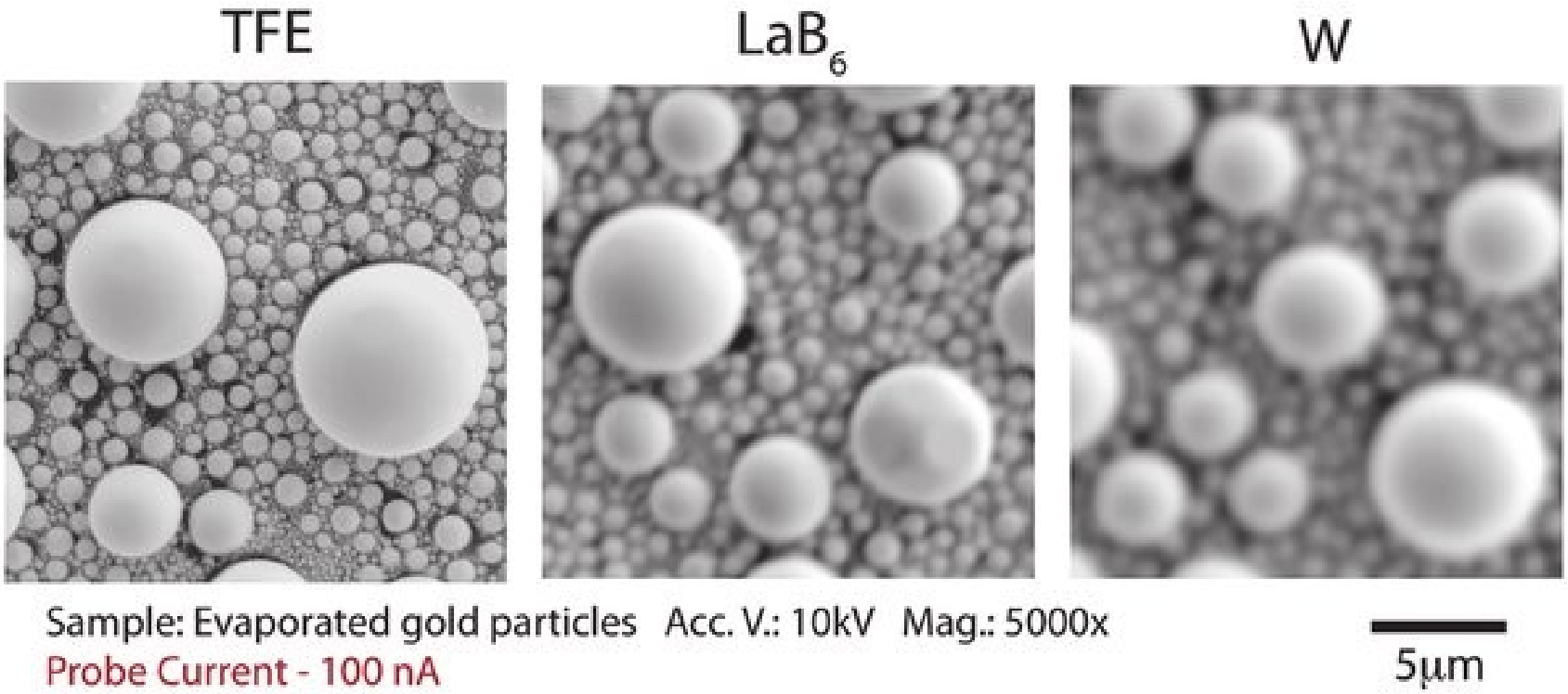
Comparison od W, LaB_6, cold and thermal FEG



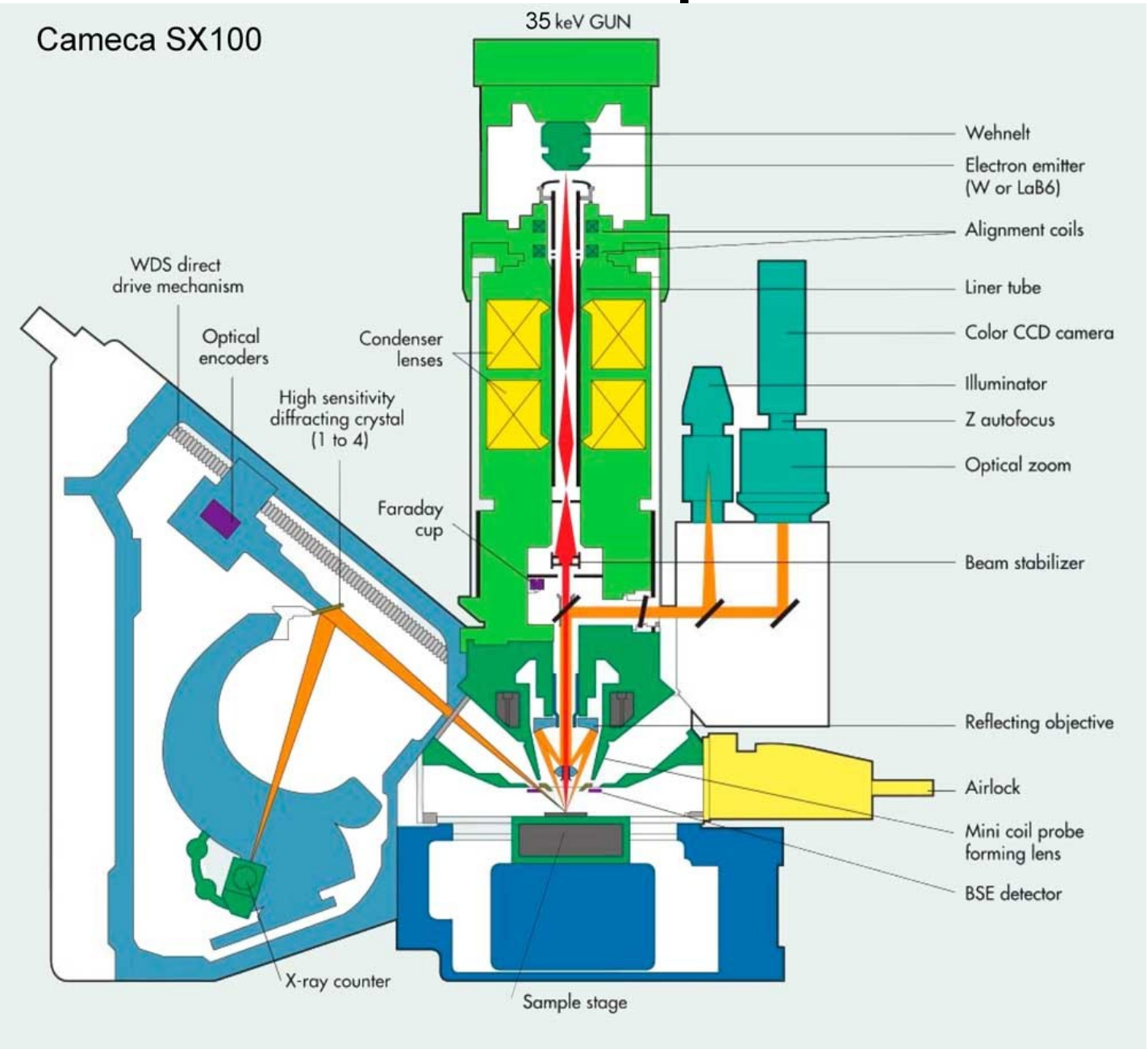


Reed, 2005: Electron Microprobe Analysis and scanning Electron Microscopy in Geology.

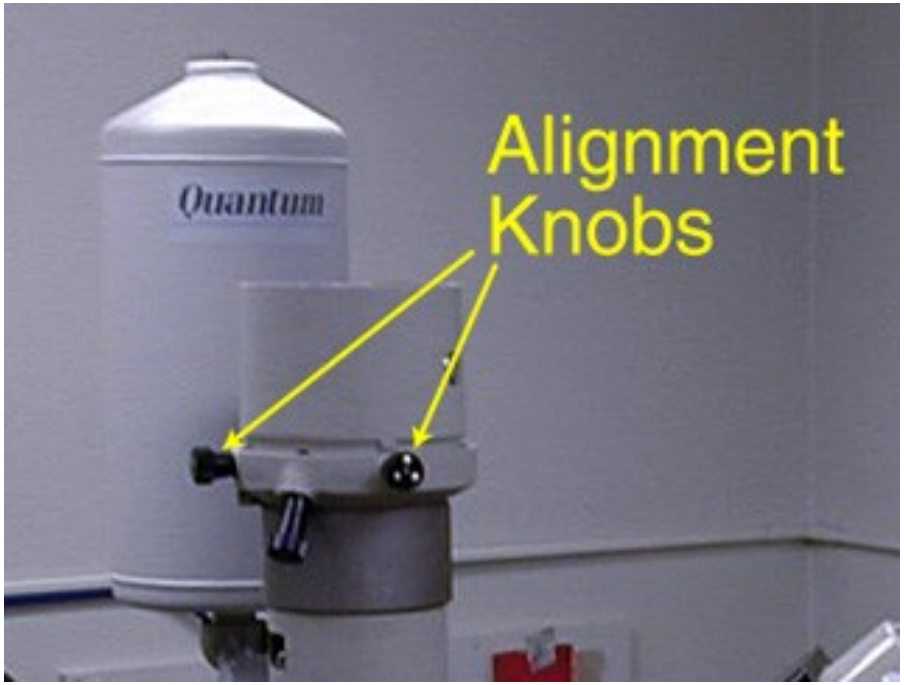
Comparison od W, LaB_6, and thermal FEG

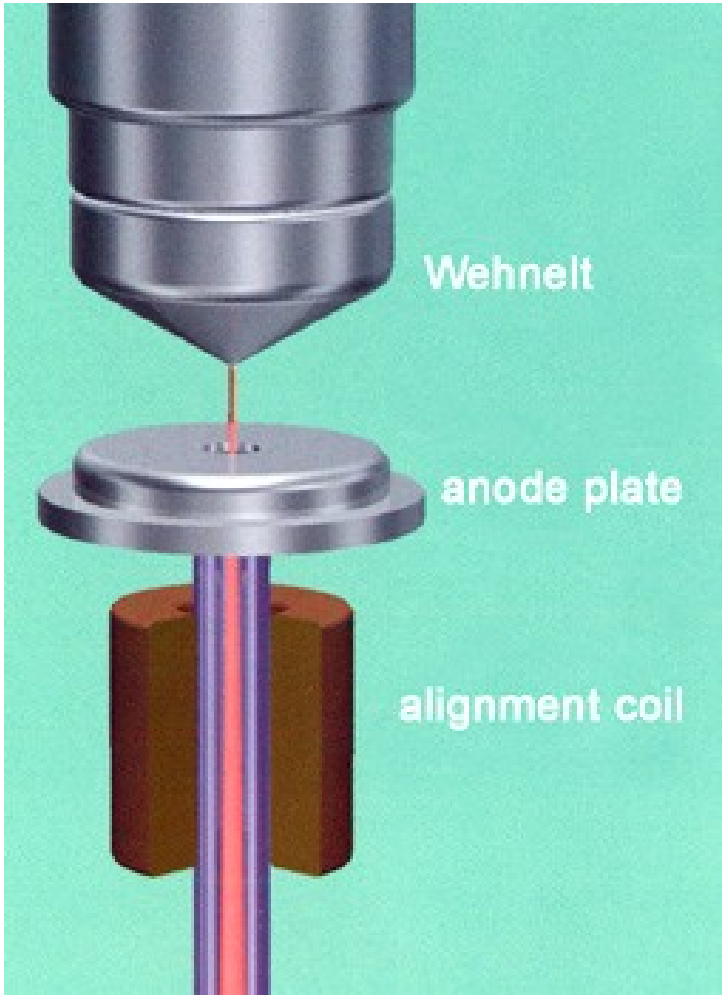


electron optics

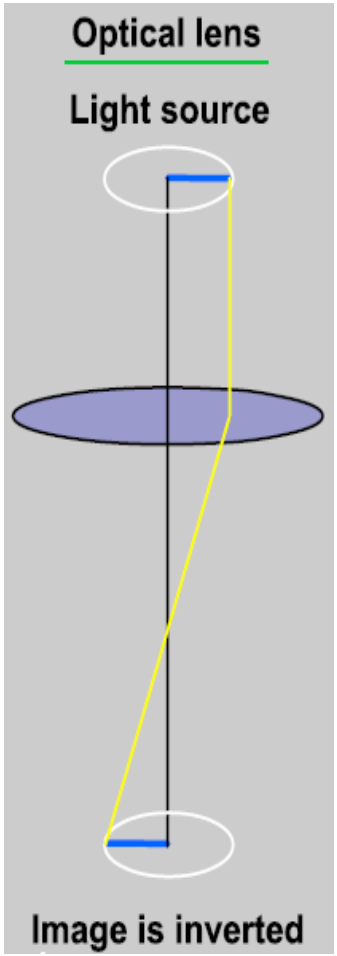


Additional alignment of the electron source/beam





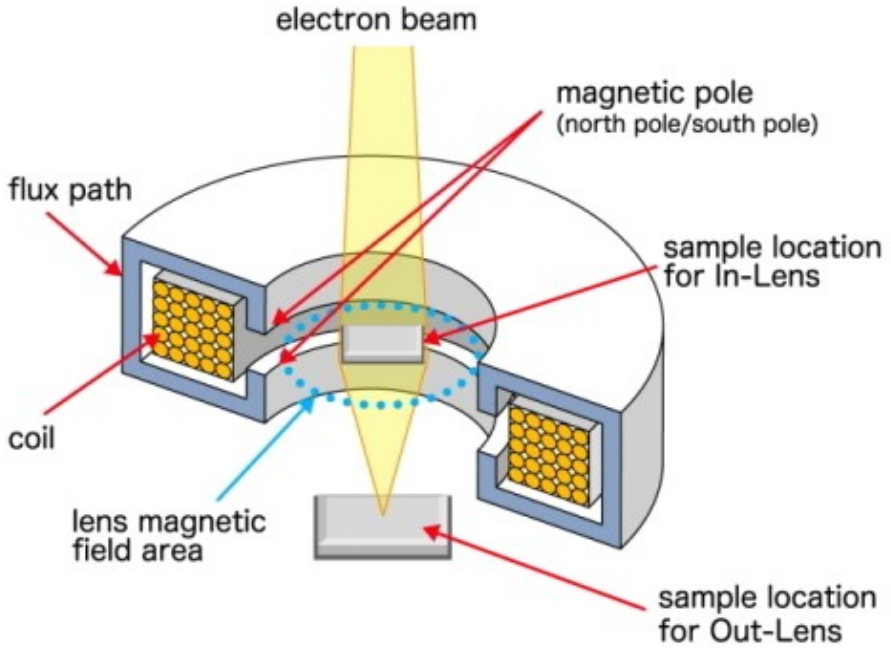
Principle of elmg lenses

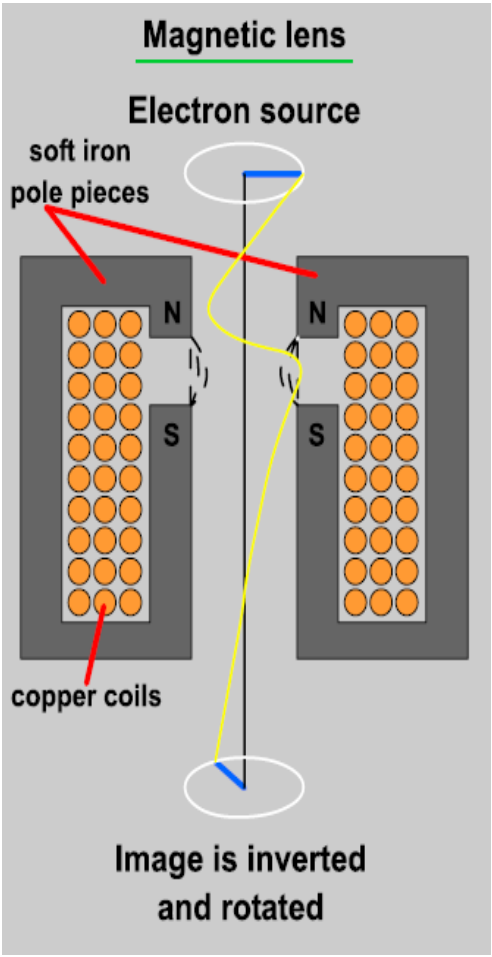


- A charged (q) particle moving through a magnetic field of strength B, with a speed v will feel a Lorentz force (F) with a magnitude of:

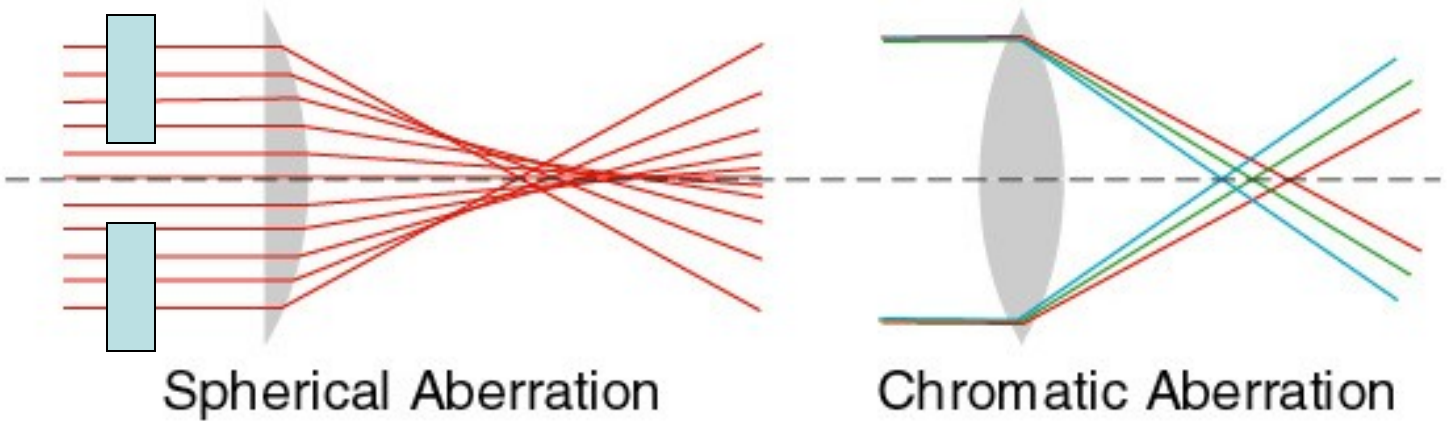
$$\mathbf{F} = q\mathbf{v} \times \mathbf{B}$$

- The force changed the direcion of the movement and not the energy (speed) of the particle.





Defects of elmg. lenses

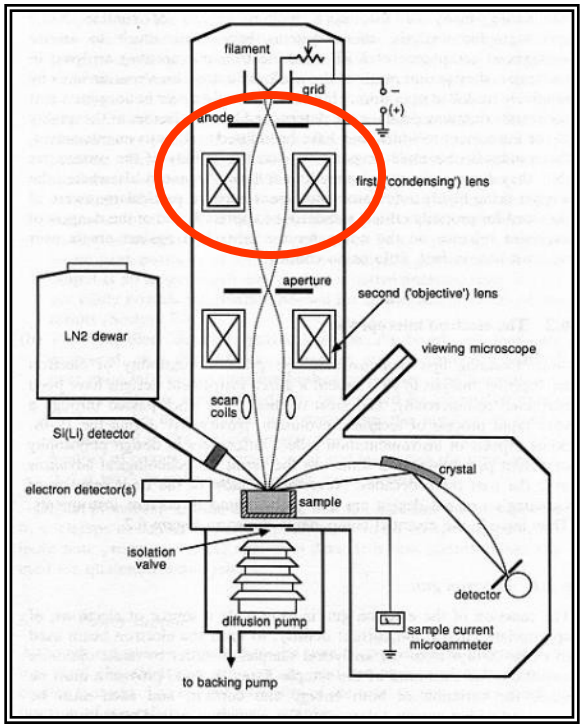


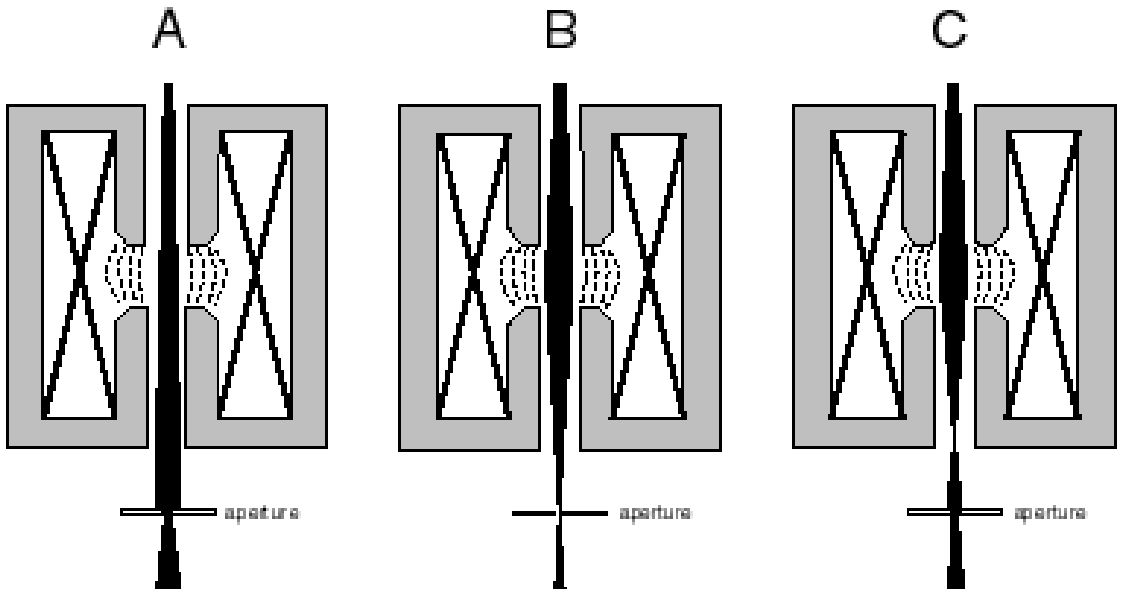
Can be minimized by insertion of the aperture

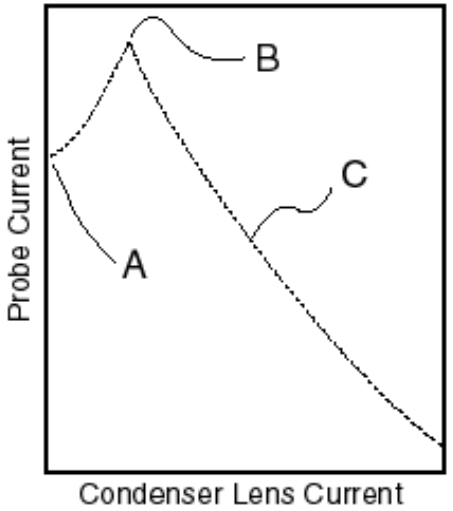
is minimal, bcs the electrons have the same energy

condenser lens

- Electron beam is divergent when passing through anode
- Condenser lens(es) collimates or converges the beam.
- Position of the focal point controls the amount of electrons passing through the aperture
  - Current regulation in range (X0 pA-X00 nA)

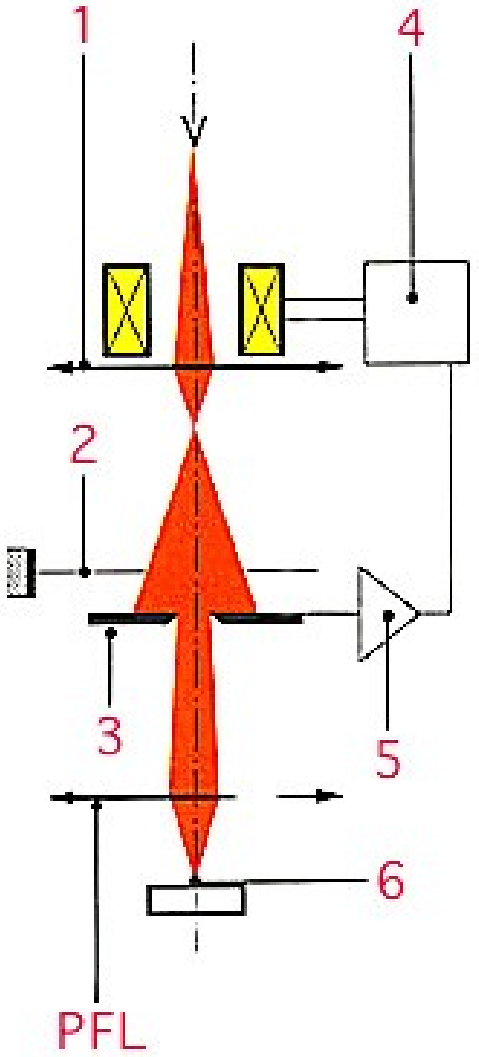






Additional beam current regulator

Fluctuation of the heating current or condenser lens coil current caused little variation of the beam current

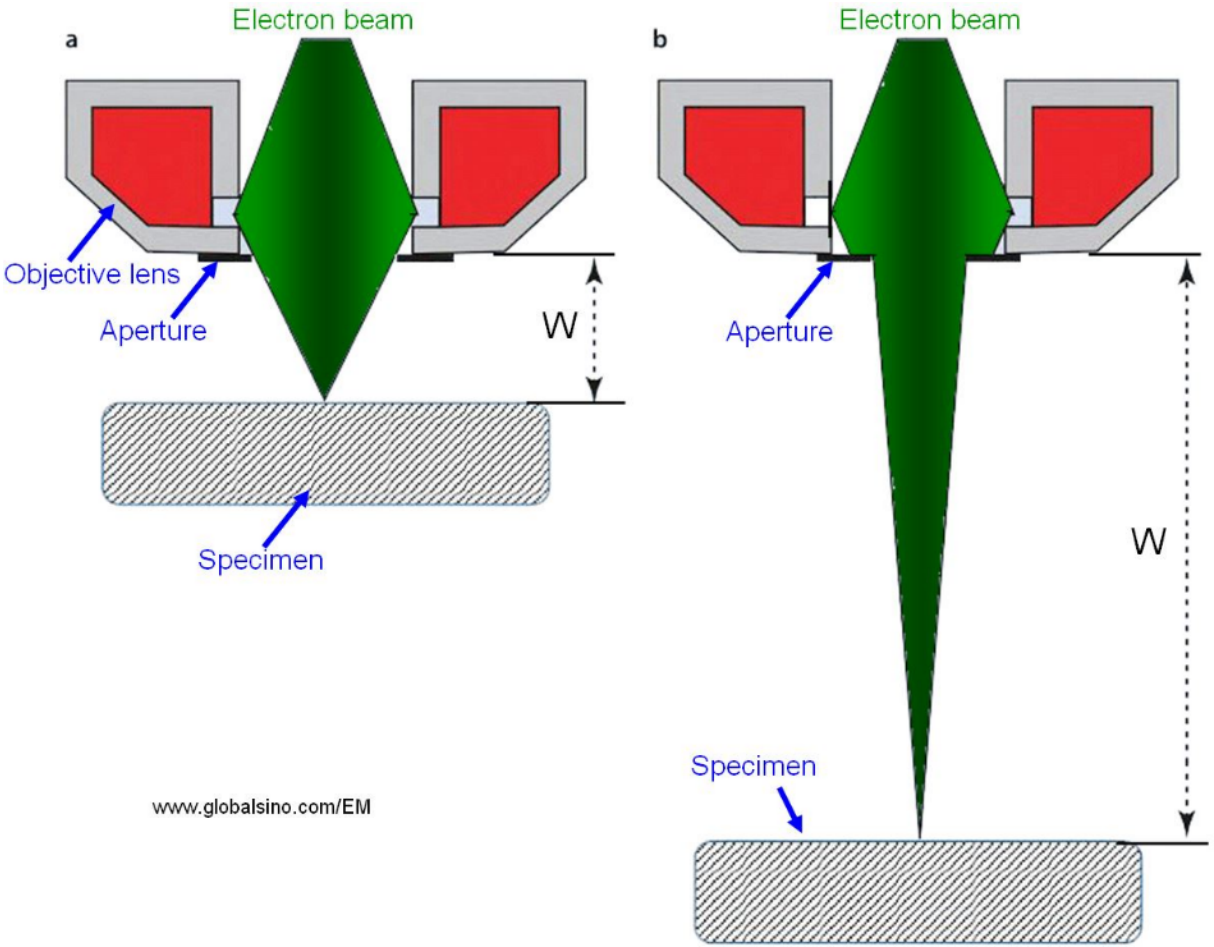


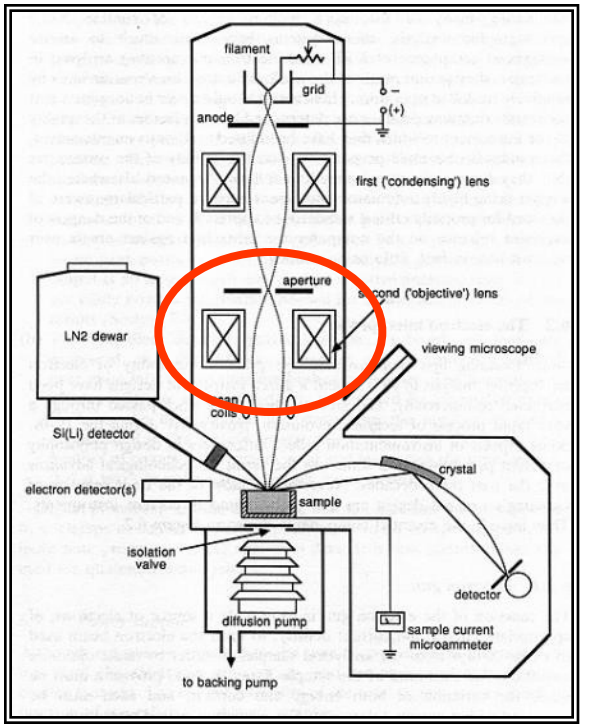
1-condenser lens
PFL-probe-forming lens - objective lens
2-limiting aperture of the regulator
3-collecting aperture of the regulator
4-elm lens power supply
5-amplifier, electronic
6-sample

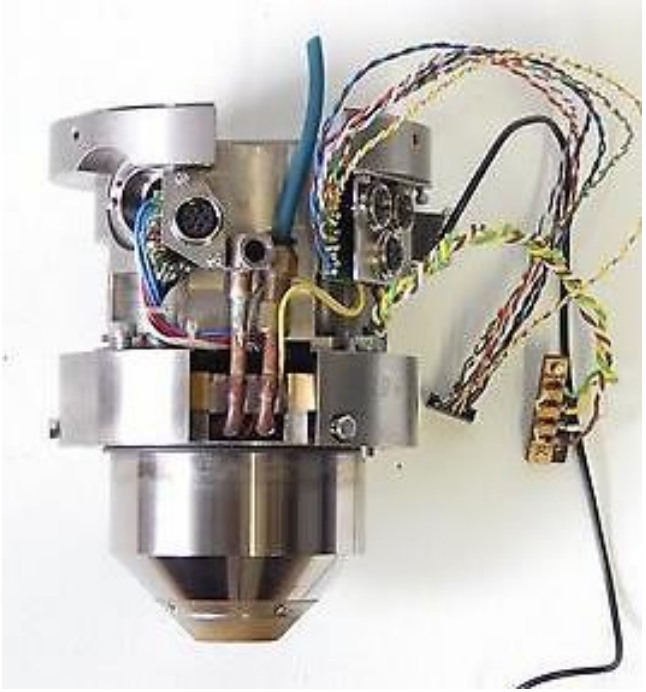
http://www4.nau.edu/microanalysis/Microprobe-SEM/Instrumentation.html

Objective lens

- Probe forming lens, focuses electron beam to the sample surface, defocuses electron beam

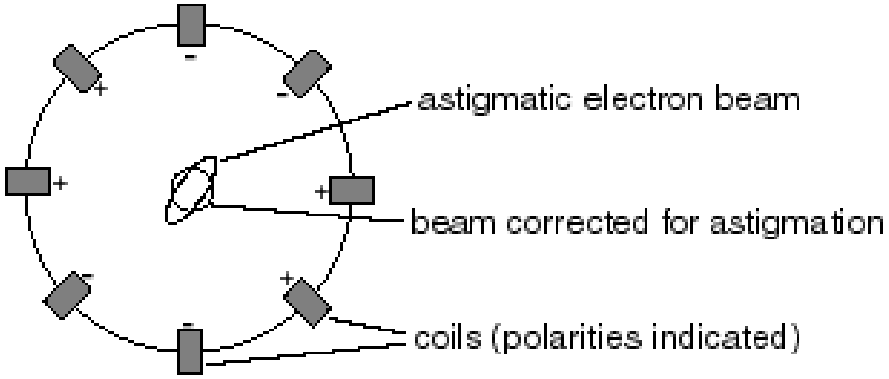




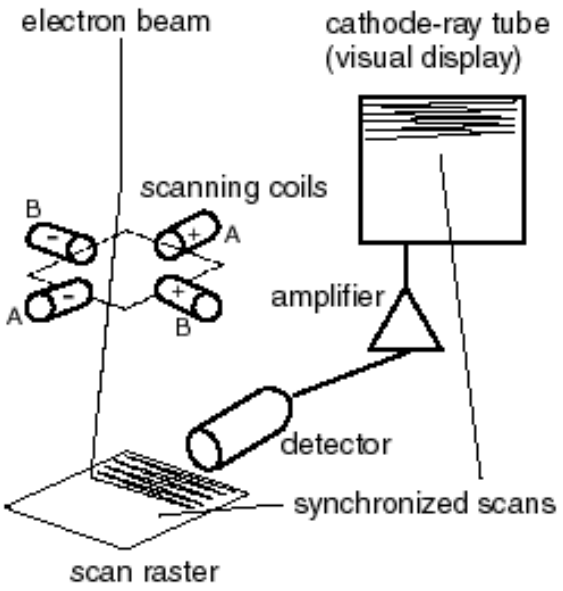


Stigmator and scanning coils

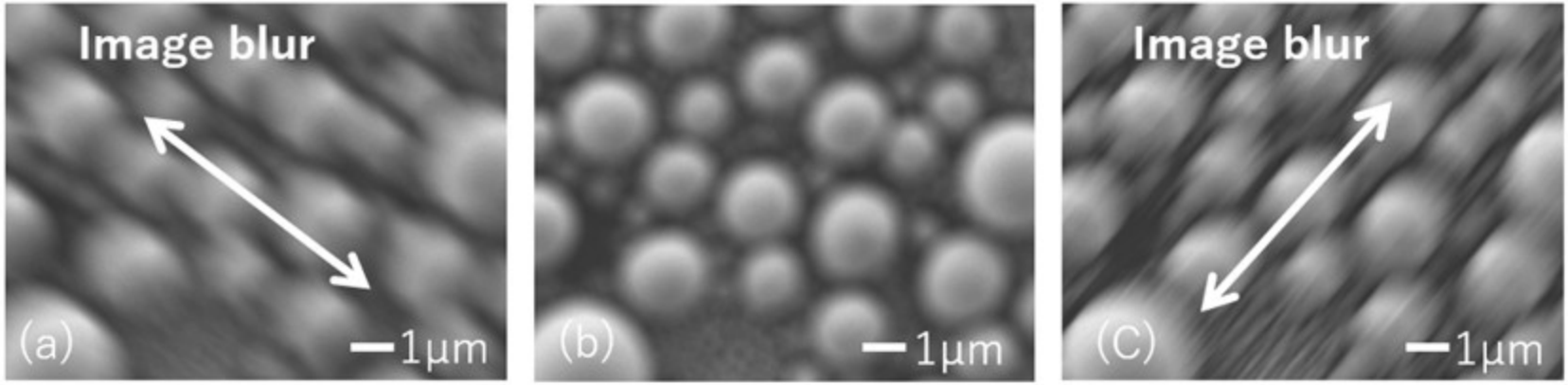
- stigmator – set of coils for corrections of abberation and shape of the beam



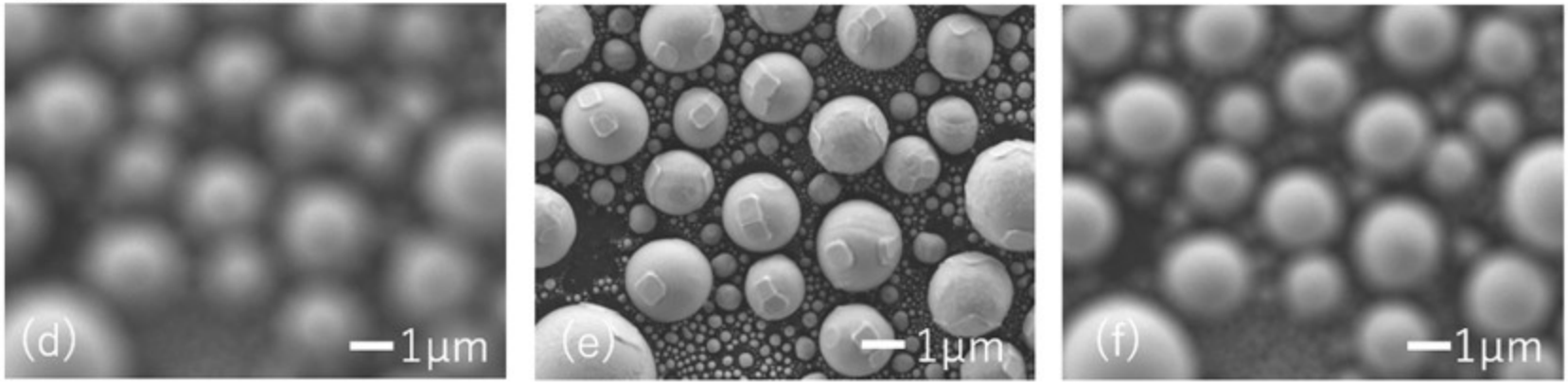
- Scanning coils for moving (scanning) of the electron beam on the sample surface



With astigmatism



Without astigmatism



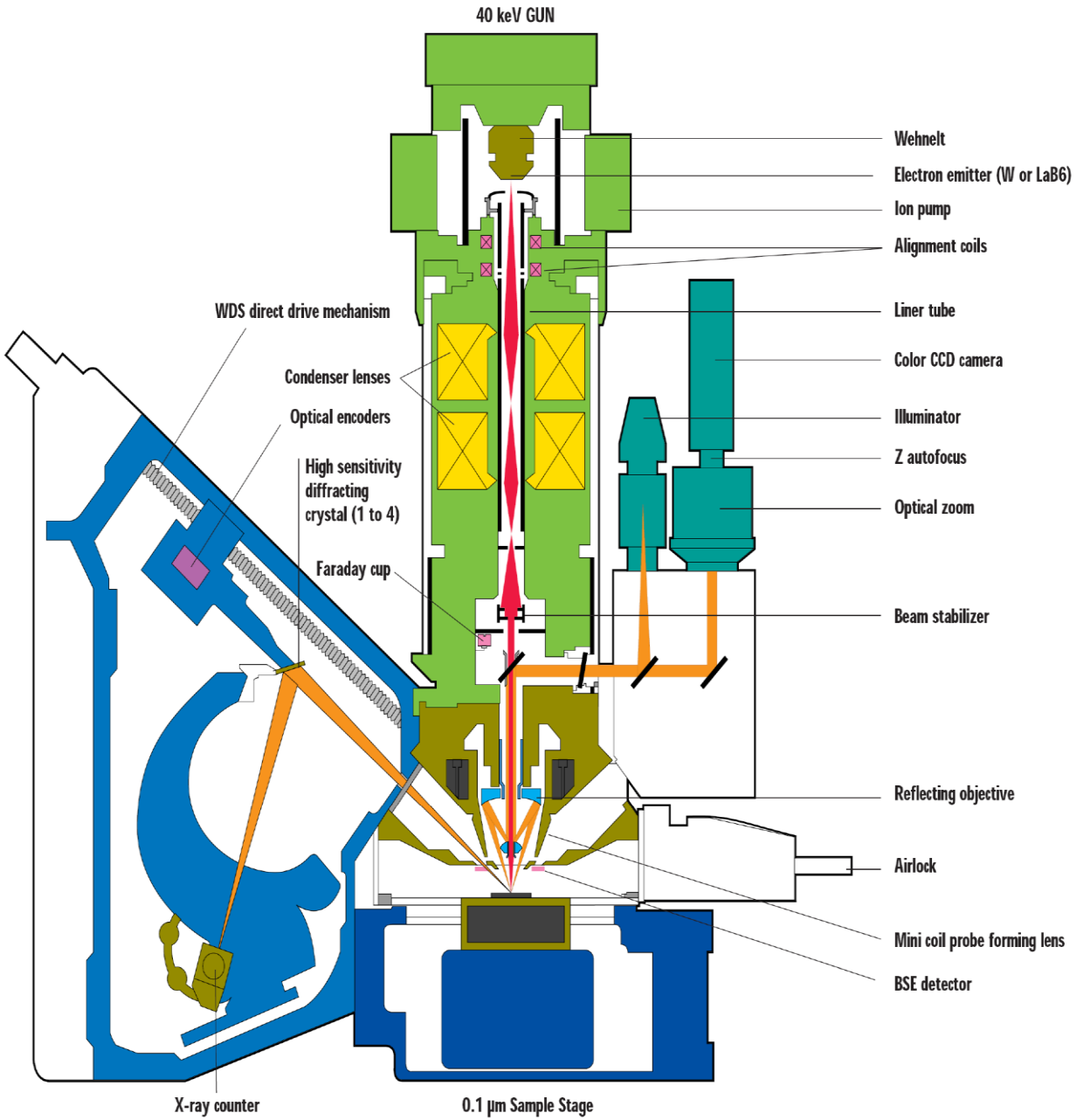
Over focus

Exact focus

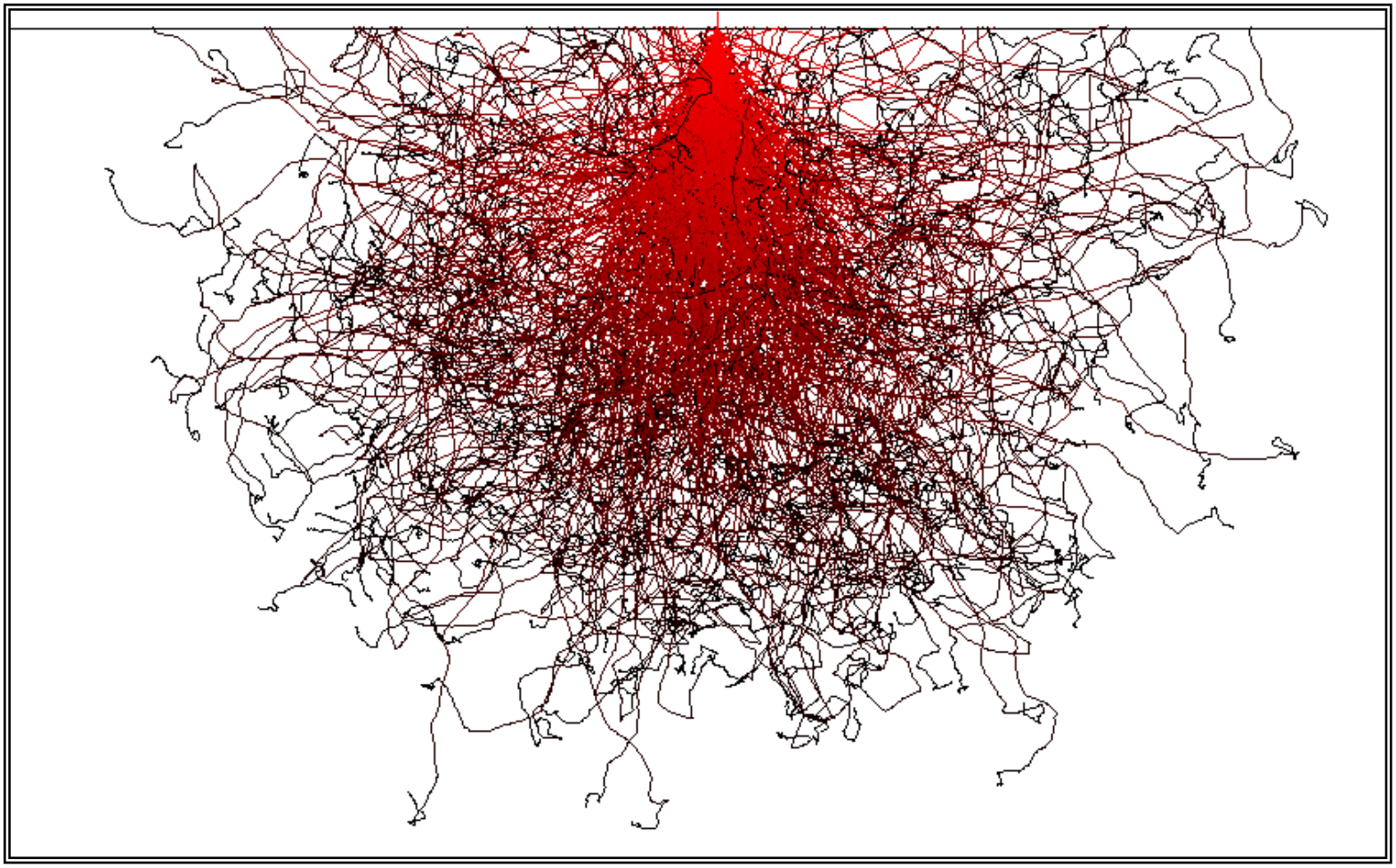
Under focus

SX 100

ELECTRON PROBE MICROANALYZER

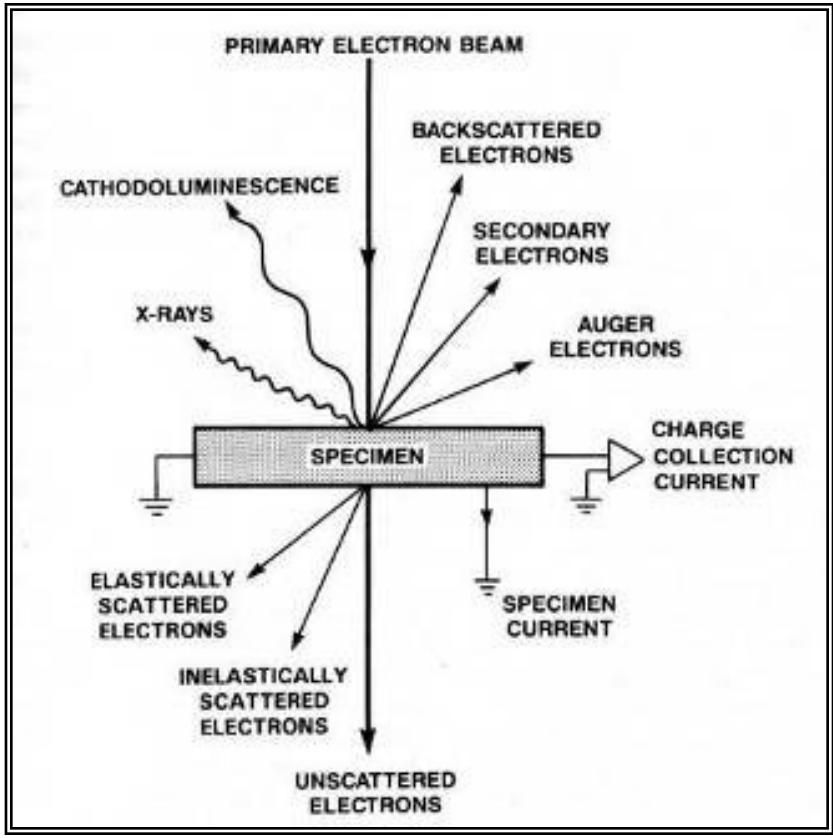


Interaction of the electron beam with a sample



Interaction of the electron beam with a sample

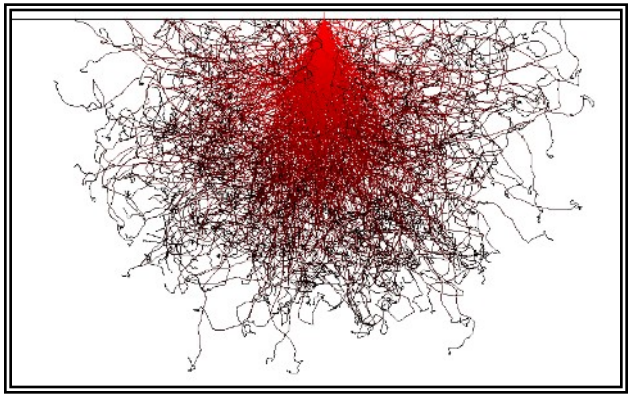
- Interaction od accelerated electrons with mass of sample causes production of several kinds of photons and electrons.
  - Elastic collision – changing of trajectory with minimal changes of energy
    - Back scattered electrons BSE
    - Transmitted electrons TE
  - inelastic collision – loosing of energy, interactions with electrons in atom´s shells of sample
    - Secondary electrons SE
    - Photons in visible range – cathodoluminescence CL
    - Auger electrons
    - Characteristic X-ray
    - Continual X-ray
    - heat

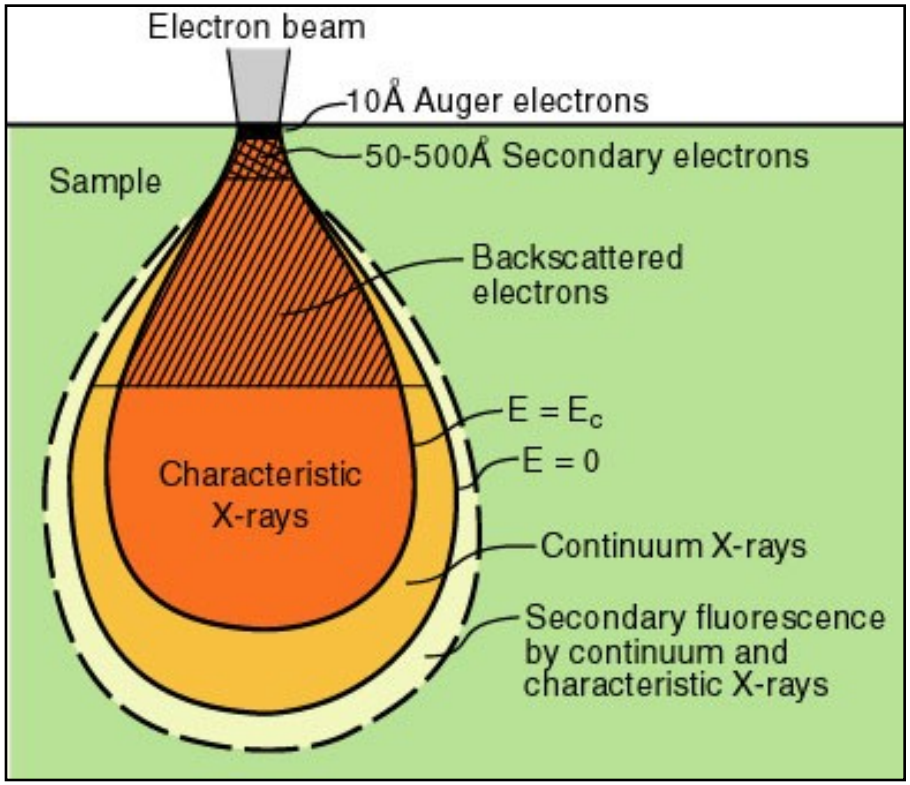


- Detection of these signals can be used for a detailed characterization of the sample

Excitation volume

- A space, where interactions take a place
- Increases with increasing electrons energy
- Decreases with increasing mean Z of sample
- The shape depends on a beam size and a mean Z





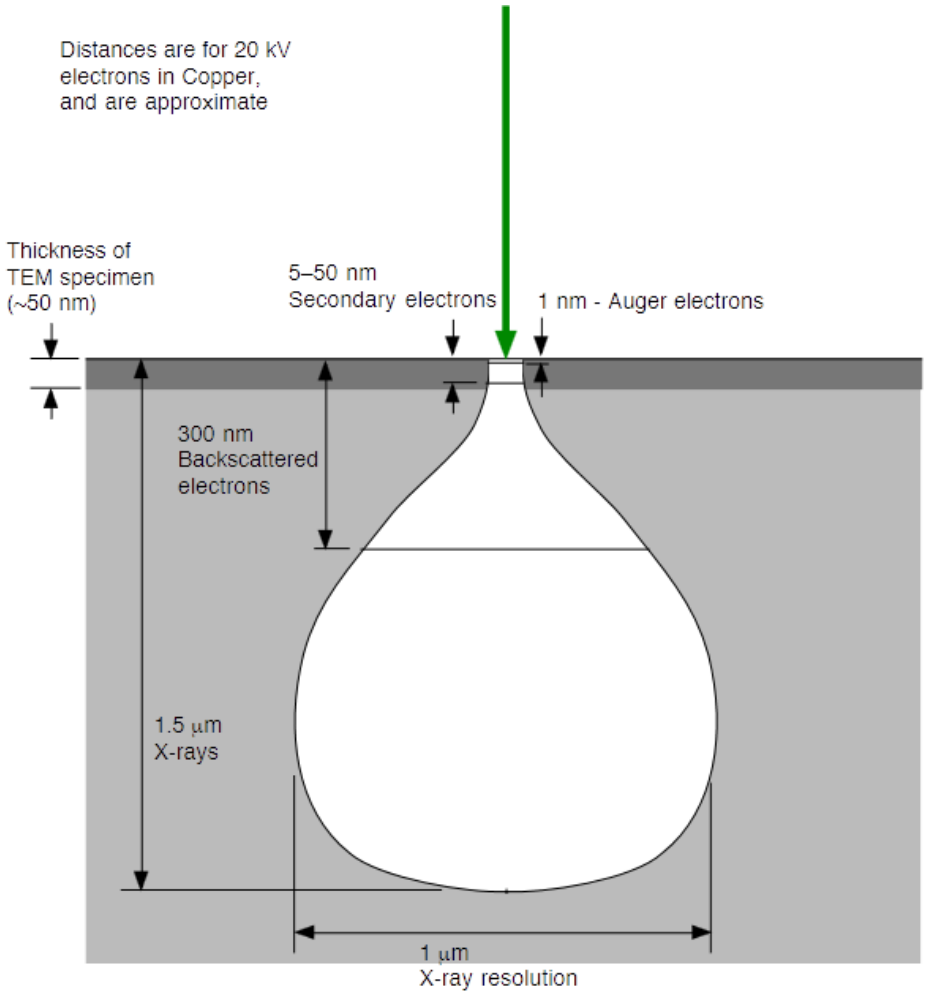
$$R = \frac{0.0276\, A\, E_0^{1.67}}{Z^{0.889}\rho}$$

A = atomic mass

E_0 = beam energy in keV

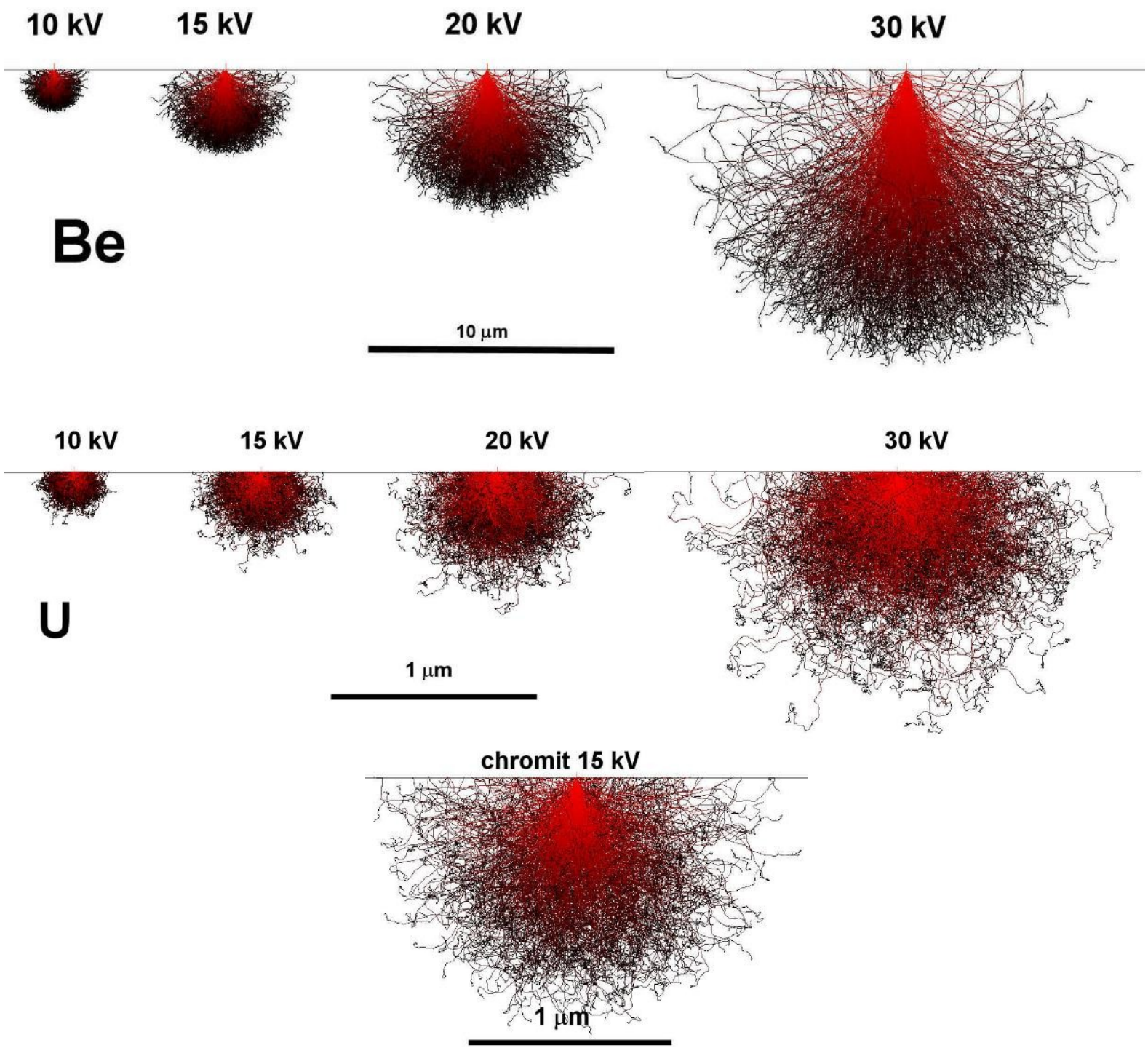
Z = atomic number

ρ = density in g/cm^3



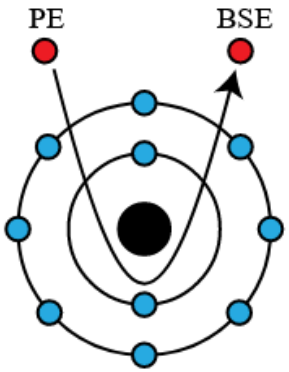
Interaction volume for electrons in a bulk sample. Distances are for 20 kV electrons in Cu. For Al, multiply by 3

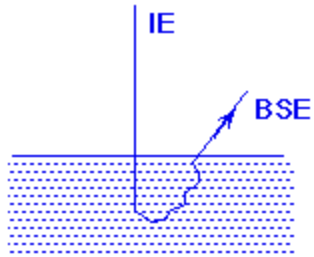
Target	5 keV	10 keV	20 keV	30 keV
Aluminium	0.41 μm	1.32	4.2	8.3
Copper	0.15	0.46	1.47	2.89
Gold	0.085	0.27	0.86	1.70


10 kV
15 kV
20 kV
30 kV
Be
10 µm
10 kV
15 kV
20 kV
30 kV
U
1 µm
chromit 15 kV
1 µm

Back-scattered electrons- BSE

- Elastic interations
- BSE –Back-Scattered Electrons
- BEI – Backscattered Electron Image





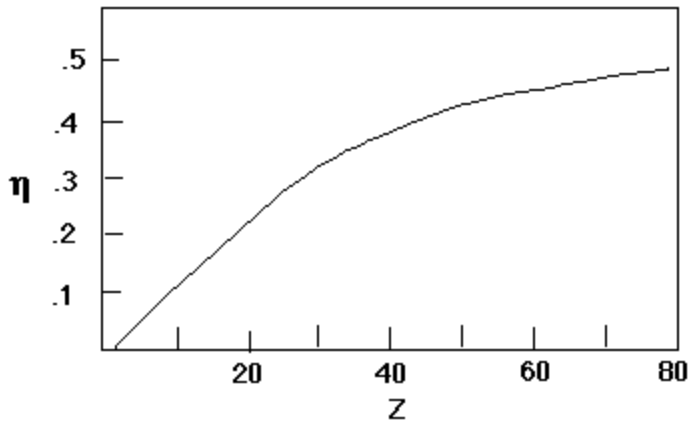
- $E_e \approx E_0$, $\Delta E < 1$ eV
- Generally, BSE are considered all electrons >50eV

- Production of BSE - η_b (back-scattering coefficient), is dependent on a mean atomic number Z of sample

$$\eta = \frac{N_{BSE}}{N_{IE}}$$

Z	η
10	12
20	23
30	32
40	37
50	43
60	45
70	48
80	50

$$\eta = -0.0254 + 0.016\,Z - 0.000186\,Z^2 + 8.3 \times 10^{-7} Z^3$$



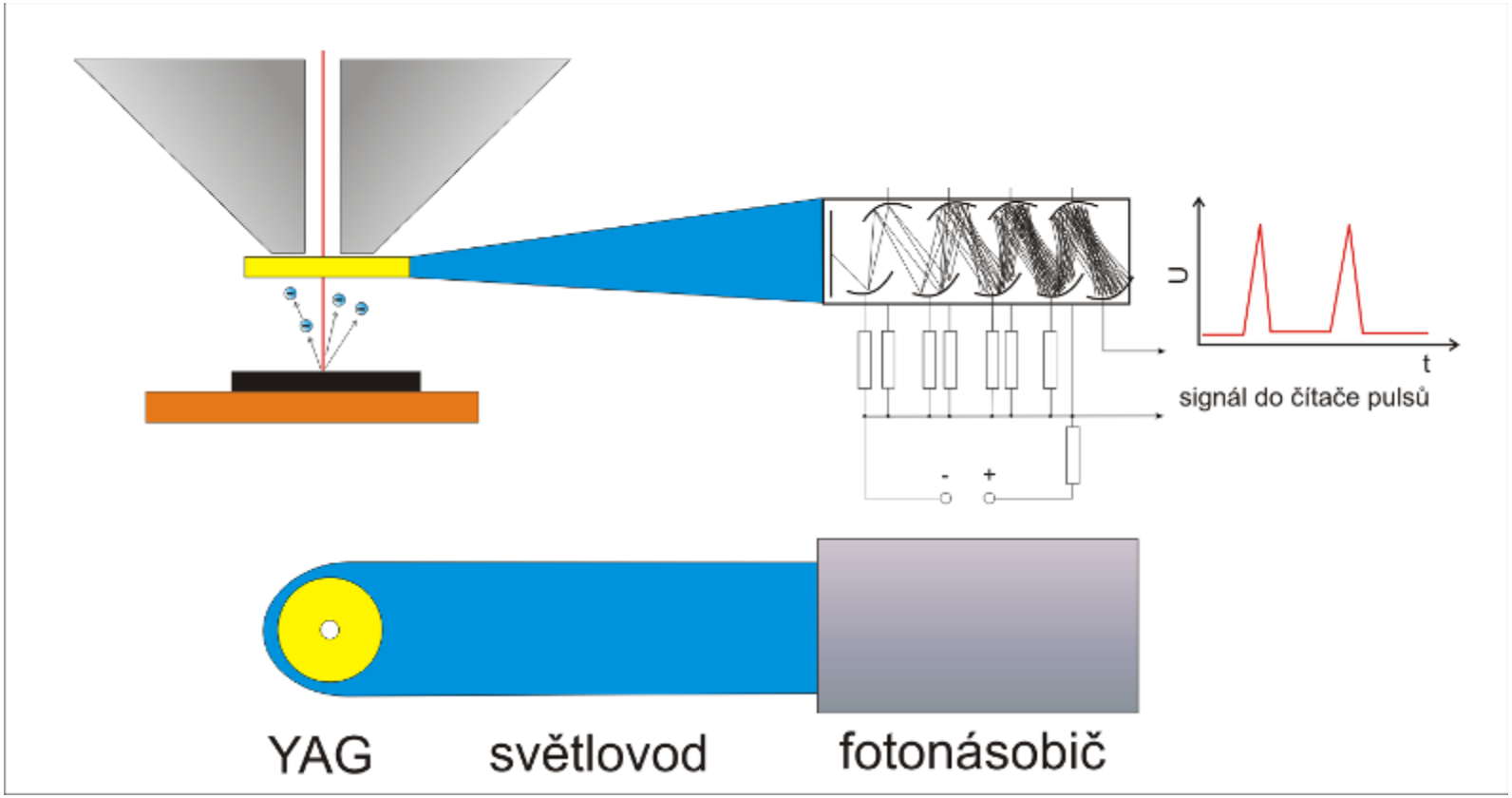
$$Z = w_1 z_1 + w_2 z_2 + w_3 z_3 \ldots + w_n z_n$$

$$Z_{SiO_2} = 0.4674 \times 14 + 0.5326 \times 8 = 10.8044$$

- **BSE give us information about a phase contrast in the analysed area**

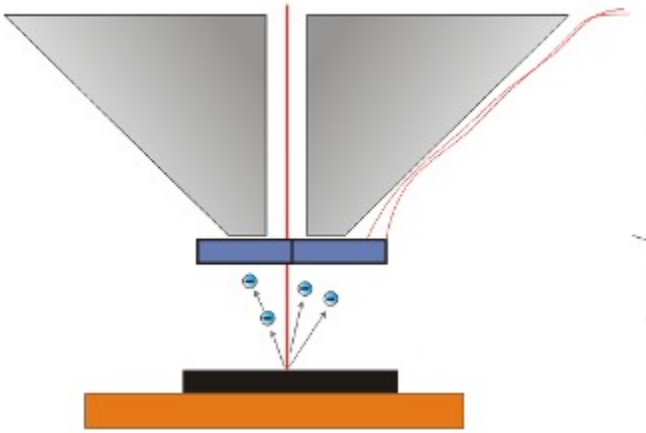
Scintillation type of BSE detector

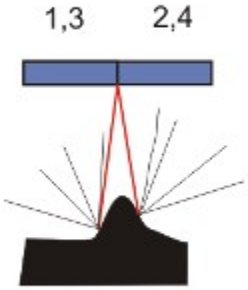
- „ROBINSON“ type of detector

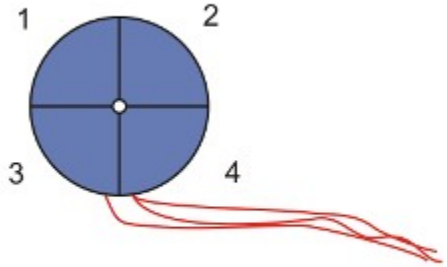


semiconductor type of BSE detector

- solid state detector

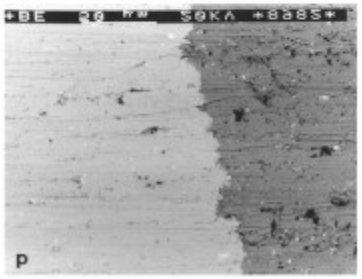


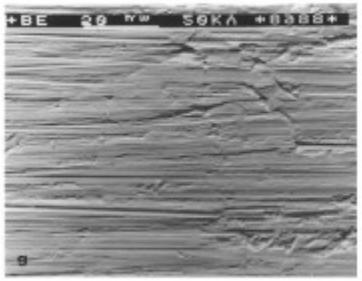




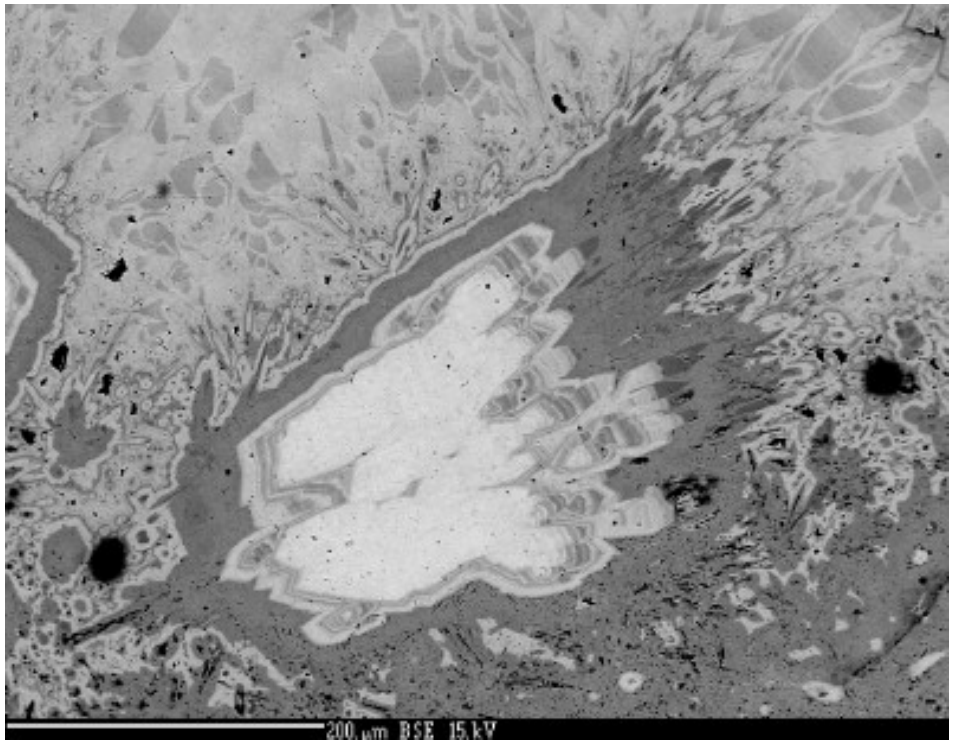
1+2+3+4 - obraz fázového kontrastu (compo)

(1+3)-(2+4) - obraz topografický (topo)

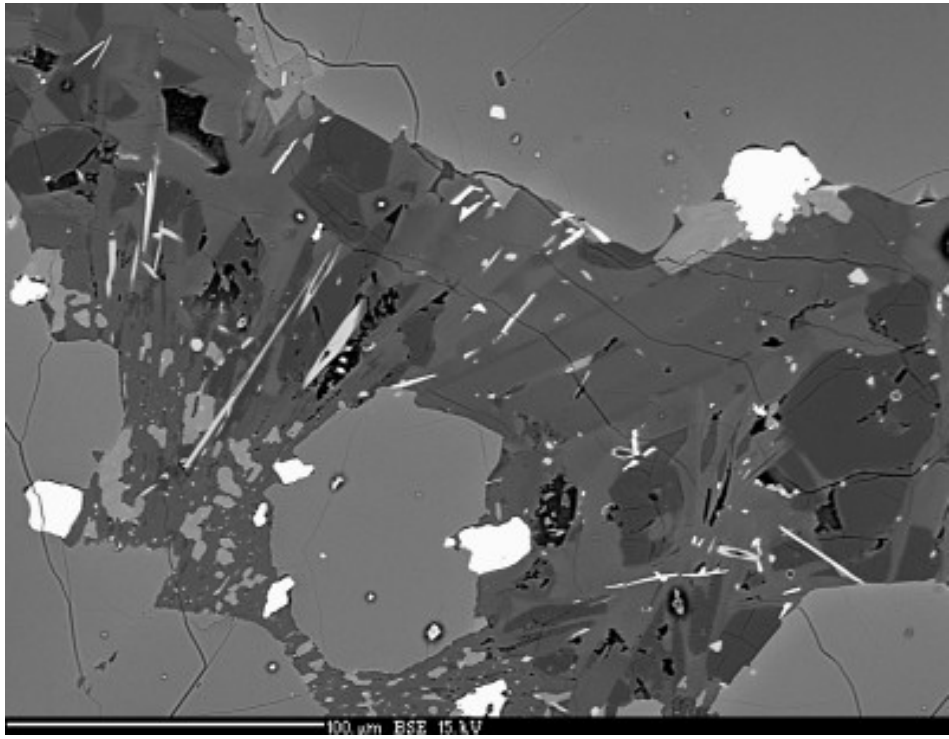




BSE images



chemical variability of Ca-Pb phosphate mineral
Ca-rich pyromorphite



bazaltic rock with olivine and other minerals

Secondary electrons - SE

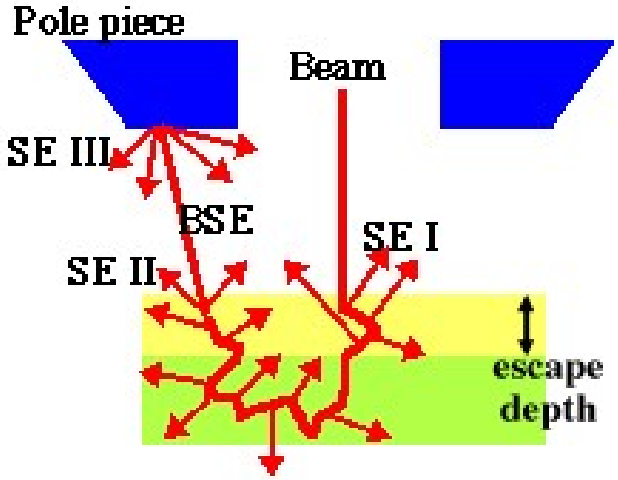
- SE - Secondary Electrons
- SEI - Secondary Electrons Image

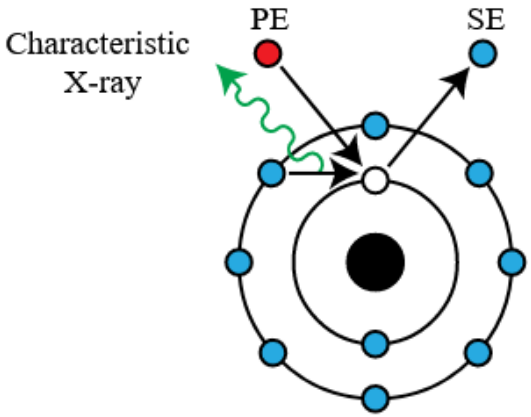
- SE are emitted form electron shells of atoms due to collision with primary

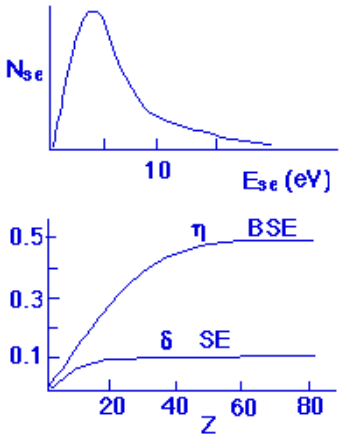
- energy up to 50 eV usually 2-10 eV
- SE can escape from only a thin surface layer ~500 Å, owing to their low energy

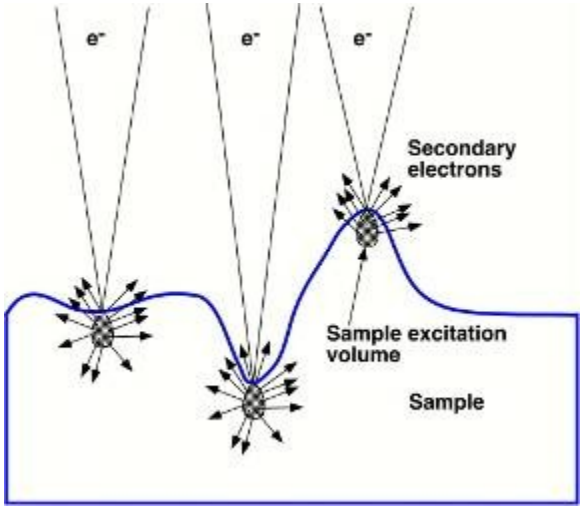
- The number of escaped electrons is mainly a function of morphology, effect of Z is minimal.

- One accelerated electron (10-30 keV) release about δ = 0.1-0.2 SE



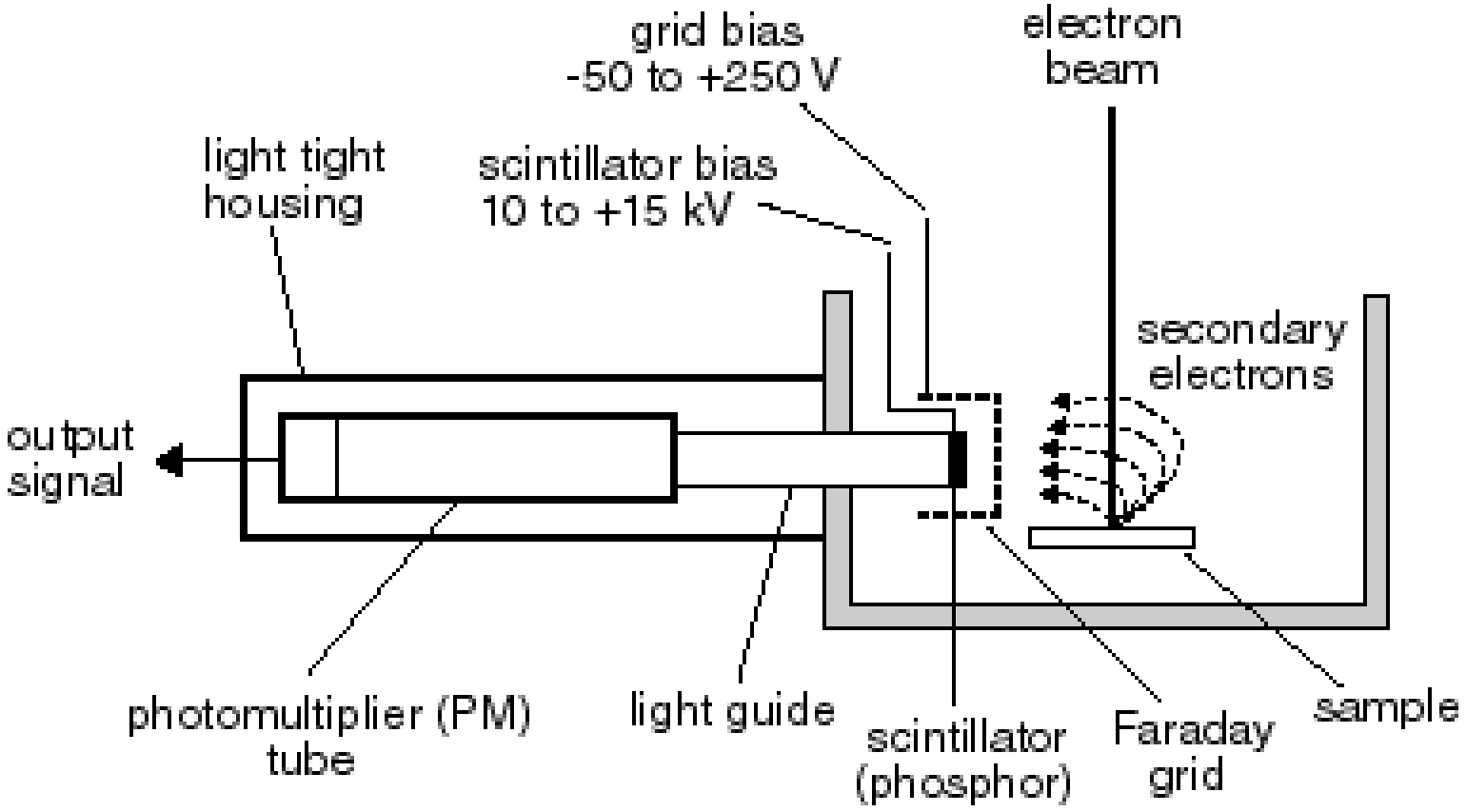




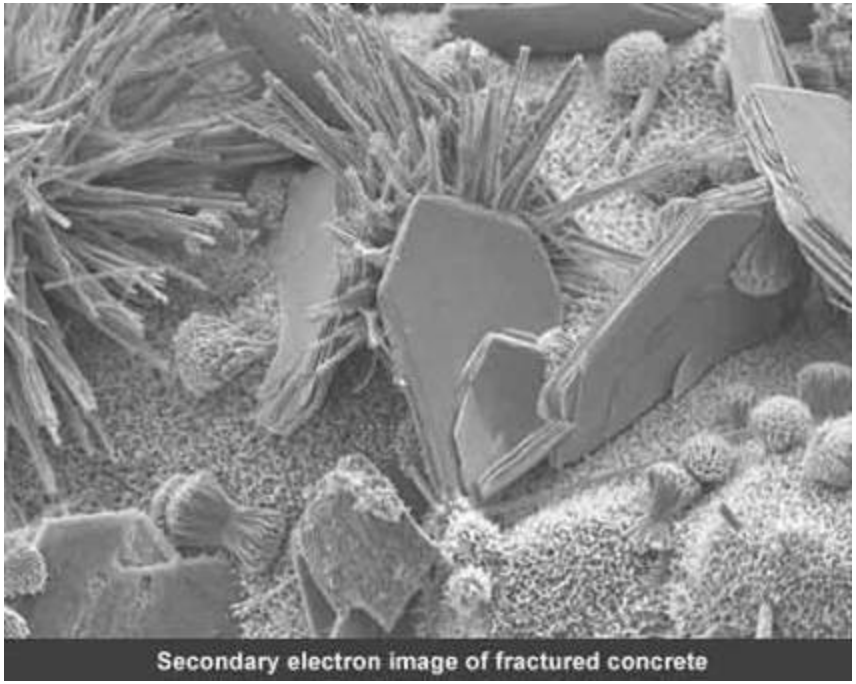


Detection of SE

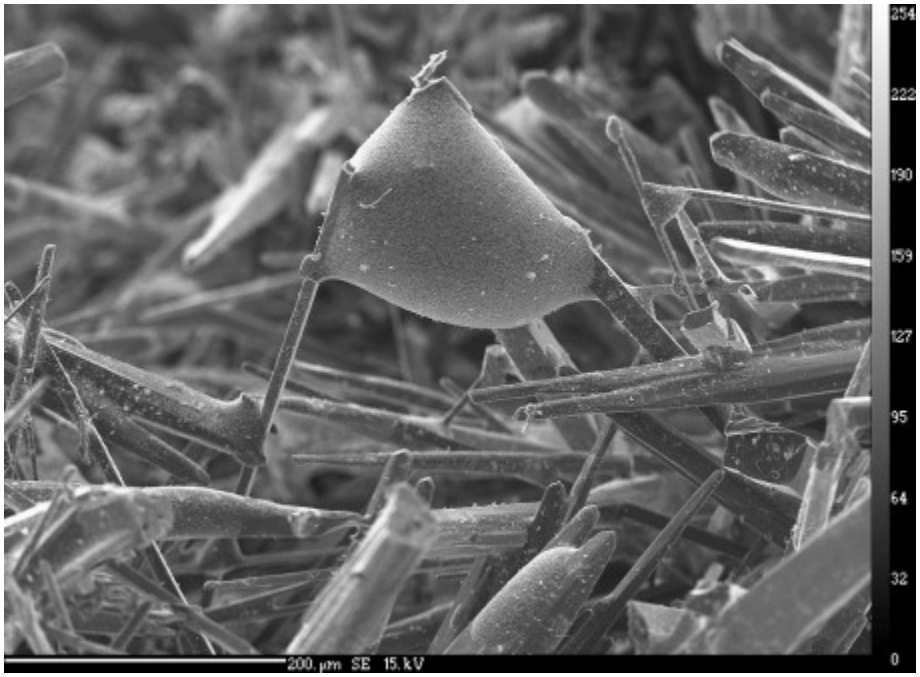
- Everhart and Thornley detector

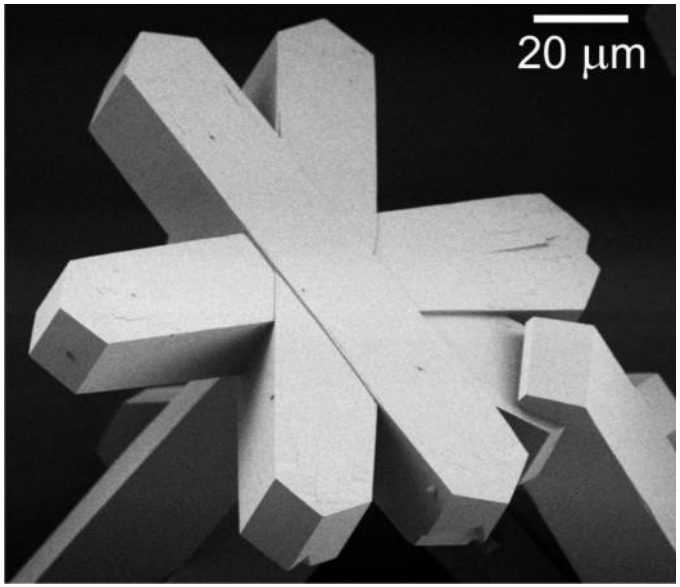


SE images

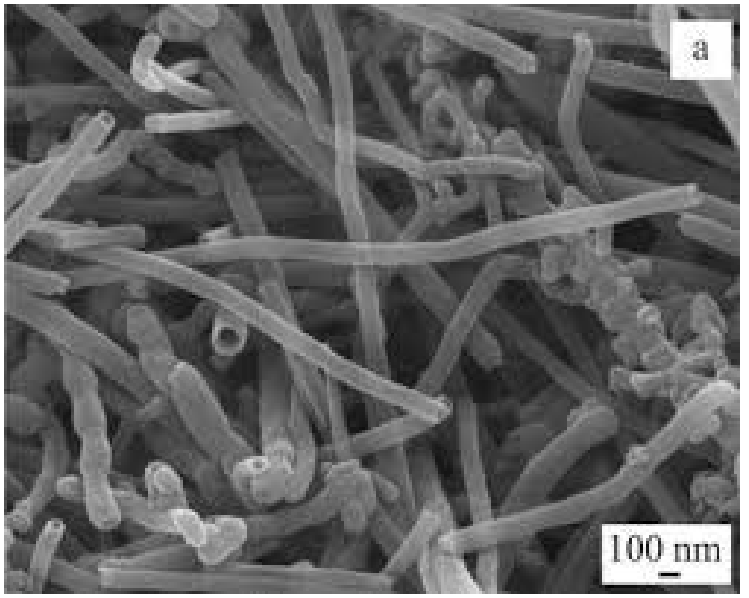


Secondary electron image of fractured concrete

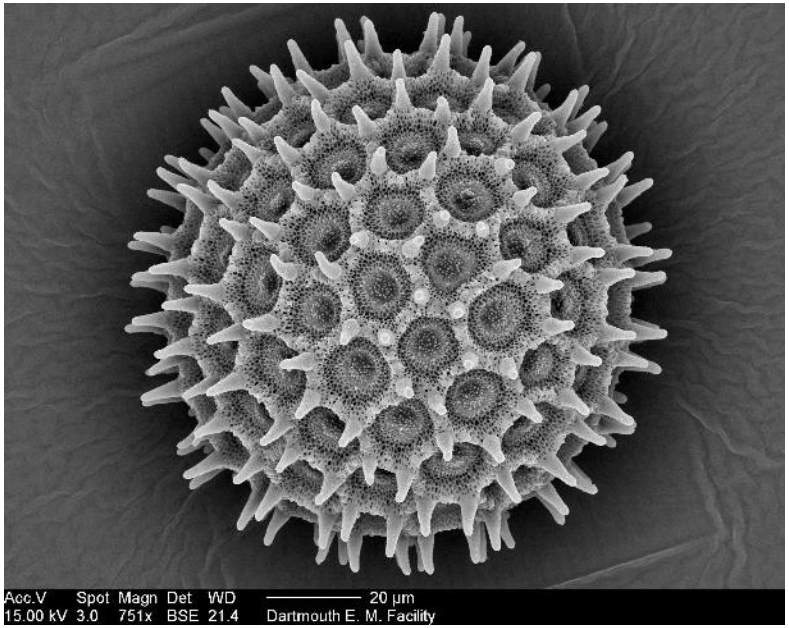




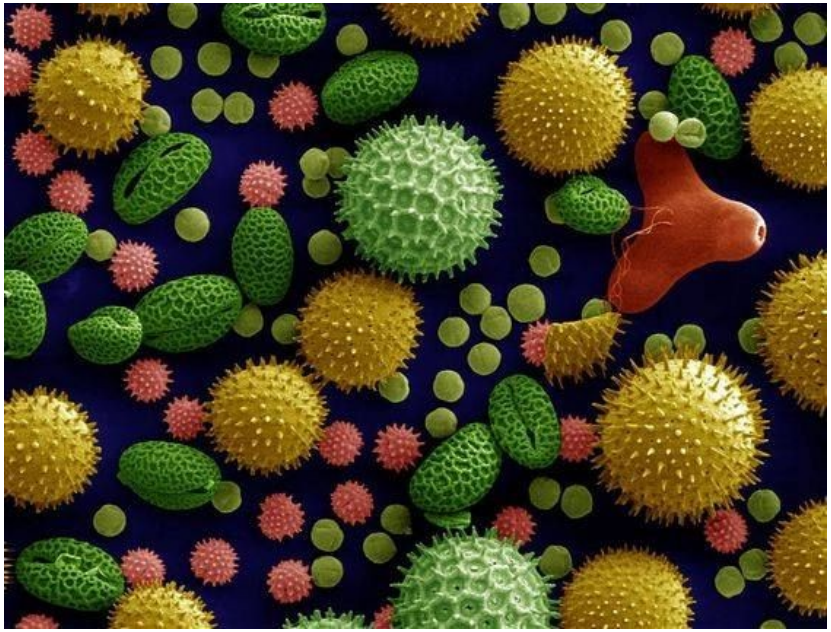
Synthetic zeolite



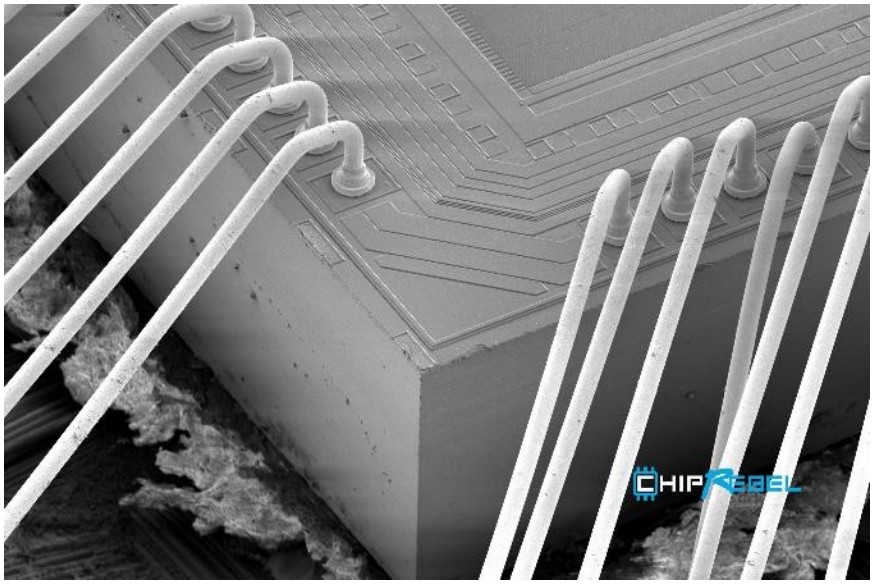
carbon nanovires

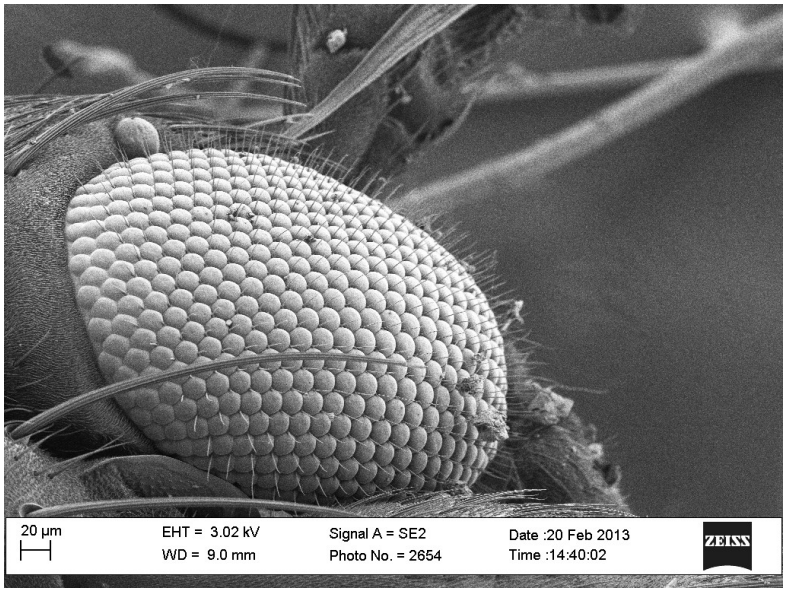


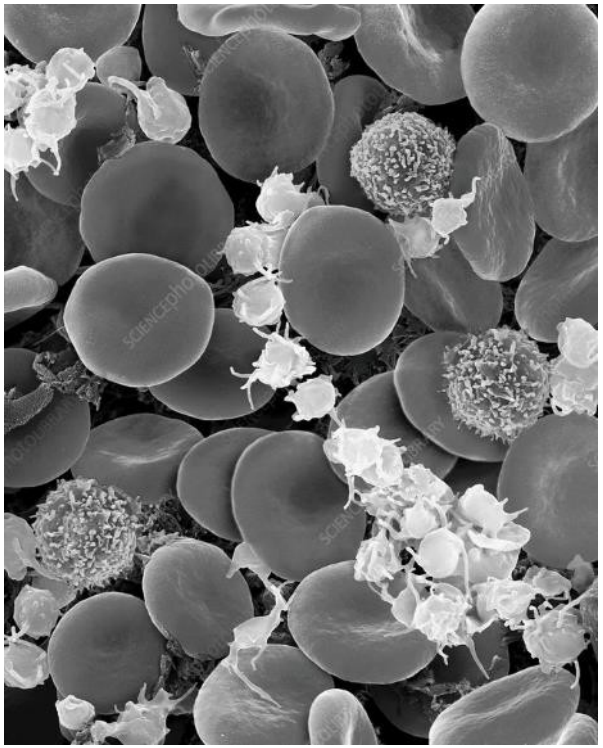
Polen particle

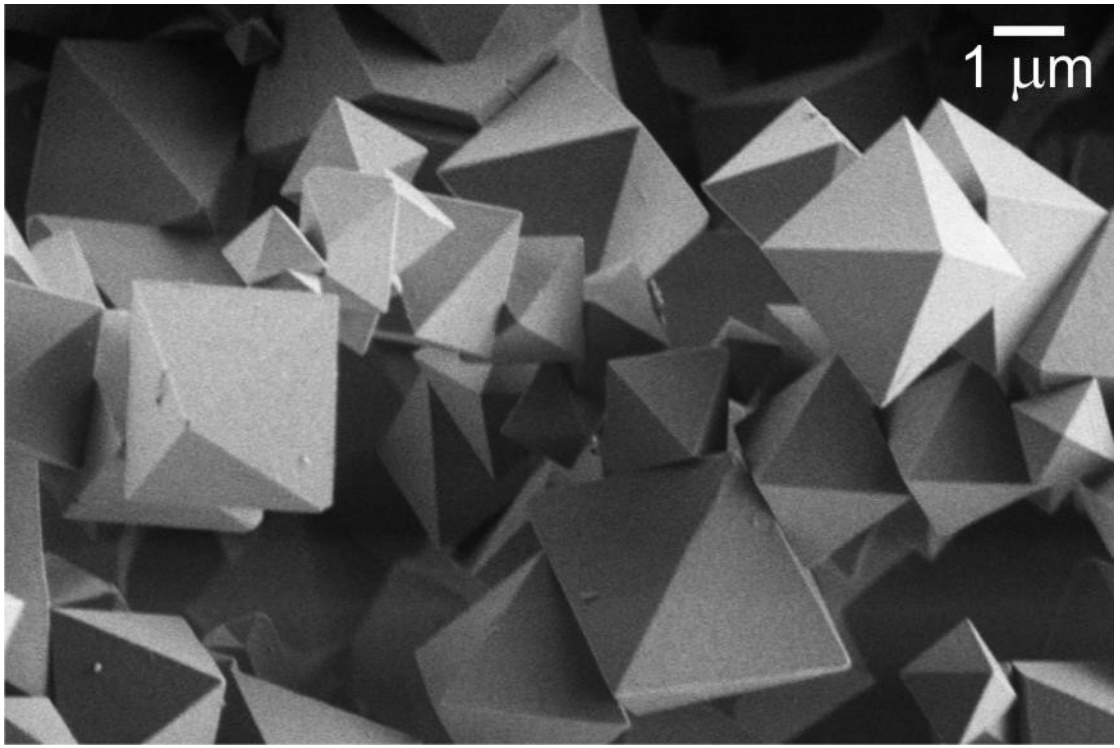


Photoshopized SEM images


CHIP


20 µm
EHT = 3.02 kV
WD = 9.0 mm
Signal A = SE2
Photo No. = 2654
Date :20 Feb 2013
Time :14:40:02
ZEISS

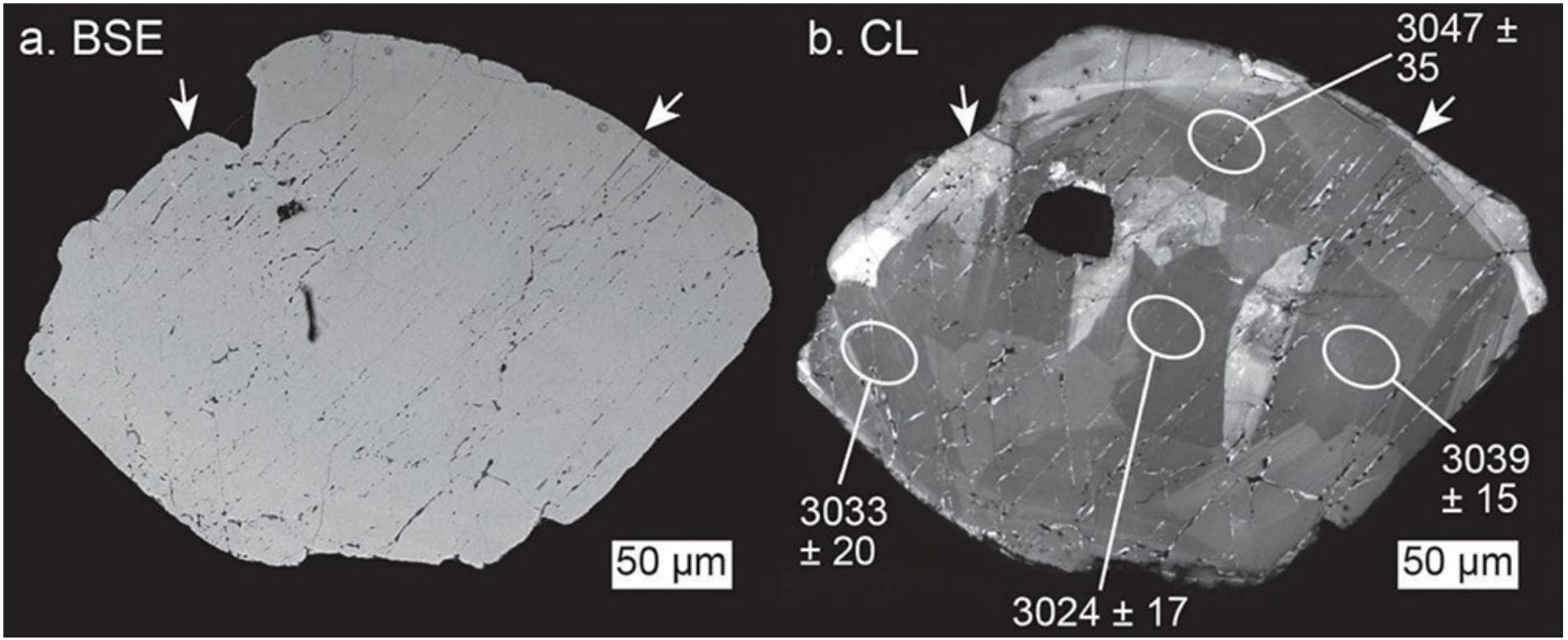



1 µm

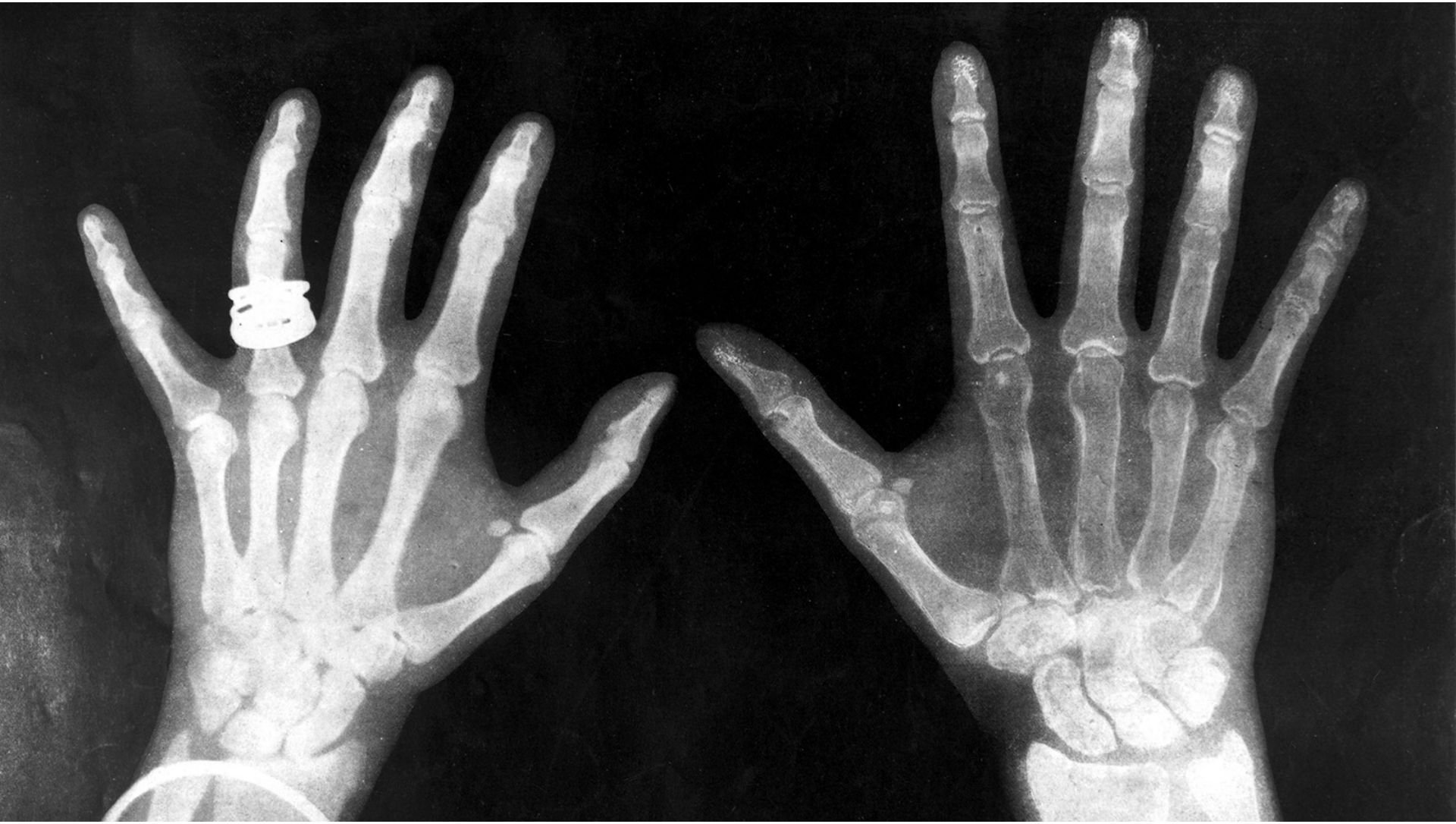
cathodoluminescence

- Production of photons in visible range
  - Shows changes in concentration of the CL activators (Mn, REE,…) in ppm
  - Information about growth history, zonation
- Scintillation detector – panchromatic image
- CL spectrometer – spectral characteristic of CL light

CL of natural diamond caused by variation of N content

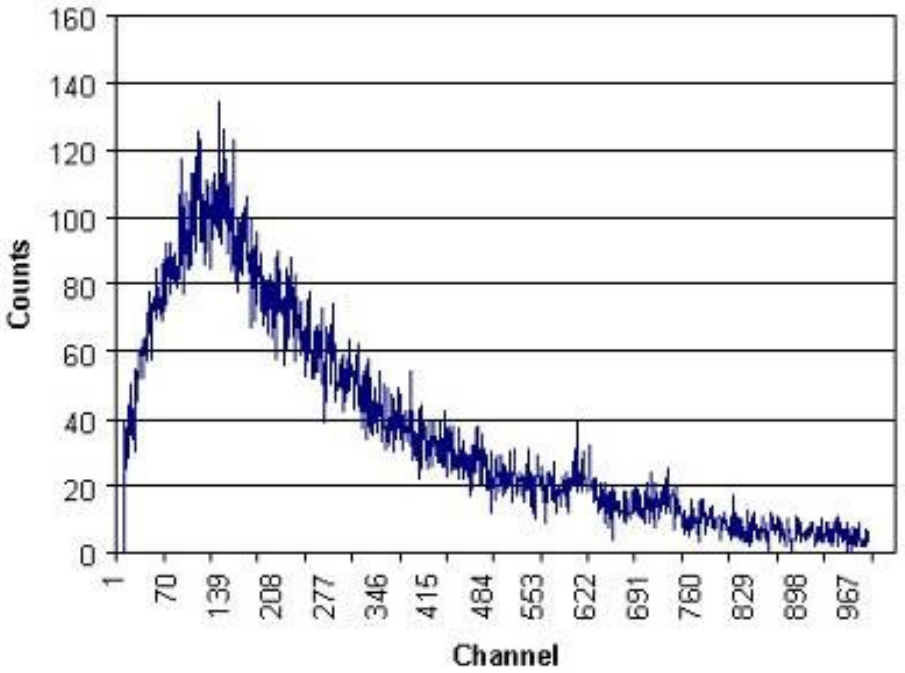

a. BSE
50 µm
b. CL
3047 ± 35
3033 ± 20
3024 ± 17
3039 ± 15
50 µm

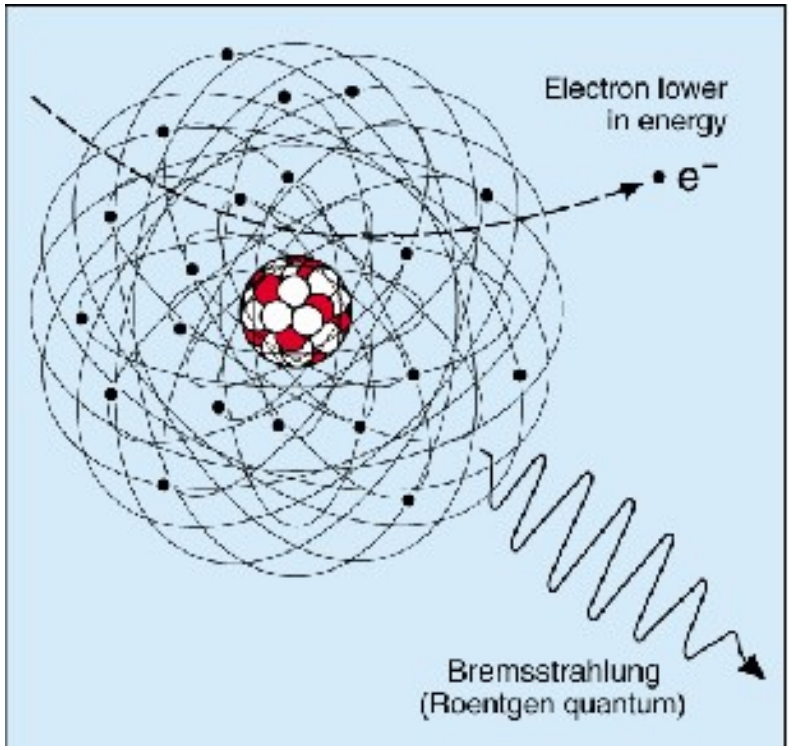
X-rays



continuum X-ray

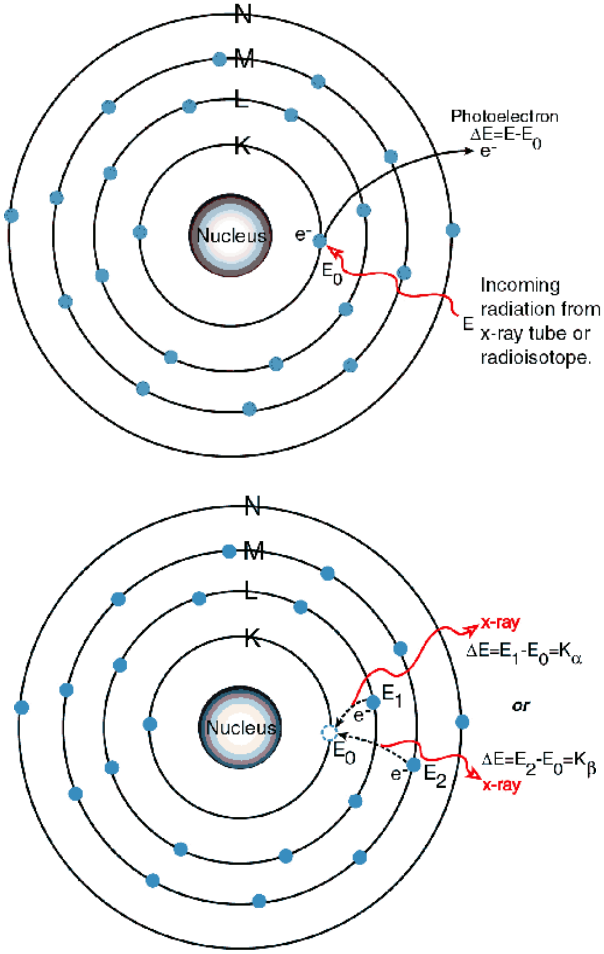
- Continuum
  - Inelastic collision
  - Braking radiation - **bremsstrahlung**
  - Energy 0 eV to ~ U_{acc}





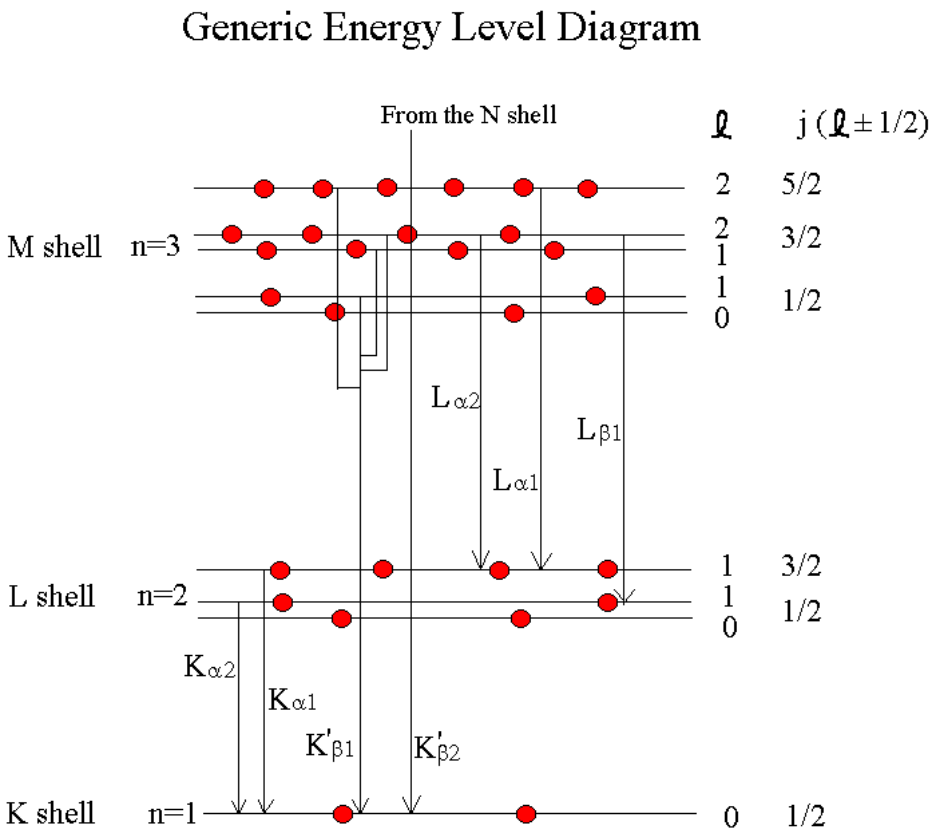
Characteristic X-ray

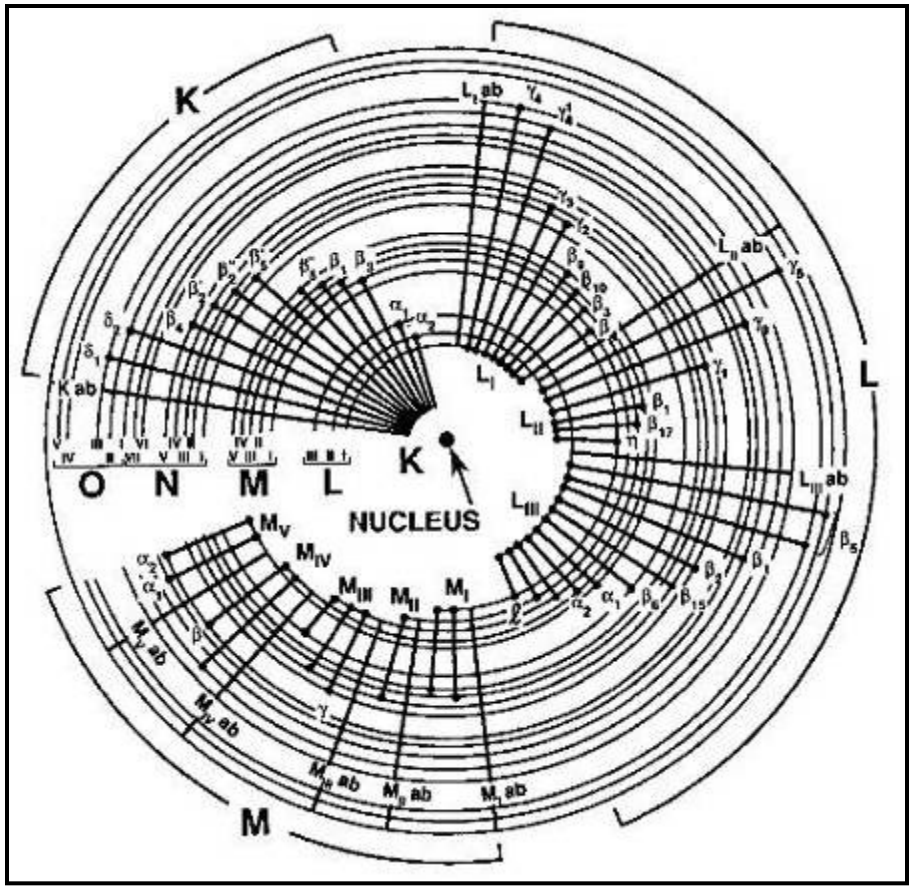
- ~ 0.X % of accelerated e^- hit e^- in atom shell and knock it out – SE
- The vacancy in orbital is filled by jumping of e^- from higher energy orbitals
- The transition is followed by emission of quantum of E in form of X-ray photon
- Several types of transition
- K, L, M lines



http://www.matter.org.uk/tem/electron_atom_interaction/x-ray_and_auger.htm

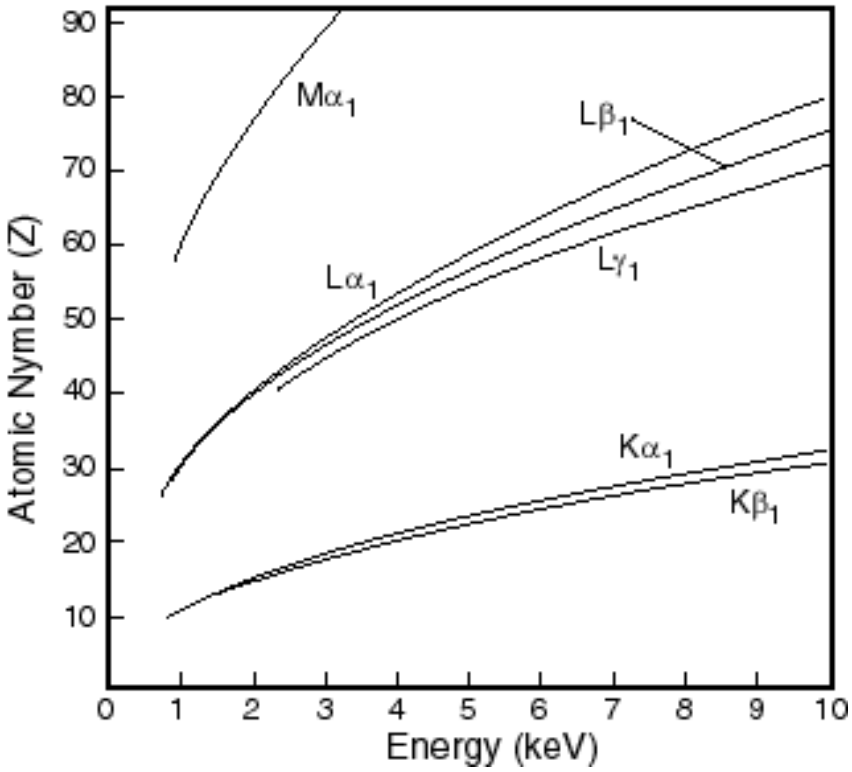
Characteristic X-ray



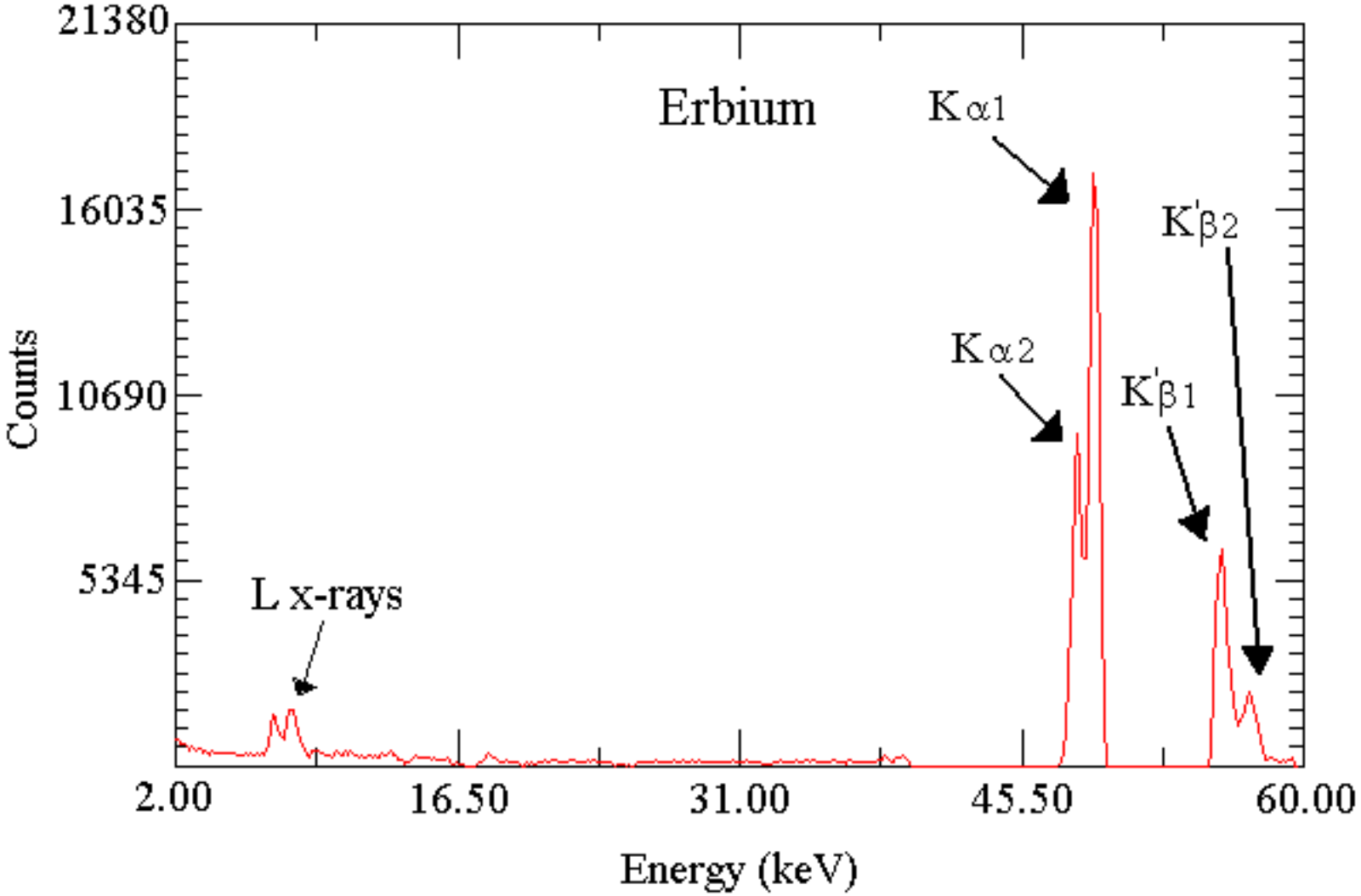


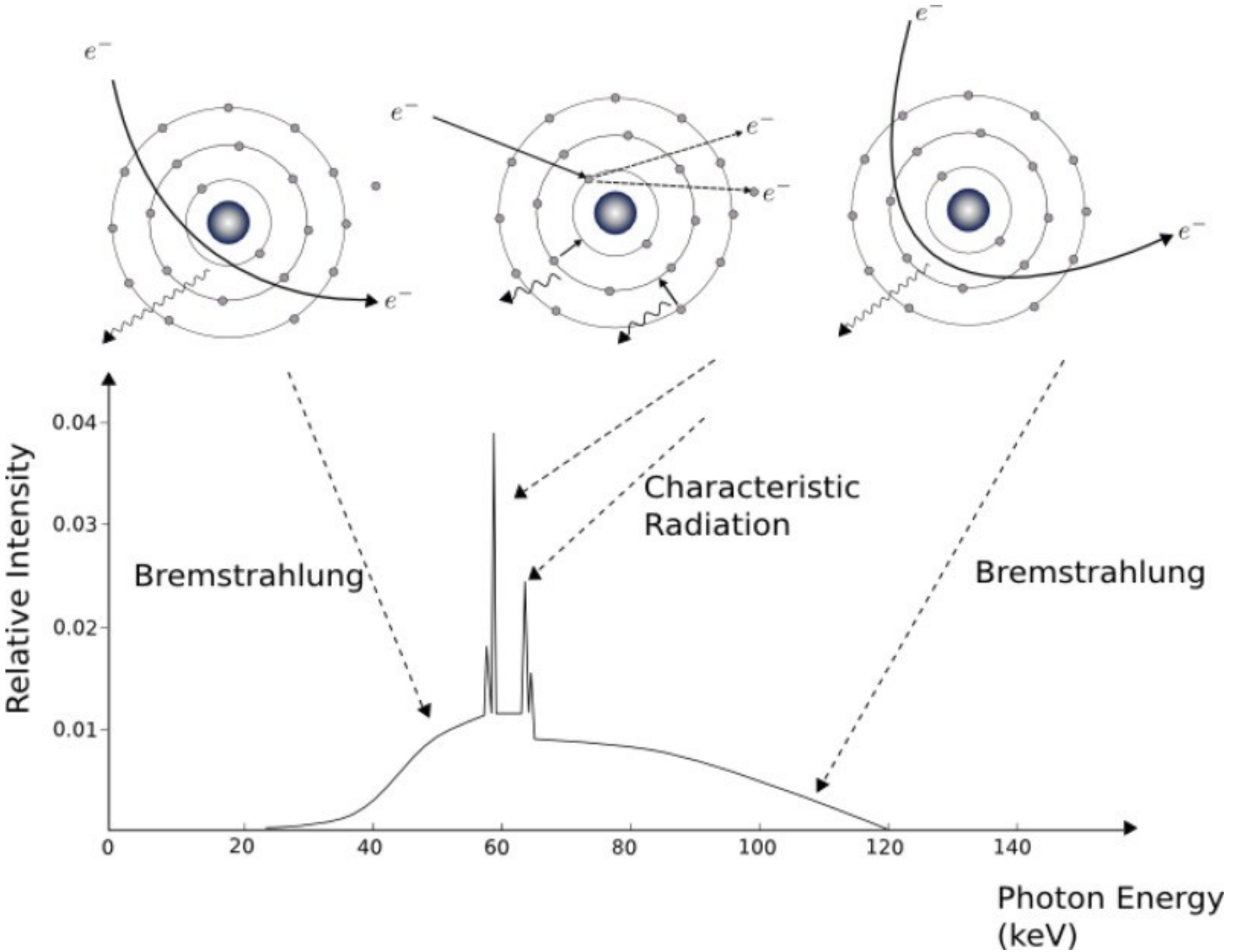
Characteristic X-ray

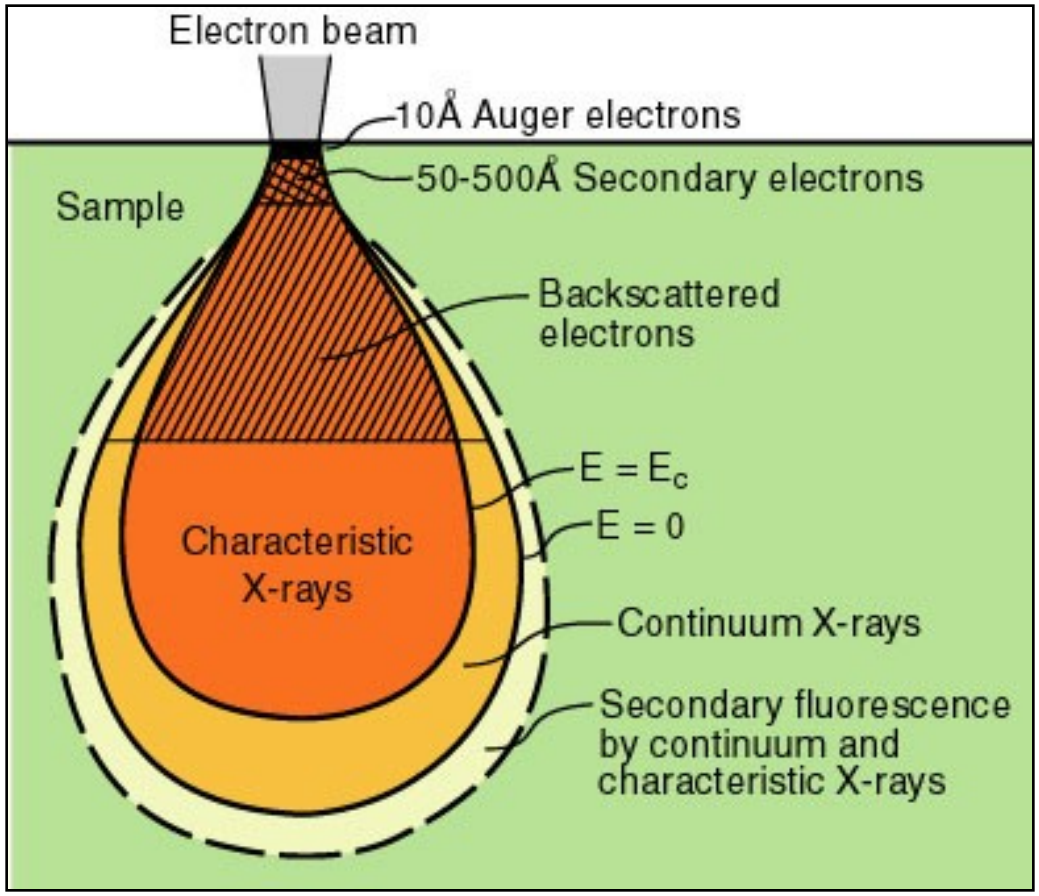
Source Shell	Shell Filled						
	K	L_I	L_{II}	L_{III}	M_{III}	M_{IV}	M_V
L_I							
L_{II}	$K\alpha_2$ (50)						
L_{III}	$K\alpha_1$ (100)						
M_I			$L\eta$ (1)	L_1 (2)			
M_{II}	$K\beta_3$ (1)	$L\beta_4$ (5)		Lt (0.01)			
M_{III}	$K\beta_1$ (20)	$L\beta_3$ (6)	$L\beta_{17}$	L_S (0.01)			
M_{IV}	$K\beta_{5'}$	$L\beta_{10}$	$L\beta_1$ (50)	$L\alpha_2$ (10)			
M_V	$K\beta_{5''}$	$L\beta_9$		$L\alpha_1$ (100)			
N_I			$L\gamma_5$ (0.1)	$L\beta_6$ (0.1)			
N_{II}	$K\beta_{2''}$ (5)	$L\gamma_2$ (1)					
N_{III}	$K\beta_{2'}$	$L\gamma_3$ (2)					
N_{IV}	$K\beta_4$		$L\gamma_1$ (10)	$L\beta_{15}$ (1)	$M\gamma_2$ (1)		
N_V	$K\beta_4$			$L\beta_2$ (20)	$M\gamma_1$ (1)		
N_{VI}			$L\nu$			$M\beta_1$ (50)	$M\alpha_2$ (100)
N_{VII}			$L\nu$				$M\alpha_1$ (100)
O_I		$L\gamma_4$	$L\gamma_8$	$L\beta_7$			
O_{II}	$K\delta_2$ (0.1)	$L\gamma_4$					
O_{III}	$K\delta_{1'}$ (0.1)						
O_{IV}			$L\gamma_8$	$L\beta_5$			
O_V				$L\beta_5$			



Characteristic X-ray




e^-
e^-
e^-
e^-
e^-
e^-
Relative Intensity
0.04
0.03
0.02
0.01
Bremstrahlung
Characteristic
Radiation
Bremstrahlung
0
20
40
60
80
100
120
140
Photon Energy
(keV)


Electron beam
10Å Auger electrons
50-500Å Secondary electrons
Sample
Backscattered electrons
E = E$_c$
E = 0
Characteristic X-rays
Continuum X-rays
Secondary fluorescence by continuum and characteristic X-rays

Electron microanalysis

- Relativelly non-destructive method to analyse the chemical composition of „usually“ inorganic sample
- Small sample needed - few microns
- Detection of characteristic X-ray from the sample

Electron microanalysis

- **Energy-dispersive X-Ray spectroscopy** (EDS, EDX)
  - quantum nature of radiation
  - semiconductor type of detector

- **Wavelength-dispersive X-Ray spectroscopy** (WDS, WDX)
  - wave nature of radiation
  - based on the X-ray diffraction

- Accelerating voltage 15 kV for silicates, 20-25 kV for sulphides and alloys

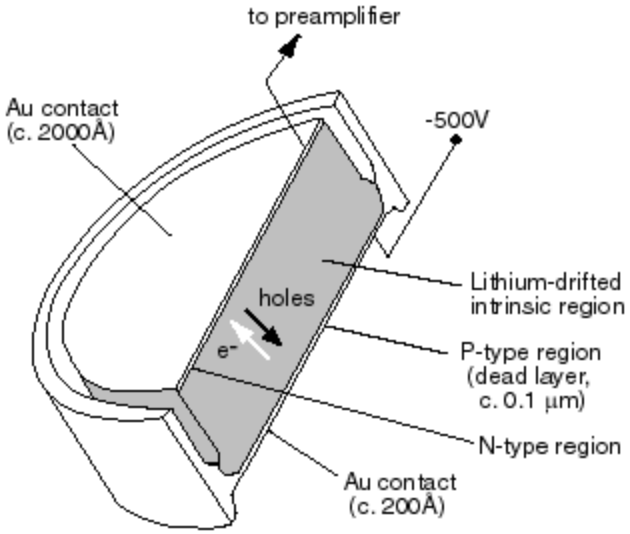
energy dispersive spectroscopy

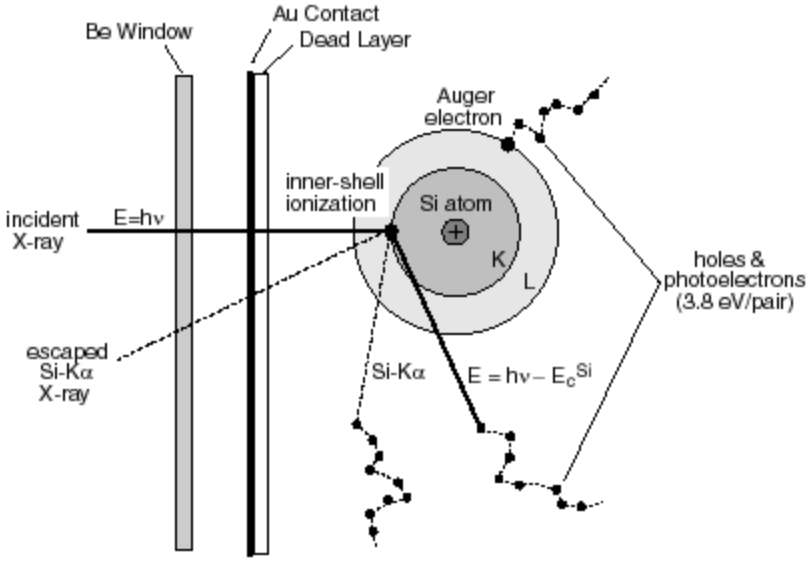
- Semiconductor detector Si:Li
  - area 10-40mm^2
  - bias 500-600V

- X-ray generates pairs electron-hole

- The higher E of X-ray, the more pairs of electron-hole => higher current pulse

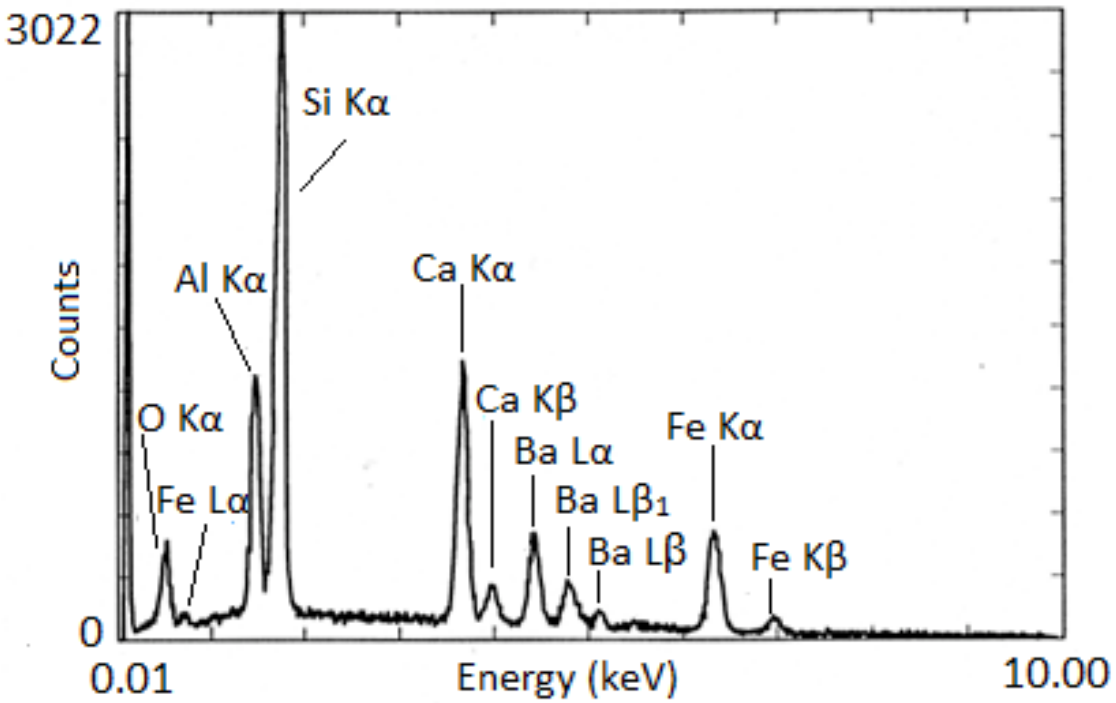
- Old types: Na - U
- Modern types: (Be) B - U
- Cooling od detector (LN_2, peltier module)





energy dispersive system (EDS, EDX)

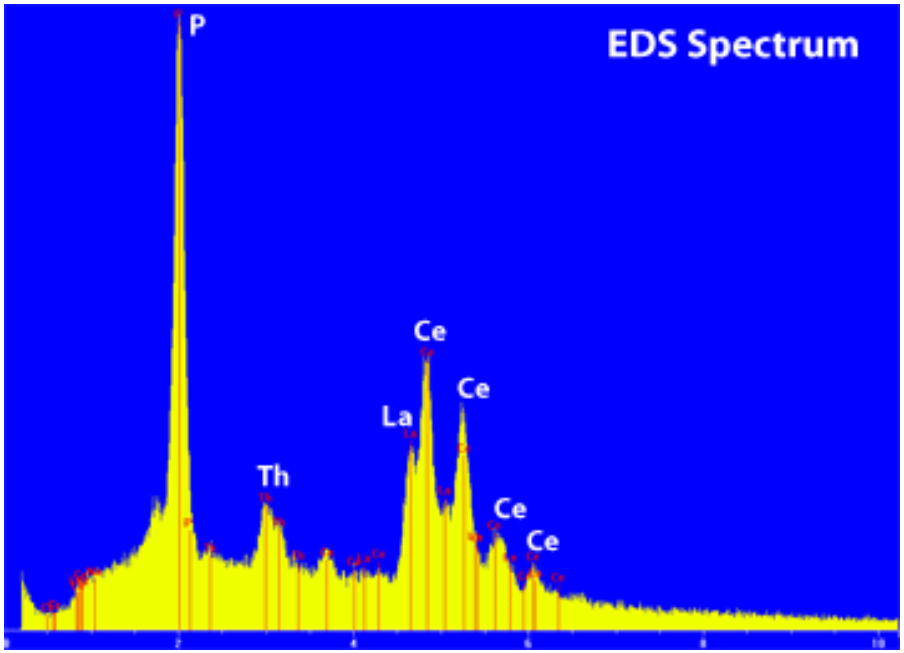
- advantages
  - The whole spectrum at once
  - Fast analysis 30, 60 s
  - Cheaper than WDS
- disadvantages
  - Poor resolution 130 -150 eV per channel
  - Common overlaps Pb-Bi-S, Mo-S, As-Mg, Na-Zn, Ba-Ti
  - Higher detection limit 0.1-1.0 hm.%

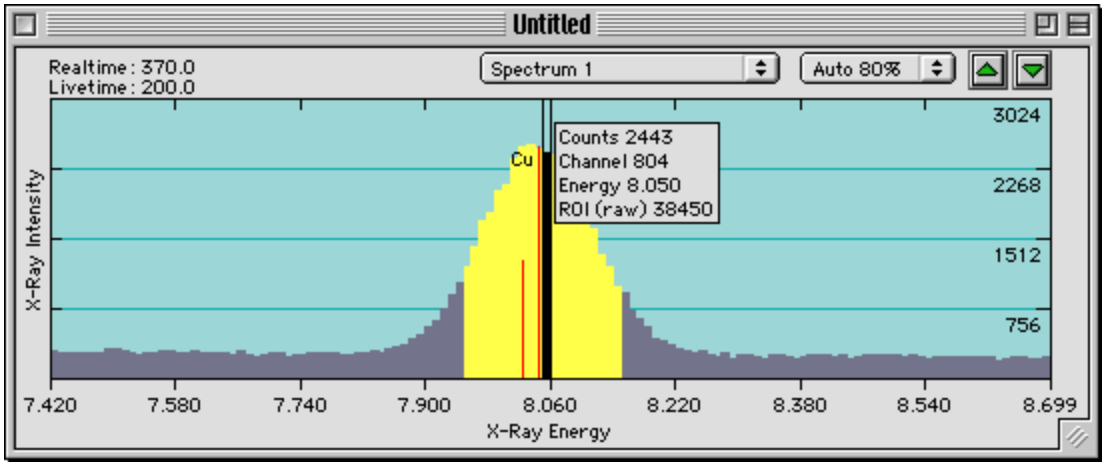


energy dispersive system (EDS, EDX)

- Peak position on x-axis - energy
- Peak height (area) - concentration
- Concentration of element
  - Ratio between peak area of unknown (sample) and peak area of standard

- $c_{unk}=I_{unk}/I_{std} \times c_{std}$



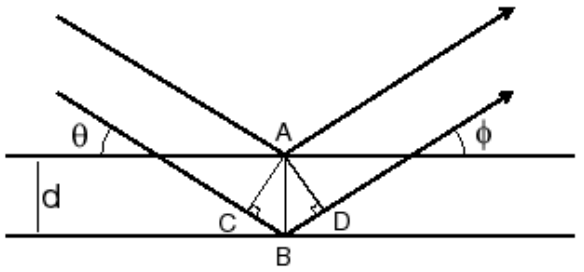


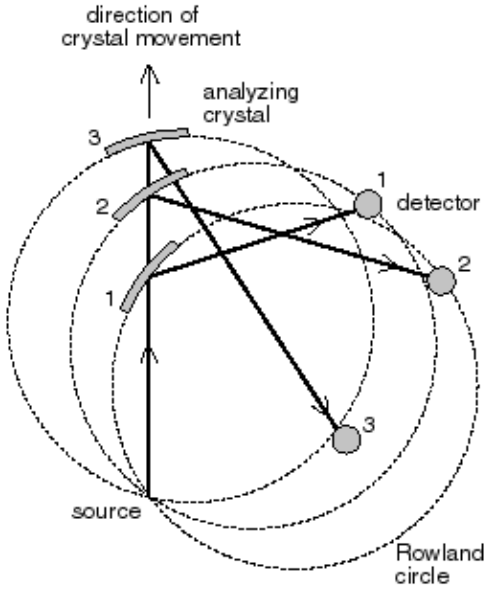
wavelength dispersive system (WDS, WDX)

- Based of wave characteristic of radiation
- Based on X-ray diffraction on a crystal – monochromator
- Radiation source, monochromator and detector lay on a Rowland circle
- If the Bragg equation is comply, a radiation is diffracted towards a detector.
- If not, a radiation is absorbed by a crystal

$$n\lambda = 2d \sin\theta$$

where, n = an integer (1, 2, 3...),
λ = wavelength,
d = d-spacing of the crystal,
and θ = incident angle (measured from crystal surface)





WDS-crystals

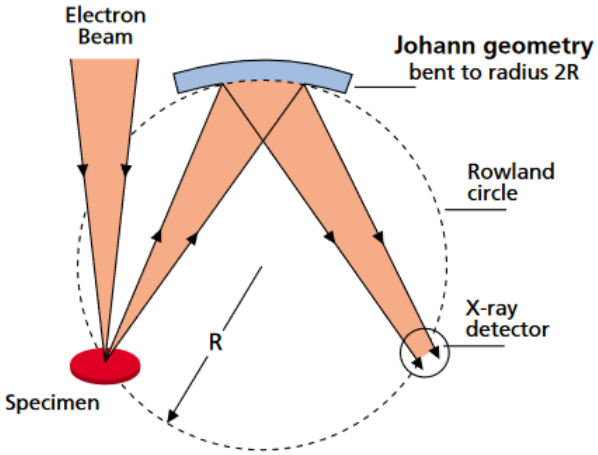


Figure 2a

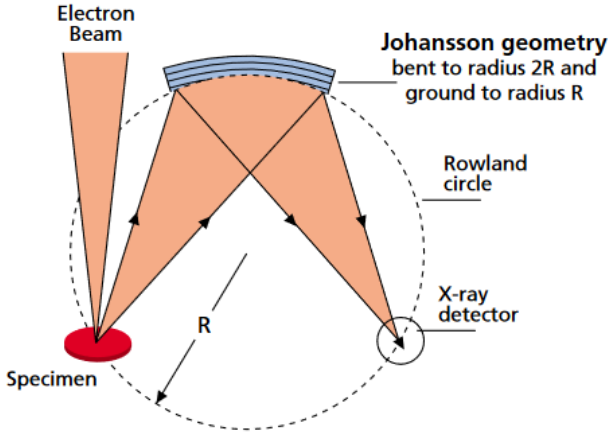
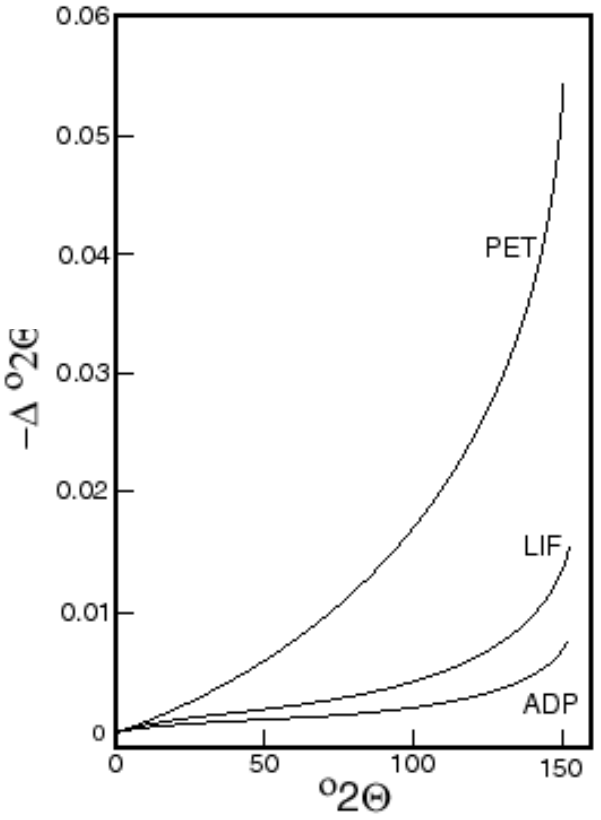


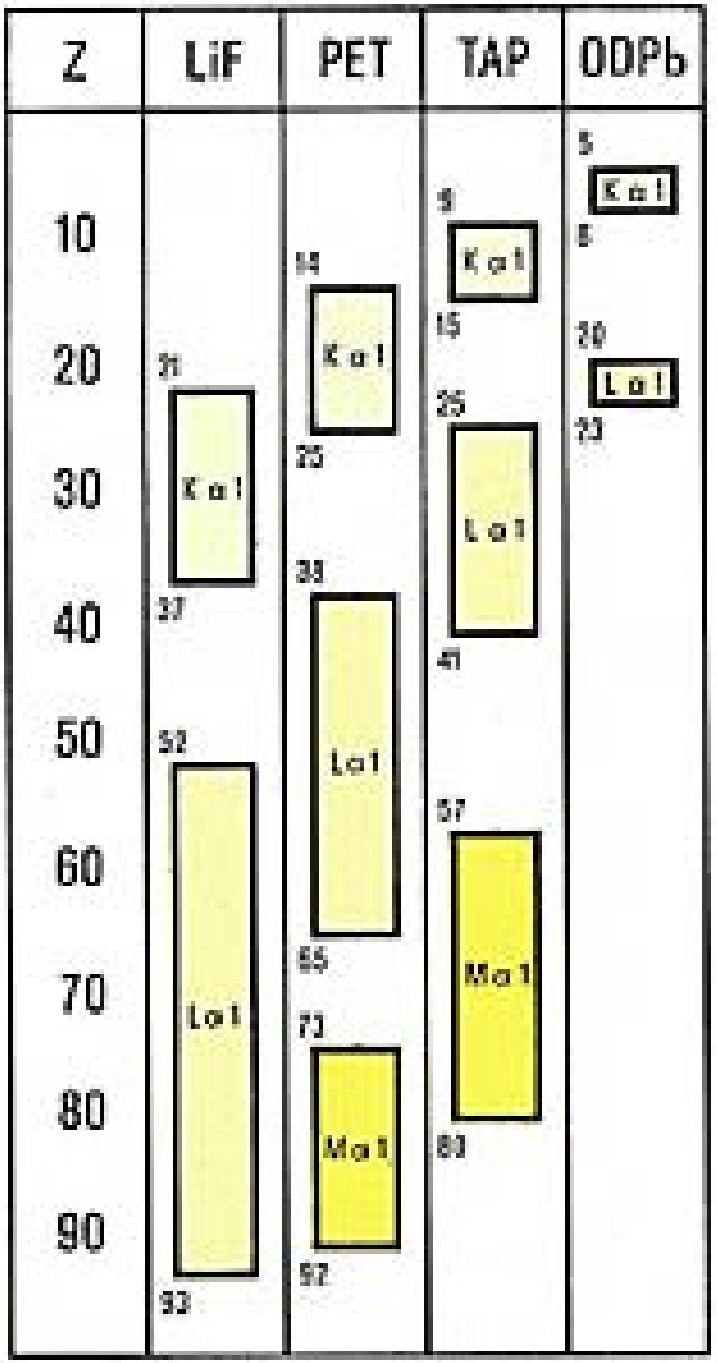
Figure 2b



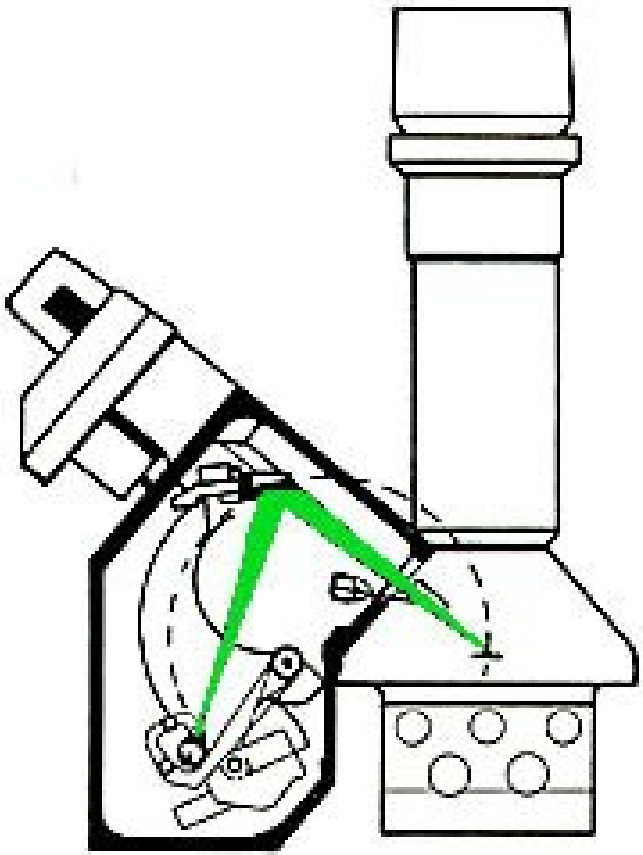
- **sensitive to temperature variation**
  - **changes of diffraction angle**

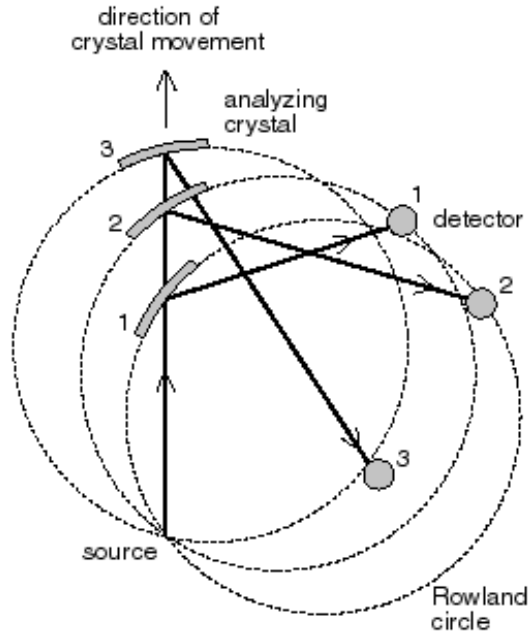
WDS crystals

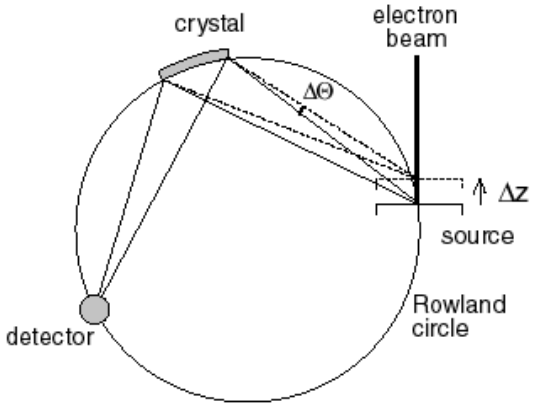
- Lithium fluoride 200 (**LIF**), 2d = 4.028 Å
- Potassium acid pthalate 1011 (KAP), 2d = 26.6 Å
- Ammonium dihydrogen phosphate 011 (ADP), 2d = 10.648 Å
- Rubidium acid pthalate (RAP), 2d = 26.1 Å
- Pentaerythritol 002 (**PET**), 2d = 8.742 Å
- Thallium acid pthalate 1011 (**TAP**), 2d = 25.75 Å, and
- Lead sterate or Lead octodecamoate (ODPB), 2d = 100 Å



WDS – arrangement

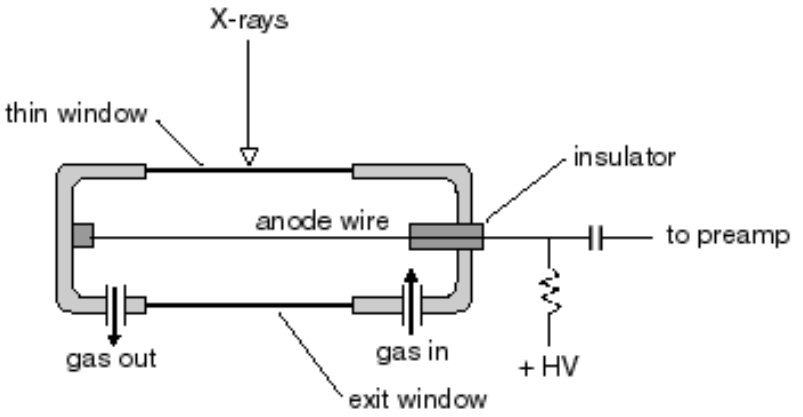


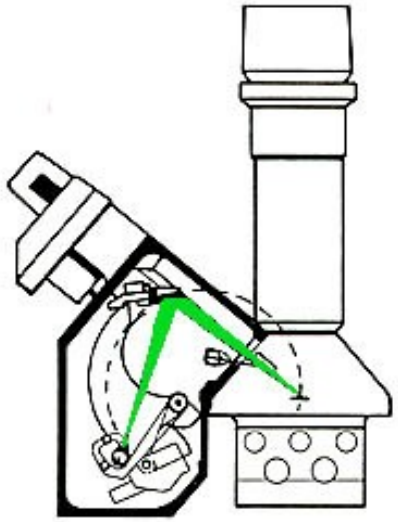


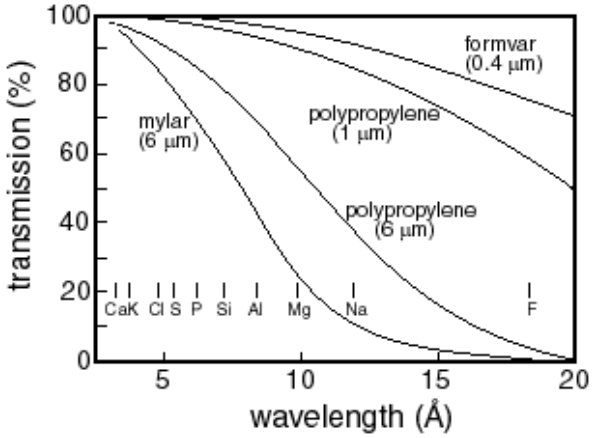


WDS - detector

- Proportional gas detector
- „gas flow“ of sealed type
- argon methane 9:1, krypton
- Diffracted X-ray ionize gas in detector
  - methane works as a discharge quencher

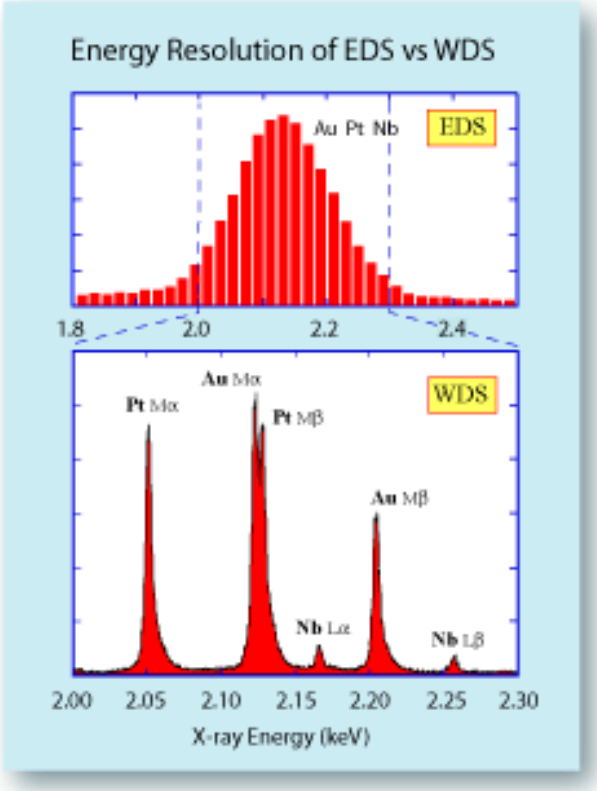






wavelength dispersive system (WDS, WDX)

- advantages
  - Excellent spectral resolution 6 eV per channel
  - Low detection limits 0.0X wt.%
  - Less spectral coincidences
  - Up to 5 spectrometers
- disadvantages
  - Analysis takes more time X minutes
  - Sensitive to quality of the surface
  - More expensive device
  - Measure only selected elements



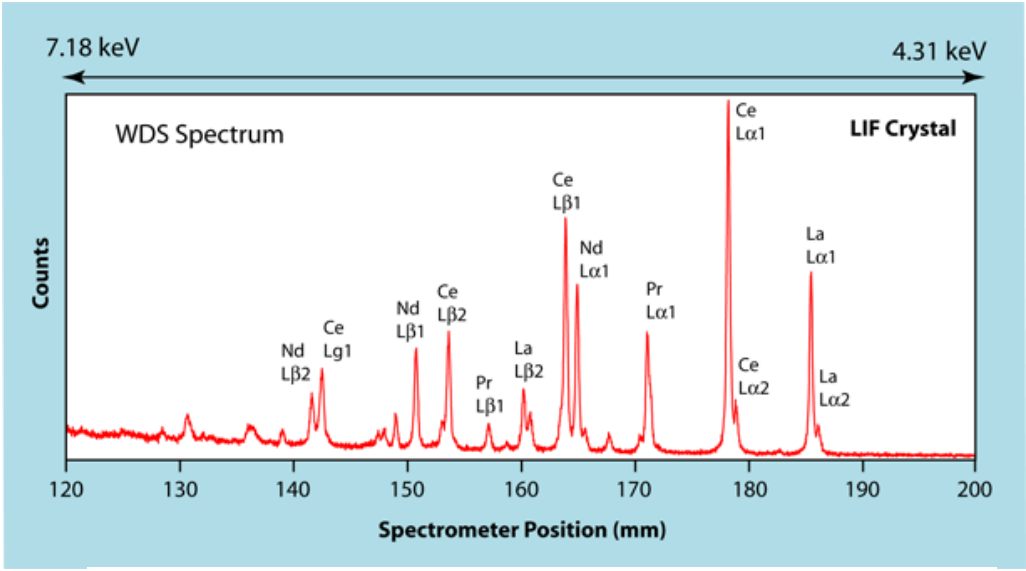
WDS – principles of measurement

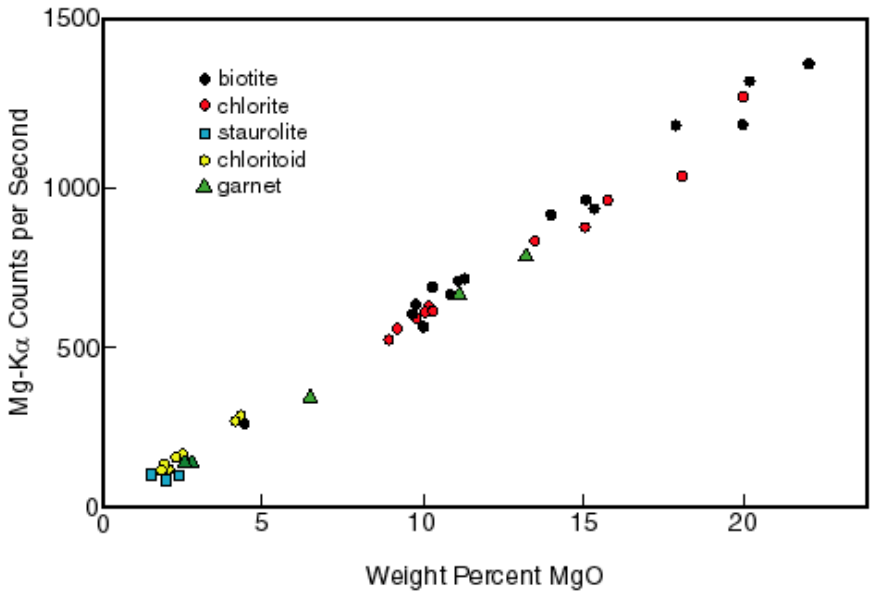
- countrate on the peak maxima
- countrate on background.

- Countrate depends on:
  - concentration, analytical line, accelerating voltage and beam current
  - cts.s^{-1}.nA^{-1} (for certain kV)

- Comparison of countrate (I) of unknown with the countrate of standards – concentration (c)

- $c_{unk}=I_{unk}/I_{std}$ x c_{std}





EDS, WDS - ZAF corrections

- theoretically

$$C^{A}_{unk} = C^{A}_{std}\left(\frac{I^{A}_{unk}}{I^{A}_{std}}\right)$$

where C^{A}_{unk} = concentration of A in the unknown, C^{A}_{std} = concentration of A in the standard, I^{A}_{unk} = the background-corrected intensity of A X-rays in the unknown, and I^{A}_{std} = background-corrected intensity of A X-rays in the standard.

- Z – correction to BSE
  - The more production of BSE, the less electrons can generate characteristic X-ray
  - Production of BSE depends on Z
  - Correction for loss of characteristic X-rays due to production of BSE
- A – characteristic X-rays is partially absorbed by matter of sample
  - absorption depends of X-ray energy and chemical composition of the sample
- F – absorbed X-ray can cause an emission of secondary X-rays of lower energy
  - fluorescence

Sample preparation for electron microscopy and microanalysis

Sample preparation for electron microscopy and microanalysis

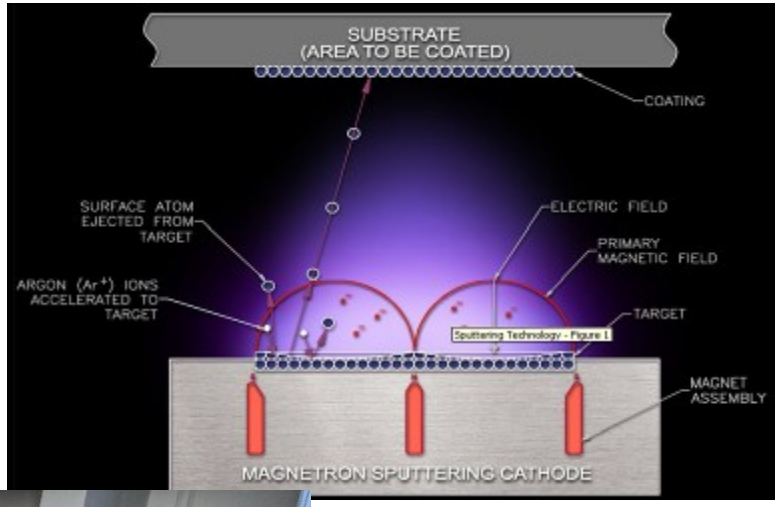
- High vacuum devices
- Sample surface must be electrically conductive
  - For analysis – C
  - For images only – Au, Ir, Pd,…

- High vacuum devices (environmental microscopes)
  - Observe the sample without coating
  - Higher pressure (worse vacuum) in chambre

- For proper microanalysis
  - polished surface perpendicular to the electron beam

Au, Pt, It coating

- For electron microscopy
- Morphological samples
- Magetron sputtering coaterd
- About 20-30 minutes a batch



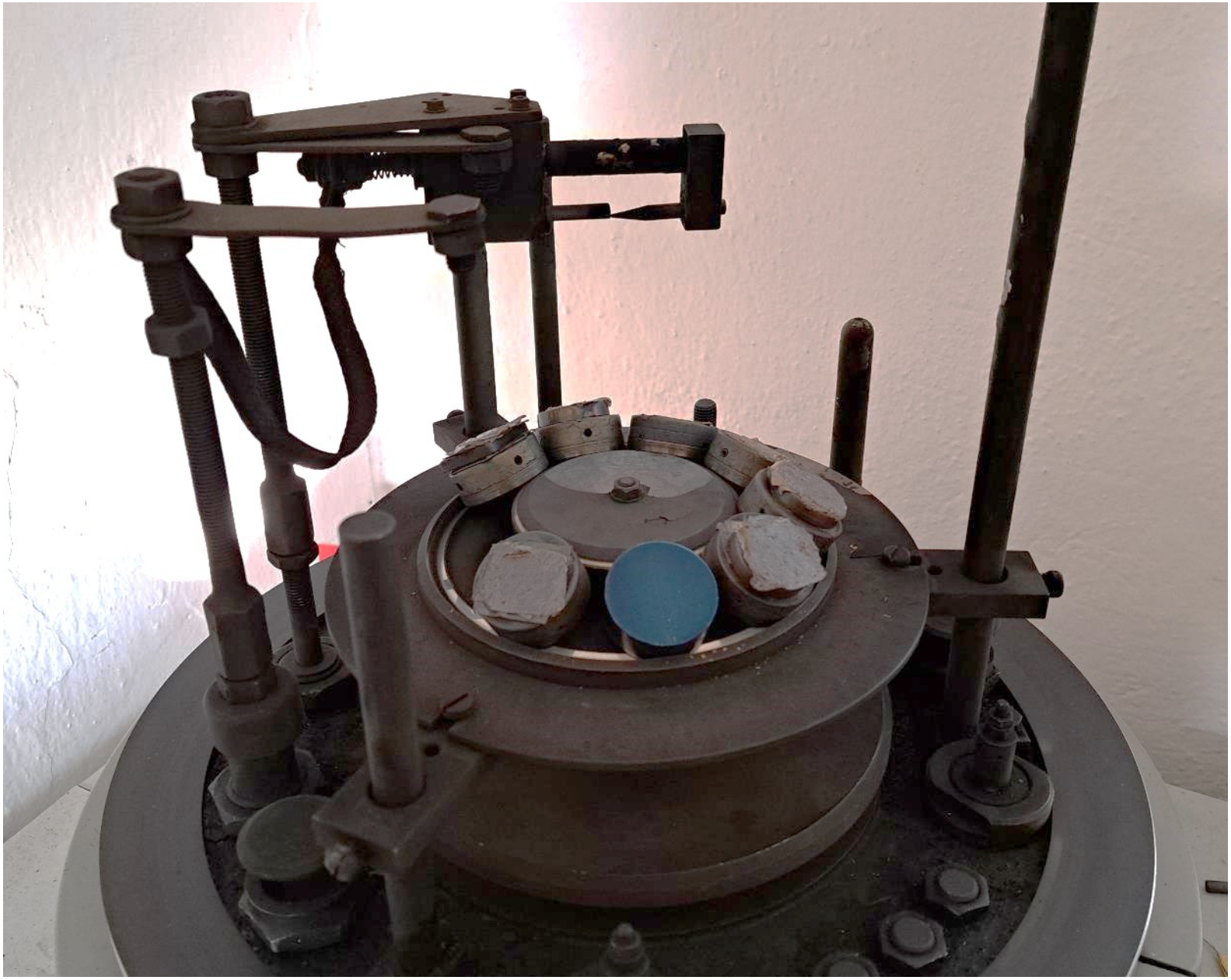
Carbon coating

- Carbon vacuum coater
- For microanalysis
- Selfheated graphite electrods emitte carbon atoms
- Graphite electrods, carbon string
- Batch time 0.5-3 hours.

- Necessary to clean the surface form a dirty and oily stuff

- For high high-quality analysis is necessary make a homogeneous thickness and the same thickness as on standards

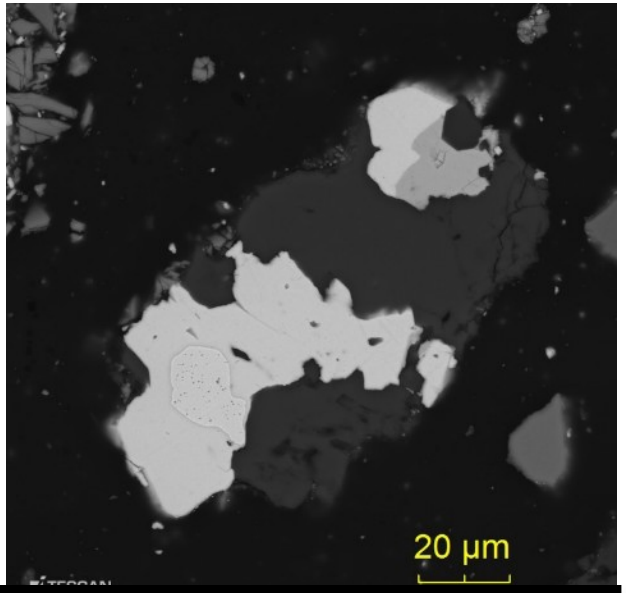
- Carbon layer is sensitive to scratch

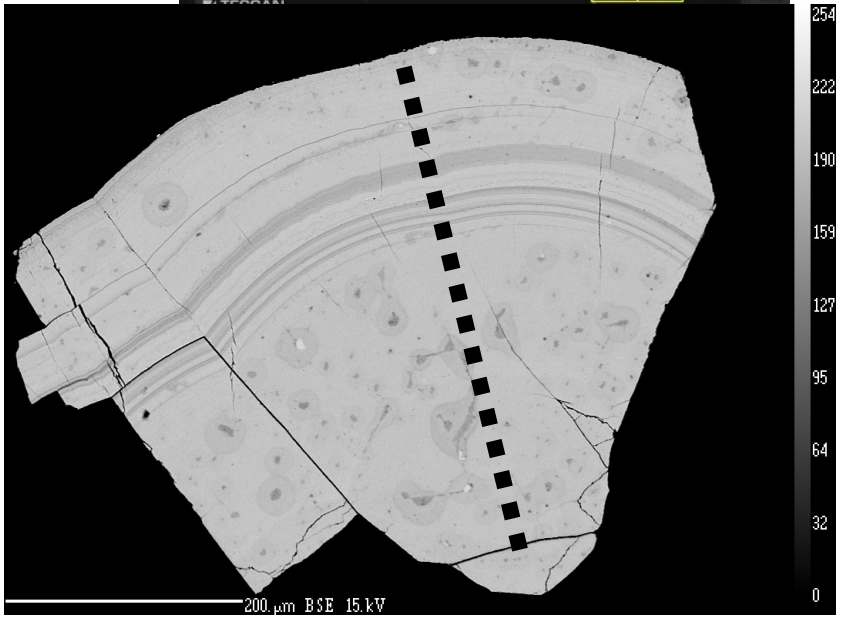


Analytical outputs of Cameca SX 100 electron microprobe

Spot WDS analysis

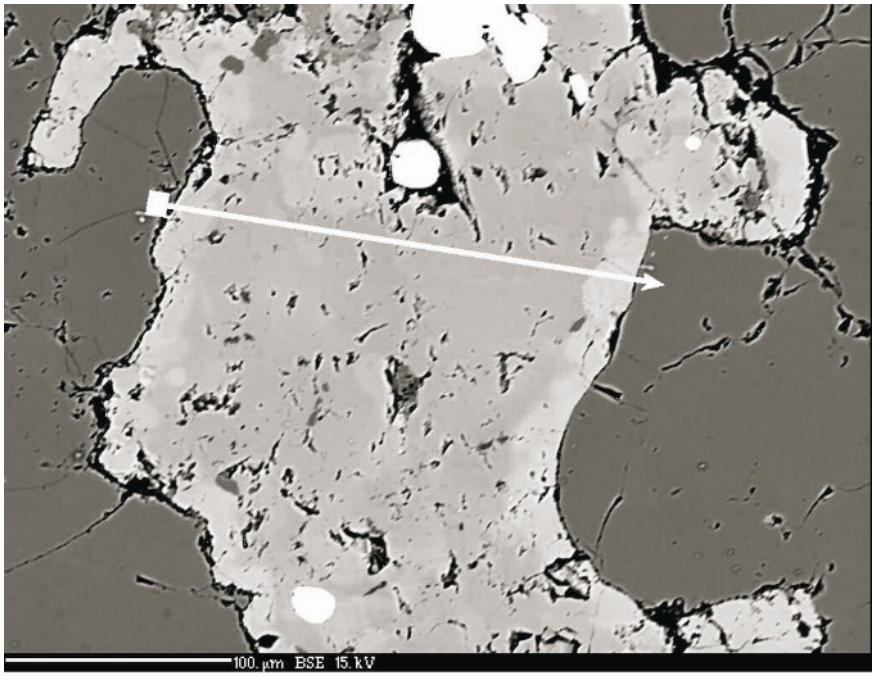
- Minimal grain size (3x3 μm)
- Focused beam ~0,5 μm to a large area analysis 50x50 μm
- 5 elements at once
- Time of analysis 3-20 minutes
- Depends on number of elements and required detection limit.

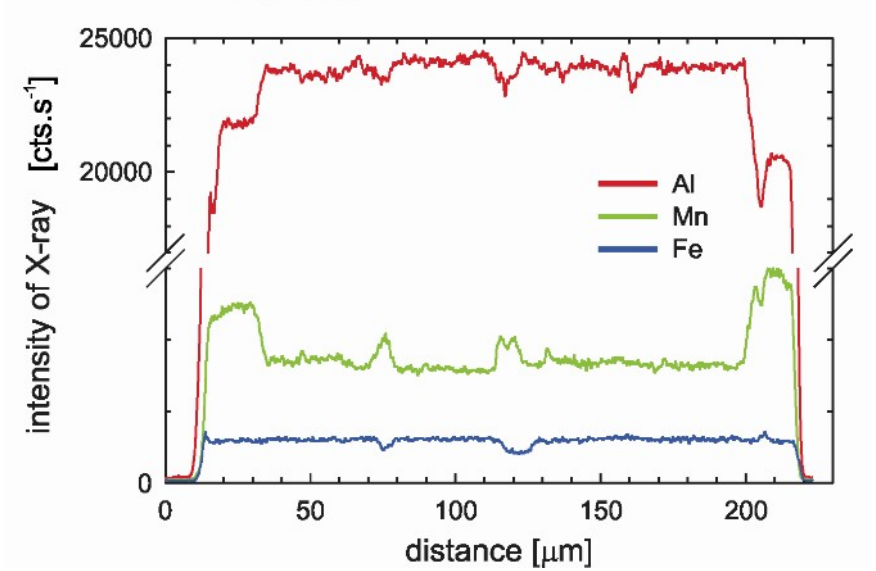




Oxide															
DataSet/Point	TiO2	CaO	Fe2O3	Mn2O3	Na2O	SiO2	Al2O3	MgO	K2O	Cr2O3	BaO	P2O5	Total	Comment	Date
1 / 1 .	0.081	0	4.308	16.59	0.004	34.818	44.17	0.112	0	0.004	0	0.008	100.102	koj5	5/5/2006 9:32
2 / 1 .	0.083	0.017	4.352	17.06	0	34.252	43.579	0.118	0.001	0.013	0.001	0	99.49	koj5	5/5/2006 9:36
3 / 1 .	0.06	0	4.32	17.434	0.041	34.296	43.955	0.105	0.012	0.007	0.012	0.032	100.275	koj5	5/5/2006 9:41
4 / 1 .	0.061	0.003	4.265	16.478	0.028	34.058	44.28	0.124	0	0.005	0	0.017	99.361	koj5	5/5/2006 9:55
Det.Lim ppm															
DataSet/Point	Tit	Ca	Fe	Mn	Na	Si	Al	Mg	K	Cr	Ba	P	O	Comment	Date
1 / 1 .	220	381	887	662	471	434	437	204	1	280	696	374		koj5	5/5/2006 9:32
2 / 1 .	209	335	774	626	482	394	431	217	326	272	643	341		koj5	5/5/2006 9:36
3 / 1 .	219	344	848	611	428	402	428	215	299	268	650	319		koj5	5/5/2006 9:41
4 / 1 .	225	362	845	675	452	430	420	214	312	275	679	347		koj5	5/5/2006 9:55
StdDev wt%															
DataSet/Point	Tit	Ca	Fe	Mn	Na	Si	Al	Mg	K	Cr	Ba	P	O	Comment	Date
1 / 1 .	0.02	0.031	0.244	0.378	0.039	0.363	0.551	0.022	0	0.023	0.055	0.031		koj5	5/5/2006 9:32
2 / 1 .	0.019	0.029	0.237	0.378	0.038	0.352	0.539	0.023	0.027	0.023	0.053	0.028		koj5	5/5/2006 9:36
3 / 1 .	0.02	0.028	0.238	0.384	0.04	0.353	0.543	0.022	0.026	0.022	0.054	0.028		koj5	5/5/2006 9:41
4 / 1 .	0.02	0.03	0.242	0.377	0.04	0.358	0.552	0.023	0.026	0.023	0.055	0.03		koj5	5/5/2006 9:55

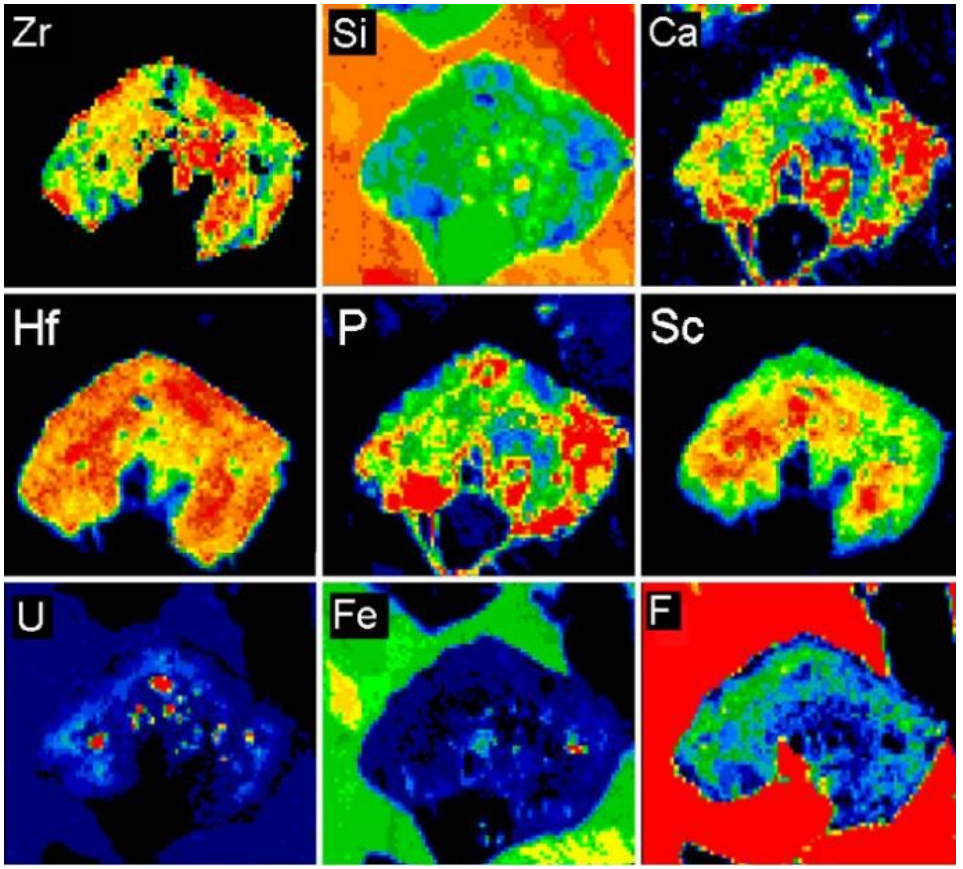
Line profile





X-ray mapping

	10ms	100ms	1000ms
64x64	45s	6m 30s	1h 8m 16s
128x128	2m 44s	27m 20s	4h 33m 4s
256x256	10m 15s	1h 49m 13s	18h 12m 16s
512x512	43m 4s	7h 16m 54s	72h 49m 4s



Angle WDS scan