SEM – Extended facilities

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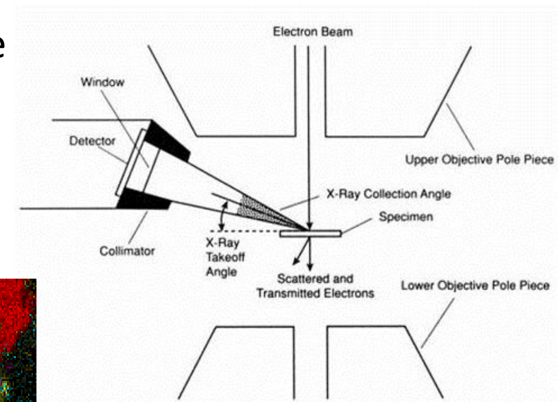
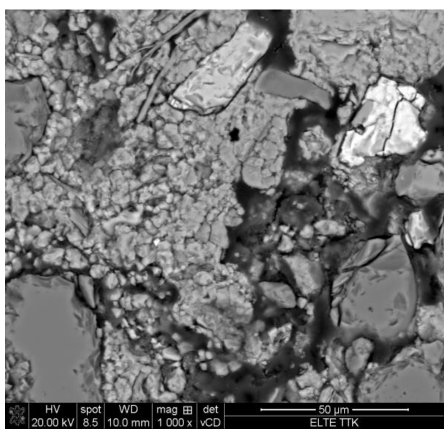
Electron Microscopy (EDS / EDX)

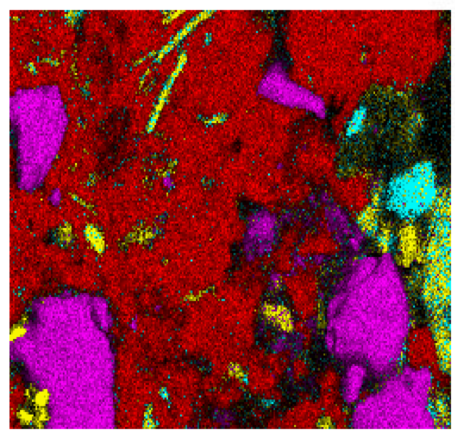
Detection of X-ray photons

- An X-ray detector is used to detect X-ray photons emitted by an electron beam induced excitation of inner shells of the atoms.
- The energy of an X-ray photon is specific to the excited atom.
- The resulting spectrum therefore reflects the atomic composition of the sample.
- This energy is the basis for dispersive X-ray microanalysis
  (energy dispersive X-ray microanalysis = EDX).



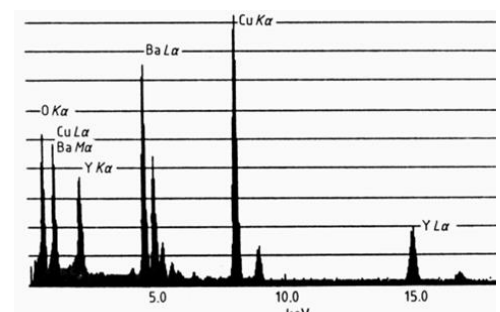


backscattered electron image



X-ray colored map

elemental mapping (root fossilisation)



$YBa_2Cu_3O_7$ high-temperature superconductor sample EDX spectrum

Energy dispersive X-ray elemental analysis

X-ray spectroscopy - a basic phenomenon

Excitation of **inner shells** (core electrons).

- It requires energy on the order of hundreds of *eV - keV*.
  (The energy for e.g. *Cu K* shell is ~ *9 keV*.)

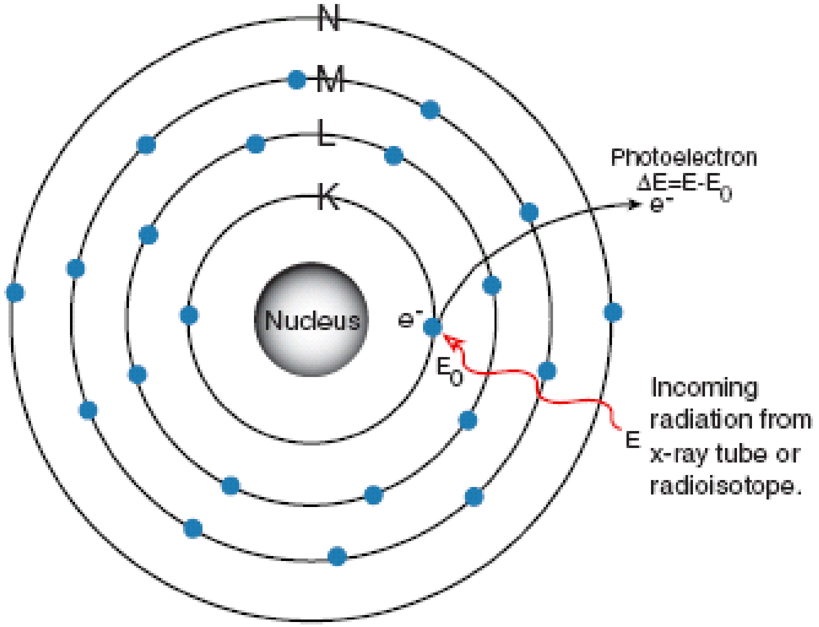
Notation:

n = 1, 2, 3, ... principal quantum number is replaced by *K, L, M, …*

l = 1, 2,...n-1 azimuthal quantum number (orbital momentum) *s, p, d, f, …*

The energy of the electron also depends to a small extent on the spin momentum (*s*). The value characterizing the total pulse moment:

$j = 1/2, 3/2, ...$



Energy dispersive X-ray elemental analysis

X-ray spectroscopy - a basic phenomenon

Excitation of **inner shell**s (core electrons).

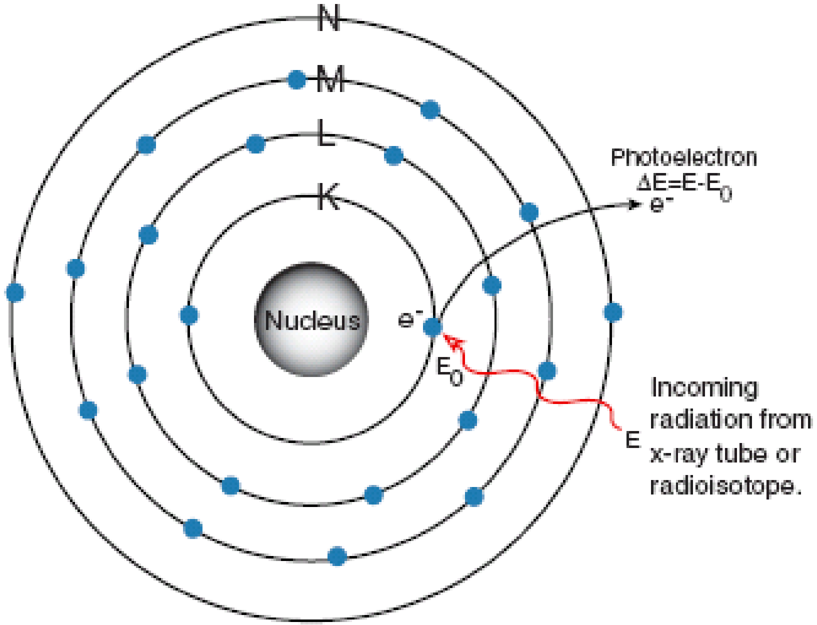
Selection rule for l and j:

$$\Delta l = \pm 1; \quad \Delta j = 0, \pm 1$$

$K_{\alpha 1}$ line: L_3 -> K
$K_{\alpha 2}$ line: L_2 -> K

K_b line: $M_{2,3}$ -> K

$L_{\alpha 1}$ line: M_3 -> L_1
…



Excitation:

- min. ~$1.6 \times E_0$
- an electron is ejected
- rearrangement energy
- difference -> photon

Energy dispersive X-ray elemental analysis

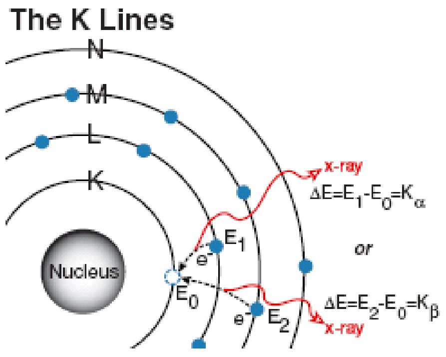
X-ray spectroscopy - a basic phenomenon

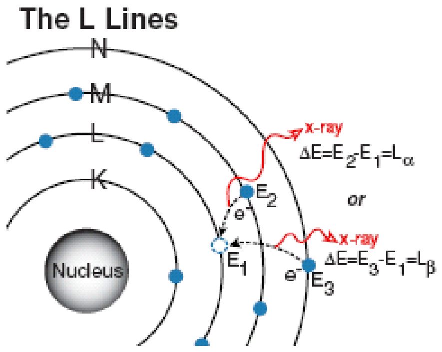
If the electron created on the **K** shell is a vacancy from the **L** shell is loaded: K_α line. (*Cu* K_α *E ~ 8 keV*).

If the electron created on the **K** shell is a vacancy from the **M** shell is loaded: K_β line. (*Cu* K_β *E ~ 8.9 keV*).

Then a vacancy occurs on the L shell, which is filled from a higher shell, etc.
This is how the spectrum characteristic of an atom is created, which is suitable for its identification.

(If the energy generated during the filling of the electron vacancy does not leave the atom in the form of a photon, but is transferred to an electron in the outer shell, then this electron is ejected, this is the Auger electron.
KL_1L_{23} and **MNN** transitions are common.)





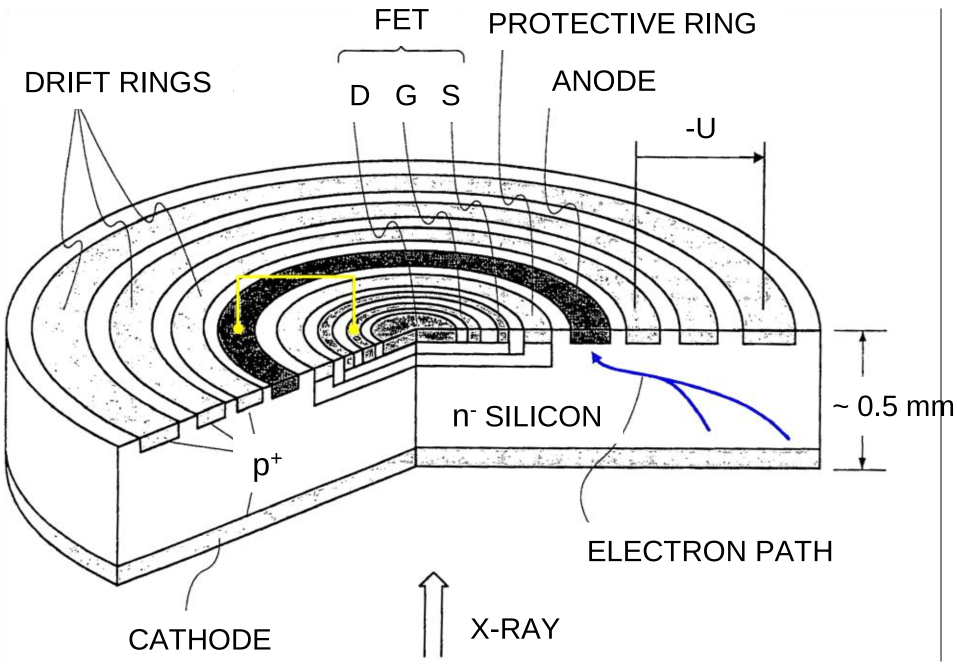
Energy dispersive X-ray elemental analysis

X-ray spectroscopy - detection

Silicon Drift Detector (SDD - Szilícium drift detektor)

Base material: ***n***-type doped semiconductor from high-purity *Si* single crystal.
There is **NO any *Li*** doping!

- A continuous ***p***-type layer on one surface and
- rings on the other side
  -> shaped potential field
- Absorbed photons create electron-hole pairs
- Electrons migrating inwards enter the FET formed in the middle.
- Small anode
  -> fast (800,000 cps)
  -> low dead time
  -> quick analysis



Energy dispersive X-ray elemental analysis

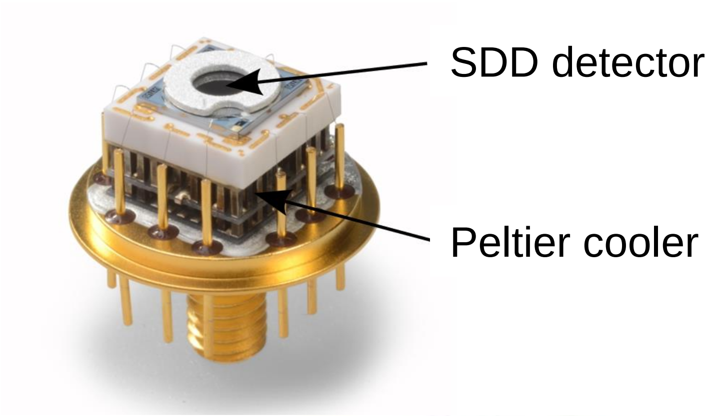
X-ray spectroscopy - detection

Silicon Drift Detector (SDD - Szilícium drift detektor) II.

- can be stored at room temperature
- can be operated near room temperature
- Peltier cooling for noise reduction
- large surface area, large solid angle
- at moderate beam intensity: it can already be analyzed with the imaging beam
- the wearing and damaging effect of the e-beam is small
- intense electron beam: fast, accurate analysis,
- element map

- *ΔE/E = 130 eV / 5899 eV* (*Mn Kα*)

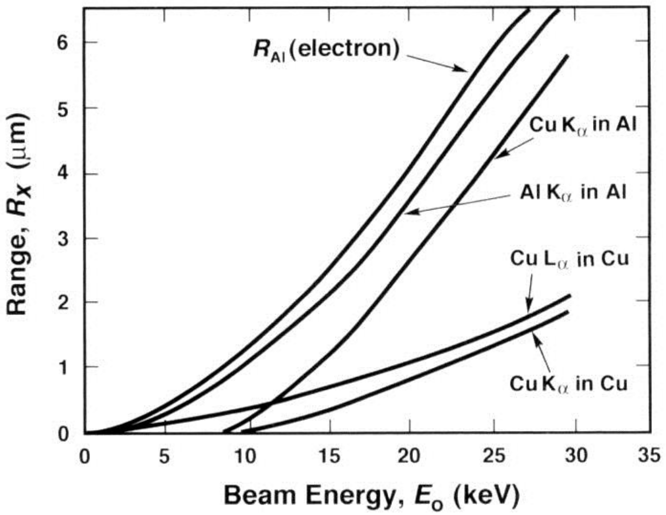
ΔE/E = **2.2 %**

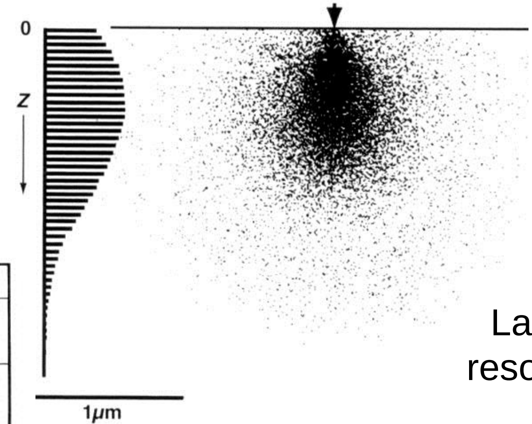


Ametek EDAX „Apollo X”

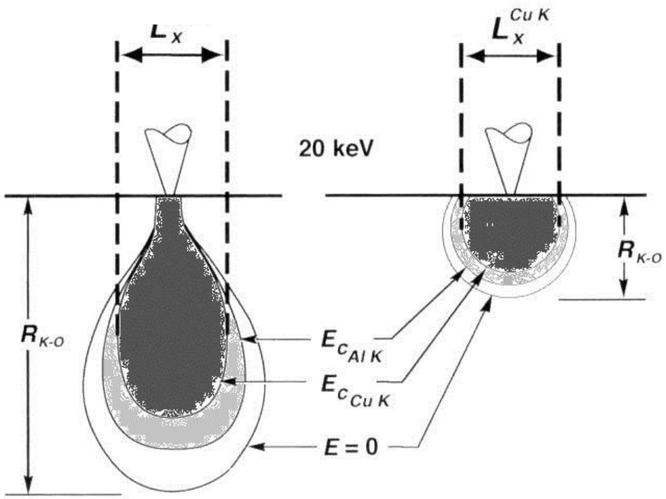
EDX elemental analysis – Resolutions

information depth





Lateral resolution



Anderson – Hasler

$$R_{X\text{-}Ray} = 0{,}064 \cdot (E_0^{1{,}68} - E_c^{1{,}68})/\rho$$

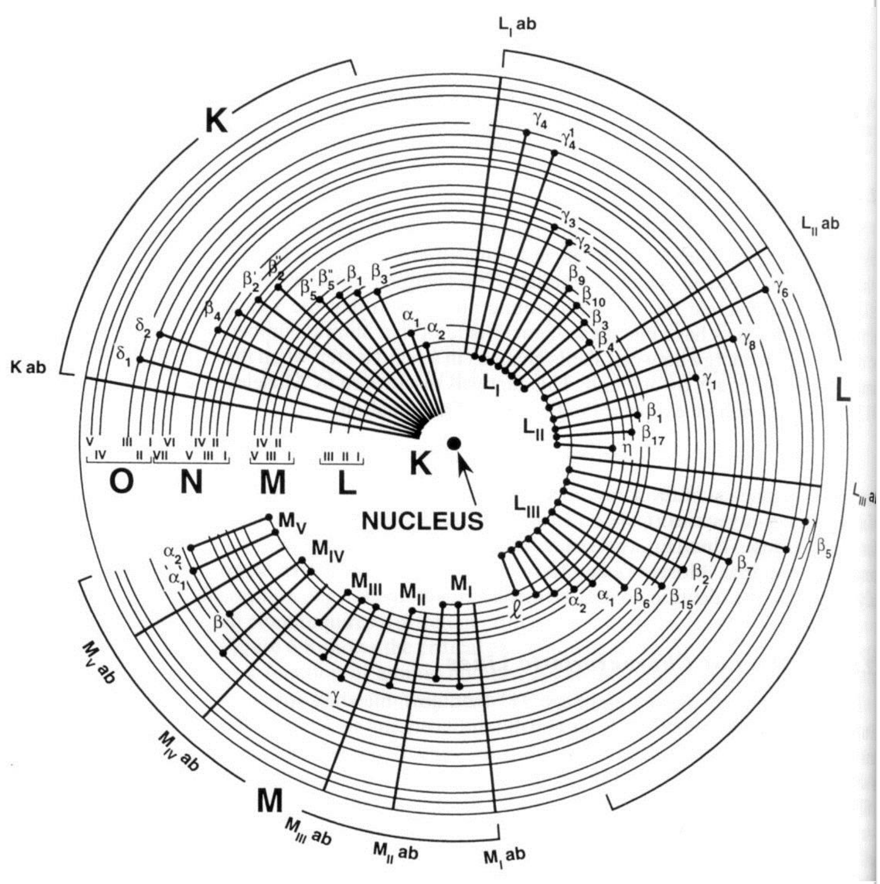
[μm] [keV] [g/cm^3]

Cu atoms in Al substrate

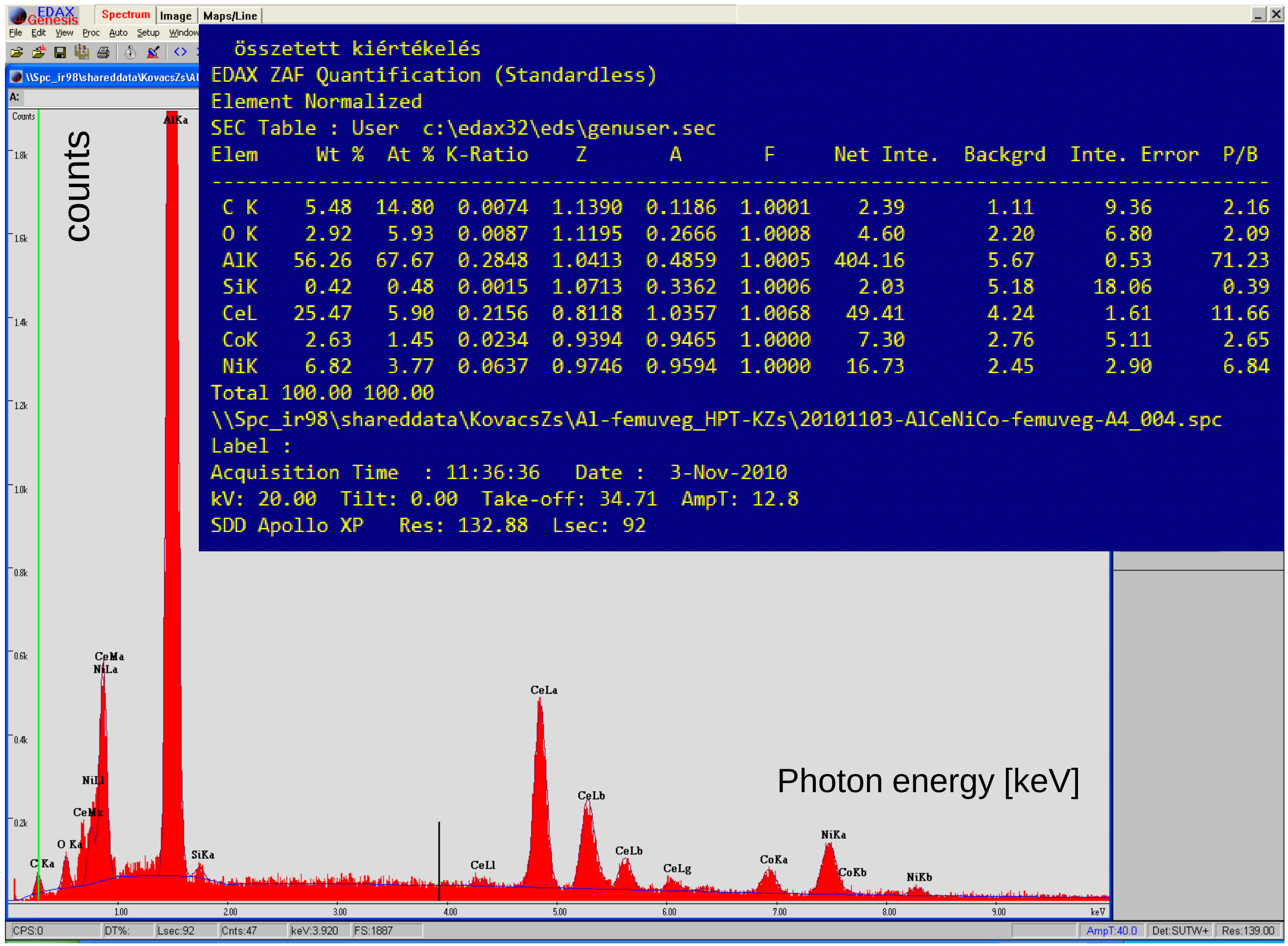
Al atoms in Cu substrate

EDX elemental analysis – Data processing

- measurement of the energy of the generated X-ray photons: SDD (spectral line location)
- count (spectral line height)
- spectrum analyzer program data storage and data processing
- the number of photons is directly proportional to the concentration of excited atoms (good approximation)
- the result can be displayed on a computer screen



EDX elemental analysis – Example

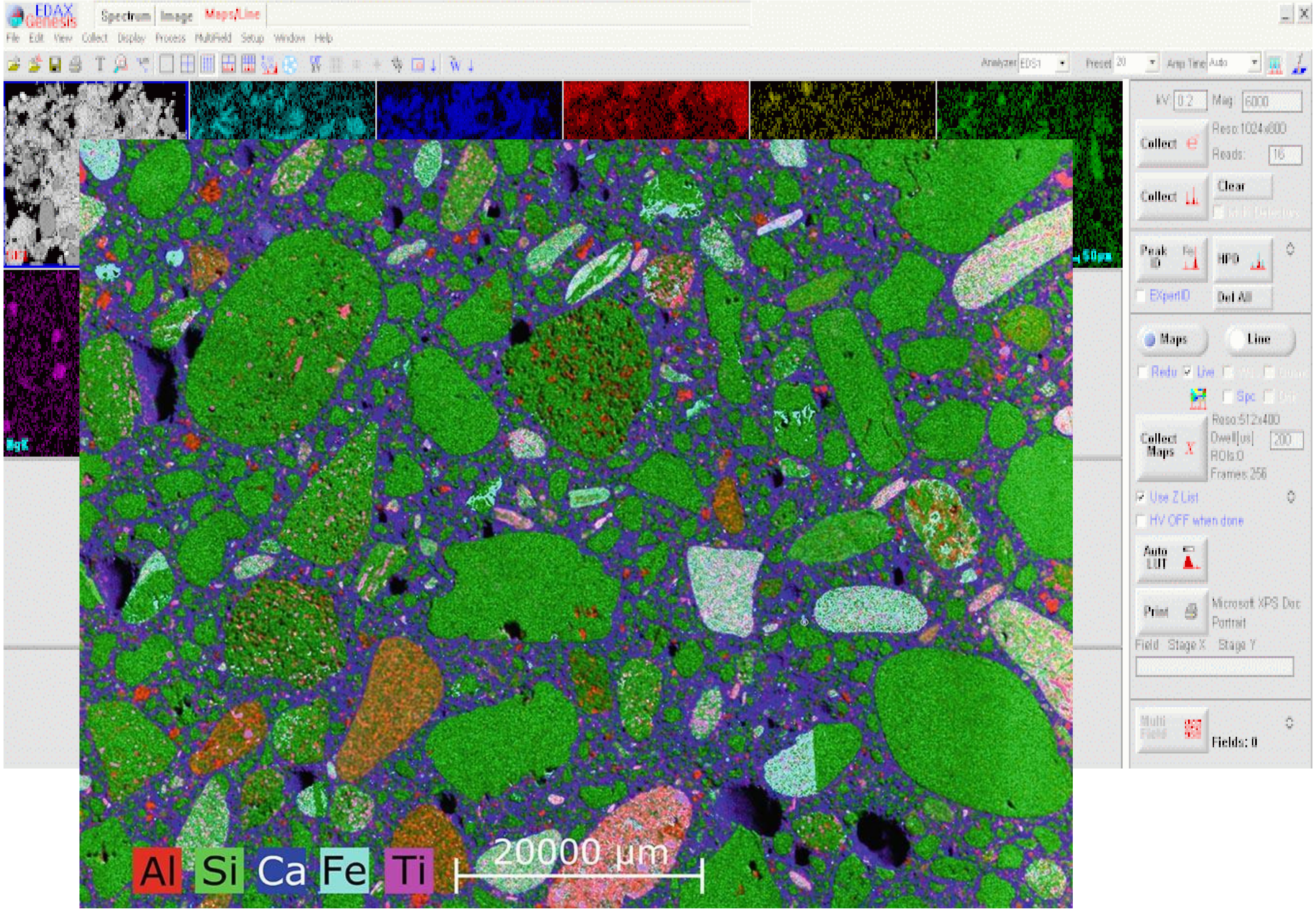


összetett kiértékelés
EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : User c:\edax32\eds\genuser.sec

Elem	Wt %	At %	K-Ratio	Z	A	F	Net Inte.	Backgrd	Inte. Error	P/B
C K	5.48	14.80	0.0074	1.1390	0.1186	1.0001	2.39	1.11	9.36	2.16
O K	2.92	5.93	0.0087	1.1195	0.2666	1.0008	4.60	2.20	6.80	2.09
AlK	56.26	67.67	0.2848	1.0413	0.4859	1.0005	404.16	5.67	0.53	71.23
SiK	0.42	0.48	0.0015	1.0713	0.3362	1.0006	2.03	5.18	18.06	0.39
CeL	25.47	5.90	0.2156	0.8118	1.0357	1.0068	49.41	4.24	1.61	11.66
CoK	2.63	1.45	0.0234	0.9394	0.9465	1.0000	7.30	2.76	5.11	2.65
NiK	6.82	3.77	0.0637	0.9746	0.9594	1.0000	16.73	2.45	2.90	6.84
Total	100.00	100.00								

\\Spc_ir98\shareddata\KovacsZs\Al-femuveg_HPT-KZs\20101103-AlCeNiCo-femuveg-A4_004.spc
Label :
Acquisition Time : 11:36:36 Date : 3-Nov-2010
kV: 20.00 Tilt: 0.00 Take-off: 34.71 AmpT: 12.8
SDD Apollo XP Res: 132.88 Lsec: 92

EDX analysis – Element mapping

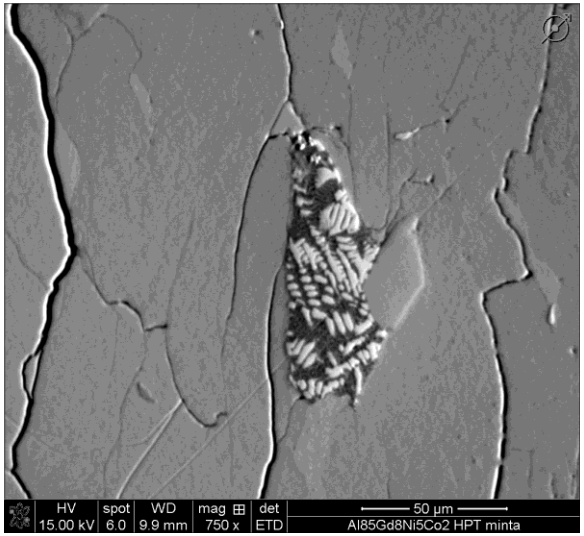


EDX analysis – Limitations, artifacts

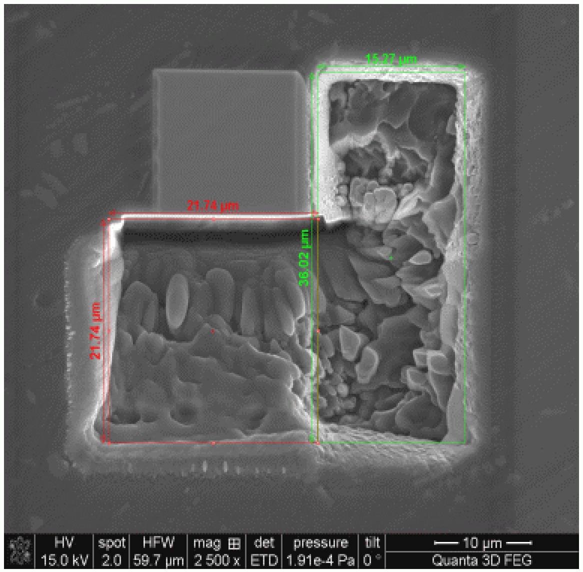
- nearby peaks – overlapping in real detectors
  - literature example: PtAuNb alloy *2.05 ... 2.25 keV*
- "ghost" peaks at energy sums
  - reason: superimposed impulses
  - avoidance (reduction): counting dead time, software recognition
- inhomogeneous patterns, shaded areas
  - rough, hollow surface, porous material
- wrong peak identification
  - if we rely on the built-in software without criticism
- omitted elements
  - we can measure from beryllium

EDX analysis – Field of application

- Industry / research
  - minerals
  - metals and alloys
  - ceramics
  - glass
- A unique composition of grains of a few *µm* ... a few *mm*
  - research + development,
  - quality control,
  - error analysis,
  - more recently error correction
- Semiconductor production and development
  - ∞ bibliography



EDX analysis – Dual-beam specialities: FIB + EDS



1

depth EDS map

2

ion beam excitation:
side effect: ion sputtering
(non-destructive)