PROCESS CHEMISTRY CENTRE

ÅBO AKADEMI UNIVERSITY

Nordic Course in Analytical Tools for Combustion

SEM/EDX

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The SEM at Åbo Akademi Univeristy

Electrone microscope vs light microscope

- Maximum magnification with light microscope limited by the the wavelength of light (max 2 000)
- With electrone microscopes possible magnifications up to 2 000 000 times (TEM)
- With scanning electrone microscope magnifications up to 200 000 times (possible to discern particles of a few nanometers)
- Acuity depth is ~1000 times better than with light microscopes

The SEM

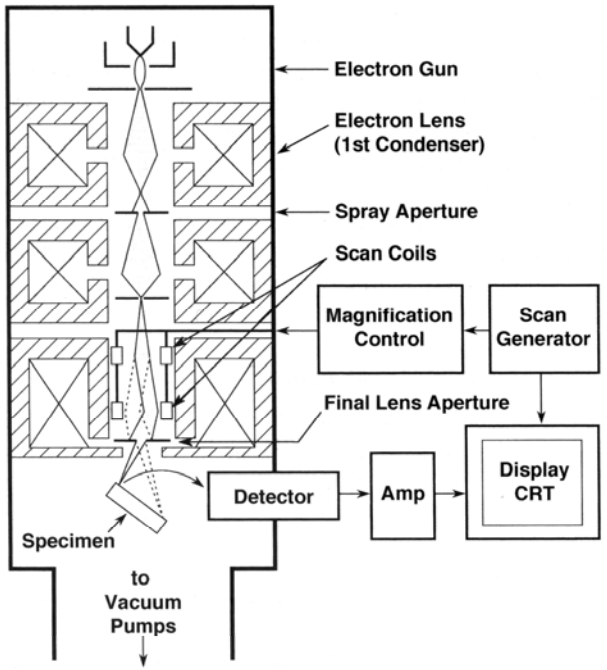


Figure 2.2. Schematic drawing of the electron column showing the electron gun, lenses, the deflection system, and the electron detector.

- The most important parts
  - Electron source
  - Column (electron optics)
  - Sample chamber
  - Vacuum pumps
  - Detectors

The idea behind the SEM

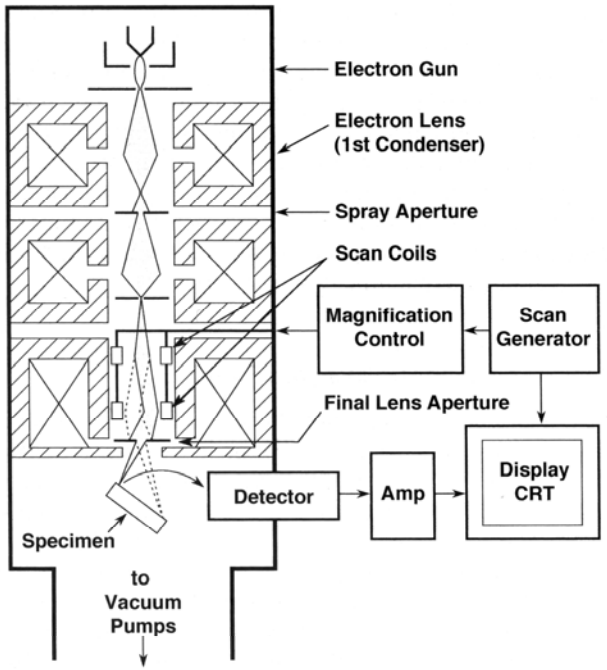
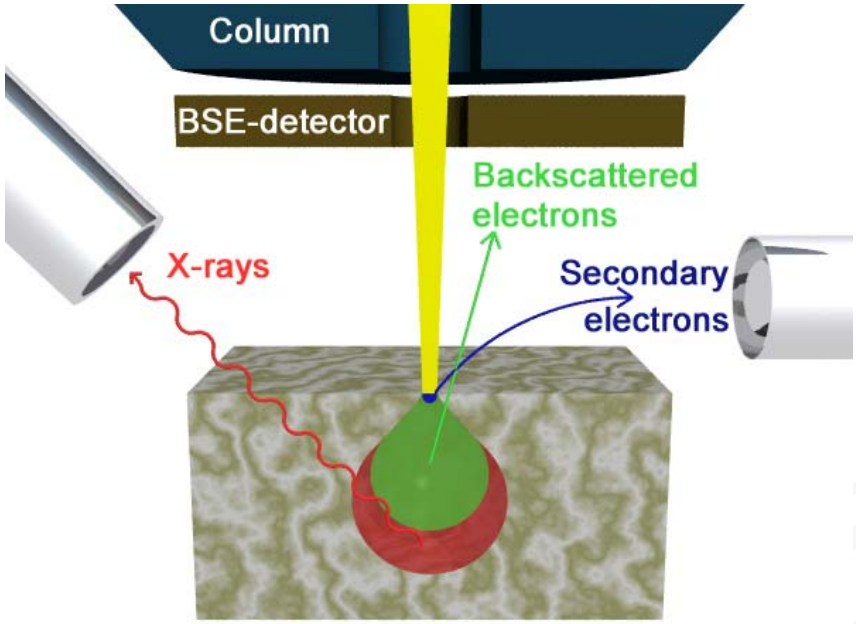


Figure 2.2. Schematic drawing of the electron column showing the electron gun, lenses, the deflection system, and the electron detector.

- SEM stands for Scanning Electron Microscope
- The electrons are generated at the electron gun at the top of the column
- The electron beams strength and focus are controlled in the column by electromagnets and apertures. The scan is also generated here
- The electrons interract with the atoms in the sample
- The products of the interactions are measured and analyzed

The detectors



- The most commonly measured signals
  - Secondary electrons (SE)
  - Backscattered electrons (BSE)
  - X-rays

The sample

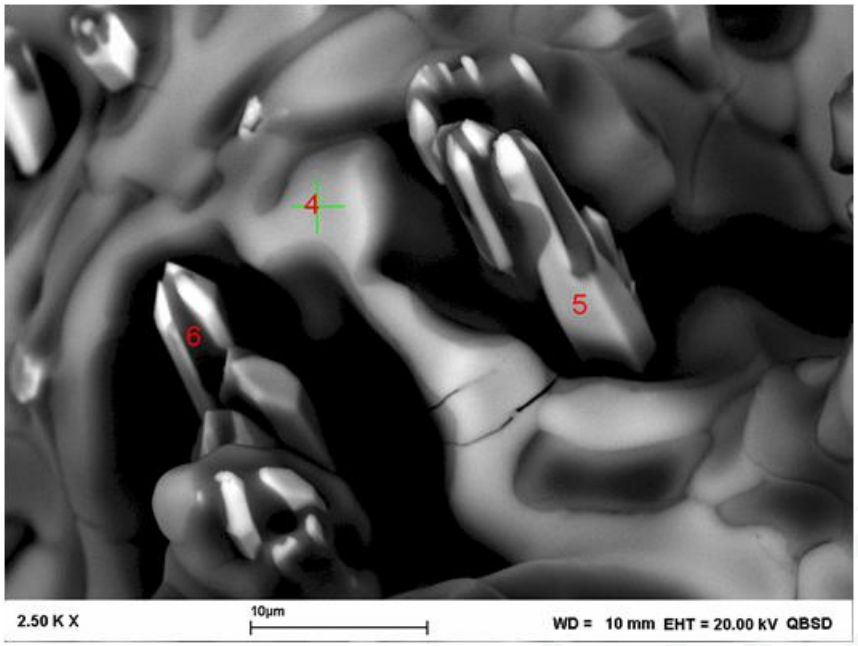
- For a high vacuum SEM the sample has to be
  - Solid
  - Dry
  - Conducting or be made conducting
  - Free from fats and oils
- Non-conducting samples start to charge and will break and xxxx the electron beam
- Can be made conducting by sputtering with, for example, carbon or gold
- Is mountable on the sample table

Energy Dispersive Spectrometry

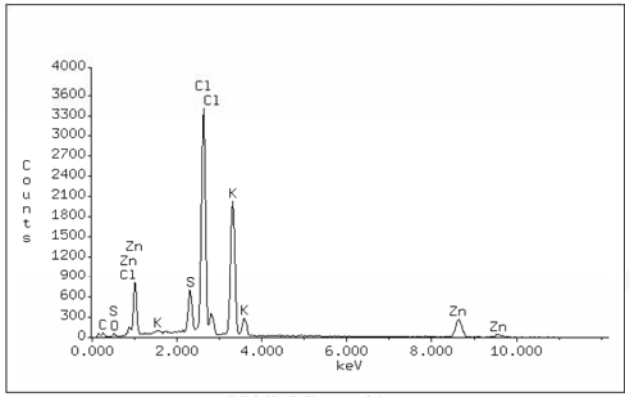
EDXA

- Short for Energy Dispersive X-ray Analysis
- The X-rays generated in the sample by the electron beam are measured for their energy
- Used for quantitative and qualitative measurement of the elements
- A powerful tool as you can choose what to analyze in the SEM

EDXA – Point analysis



ThermoNORAN



DB068r 2.5kx spot04

Accelerating Voltage: 20 KeV
Take Off Angle: 34.9912°
Live Time: 20 seconds
Dead Time: 13.814

Quantitative Analysis

Tue Apr 29 18:14:04 2008

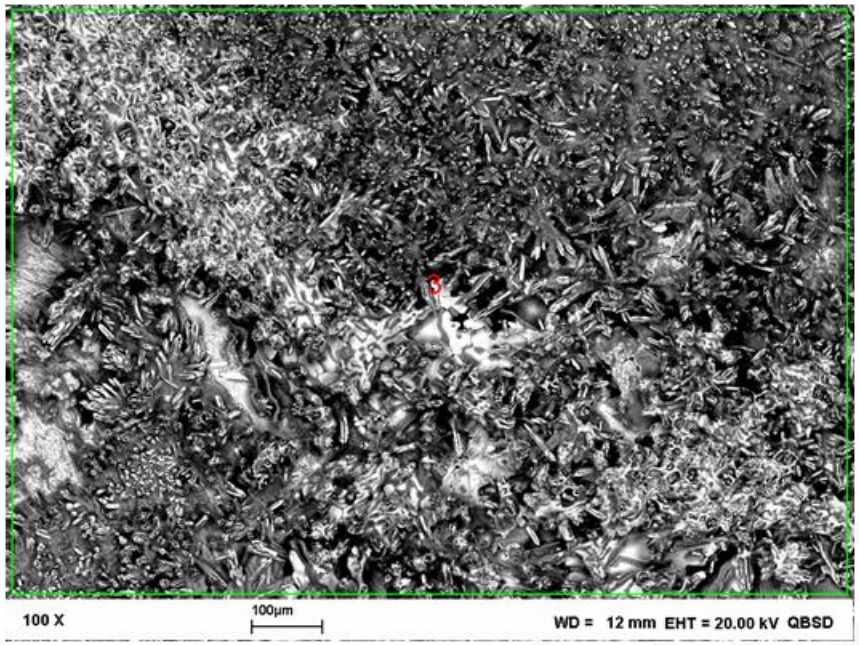
DB068r 2.5kx spot04

PROZA Correction Acc.Volt.= 20 kV Take-off Angle=34.99 deg
Number of Iterations = 5

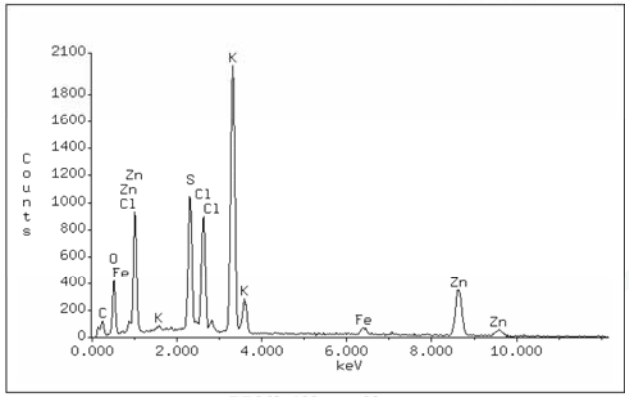
Element	k-ratio (calc.)	ZAF	Element Wt %	Atom %	Rel. Err. (1-Sigma)	No. of Cations
O -K	0.0050	8.168	4.11	9.89	15.74%	---
S -K	0.0517	1.129	5.84	7.02	2.54%	17.027
Cl-K	0.3095	1.172	36.27	39.40	0.84%	95.565
K -K	0.2371	1.280	30.34	29.89	0.97%	72.501
Zn-K	0.2072	1.131	23.43	13.80	3.38%	33.485
Total			100.00	100.00		218.578

The number of cation results are based upon 24 Oxygen atoms

EDXA – Area analysis



ThermoNORAN



DB068r 100x area03

Accelerating Voltage: 20 KeV
Live Time: 35 seconds

Take Off Angle: 37.8028°
Dead Time: 14.649

Quantitative Analysis

Tue Apr 29 18:13:33 2008

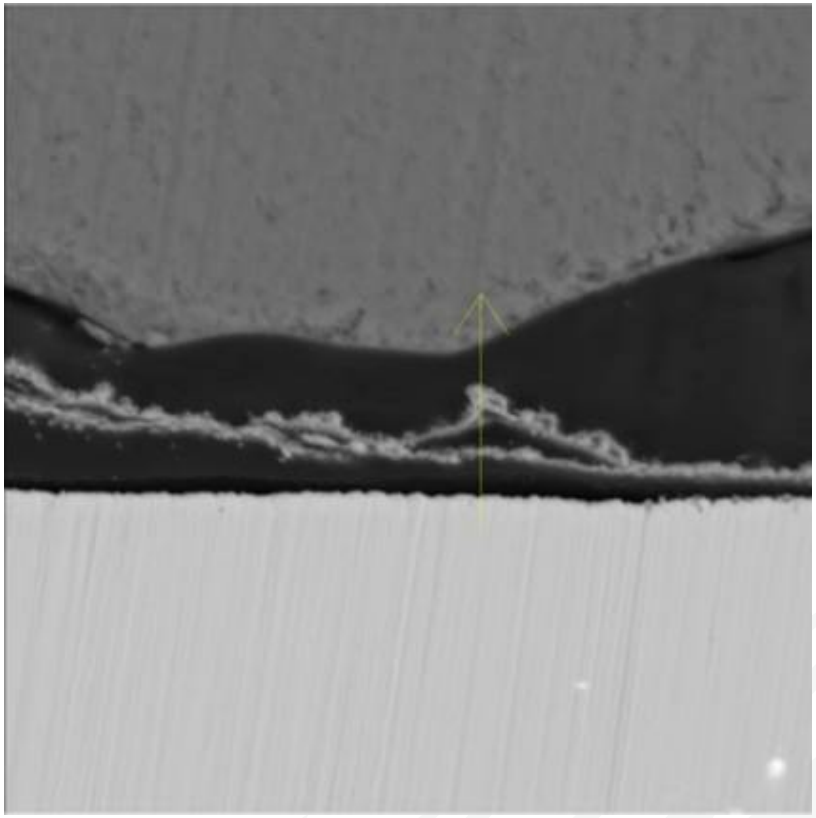
DB068r 100x area03

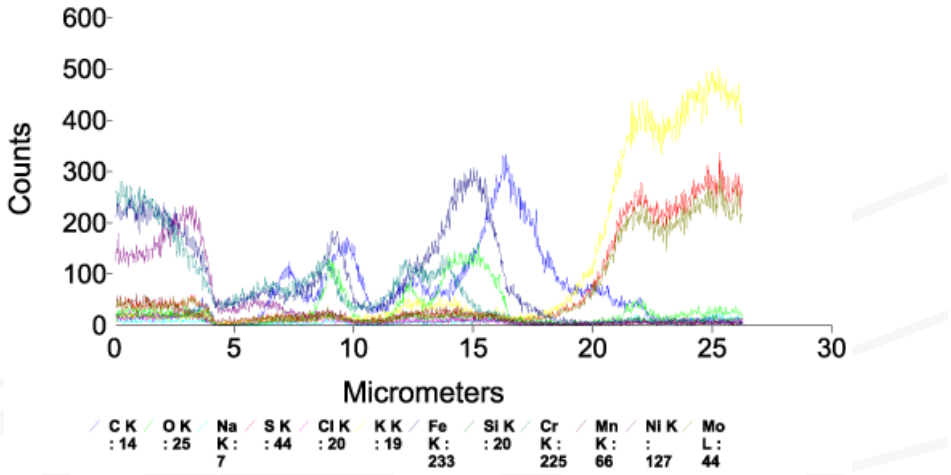
PROZA Correction Acc.Volt.= 20 kV Take-off Angle=37.80 deg
Number of Iterations = 5

Element	k-ratio (calc.)	ZAF	Element Wt %	Atom %	Rel. Err. (1-Sigma)	No. of Cations
O -K	0.0475	5.589	26.52	50.05	2.81%	---
S -K	0.0774	1.236	9.57	9.01	1.05%	4.320
Cl-K	0.0718	1.278	9.18	7.82	1.97%	3.750
K -K	0.2095	1.186	24.85	19.18	0.98%	9.199
Fe-K	0.0164	1.098	1.80	0.97	14.45%	0.467
Zn-K	0.2419	1.161	28.08	12.96	3.01%	6.217
Total			100.00	100.00		23.954

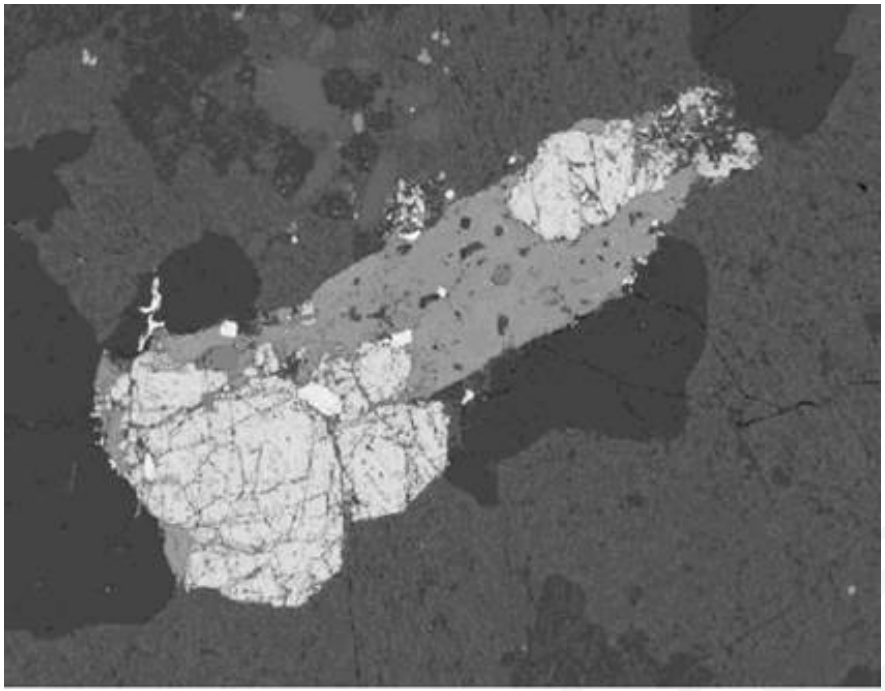
The number of cation results are based upon 24 Oxygen atoms

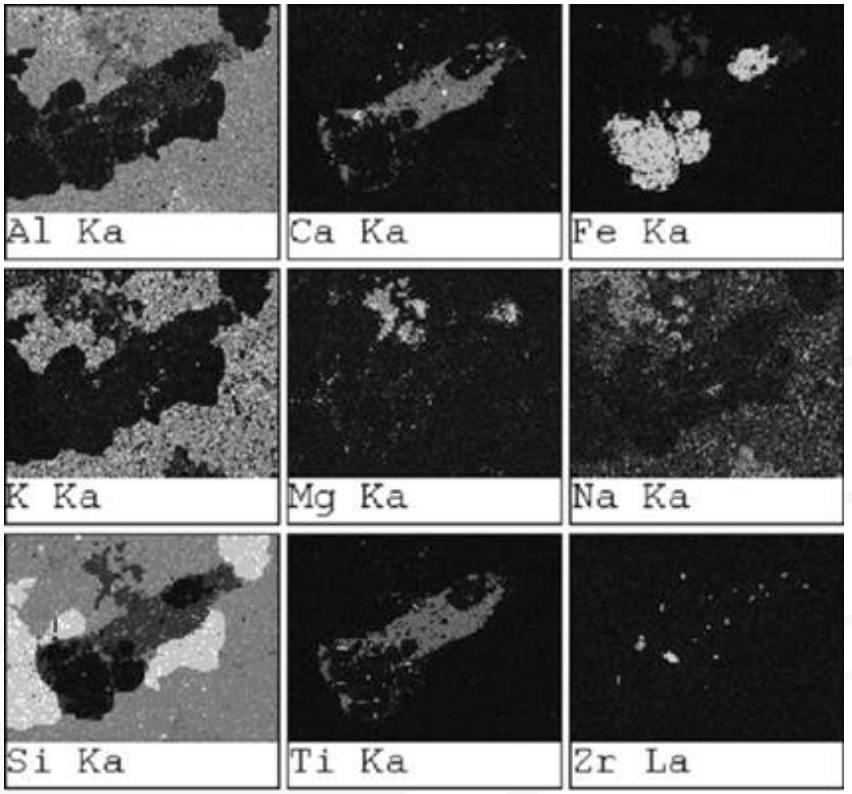
EDXA – Line analysis





EDXA – X-ray maps





Limitations with EDXA 1

- Elements lighter than Na give poor quantitative results
- Samples need to be conductive or made conductive with C or Au sputtering
- Au coating on samples interfer with quantitative analysis
- Quantitative calculations expect flat and homogenous samples
- Sum peaks (not a problem in ***newer*** detectors)
- Escape peaks (not a problem in ***newer*** detectors)

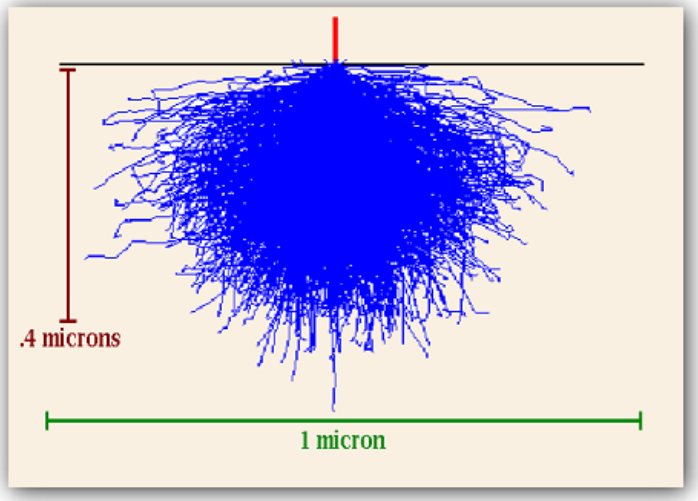
Limitations with EDXA 2

- Can only analyze elements, not compounds
- Qualitative analysis, a peak is enough
- Quantitative analysis, the optimal range for analysis is 1-10 keV
- A volume is always analyzed, not a spot!

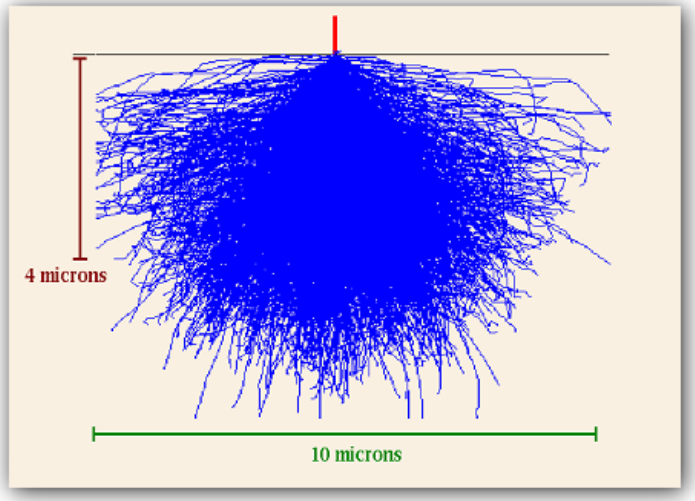
Electron Beam & Sample Interaction

Interaction Volume Simulation

Carbon Sample



Accelerating Voltage = 5KV

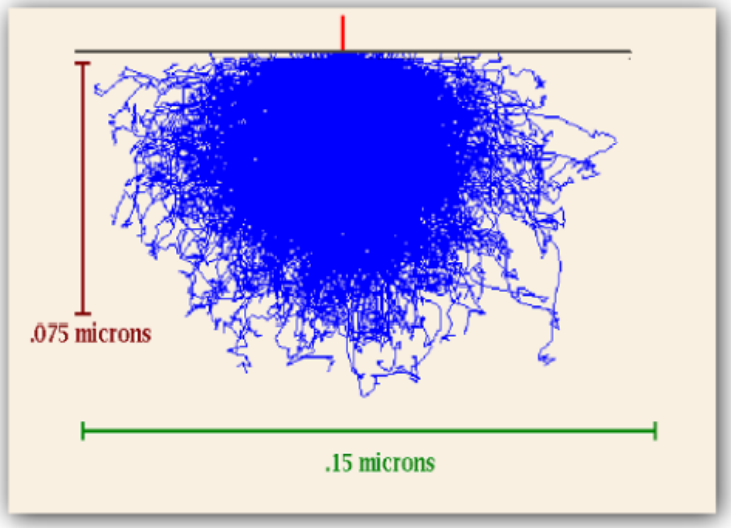


Accelerating Voltage = 25KV

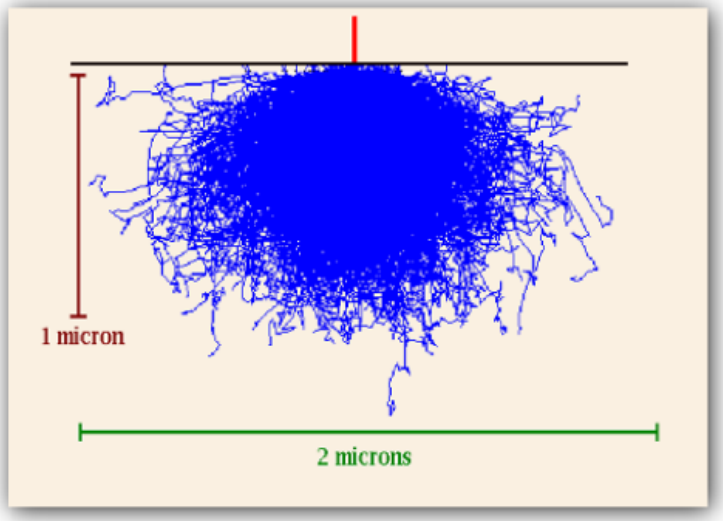
Electron Beam & Sample Interaction

Interaction Volume Simulation

Gold (Au) Sample

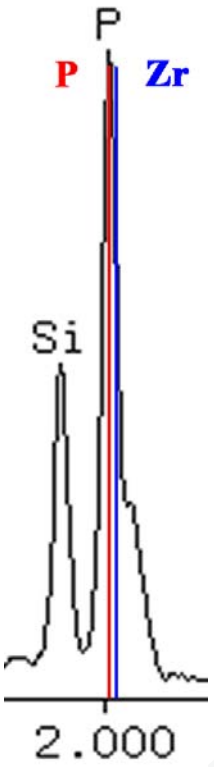


Accelerating Voltage = 5KV



Accelerating Voltage = 25KV

Overlapping peaks



- Sometimes the resolution of the detector isn’t good enough to resolve two peaks
- Usually there are other peaks for the elements that might be used
- Sometimes there are not and you’re stuck in an unfortunate situation
- The EDXA software will try to deconvolute the peaks

Examples of overlapping peaks

Element	Kα1	Kα2	Kβ1	Lα1	Lα2	Lβ1	Lβ2	Lγ1
11 Na	1.041	1.041	1.071					
30 Zn	8.639	8.616	9.572	1.012	1.012	1.035		
16 S	2.308	2.307	2.464					
42 Mo	17.479	17.374	19.608	2.293	2.290	2.395	2.518	2.624
82 Pb	74.969	72.804	84.936	10.552	10.450	12.614	12.623	14.764

SEM demonstration on lab tour