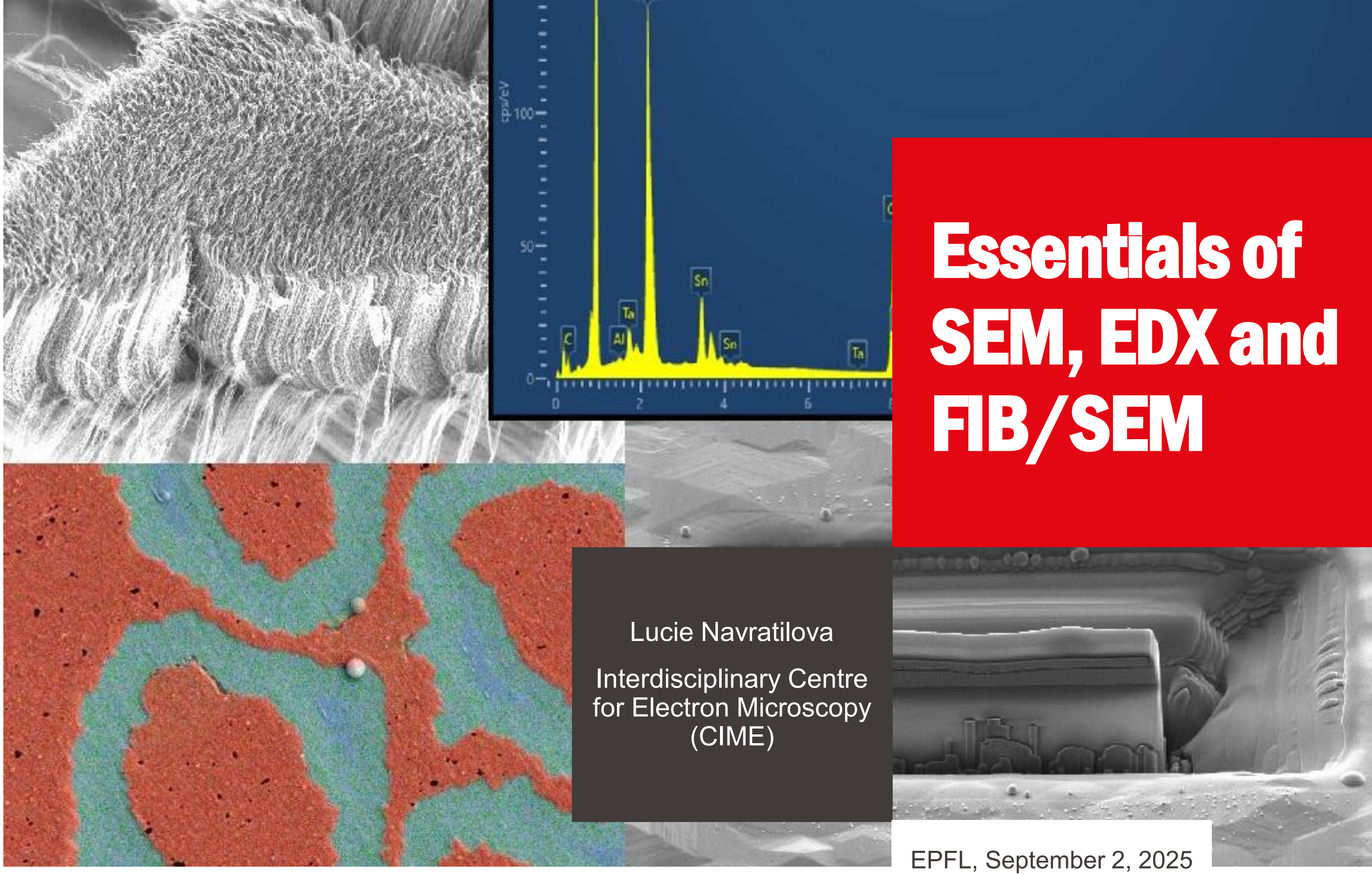


Essentials of SEM, EDX and FIB/SEM

Lucie Navratilova

Interdisciplinary Centre for Electron Microscopy (CIME)

EPFL, September 2, 2025

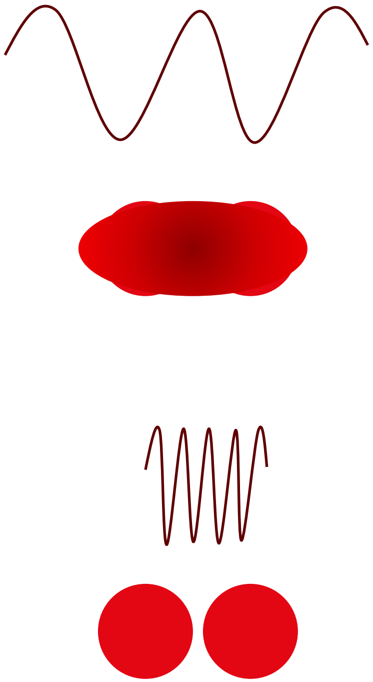
2025 Characterization of Materials Summer School

SEM

What is a SEM?

- SEM = Scanning Electron Microscope
- Focused beam of electrons to scan a sample’s surface
- Magnified images of objects and features that are not visible with light microscopes

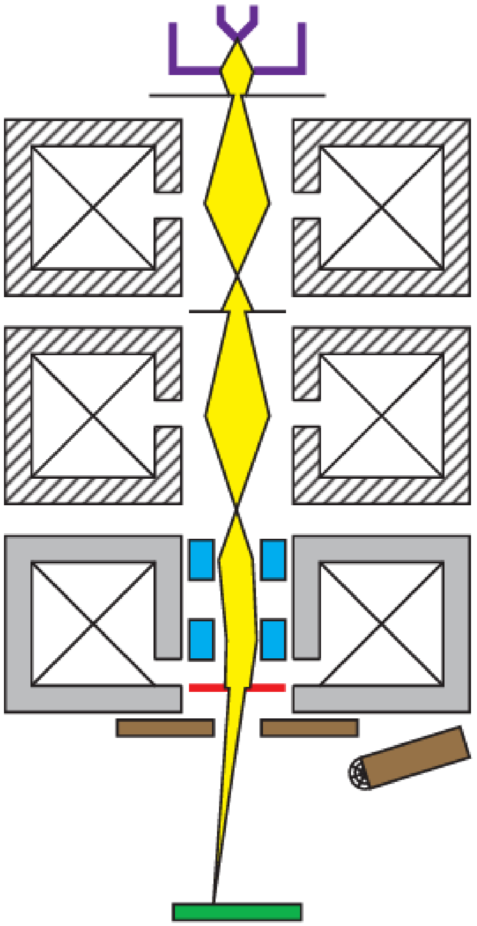
Electrons vs. light



- Resolution: the ability to distinguish between two closely spaced objects as separate entities
- Light microscopes: resolution limited by the wavelength of visible light ~200nm
- Electrons have wave/particle duality
- Electron microscopes:
  - resolution below 1nm for TEM (transmission electron microscope)
  - SEM resolution ~1nm possible (30kV)

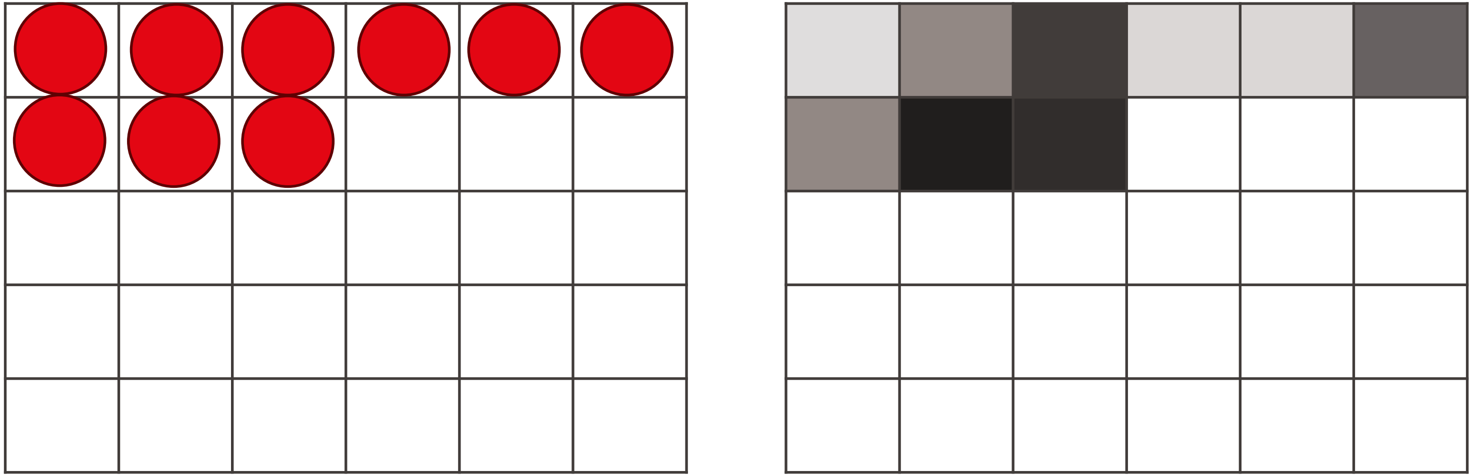
How SEM works?

- Source of electrons
- System of lenses (condenser and objective), apertures and scan coils
- Beam interactions with the sample surface
- Detectors

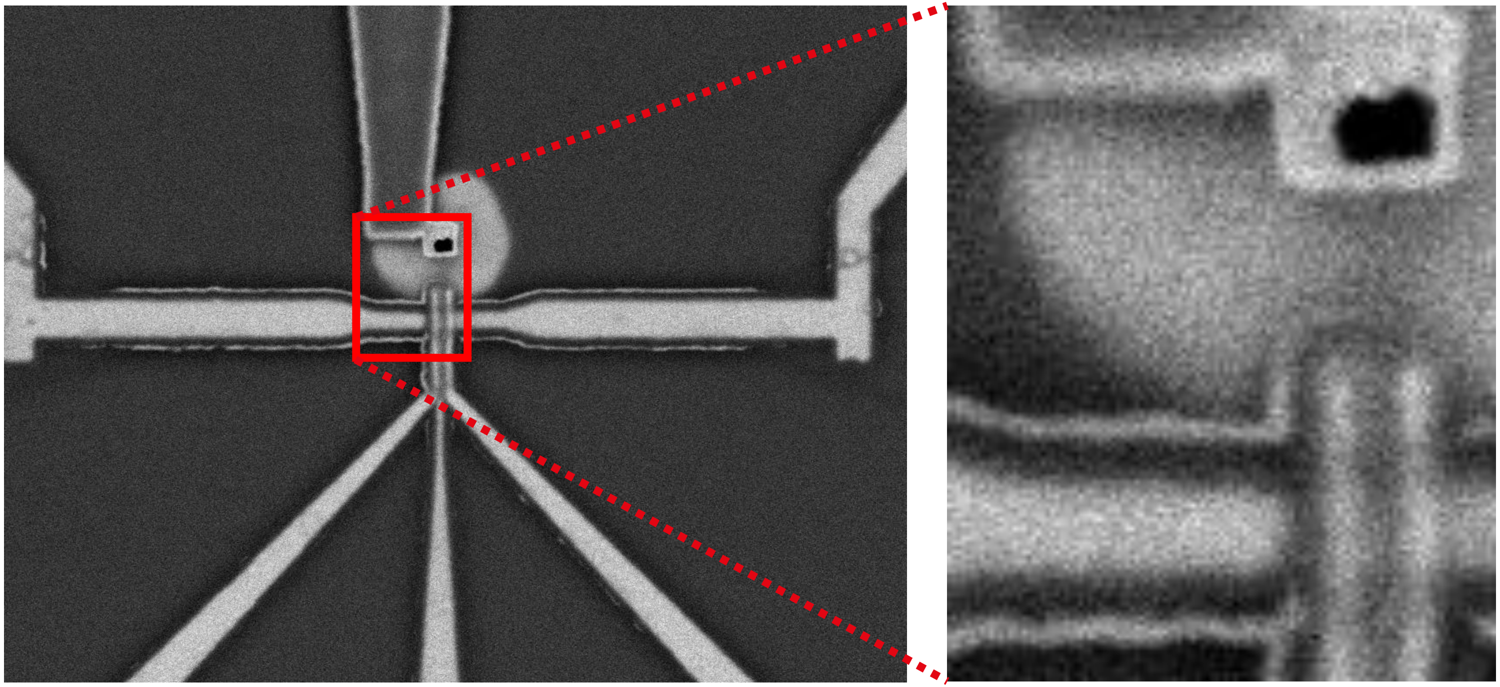


SEM image generation

- Intensity of each pixel is proportional to signal received

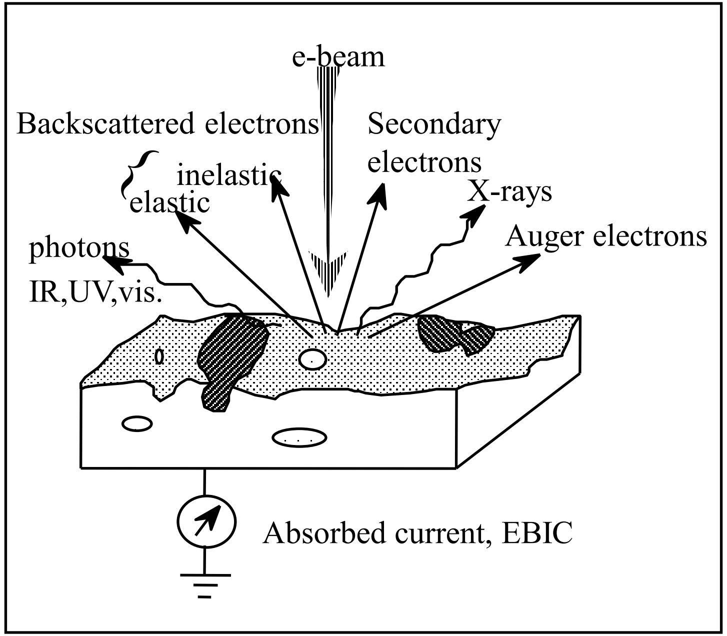


SEM image generation

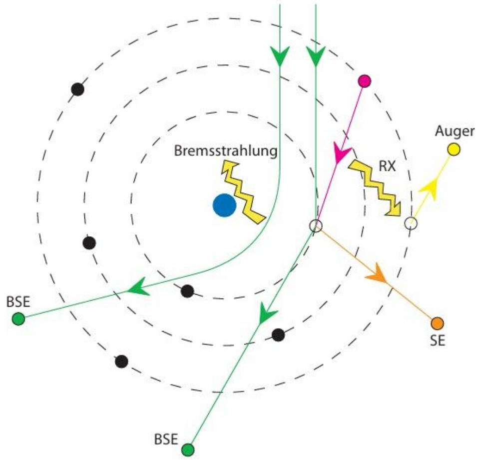


SEM signals

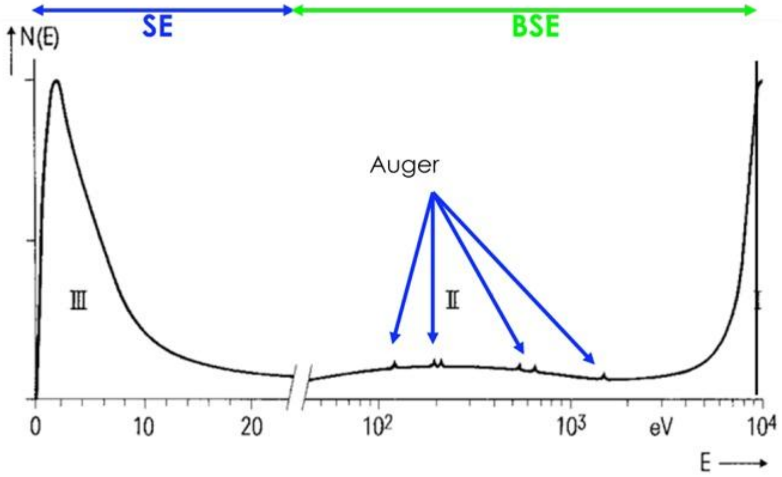
- Secondary electrons (SE)
- Backscattered electrons (BSE)
- Auger electrons
- Photons: visible, UV, IR, X-Rays
- Phonons, Heating,
- Absorption of incident electrons (EBIC-current)



SEM signals



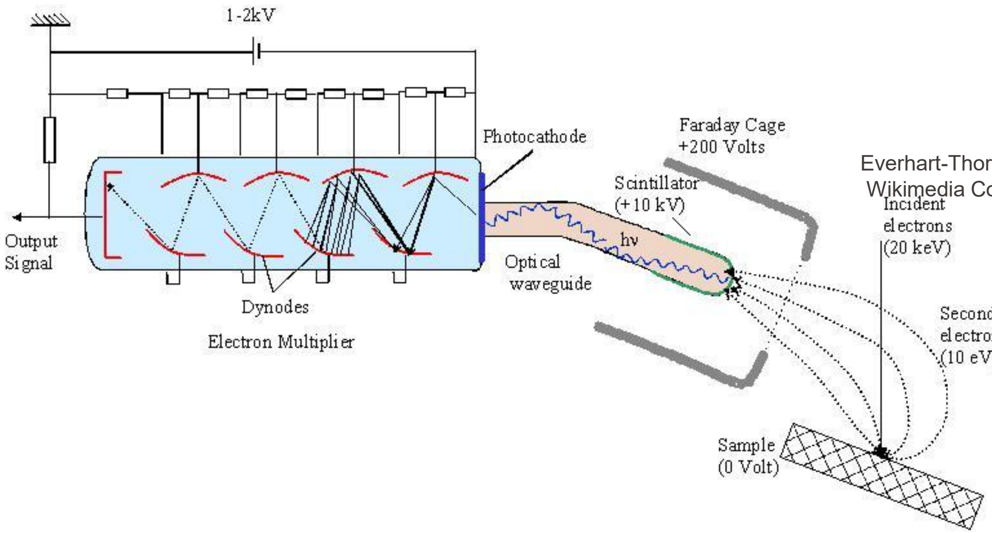
Incident electrons interactions with an atom



Energy spectrum of electrons leaving a sample

SEM detectors

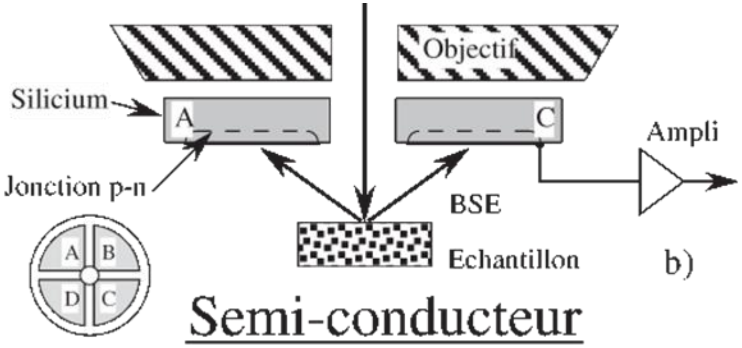
- **Scintillator / Photomultiplier**
- Everhart-Thornley detector (ETD)



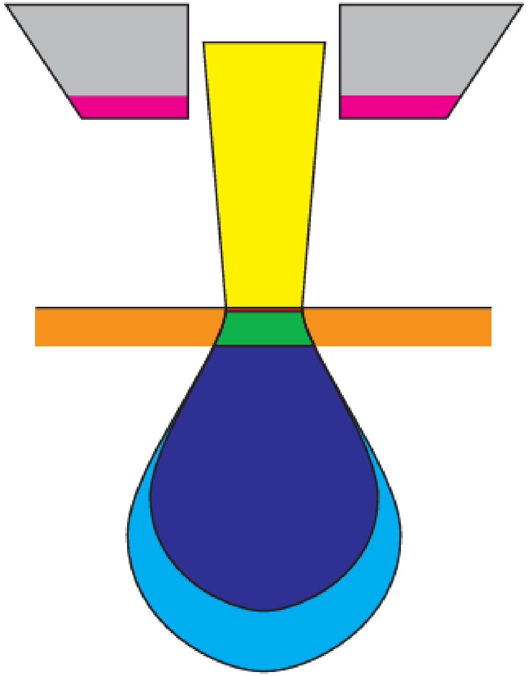
Everhart-Thornley detector
Wikimedia Commons

SEM detectors

- **Semiconductor detectors**
- Si diode with a p-n junction
- Electron-hole pairs
- Large collection angle
- Some diodes are split in 2 or 4 quadrants
- Slow (not responsive to rapid changes in signal intensity)

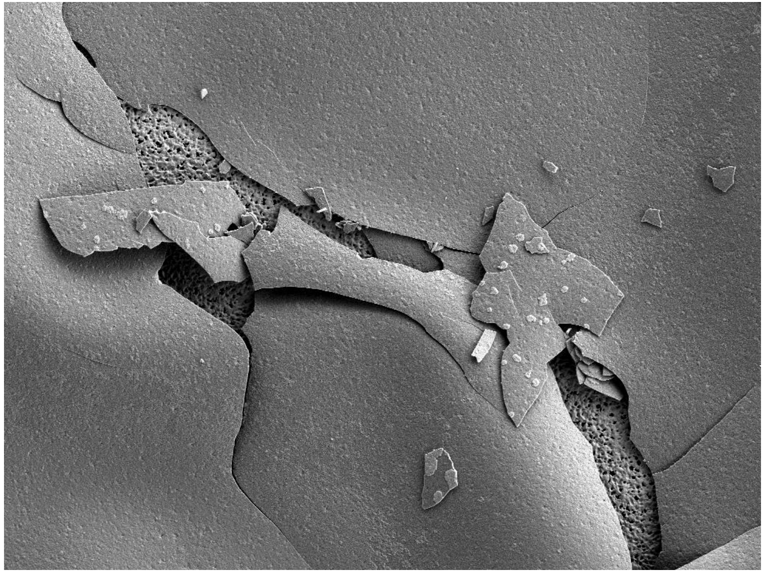


SEM interaction volume

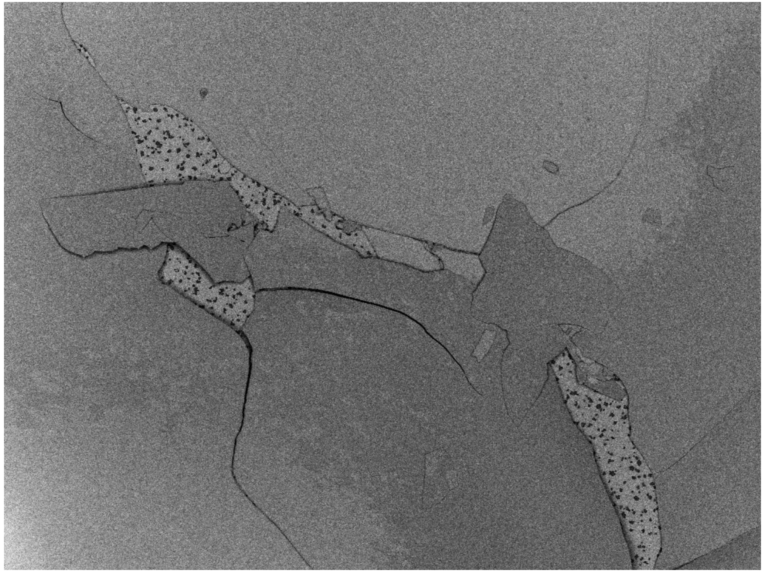


- Incident beam
- Auger electrons
- Secondary electrons (SE1)
- Backscattered electrons (BSE)
- X-Rays
- Secondary electrons (SE2)
- Secondary electrons (SE3)

SE vs. BSE image

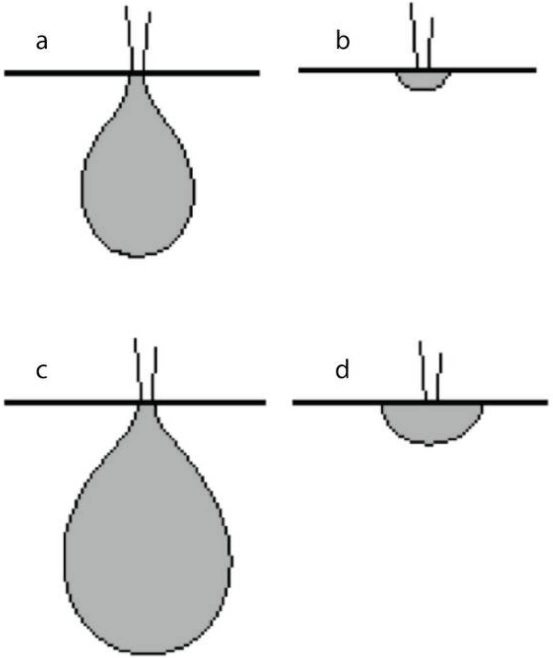


SE => topography



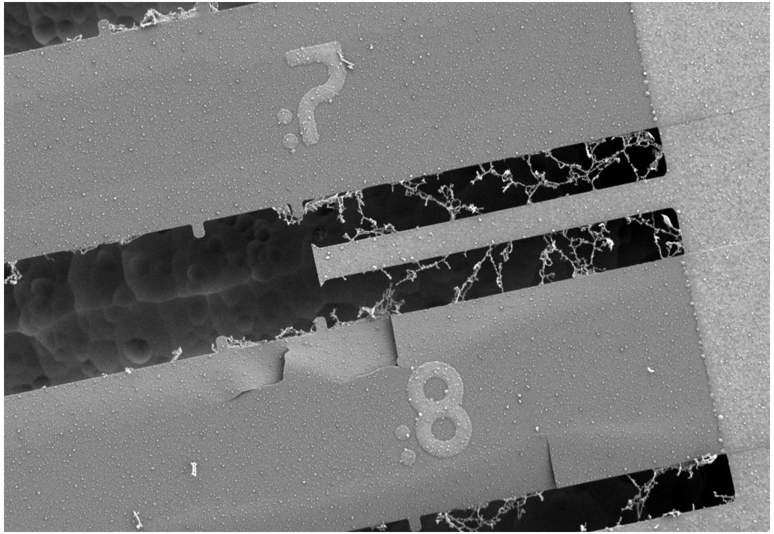
BSE => mass density contrast

SEM interaction volume

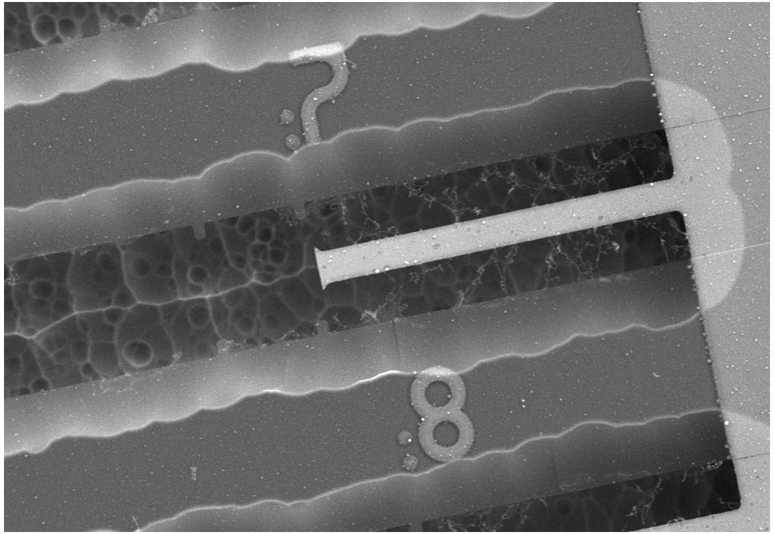


- Low Z vs. high Z
- Low vs. high acceleration voltage

SEM interaction volume



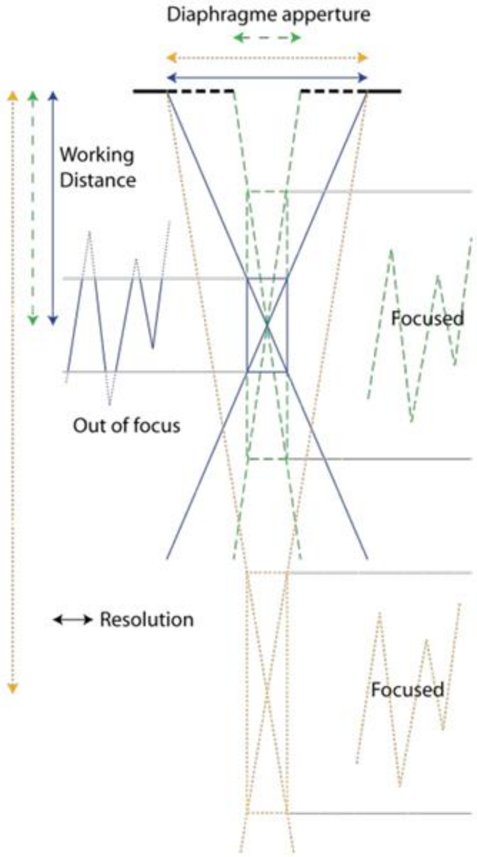
2 kV



10 kV

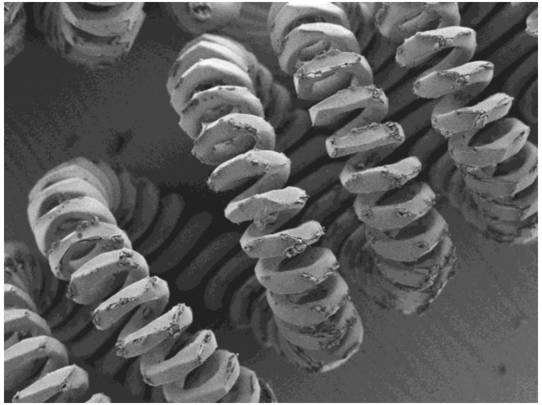
Depth of field

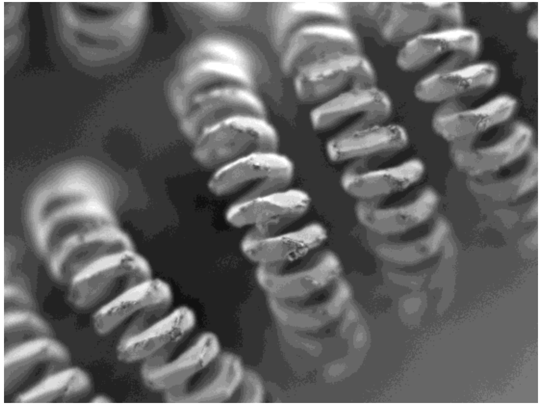
- The range of distances where an image appears acceptably sharp
- Probe convergence angle
  - Aperture size
  - Working distance



Depth of field

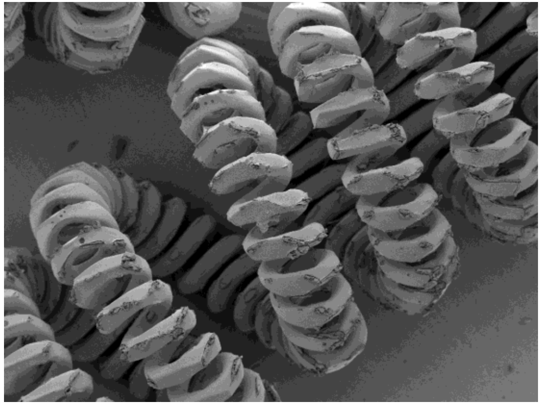
WD 5mm
Aperture size
30um

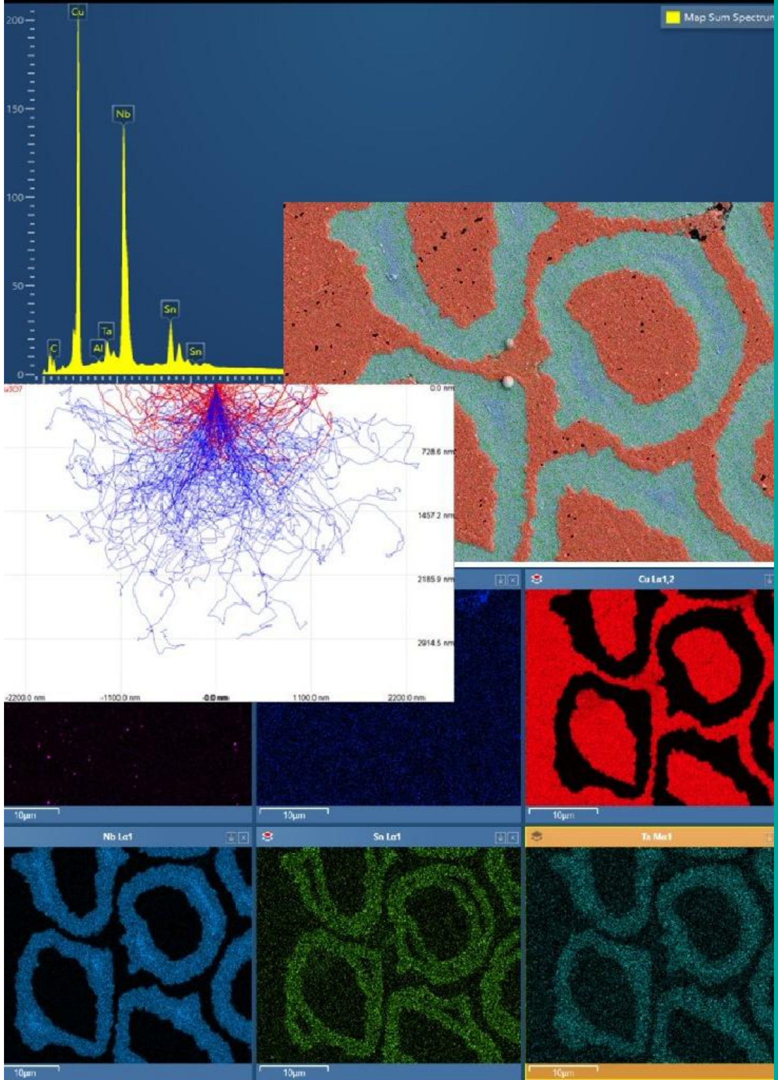




WD 5mm
Aperture size
120um

WD 15mm
Aperture size
30um

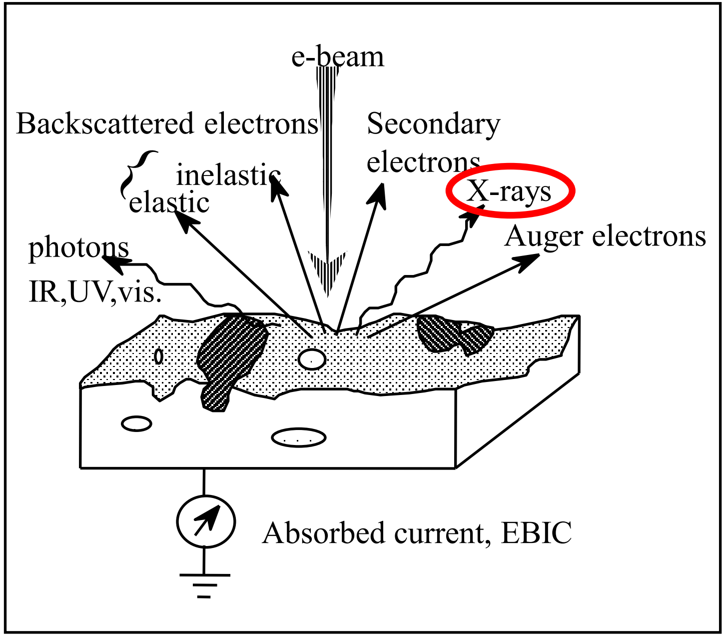



Map Sum Spectrum
200
150
100
50
0
Cu
Nb
Sn
Ta
Al
C
Sn
0.0 nm
728.6 nm
1457.2 nm
2185.9 nm
2914.5 nm
-2200.0 nm
-1100.0 nm
0.0 nm
1100.0 nm
2200.0 nm
Cu Lα1,2
Nb Lα1
Sn Lα1
Ta Mα1
10µm

EDX

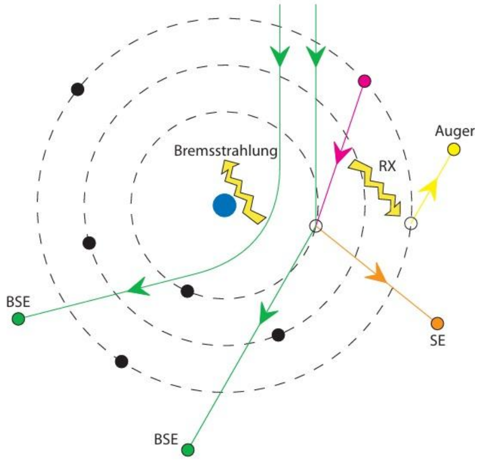
What is an EDX?

- Energy Dispersive X-ray Microanalysis
- Energy Dispersive Spectroscopy (EDS)
- Energy Dispersive X-ray spectroscopy (EDXS)
- Elemental analysis or chemical characterization technique in electron microscopes (SEM and TEM)



X-Rays generation

- Inelastic scattering of electrons at atoms
  - Continuum X-Ray production (Bremsstrahlung, Synchrotron, white radiation)
  - Characteristic X-Ray emission



X-Rays generation

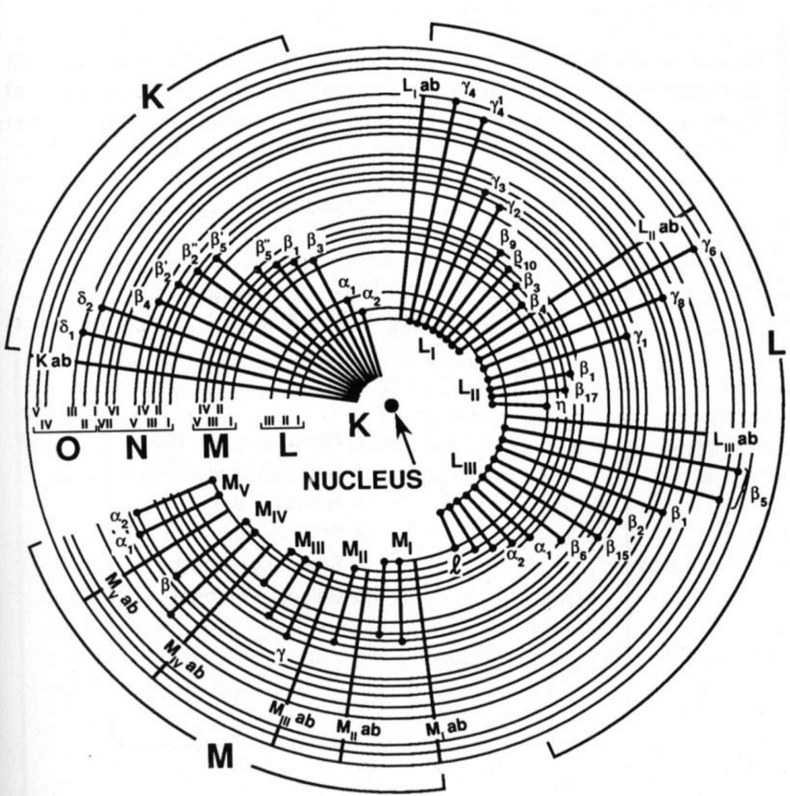
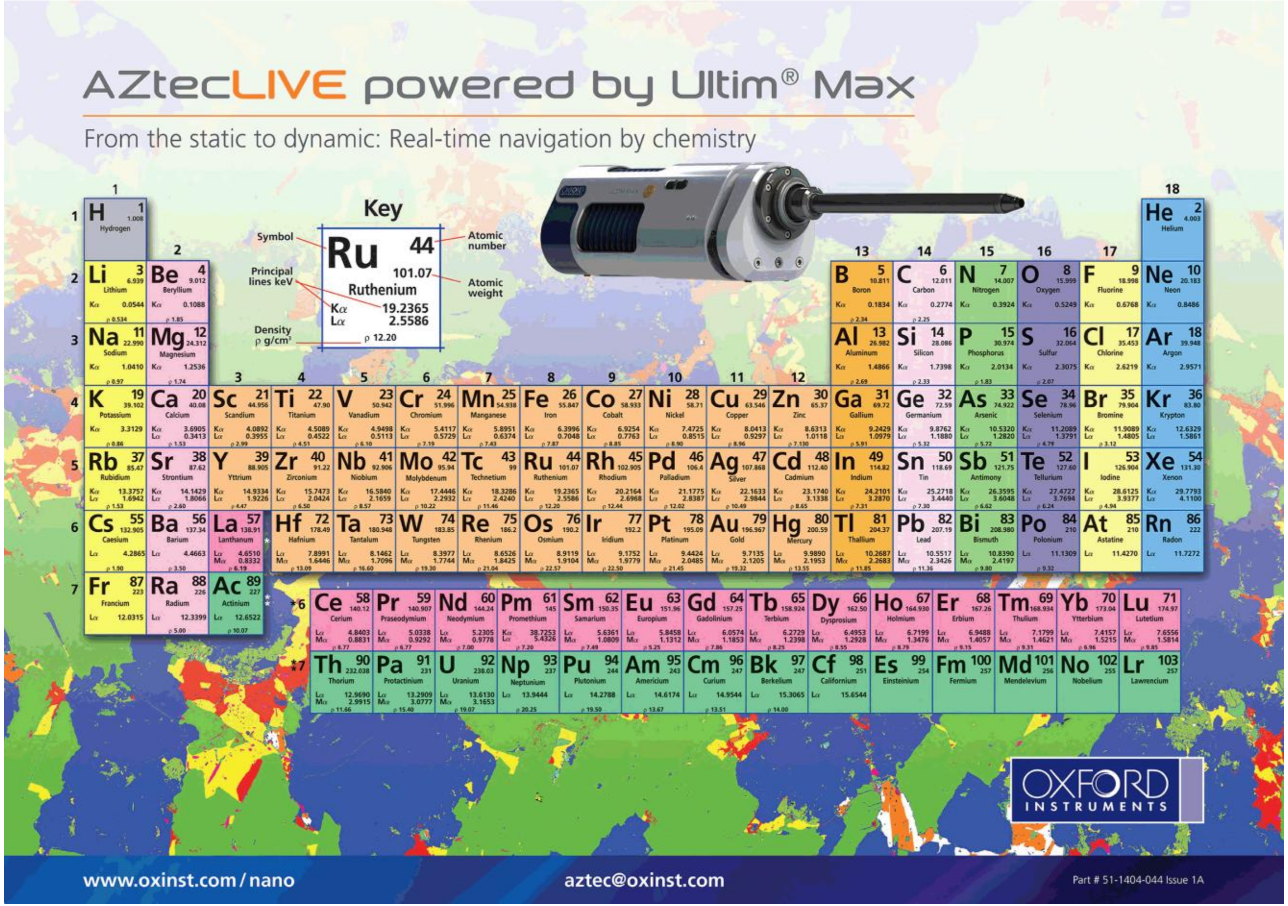


Figure 3.37. Comprehensive energy-level diagram showing all electron transitions which give rise to *K*, *L*, and *M* x rays (Woldseth, 1973).

Table of elements



AZtecLIVE powered by Ultim® Max

From the static to dynamic: Real-time navigation by chemistry

Key

Symbol	Atomic number	Atomic weight	Name	Principal lines keV	Density ρ g/cm³
Ru	44	101.07	Ruthenium	Kα 19.2365 Lα 2.5586	ρ 12.20

Period	Group	Symbol	Atomic number	Atomic weight	Name	Principal lines keV	Density ρ g/cm³
1	1	H	1	1.008	Hydrogen		
1	18	He	2	4.003	Helium		
2	1	Li	3	6.939	Lithium	Kα 0.0544	ρ 0.534
2	2	Be	4	9.012	Beryllium	Kα 0.1088	ρ 1.85
2	13	B	5	10.811	Boron	Kα 0.1834	ρ 2.34
2	14	C	6	12.011	Carbon	Kα 0.2774	ρ 2.25
2	15	N	7	14.007	Nitrogen	Kα 0.3924	
2	16	O	8	15.999	Oxygen	Kα 0.5249	
2	17	F	9	18.998	Fluorine	Kα 0.6768	
2	18	Ne	10	20.183	Neon	Kα 0.8486	
3	1	Na	11	22.990	Sodium	Kα 1.0410	ρ 0.97
3	2	Mg	12	24.312	Magnesium	Kα 1.2536	ρ 1.74
3	13	Al	13	26.982	Aluminum	Kα 1.4866	ρ 2.69
3	14	Si	14	28.086	Silicon	Kα 1.7398	ρ 2.33
3	15	P	15	30.974	Phosphorus	Kα 2.0134	ρ 1.83
3	16	S	16	32.064	Sulfur	Kα 2.3075	ρ 2.07
3	17	Cl	17	35.453	Chlorine	Kα 2.6219	
3	18	Ar	18	39.948	Argon	Kα 2.9571	
4	1	K	19	39.102	Potassium	Kα 3.3129	ρ 0.86
4	2	Ca	20	40.08	Calcium	Kα 3.6905 Lα 0.3413	ρ 1.53
4	3	Sc	21	44.956	Scandium	Kα 4.0892 Lα 0.3955	ρ 2.99
4	4	Ti	22	47.90	Titanium	Kα 4.5089 Lα 0.4522	ρ 4.51
4	5	V	23	50.942	Vanadium	Kα 4.9498 Lα 0.5113	ρ 6.10
4	6	Cr	24	51.996	Chromium	Kα 5.4117 Lα 0.5729	ρ 7.19
4	7	Mn	25	54.938	Manganese	Kα 5.8951 Lα 0.6374	ρ 7.43
4	8	Fe	26	55.847	Iron	Kα 6.3996 Lα 0.7048	ρ 7.87
4	9	Co	27	58.933	Cobalt	Kα 6.9254 Lα 0.7763	ρ 8.85
4	10	Ni	28	58.71	Nickel	Kα 7.4725 Lα 0.8515	ρ 8.90
4	11	Cu	29	63.546	Copper	Kα 8.0413 Lα 0.9297	ρ 8.96
4	12	Zn	30	65.37	Zinc	Kα 8.6313 Lα 1.0118	ρ 7.130
4	13	Ga	31	69.72	Gallium	Kα 9.2429 Lα 1.0979	ρ 5.91
4	14	Ge	32	72.59	Germanium	Kα 9.8762 Lα 1.1880	ρ 5.32
4	15	As	33	74.922	Arsenic	Kα 10.5320 Lα 1.2820	ρ 5.72
4	16	Se	34	78.96	Selenium	Kα 11.2089 Lα 1.3791	ρ 4.79
4	17	Br	35	79.904	Bromine	Kα 11.9089 Lα 1.4805	ρ 3.12
4	18	Kr	36	83.80	Krypton	Kα 12.6329 Lα 1.5861	
5	1	Rb	37	85.47	Rubidium	Kα 13.3757 Lα 1.6942	ρ 1.53
5	2	Sr	38	87.62	Strontium	Kα 14.1429 Lα 1.8066	ρ 2.60
5	3	Y	39	88.905	Yttrium	Kα 14.9334 Lα 1.9226	ρ 4.47
5	4	Zr	40	91.22	Zirconium	Kα 15.7473 Lα 2.0424	ρ 6.50
5	5	Nb	41	92.906	Niobium	Kα 16.5840 Lα 2.1659	ρ 8.57
5	6	Mo	42	95.94	Molybdenum	Kα 17.4446 Lα 2.2932	ρ 10.22
5	7	Tc	43	99	Technetium	Kα 18.3286 Lα 2.4240	ρ 11.46
5	8	Ru	44	101.07	Ruthenium	Kα 19.2365 Lα 2.5586	ρ 12.20
5	9	Rh	45	102.905	Rhodium	Kα 20.2164 Lα 2.6968	ρ 12.44
5	10	Pd	46	106.4	Palladium	Kα 21.1775 Lα 2.8387	ρ 12.02
5	11	Ag	47	107.868	Silver	Kα 22.1633 Lα 2.9844	ρ 10.49
5	12	Cd	48	112.40	Cadmium	Kα 23.1740 Lα 3.1338	ρ 8.65
5	13	In	49	114.82	Indium	Kα 24.2101 Lα 3.2870	ρ 7.31
5	14	Sn	50	118.69	Tin	Kα 25.2718 Lα 3.4440	ρ 7.30
5	15	Sb	51	121.75	Antimony	Kα 26.3595 Lα 3.6048	ρ 6.62
5	16	Te	52	127.60	Tellurium	Kα 27.4727 Lα 3.7694	ρ 6.24
5	17	I	53	126.904	Iodine	Kα 28.6125 Lα 3.9377	ρ 4.94
5	18	Xe	54	131.30	Xenon	Kα 29.7793 Lα 4.1100	
6	1	Cs	55	132.905	Caesium	Lα 4.2865	ρ 1.90
6	2	Ba	56	137.34	Barium	Lα 4.4663	ρ 3.50
6	3	La *	57	138.91	Lanthanum	Lα 4.6510 Mα 0.8332	ρ 6.19
6	4	Hf	72	178.49	Hafnium	Lα 7.8991 Mα 1.6446	ρ 13.09
6	5	Ta	73	180.948	Tantalum	Lα 8.1462 Mα 1.7096	ρ 16.60
6	6	W	74	183.85	Tungsten	Lα 8.3977 Mα 1.7744	ρ 19.30
6	7	Re	75	186.2	Rhenium	Lα 8.6526 Mα 1.8425	ρ 21.04
6	8	Os	76	190.2	Osmium	Lα 8.9119 Mα 1.9104	ρ 22.57
6	9	Ir	77	192.2	Iridium	Lα 9.1752 Mα 1.9779	ρ 22.50
6	10	Pt	78	195.09	Platinum	Lα 9.4424 Mα 2.0485	ρ 21.45
6	11	Au	79	196.967	Gold	Lα 9.7135 Mα 2.1205	ρ 19.32
6	12	Hg	80	200.59	Mercury	Lα 9.9890 Mα 2.1953	ρ 13.55
6	13	Tl	81	204.37	Thallium	Lα 10.2687 Mα 2.2683	ρ 11.85
6	14	Pb	82	207.19	Lead	Lα 10.5517 Mα 2.3426	ρ 11.36
6	15	Bi	83	208.980	Bismuth	Lα 10.8390 Mα 2.4197	ρ 9.80
6	16	Po	84	210	Polonium	Lα 11.1309	ρ 9.32
6	17	At	85	210	Astatine	Lα 11.4270	
6	18	Rn	86	222	Radon	Lα 11.7272	
7	1	Fr	87	223	Francium	Lα 12.0315	
7	2	Ra	88	226	Radium	Lα 12.3399	ρ 5.00
7	3	Ac **	89	227	Actinium	Lα 12.6522	ρ 10.07
*6		Ce	58	140.12	Cerium	Lα 4.8403 Mα 0.8831	ρ 6.77
*6		Pr	59	140.907	Praseodymium	Lα 5.0338 Mα 0.9292	ρ 6.77
*6		Nd	60	144.24	Neodymium	Lα 5.2305 Mα 0.9778	ρ 7.00
*6		Pm	61	145	Promethium	Kα 38.7253 Lα 5.4326	ρ 7.20
*6		Sm	62	150.35	Samarium	Lα 5.6361 Mα 1.0809	ρ 7.49
*6		Eu	63	151.96	Europium	Lα 5.8458 Mα 1.1312	ρ 5.25
*6		Gd	64	157.25	Gadolinium	Lα 6.0574 Mα 1.1853	ρ 7.86
*6		Tb	65	158.924	Terbium	Lα 6.2729 Mα 1.2398	ρ 8.25
*6		Dy	66	162.50	Dysprosium	Lα 6.4953 Mα 1.2928	ρ 8.55
*6		Ho	67	164.930	Holmium	Lα 6.7199 Mα 1.3476	ρ 8.79
*6		Er	68	167.26	Erbium	Lα 6.9488 Mα 1.4057	ρ 9.15
*6		Tm	69	168.934	Thulium	Lα 7.1799 Mα 1.4621	ρ 9.31
*6		Yb	70	173.04	Ytterbium	Lα 7.4157 Mα 1.5215	ρ 6.96
*6		Lu	71	174.97	Lutetium	Lα 7.6556 Mα 1.5814	ρ 9.85
**7		Th	90	232.038	Thorium	Lα 12.9690 Mα 2.9915	ρ 11.66
**7		Pa	91	231	Protactinium	Lα 13.2909 Mα 3.0777	ρ 15.40
**7		U	92	238.03	Uranium	Lα 13.6130 Mα 3.1653	ρ 19.07
**7		Np	93	237	Neptunium	Lα 13.9444	ρ 20.25
**7		Pu	94	244	Plutonium	Lα 14.2788	ρ 19.50
**7		Am	95	243	Americium	Lα 14.6174	ρ 13.67
**7		Cm	96	247	Curium	Lα 14.9544	ρ 13.51
**7		Bk	97	247	Berkelium	Lα 15.3065	ρ 14.00
**7		Cf	98	251	Californium	Lα 15.6544	
**7		Es	99	254	Einsteinium		
**7		Fm	100	257	Fermium		
**7		Md	101	256	Mendelevium		
**7		No	102	255	Nobelium		
**7		Lr	103	257	Lawrencium		

OXFORD INSTRUMENTS

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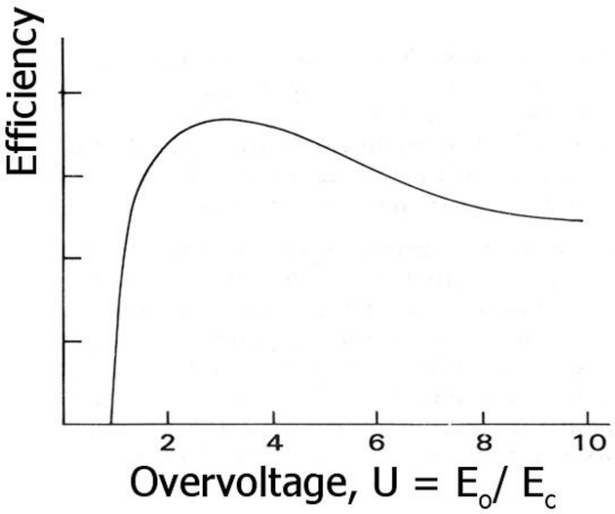
Part # 51-1404-044 Issue 1A

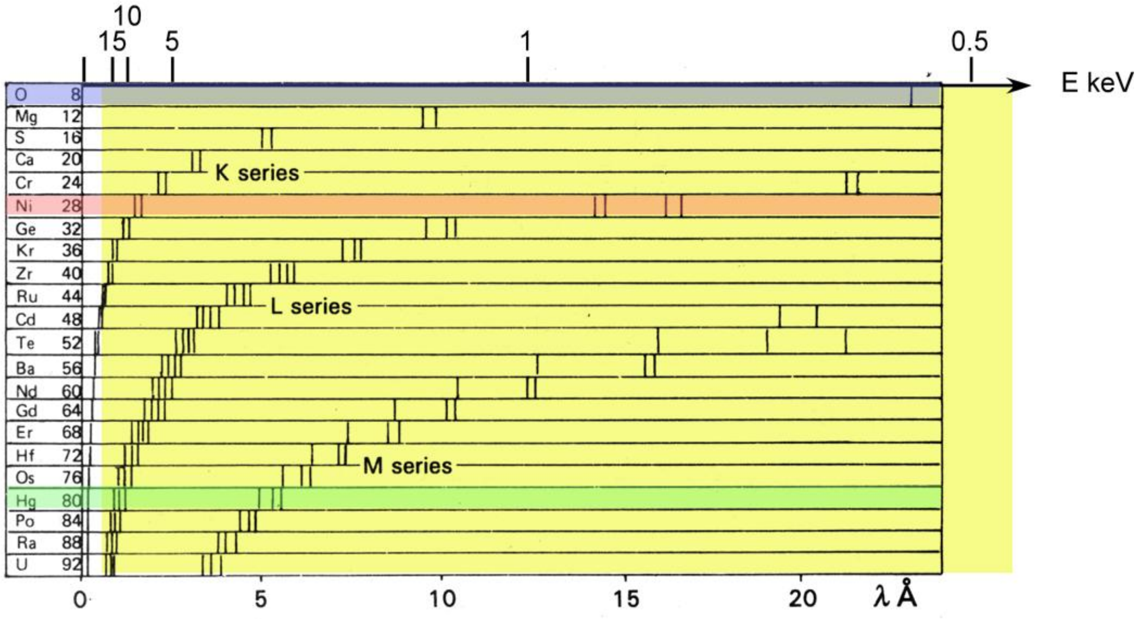
X-Rays generation efficiency

- Overvoltage $U=E_o/E_{ionization}$



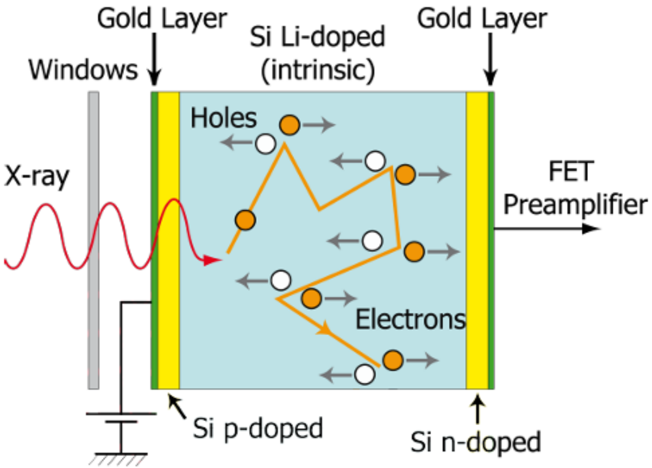
Characteristic lines: Moseley’s Law

There is a relationship between the energy of the Characteristic X-ray lines for each element and its atomic number: the energy is proportional to the square of the atomic number, Z.



Known ambiguities:
Al Ka = Br Ll
S Ka = Mo Ll

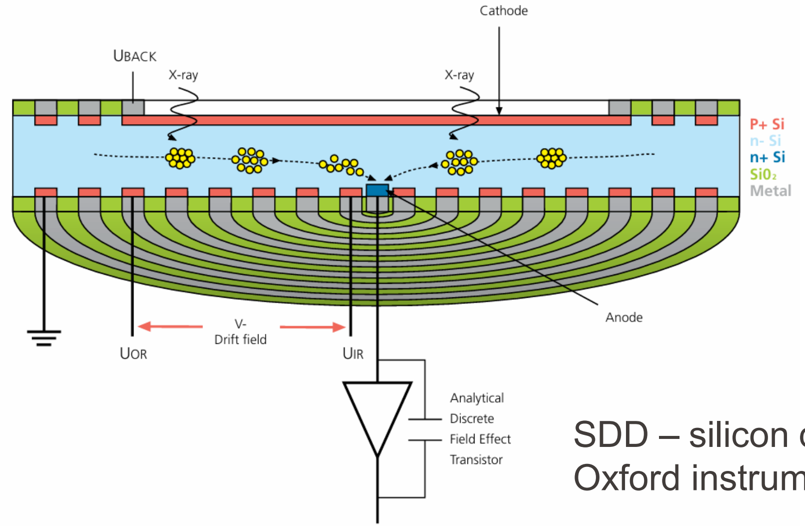
How EDX detector works



- Li-doped Si single crystal semiconductor
- X-Ray is converted into a charge by the ionization of atoms in the semiconductor crystal
- Electron-hole pairs – number proportional to the energy of the X-Ray
- Electrons collected to the anode
- X-Ray energies are measured as electric voltage pulses
- Cooling by liquid nitrogen – to prevent Li diffusion and to reduce a dark current (thermal noise)

Modern EDX detector

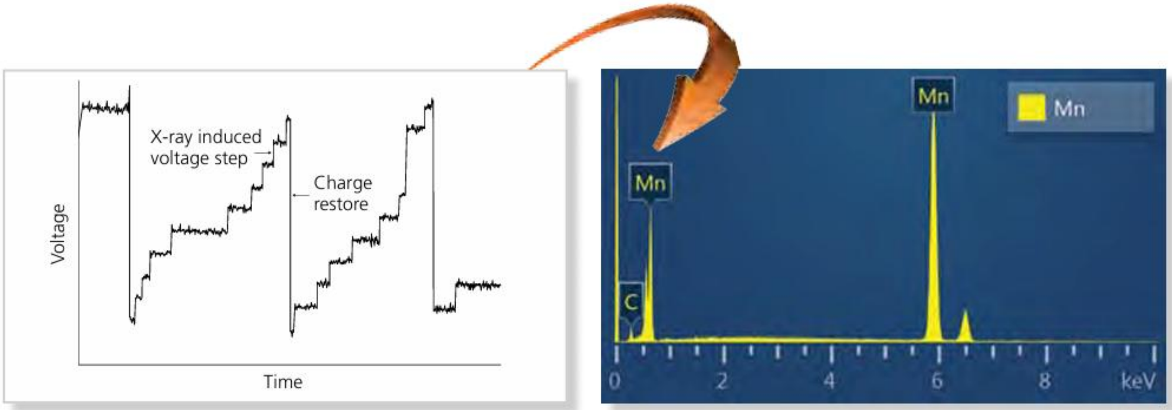
- SDD sensor – high purity silicon with a large area contact
- Central small anode contact, surrounded by a number of concentric drift electrodes
- X-Ray is converted into an electron cloud
- Electrons drifted down a field gradient applied between the drift rings
- Peltier cooling



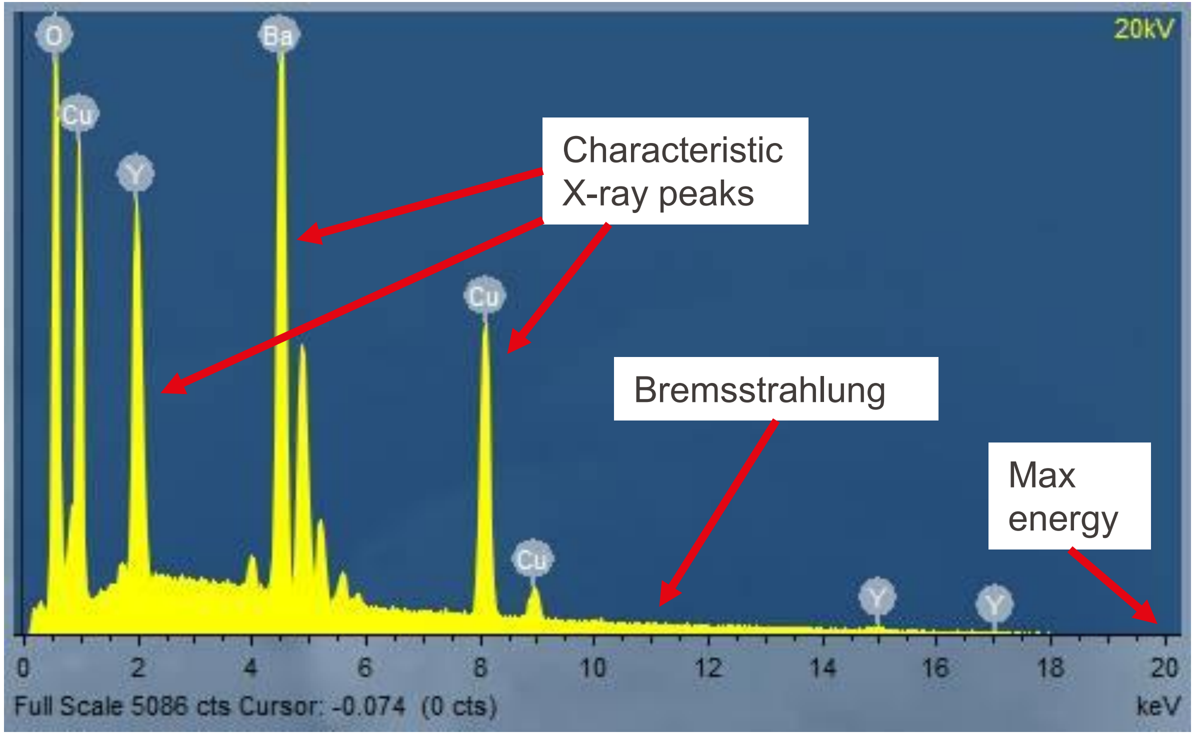
SDD – silicon drift detector
Oxford instruments

How EDX detector works

- The charge is converted into the voltage signal by the FET preamplifier
- The output from the preamplifier is a voltage “ramp” – X-ray charge + leakage current
- The voltage signal is input into the pulse processor for measurement



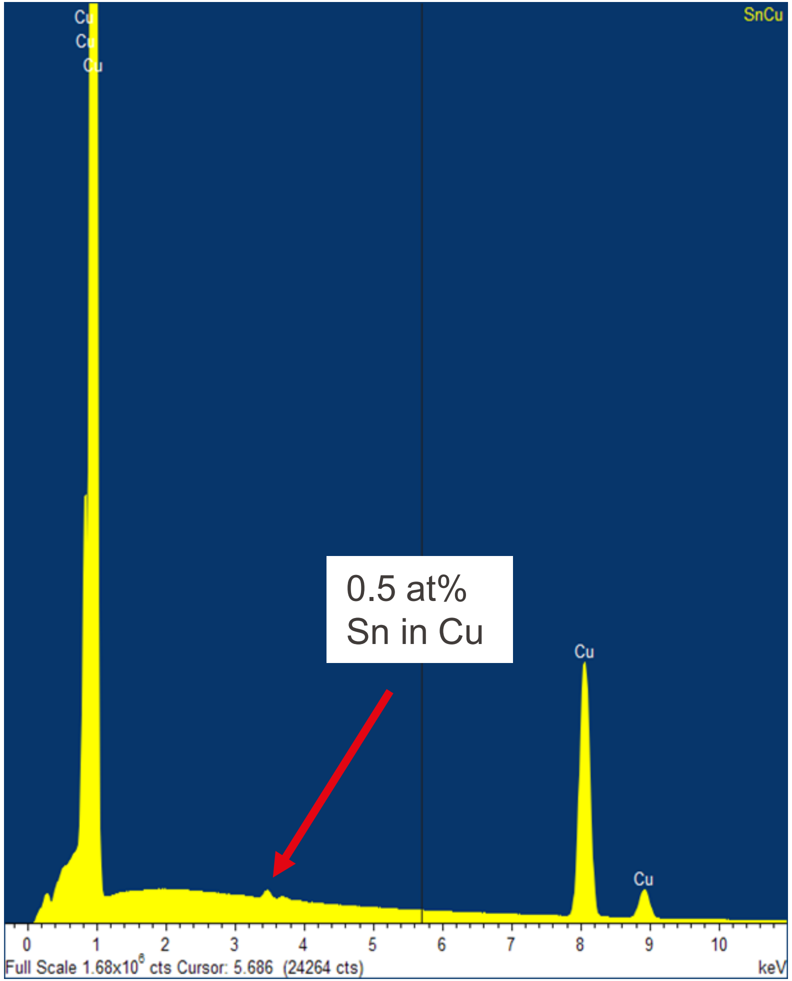
EDX spectrum



Spectrum simulation $YBa_2Cu_3O_7$ at 20kV

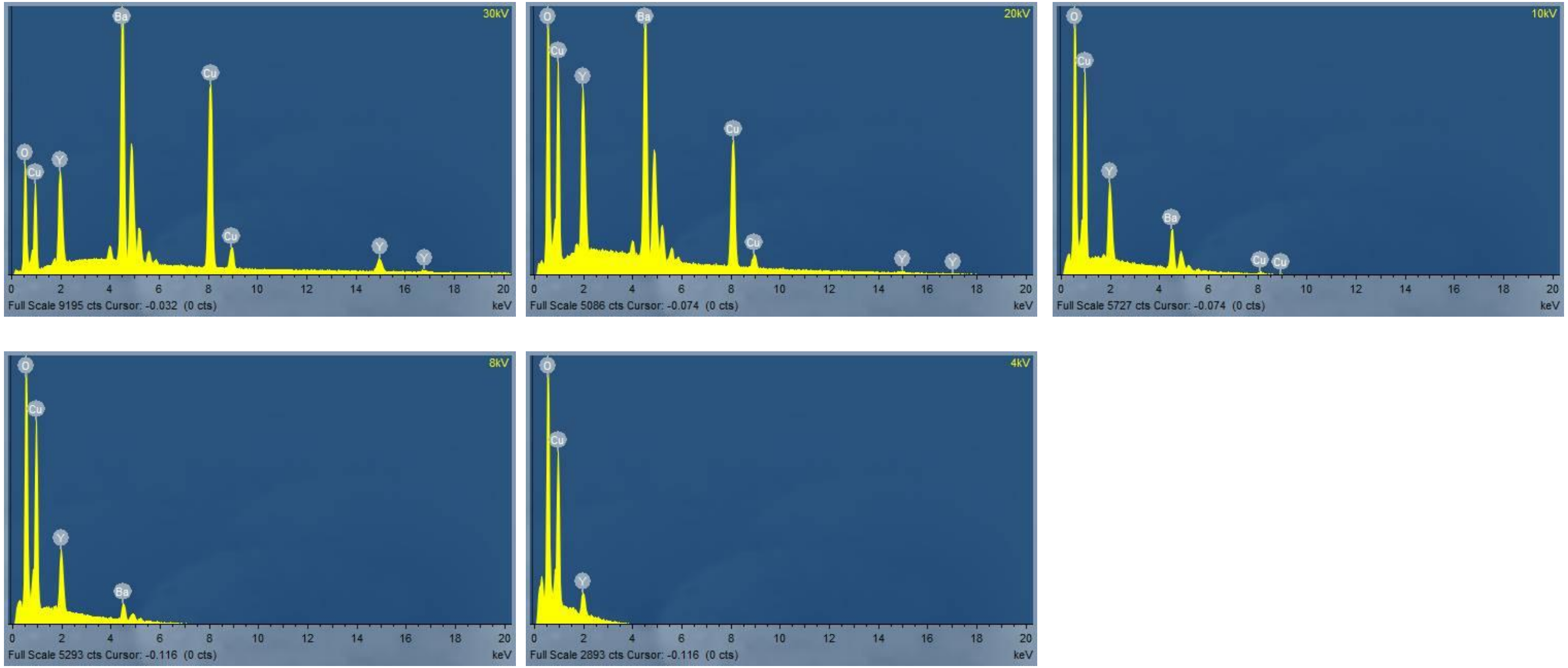
Detection limit

- Acquisition under best conditions => detection of ~0,5 at% for almost all elements for acquisition times of 100sec



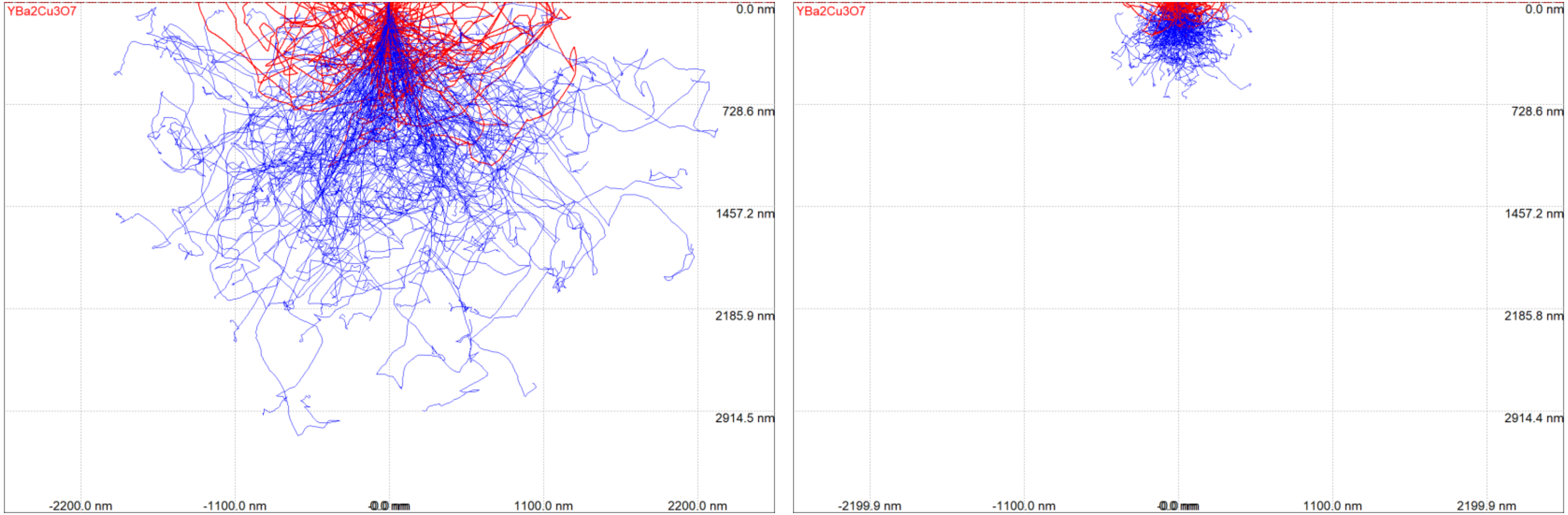
0.5 at%
Sn in Cu

Choice of HV in SEM



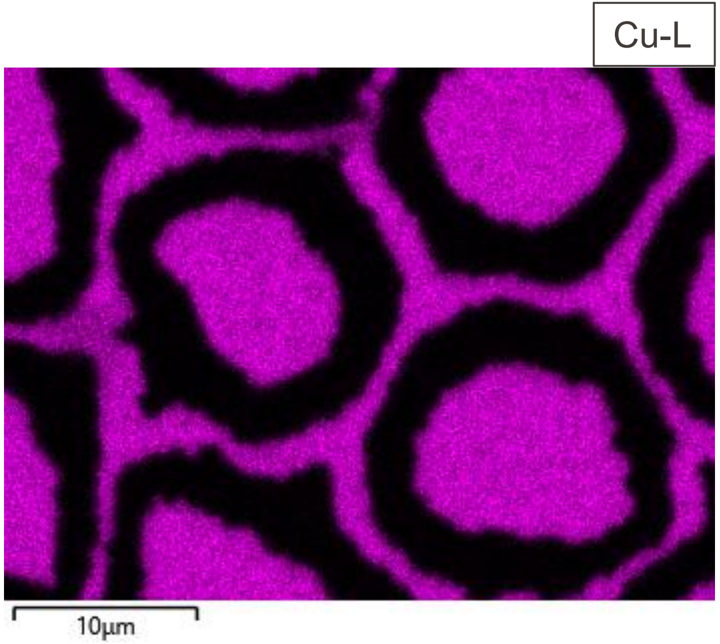
Sample: $YBa_2Cu_3O_7$

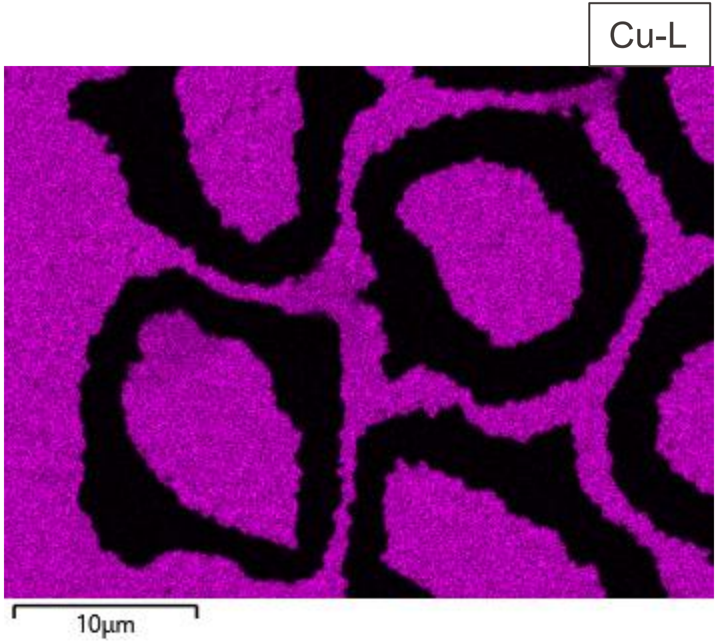
20kV vs. 8kV



Monte-Carlo Simulation of electron trajectories with CASINO

20kV vs. 8kV resolution

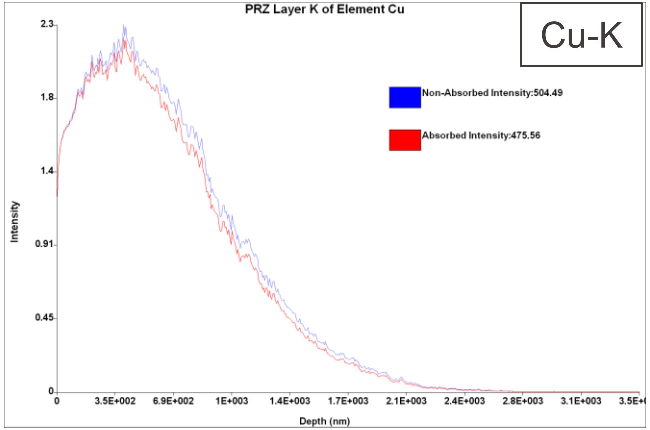


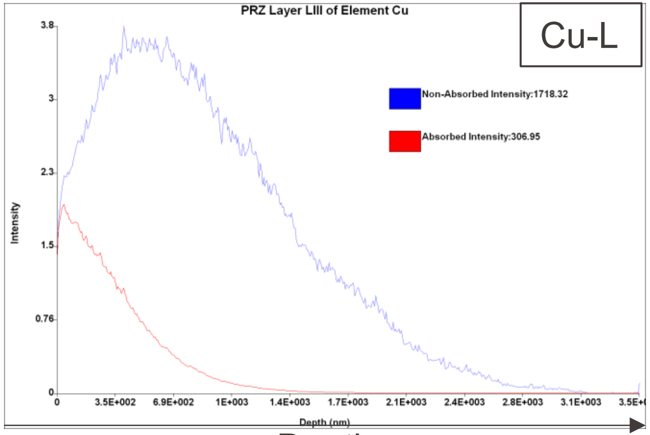


20kV vs. 8kV – absorption issue

X-rays generated
X-rays detected

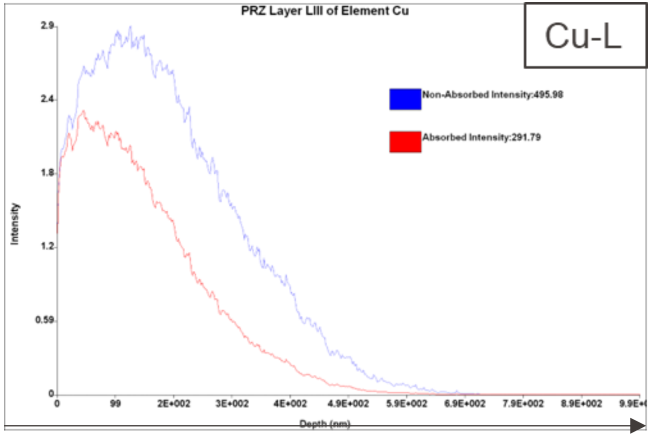
20kV





Depth

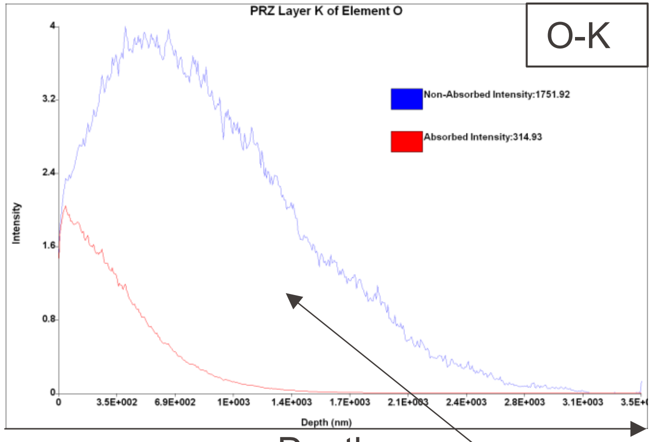
8kV



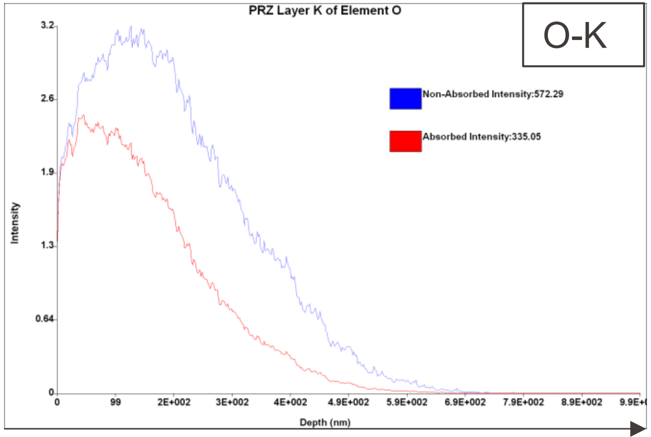
Depth

20kV vs. 8kV – absorption issue

20kV



Depth



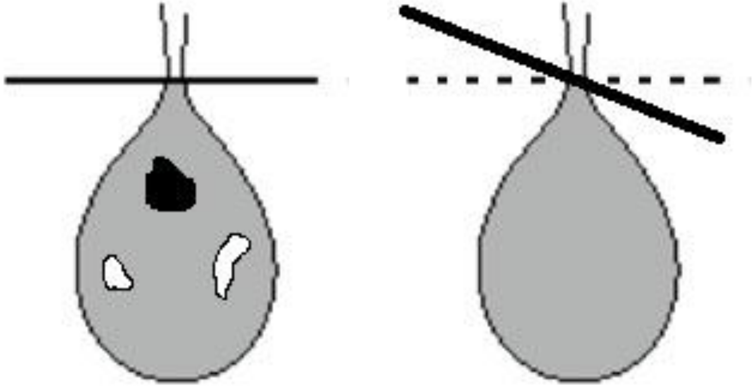
Depth

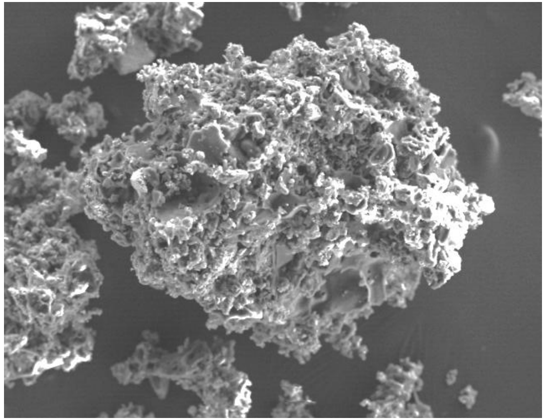
8kV

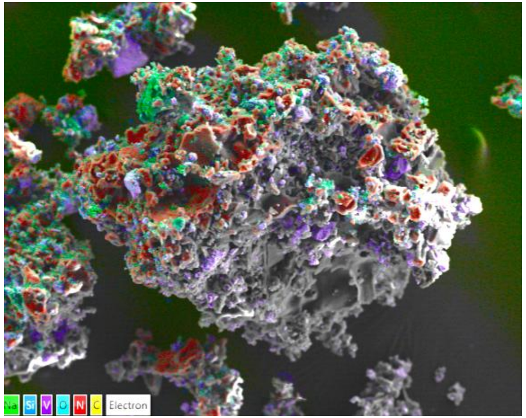
Not detected by detector
due to absorption

Ideal sample

- Flat surface without contamination
- Horizontal orientation
- Homogeneous sample (no inclusions, voids,...)







Quantitative EDX in SEM

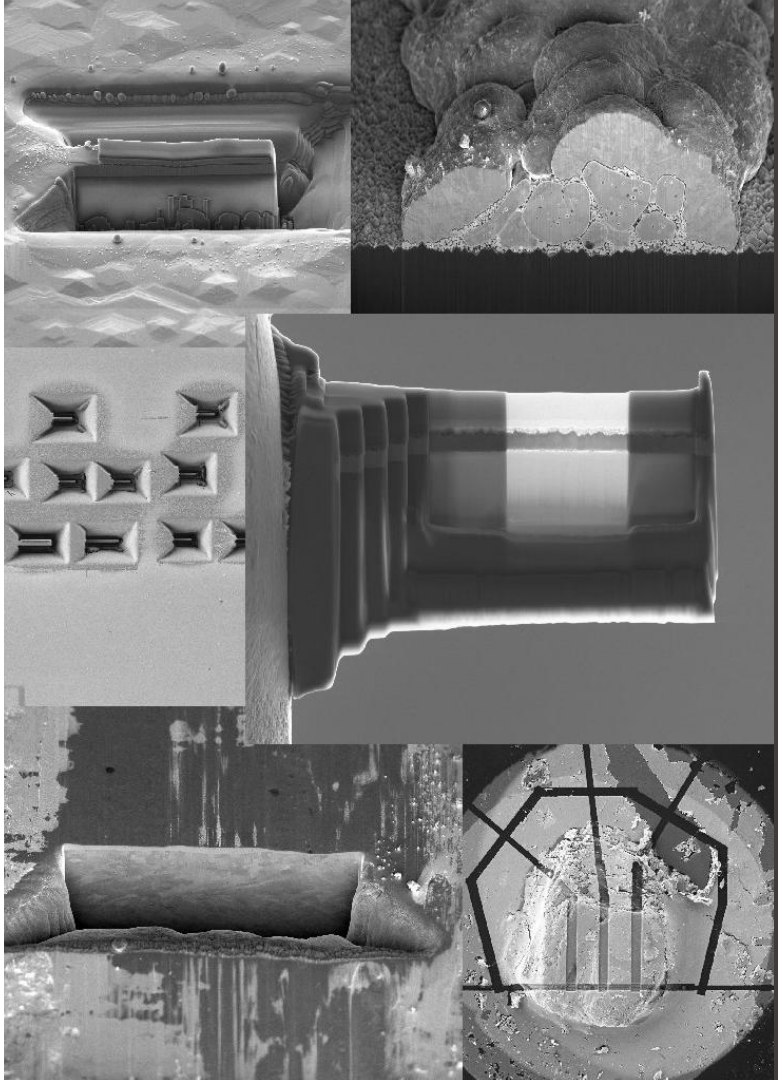
When the going gets tough…

- Acquisition under best conditions - sample, position, HV,...
- Comparison with a standard sample (known concentration, same conditions)
- Standardless – theoretical calculation, possible with high accuracy, light elements are critical

- Correction methods

$$[Z \times A \times F]\frac{c_i}{c_i^{std}} = \frac{I_i}{I_i^{std}} = k_i$$

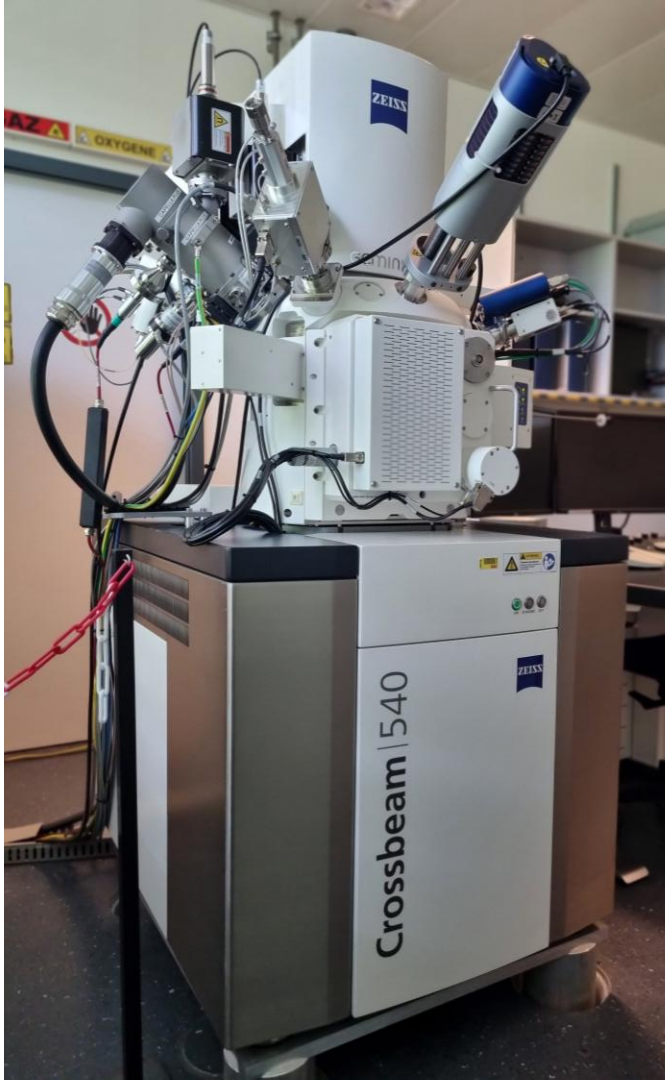
- Z – how the electron beam penetrates in the sample and loose energy (Z and density dependant)
- A – absorption of the X-rays along the path to sample surface
- F – adds some photons when (secondary) fluorescence occurs



FIB/SEM

What is a FIB/SEM?

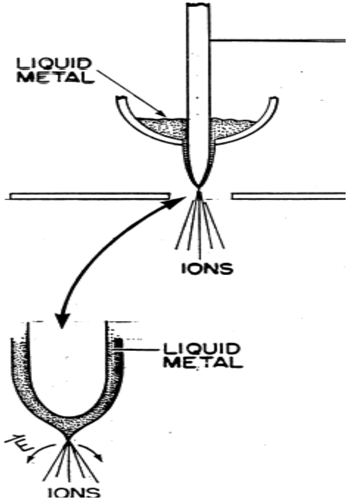
- FIB = Focused Ion Beam
- FIB/SEM = dual beam/ cross beam microscope
- SEM goes 3D

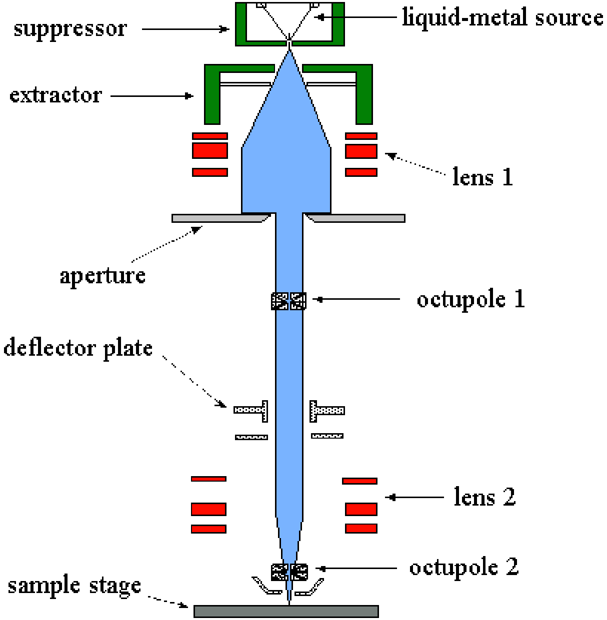


Ion column

- Source: liquid metal ion source (LMIS)
- Ga, Au, Ir
- (Xe, Ar, N, O)

- Why Ga?
  - Metallic
  - Low melting point
  - In the middle of periodic table
  - No overlap for EDX





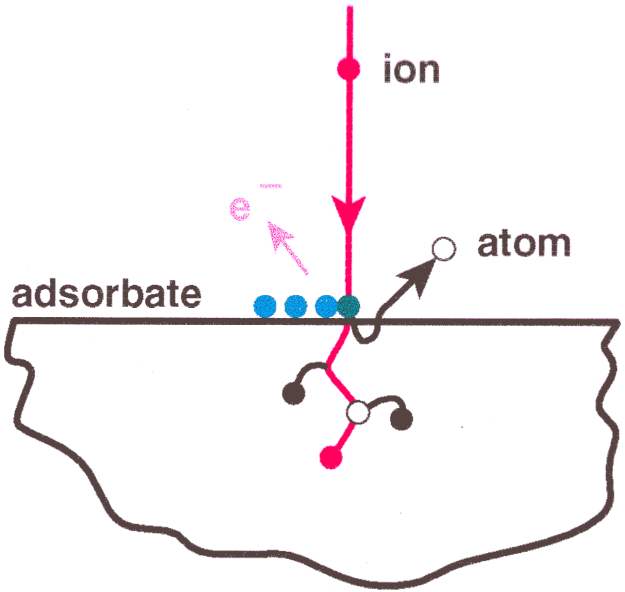
Schematic diagram of a FIB ion column
Source: IBM Almaden Research Center

Electrons vs. Ions

- **Electrons:**
- Small
- Inner shell reactions
- High penetration depth
- Low mass – higher speed for given energy
- Negative
- Magnetic lens

- **Ions:**
- Big
- Outer shell reactions (no X-Rays)
- High interaction probability
- Less penetration depth
- Can remain trapped – doping
- High mass – slow speed but high momentum
  - Milling
- Positive
- Electrostatic lenses

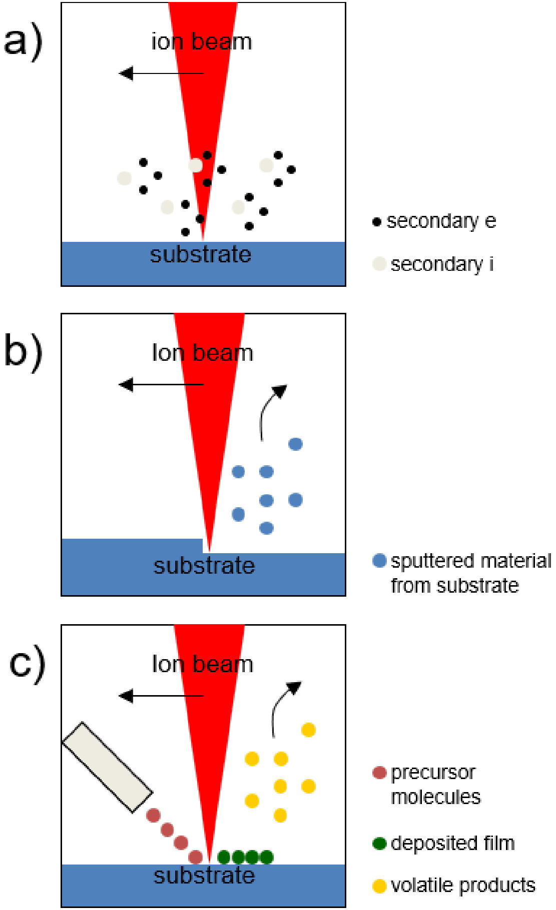
Ion Solid interaction



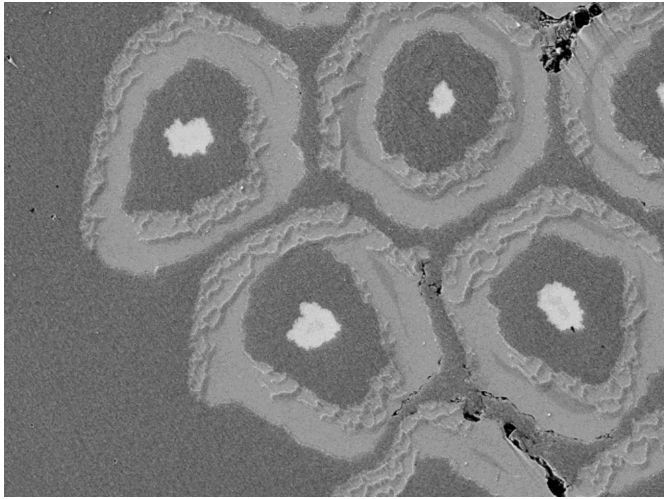
- SE emission – SE image (2-3 SE per ion)
- Sputtering
- Damage – displacement of atoms in the solid
- Ion implantation

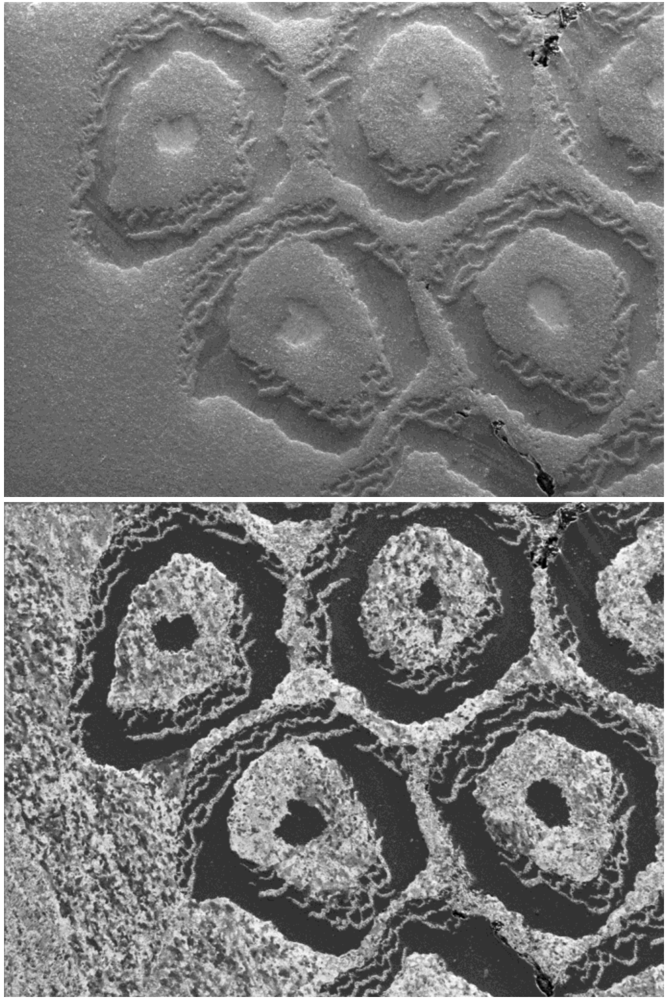
3 basic operating modes

- FIB imaging
  - Emission of secondary electrons and ions
  - Low ion currents
- FIB milling
  - Sputtering of substrate atoms
  - High ion currents
- FIB deposition/enhanced etching
  - Gas assisted chemical interactions

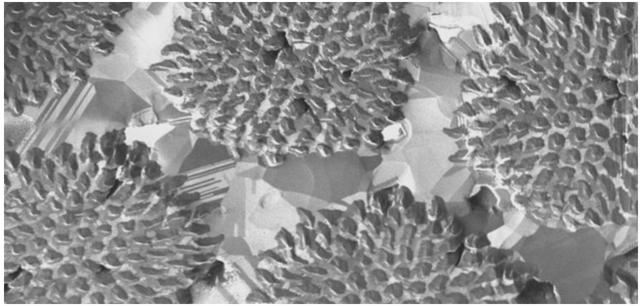


SEM image vs. FIB image

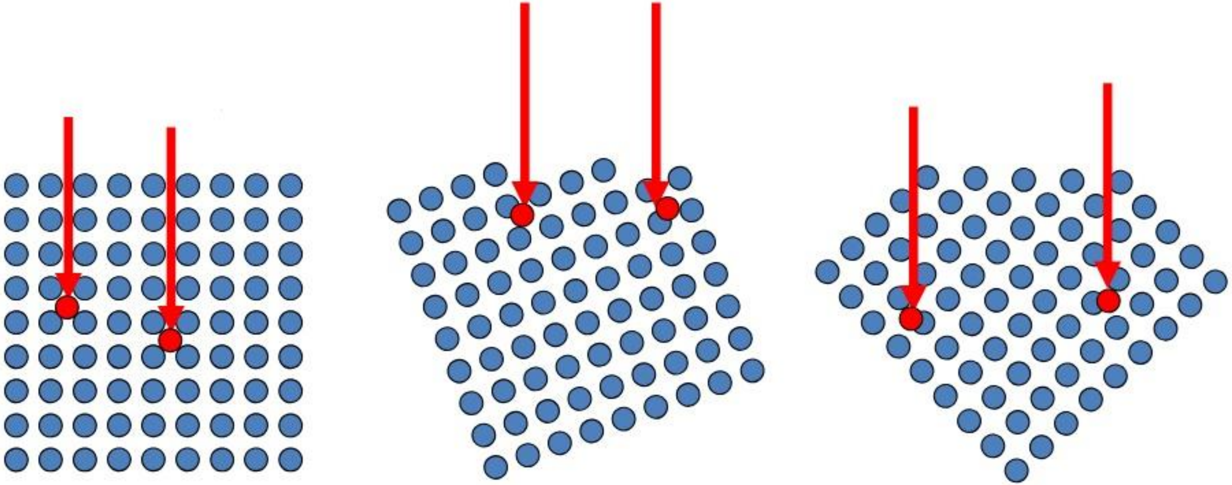




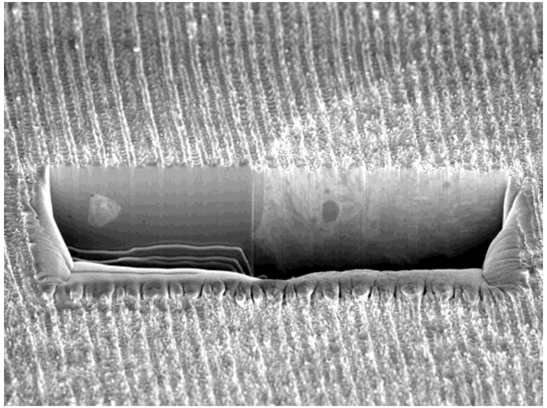
FIB imaging
Channelling contrast

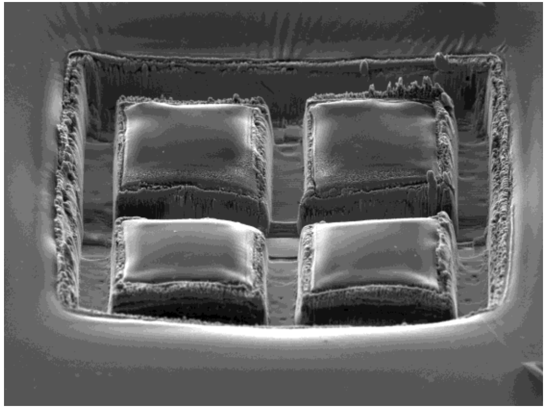


- Atom columns aligned with the ion trajectory => deeper penetration => less sputtering and less SE electrons



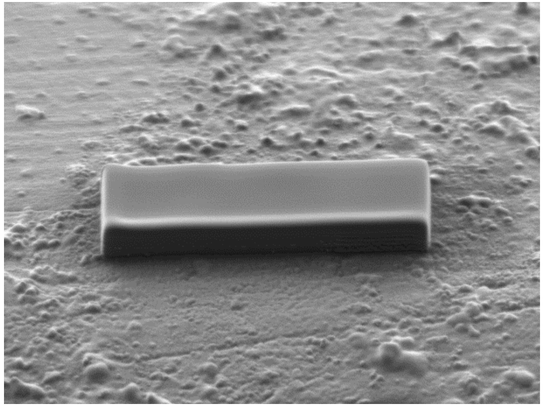
FIB milling

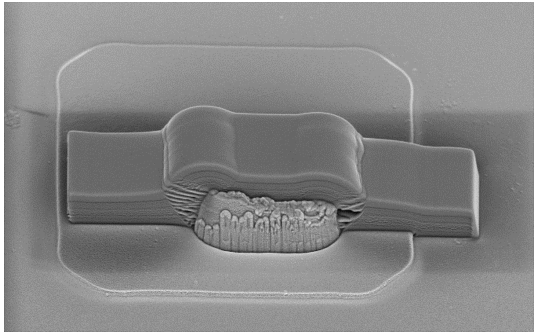




Material	Sputter rate (μm^3/nC)
C	0.18
Al	0.29-0.37
Si	0.15-0.27
Ti	0.37-0.46
Fe	0.29
W	0.11-0.12
Au	1.5-1.8
SiO_2	0.19-0.24
TiO	0.15
Al_3O_2	0.30 - 0.42

FIB deposition





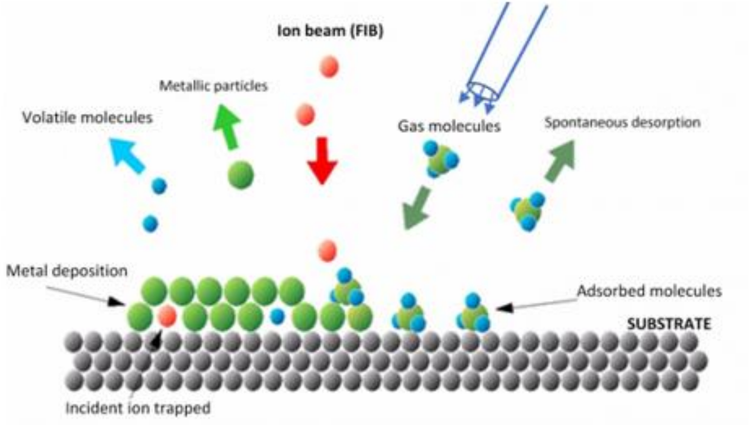
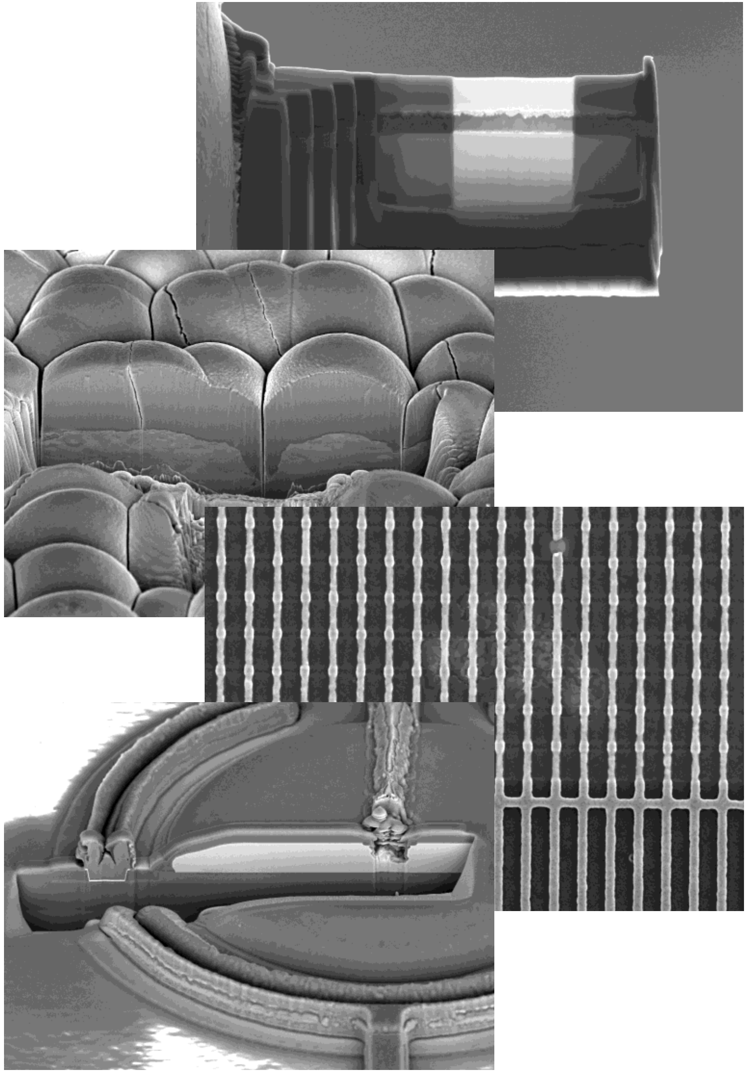


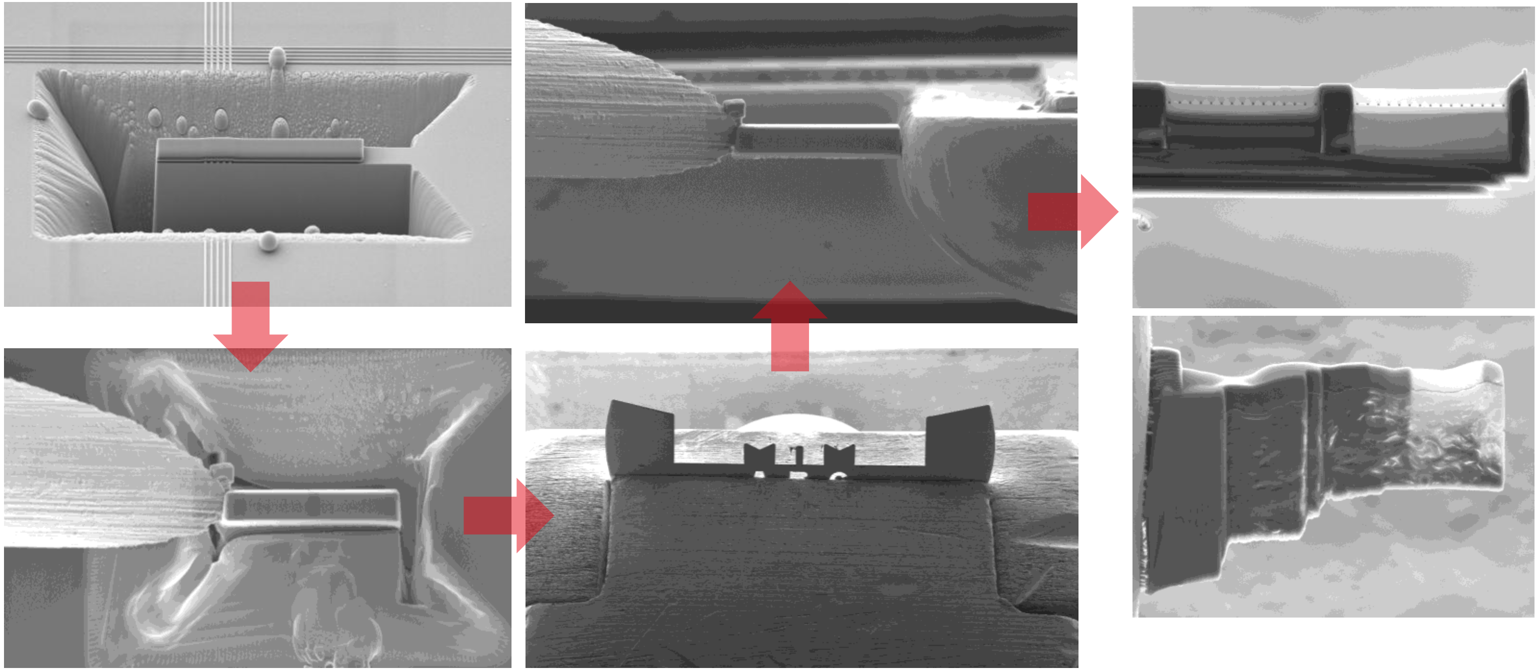
Image source: Orsay Physics

FIB applications

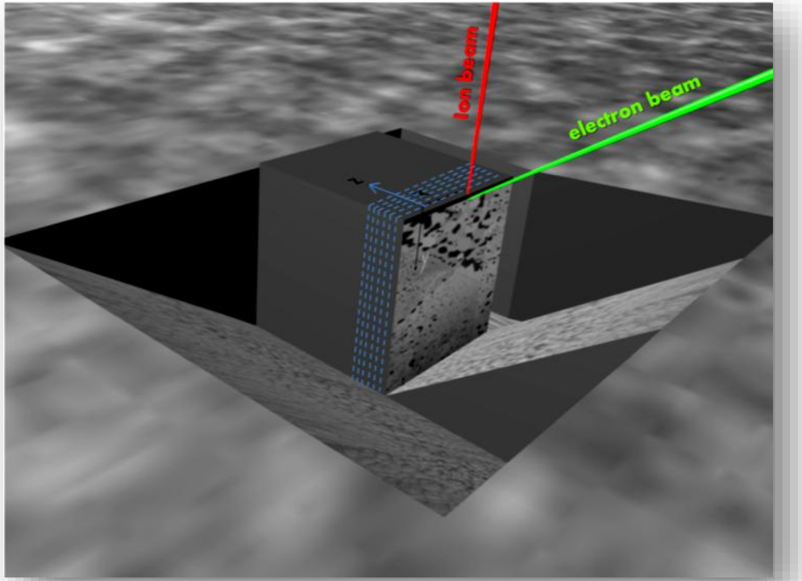
- TEM lamella preparation
  - Precise selection of the position
  - Preparation of heterogenous samples
- 3D nano-tomography
- Failure analysis
- Chip modification
- Nanofabricated structures

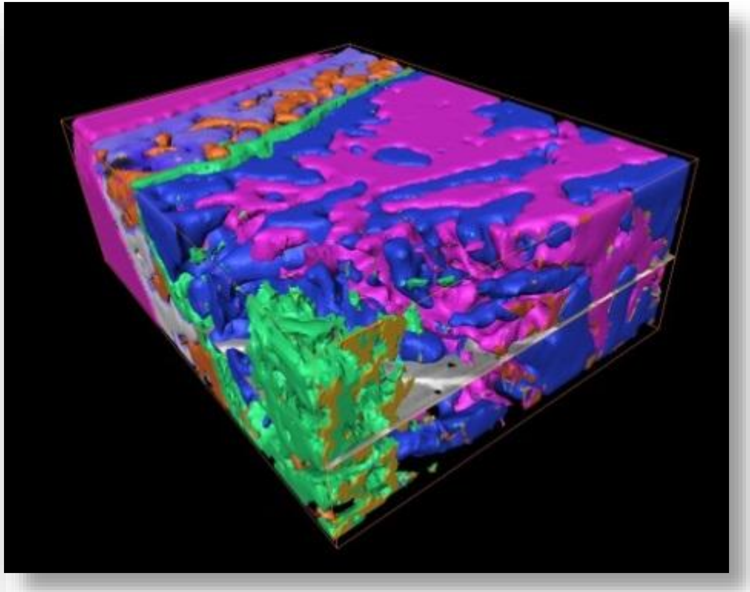


FIB applications
TEM lamella preparation



FIB applications
3D nano-tomography





Summary

- SEM – magnified images with better resolution than light microscope
  - Signal: SE, BSE
  - Volume of interaction – low Z vs. high Z, low vs. high acceleration voltage
  - Depth of field – convergence angle
- EDX – chemical composition
  - accuracy is affected by the nature of the sample
- FIB – from 2D SEM to small 3D volume analysis
  - milling, deposition
  - TEM lamella preparation, 3D tomography, nanofabrication

Thank you for your attention