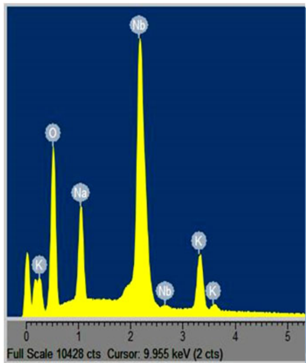


Essentials of SEM, EDX and FIB/SEM

Marco Cantoni
Ecole Polytechnique Fédérale Lausanne

Centre Interdisciplinaire de Microscopie Electronique
(EPFL-CIME)

Outline

SEM

- Basics, Signals, Detectors

EDX

- X-ray generation, detection, quantification

FIB/SEM

- Basics, Tomography

Some typical SEM

ZEISS NVISION

At CIME:
Zeiss NVision40
(FIB)

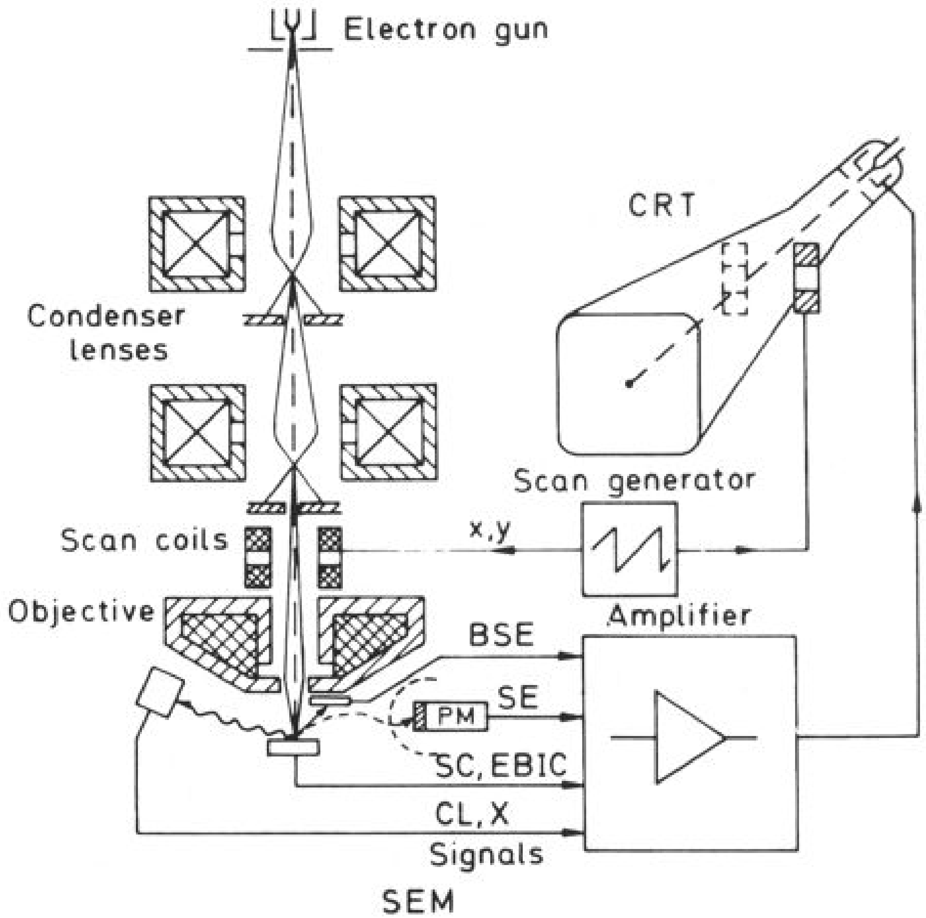
Electron beam resolution
- (site survey required to determine attainable resolution)
- Resolution @ optimum WD
  - 0.8 nm at 15 kV
  - 0.8 nm at 2 kV
  - 0.9 nm at 1 kV
  - 1.5 nm at 200 V
- Resolution @ coincident point
  - 0.8 nm at 15 kV
  - 0.9 nm at 5 kV
  - 1.2 nm at 1 kV

Maximum horizontal field width
- E-beam: 1.5 mm at beam coincident point (WD 4 mm)

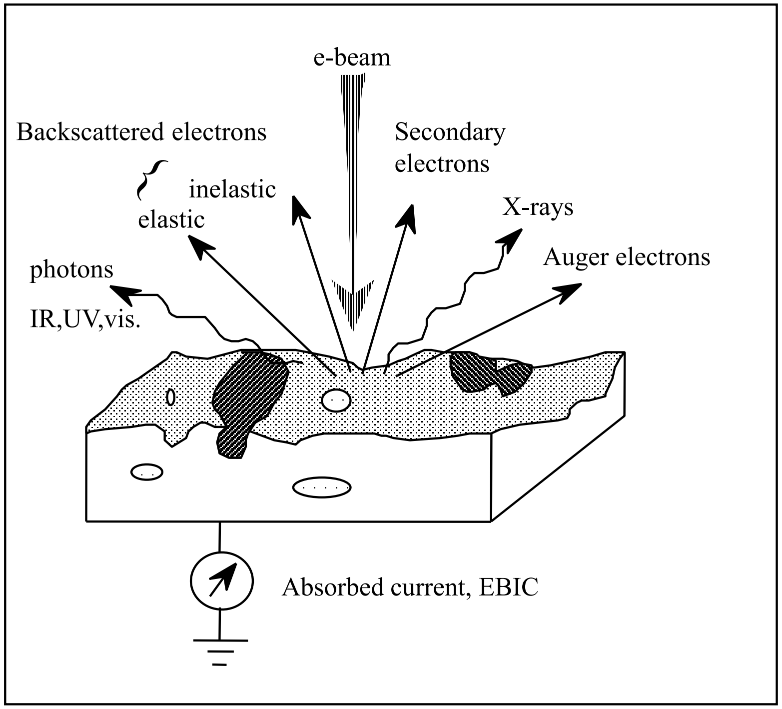
Landing energy range
- 50 V – 30 kV

Probe current
- E-beam: 1 pA to 22 nA

Image formation



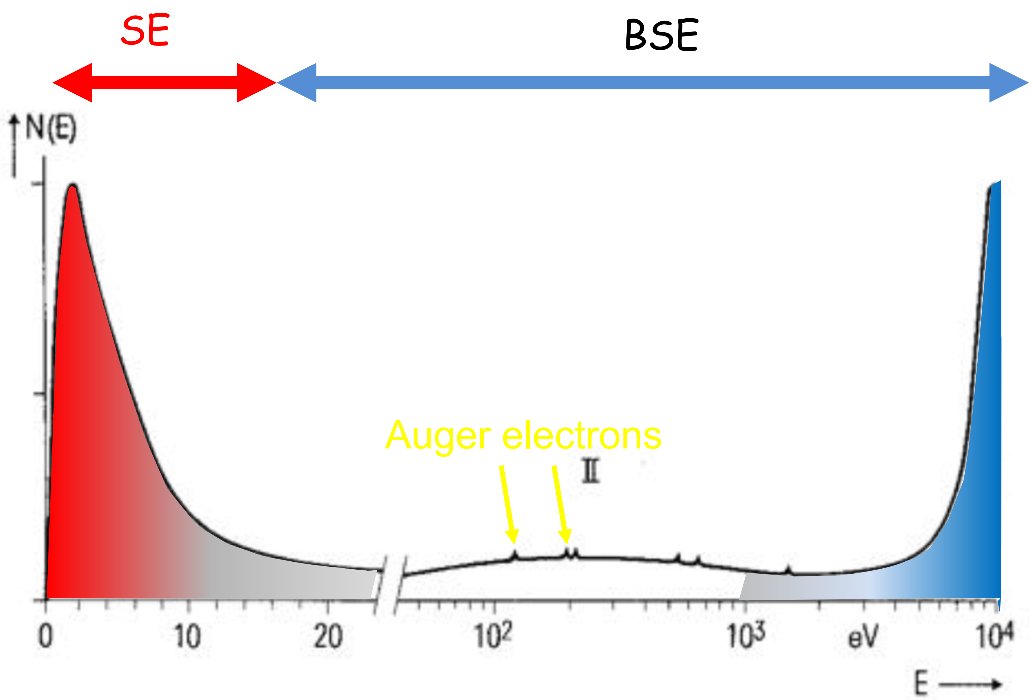
SEM, signals



- Secondary electrons (~0-30eV), **SE**
- Backscattered electrons (~eVo), **BSE**
- Auger electrons
- Photons: visible, UV, IR, **X-rays**
- Phonons, Heating
- Absorbtion of incident electrons (EBIC-Current)

SEM imaging with electrons

- Energy spectrum of electrons leaving the sample

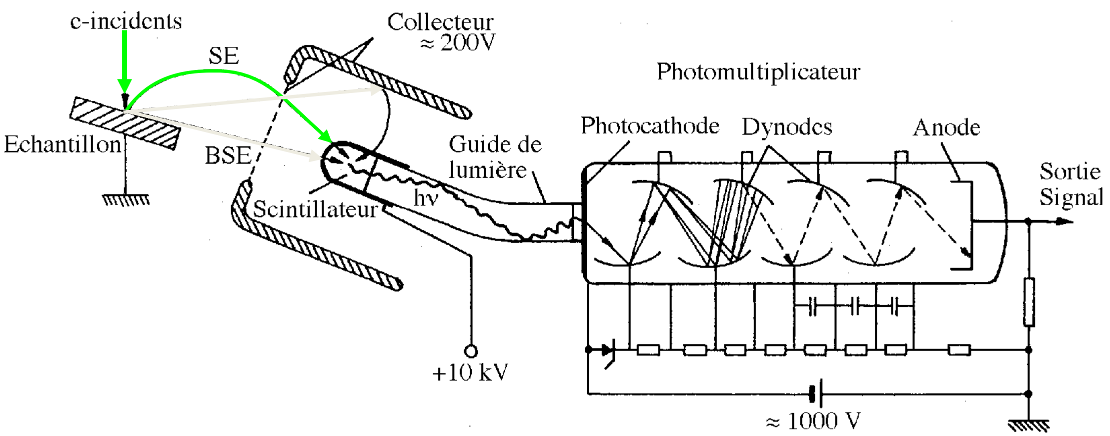


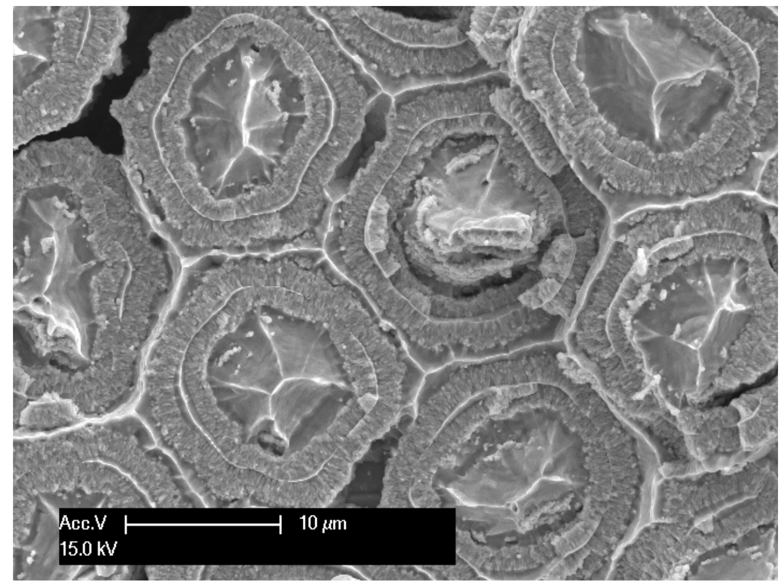
SE: secondary electrons 0-50eV
BSE: backscattered electrons E>50eV

Electron detectors I, secondary electrons

Everhart-Thornley detector

Photomultiplier



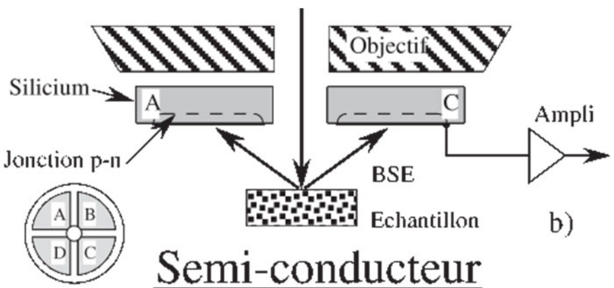


Collects and detects lower energy (<100eV) electrons: **SEM secondary electrons**

Topography information

Electron detectors II, backscattered electrons

- Semiconductor type



BSE semiconductor detector: a silicon diode with a p-n junction close to its surface collects the BSE (3.8eV/ehole pair)

- large collection angle
- slow (poor at TV frequency)
- some diodes are split in 2 or 4 quadrants to bring spatial BSE distribution info

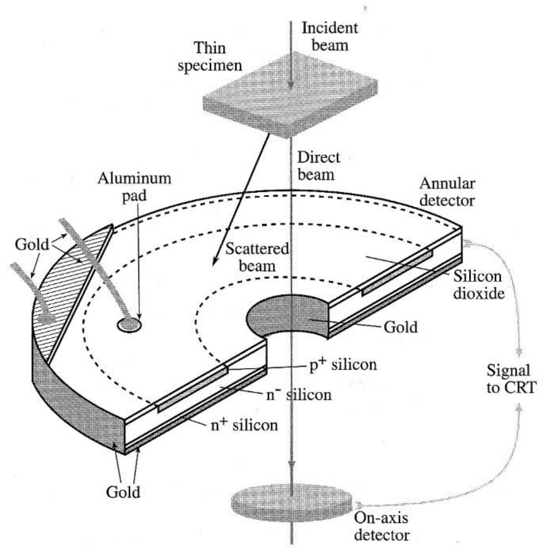
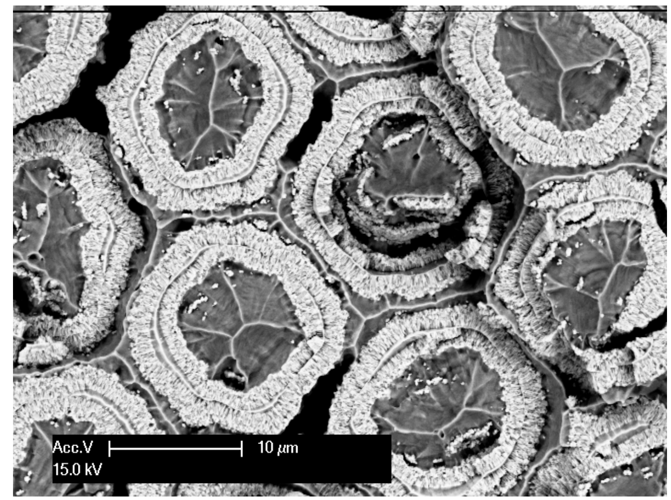


Figure 7.1. Semiconductor detector of the surface-barrier type, shown in a configuration where it would be used to detect high-energy forward-scattered electrons. The direct beam is detected by a small circular detector on the optic axis surrounded by a concentric wide-angle annular detector, which detects any scattered electrons.



« z-contrast »

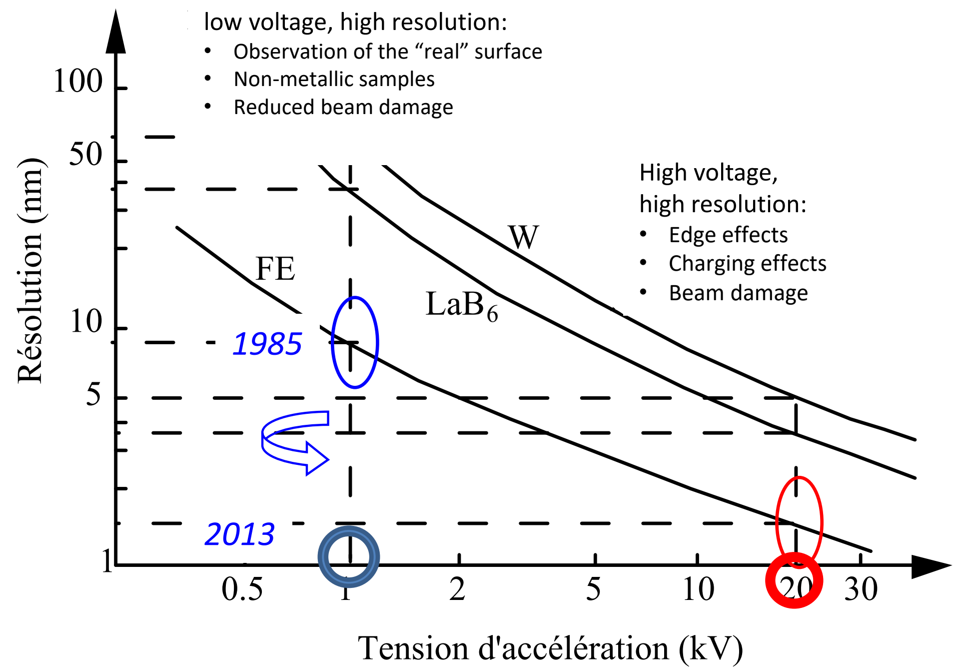
mass-density contrast

Detects higher energy (>5kV) electrons: SEM backscattered electrons

Evolution of SEM resolution

Field emission gun monochromators
beam boosters, beam deceleration,
Lens-design: In-lens, Semi-in-lens,
immersion lens
Short working distance

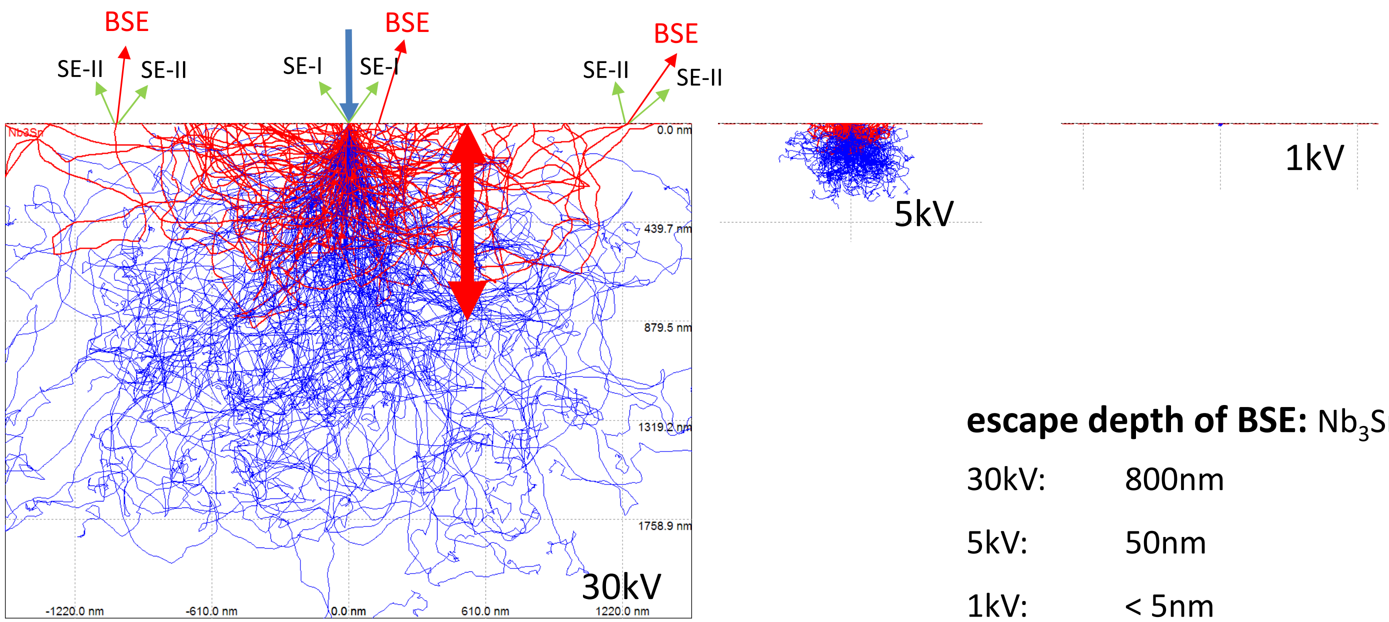
Detectors:
Everhard-Thornley (SE)
In-column (through the lens, in-lens, “in-beam”)
Low Voltage BSE detection, energy filtering (separation of materials and topography contrast)



Analytical SEM:
SDD EDX detectors (high throughput, large collection angle)
High-speed EBSD detectors
Beam currents of several 100 nA

SEM interaction volume

Electron trajectories in matter



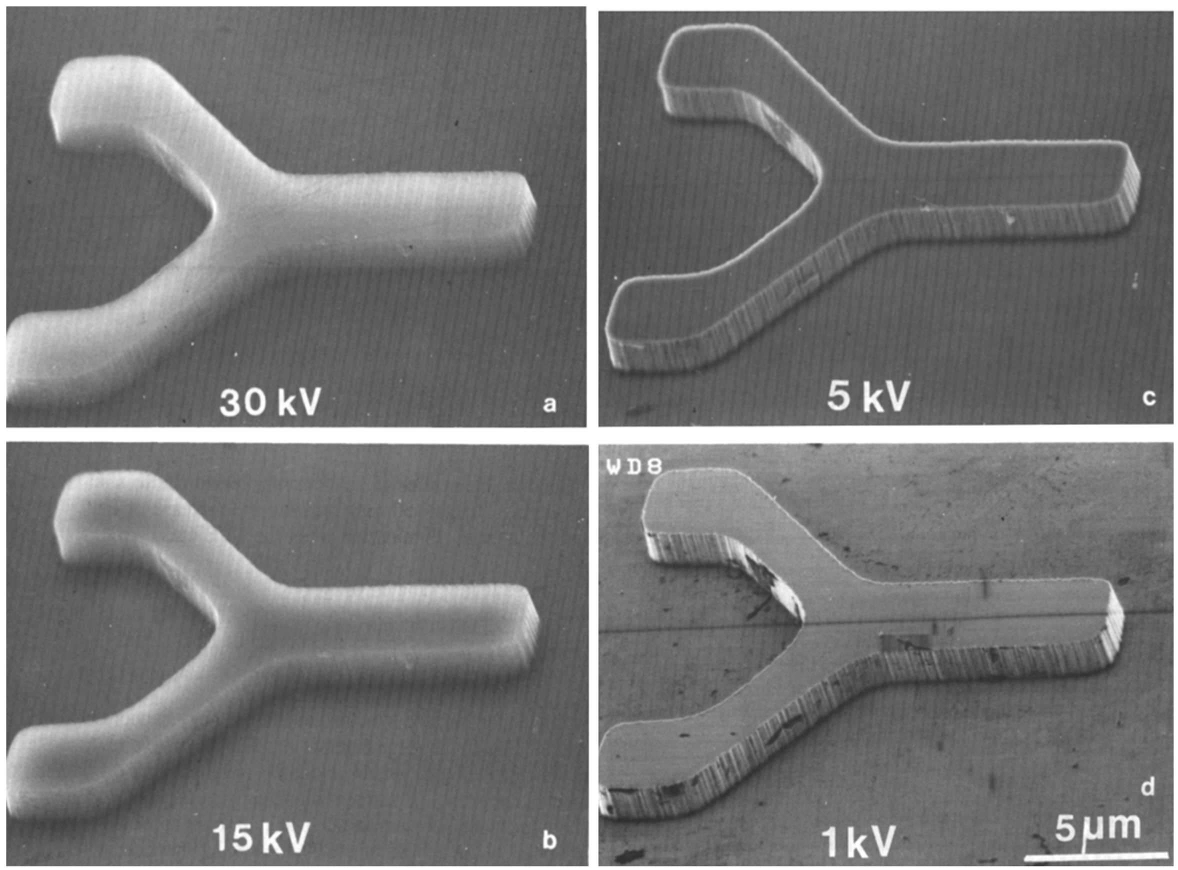
escape depth of BSE: Nb_3Sn

30kV:	800nm
5kV:	50nm
1kV:	< 5nm

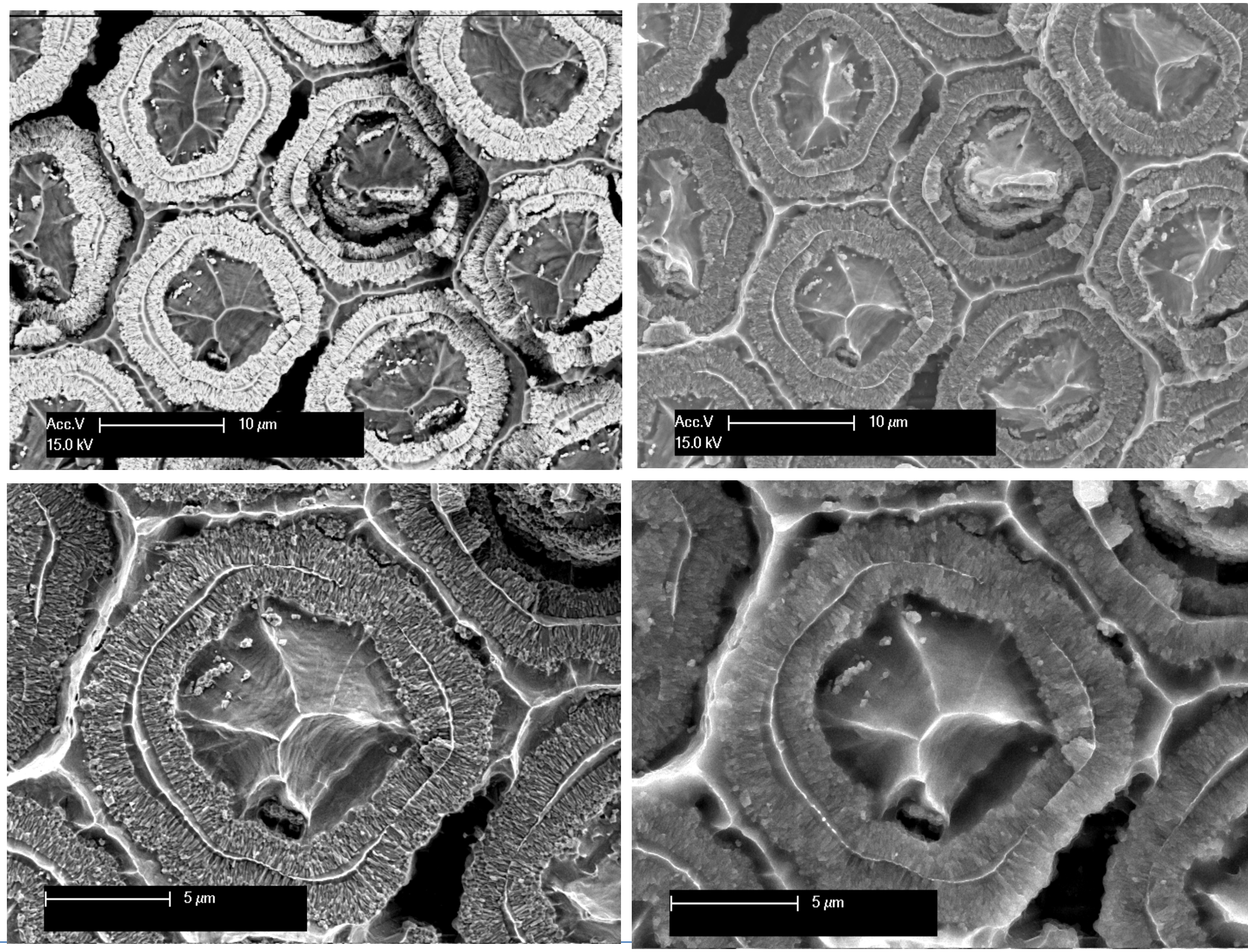
Interaction volume
Blue: scattered electron trajectory
Red: backscattered electrons (leaving the sample surface)
Green: secondary electrons

Monte-Carlo Simulation CASINO v2.42

Change in secondary electron (SE) contrast with accelerating voltage

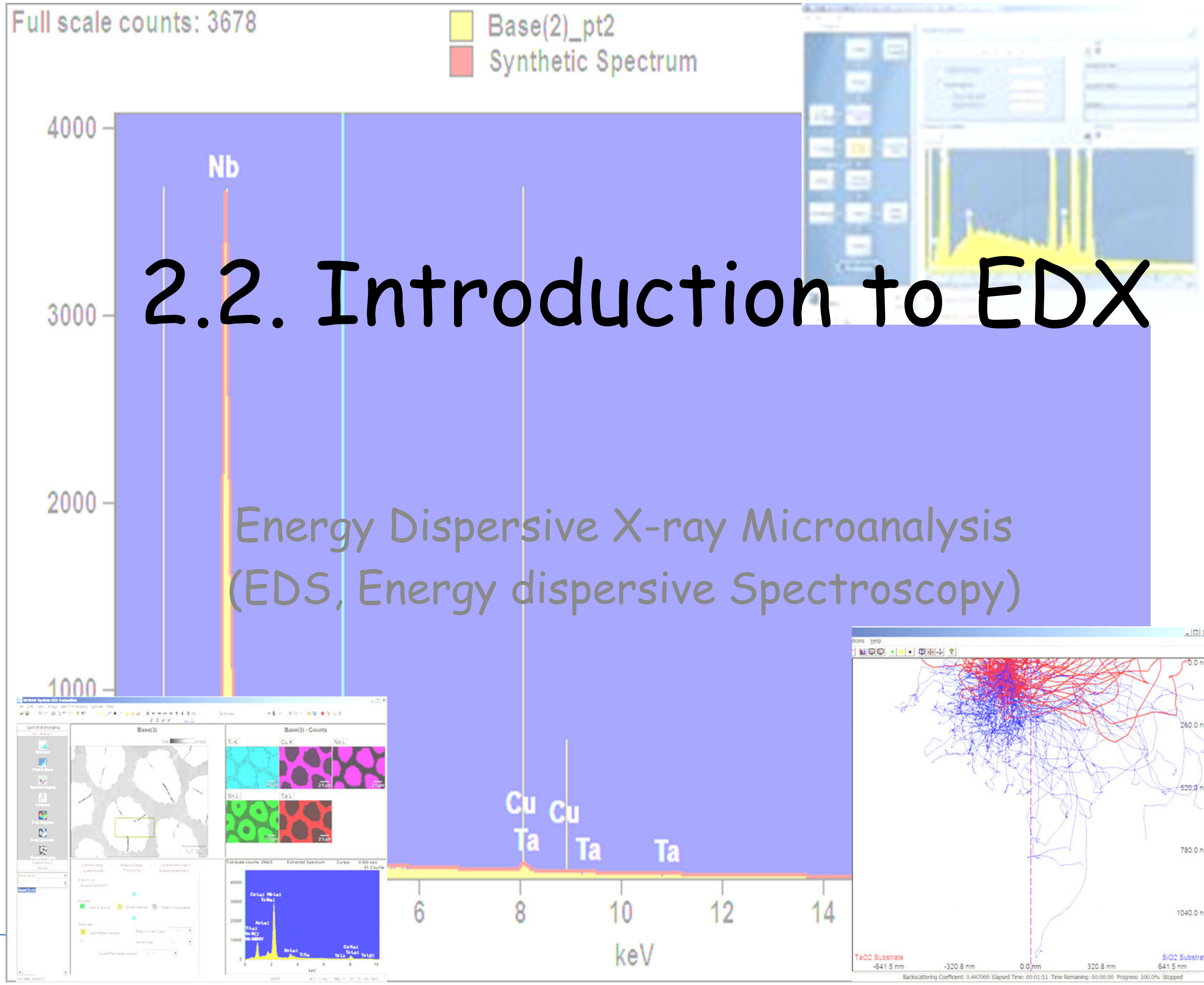


(from L.Reimer, Image formation in the low-voltage SEM)

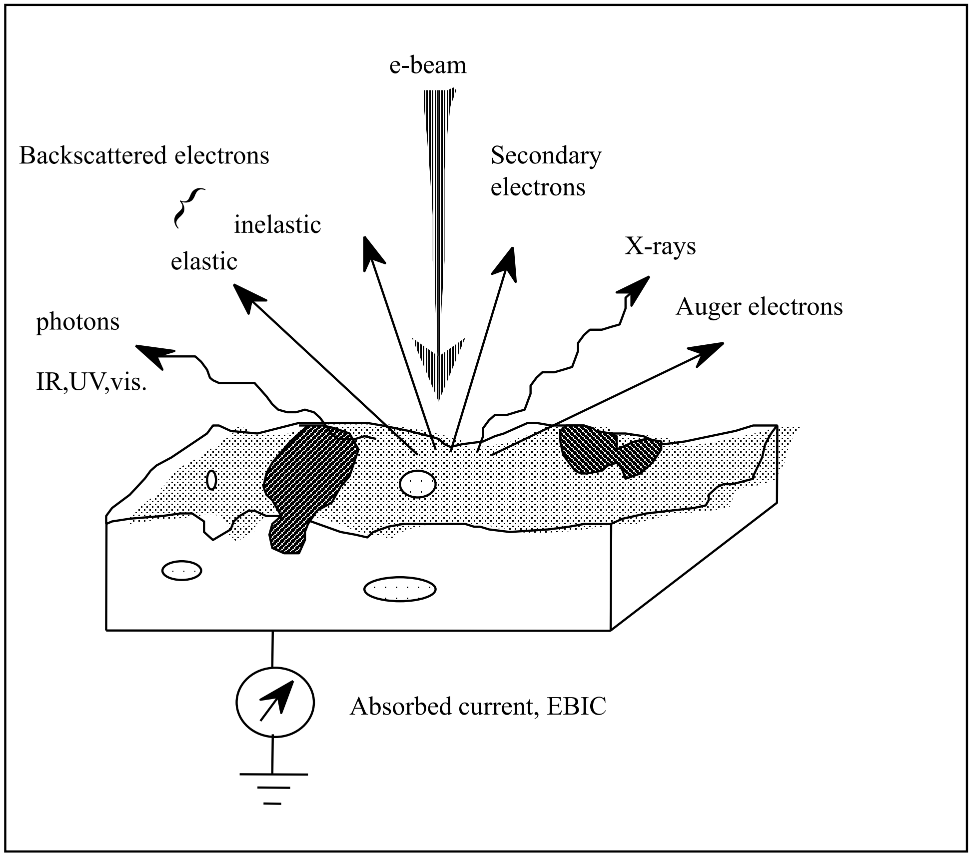

Acc.V
10 μm
15.0 kV
Acc.V
10 μm
15.0 kV
5 μm
5 μm

2.2. Introduction to EDX

Energy Dispersive X-ray Microanalysis
(EDS, Energy dispersive Spectroscopy)



SEM, signals

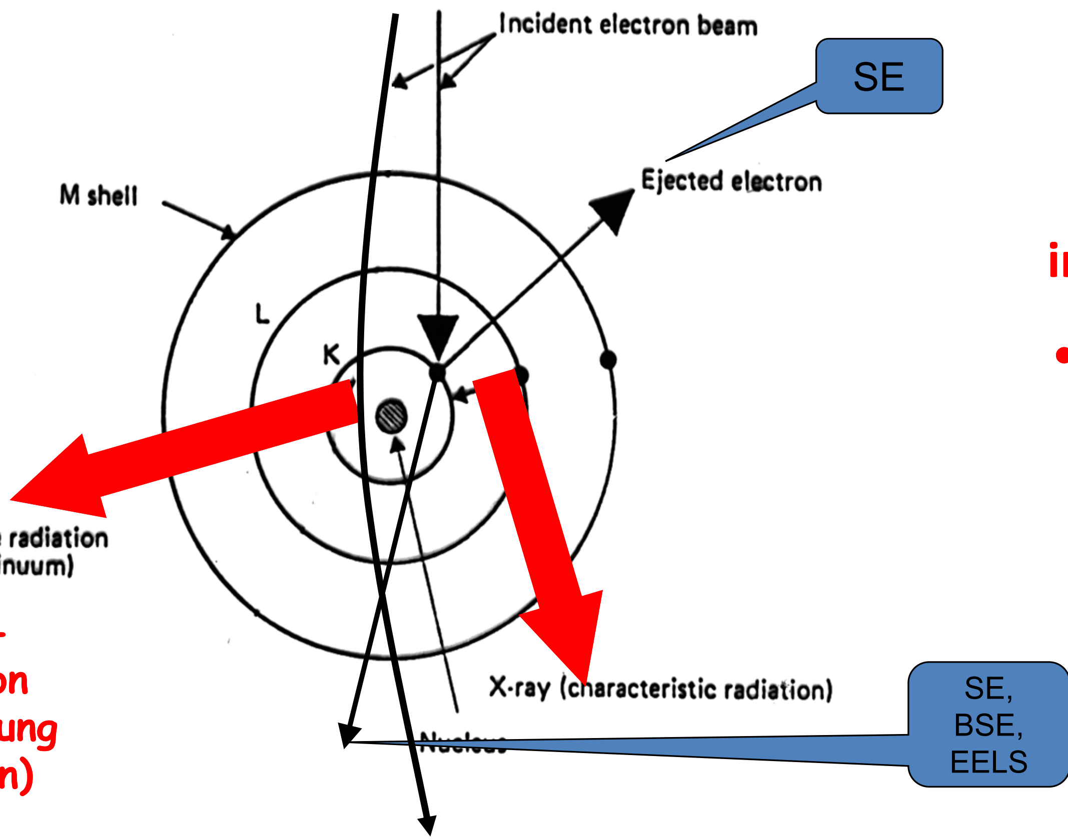


- Secondary electrons (~0-30eV), **SE**
- Backscattered electrons (~eVo), **BSE**
- Auger electrons
- Photons: visible, UV, IR, **X-rays**
- Phonons, Heating
- Absorbtion of incident electrons (EBIC-Current)

Basics of EDX

- a) Generation of X-rays
- b) Detection
- c) Quantification
  EDX in SEM, Interaction volume
  Monte-Carlo-Simulations

X-ray generation:
Inelastic scattering of electrons at atoms
$E_{electron\_in} > E_{electron\_out}$



inner shell ionization

- Characteristic X-ray emission

- Continuum X-ray production (Bremsstrahlung, Synchrotron)

Forbidden transitions !
quantum mechanics:
conservation of angular momentum

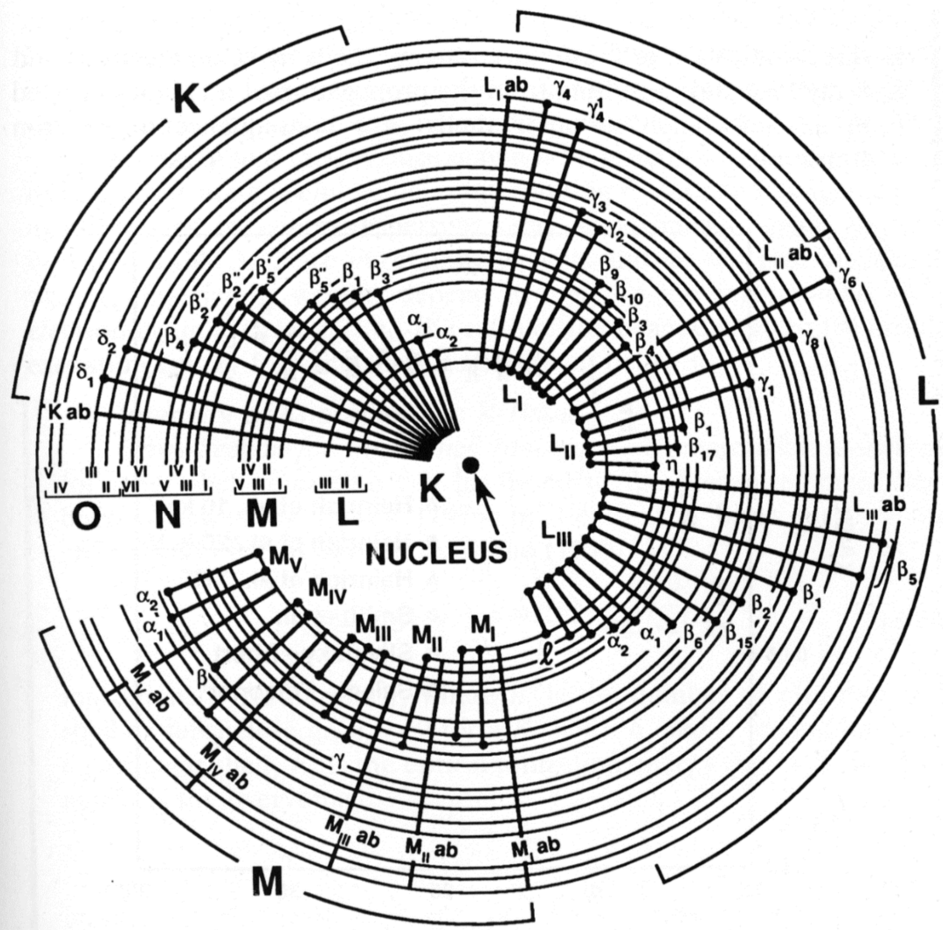


Figure 3.37. Comprehensive energy-level diagram showing all electron transitions which give rise to *K*, *L*, and *M* x rays (Woldseth, 1973).

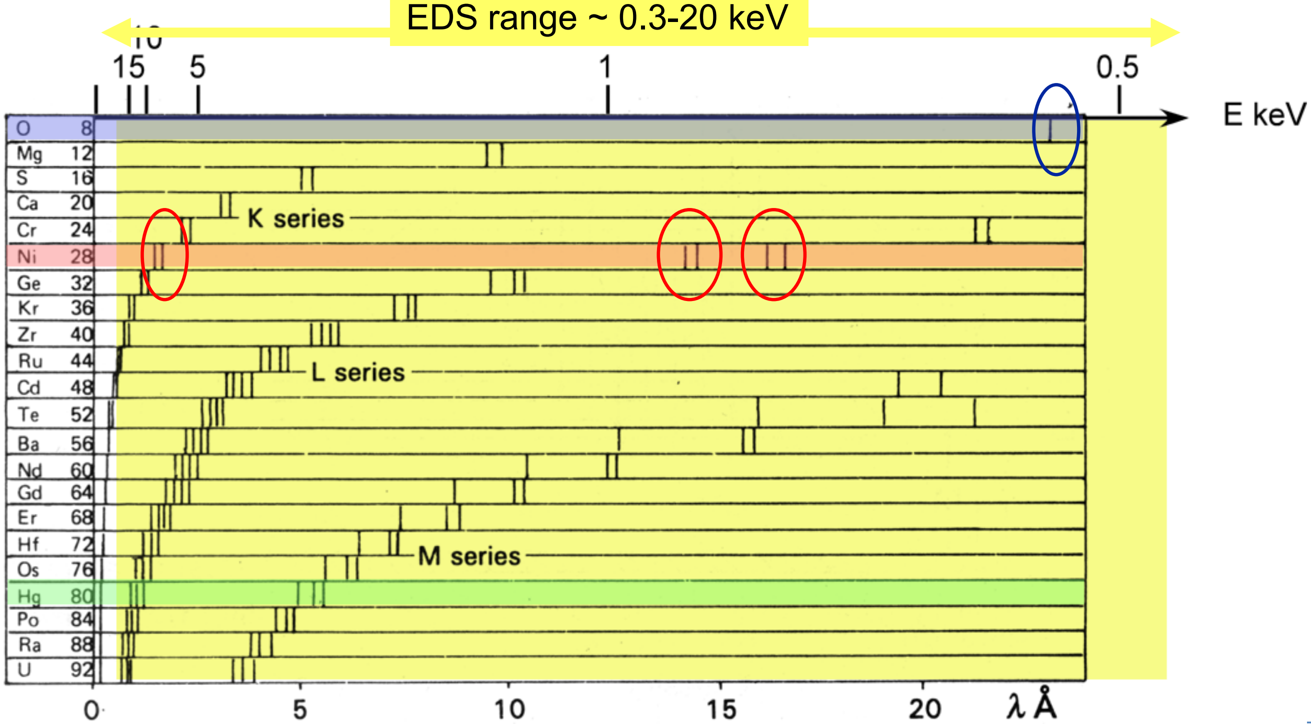
Characteristic lines: Moseley's Law

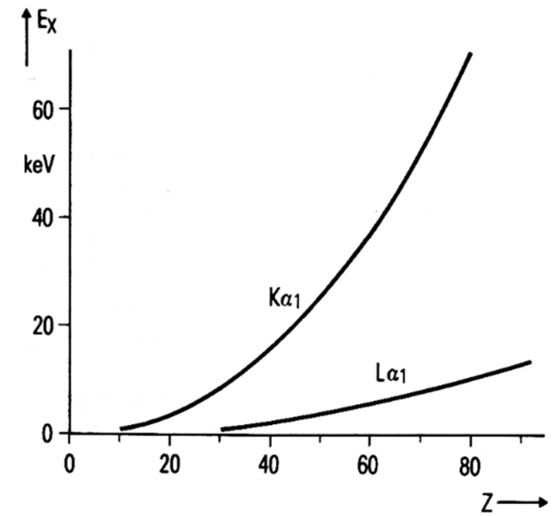
Frequency ν of X-rays emitted from K-level vs. atomic number

E= hν et λ=c/ν

$$\nu = 2.48\,10^{15}(Z-1)^2$$

with the Planck constant:h=6.626 068 76(52) × 10^{-34} J·s
and 1eV = 1.6 10^{-19} J





To assess an element
all detectables lines
MUST
be present!!!

known ambiguities:

Al Kα = Br Ll
S Kα = Mo Ll

b) Detection of X-rays (EDX)

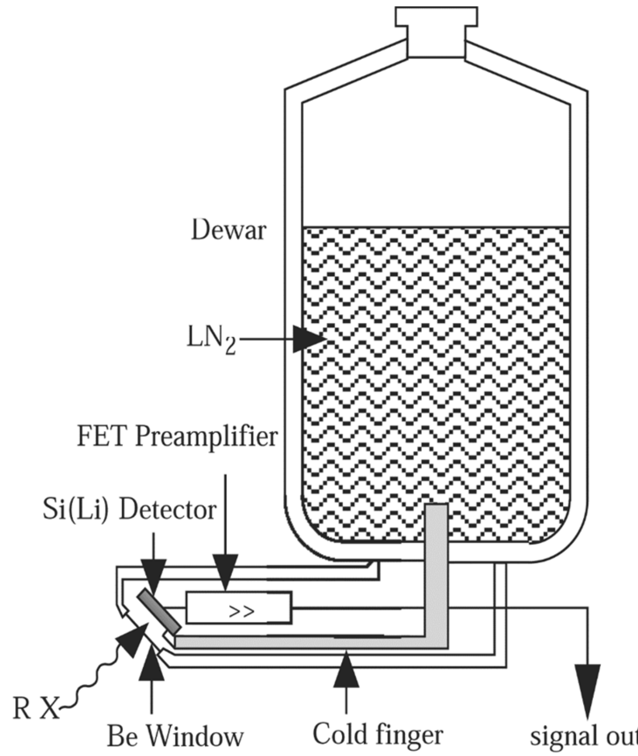
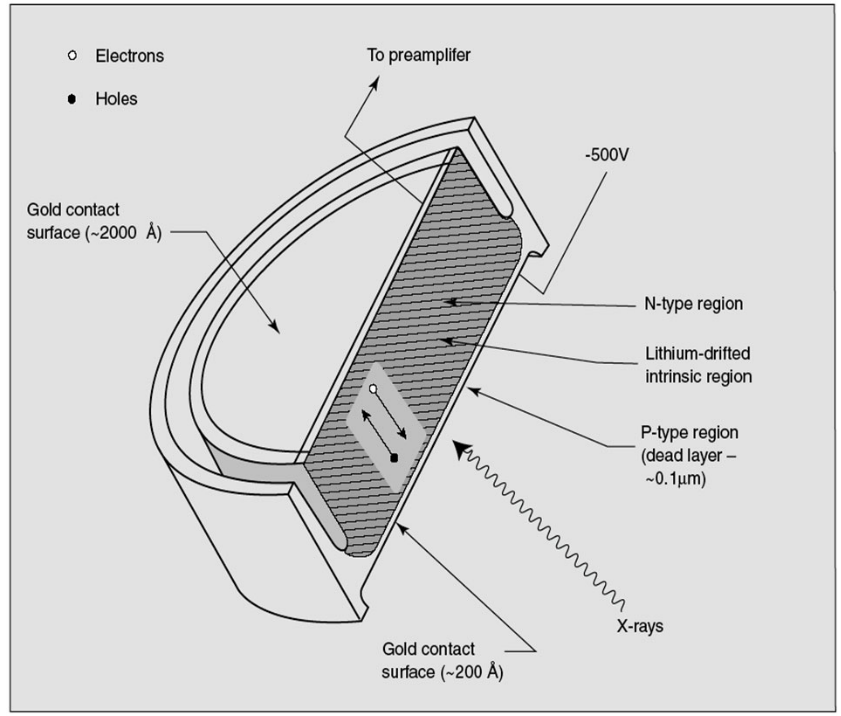
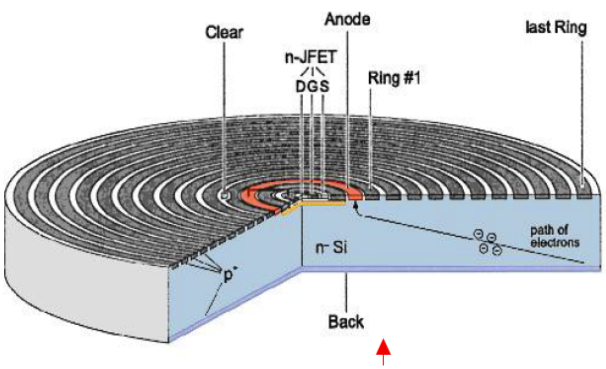


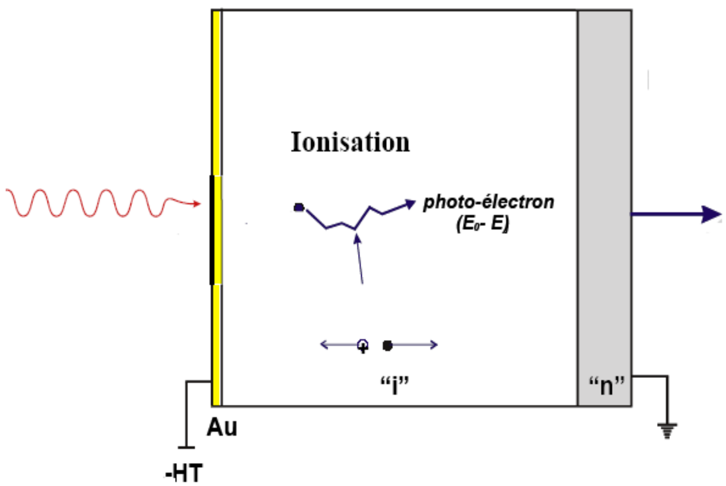
Figure 4-2. *Cross section of a typical lithium-drifted silicon detector. X-rays create electron-hole pairs in the intrinsic region of the semiconductor; these charge carriers then migrate to the electrodes under the influence of an applied bias voltage.*

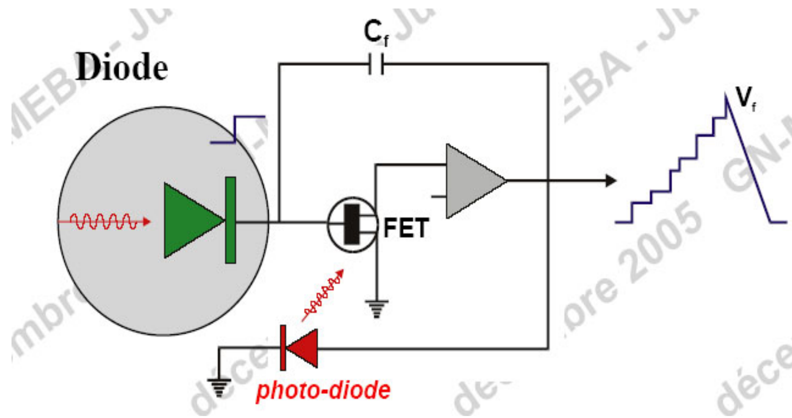


Right: Si(Li) detector
Cooled down to liquid nitrogen temperature

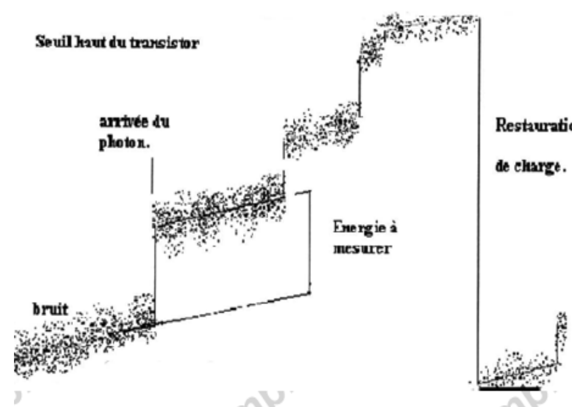


modern silicon drift (SDD) detector:
no LN cooling required



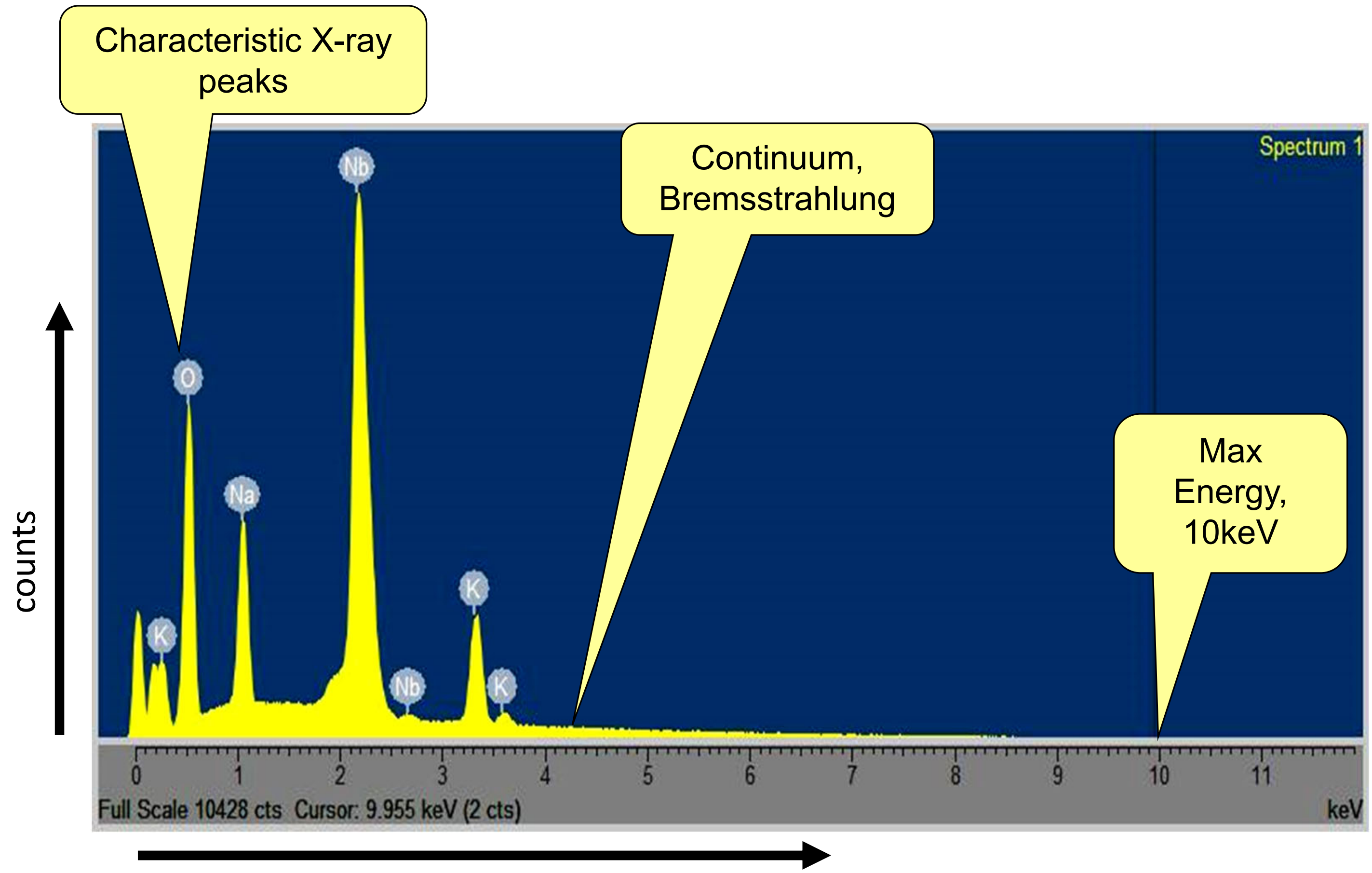


X-Ray energy conversion to electrical charges:
3.8eV / electron-hole pair in average
electronic noise+ imperfect charge collection:
130 eV resolution / Mn Ka line



- Detector acts like a diode: at room temperature the leak current for 1000V would be too high !
- The FET produces less noise if cooled !
- Li migration at room temperature !
- ->Detector cooling by L-N

EDX spectrum of (K,Na)NbO_3



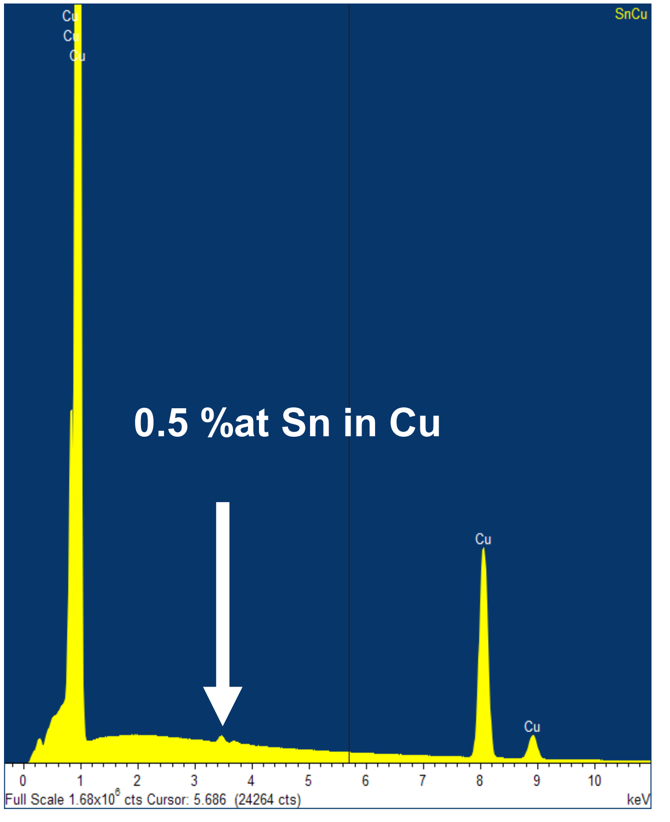
Energy in «channels» of 5/10 eV

Electron beam: 10keV

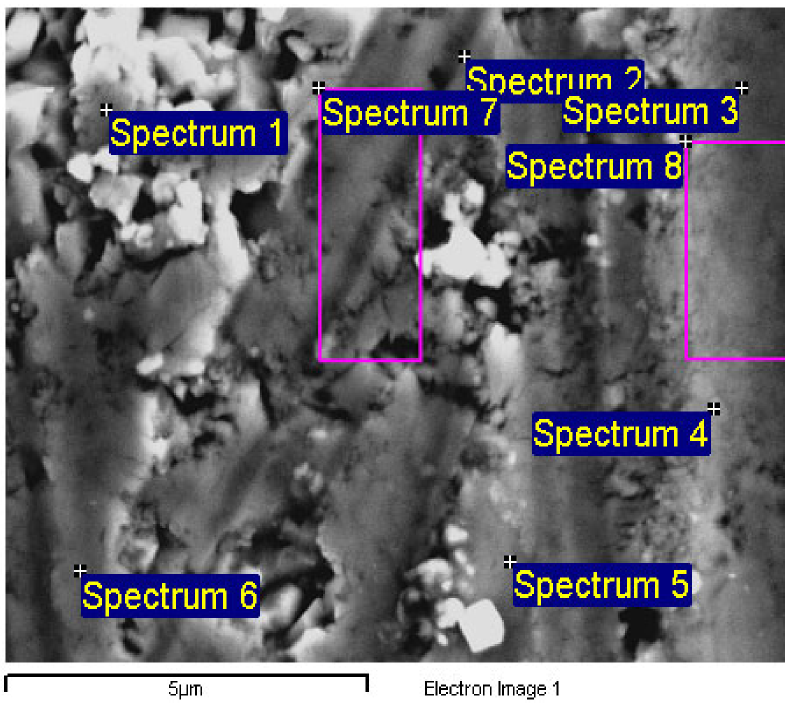
Detection limit for EDS in SEM

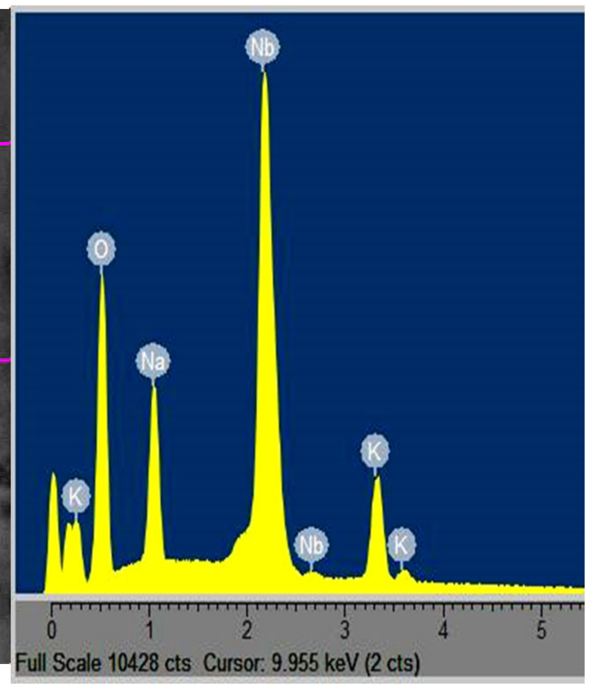
- Acquisition under best conditions
  - Flat surface without contamination (no Au coating, use C instead)
  - Sample must be homogenous at the place of analysis (interaction volume !!)
  - Horizontal orientation of the surface
  - High count rate
  - Overvoltage U=Eo/Ec >1.5-2

- For acquisition times of 100sec. : detection of ~0.5at% for almost all elements



(K,Na)NbO3, Quantification





Spectrum	Na	K	Nb	O	Total
Spectrum 1	8.19	10.18	20.70	60.93	100.00
Spectrum 2	9.59	8.66	20.75	61.00	100.00
Spectrum 3	7.82	9.54	21.13	61.51	100.00
Spectrum 4	9.79	9.37	20.36	60.48	100.00
Spectrum 5	8.86	9.35	20.77	61.02	100.00
Spectrum 6	9.46	9.07	20.63	60.84	100.00
Spectrum 7	8.89	10.25	20.37	60.49	100.00
Spectrum 8	8.60	9.40	20.86	61.14	100.00
Max.	9.79	10.25	21.13	61.51	
Min.	7.82	8.66	20.36	60.48	

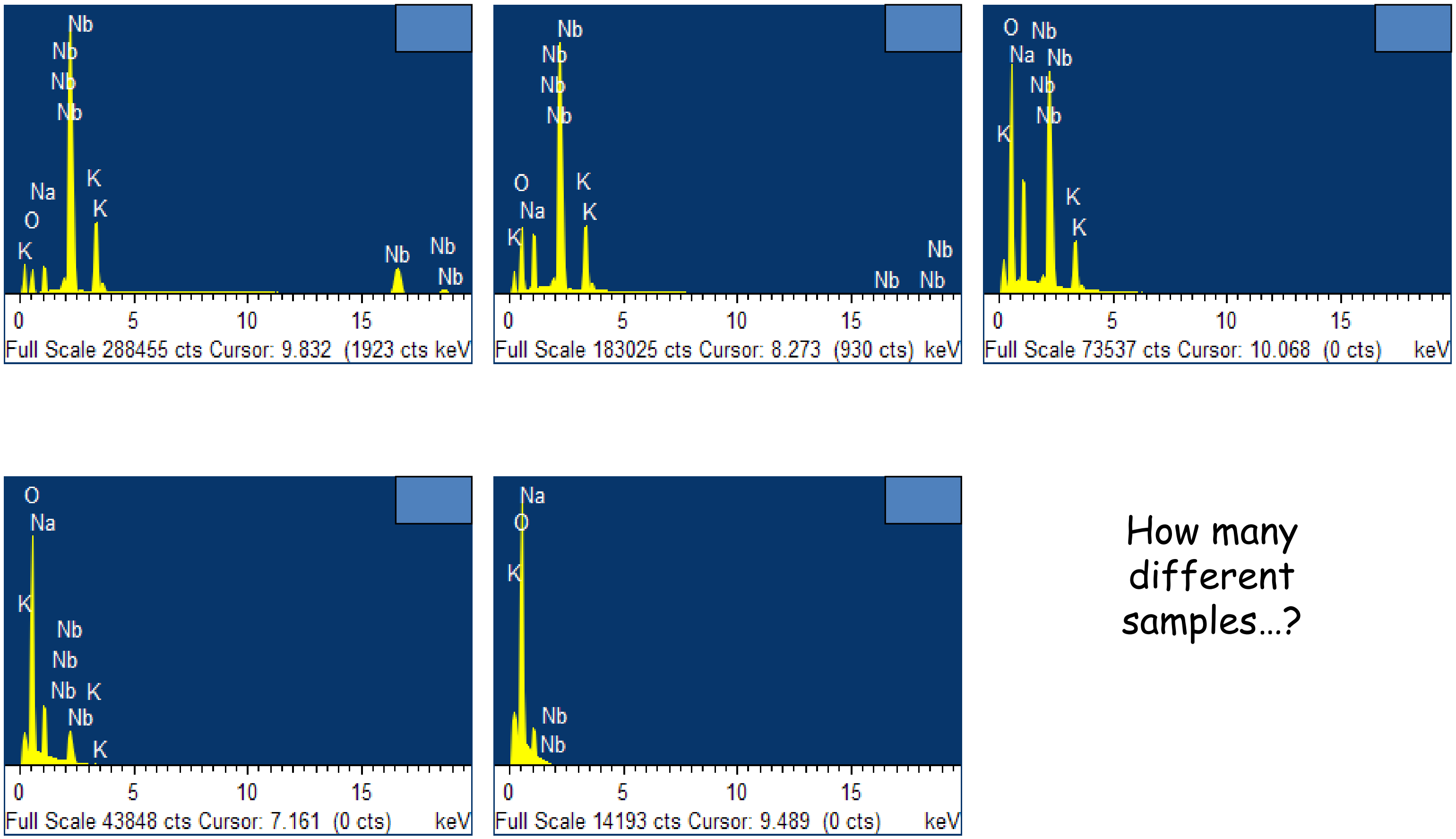
c) Quantification

Yes, but….

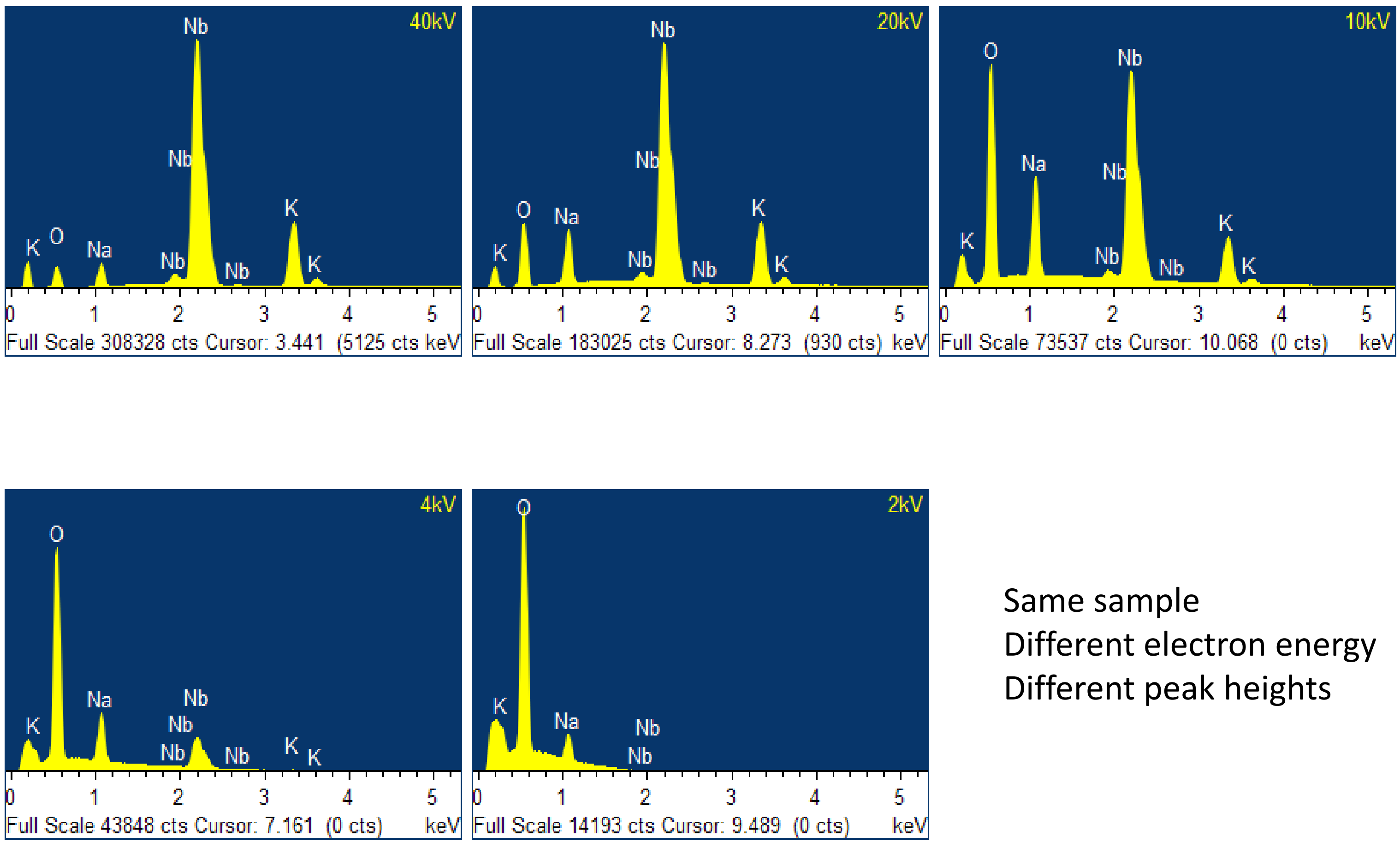
- First approach:
  compare X-ray intensity with a standard (sample with known concentration, same beam current of the electron beam)
- c_i: wt concentration of element i
- I_i: X-ray intensity of char. Line
- k_i: concentration ratio

$$\frac{c_i}{c_i^{std}} = \frac{I_i}{I_i^{std}} = k_i$$

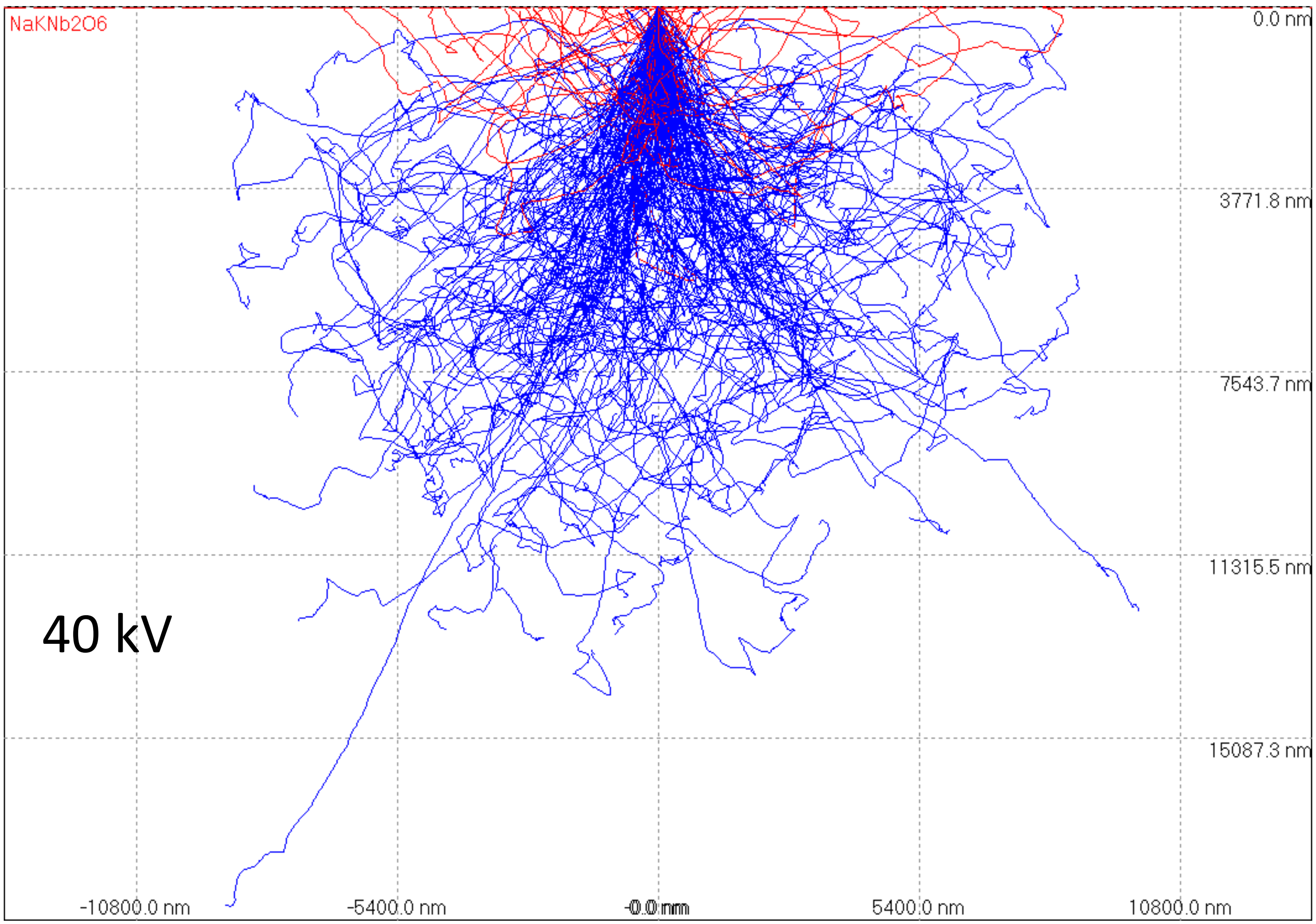
Intensity ~ Concentration…?



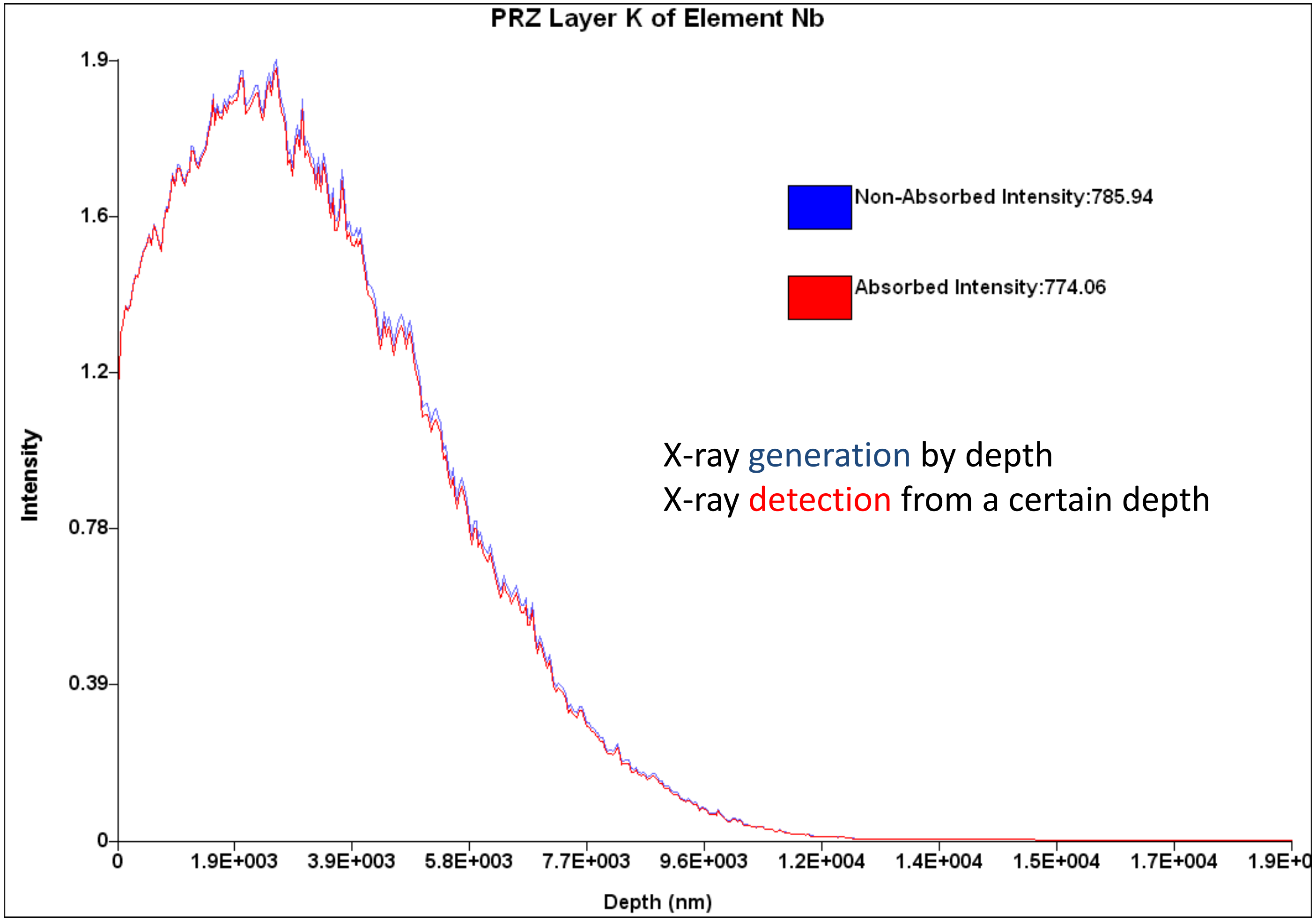
How many different samples…?



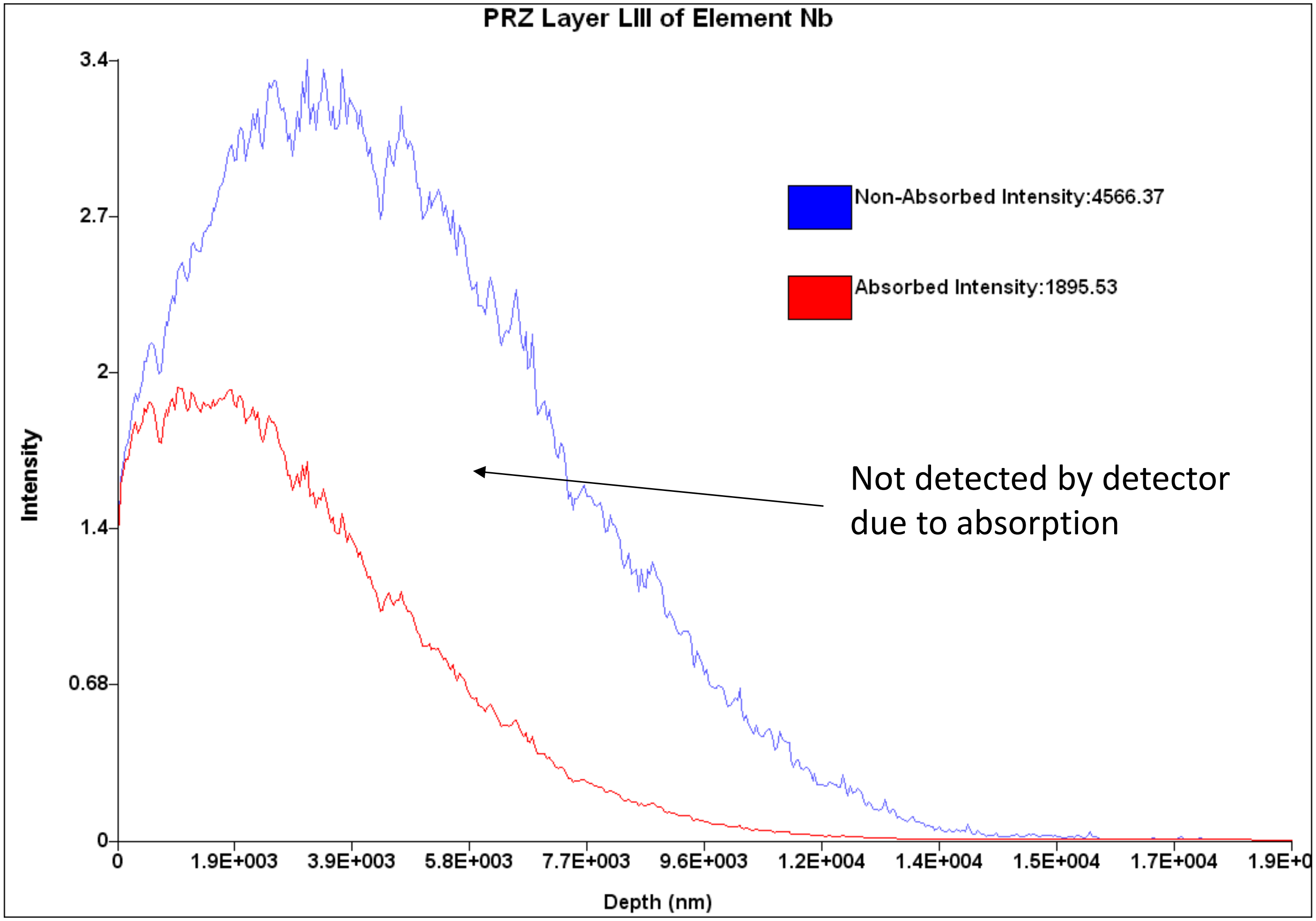
Same sample
Different electron energy
Different peak heights

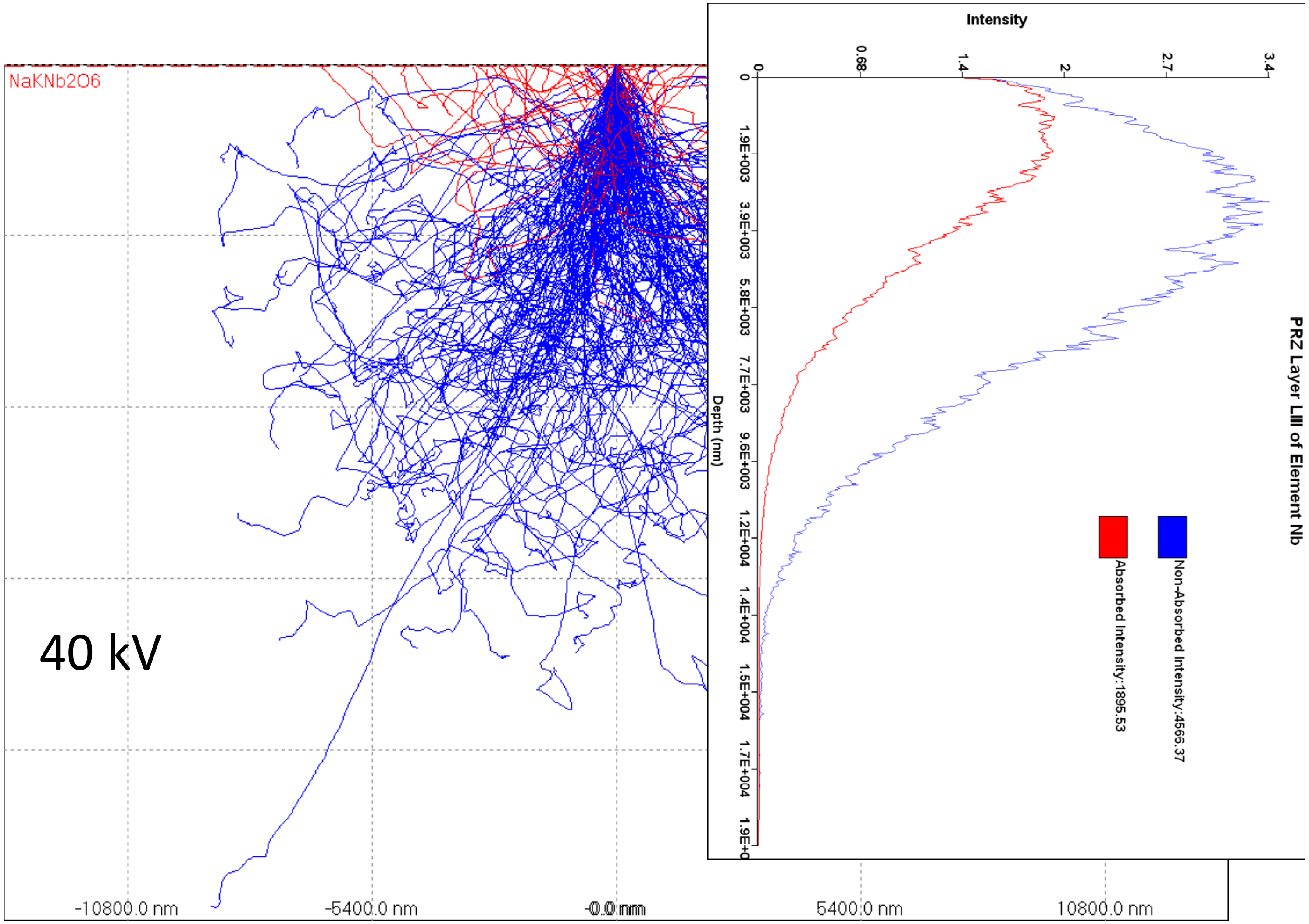


Monte-Carlo Simulation of electron trajectories with CASINO

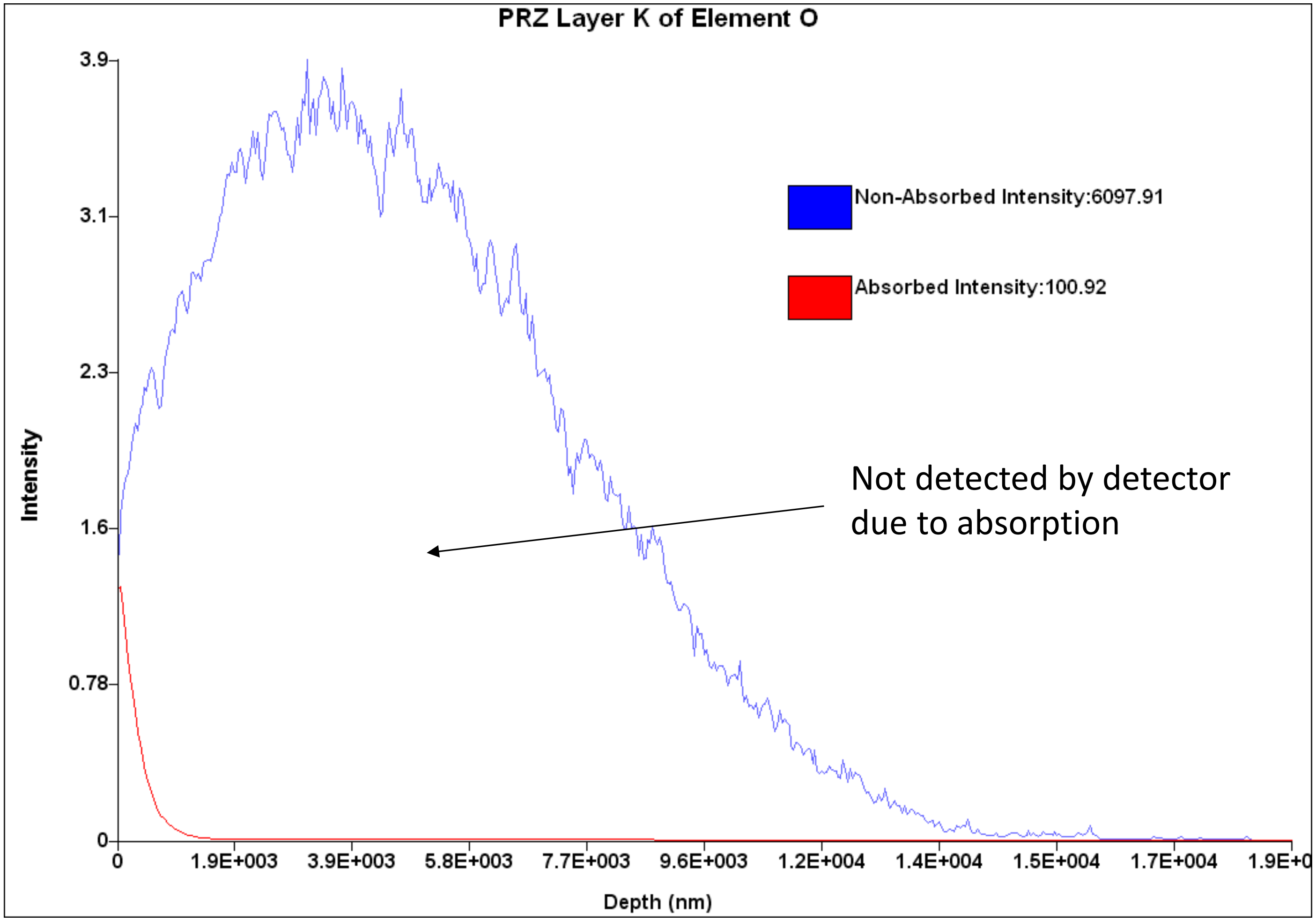


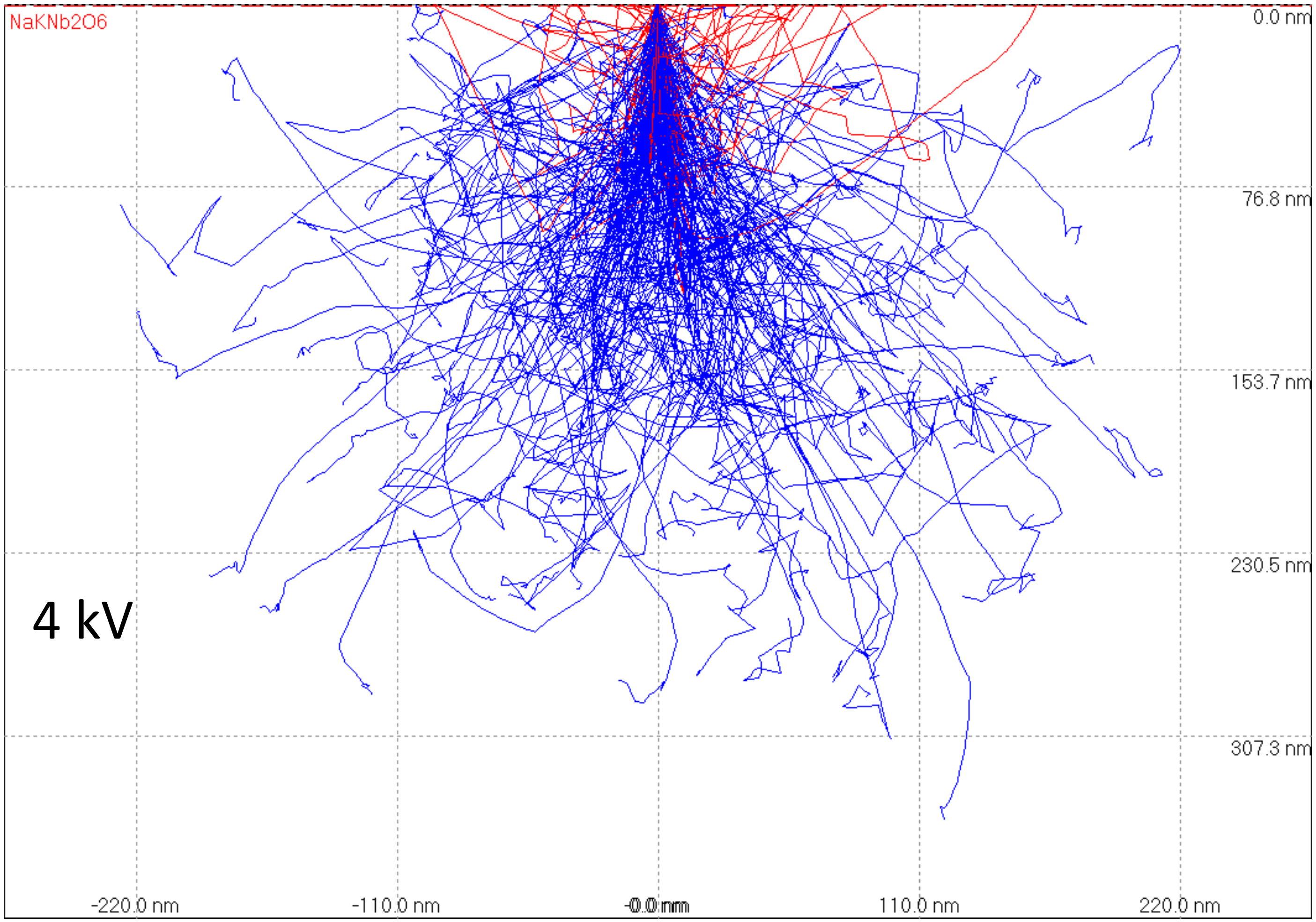
X-ray generation by depth
X-ray detection from a certain depth

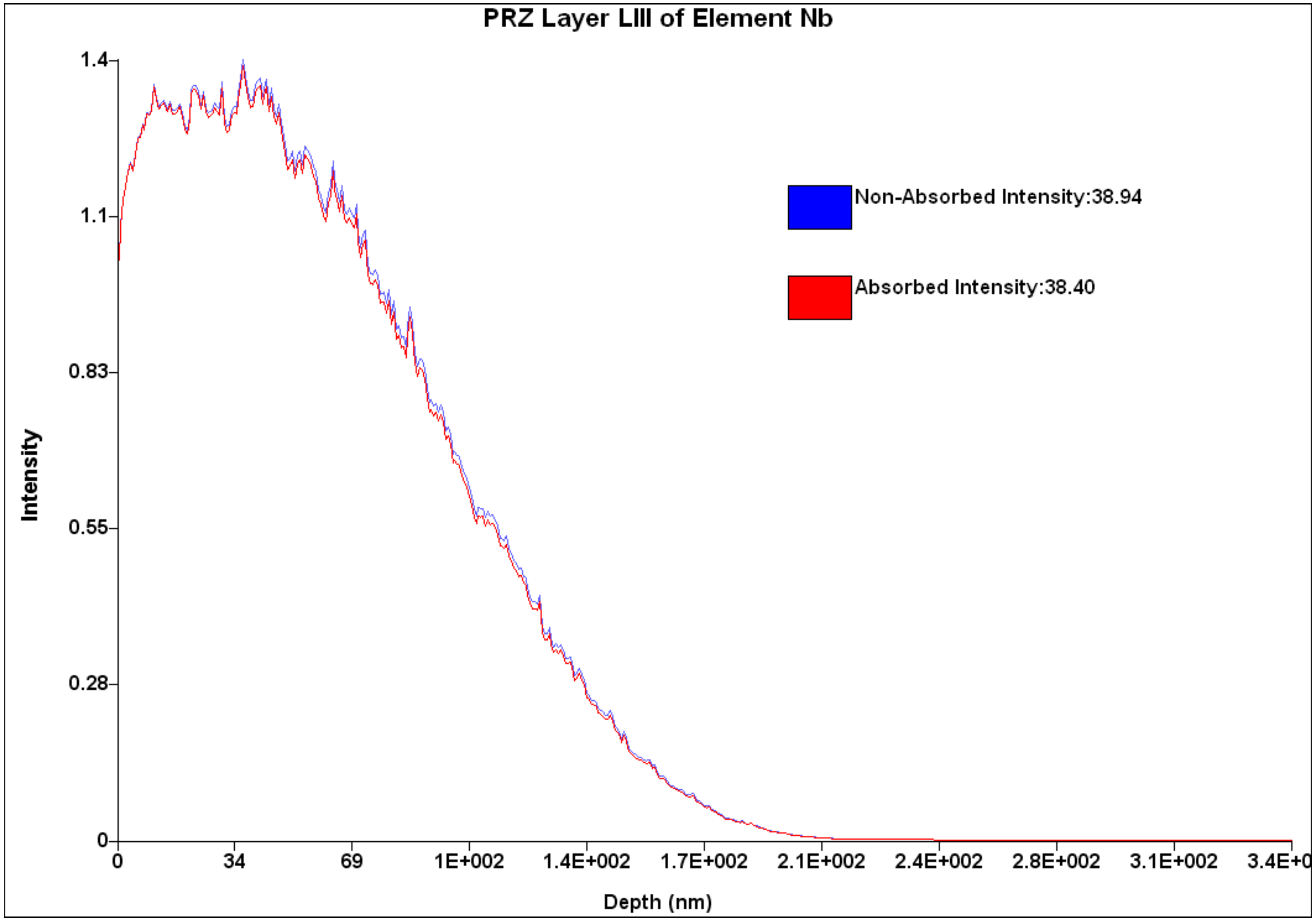

PRZ Layer LIII of Element Nb
Non-Absorbed Intensity:4566.37
Absorbed Intensity:1895.53
Intensity
Depth (nm)
3.4
2.7
2
1.4
0.68
0
1.9E+003
3.9E+003
5.8E+003
7.7E+003
9.6E+003
1.2E+004
1.4E+004
1.5E+004
1.7E+004
Not detected by detector due to absorption

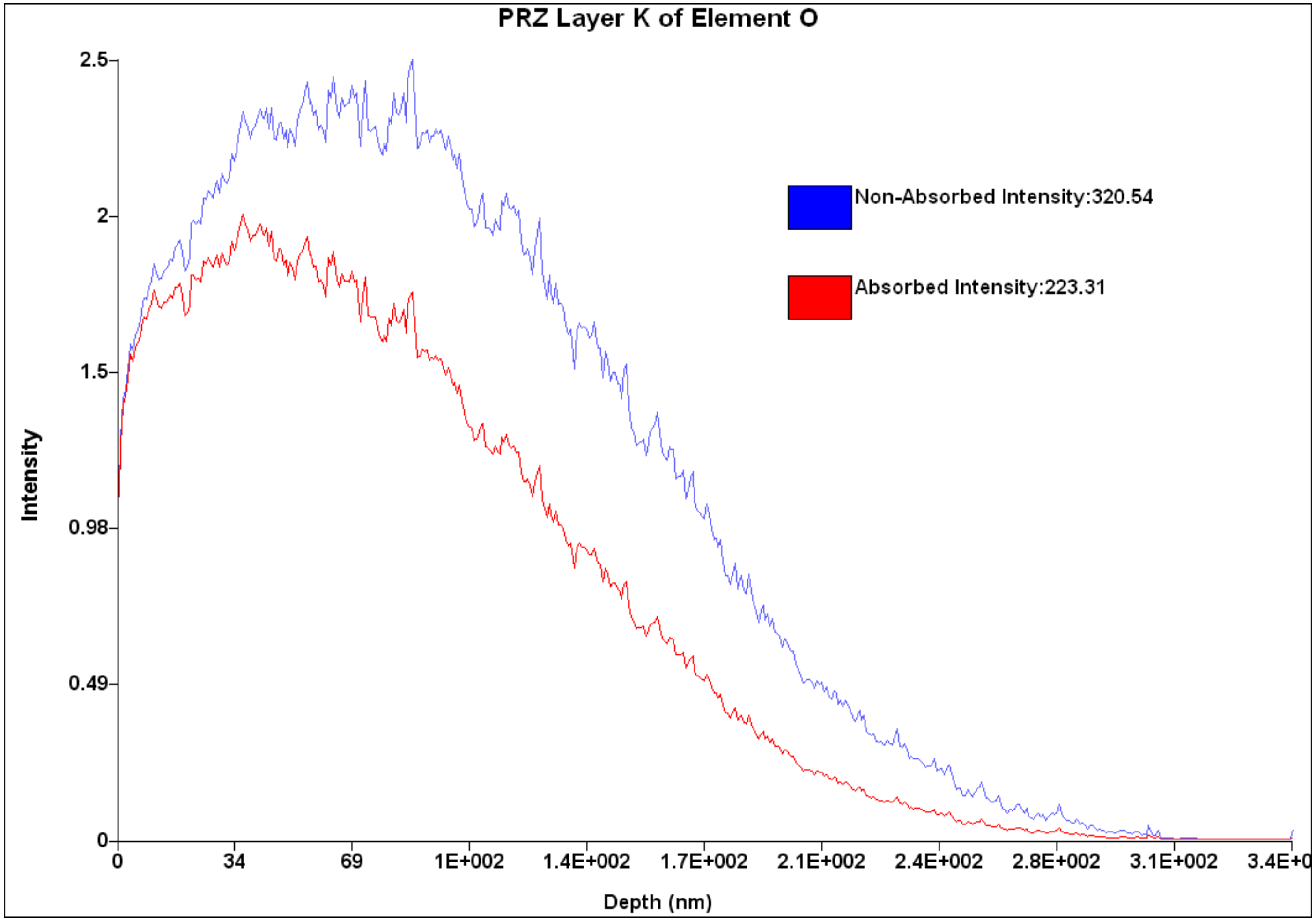

NaKNb2O6
-10800.0 nm
-5400.0 nm
0.0 nm
5400.0 nm
10800.0 nm
Intensity
PRZ Layer LIII of Element Nb
Depth (nm)
Absorbed Intensity:1895.53
Non-Absorbed Intensity:4566.37

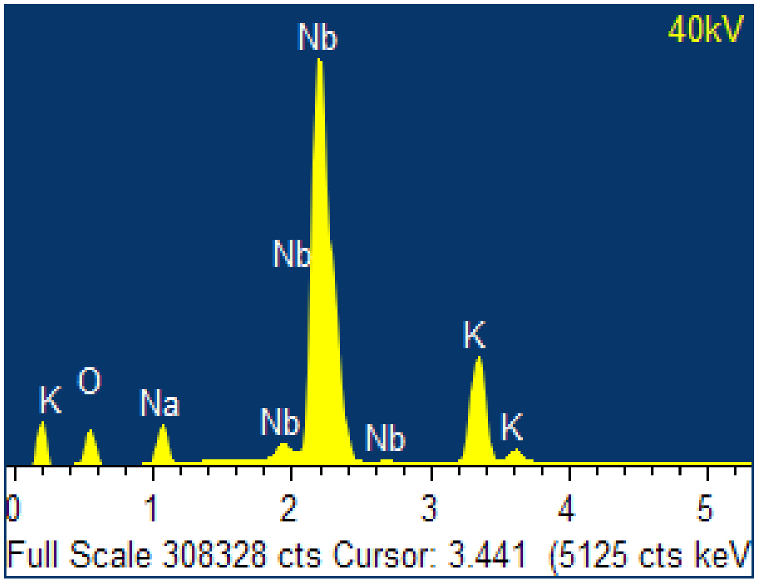
40 kV

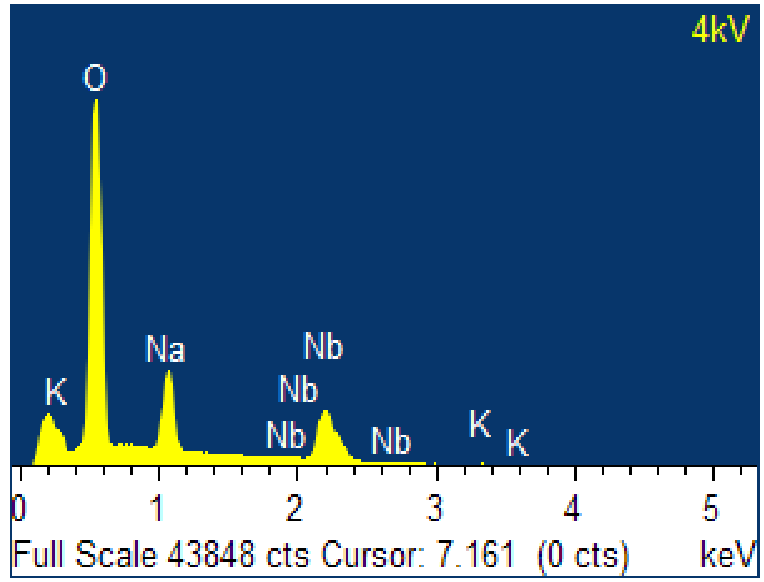

PRZ Layer K of Element O
Non-Absorbed Intensity:6097.91
Absorbed Intensity:100.92
Intensity
Depth (nm)
3.9
3.1
2.3
1.6
0.78
0
1.9E+003
3.9E+003
5.8E+003
7.7E+003
9.6E+003
1.2E+004
1.4E+004
1.5E+004
1.7E+004
Not detected by detector due to absorption

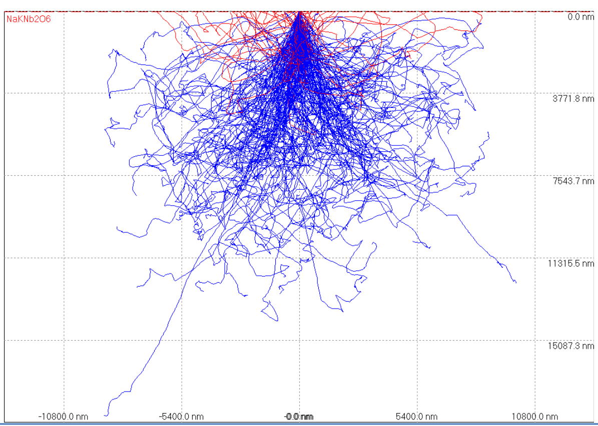

NaKNb2O6
0.0 nm
76.8 nm
153.7 nm
230.5 nm
307.3 nm
-220.0 nm
-110.0 nm
0.0 nm
110.0 nm
220.0 nm
4 kV

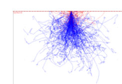

PRZ Layer LIII of Element Nb
Non-Absorbed Intensity:38.94
Absorbed Intensity:38.40
Intensity
1.4
1.1
0.83
0.55
0.28
0
0
34
69
1E+002
1.4E+002
1.7E+002
2.1E+002
2.4E+002
2.8E+002
3.1E+002
3.4E+0
Depth (nm)


PRZ Layer K of Element O
Non-Absorbed Intensity:320.54
Absorbed Intensity:223.31
Intensity
2.5
2
1.5
0.98
0.49
0
0
34
69
1E+002
1.4E+002
1.7E+002
2.1E+002
2.4E+002
2.8E+002
3.1E+002
3.4E+0
Depth (nm)


40kV
Nb
Nb
K
K
O
Na
Nb
Nb
K
0
1
2
3
4
5
Full Scale 308328 cts Cursor: 3.441 (5125 cts keV


4kV
O
Na
Nb
Nb
K
Nb
Nb
K
K
0
1
2
3
4
5
Full Scale 43848 cts Cursor: 7.161 (0 cts) keV

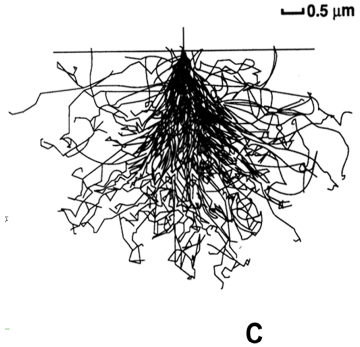

NaKNb2O6
0.0 nm
3771.8 nm
7543.7 nm
11315.5 nm
15087.3 nm
-10800.0 nm
-5400.0 nm
0.0 nm
5400.0 nm
10800.0 nm

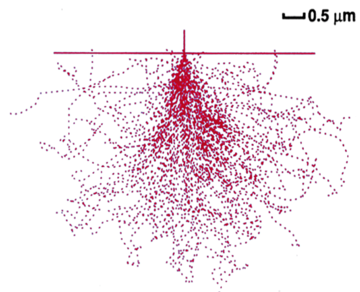


Quantification

When the going gets tough…..

Correction matrix

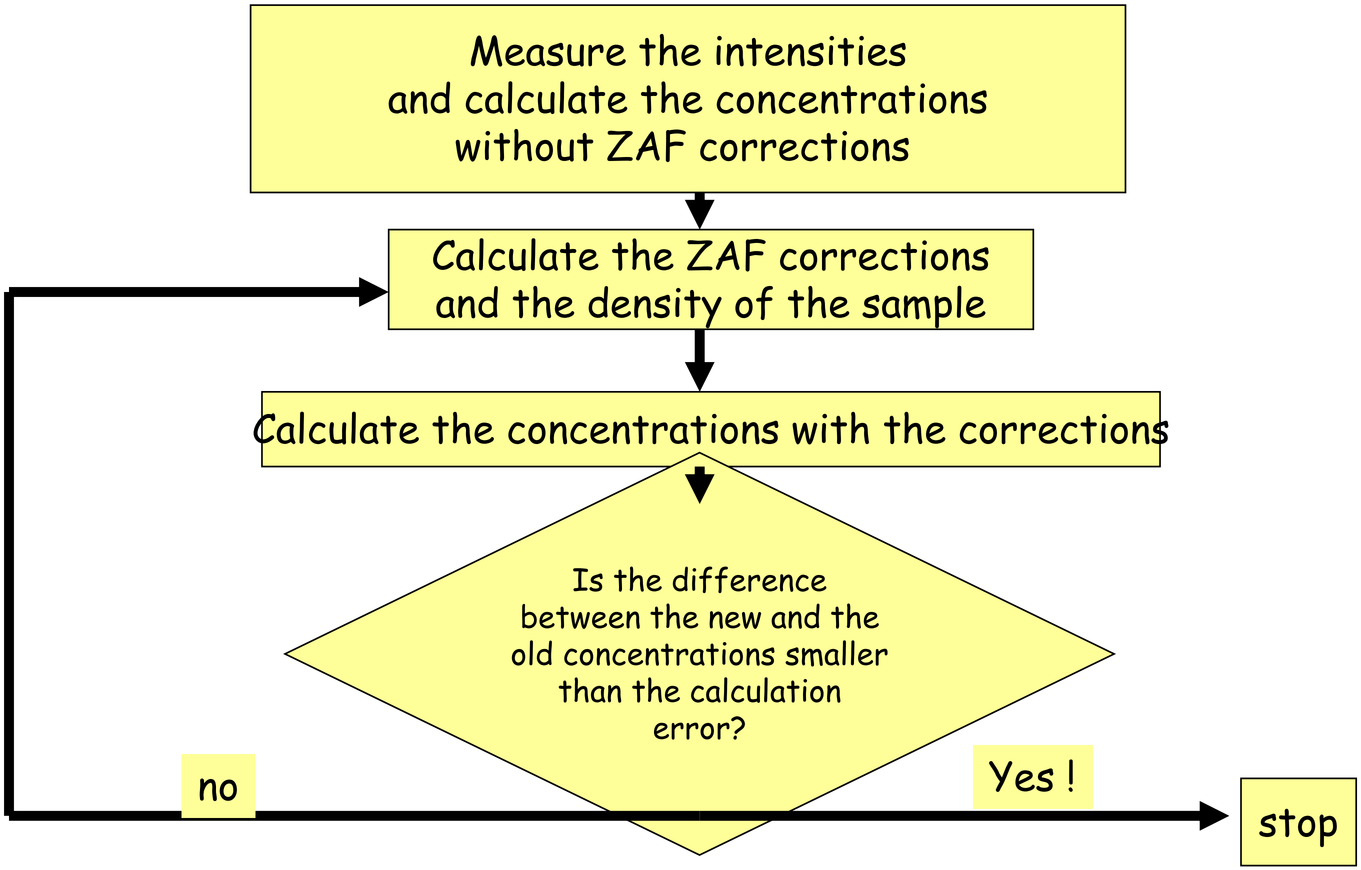




$$[Z \times A \times F]\frac{c_i}{c_i^{std}} = \frac{I_i}{I_i^{std}} = k_i$$

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

Flow chart of quantification



Correction methods:

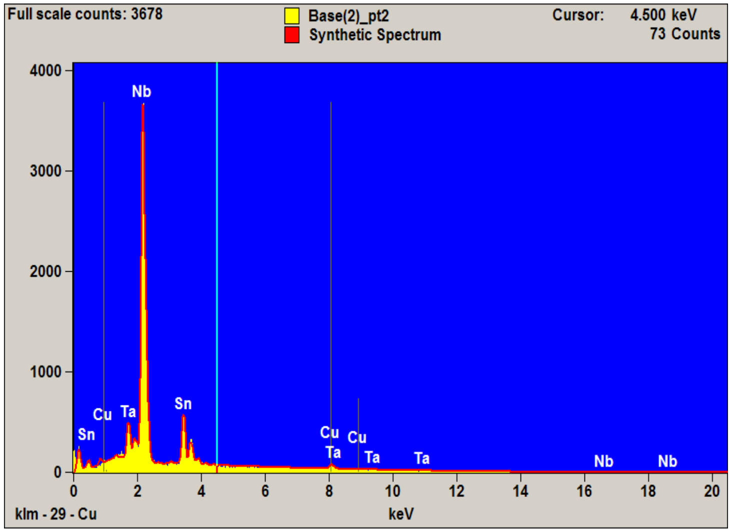
- ZAF (purely theoretical)
- PROZA Phi-Rho-Z
- PaP (Pouchou and Pichoir)
- XPP (extended Puchou/Pichoir)

- with standards (same HT, current, detector settings)
- Standardless: theoretical calculation of I_{std}
- Standardless optimized: « hidden » standards, user defined peak profiles

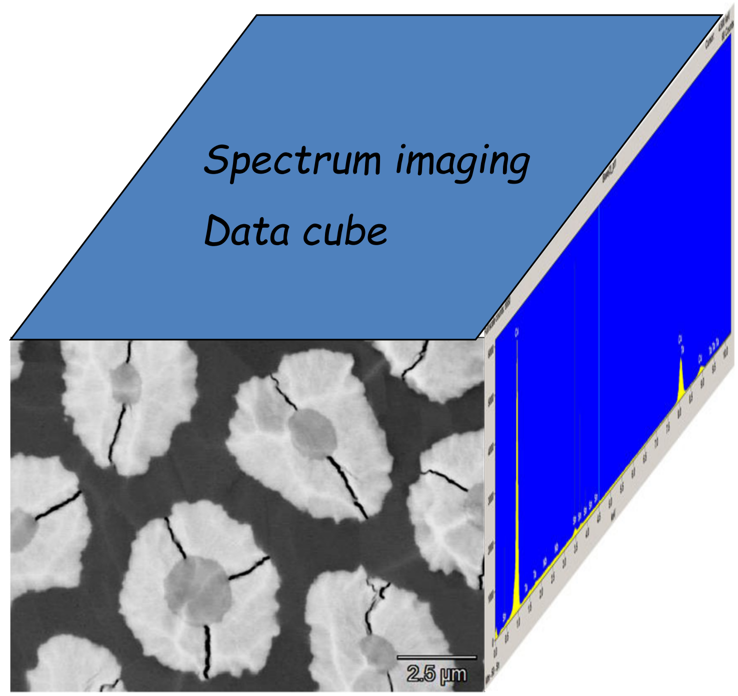
Quantitative EDX in SEM

- Acquisition under best conditions
  - Flat surface without contamination, horizontal orientation of the surface (no Au coating, use C instead)
  - Sample must be homogenous at the place of analysis (interaction volume !!)
  - High count rate (but dead time below 30%)
  - Overvoltage U=Eo/Ec >1.5-2
- For acquisition times of 100sec. :
detection of ~0.5at% possible for almost all elements

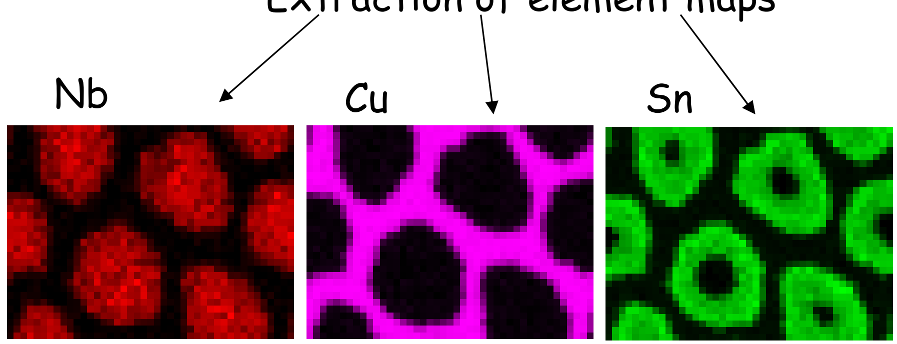
- Standardless quantification
- possible with high accuracy (intensities of references under the given conditions can be calculated for a great range of elements), test with samples of known composition, light elements (like O) are critical…
- Spatial resolution depends strongly on HT and the density of the sample



Synthesized spectrum



Extraction of element maps

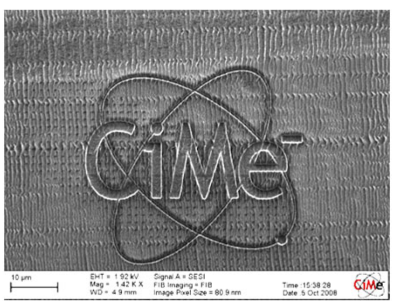


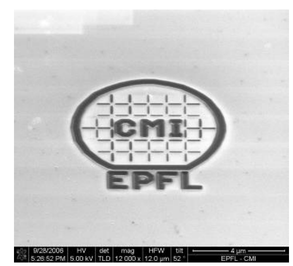
Element “Mapping”

FIB @ EPFL

@CIME: since summer 2008
ZEISS NVision 40

@CMI: since 2004
FEI Nova nanolab 600
clean room installation

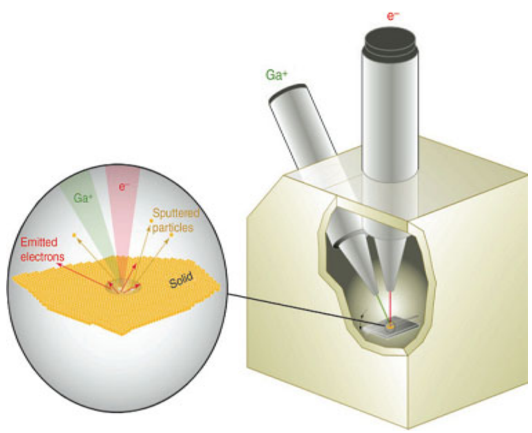




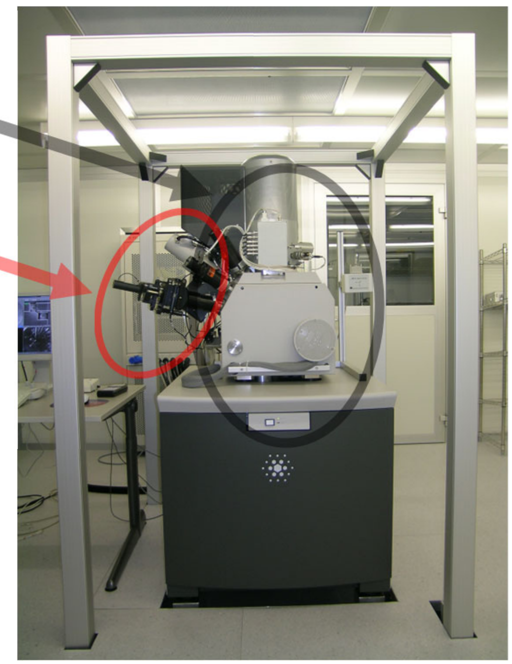
A modern FIB (Focused Ion Beam) system in a research lab (« lab » systems)

a complete state of the art (high -performance) SEM equipped with

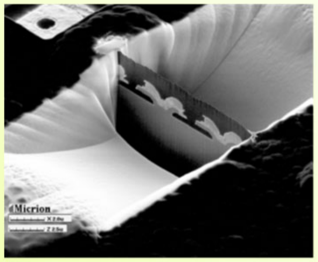
a) focused ion column
b) Gas injector system
c) micromanipulators

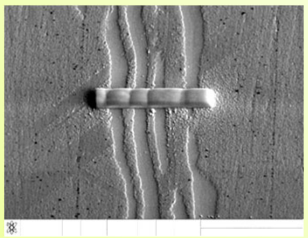


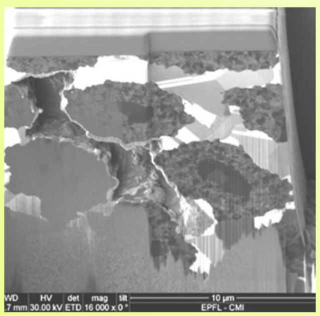
Dual beam ®, crossbeam ®

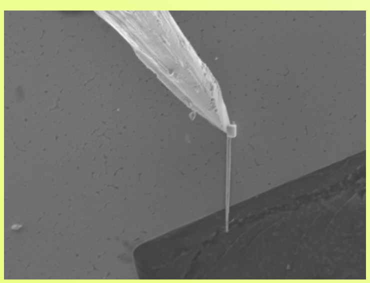


Dual Beam Nova 600 Nanolab
from FEI Company









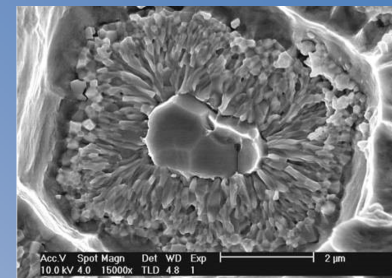
Ion Beam & Electron Beam

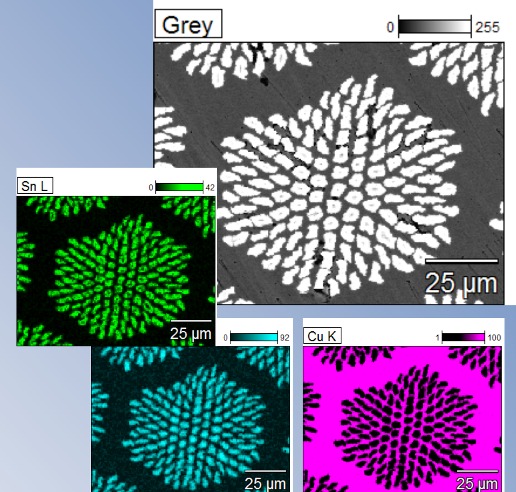
SEM: Imaging and Analysis

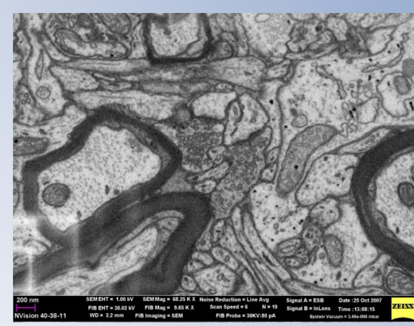
- High resolution (1-2nm) SEM
- SE, general imaging, topography contrast
- BSE, chemical (mass density contrast) contrast
- EDX microanalysis (point analysis and element mapping)
- Low voltage SE and BSE imaging (small interaction volume=high resolution), compatible with "non"-conducting and biological specimens

FIB: Nano-machining

- Machining (sputtering)
- chemically assisted deposition and etching (gas injector system)
- Ion beam induced imaging (channeling contrast), SE and SI
- Micromanipulation (multiple micromanipulators) of small objects (<10nm precision)
- ***Nano-scale "laboratory"***







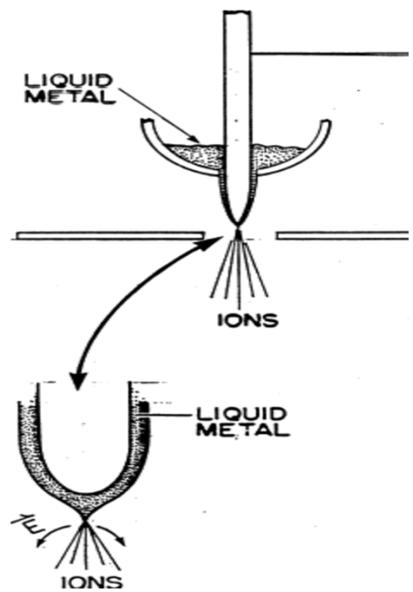
Focused Ion Beam

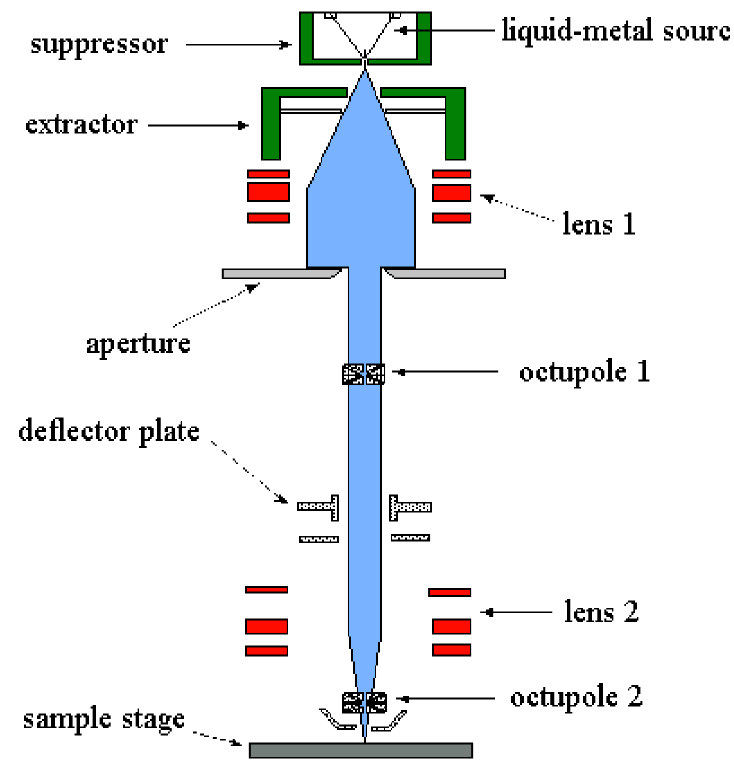
•Mainly developed in 1970's and 80's (Escovitz, Levi-Setti, Orloff, Swanson…)

•Ion column structure similar to that of SEM

•Source: Liquid Metal Ion Source (LMIS). Ex: Ga, Au, Be, Si, Pd, B, P, As, Ni, Sb, alloys …

•Principle: A strong electromagnetic field causes the emission of positively charged ions





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

SIM = Scanning Ion Microscope

The FIB history

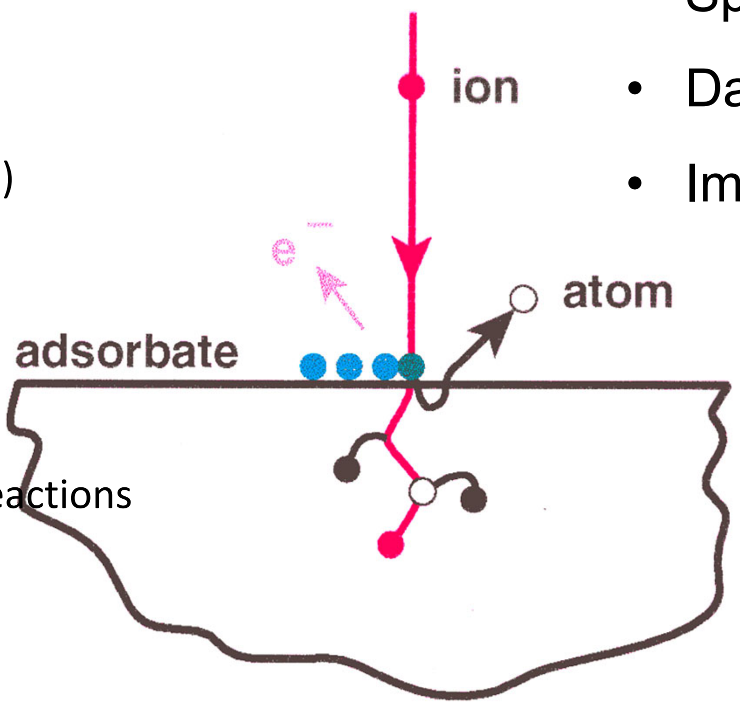
- Developed in early 70's for semi conductors (mask repair, failure analysis, chip modification, Escovitz, Levi-Setti, Orloff, Swanson…)
- Soon recognised as a "super-microscope", escaped the semi-conductor world to enter electron microscopy labs (2000, first "small" dual beam released)
- Different specific needs (quality control <-> high end research)

- **New developments**
  - low kV capability, 3D nano-tomography, lift-out techniques
  - 2000-2007: 3 generations of machines (DB235 -> Strata400, Nova600->HELIOS)
  - strong competition between FEI and ZEISS (taking advantage of their excellent SEM column)

Ion Solid interaction

- Secondary electron emission (SE-image) 2-3 SE per Ion !

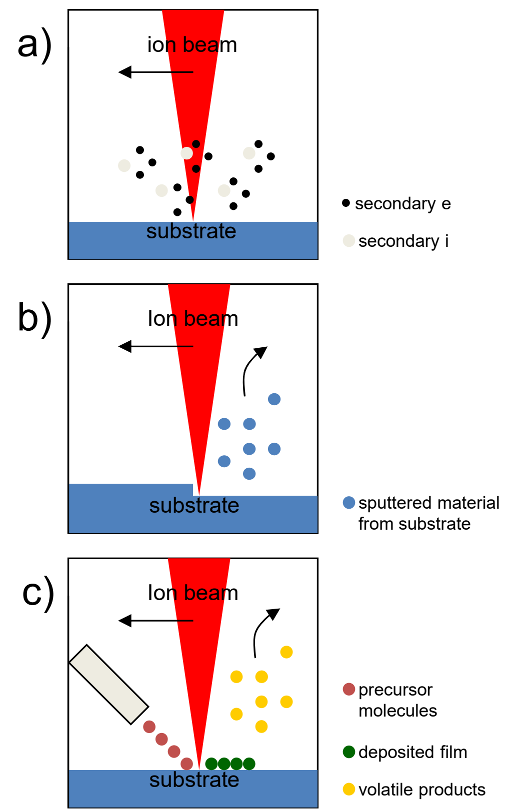
- Surface chemical reactions
  - deposition
  - enhanced etching



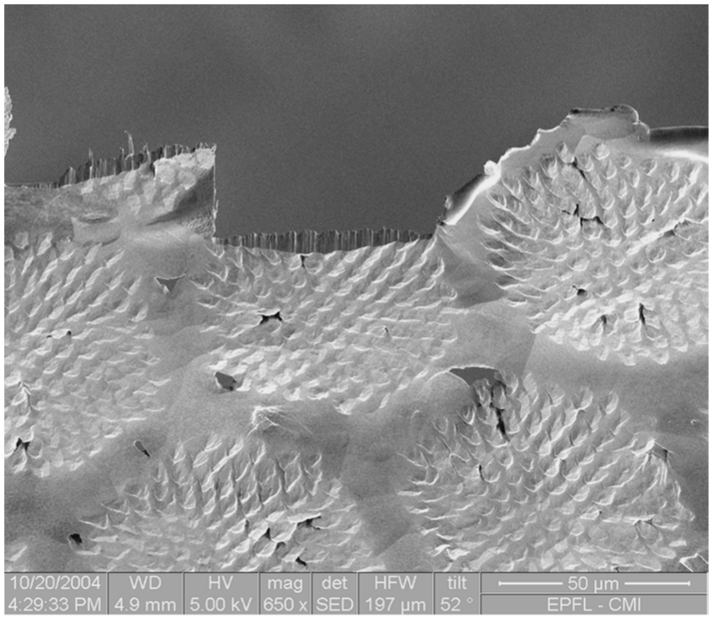
- Sputtering
- Damage
- Implantation

3 basic "operating modes"

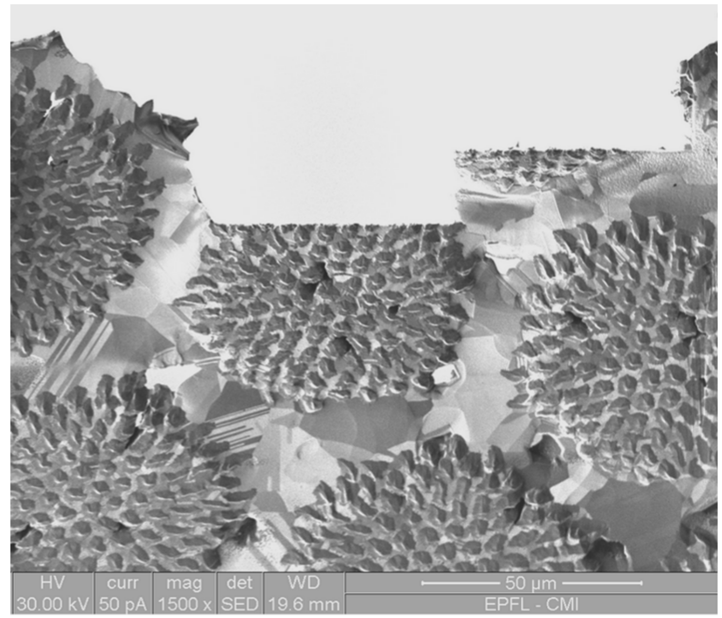
- Emission of secondary ions and electrons
  - FIB **imaging** ☛ a)
    low ion current
- Sputtering of substrate atoms
  - FIB **milling** ☛ b)
    high ion current
- Chemical interactions (gas assisted)
  - FIB **deposition**
  - Enhanced (preferrential) etching ☛ c)
- Other effects:
- Ion implantation
- Displacement of atoms in the solid
  - Induced damage
- Emission of phonons
  - Heating



SE image contrast
FIB «imaging» mode



e-beam 5kV

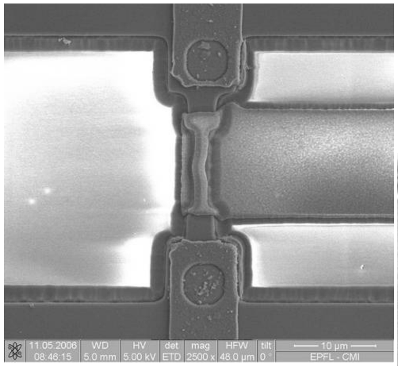


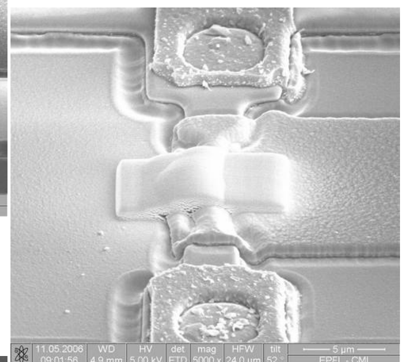
ion-beam 30kV 50pA

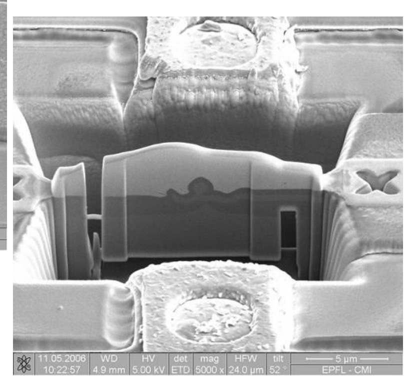
material (sputtering) contrast
orientational contrast

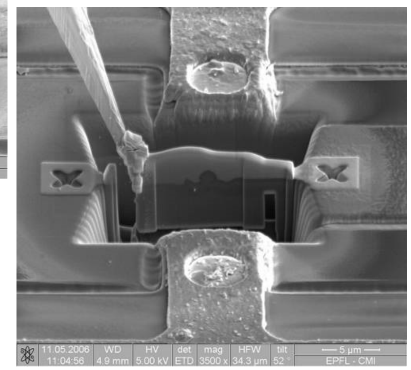
TEM lamella preparation of a Si nano-wire

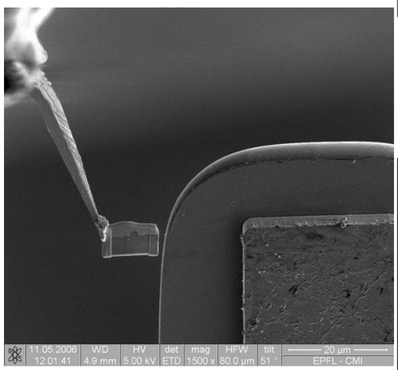
M. Pavius, V. Pott, CMI

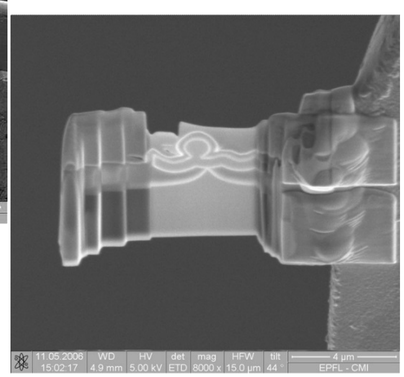






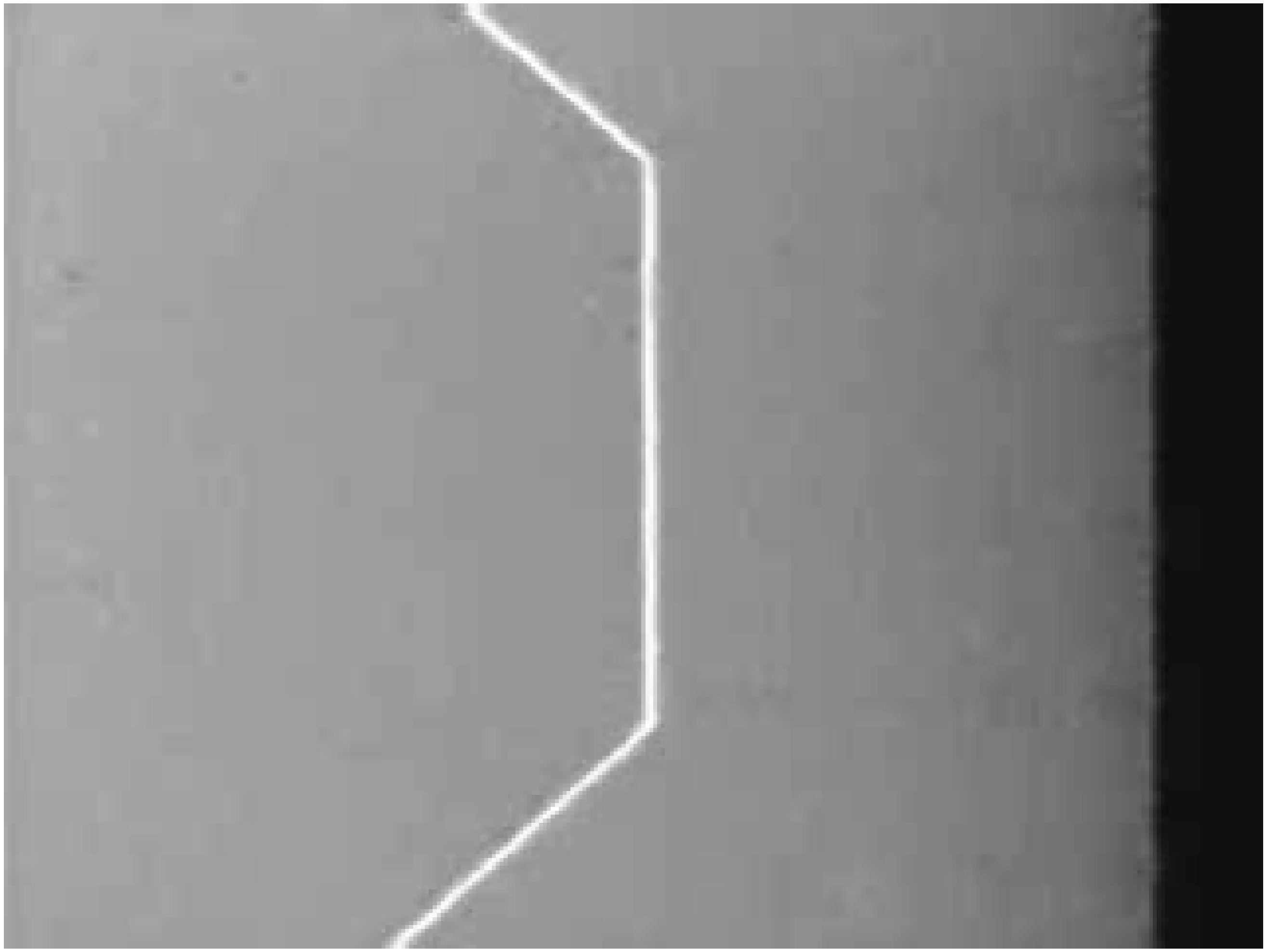




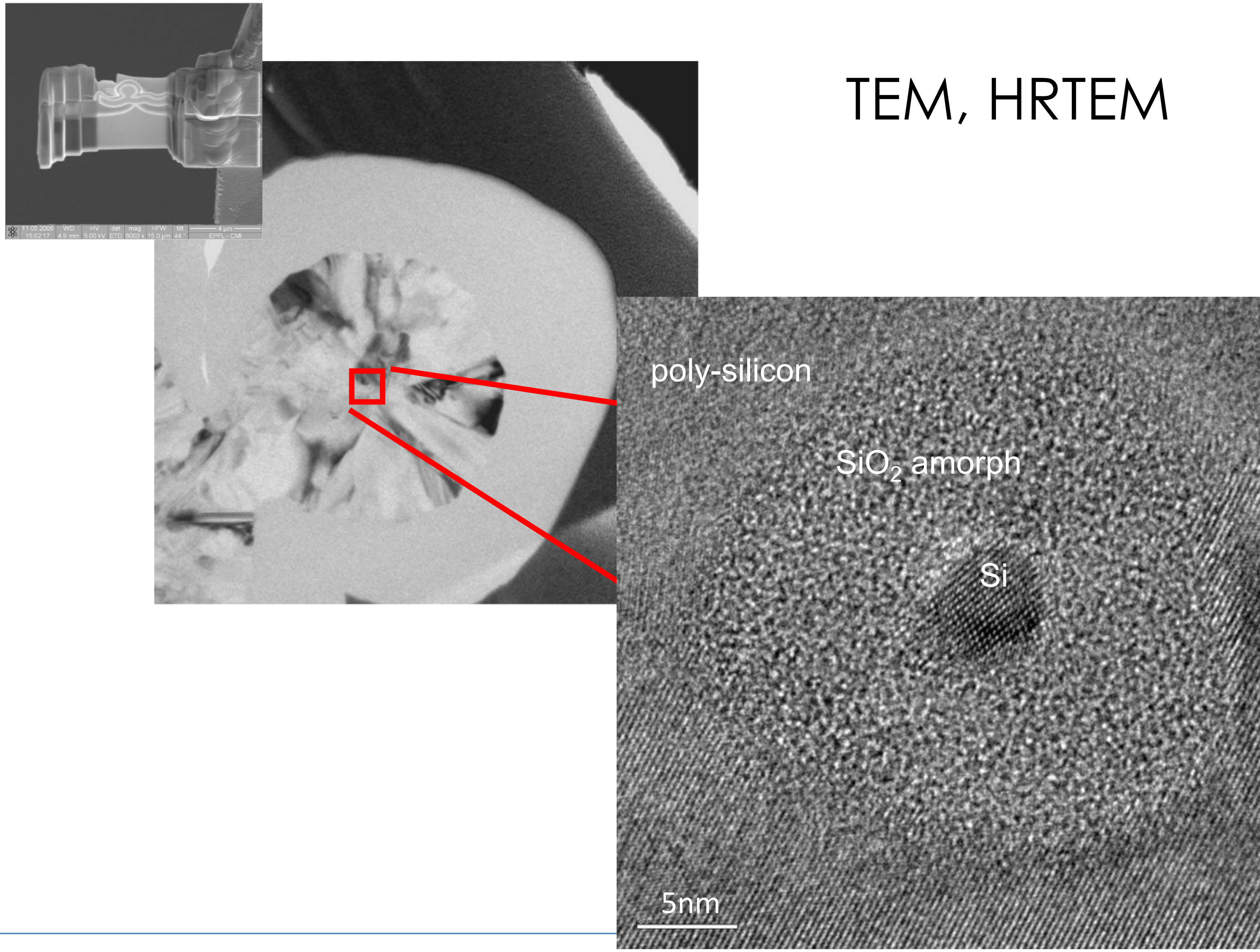


FIB «milling» and «deposition» mode

c) TEM preparation in-situ lift-out movie



TEM, HRTEM



TEM lamellae by FIB

Focused Ion Beam adds a new dimension to TEM specimen preparation

Take care of artifacts !!!

- Large (10x5um) flat areas with uniform thickness (50-80 nm)
- Preparation of heterogeneous samples with "difficult" material combinations becomes possible
- Precise selection of the lamella position possible (devices)

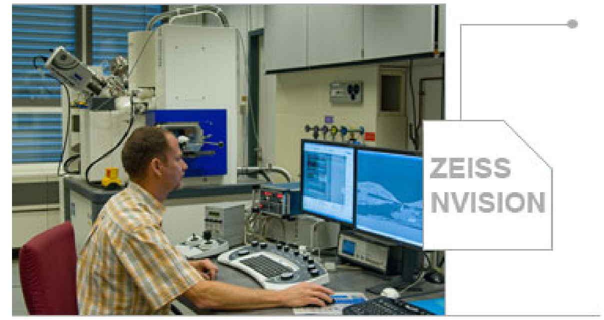
Since August 2008: Zeiss NVision 40

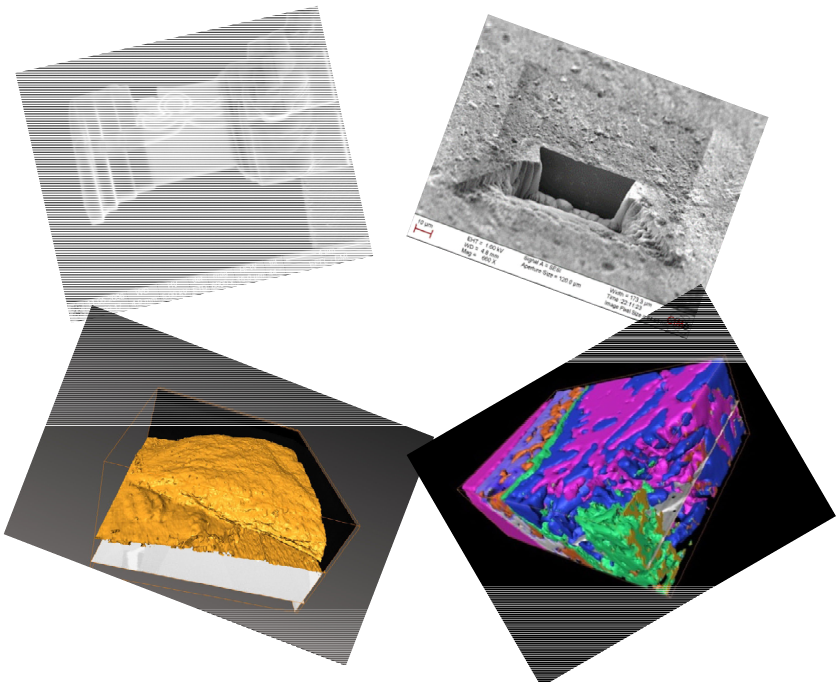
e-beam: ZEISS Gemini, 1-30kV, 1nm@30kV, 2.5nm@1 kV

EDS X-MAX (SDD) 80mm^2 detector, Kleindiek micromanipulator (TEM prep)

Since 2011: ATLAS-3D (scan generator & software)

2-3 Ga Sources / year (~5000 beam hours)

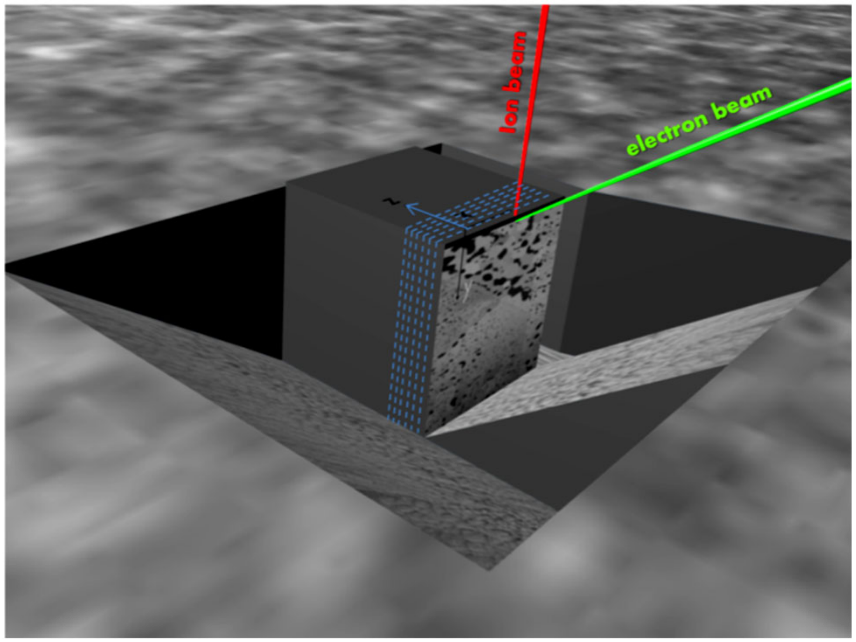




FIB Applications @ CIME

- ***Materials Science:***
  - *TEM Lamellae preparation*
  - *cross-sectioning, SE/BSE imaging, EDX*
  - *Micro& nano-patterning*
  - ***3D reconstruction***
  - ***3D EDX***
- ***Life Science:***
  - ***Serial Sectioning of cells and brain tissue: SUPER-STACKS***

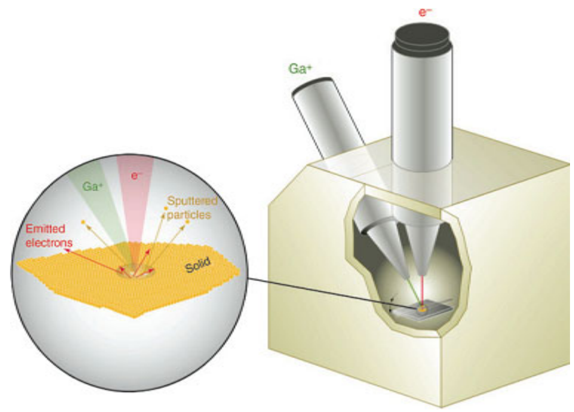
FIB/SEM volume reconstruction: FIB Nano-Tomography

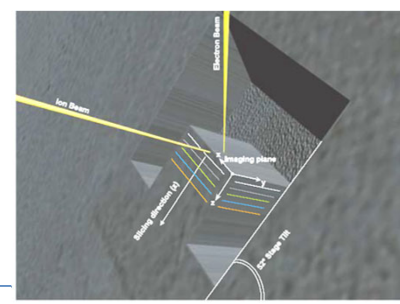


Three-dimensional analysis of porous $BaTiO_3$ ceramics using FIB nanotomography

L. HOLZER, F. INDUTNYI, PH. GASSER, B. MÜNCH & M. WEGMANN
EMPA, Swiss Federal Laboratories for Materials Testing and Research, Ueberlandstrasse 129, 8600 Dübendorf, Switzerland

Journal of Microscopy, Vol. 216, Pt 1 October 2004, pp. 84–95

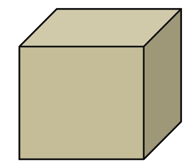




"direct" tomography: cutting and imaging

What you (detector) see is what you get !

3D FIB/SEM: volume reconstruction

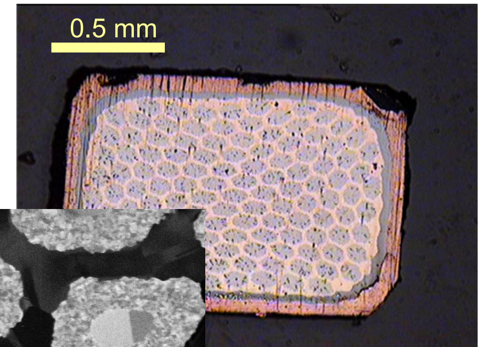


"Leitmotiv"
Isometric voxel size
x = y = z

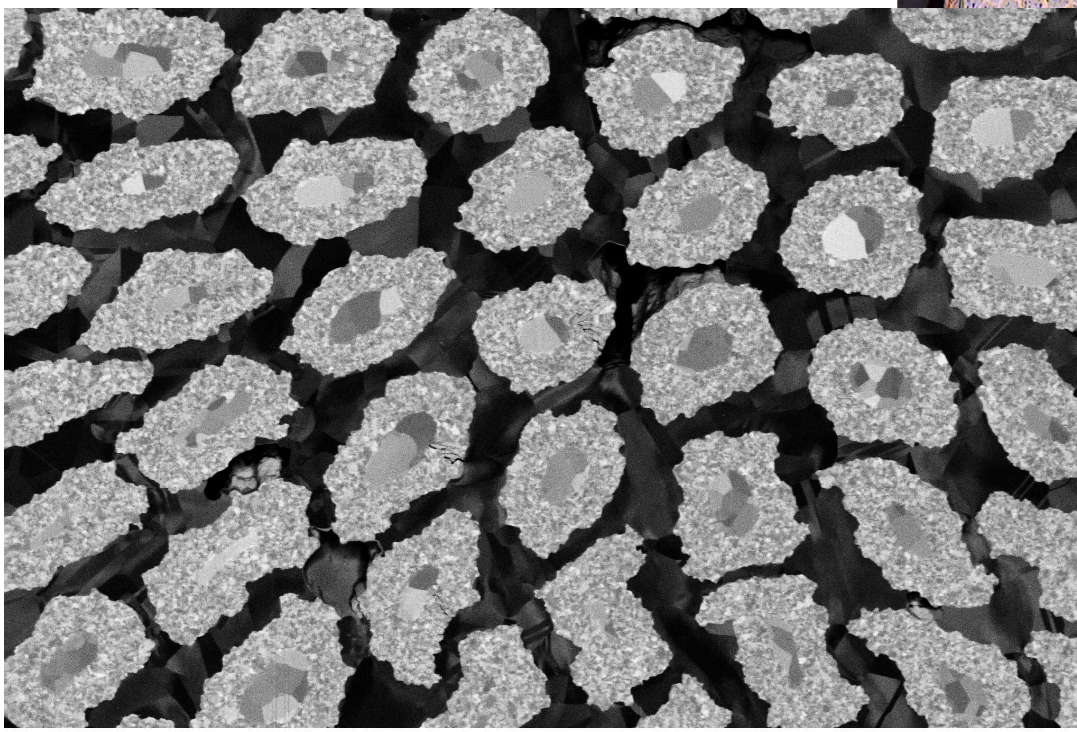
- **Isotropic resolution**
  - Slice thickness (z) = image pixel size (x,y)
  - *Z dimension ~ X or Y, typical: 10nm, possible 5nm (3nm)*
  - *Z- Resolution: low kV !!!*
- **High Throughput**
  - *Acquisition time "Image" ~1min / slice (~60 slices / hour)*

    -> high S/N ratio, beam current (1-1.5nA), detector efficiency
  - *Dwell times/pixel 5- 15µsec.*
  - *minimise overhead, no tilting, rotating, (drift correction)*

Nb3Sn multifilament Superconductors

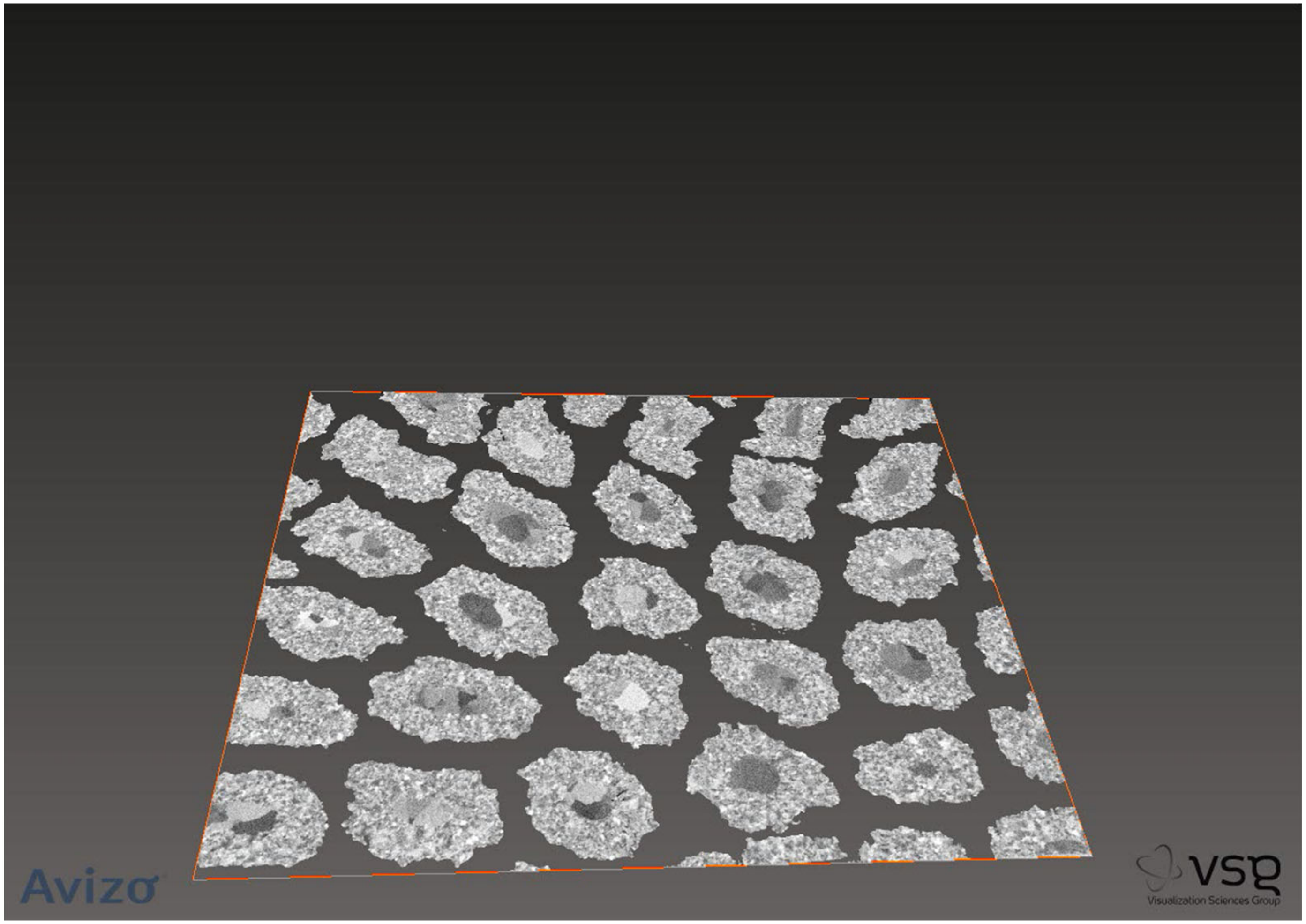


Materials & grain contrast

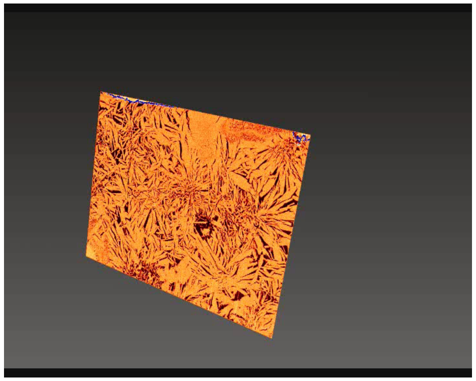


Nb$_3$Sn superconductor multifilament cable:
14’000 Nb$_3$Sn filaments (diameter ~5um) in bronze matrix
1.8kV EsB detector

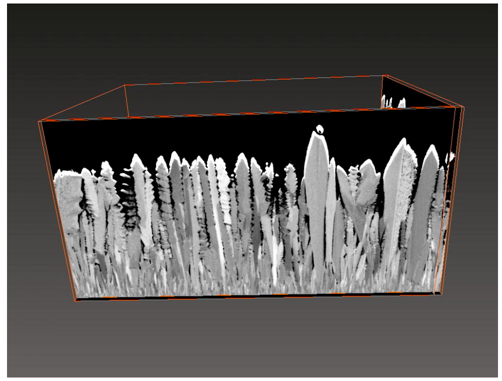
Nb_3Sn multifilament Superconductors
Materials & grain contrast



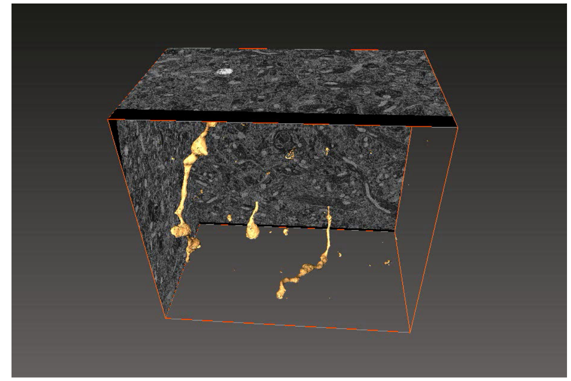
2048x1536x1700 (10x10x10nm voxel)



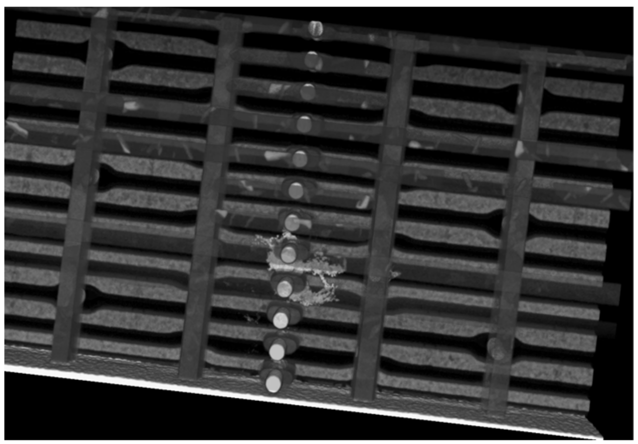
Cement, $(10nm)^3$ voxel



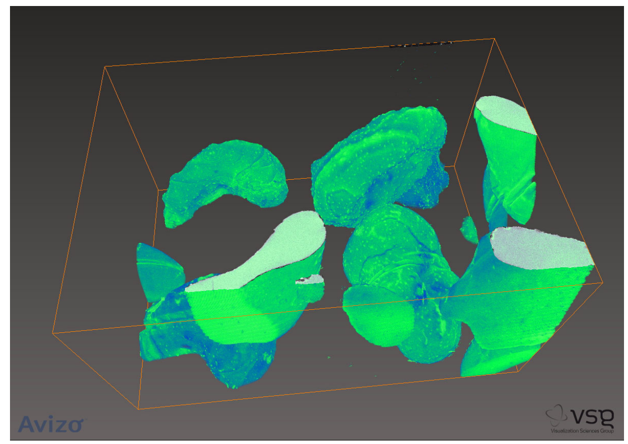
Solar cell: ZnO, $(10nm)^3$ voxel



Rat brain $(10nm)^3$ voxel



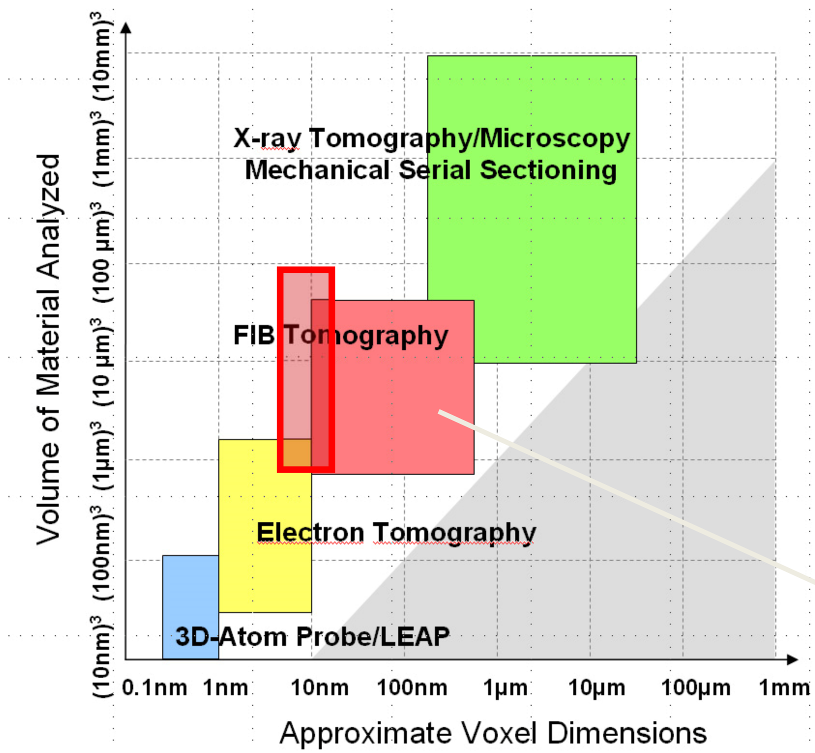
IC, $(10nm)^3$ voxel



Malaria parasite, $(10nm)^3$ voxel

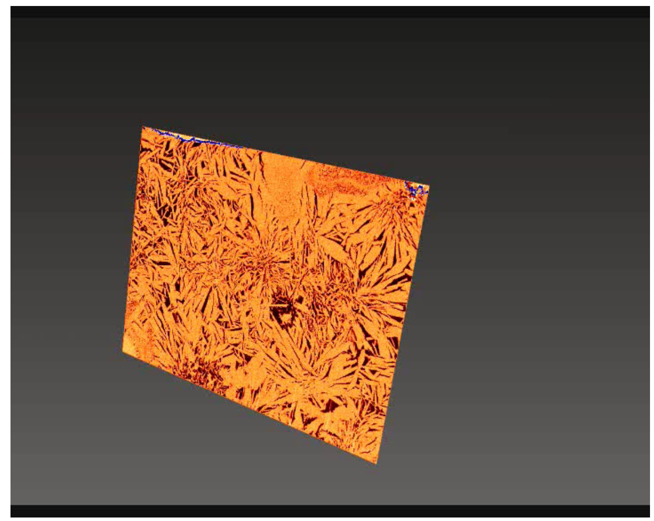
FIB/SEM Nano-Tomography, volume reconstruction
Typical voxel sizes

FIB-NT compared with other 3D-techniques



From: Uchic, Holzer and Inkson
Mat. Res. Bul.,

Ciment, A. Quennoz IMX-LMC
Materials contrast
1100 slices (20nm thickness)



New possibilities in 3D-microscopy: Combination with quantitative analytical SEM techniques: EDX, EBSD

conclusion

- FIB Nano-Tomography (FIB-NT) is “straight forward”
- low kV imaging in a SEM/FIB, the right selection of your detector, high resolution in z direction
- Applications in Materials Science and Life Science
  - easier segmentation with multiple detectors
- 3D EDX is possible

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Volume 39, Issue 4, April 2014, Pages 354-360
Marco Cantoni and Lorenz Holzer