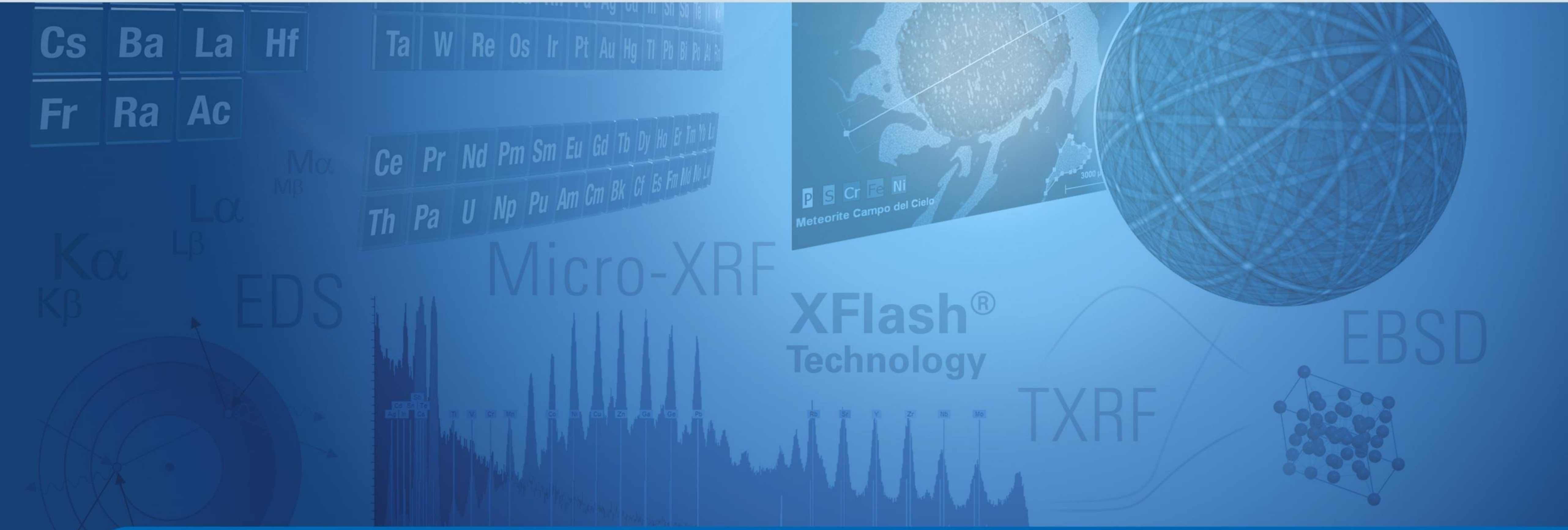
Advanced Application Examples using SEM-EDS

Bruker Nano Analytics, Berlin, Germany
Webinar, March, 2021



Presenters

Max Patzschke

Application Scientist EDS
Bruker Nano Analytics, Berlin, Germany

Dr. Ifat Kaplan-Ashiri

Associate Staff Scientist
Weizmann Institute of Science

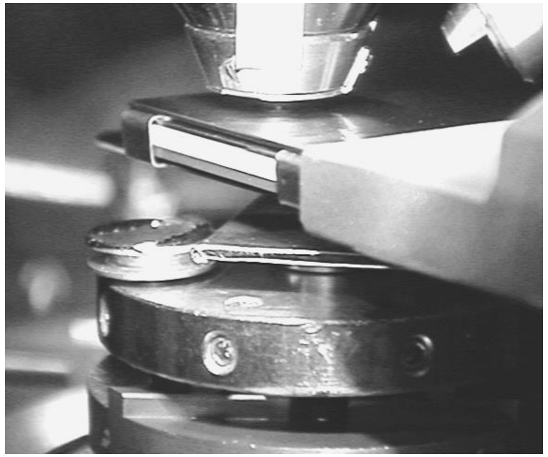
Bruker Nano Analytics
Product Line – EM Analyzers

- EDS
- WDS
- EBSD & TKD
- Micro-XRF on SEM
- EDS/FlatQUAD

Flexible combination of up to four analytical methods controlled by a single user interface.

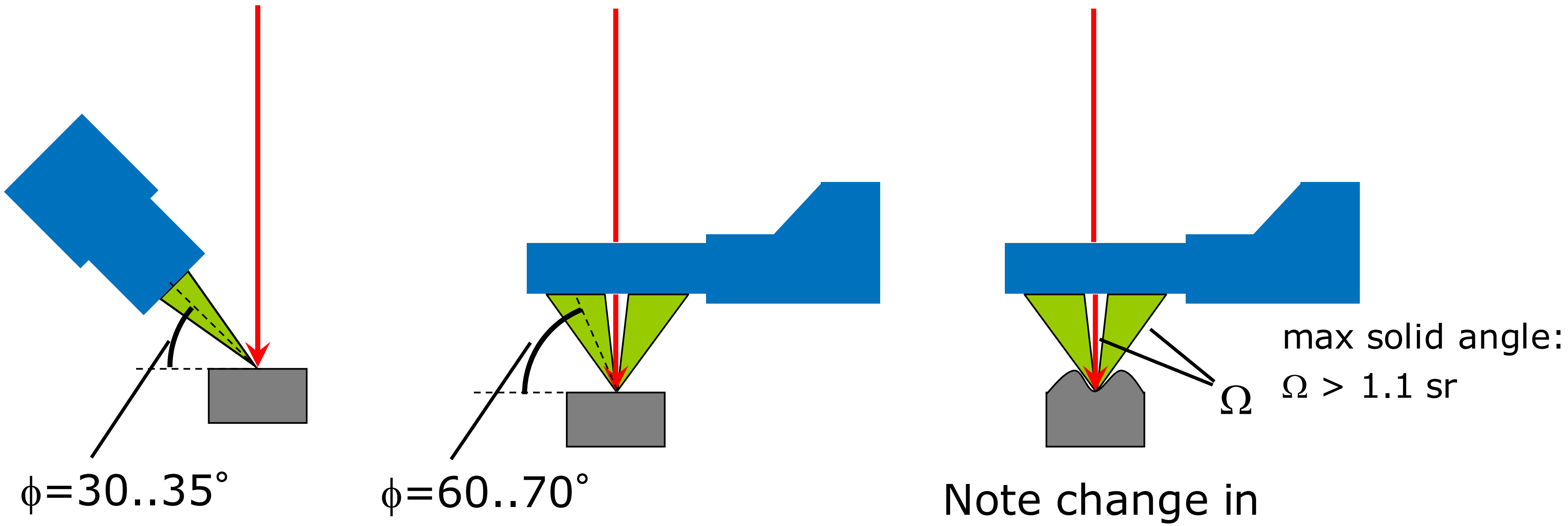
Facts of the XFlash® FlatQUAD detector

- Annular design, 4x15 mm^2 = 60 mm^2
- Placed between pole piece and sample (hole in the center for the primary beam)
- Energy resolution Mn $K\alpha \leq 129$ eV
- Combination of high count rate capability and high solid angle ($\Omega \sim 1.1$ sr)



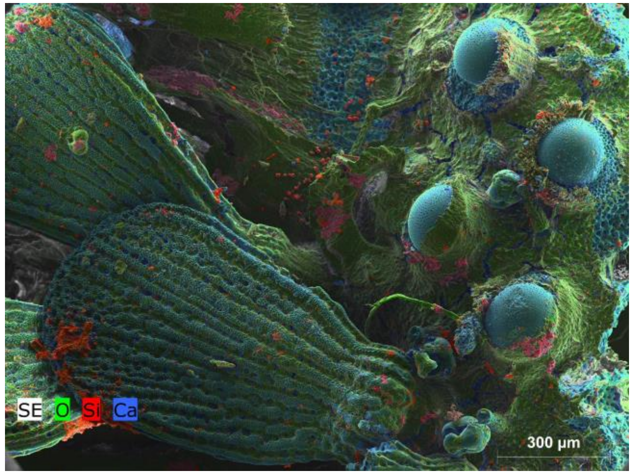
Advantage of the annular design of the XFlash® FlatQUAD

Take-off angle comparison: XFlash® FlatQUAD vs. conventional SDDs:

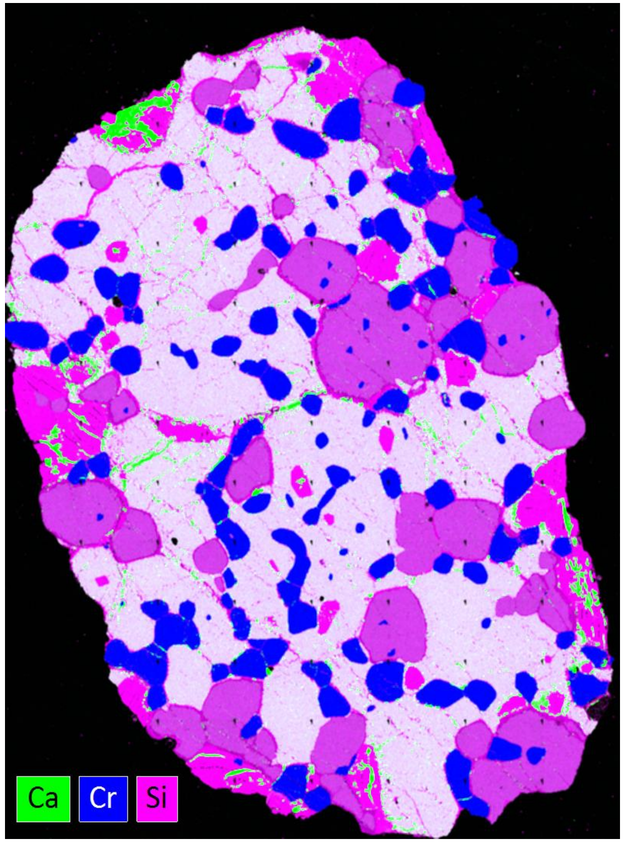


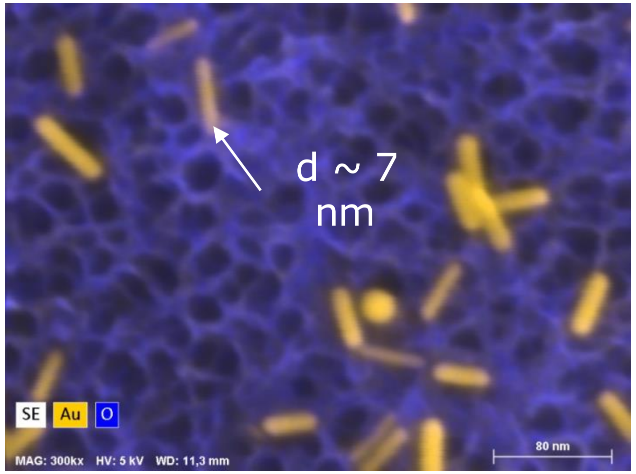
Note change in topography of sample

Advantage of the XFlash® FlatQUAD



- Life Science





- Nanomaterial
- High speed mapping of entire thin section

SEM-EDS Analysis – Materials and Life Science Applications

Ifat Kaplan-Ashiri

Electron Microscopy Unit

Weizmann Institute of Science

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Bruker Webinar - March 11th, 2021

Outline:

1- Introduction to SEM and EDS

2- EDS paramters/considerations

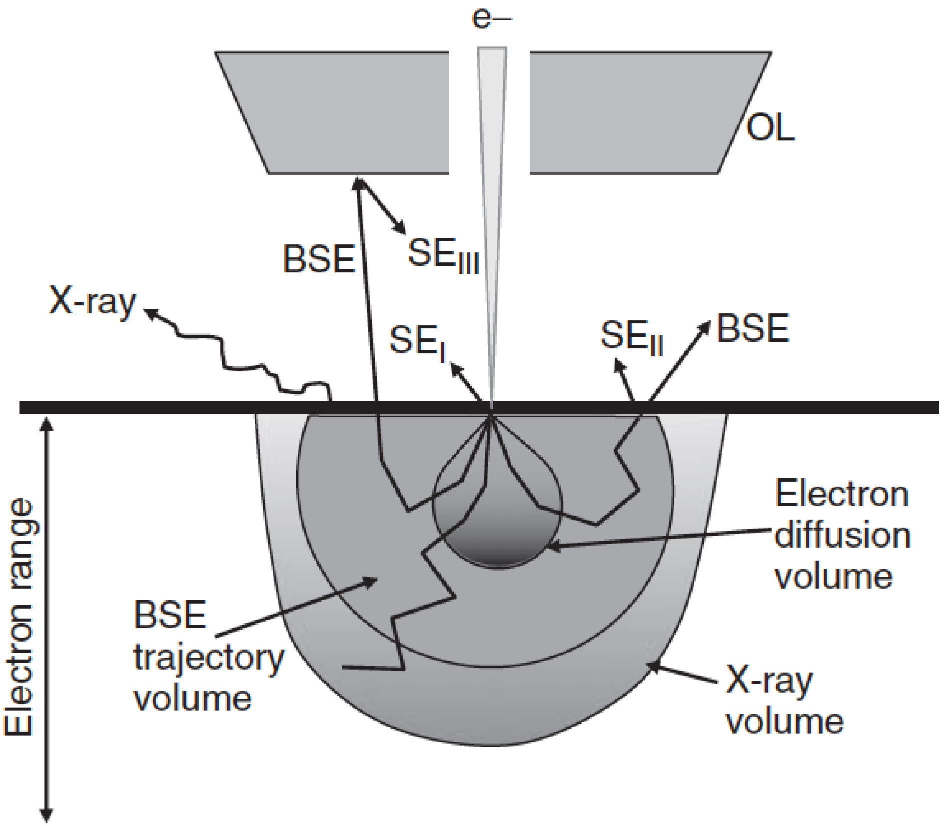
3- Applications:

Nanomaterials

Cryo EDS in life science

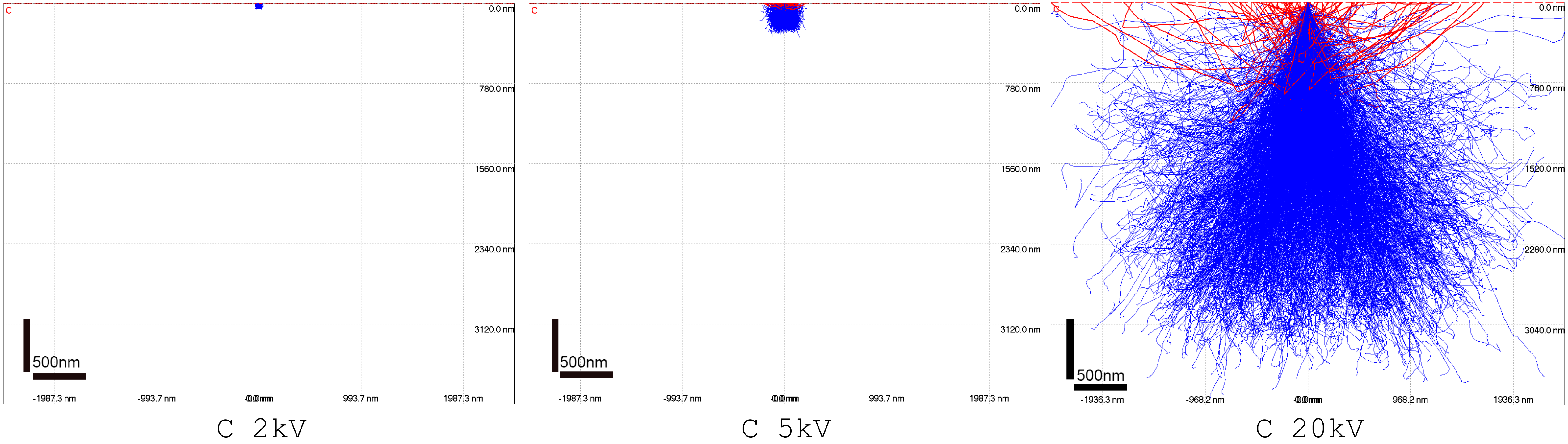
5- Summary

Scanning Electron Microscopy



Low Voltage Electron Microscopy: Principles and Applications, D.C.Bell and N. Erdman

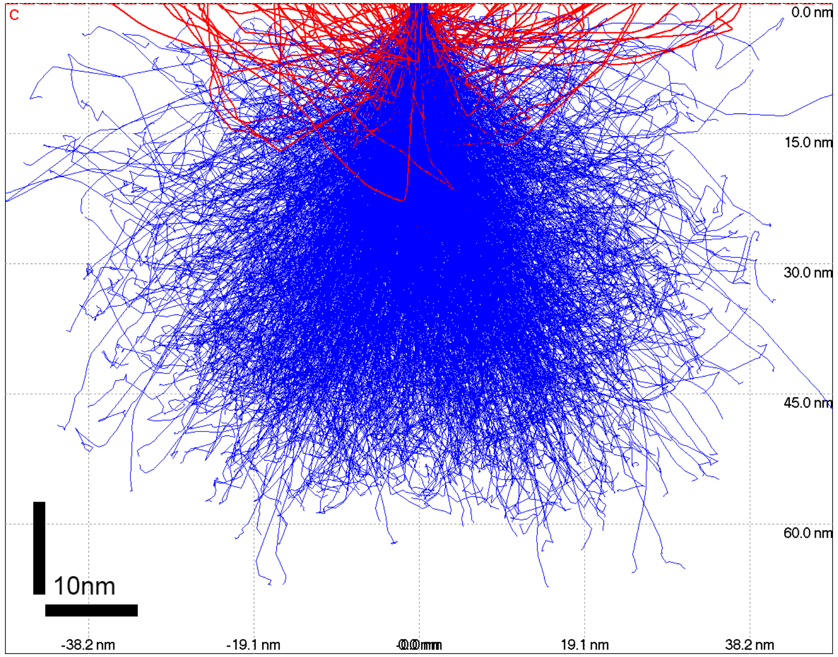
The Volume of Interaction



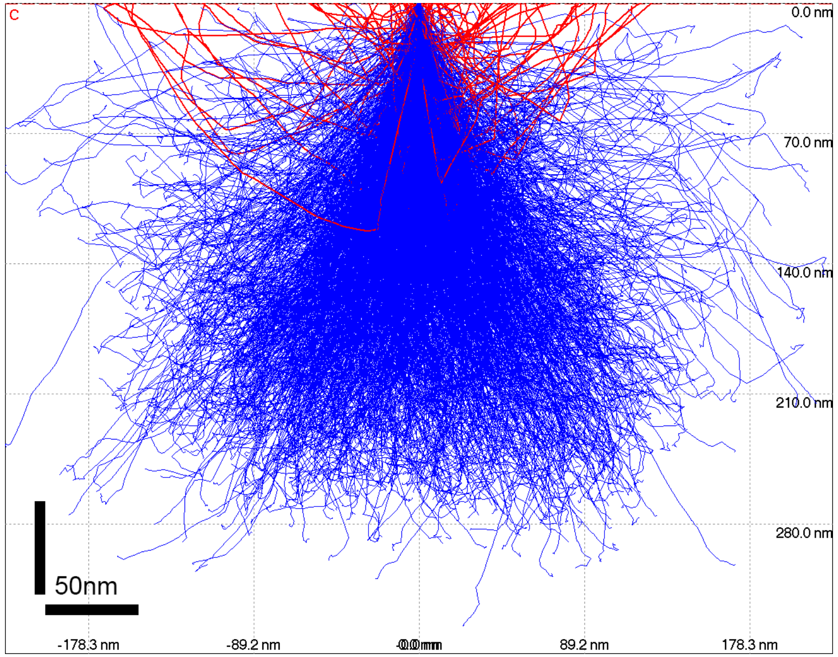
Monte Carlo simulations of electron trajectories

CASINO (monte CArlo SImulation of electroN trajectory in sOlids) Version 2.24 simulator (Copyright © 2001: D. Drouin, A. Réal Couture, R. Gauvin,P. Hovongton, P. Horny, and H. Demers)

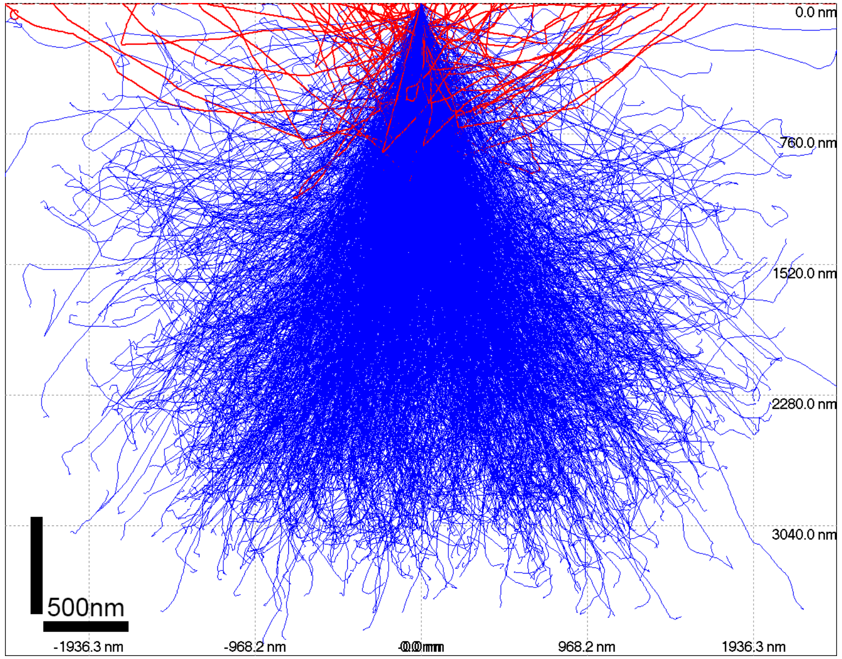
The Volume of Interaction



C 2kV

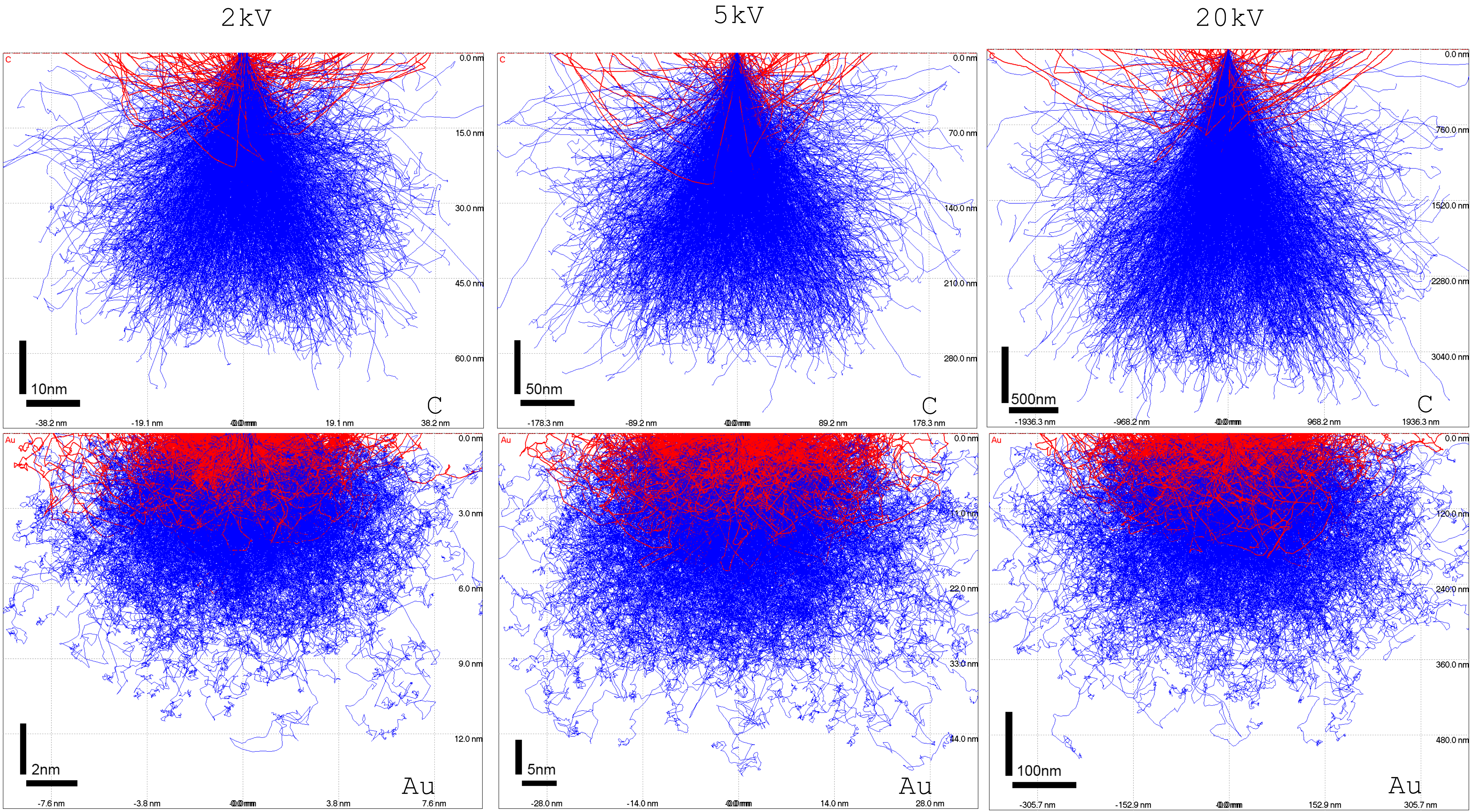


C 5kV

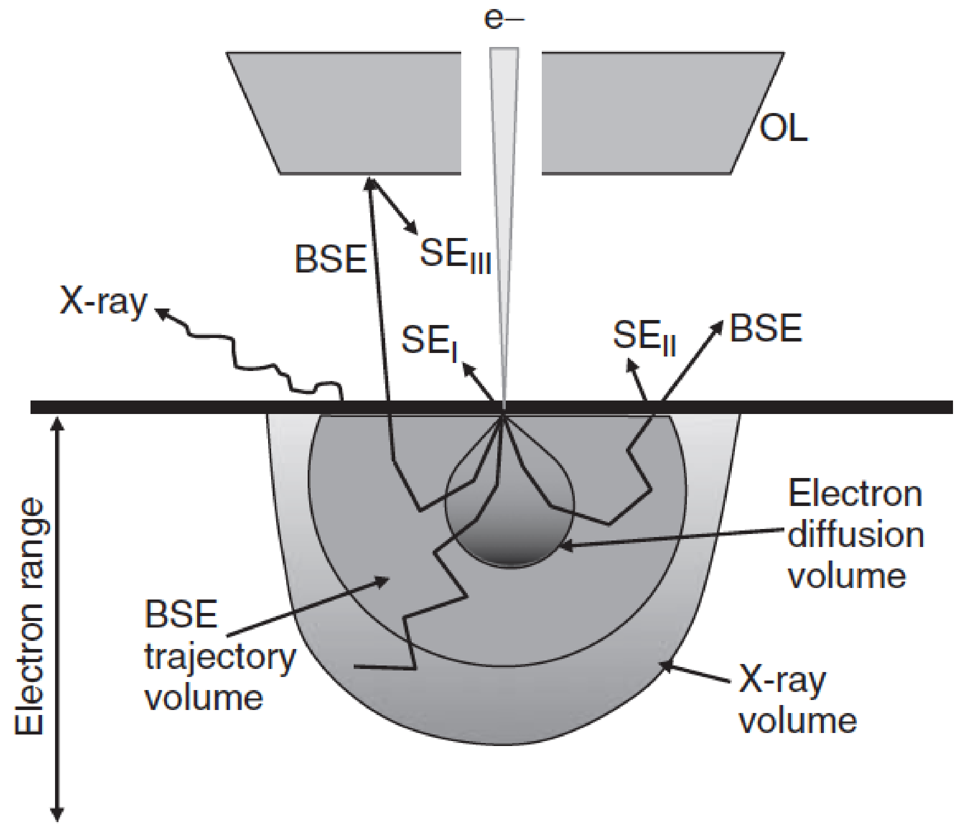


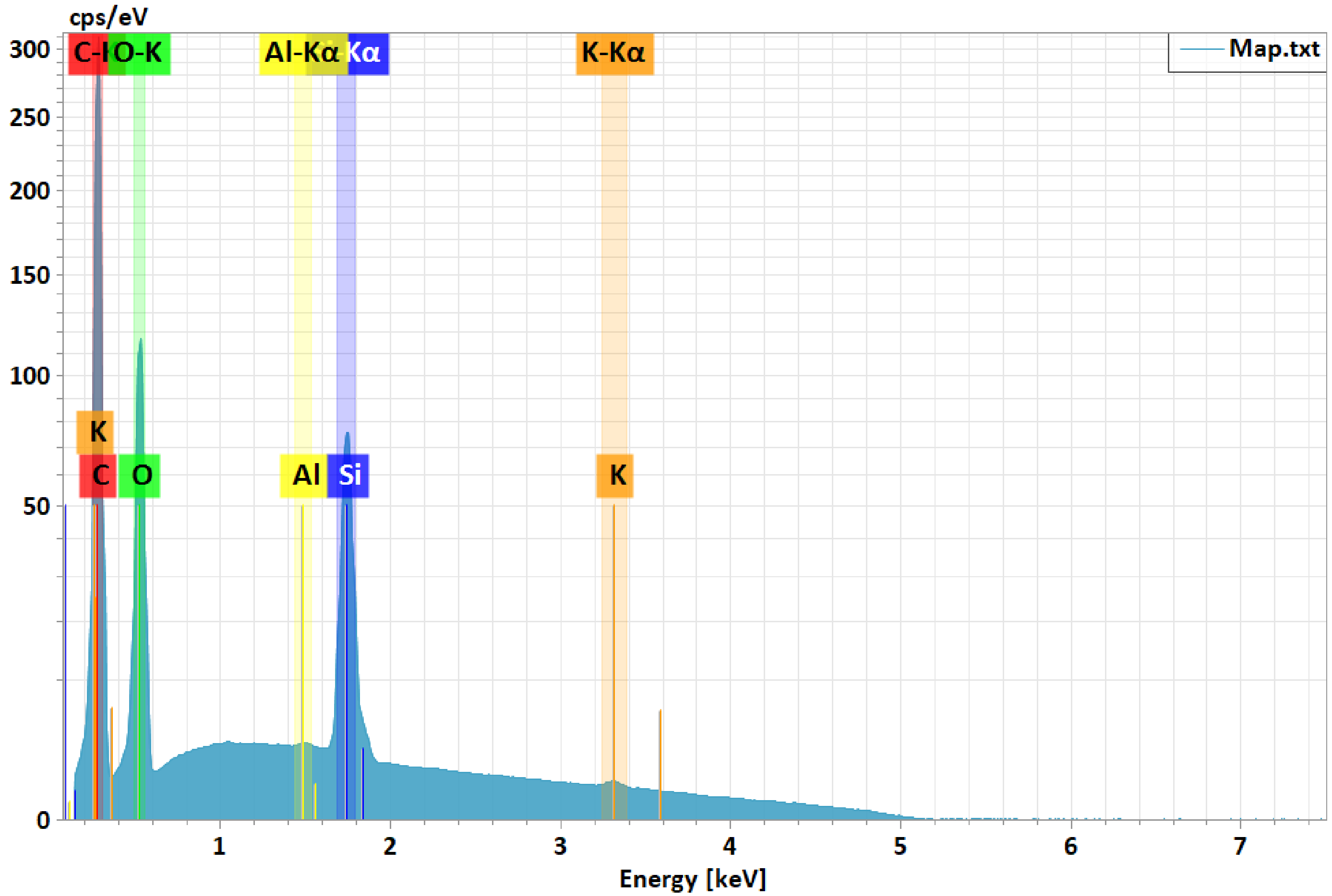
C 20kV

Monte Carlo simulations of electron trajectories


2kV
5kV
20kV
C
0.0 nm
15.0 nm
30.0 nm
45.0 nm
60.0 nm
10nm
-38.2 nm
-19.1 nm
0.0 nm
19.1 nm
38.2 nm
70.0 nm
140.0 nm
210.0 nm
280.0 nm
50nm
-178.3 nm
-89.2 nm
89.2 nm
178.3 nm
760.0 nm
1520.0 nm
2280.0 nm
3040.0 nm
500nm
-1936.3 nm
-968.2 nm
968.2 nm
1936.3 nm
Au
3.0 nm
6.0 nm
9.0 nm
12.0 nm
2nm
-7.6 nm
-3.8 nm
3.8 nm
7.6 nm
11.0 nm
22.0 nm
33.0 nm
44.0 nm
5nm
-28.0 nm
-14.0 nm
14.0 nm
28.0 nm
120.0 nm
240.0 nm
360.0 nm
480.0 nm
100nm
-305.7 nm
-152.9 nm
152.9 nm
305.7 nm

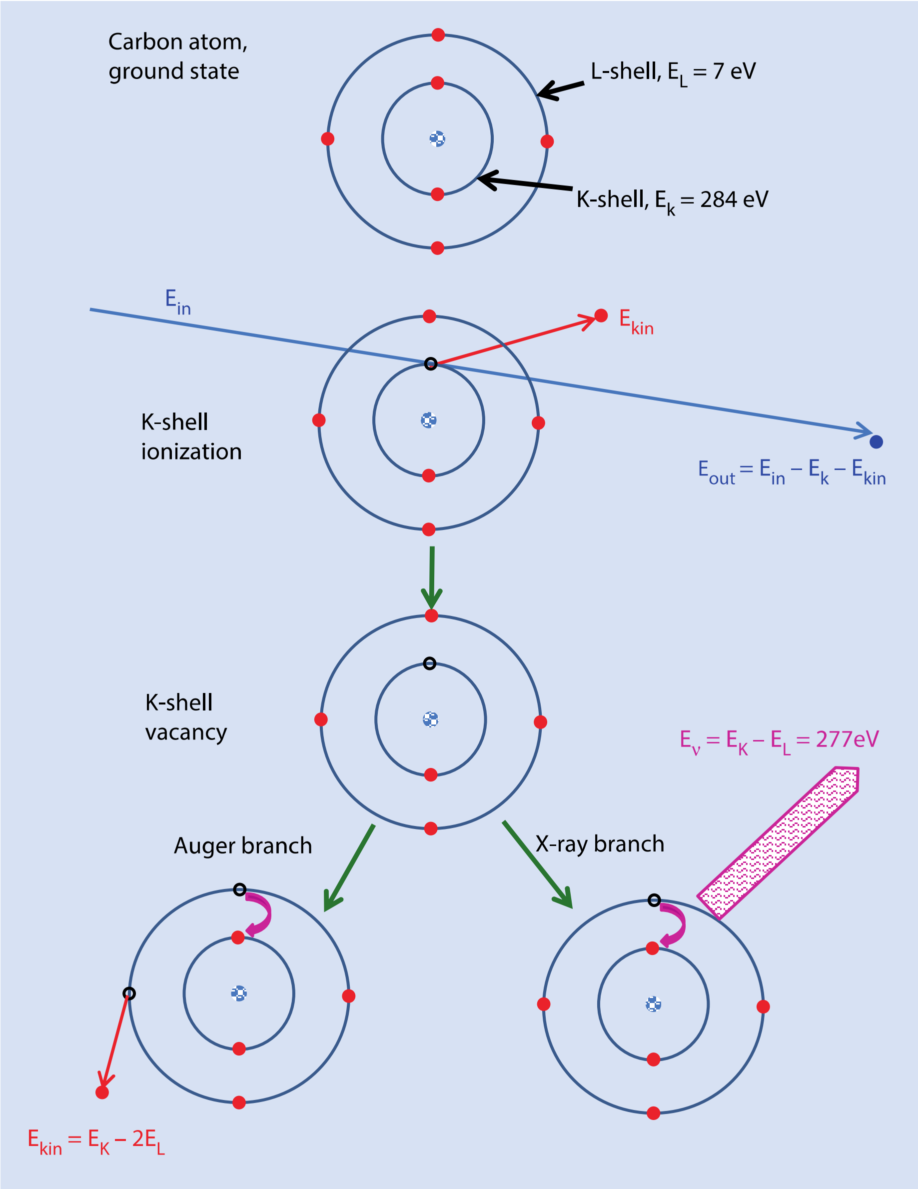
Measuring Elemental Composition
Energy Dispersive x-ray Spectroscopy - EDS

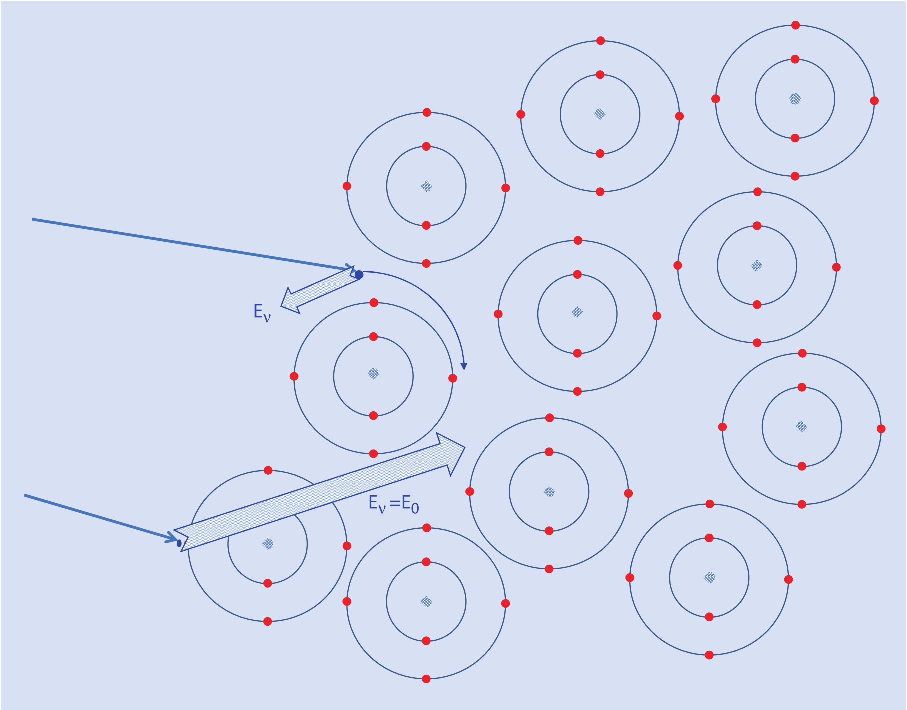




Low Voltage Electron Microscopy: Principles and Applications, D.C.Bell and N. Erdman

The Process of x-ray Generation

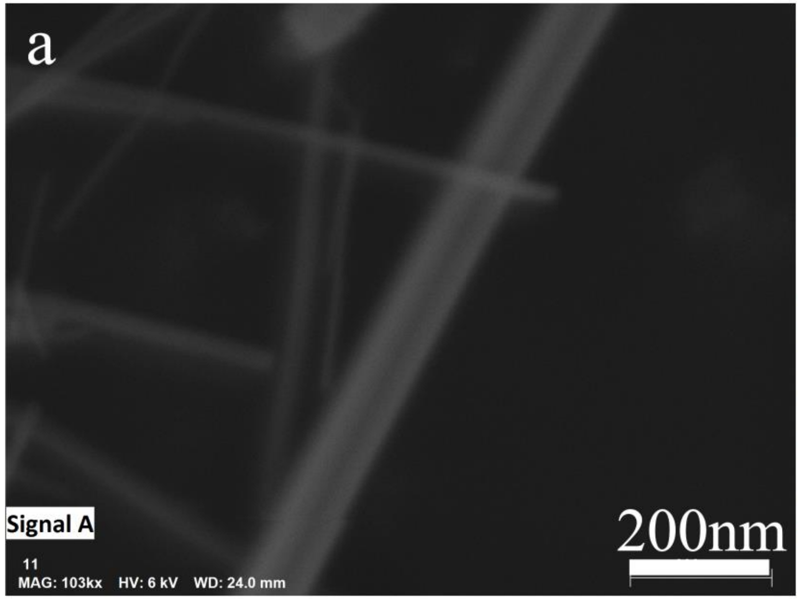


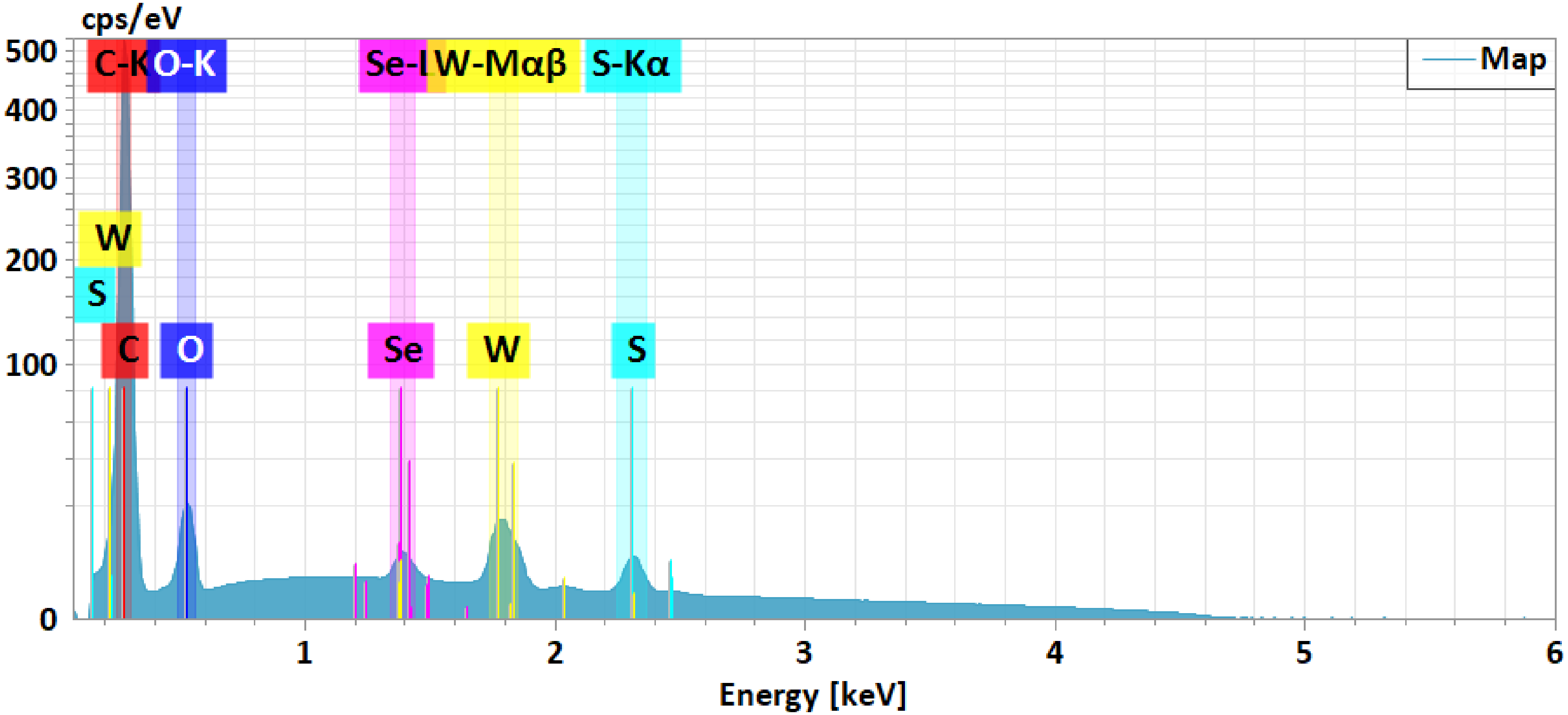


The incident electron is inelastically scattered. The difference in energy from an electron transition is expressed either as the ejection of an energetic electron with characteristic energy (Auger process) or by the emission of a characteristic x-ray photon.

J.I. Goldstein et al. Scanning Electron Microscopy and X-ray Microanalysis, Fourth Edition, 2018

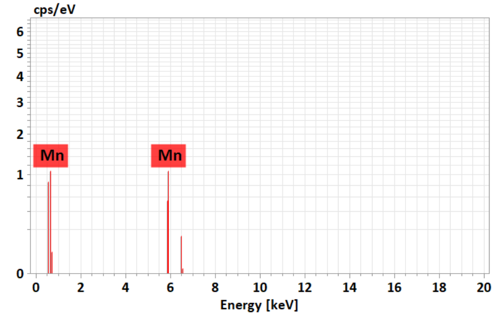
Measuring Elemental Composition
Energy Dispersive x-ray Spectroscopy - EDS

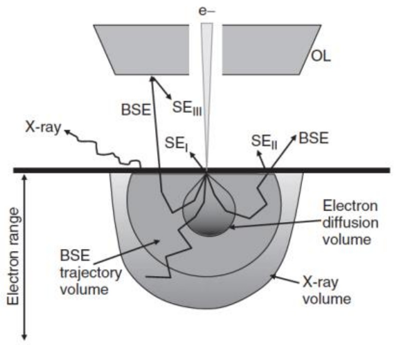


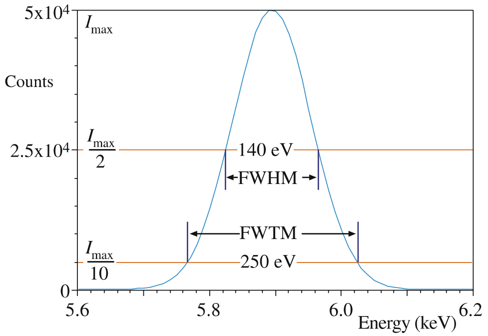


EDS Parameters

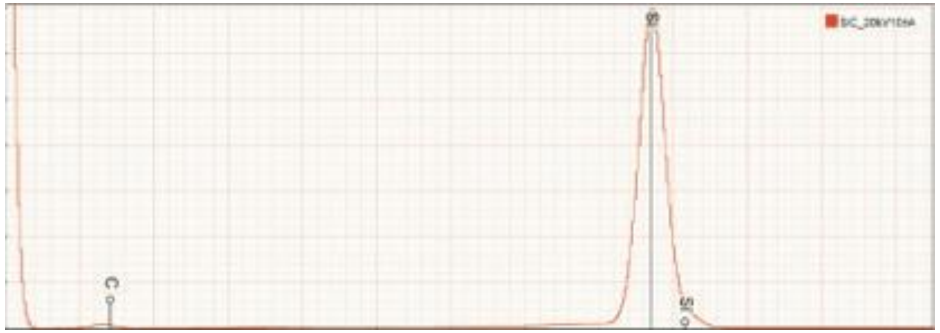
1. The critical ionization energy







2. Volume of interaction
(set the spatial resolution)



3. Signal to noise

4. Spectral resolution

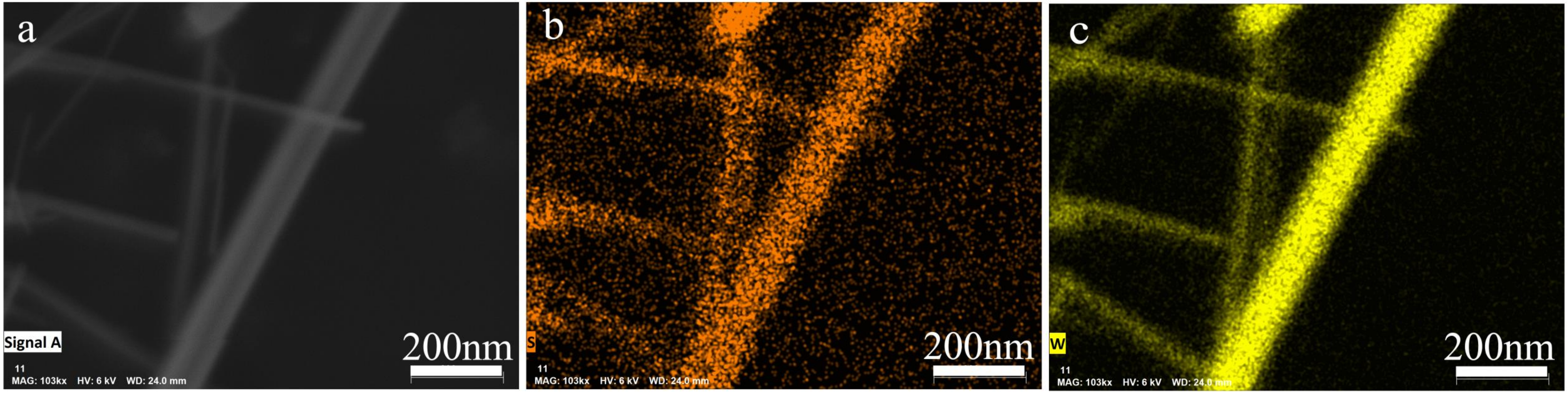
5. Minimum detected mass

6. Acquisition time
(Beam damage)

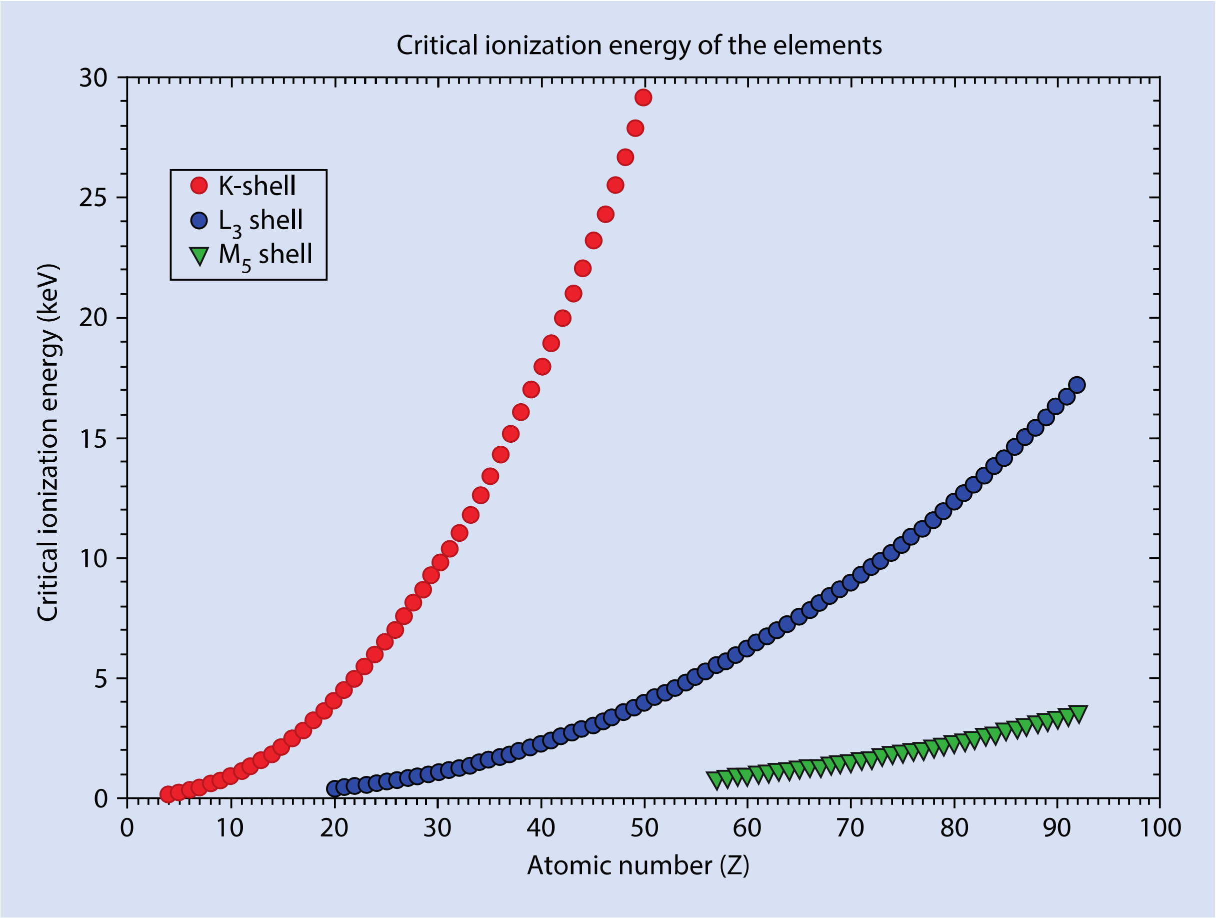
Sample (composition, preparation, topography)

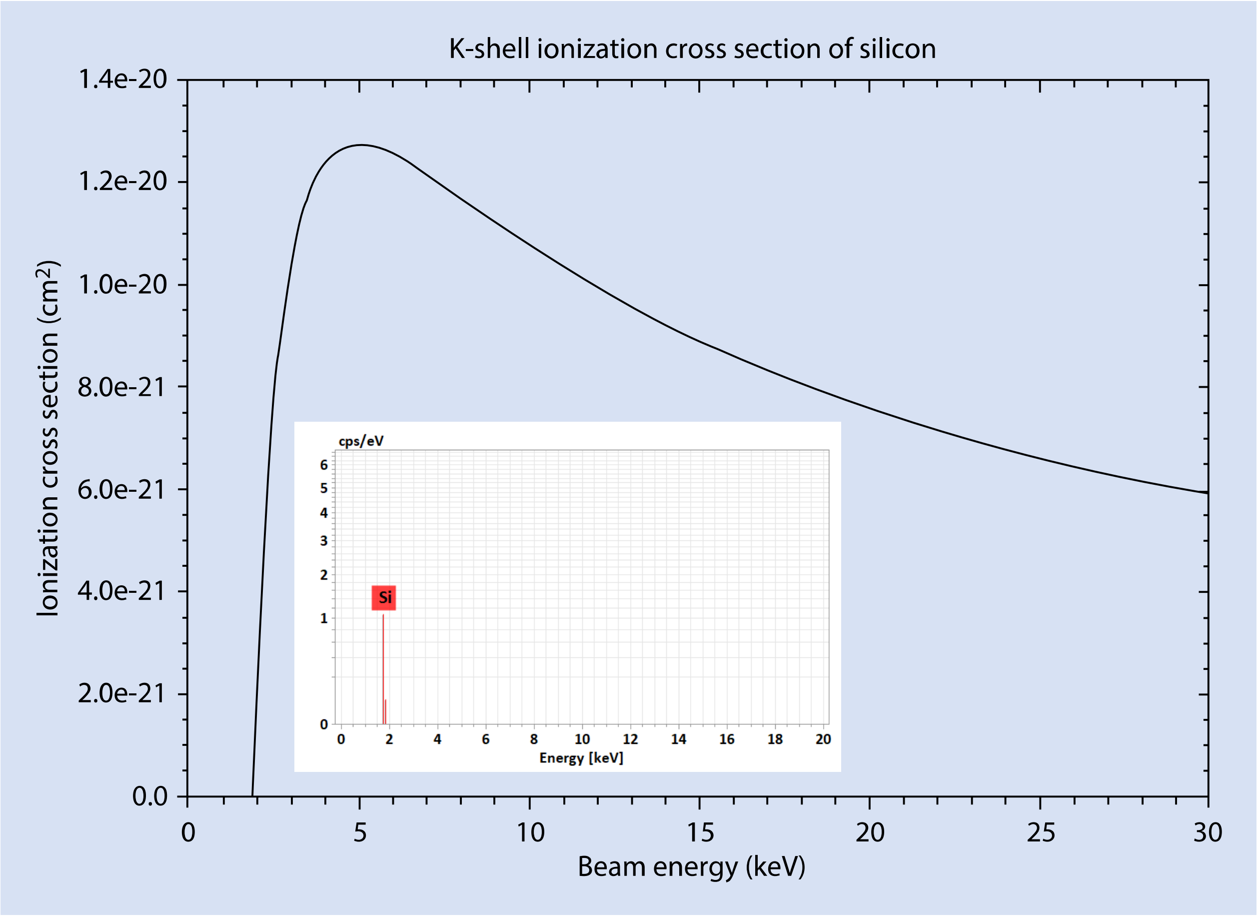
Instrument (EDS, SEM)

SEM-EDS of Nanoscale Materials



Critical Ionization Energy

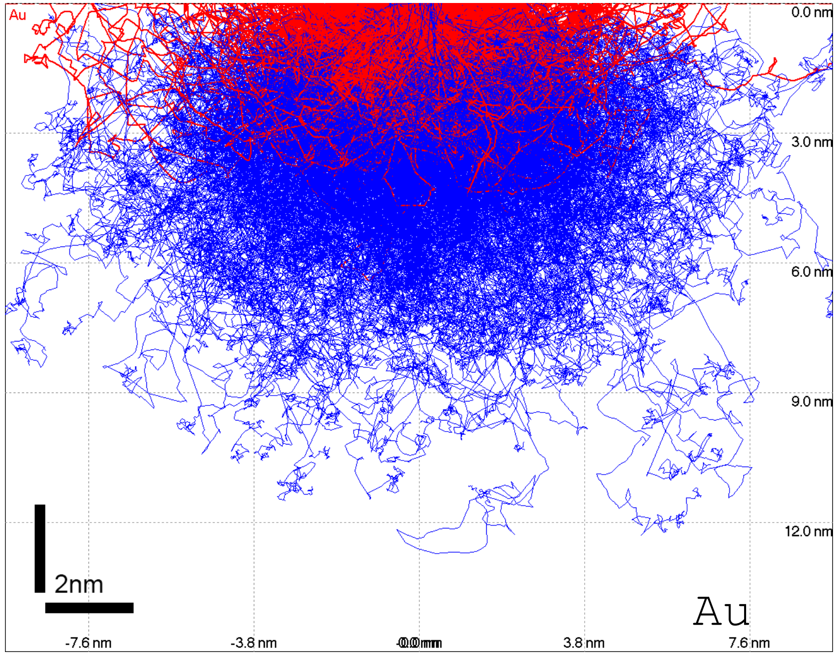




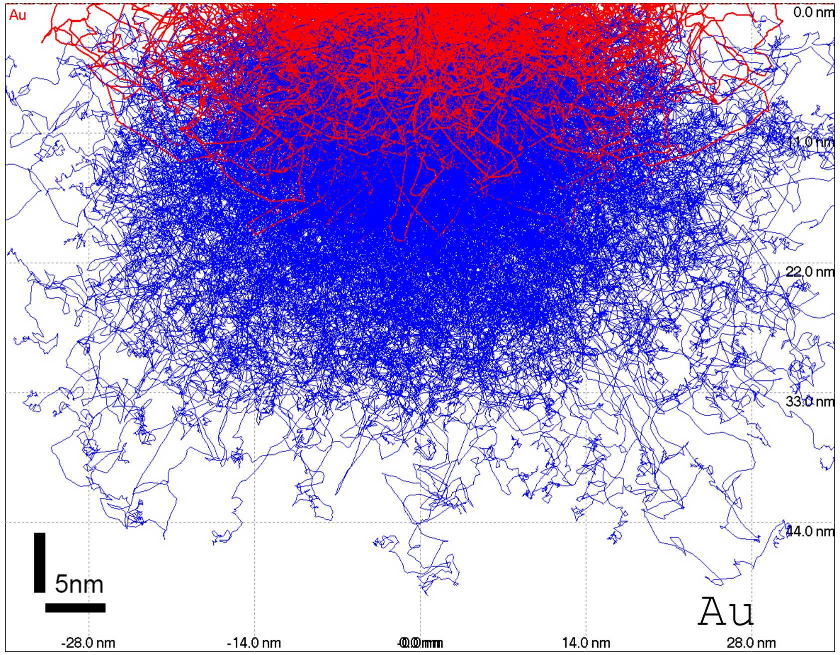
What is the limit ?

J.I. Goldstein et al. Scanning Electron Microscopy and X-ray Microanalysis, Fourth Edition, 2018

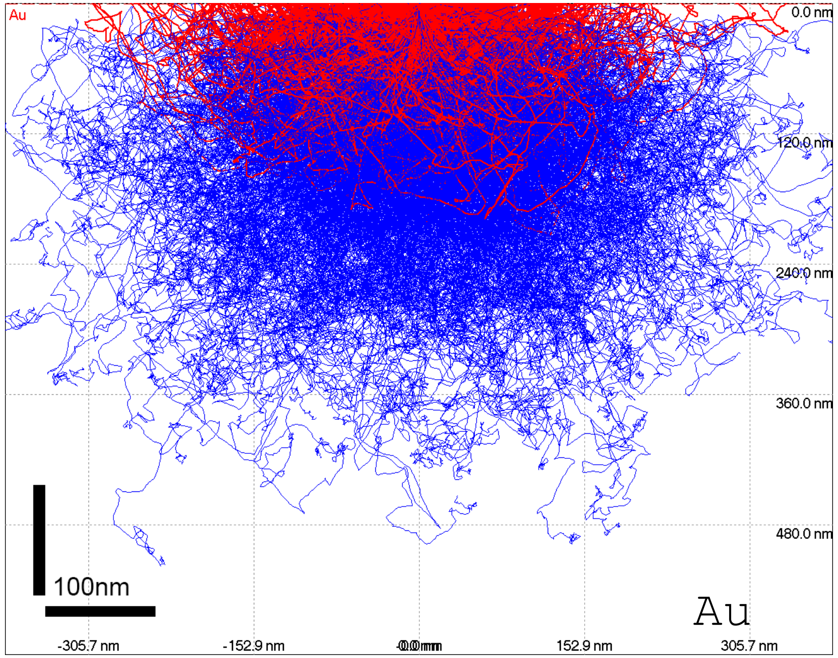
Volume of Interaction and Spatial Resolution



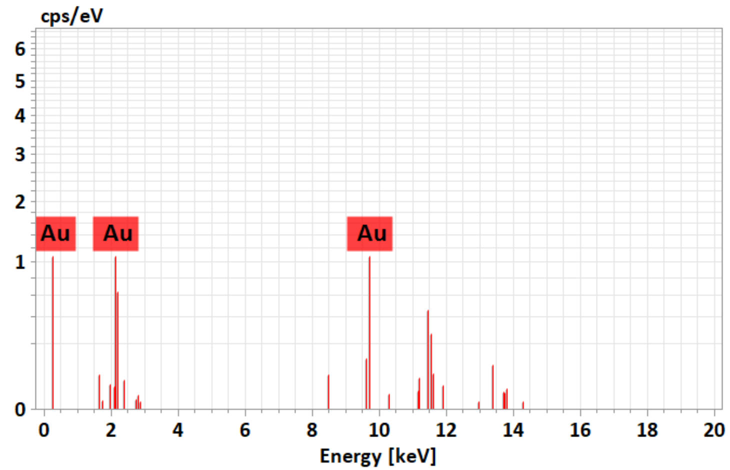
2kV



5kV



20kV



X-Ray Peak-to-Background Ratio

The most important factor in determining the limits of detection in x-ray spectrometric analysis is the presence of the continuum background, that is, noncharacteristic radiation at the same energy as the characteristic radiation of interest. The peak-to-background ratio can be calculated as follow:

$$\frac{P}{B} = \frac{I_{\mathrm{c}}}{I_{\mathrm{cm}}} = \frac{1}{Z}\left(\frac{E_0 - E_{\mathrm{c}}}{E_{\mathrm{c}}}\right)^{n-1}$$

Where I_c is the characteristic x-ray intensity and I_{cm} is the continuum x-ray intensity, Z is the atomic number, E_o is the accelerating energy, E_c is the critical ionization energy and n is a constant for a particular shell.

As the accelerating voltage increases the peak to background increases.

J.I. Goldstein et al. Scanning Electron Microscopy and X-ray Microanalysis, Fourth Edition, 2018

EDS Parameters

1. The critical ionization energy

2. Volume of interaction (set the spatial resolution)

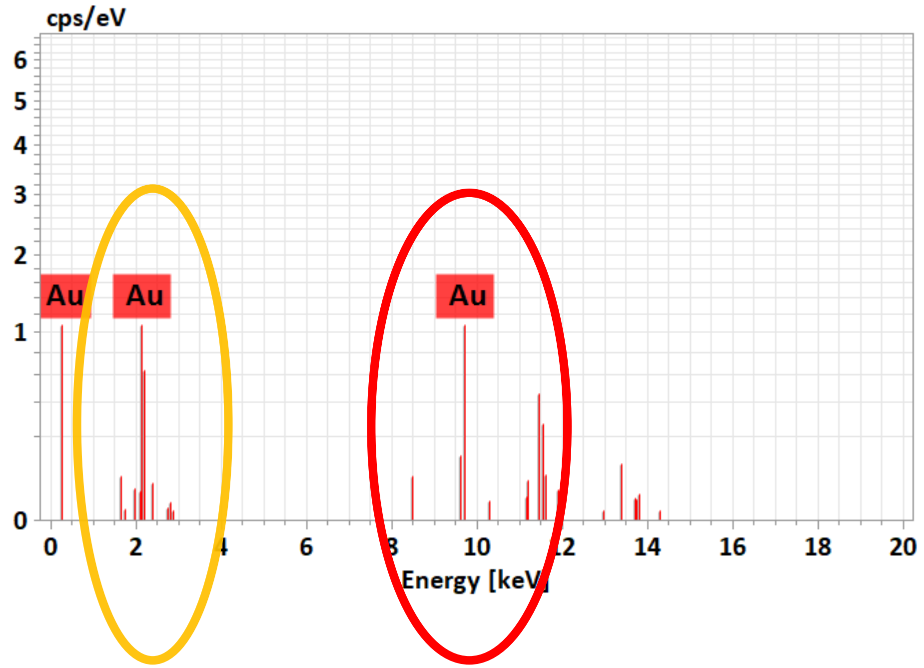
3. Signal to noise

4. Spectral resolution

5. Minimum detected mass

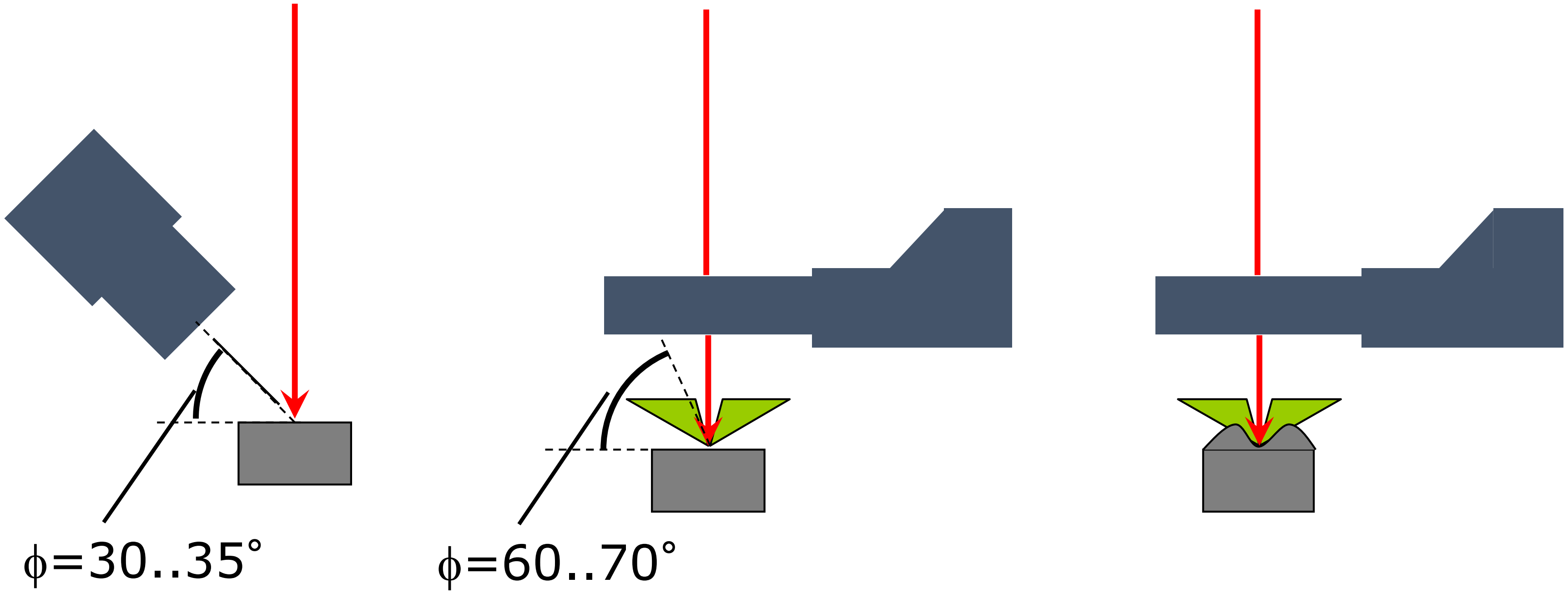
6. Acquisition time (Beam damage)

Electron beam's acceleration voltage



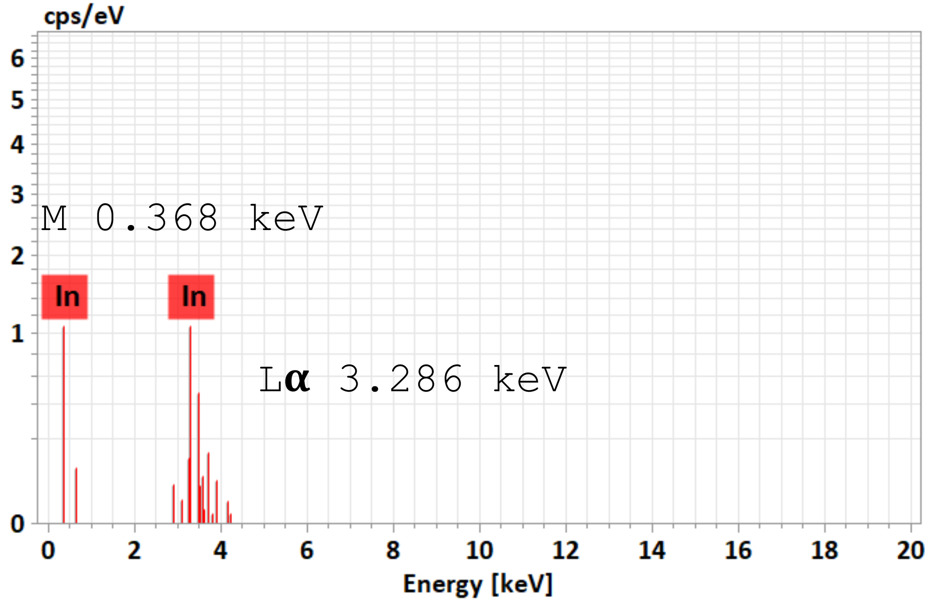
The Advantage of High take-off Angle and Annular Design

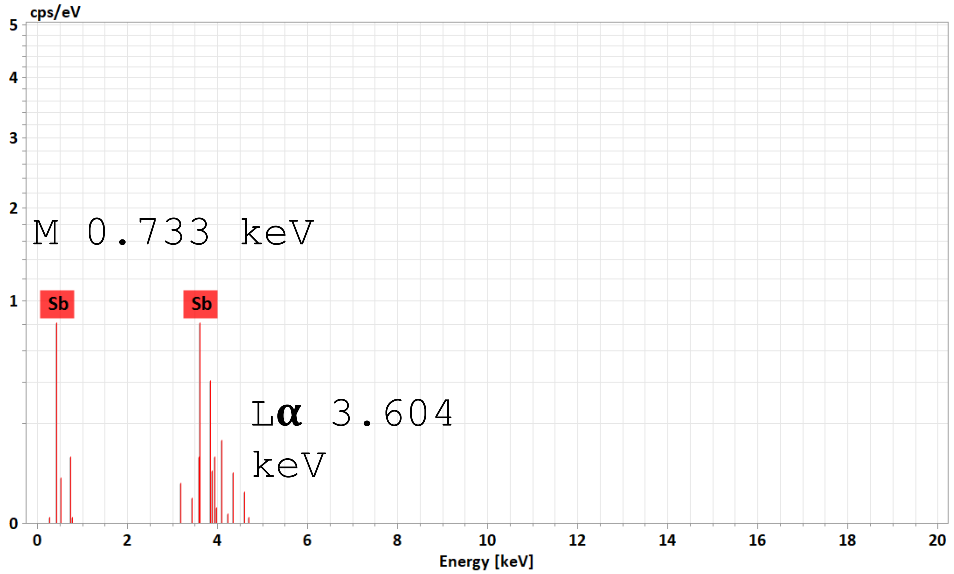
Take-off angle comparison: XFlash® 5060FQ vs. conventional SDDs:



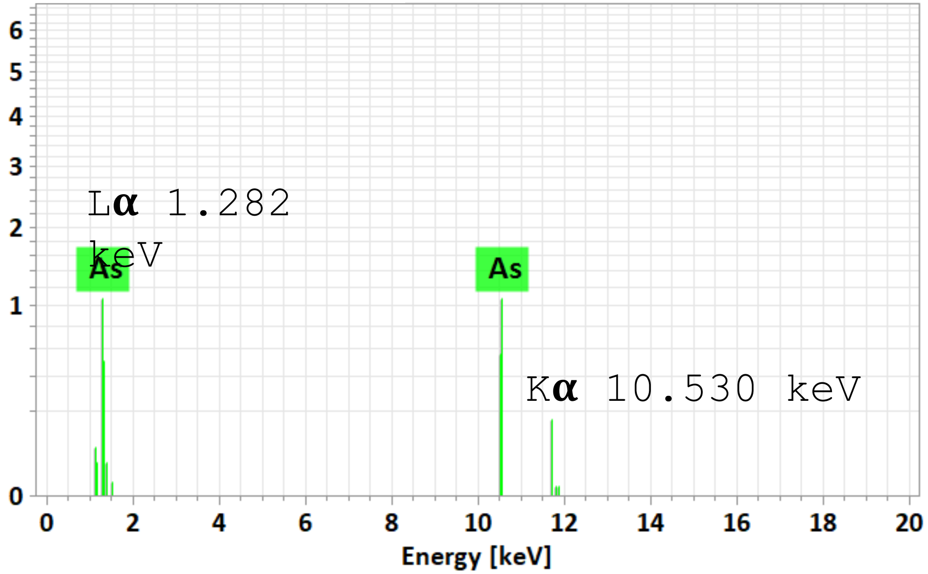
Max Patzschke, Application Scientist EDS, Bruker Nano GmbH, Berlin Germany

InAsSb Nanowires



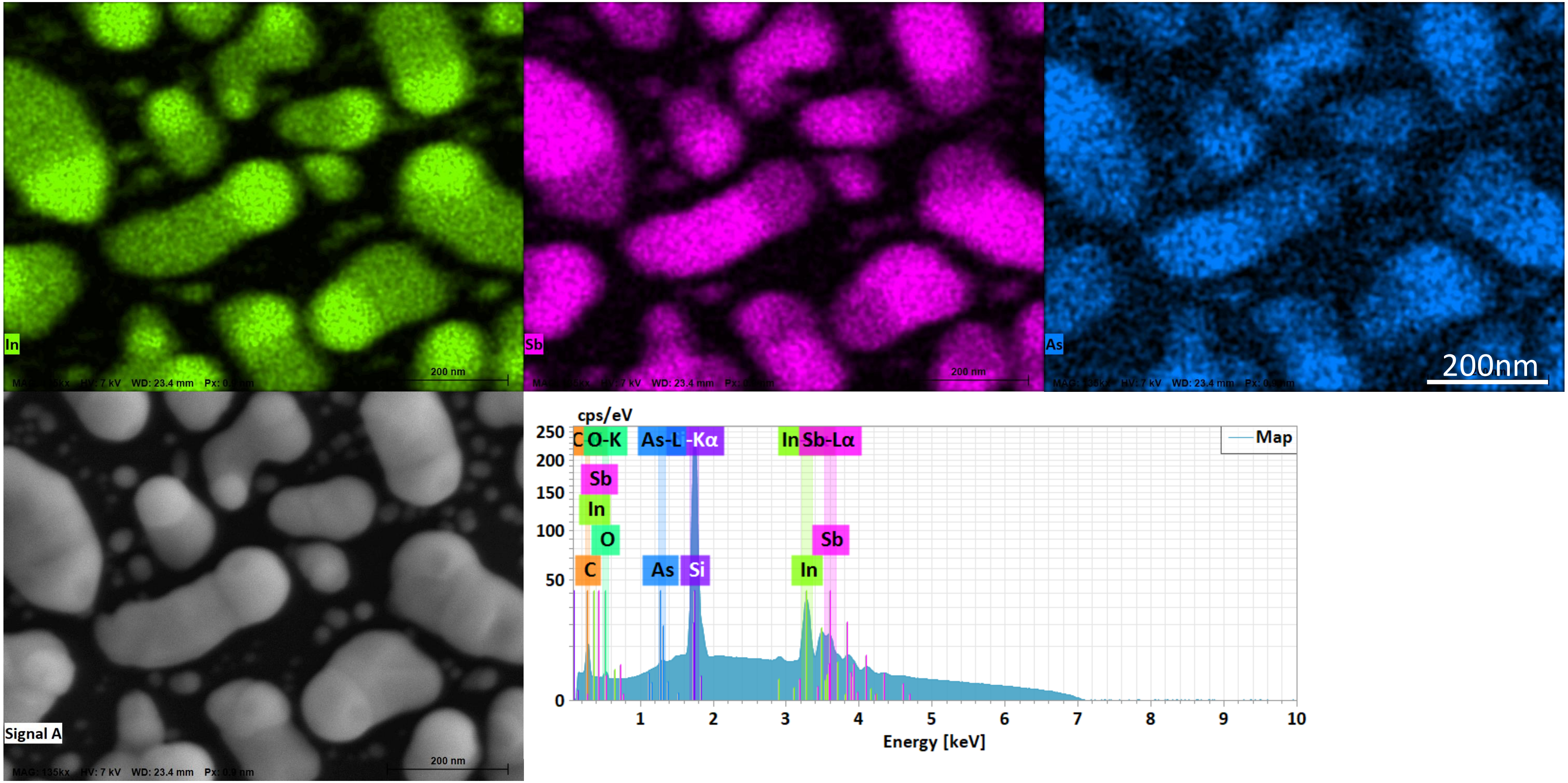


7 kV



Dr. Man Suk Song and Dr. Hadas Shtrikman , Condensed Matter Physics Dept., WIS

InAsSb Nanoparticles



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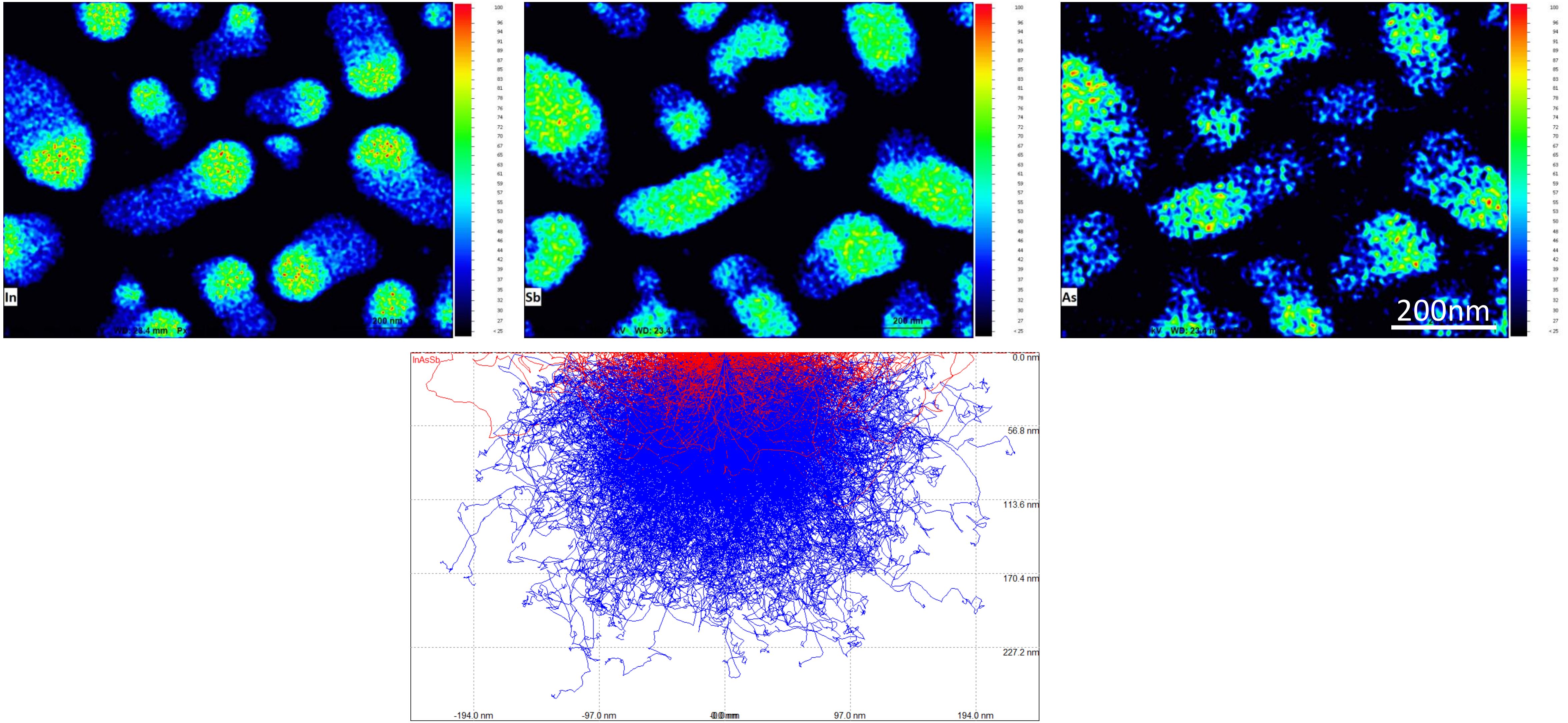
InAsSb Nanoparticles



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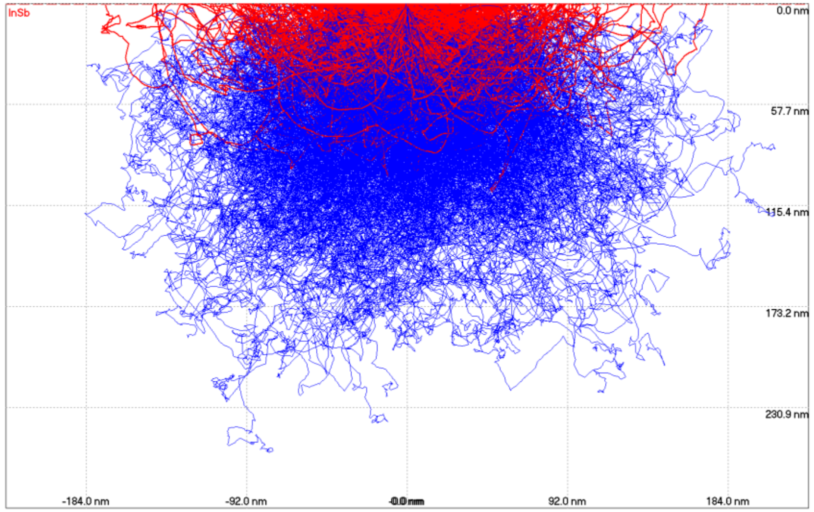
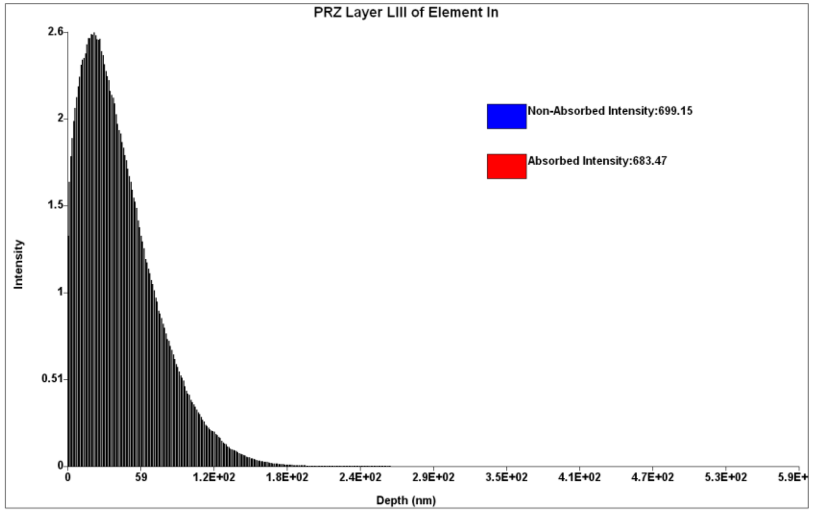
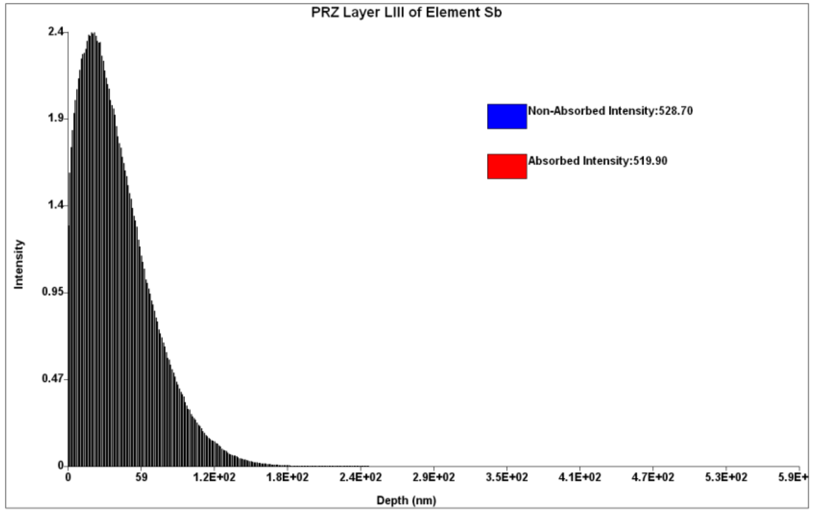
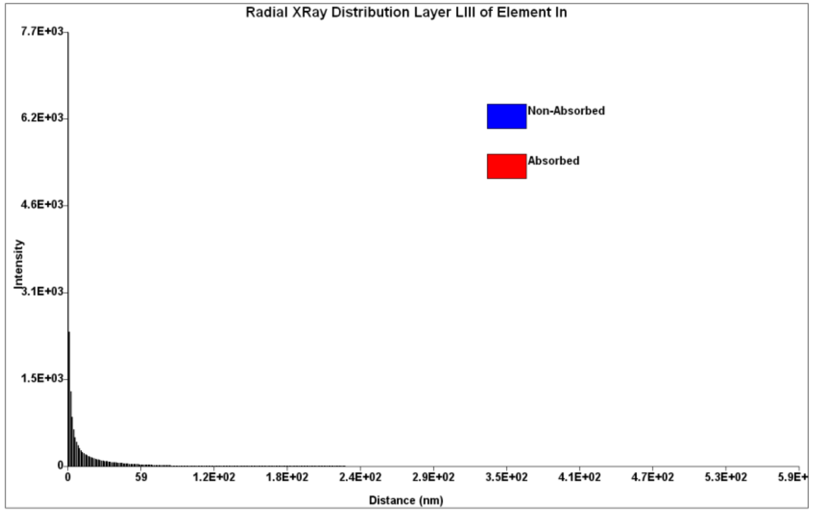
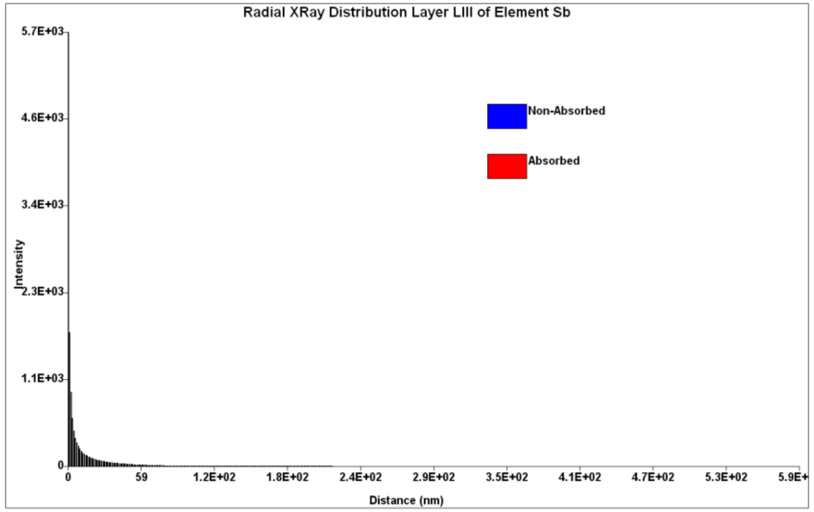
InAsSb Nanoparticles











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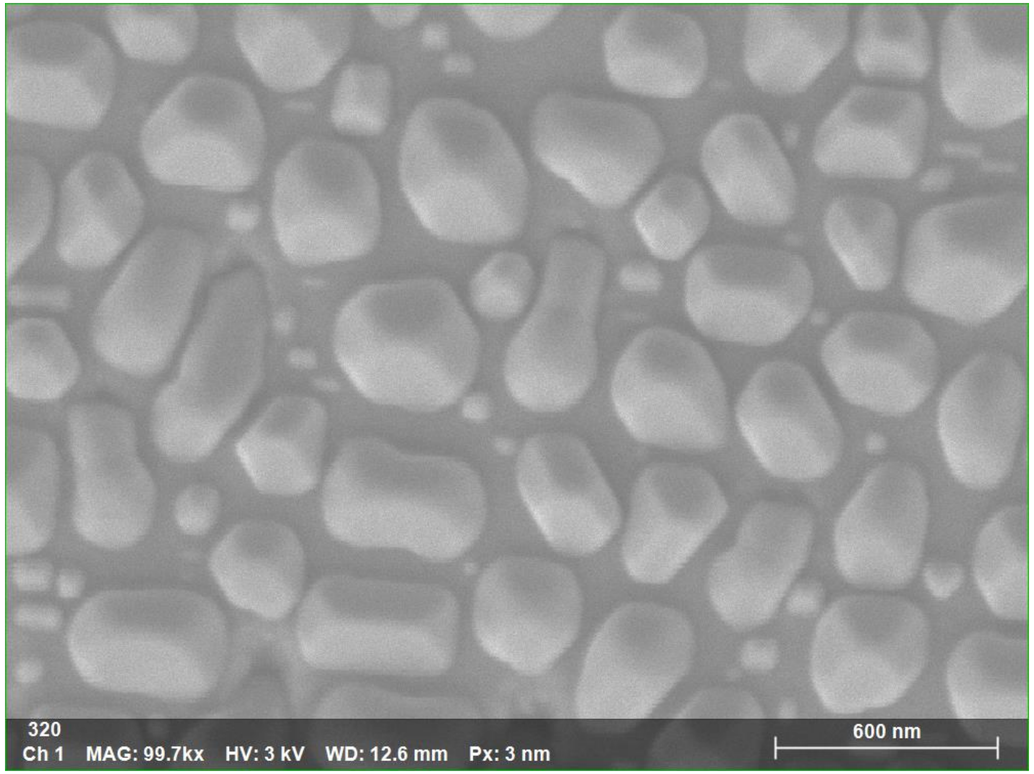
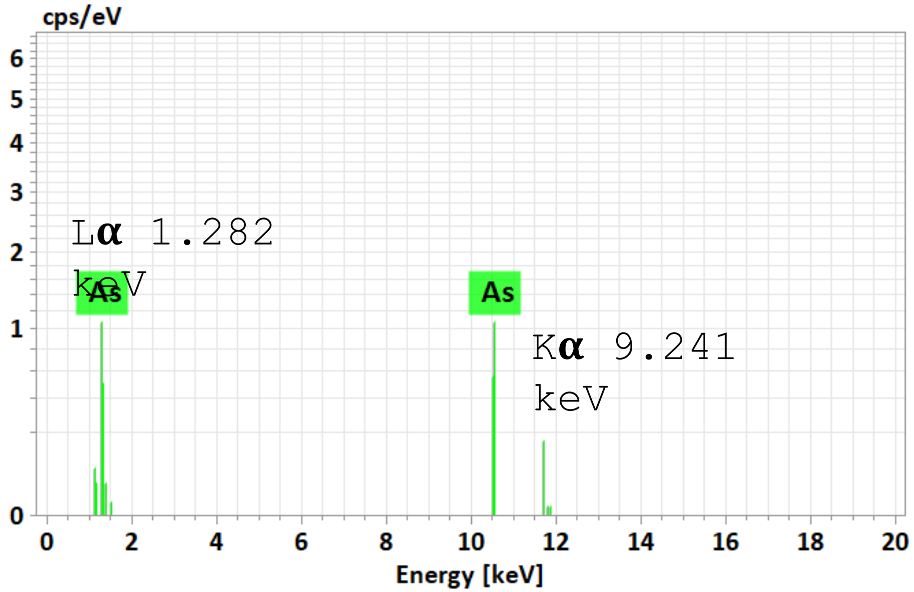
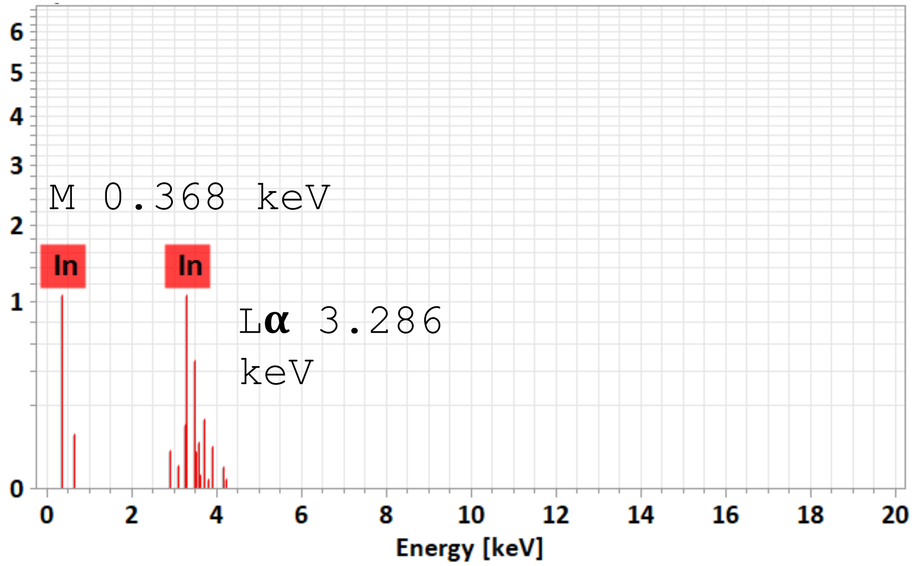
In on InAs







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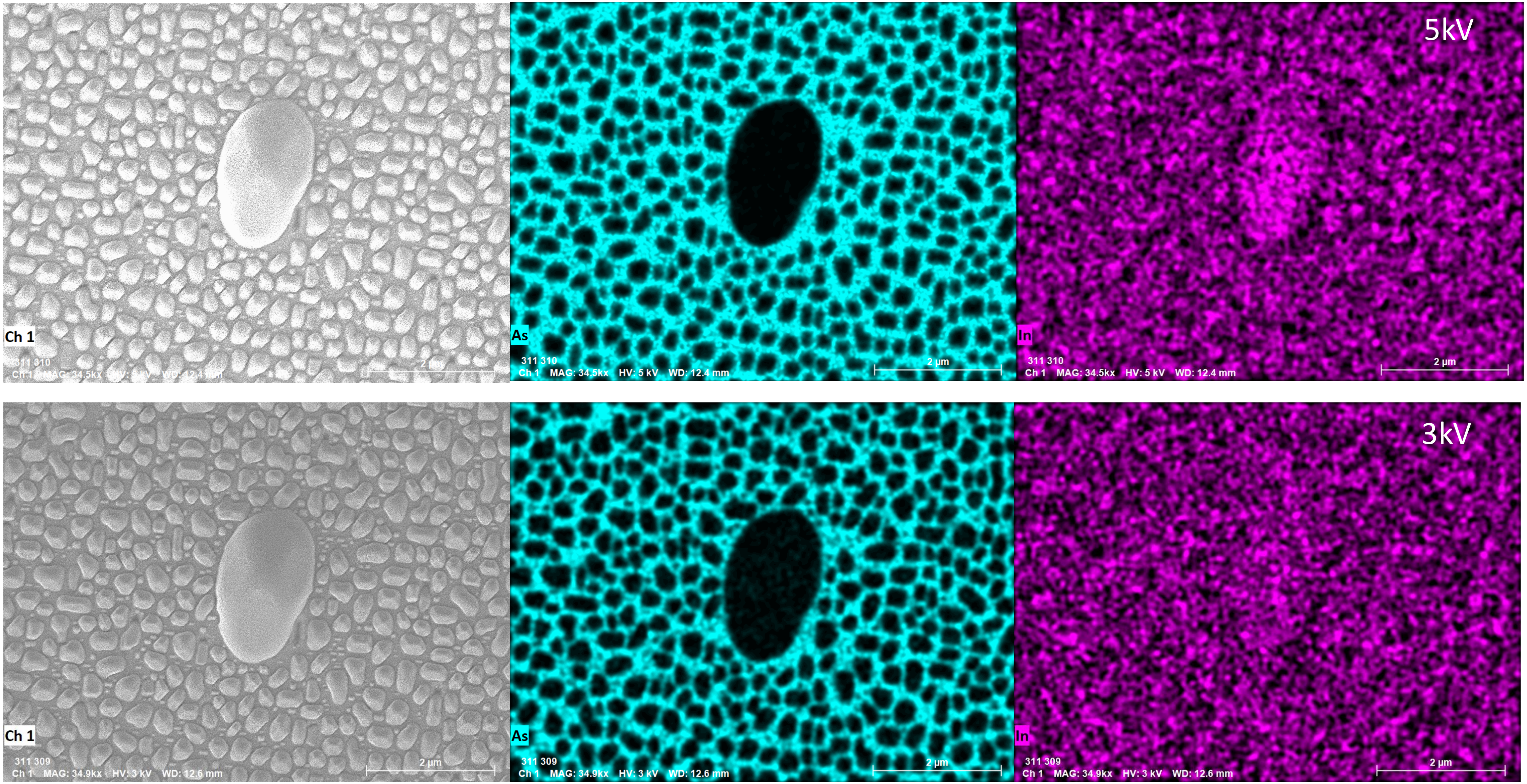
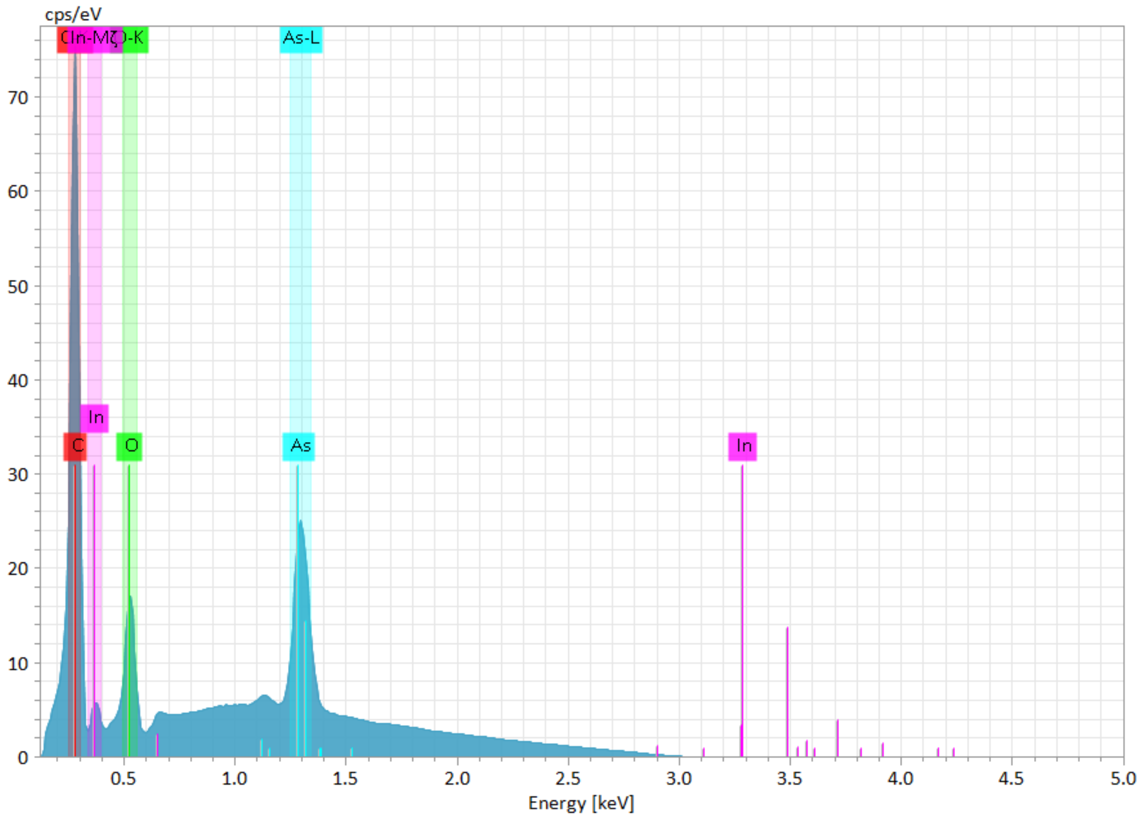
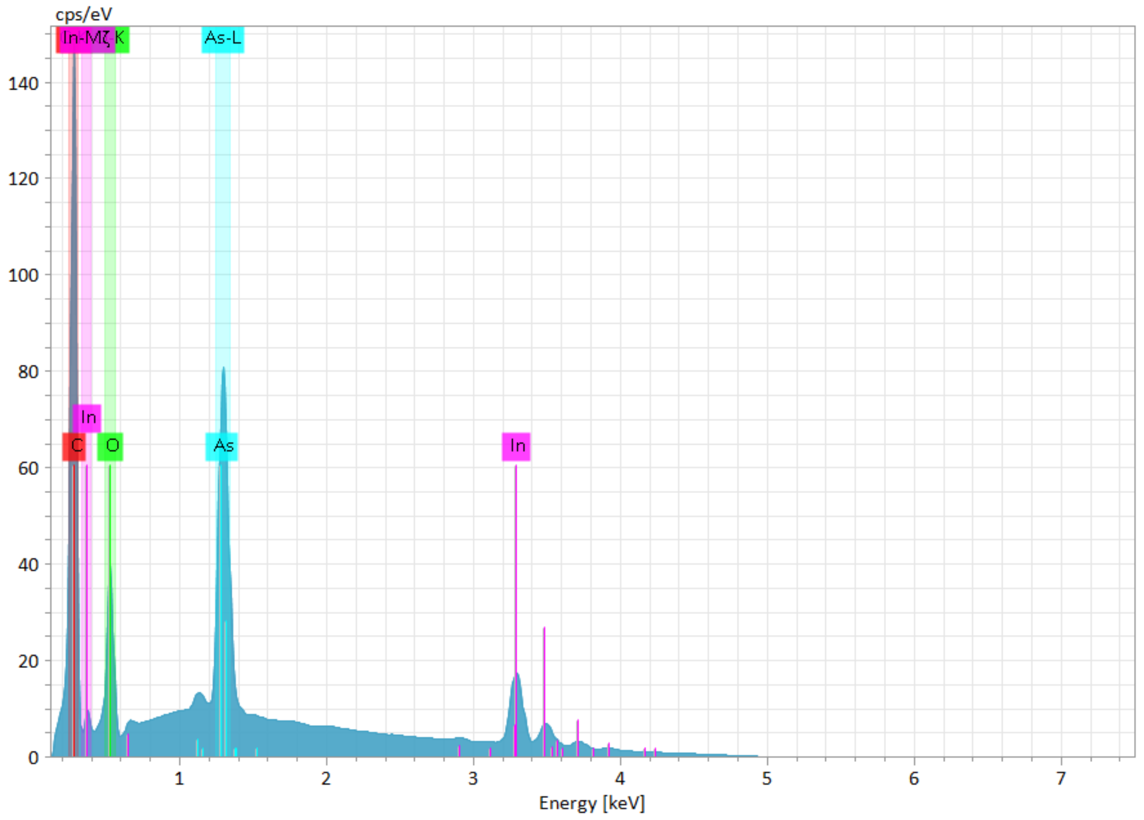
In on InAs


5kV
Ch 1
As
In
311 310
Ch 1 MAG: 34.5kx HV: 5 kV WD: 12.4 mm
2 µm
3kV
311 309
Ch 1 MAG: 34.9kx HV: 3 kV WD: 12.6 mm

In on InAs



3 kV



5 kV

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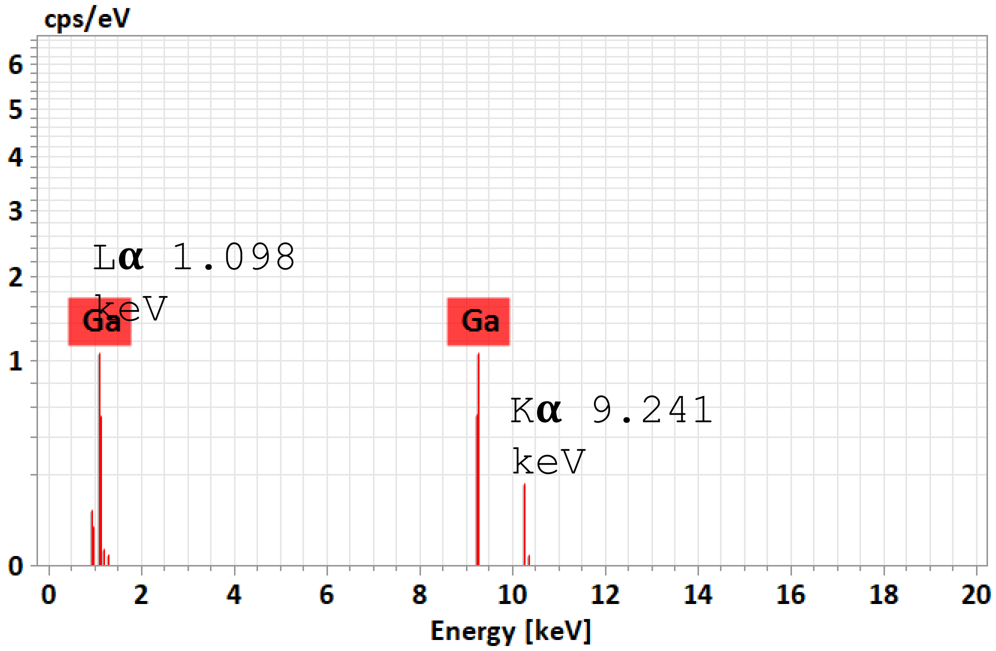
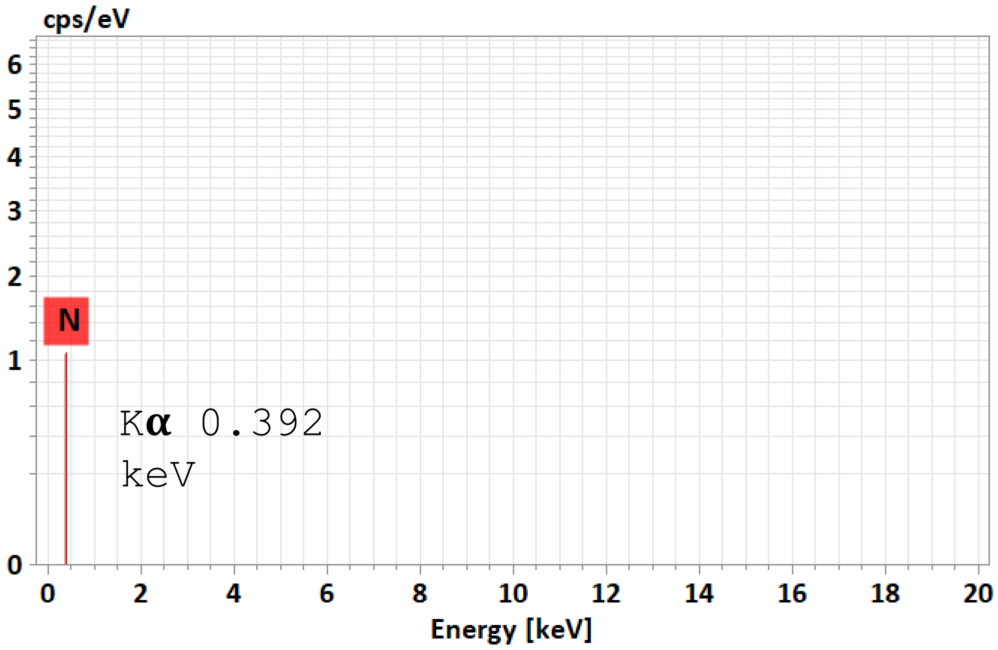
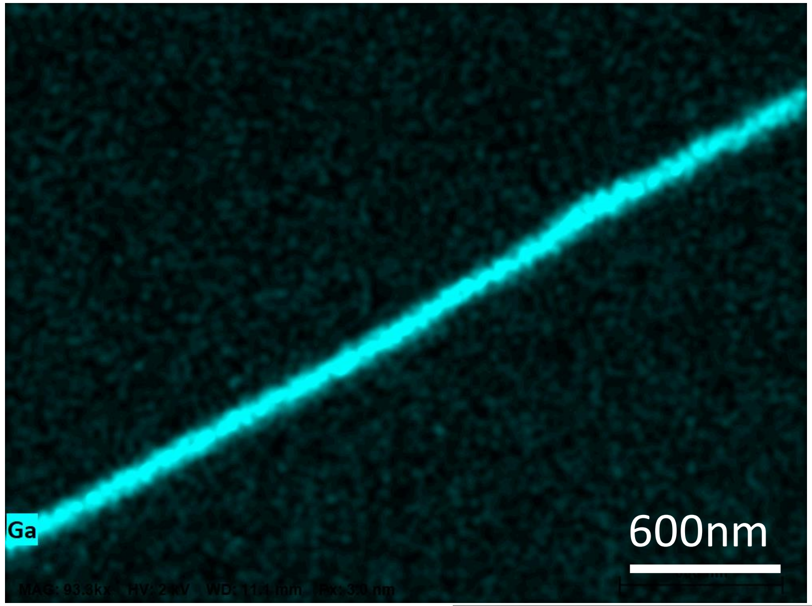
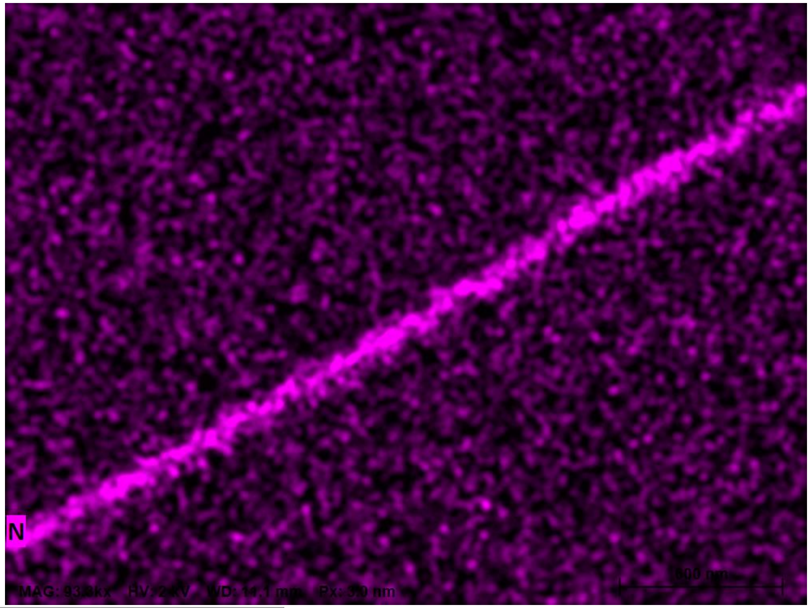
GaN Nanowires on Sapphire





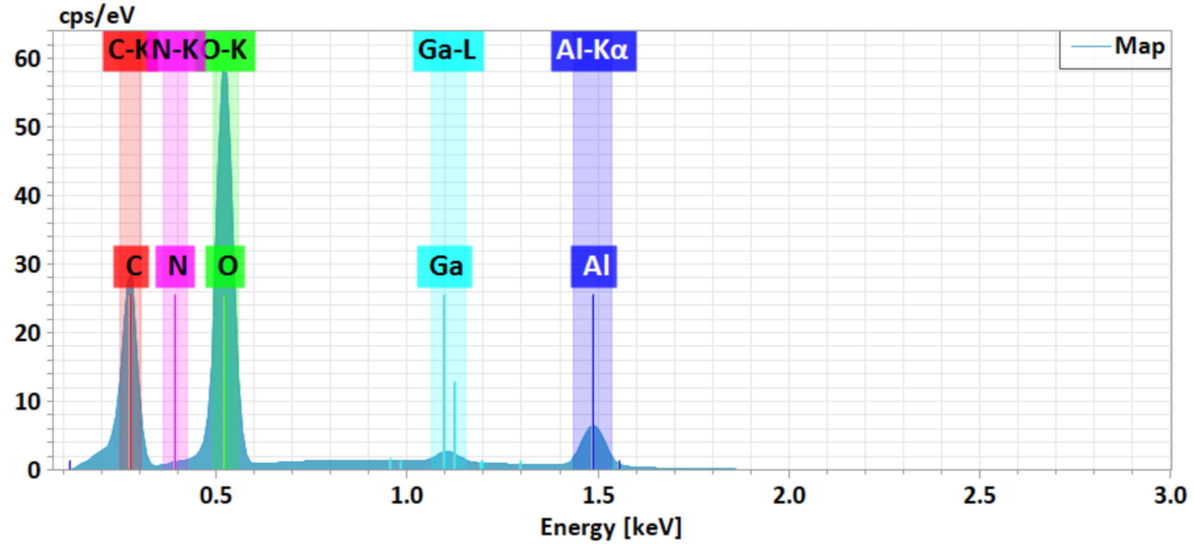
GaN Nanowires on Sapphire

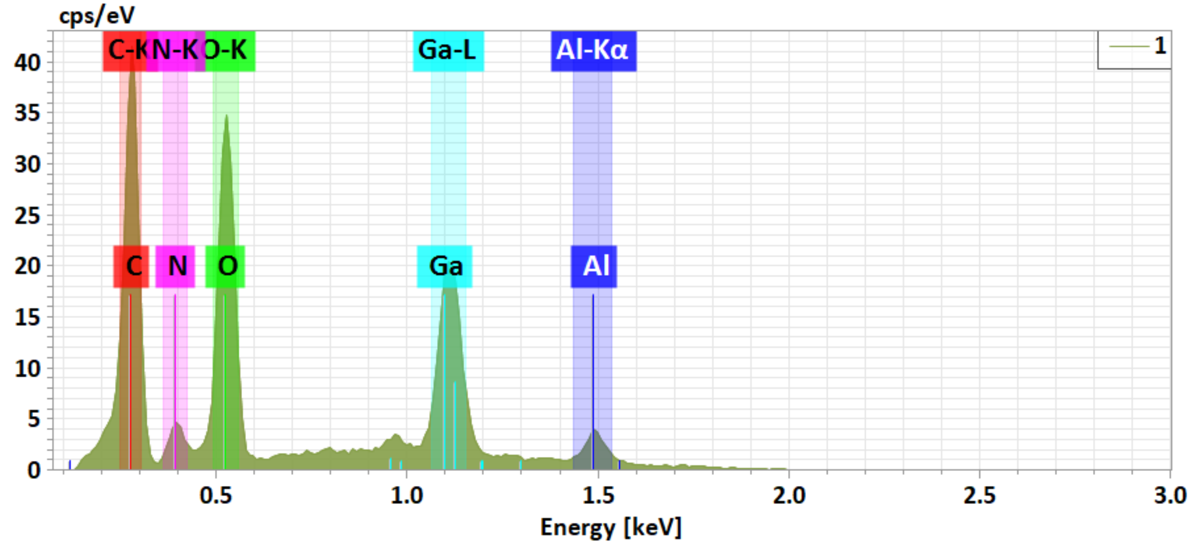




Prof. Ernesto Joselevich, Materials and Interfaces Dept., WIS

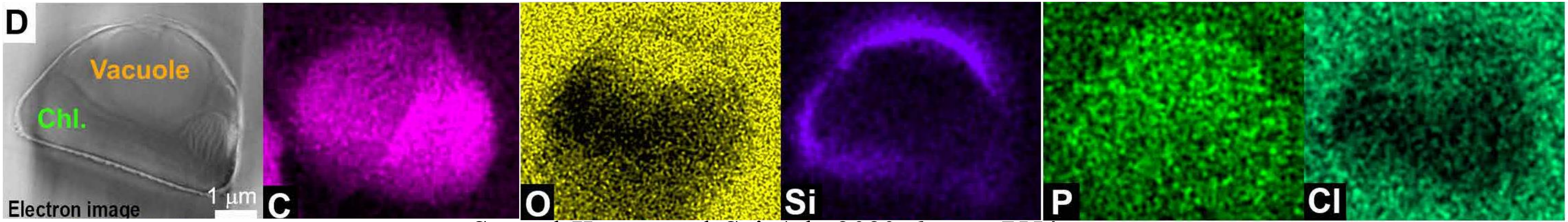
GaN Nanowires on Sapphire





Prof. Ernesto Joselevich, Materials and Interfaces Dept., WIS

SEM-EDS in Life Science



Santosh Kumar et al. Sci. Adv. 2020; 6 : eaaz7554

EDS Parameters

1. The critical ionization energy

2. Volume of interaction
(set the spatial resolution)

3. Signal to noise

4. Spectral resolution

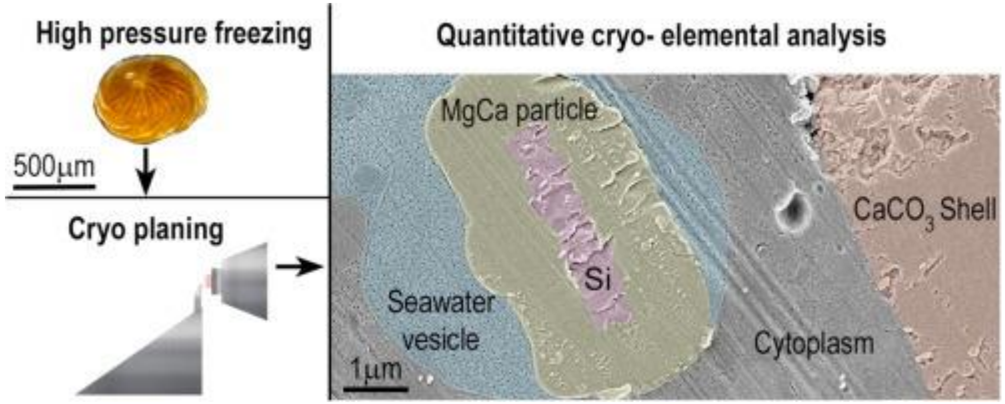
5. Minimum detected mass

6. Acquisition time
(Beam damage)

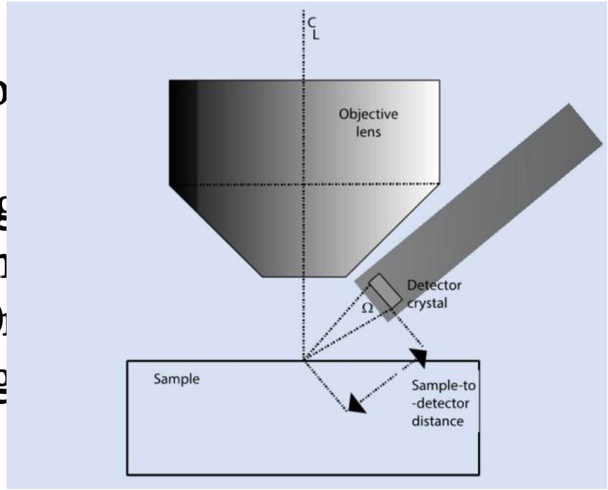
Cryo EDS

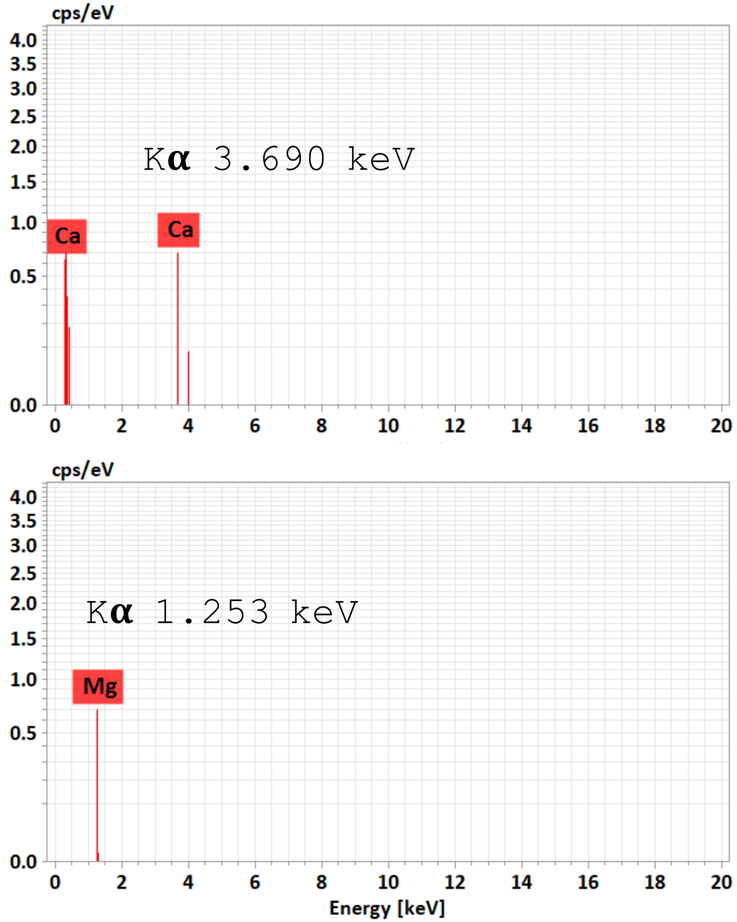
Characterization of unusual MgCa particles involved in the formation of foraminifera shells using a novel quantitative cryo SEM/EDS protocol

Gal Mor Khalifa et al. Acta Biomaterialia 77 (2018) 342–351



Cryo EDS wo
HPF
Cryo planning
SE and BS im
C coating (10
EDS mapping

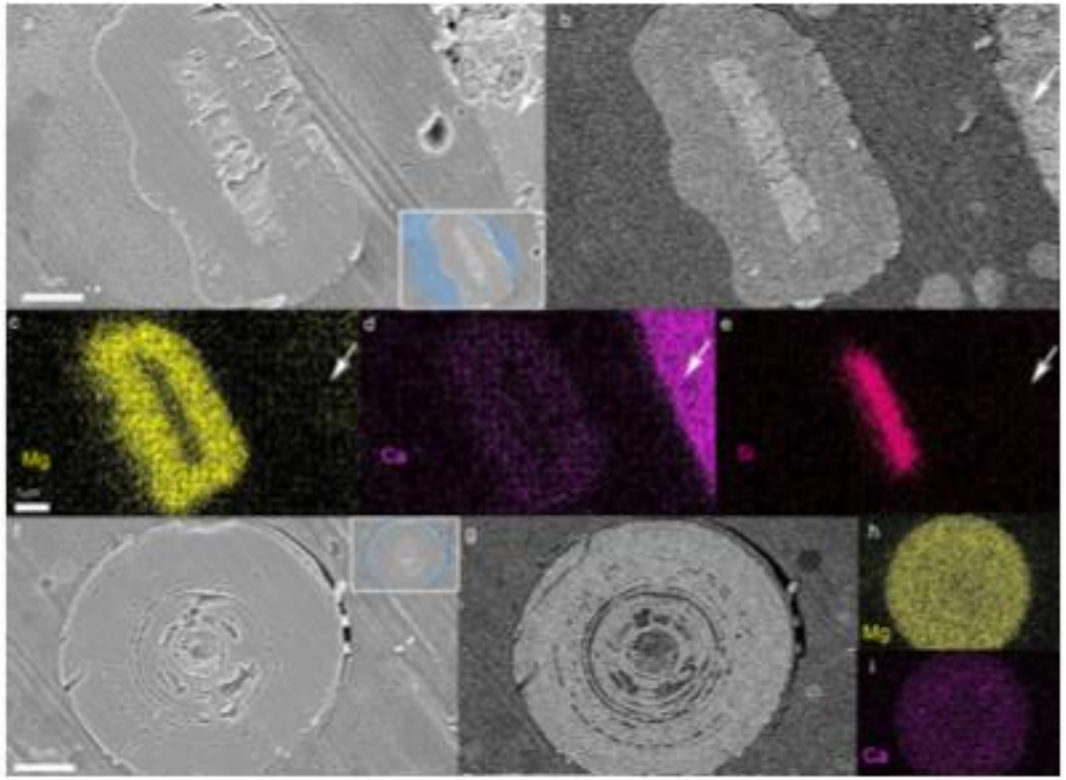


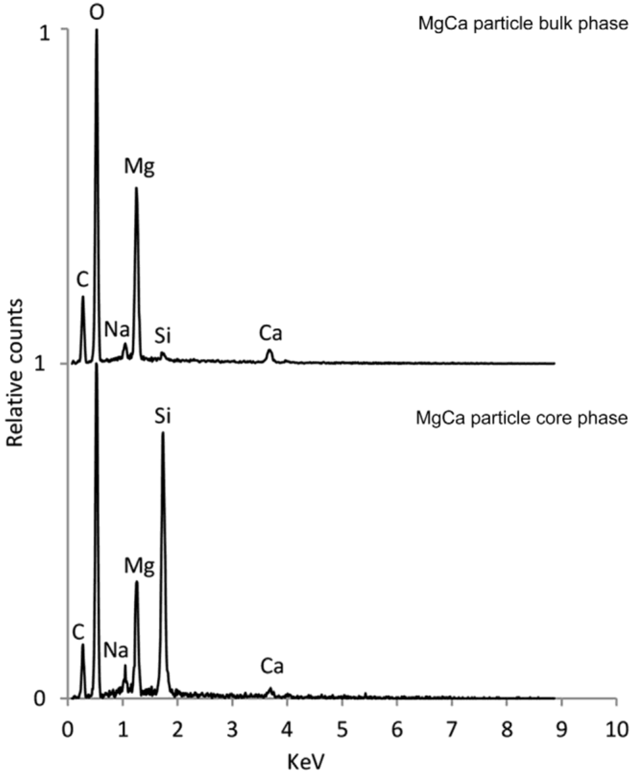


Dr. Gal Mor Khalifa, Prof. Lia Addadi and Prof. Steve Weiner group, Structural Biology Dept., WIS

Characterization of unusual MgCa particles involved in the formation of foraminifera shells using a novel quantitative cryo SEM/EDS protocol

Gal Mor Khalifa et al. Acta Biomaterialia 77 (2018) 342–351





Correlative cryo- SEM/EDS analysis of two intracellular MgCa particles

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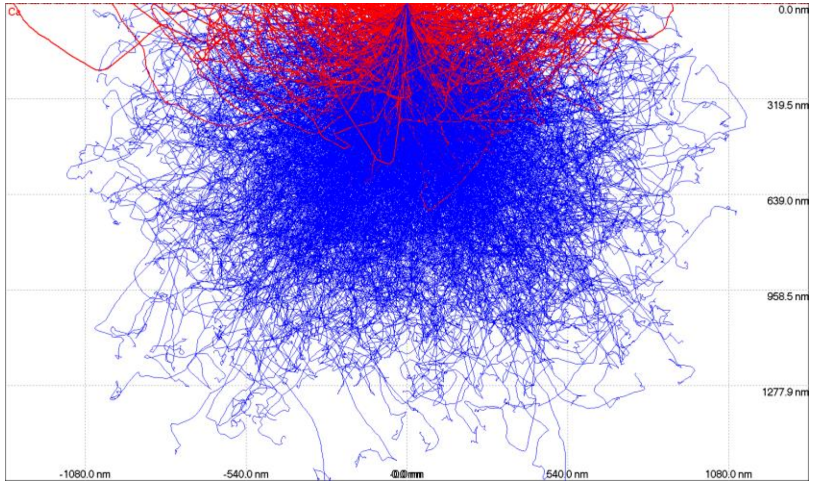
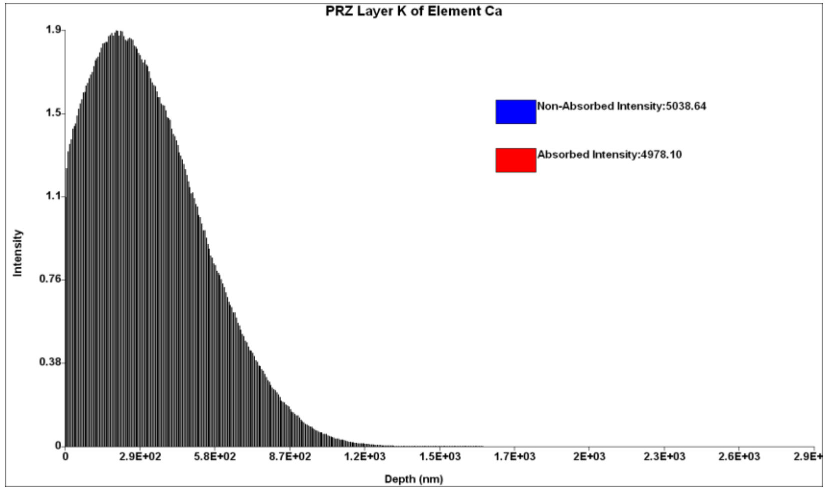
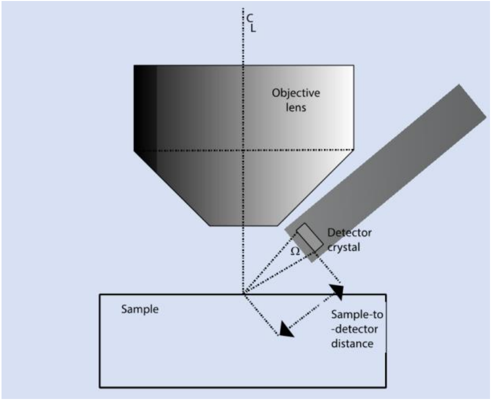
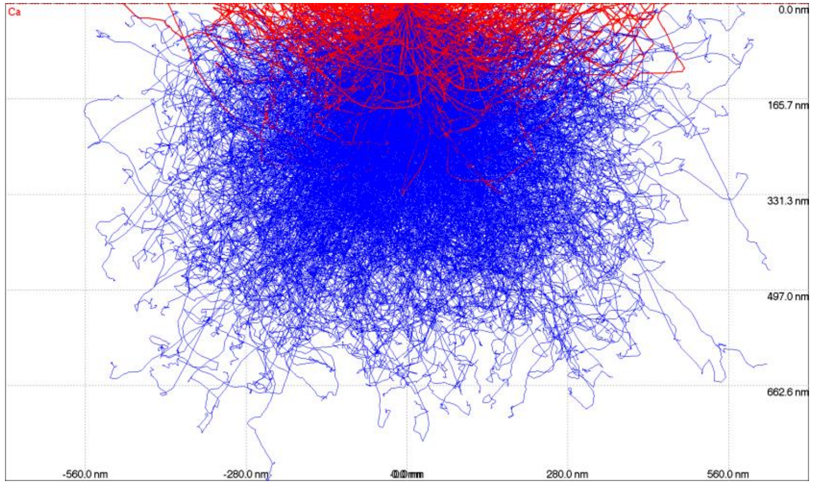
Ca - Volume of Interaction

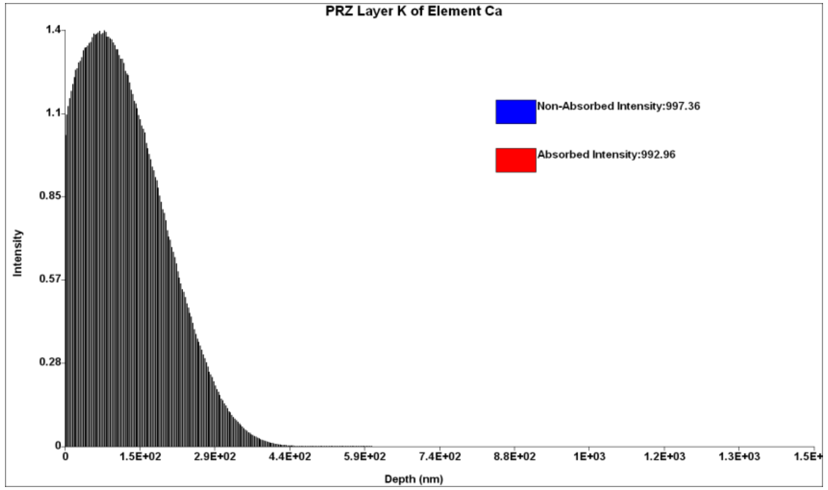




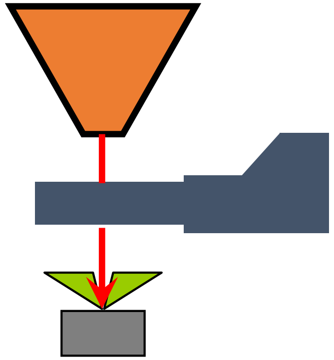
Ca 9kV





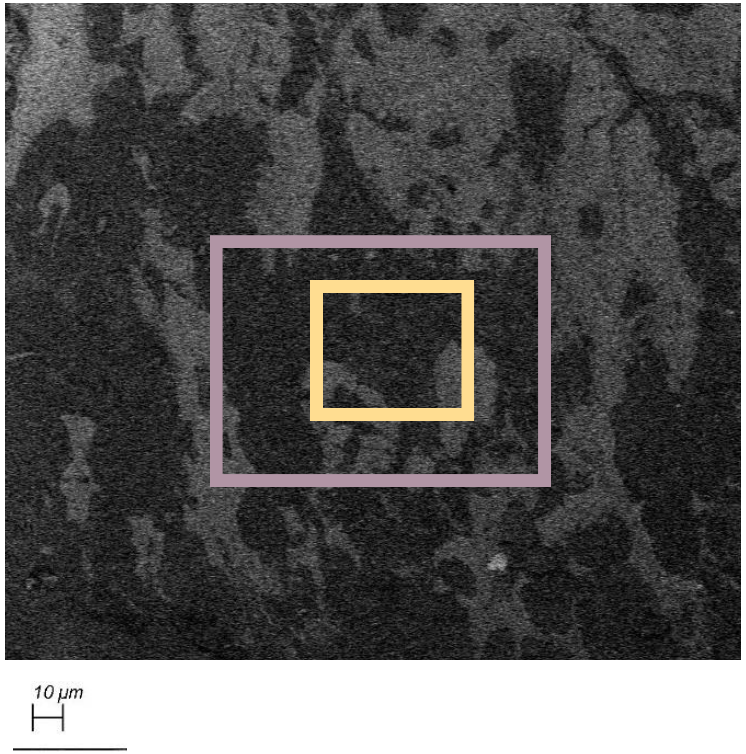


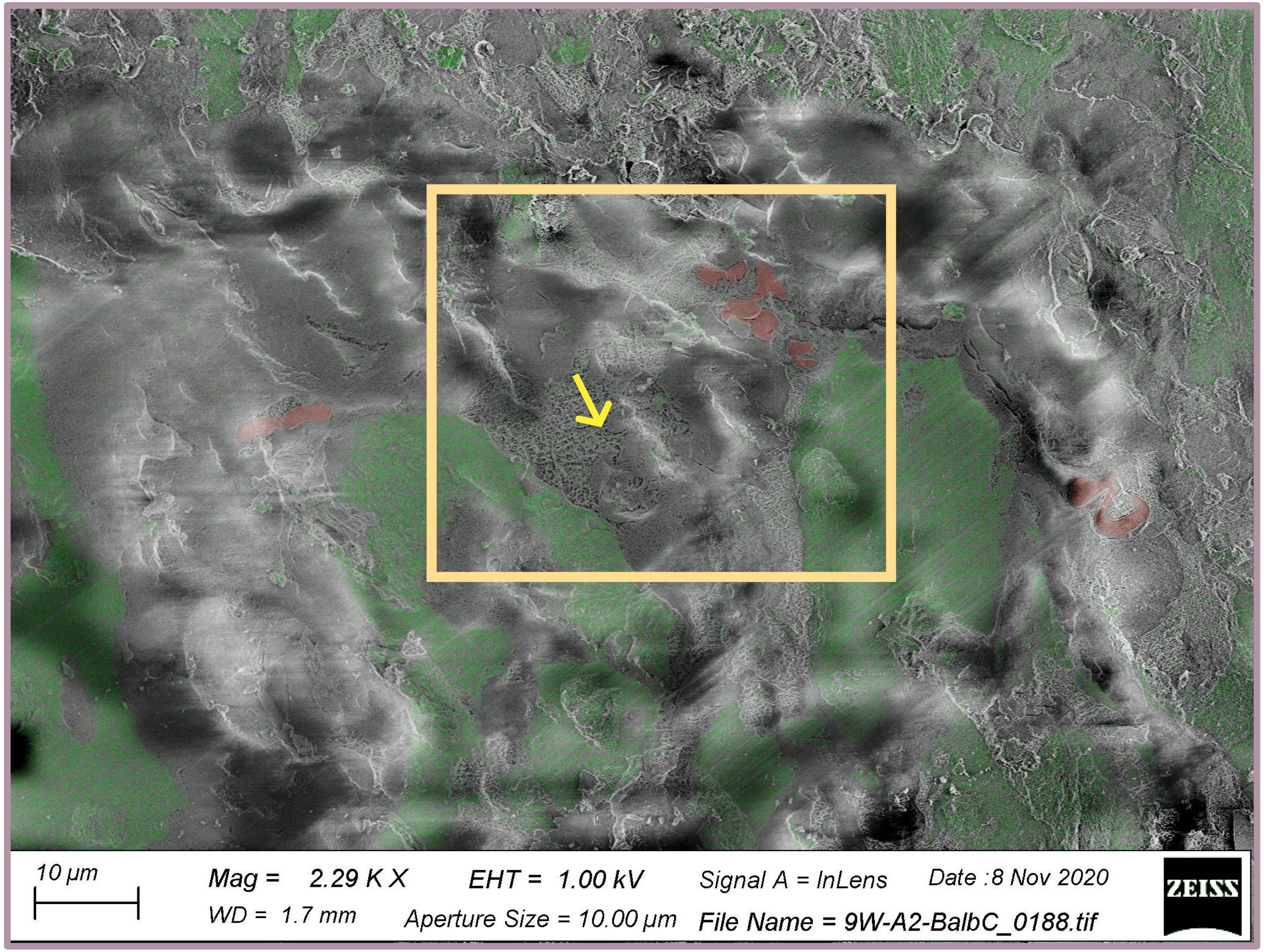
Ca 6kV



Locating Ca and P in a Bone

No need for flat surface, any topogrphy can be measured





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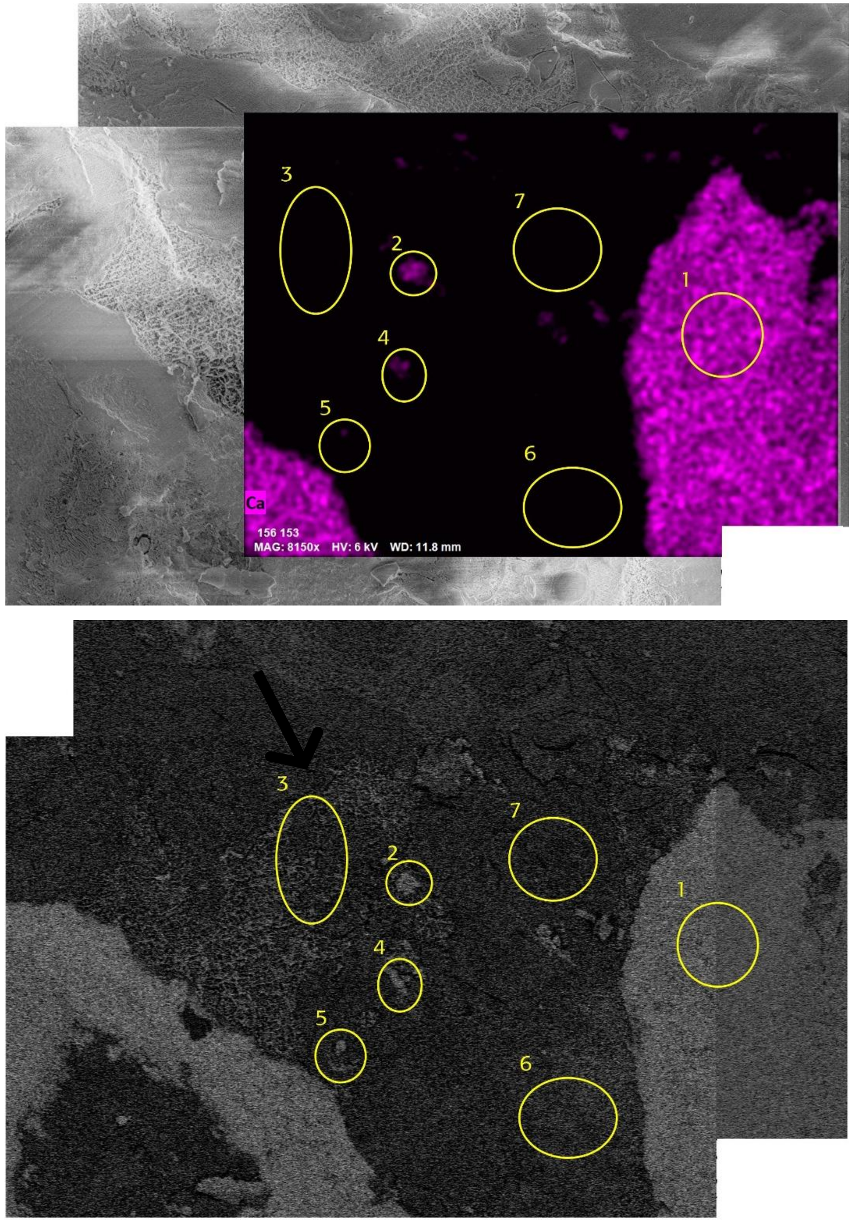
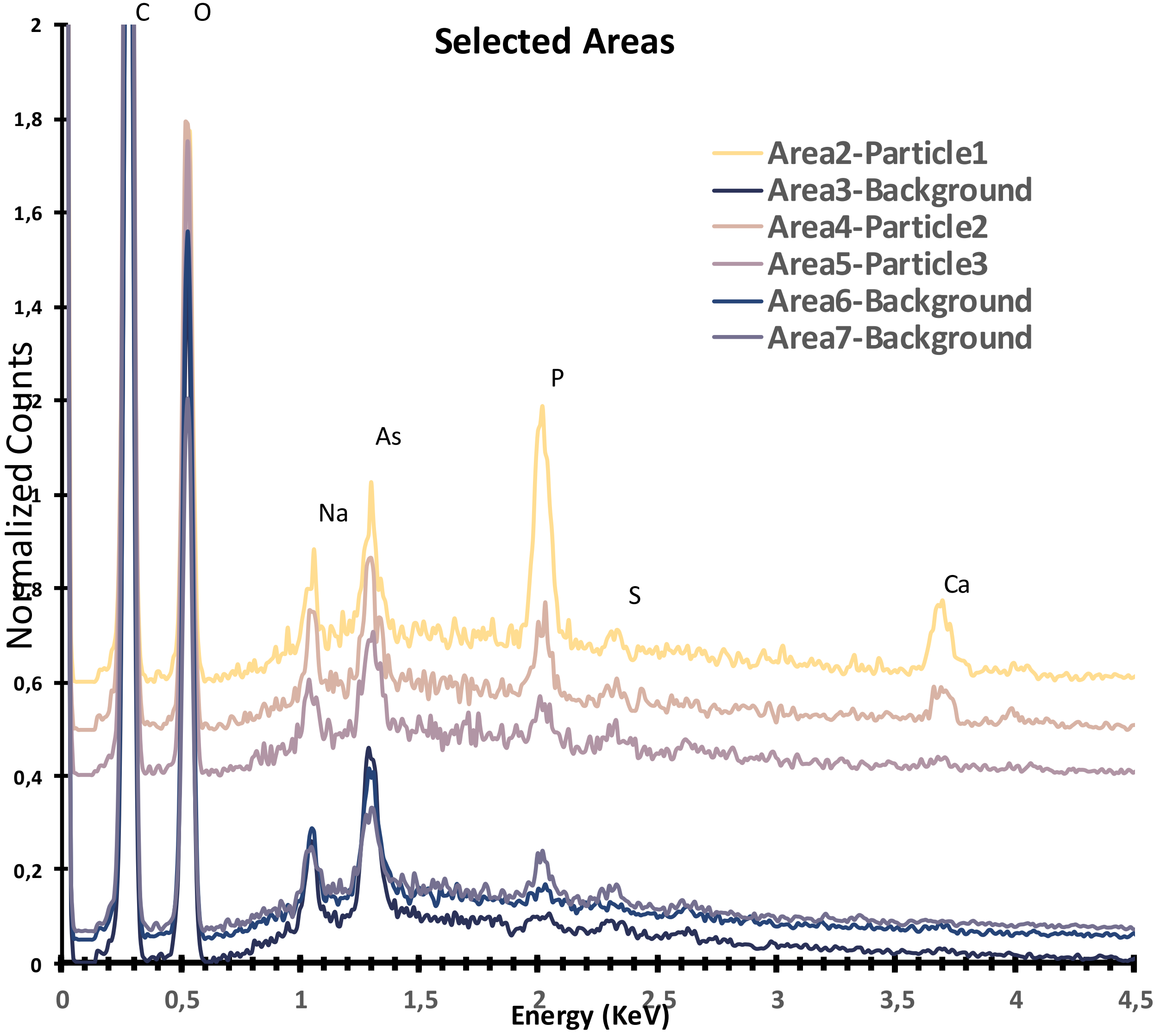
Locating Ca and P in a Bone





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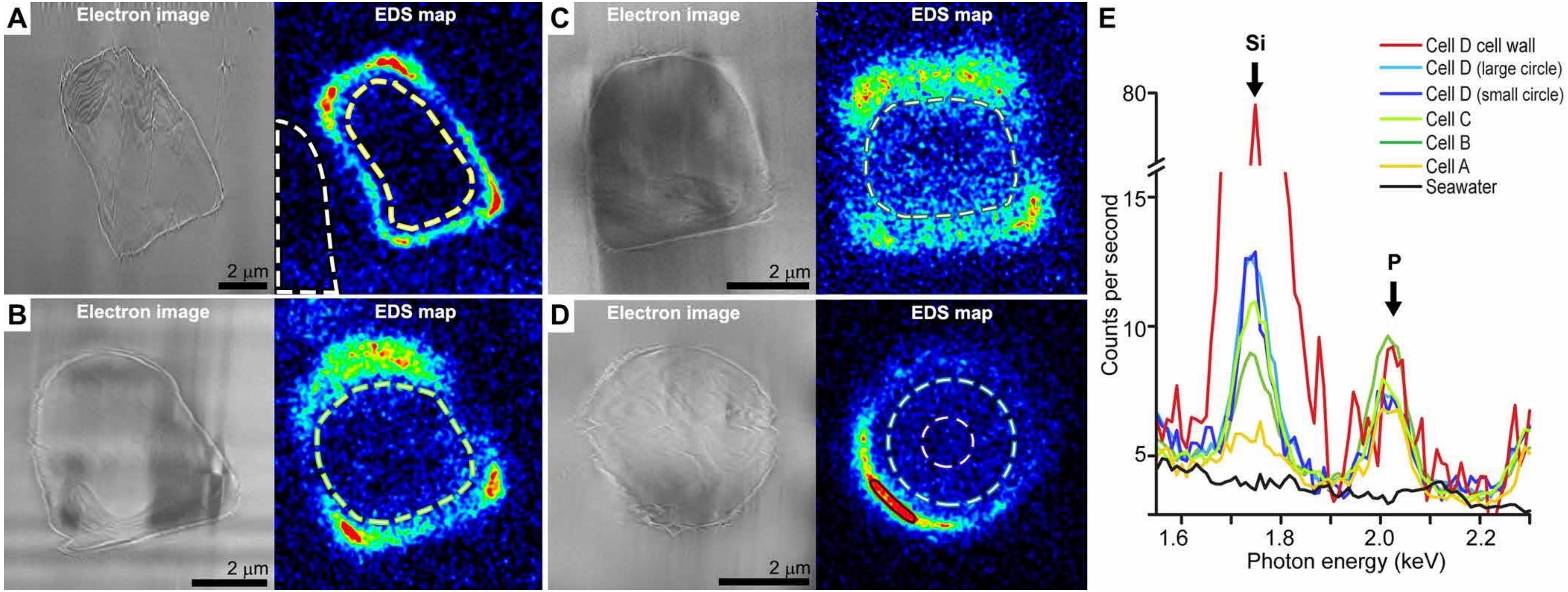
Imaging and quantifying homeostatic levels of intracellular silicon in diatoms

Santosh Kumar et al. Sci. Adv. 2020; 6 : eaaz7554



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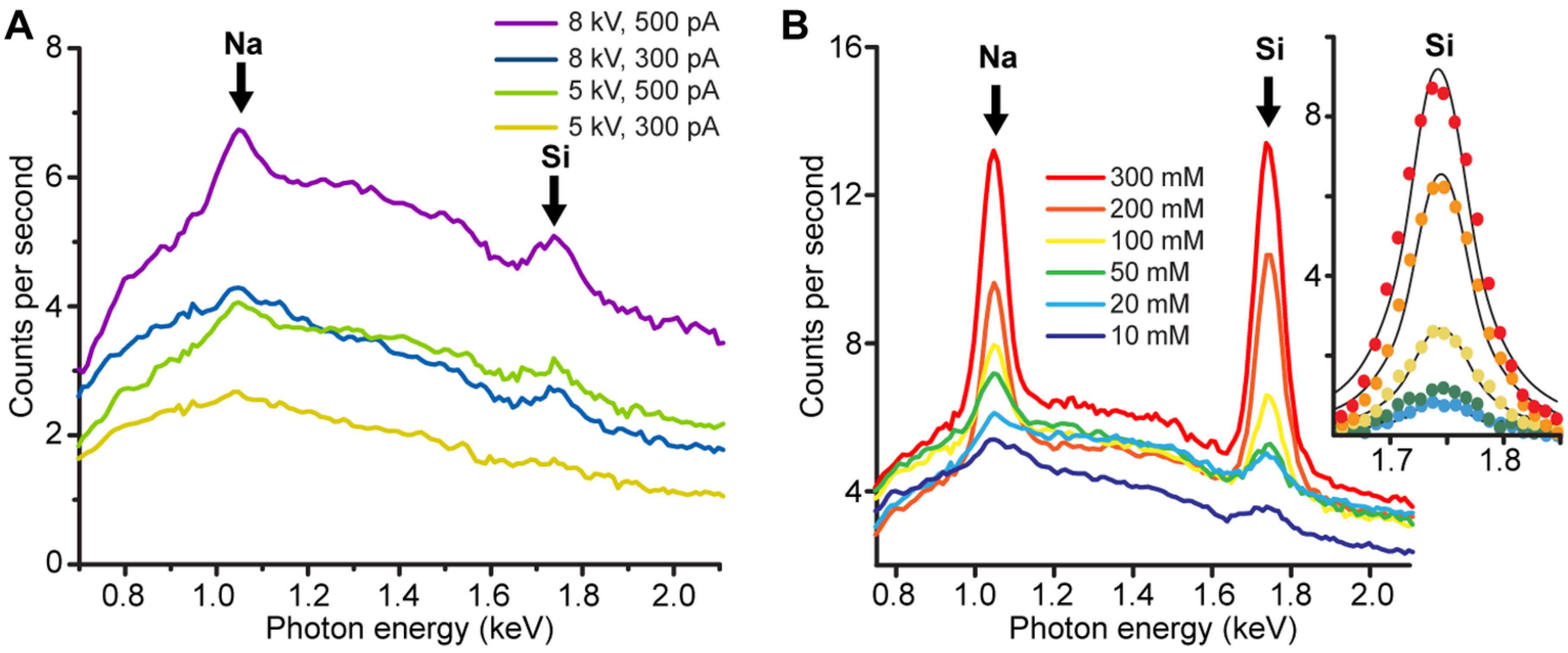
Imaging and quantifying homeostatic levels of intracellular silicon in diatoms

Santosh Kumar et al. Sci. Adv. 2020; 6 : eaaz7554



Dr. Santosh Kumar, Dr. Assaf Gal group, Structural Biology Dept., WIS

Summary

EDS is an analytic technique that can be applied in different fields, from nanomaterials to biomaterials

By using the new EDS technology, the Bruker FlatQuad we are able to break some limits of lateral resolution.

Cryo EDS is a promising but challenging field that still requires the development of better workflows

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Nano GmbH, Berlin
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Are There Any Questions?

Please type in the questions you might have in the Q&A box and press *Send*.

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