EDS Of Semiconductor Lamellae in SEM (T-SEM) and STEM
October 8th, 2020

Max Patzschke, Meiken Falke and Purvesh Soni

EDS Of Semiconductor Lamellae in SEM (T-SEM) and STEM; Quantitative Element Mapping and More

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Bruker Nano Analytics

Host

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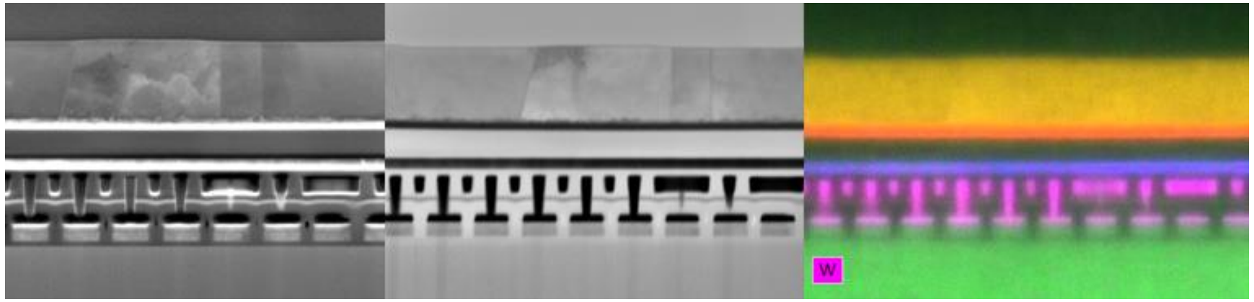
Global Product Manager EDS/TEM,
Bruker Nano Analytics

Speaker

Purvesh Soni
Application Scientist,
Bruker Nano Analytics

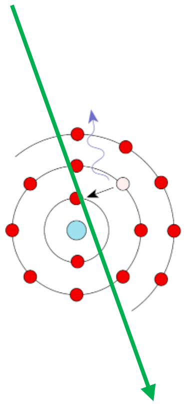
Speaker

Overview



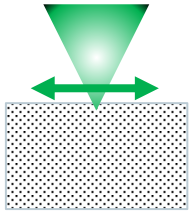
- Intro: Microscopy of bulk and e-transparent specimens
  - Quantitative EDS for lamellae in SEM (FIB) > “T-SEM” and TEM/STEM
- Specimen preparation
- Optimum specimen-detector geometries in SEM

- Examples of quantitative EDS analysis of semiconductor lamellae
  - in SEM (T-SEM)
  - and STEM and
  - in relation to available complementary analysis methods

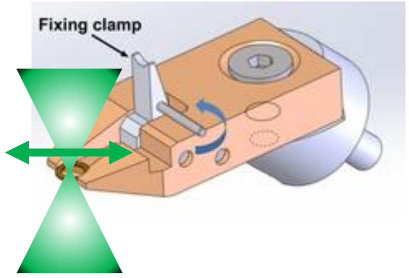


Electron Microscopy

SEM
Scanning EM

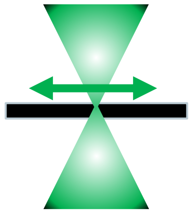


SEM: „T-SEM“

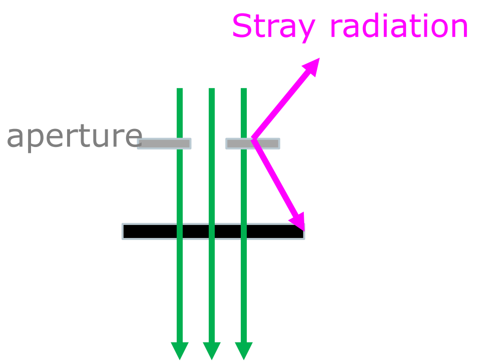


- TKD holder, patented
- Commercial STEM holders and detectors
- Home made versions

STEM
Scanning TEM

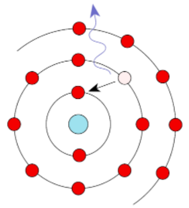


TEM
Parallel illumination



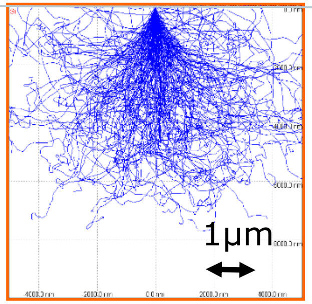
Spatial Resolution for Bulk and Electron Transparent Specimens

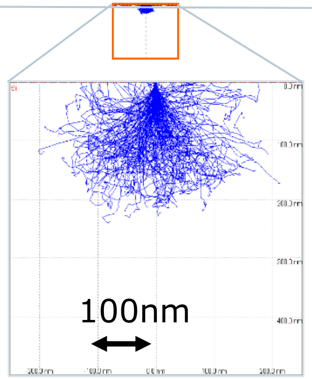
SEM: bulk



High voltage
30kV

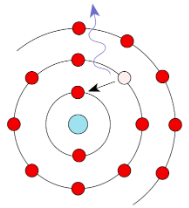
Lower voltage
4kV





Spatial Resolution for Bulk and Electron Transparent Specimens

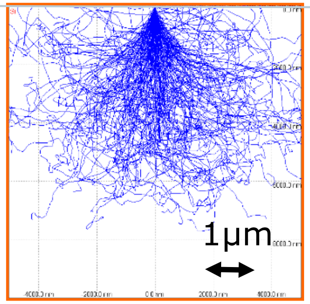
SEM: bulk

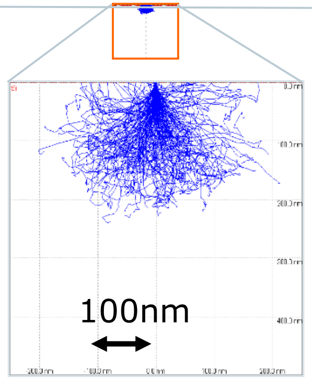


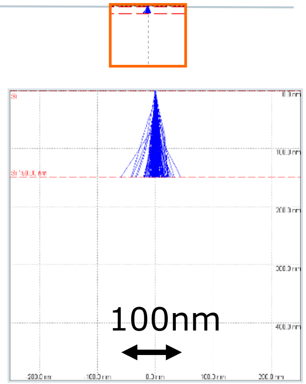
S/TEM, T-SEM (trajectories for 30kV):
thin specimen, small probe

High voltage
30kV

Lower voltage
4kV

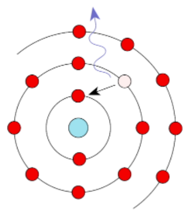






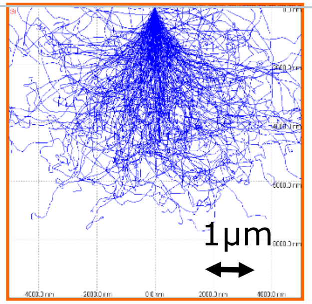
Spatial Resolution for Bulk and Electron Transparent Specimens

SEM: bulk

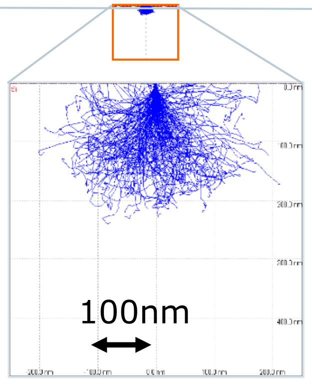


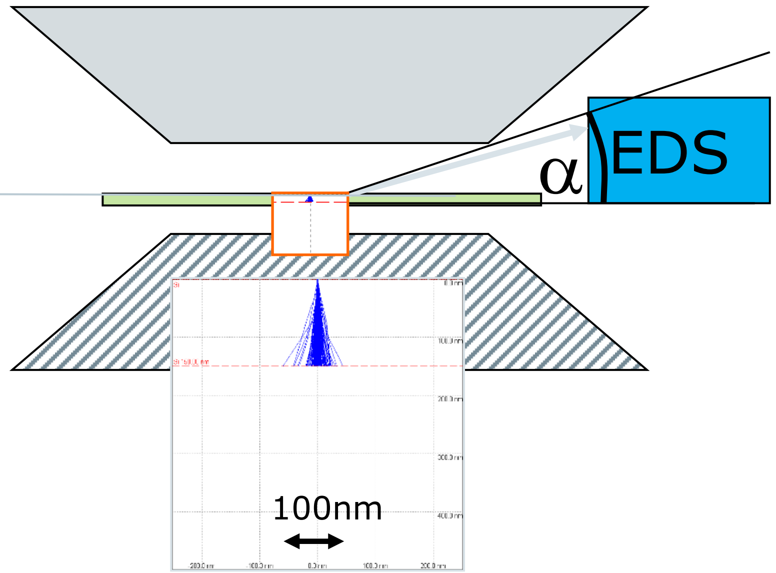
S/TEM, T-SEM (trajectories for 30kV):
thin specimen, small probe

High voltage
30kV



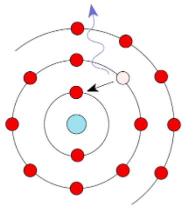
Lower voltage
4kV





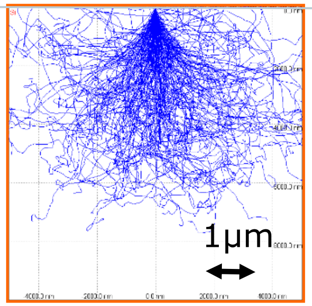
Spatial Resolution for Bulk and Electron Transparent Specimens

SEM: bulk

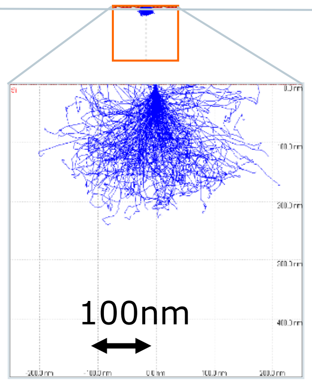


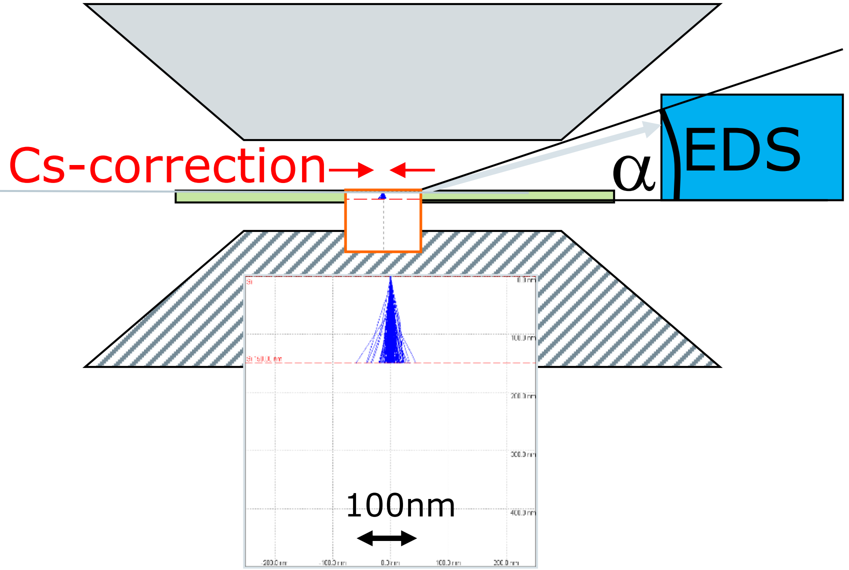
S/TEM, T-SEM (trajectories for 30kV):
thin specimen, small probe

High voltage
30kV



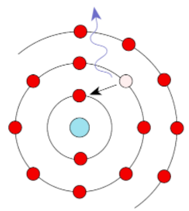
Lower voltage
4kV



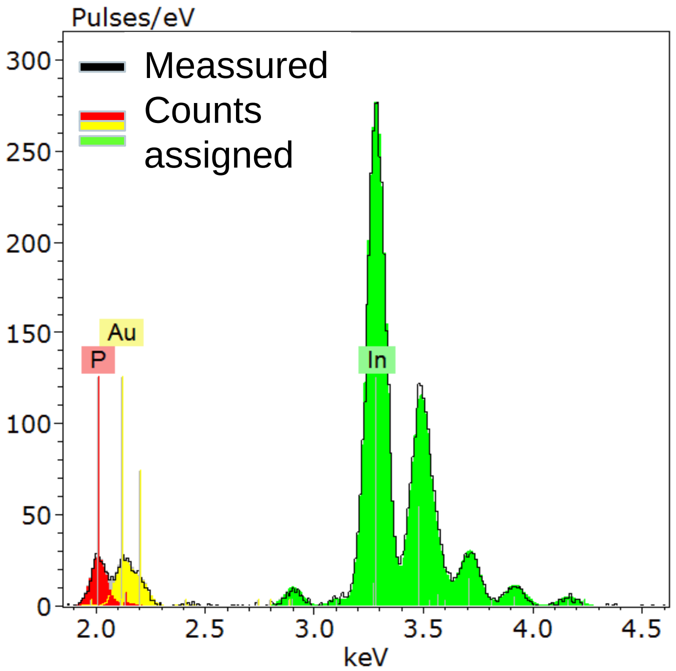


(TEM) EDS Quantification; R. Egerton 1994,

line intensity for a particular element line / transition:



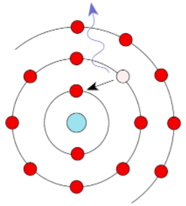
$$I_x = N_A\, \sigma_A\, \omega_A\, (\Omega/4\pi)\, \varepsilon\, N_e = n_A\, t\, \sigma_A\, \omega_A\, (\Omega/4\pi)\, \varepsilon\, N_e$$



I_x	number of X-ray photons in a characteristic peak of species A
N	number of atoms per unit volume
$n\, t$	density times thickness
σ	ionization cross section (Casnati et al., 1982, Bote et al., 2009)
ω	fluorescence yield (Hubbell et al., 1994, Krause, 1979)
$\Omega/4\pi$	solid angle / geometrical collection efficiency
ε	detection quantum efficiency (window: SLEW or no window or other)
N_e	number of incident electrons

+ absorption, fluorescence, other effects…

(TEM) EDS Quantification;
The Cliff-Lorimer Method



$$I_x = N_A\, \sigma_A\, \omega_A\, (\Omega/4\pi)\, \varepsilon\, N_e = n_A\, t\, \sigma_A\, \omega_A\, (\Omega/4\pi)\, \varepsilon\, N_e$$

Cliff and Lorimer: $\frac{I_A}{I_B} = \frac{C_A}{k_{AB}\, C_B}$; k_{AB} can be determined experimentally **or theoretically**

I_x	number of X-ray photons in a characteristic peak of species A
N	number of atoms per unit volume
$n\,t$	density times thickness
σ	ionization cross section (Casnati et al., 1982, Bote et al., 2009)
ω	fluorescence yield (Hubbell et al., 1994, Krause, 1979)
$\Omega/4\pi$	solid angle / geometrical collection efficiency
ε	detection quantum efficiency (window: SLEW or no window or other)
N_e	number of incident electrons

+ absorption, fluorescence, other effects…

(TEM) EDS Quantification;
The Zeta-Factor Method … and EELS

Bulk, thick samples:
Zeta-Factor Method,
needs Standard!

$$I_x = N_A \sigma_A \omega_A (\Omega/4\pi)\, \varepsilon\, N_e = n_A\, t\, \sigma_A \omega_A (\Omega/4\pi)\, \varepsilon\, N_e$$

G. Kothleitner, Micr. Microanal. 2014
M. Watanabe, J. of Micr. 2005:
For a **standard with known density *ρ*** **and known thickness *t*** ***ζ*** can be determined: ***D_e* (total electron dose) must be known for all measurements.**

EELS: $t/\lambda = \log_e (I^E_{total} / I^E_0)$

$$\rho\, t = \zeta_A \frac{I_A A_A}{C_A D_e} \ldots = \frac{I^E_{core}}{I^E_{0\_low}\, \sigma} \frac{AtWeight}{N_{Avog}}$$

Then, for a sample $C_{A,}$ $C_{B\ \ldots}$ and ρt are unknown with:

$$C_A + C_B = 1 \qquad \rho\, t = \frac{\zeta_A I_A + \zeta_B I_B}{D_e}$$

$$(C_1 + C_2 + \ldots = 1)$$

A_A A_B absorption coefficients

$$C_A = \frac{\zeta_A I_A}{\zeta_A I_A + \zeta_B I_B}$$

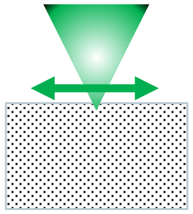
$$C_B = \ldots$$

With a few ***ζ*** data points all ***ζ*x** can be calculated from existing ***k***-factors

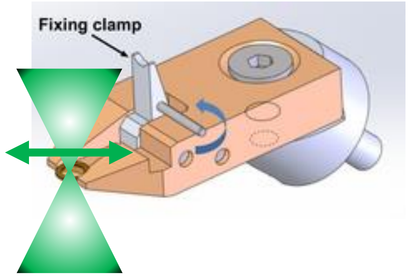
by just one click in the Esprit SW !!!

Electron Microscopy

SEM
Scanning EM

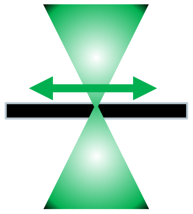


SEM: „T-SEM“

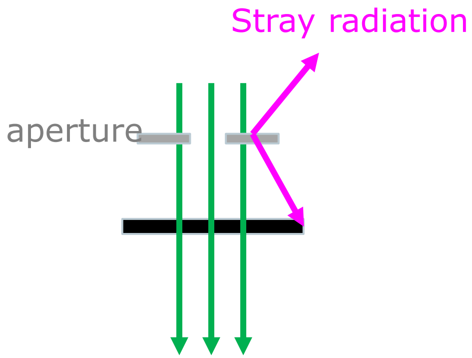


- TKD holder, patented
- Commercial STEM holders
- Home made versions

STEM
Scanning TEM



TEM
Parallel illumination

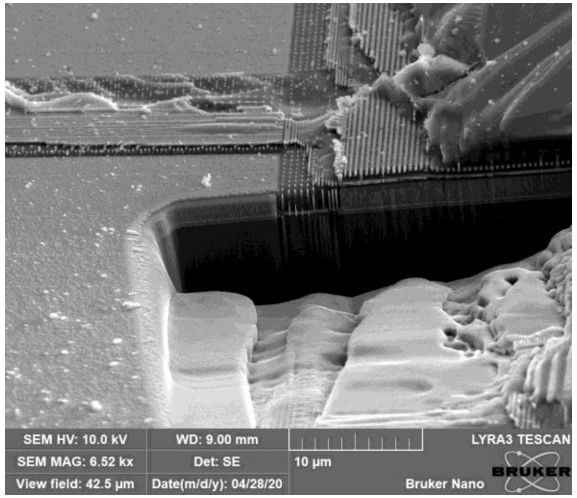


Application examples: FIB/TEM lamellae

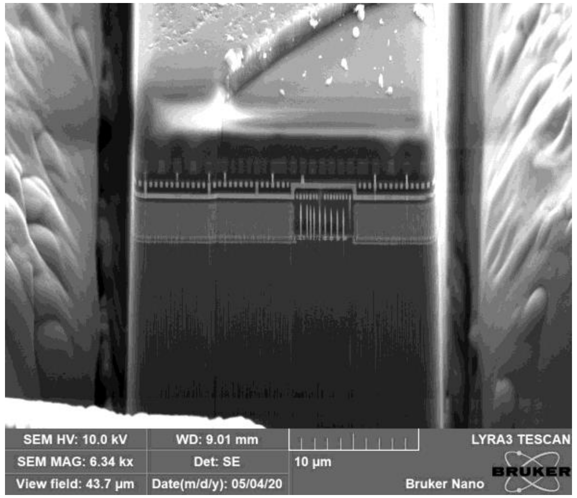
Lift out from a bulk sample

Application examples: FIB/TEM lamellae

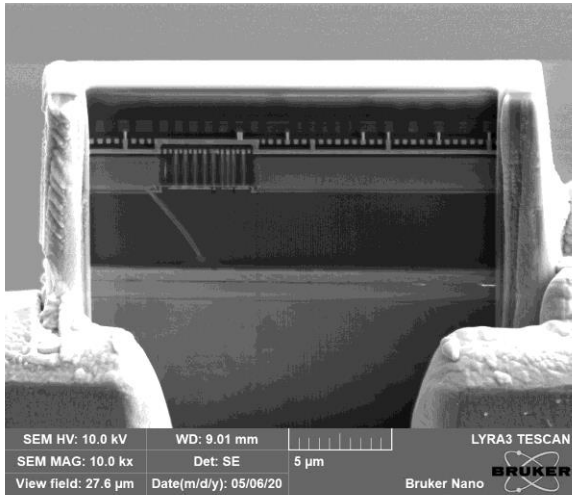
Lift out and thinning



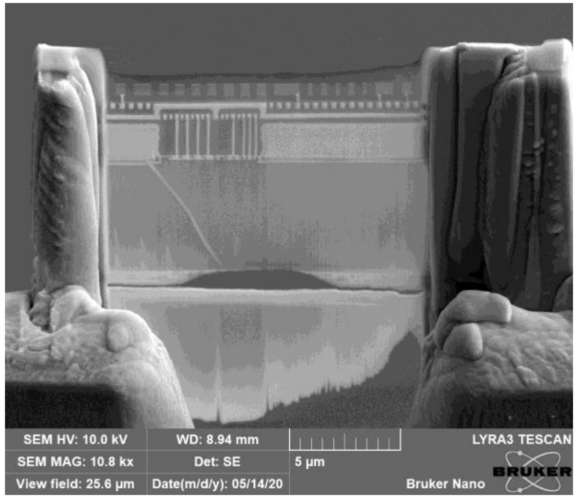
Locating ROI



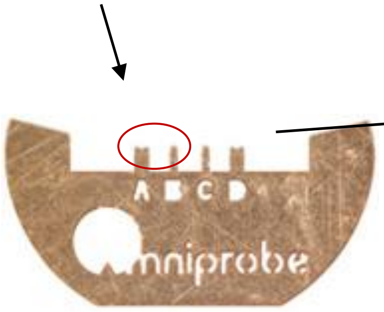
Trenches milling



Coarse thinning
(2µm to 200 nm)



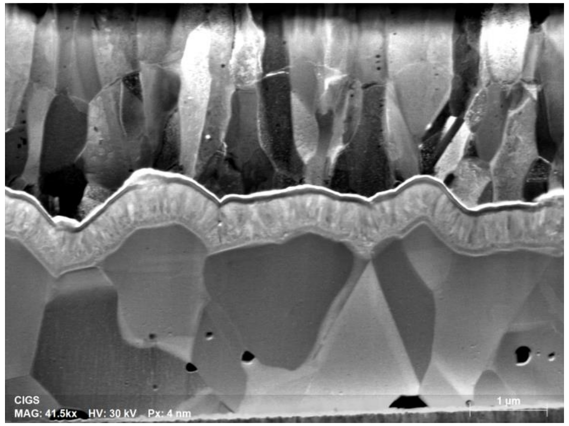
Final thinning /
Low kV milling
(50 nm)



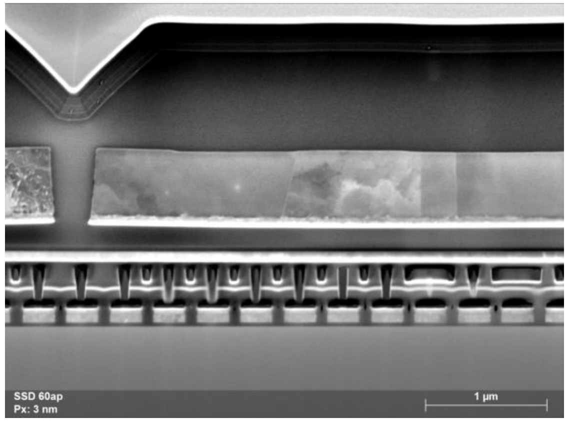
Lift out on grid

Application examples overview

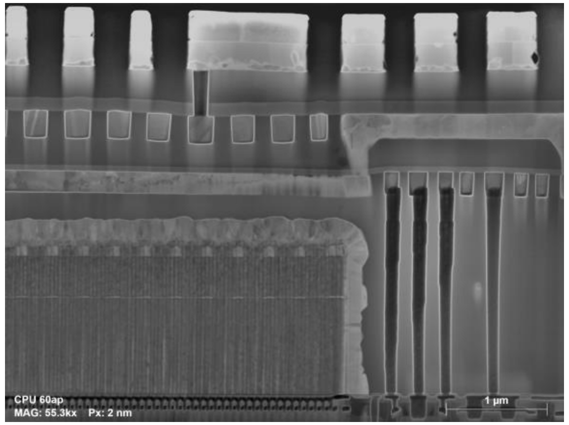
STEM images



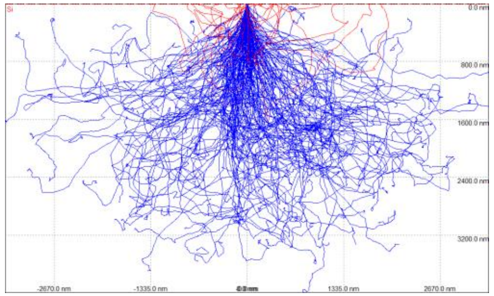
CIGS solar cell



Solid state drive



Microprocessor

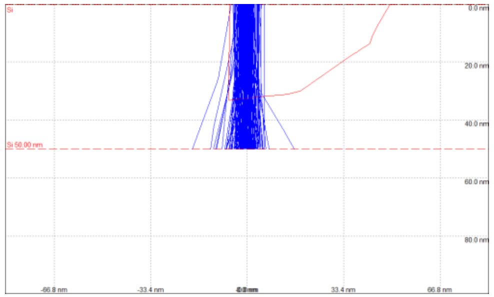


0 nm

2000 nm

4000 nm

Bulk specimen



0 nm

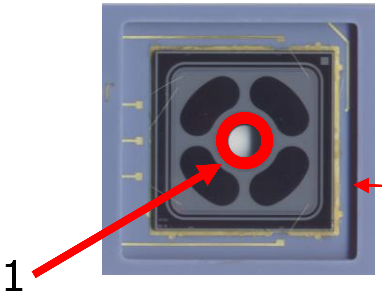
50 nm

100 nm

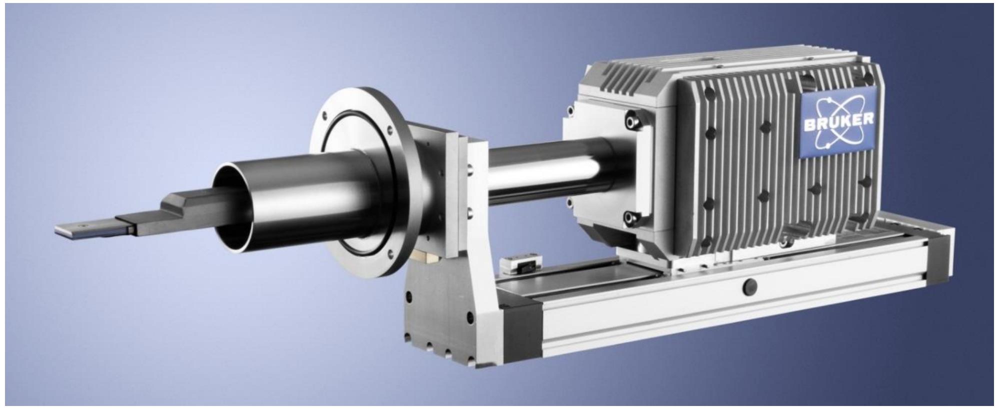
FIB/TEM lamellae

XFlash® FlatQUAD

Features



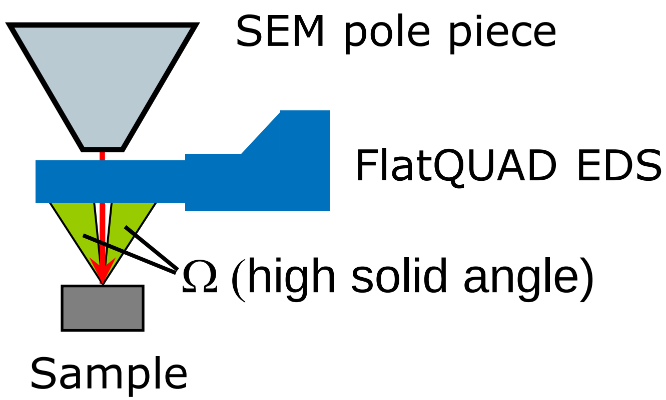
- Side entry, energy dispersive x-ray spectra detector
- Annular design; Central aperture for primary beam[1]
- 4 × SDD modules (total area 60 mm²)



Advantages:

- Optimum signal collection geometry
- High sensitivity, high signal – low noise
- Large solid angle (up to 1.1 sr)
- Shadowing minimized for topographic samples
- High count rate at low beam currents

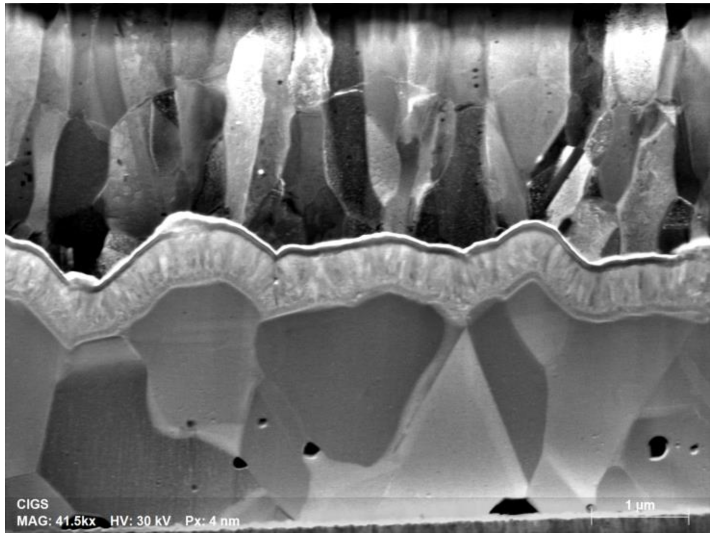
XFlash® FlatQUAD
Features and Advantages



Ideal for samples with low x-ray/EDS yield:

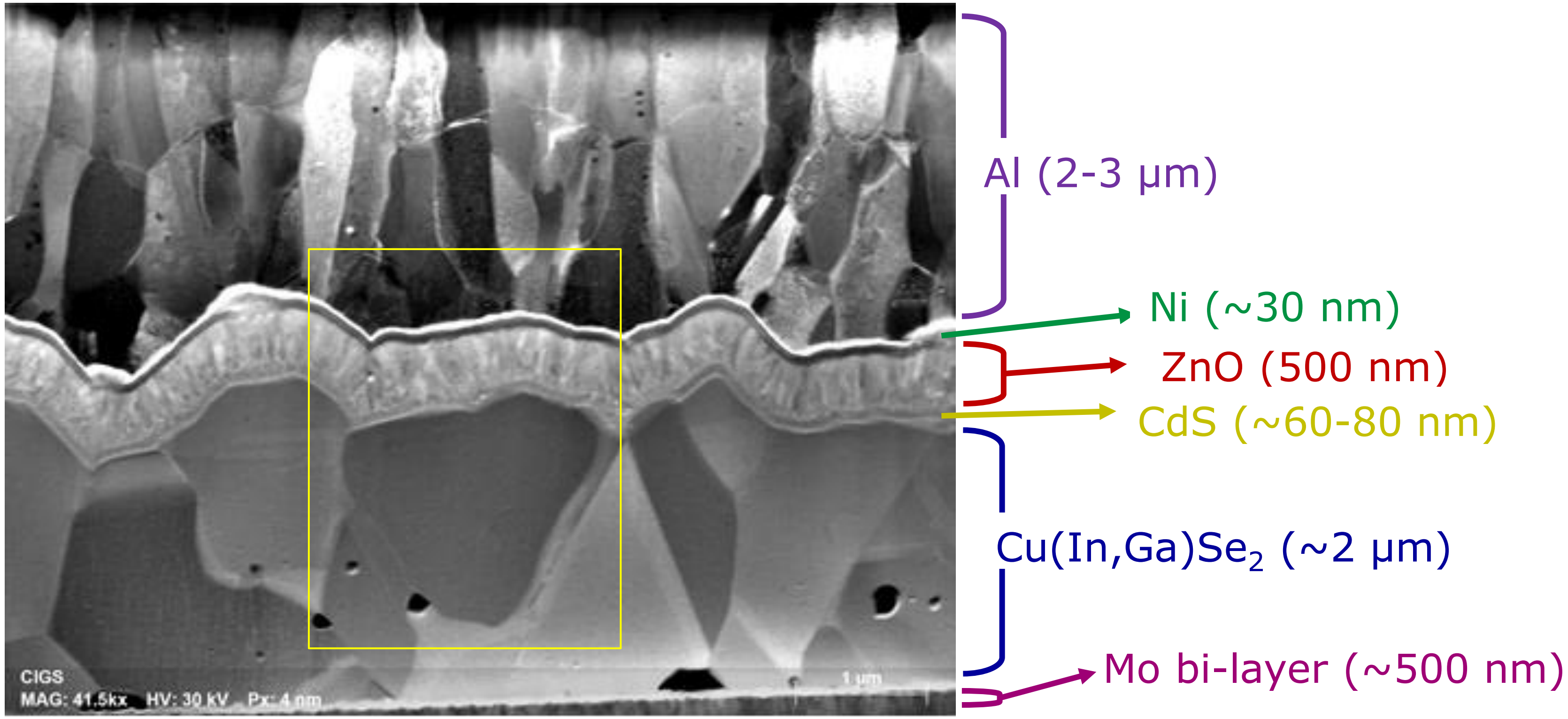
- TEM/FIB lamellae
- Thin films
- Nanoparticles
- low Z (light elements)
- Ultra-sensitive for bulk samples

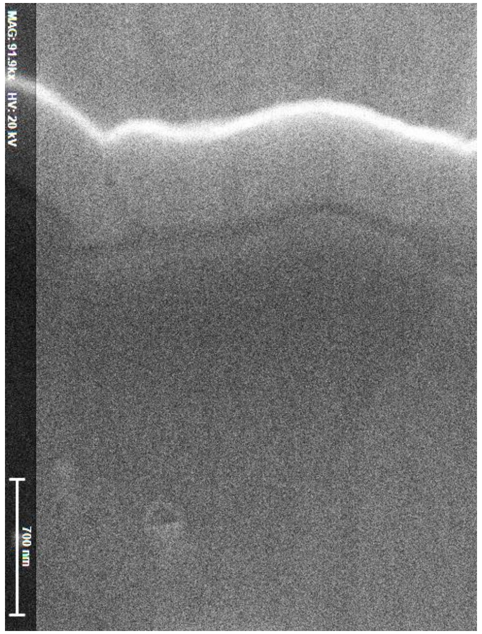
Application example: CIGS



Application example: $Cu(In,Ga)Se_2$ solar cell

STEM DF – Layered structure (FIB-Lamella)





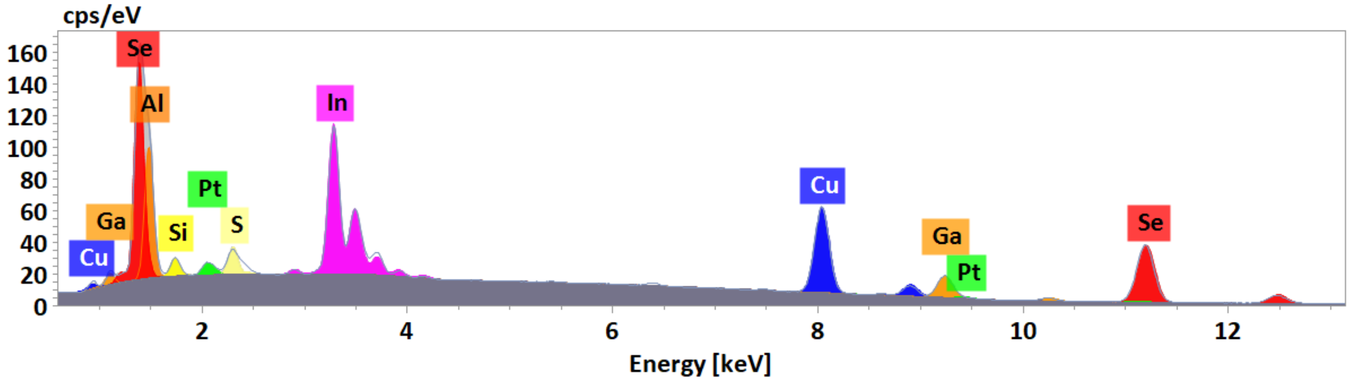
EDS mapping region

Lamella thickness ~ 70 nm

Sample courtesy: M. Raghuwanshi, RWTH Aachen University, Aachen, Germany

Application example: $Cu(In,Ga)Se_2$ solar cell

Hypermap and Quantification results

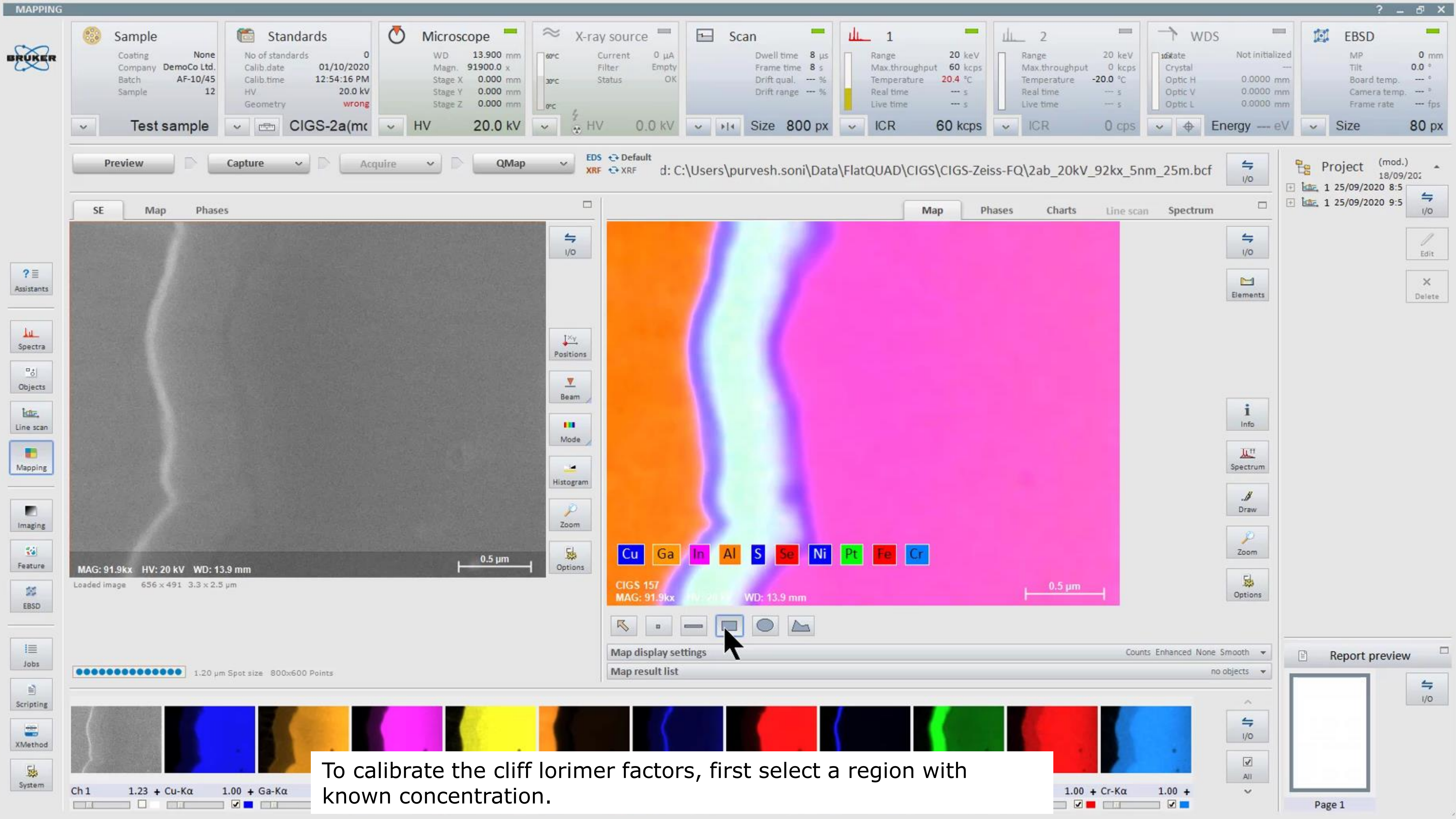


Cliff-Lorimer quantification

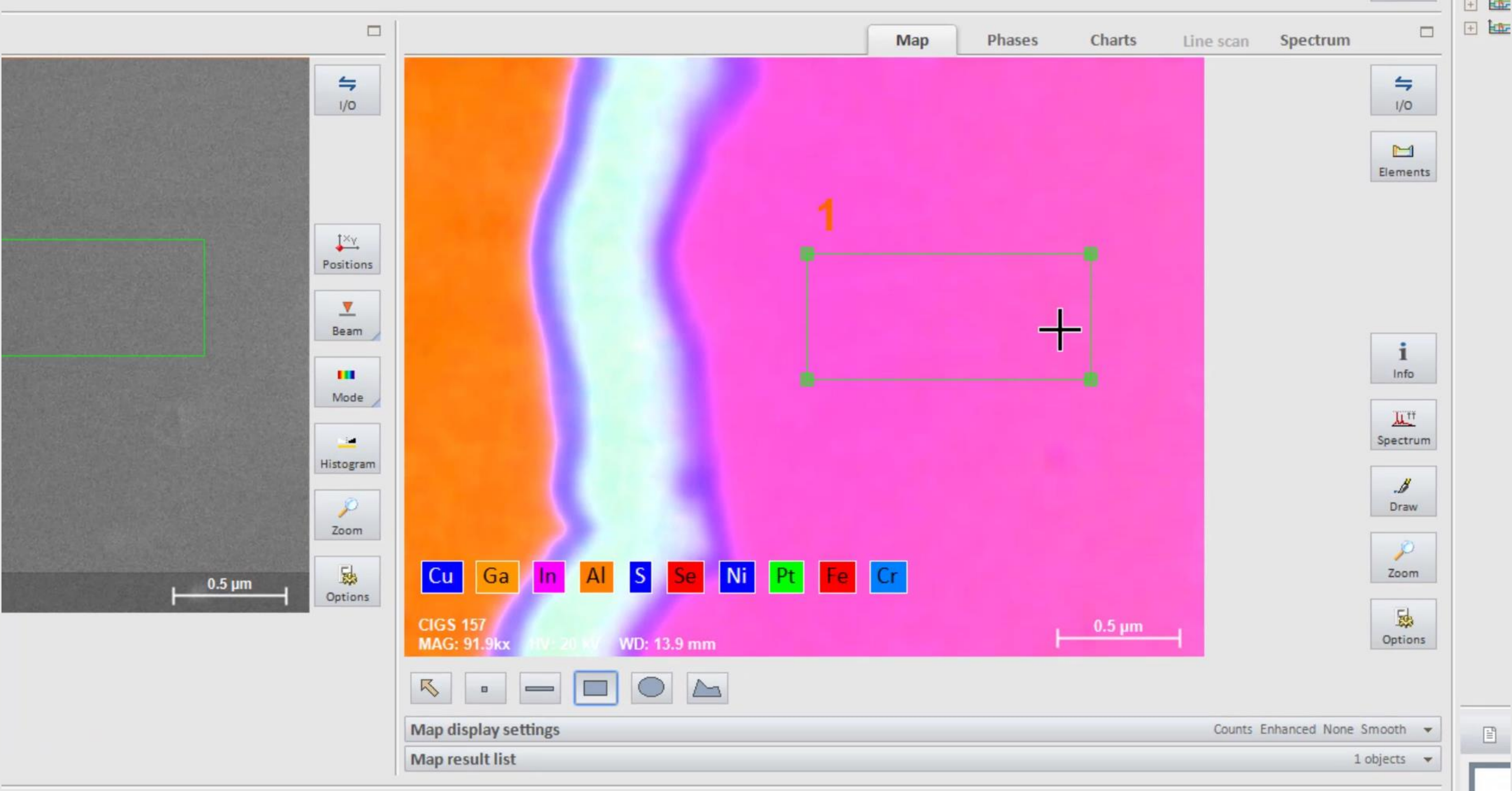
Element	Atomic No.	Line series	Net counts	Atom abs. [%]	Error (1-sigma %)
Copper	29	K	8.91E+05	21.71	0.51
Indium	49	L	1.72E+06	22.52	3.2
Gallium	31	K	2.39E+05	6.39	0.17
Selenium	34	K	7.04E+05	44.75	0.18
Sulfur	16	K	1.81E+05	4.63	0.06
Sum				**100**	

Cliff Lorimer Quantification method

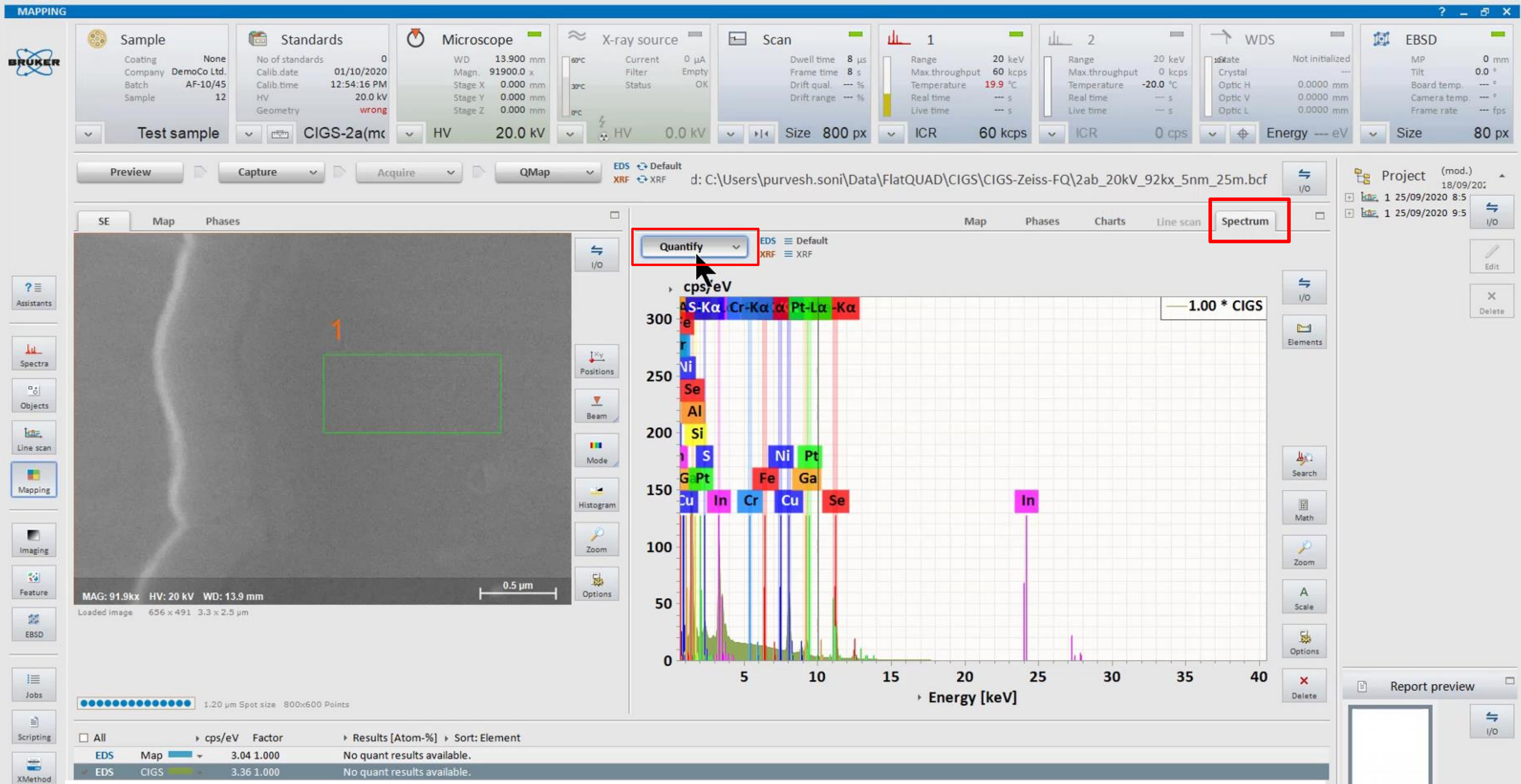
for e-transparent samples



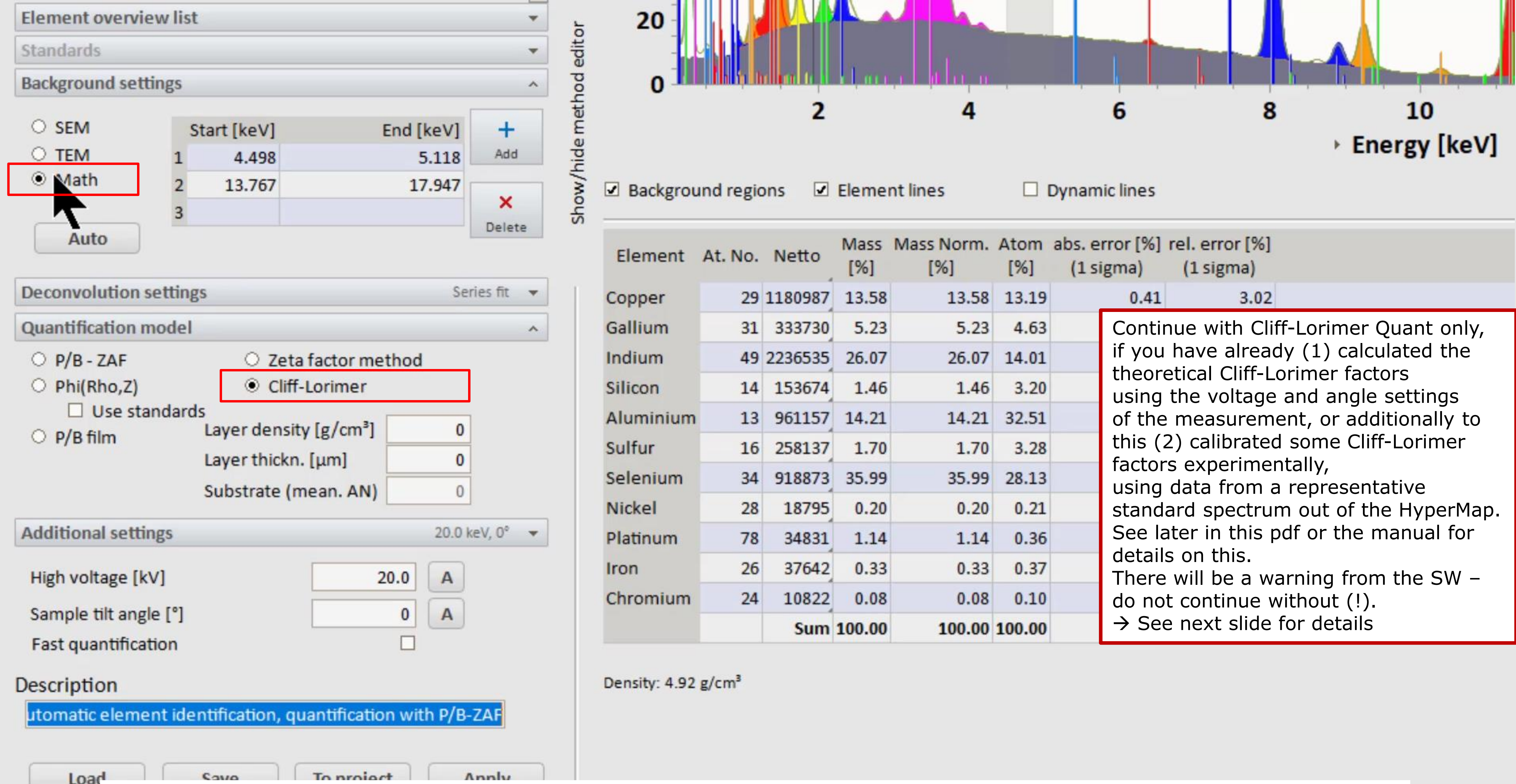
To calibrate the cliff lorimer factors, first select a region with known concentration.



This region is selected since the concentration of CIGS layer is known



Go to spectrum tab, select the spectrum from below, and click Quantify. You can also save it with the name "spectrum.spx" from the I/O button under the spectrum tab.

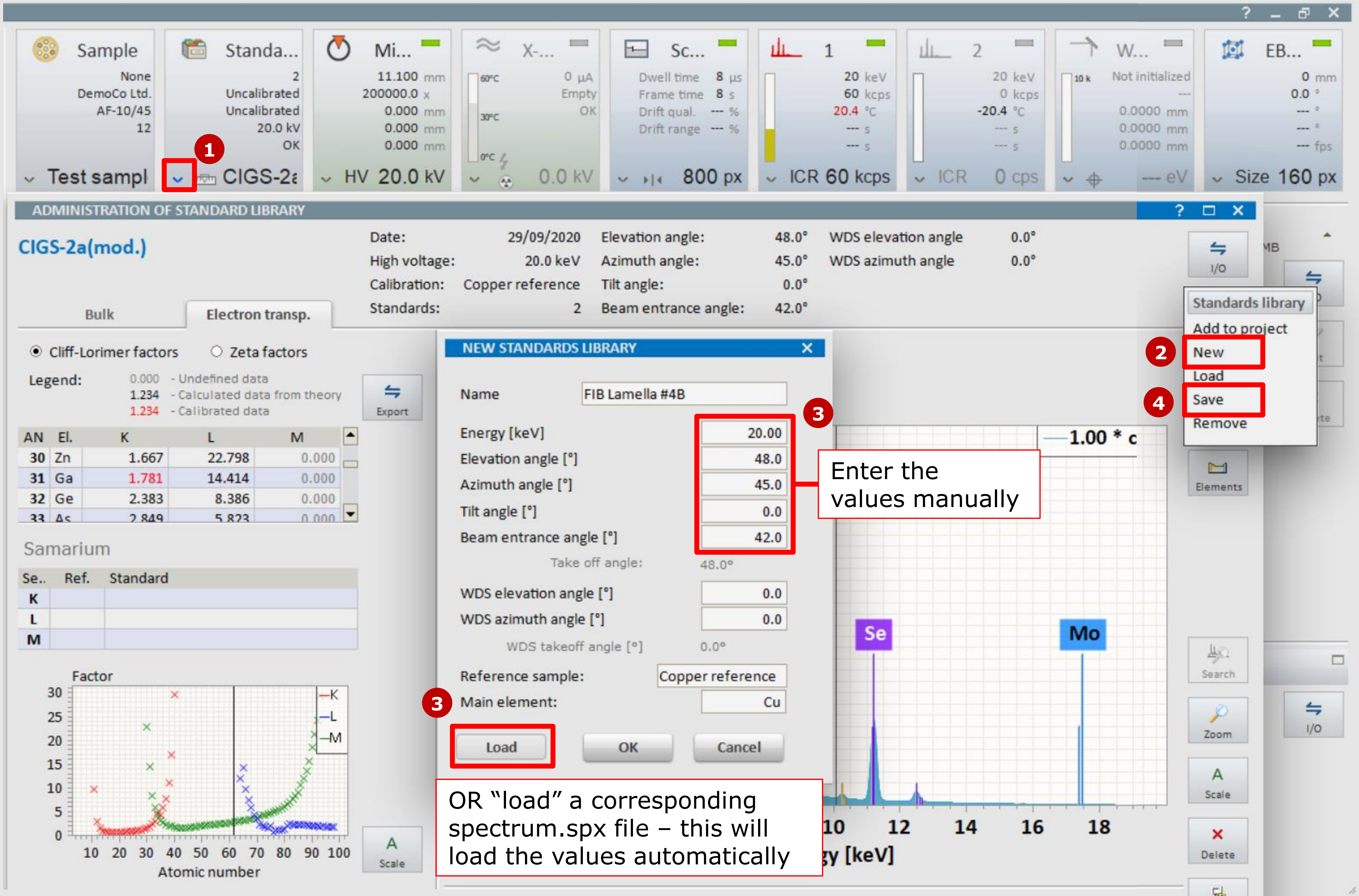


	Start [keV]	End [keV]
1	4.498	5.118
2	13.767	17.947
3		

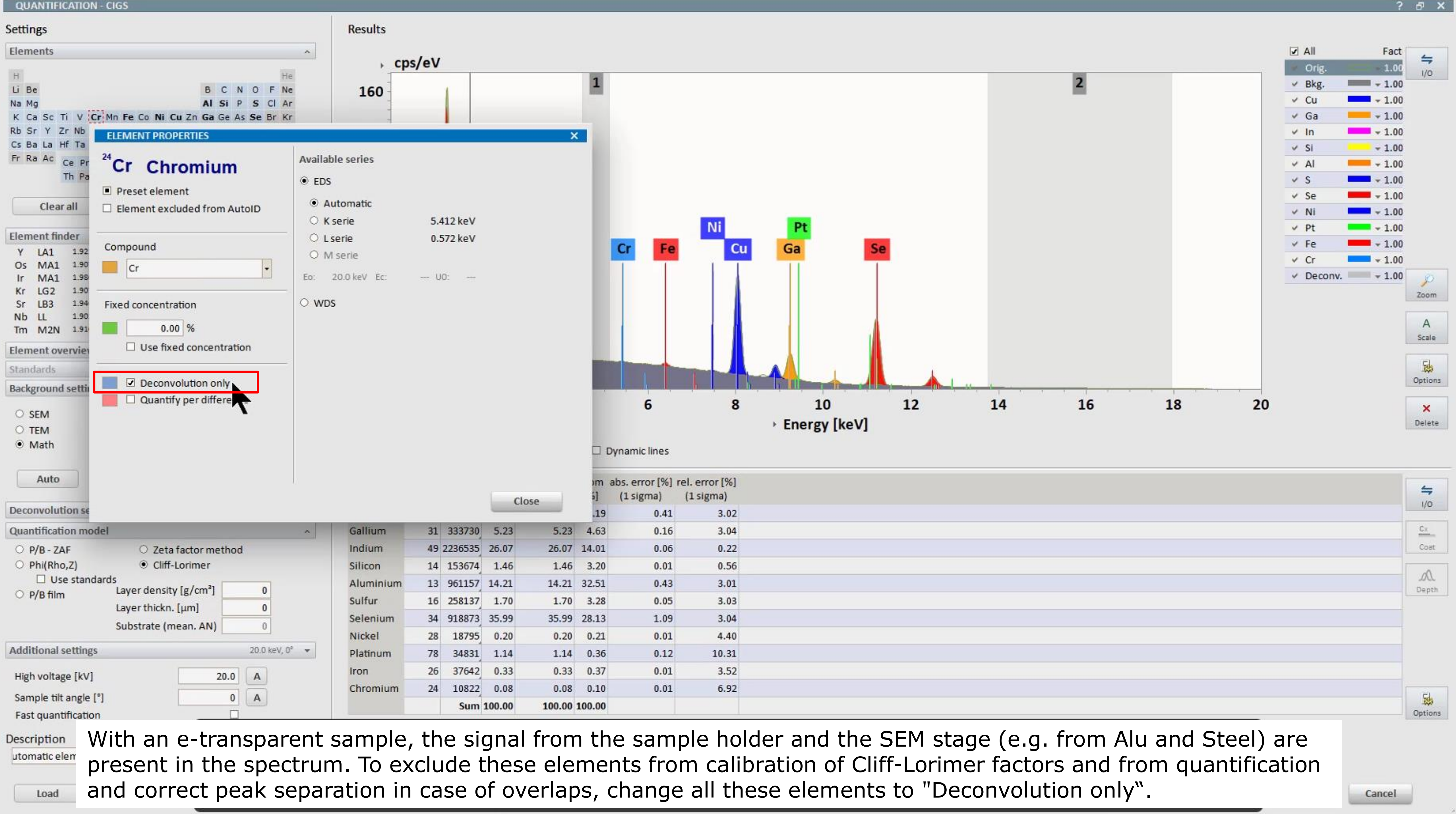
Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Copper	29	1180987	13.58	13.58	13.19	0.41	3.02
Gallium	31	333730	5.23	5.23	4.63		
Indium	49	2236535	26.07	26.07	14.01		
Silicon	14	153674	1.46	1.46	3.20		
Aluminium	13	961157	14.21	14.21	32.51		
Sulfur	16	258137	1.70	1.70	3.28		
Selenium	34	918873	35.99	35.99	28.13		
Nickel	28	18795	0.20	0.20	0.21		
Platinum	78	34831	1.14	1.14	0.36		
Iron	26	37642	0.33	0.33	0.37		
Chromium	24	10822	0.08	0.08	0.10		
		Sum	100.00	100.00	100.00		

Continue with Cliff-Lorimer Quant only, if you have already (1) calculated the theoretical Cliff-Lorimer factors using the voltage and angle settings of the measurement, or additionally to this (2) calibrated some Cliff-Lorimer factors experimentally, using data from a representative standard spectrum out of the HyperMap. See later in this pdf or the manual for details on this.
There will be a warning from the SW – do not continue without (!).
→ See next slide for details

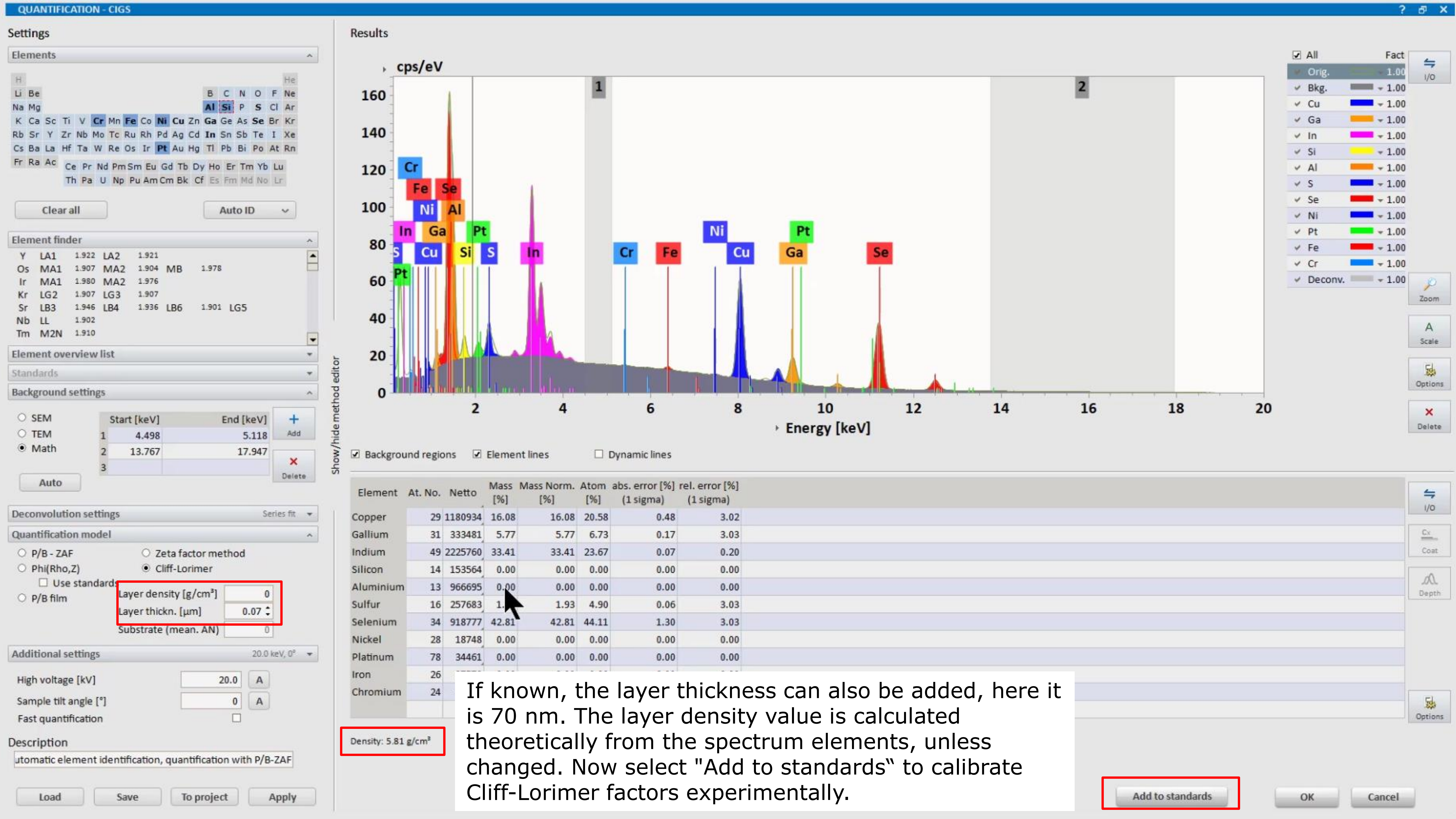
now select Cliff-Lorimer as the quantification model and Mathematical background (the mathematical bg is for samples which are neither bulk nor really thin, so that the physical SEM or TEM Background don’t work perfectly).



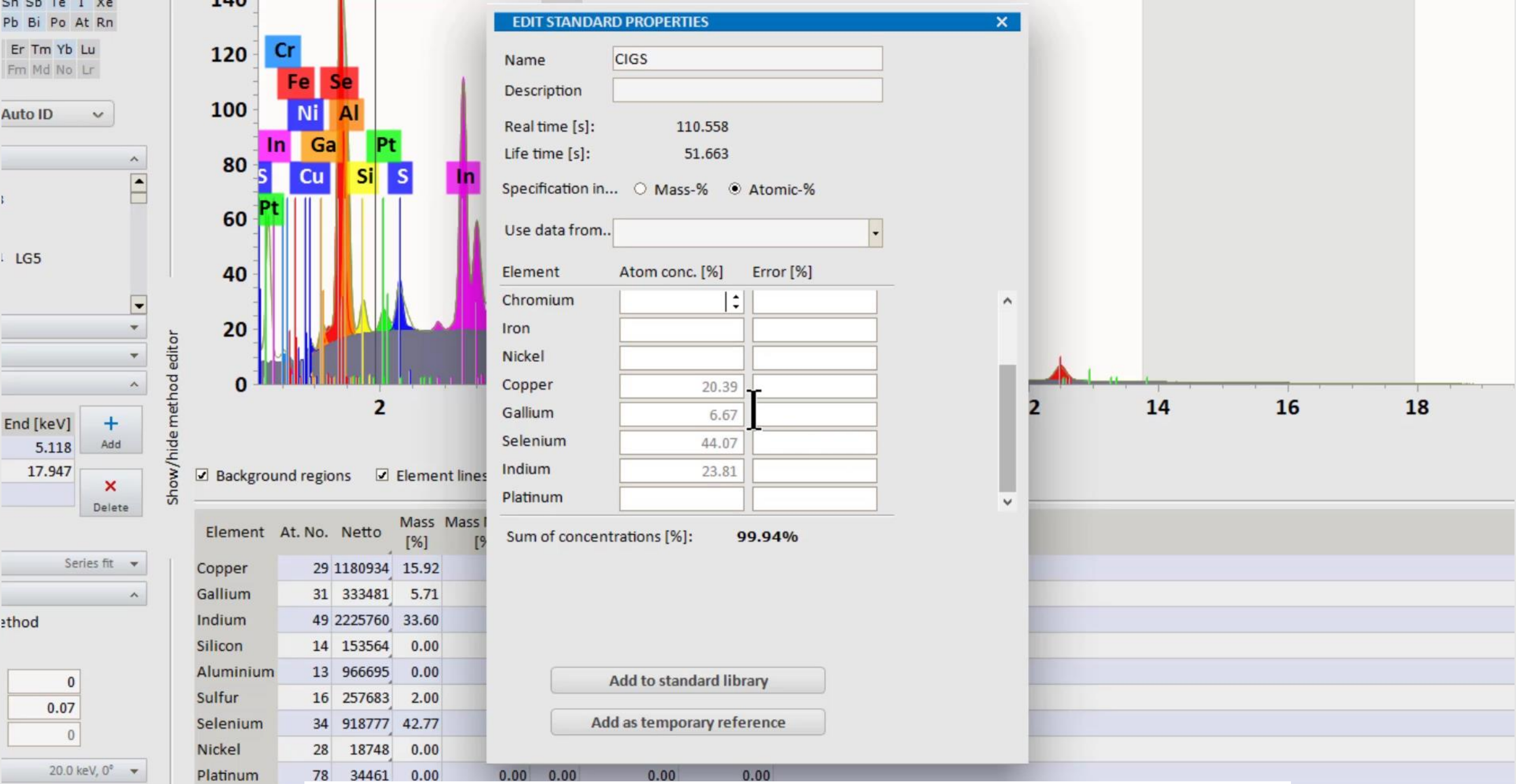
Sample
None
DemoCo Ltd.
AF-10/45
12
Test sampl
Standa...
2
Uncalibrated
Uncalibrated
20.0 kV
OK
CIGS-2a
HV 20.0 kV
Dwell time 8 µs
Frame time 8 s
800 px
ICR 60 kcps
Size 160 px
ADMINISTRATION OF STANDARD LIBRARY
CIGS-2a(mod.)
Date: 29/09/2020
High voltage: 20.0 keV
Calibration: Copper reference
Standards: 2
Elevation angle: 48.0°
Azimuth angle: 45.0°
Tilt angle: 0.0°
Beam entrance angle: 42.0°
WDS elevation angle 0.0°
WDS azimuth angle 0.0°
Bulk
Electron transp.
Cliff-Lorimer factors
Zeta factors
Legend: 0.000 - Undefined data
1.234 - Calculated data from theory
1.234 - Calibrated data
Export
AN El. K L M
30 Zn 1.667 22.798 0.000
31 Ga 1.781 14.414 0.000
32 Ge 2.383 8.386 0.000
Samarium
Se.. Ref. Standard
K
L
M
Factor
Atomic number
Standards library
Add to project
New
Load
Save
Remove
NEW STANDARDS LIBRARY
Name FIB Lamella #4B
Energy [keV] 20.00
Elevation angle [°] 48.0
Azimuth angle [°] 45.0
Tilt angle [°] 0.0
Beam entrance angle [°] 42.0
Take off angle: 48.0°
WDS elevation angle [°] 0.0
WDS azimuth angle [°] 0.0
WDS takeoff angle [°] 0.0°
Reference sample: Copper reference
Main element: Cu
Load
OK
Cancel
Enter the values manually
OR “load” a corresponding spectrum.spx file – this will load the values automatically
Se
Mo
Elements
Search
Zoom
Scale
Delete
1
2
3
4



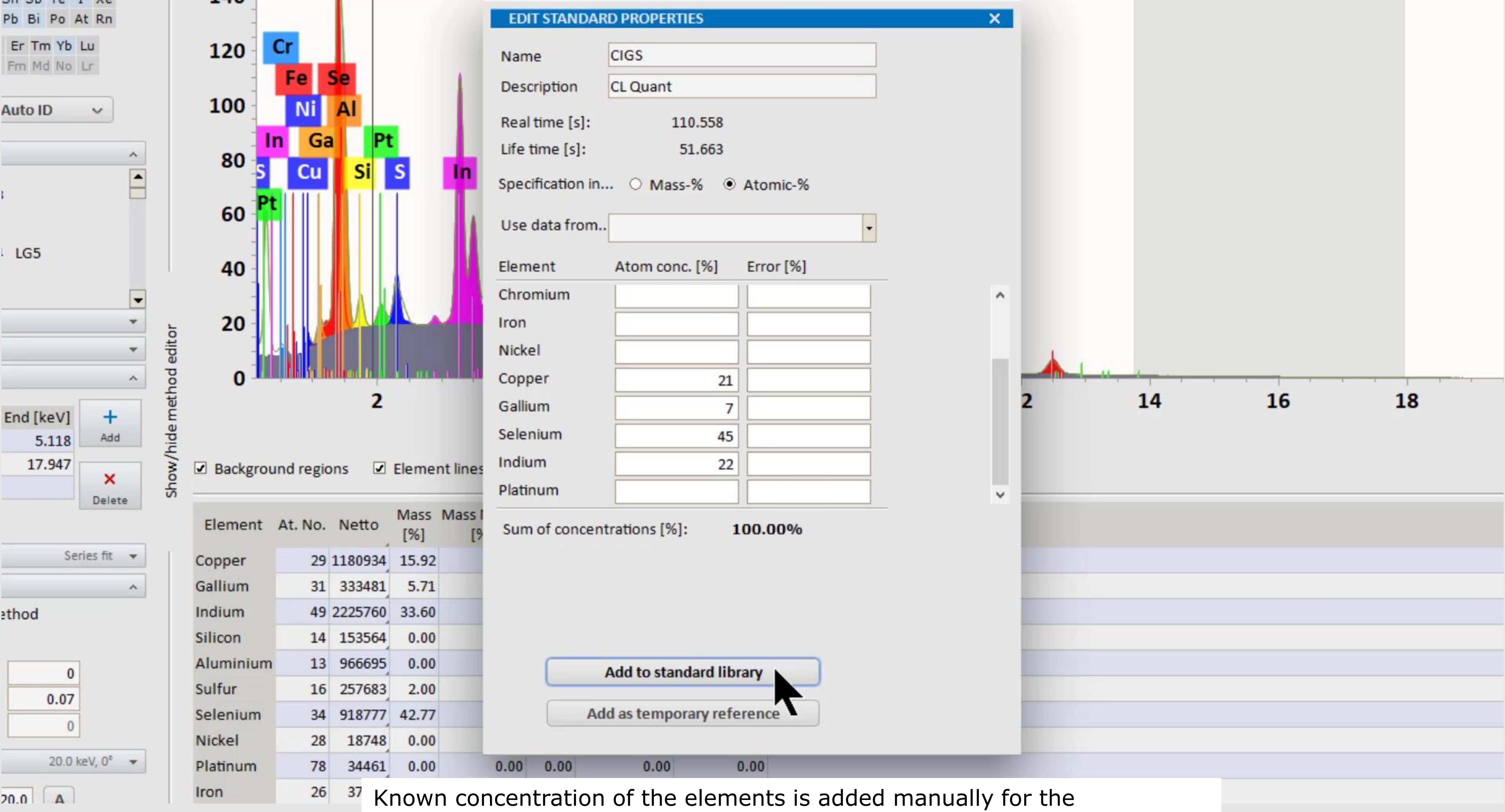
With an e-transparent sample, the signal from the sample holder and the SEM stage (e.g. from Alu and Steel) are present in the spectrum. To exclude these elements from calibration of Cliff-Lorimer factors and from quantification and correct peak separation in case of overlaps, change all these elements to "Deconvolution only".



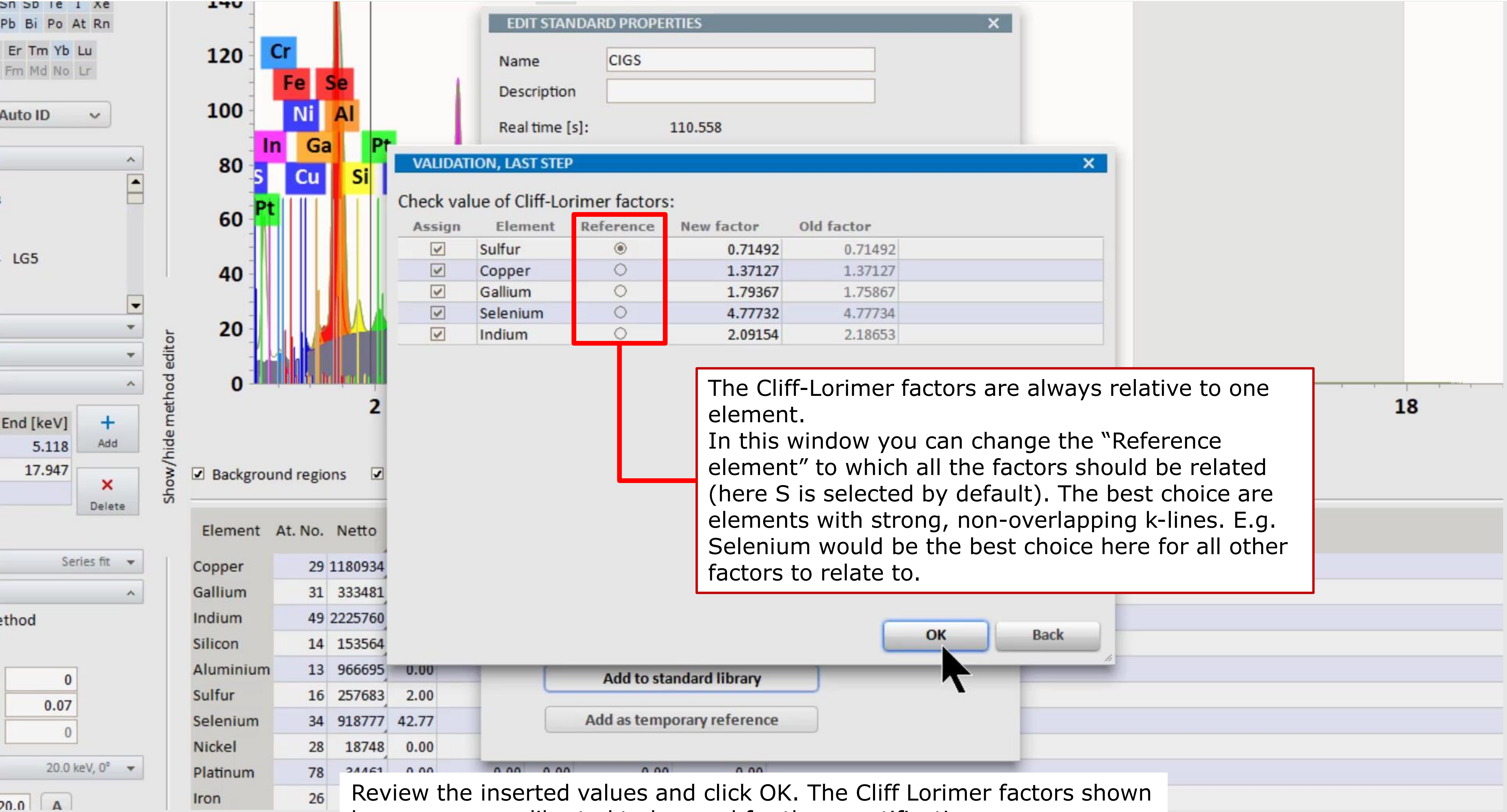
If known, the layer thickness can also be added, here it is 70 nm. The layer density value is calculated theoretically from the spectrum elements, unless changed. Now select "Add to standards" to calibrate Cliff-Lorimer factors experimentally.



These values are calculated from the theoretical Cliff-Lorimer factors, which must be replaced by known or standard concentration values



Known concentration of the elements is added manually for the experimental calibration of Cliff-Lorimer factors

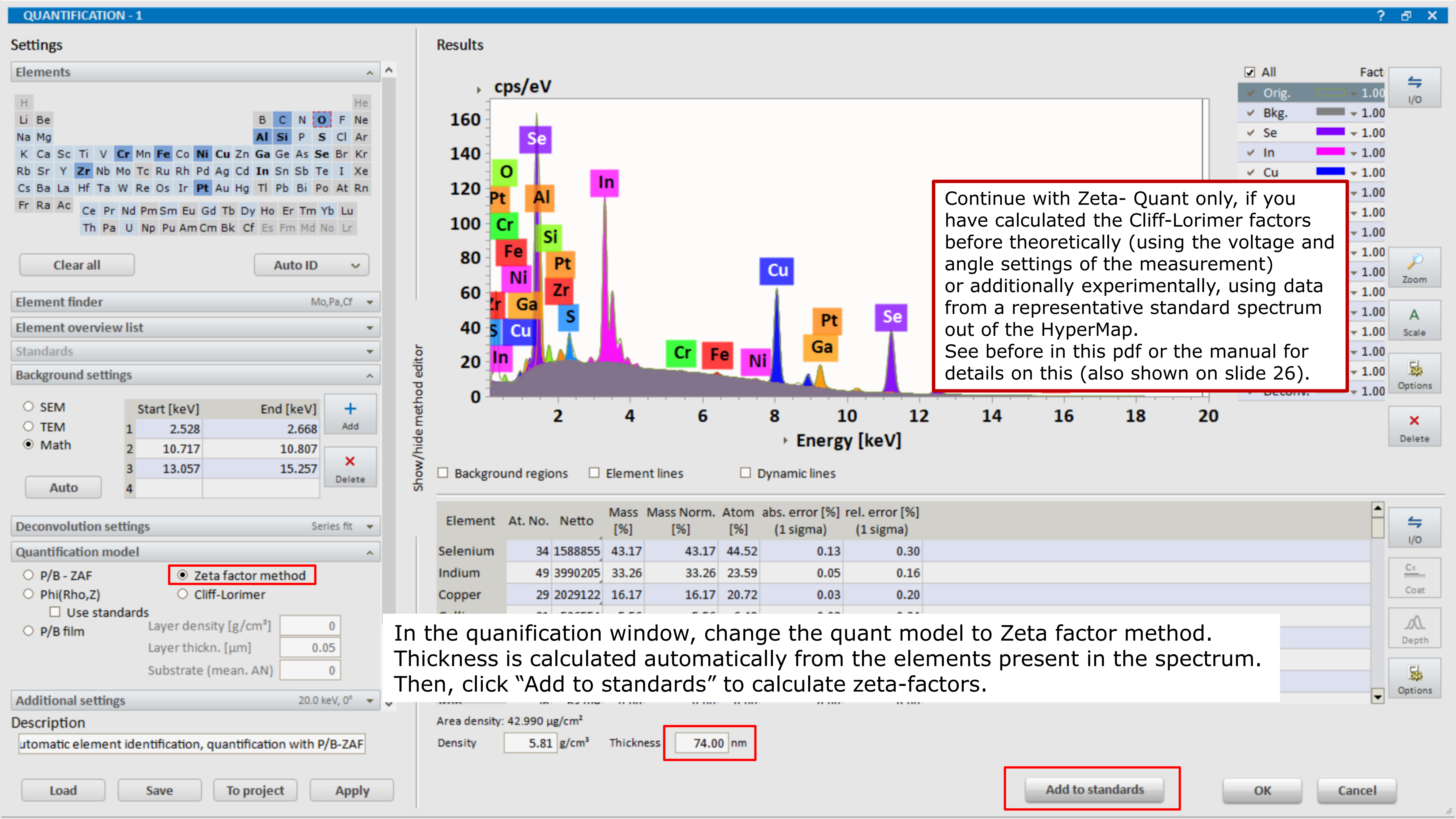


The Cliff-Lorimer factors are always relative to one element.
In this window you can change the "Reference element" to which all the factors should be related (here S is selected by default). The best choice are elements with strong, non-overlapping k-lines. E.g. Selenium would be the best choice here for all other factors to relate to.

Review the inserted values and click OK. The Cliff Lorimer factors shown here are now calibrated to be used for the quantification

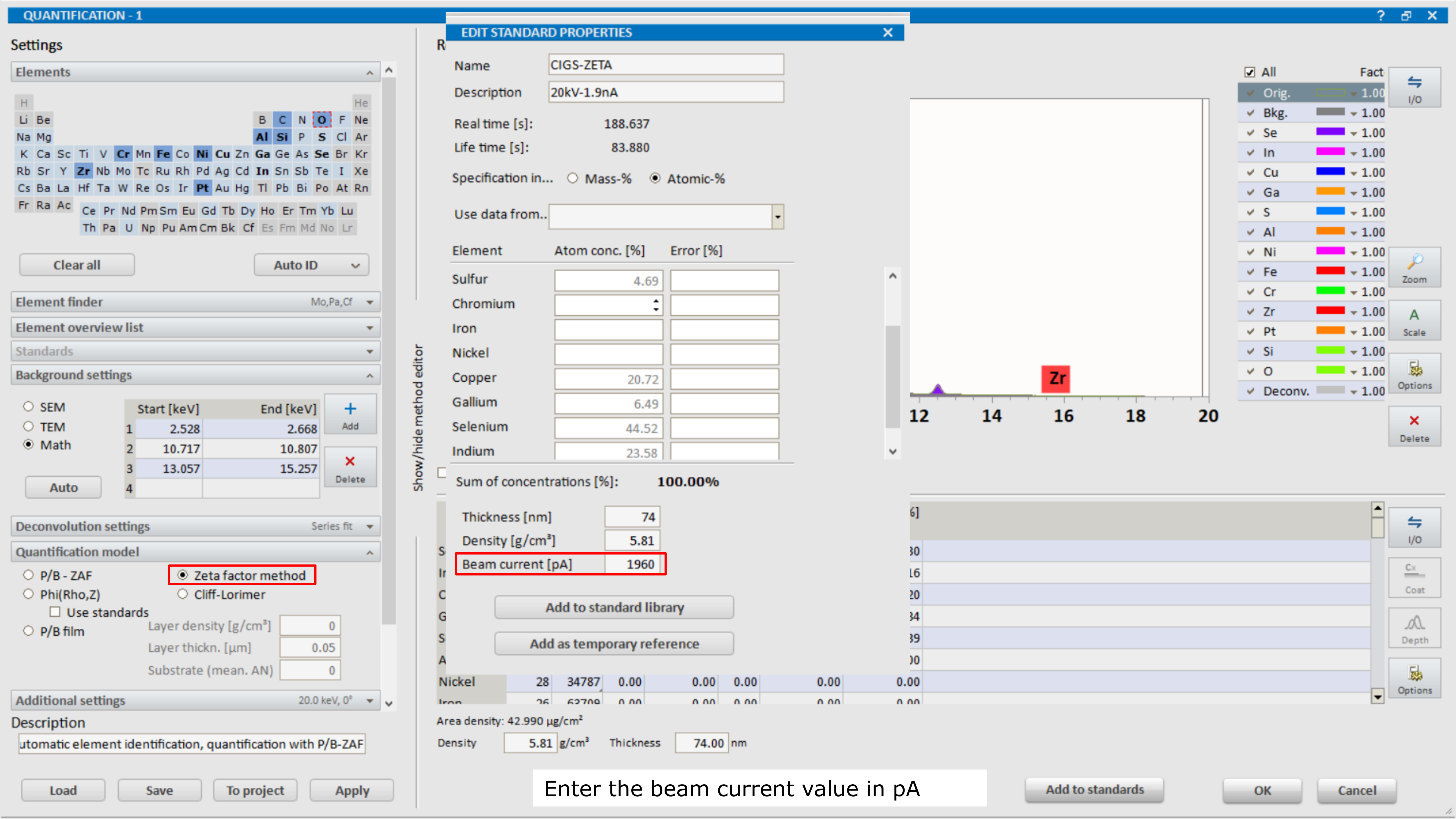
Zeta-Factor Quantification method

for e-transparent samples

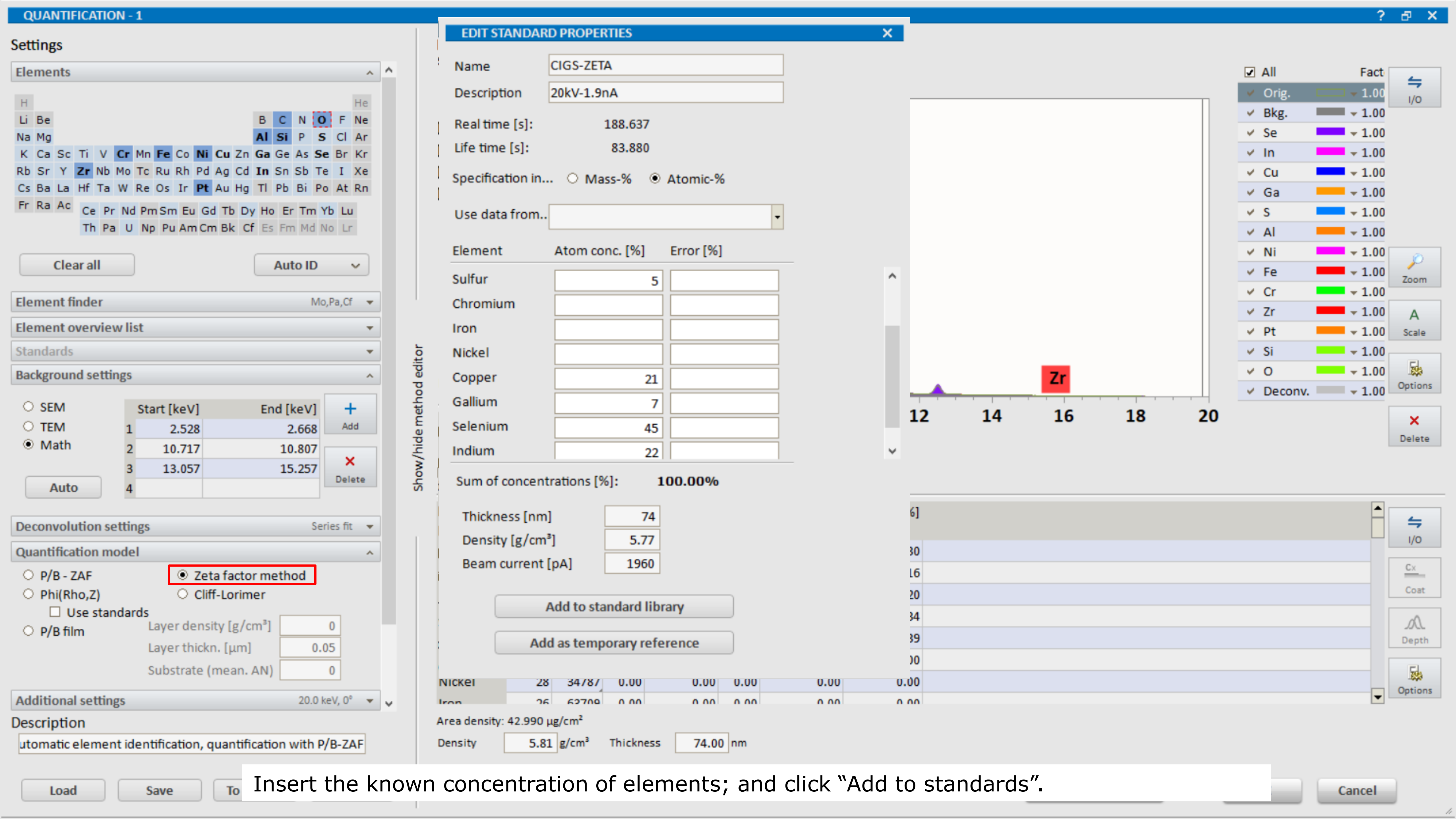


Continue with Zeta- Quant only, if you have calculated the Cliff-Lorimer factors before theoretically (using the voltage and angle settings of the measurement) or additionally experimentally, using data from a representative standard spectrum out of the HyperMap.
See before in this pdf or the manual for details on this (also shown on slide 26).

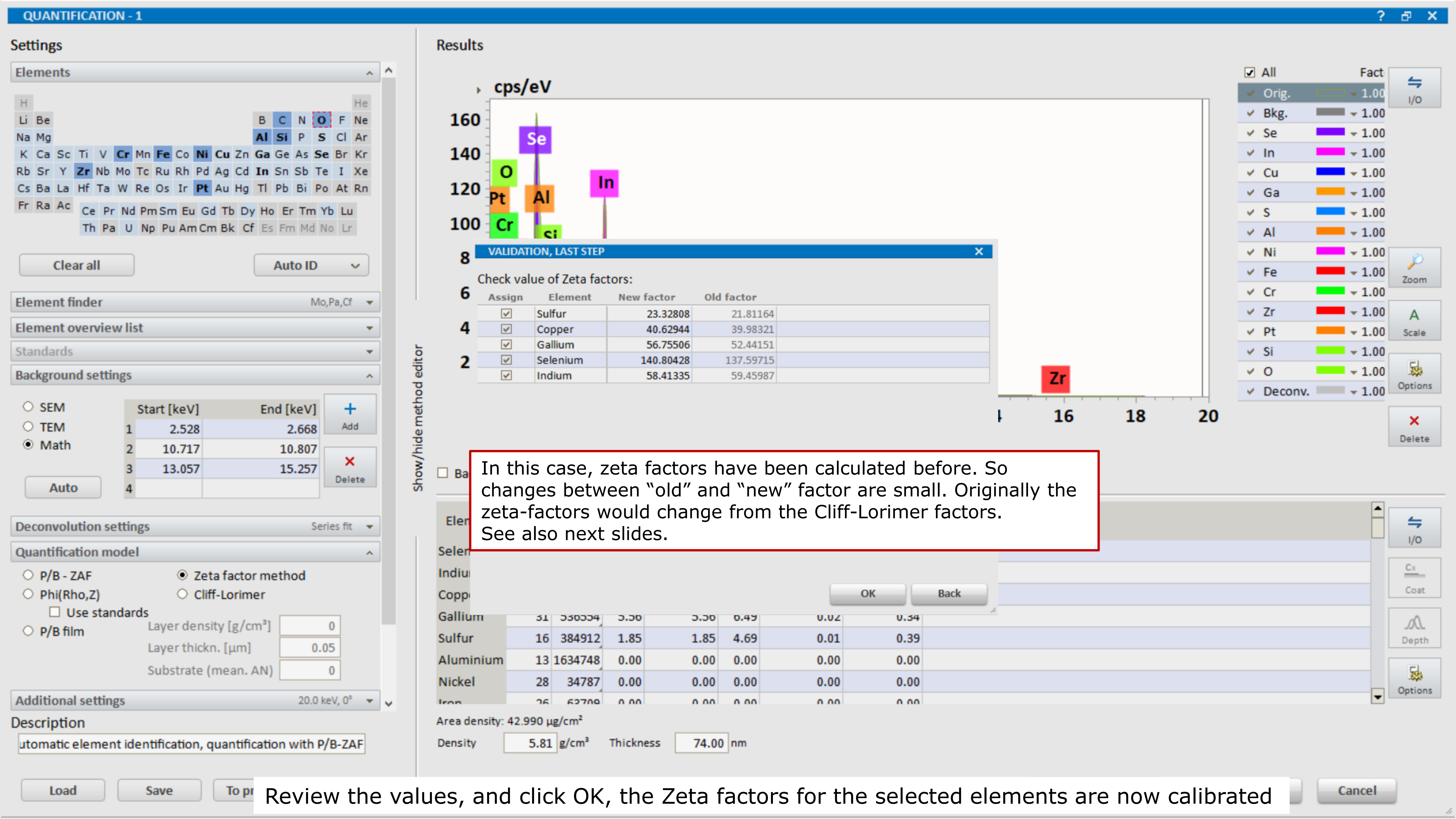
In the quanification window, change the quant model to Zeta factor method. Thickness is calculated automatically from the elements present in the spectrum. Then, click “Add to standards” to calculate zeta-factors.



Enter the beam current value in pA

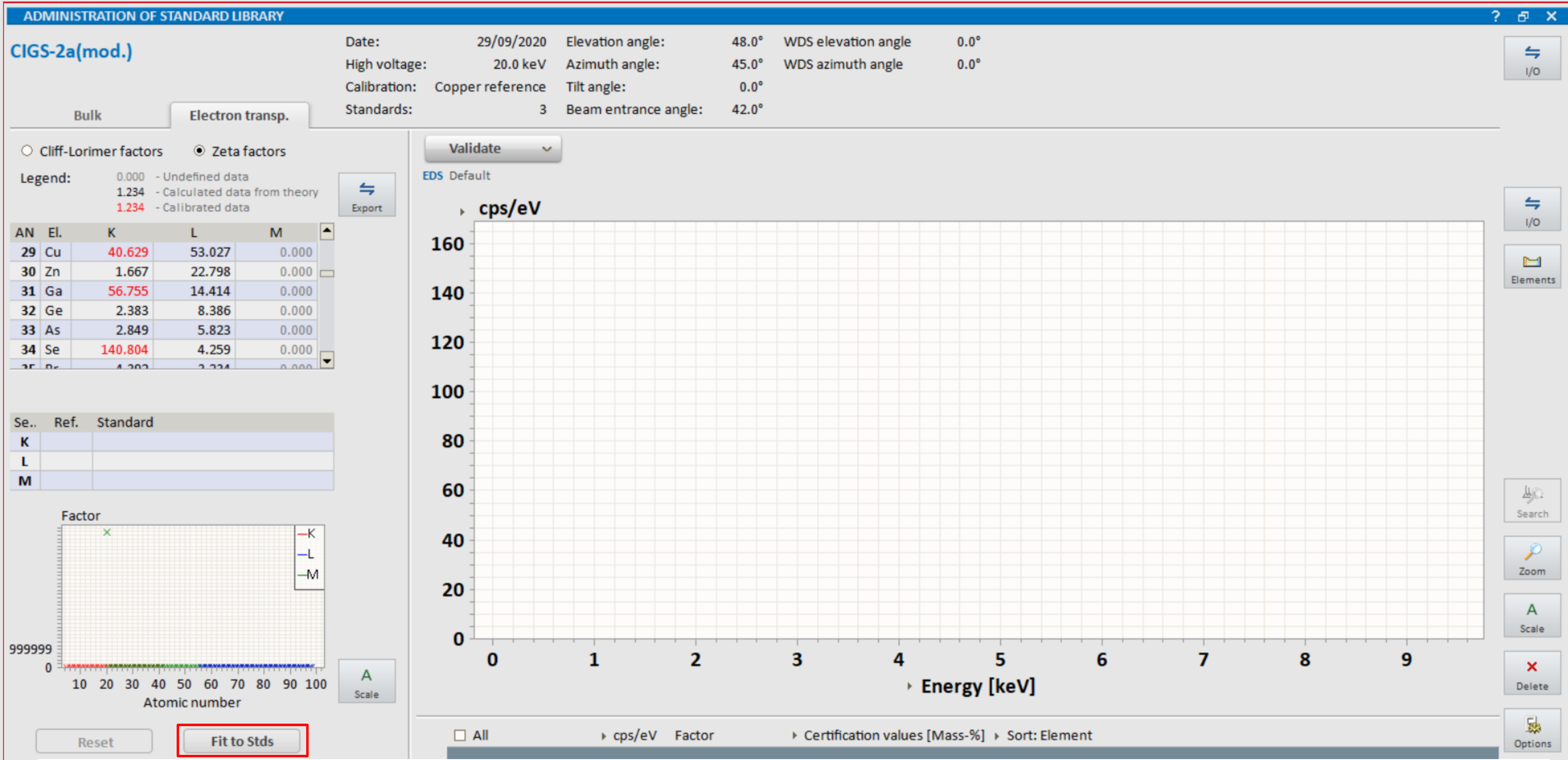


Insert the known concentration of elements; and click “Add to standards”.



In this case, zeta factors have been calculated before. So changes between "old" and "new" factor are small. Originally the zeta-factors would change from the Cliff-Lorimer factors.
See also next slides.

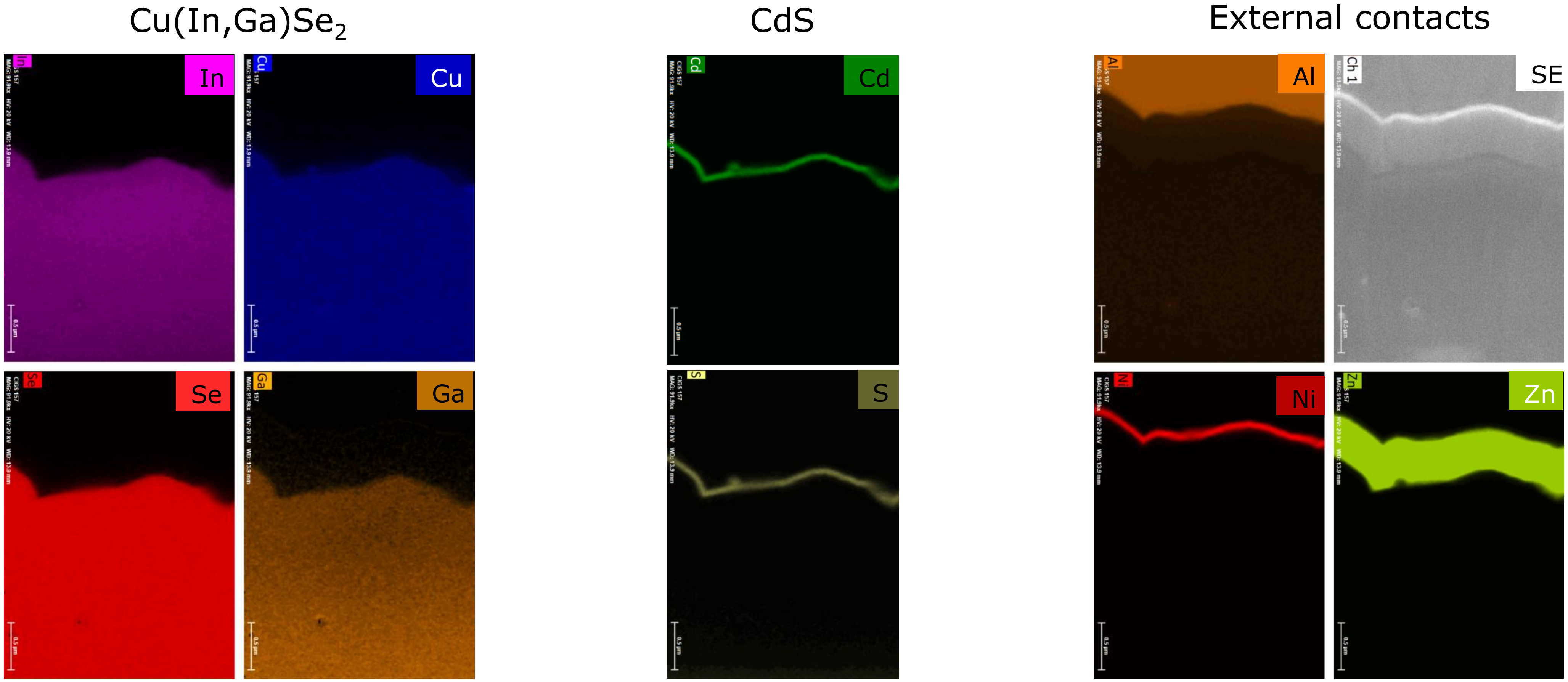
Review the values, and click OK, the Zeta factors for the selected elements are now calibrated



In the standards library, In RED, you can now see the calibrated Zeta factors for the given beam current. You can then click “fit to standards” to interpolate the theoretical zeta factors for other elements for the given e-beam conditions using the Cliff-Lorimer factors calculated before.

Application example: $Cu(In,Ga)Se_2$ solar cell (contd…)

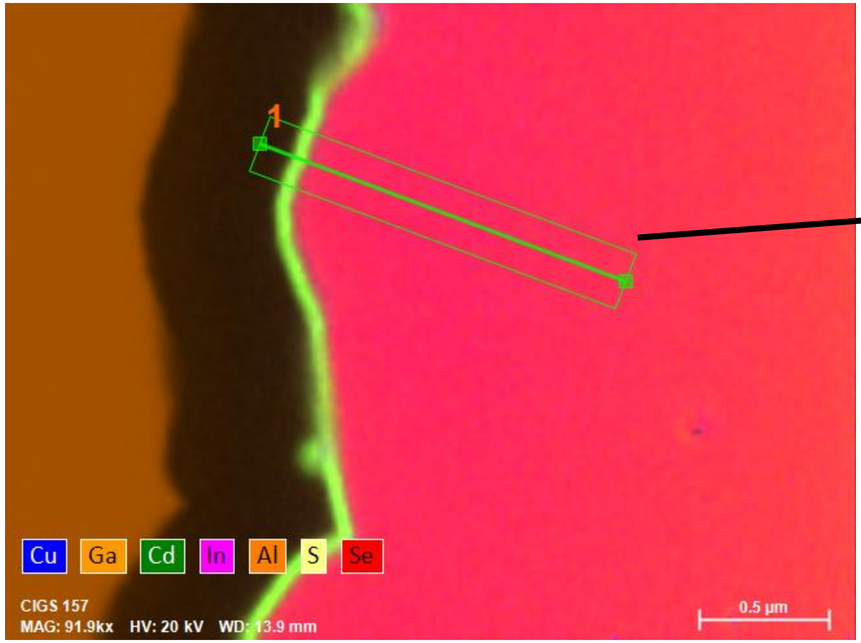
Quantified map in atomic %

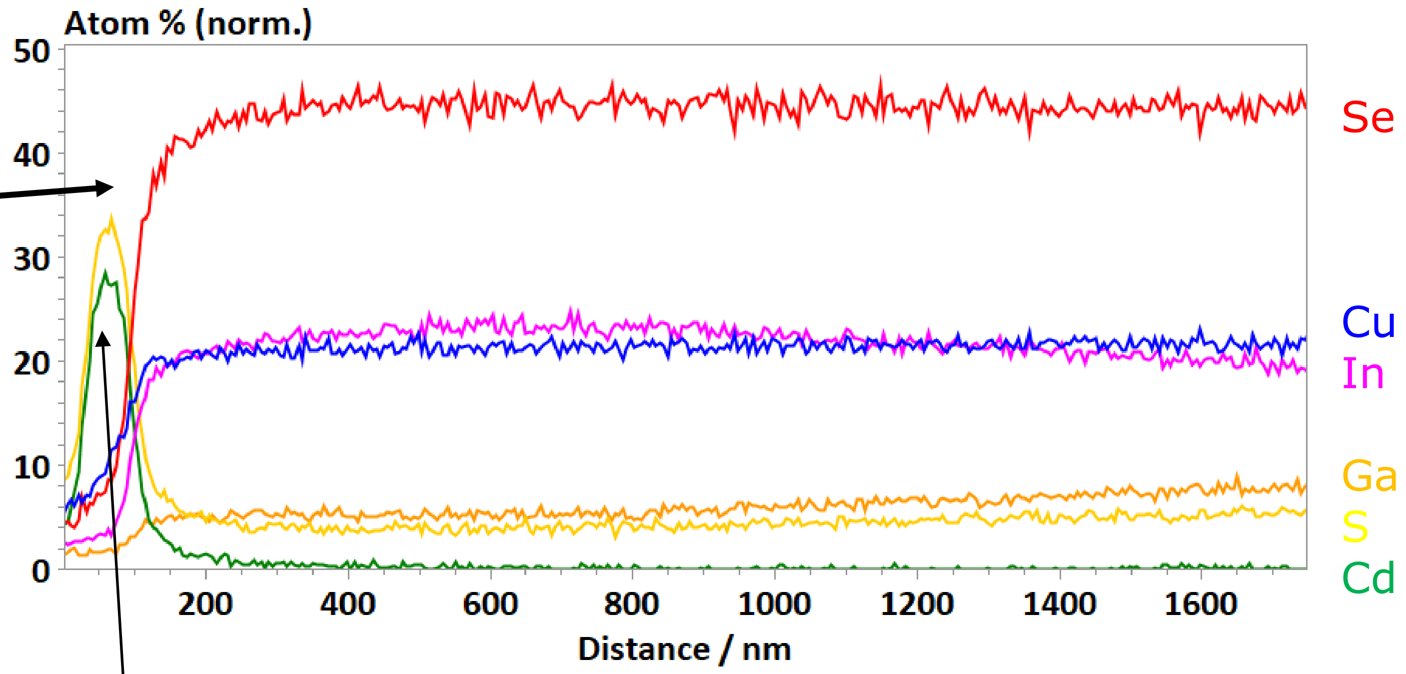


(Cliff-Lorimer quantification)

Application example: $Cu(InGa)Se_2$ solar cell

EDS Line scan in Atomic %

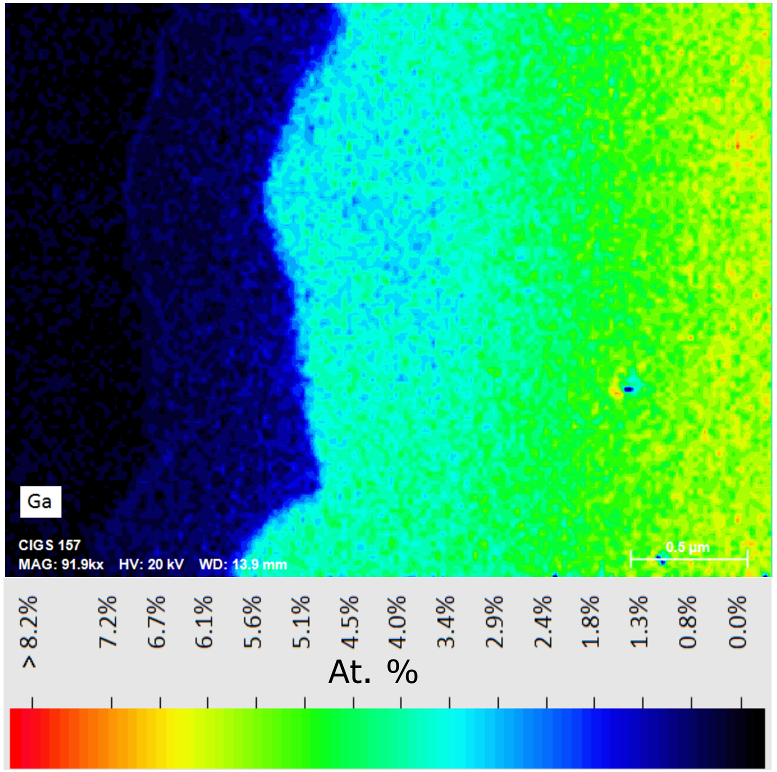


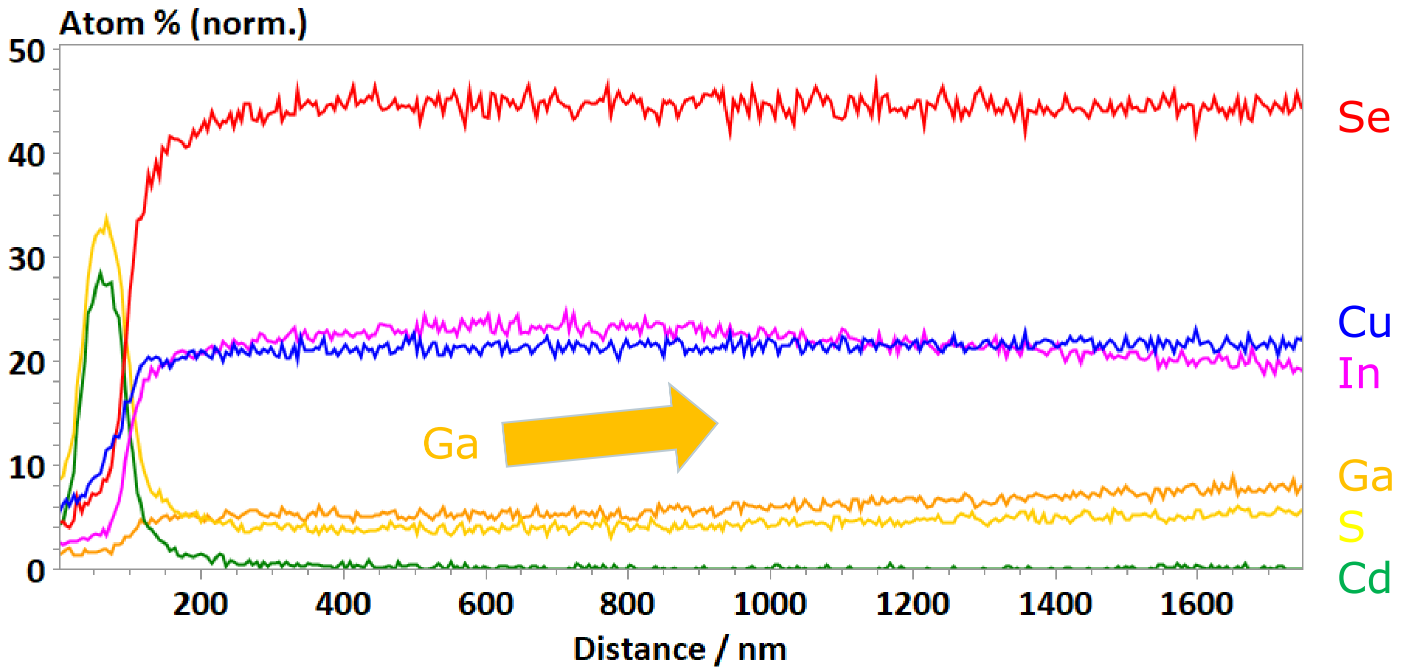


Thin CdS buffer layer (~60-80 nm)

Application example: $Cu(InGa)Se_2$ solar cell

EDS Line scan in Atomic %

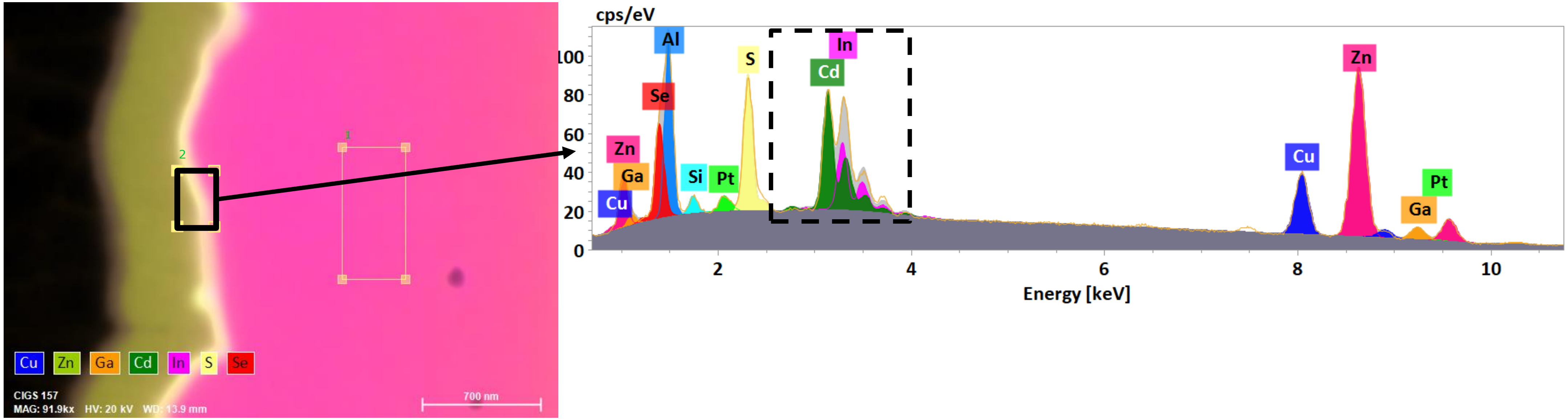




Ga grading in CIGS absorber detected by EDS

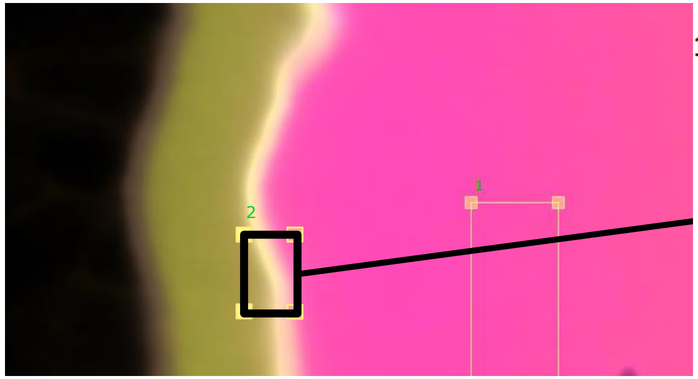
Application example: $Cu(In,Ga)Se_2$ solar cell

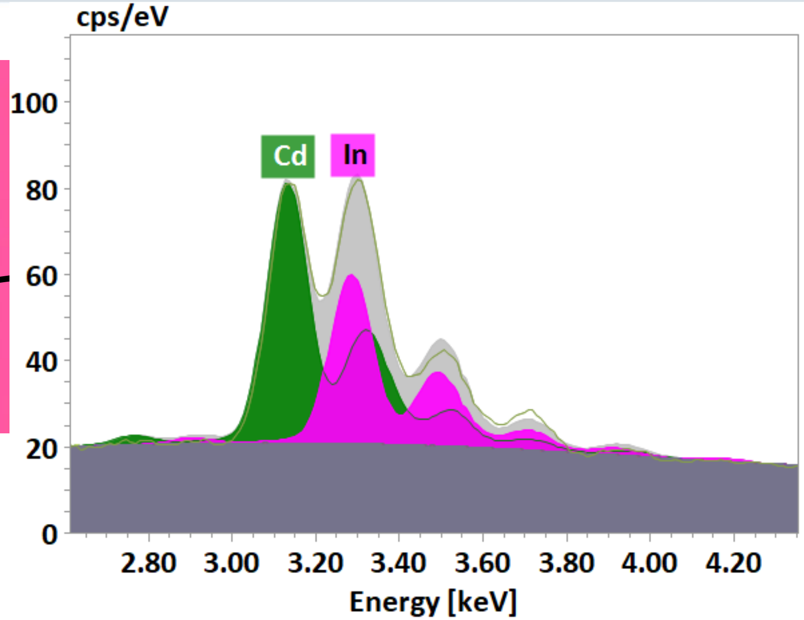
EDS mapping – automated peak deconvolution

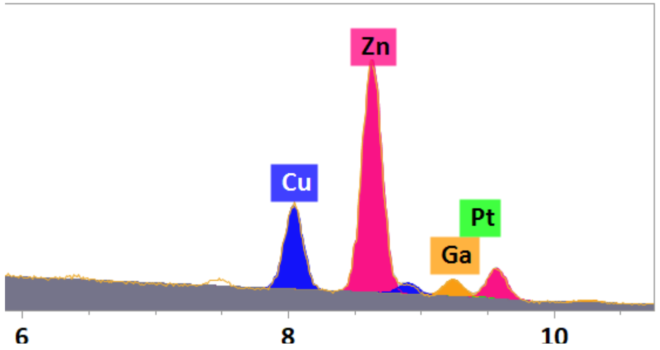


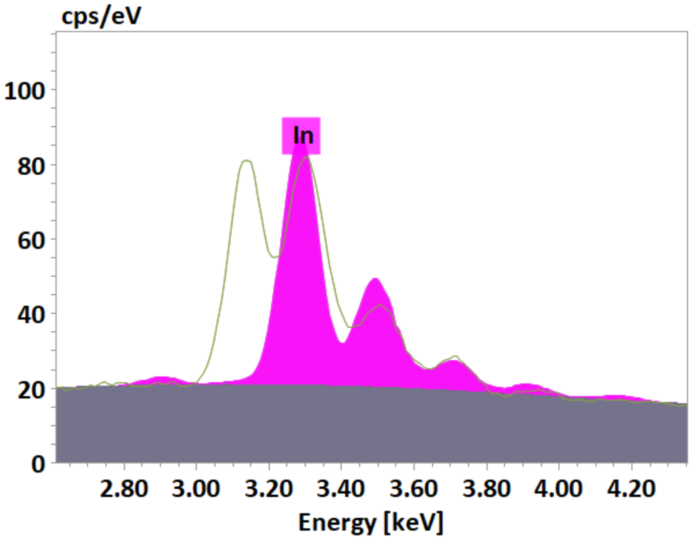
Application example: $Cu(In,Ga)Se_2$ solar cell

EDS mapping – automated peak deconvolution

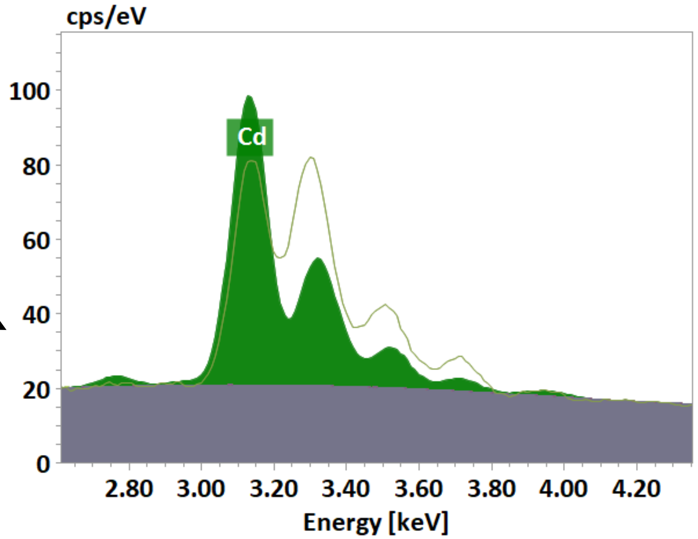




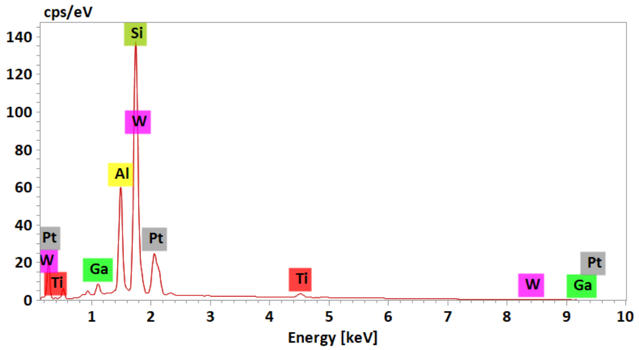




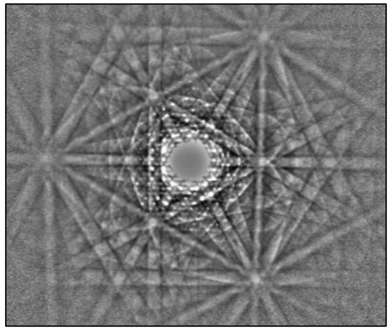
- Automated element detection and deconvolution



Simultaneous chemical (EDS) and microstructural (TKD) characterization



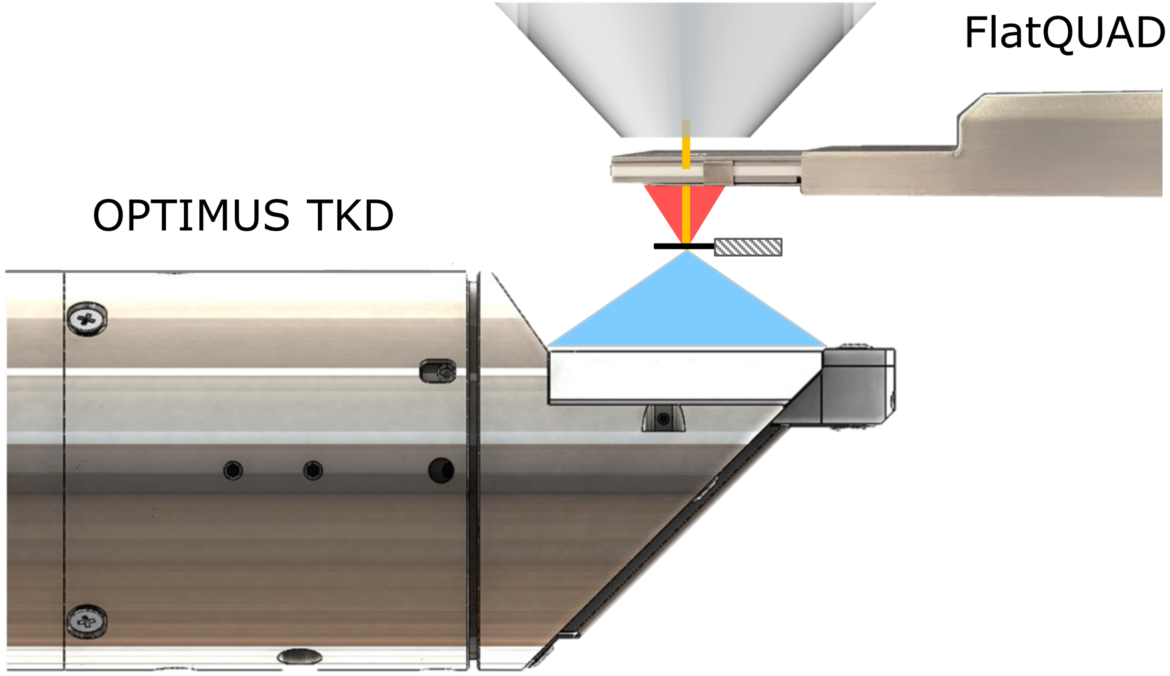
EDS spectrum

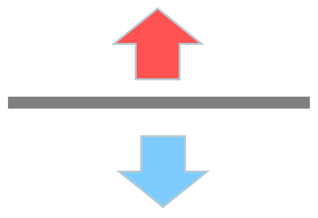


Kikuchi pattern

EDS/TKD simultaneous measurement

XFlash FlatQUAD with OPTIMUS ™ TKD

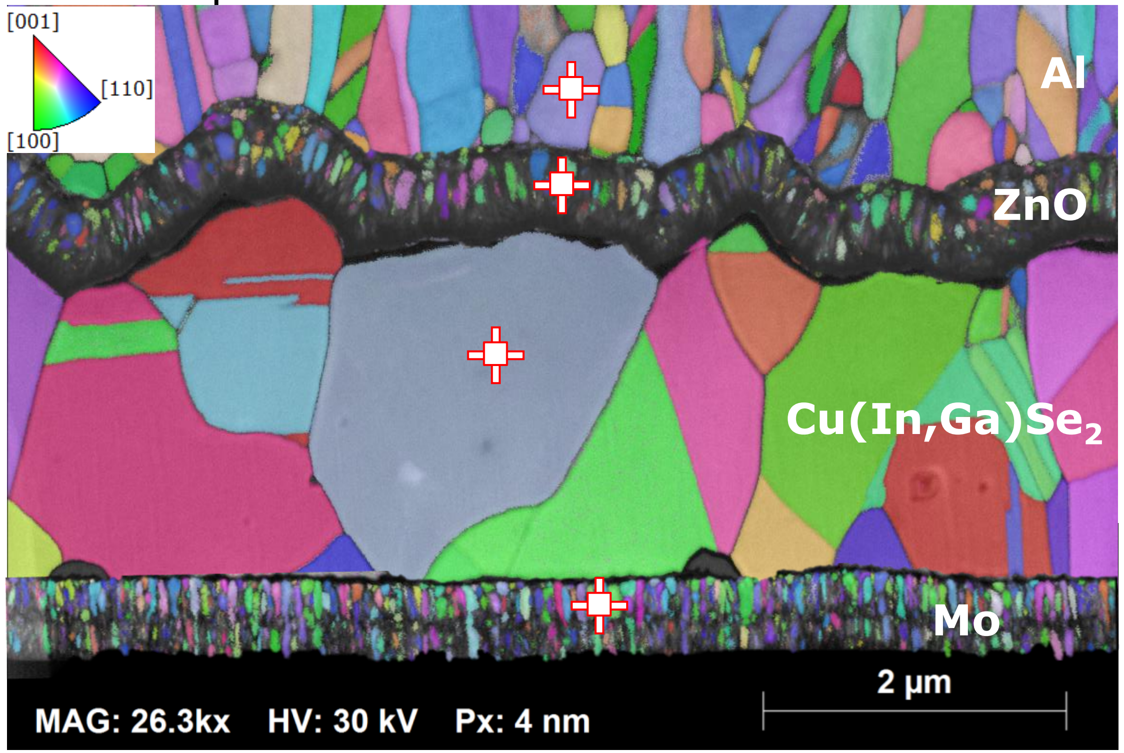


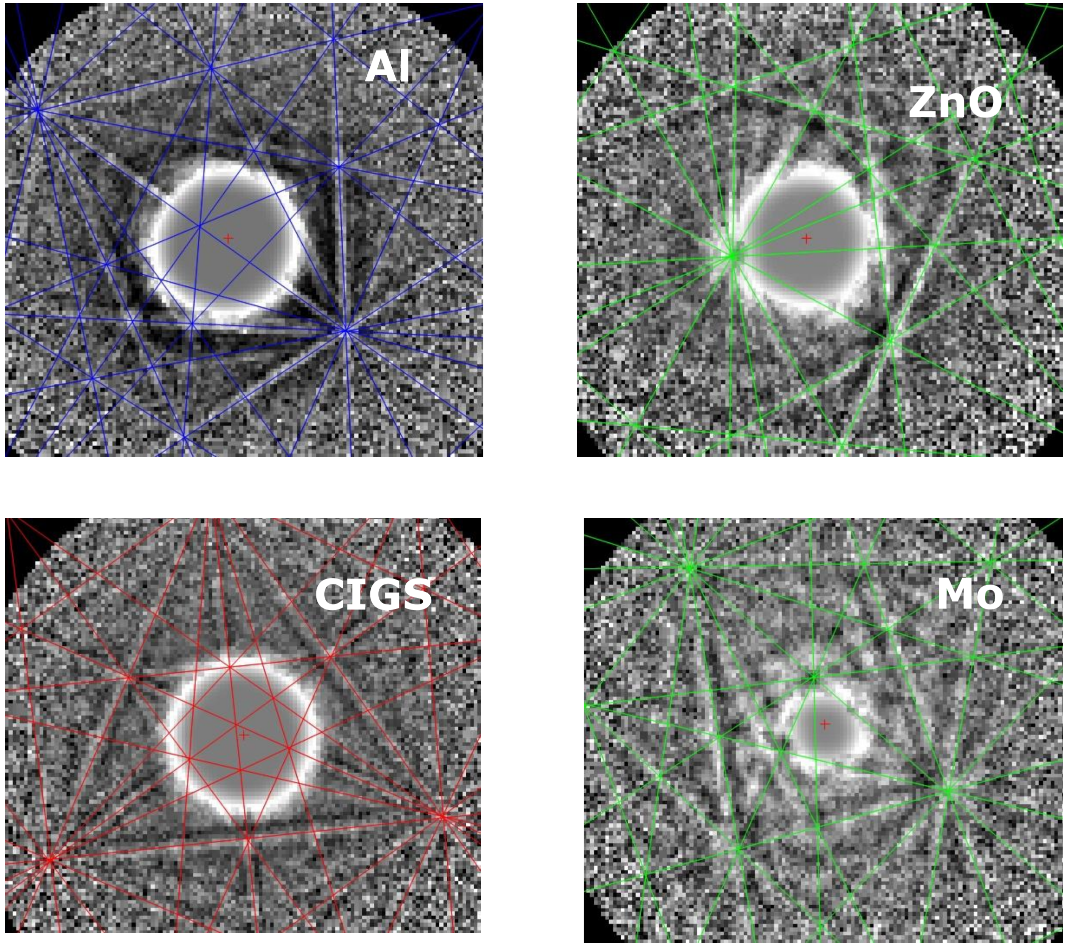


Application example: Cu(In,Ga)Se_2 solar cell

Crystallographic and orientational mapping using OPTIMUS TKD

IPF-Z map

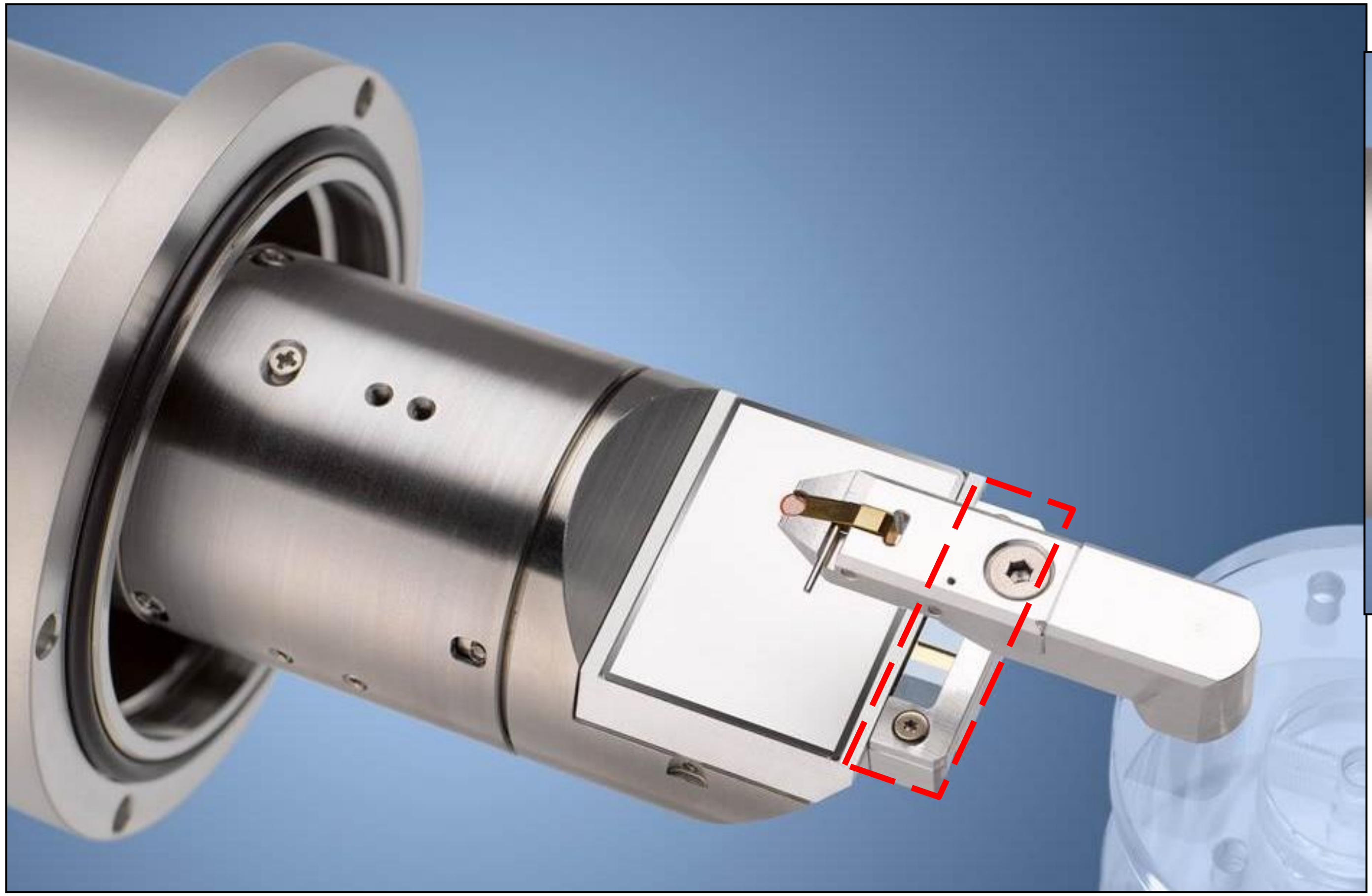


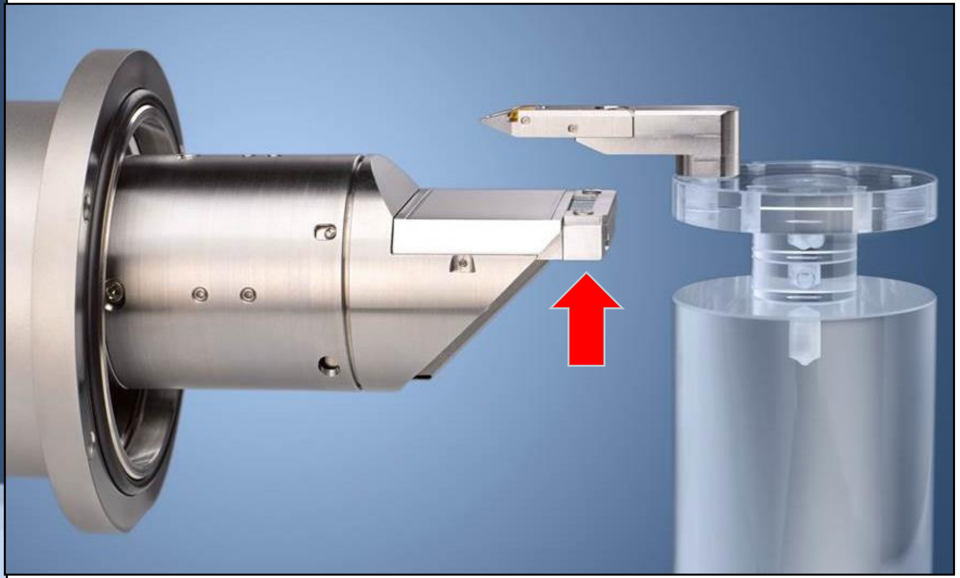


Mapping parameters:

- 30kV; 1.6 nA; Step size: 4nm
- Acq. speed: 330 fps; **Map size: 1.93 million pixels**

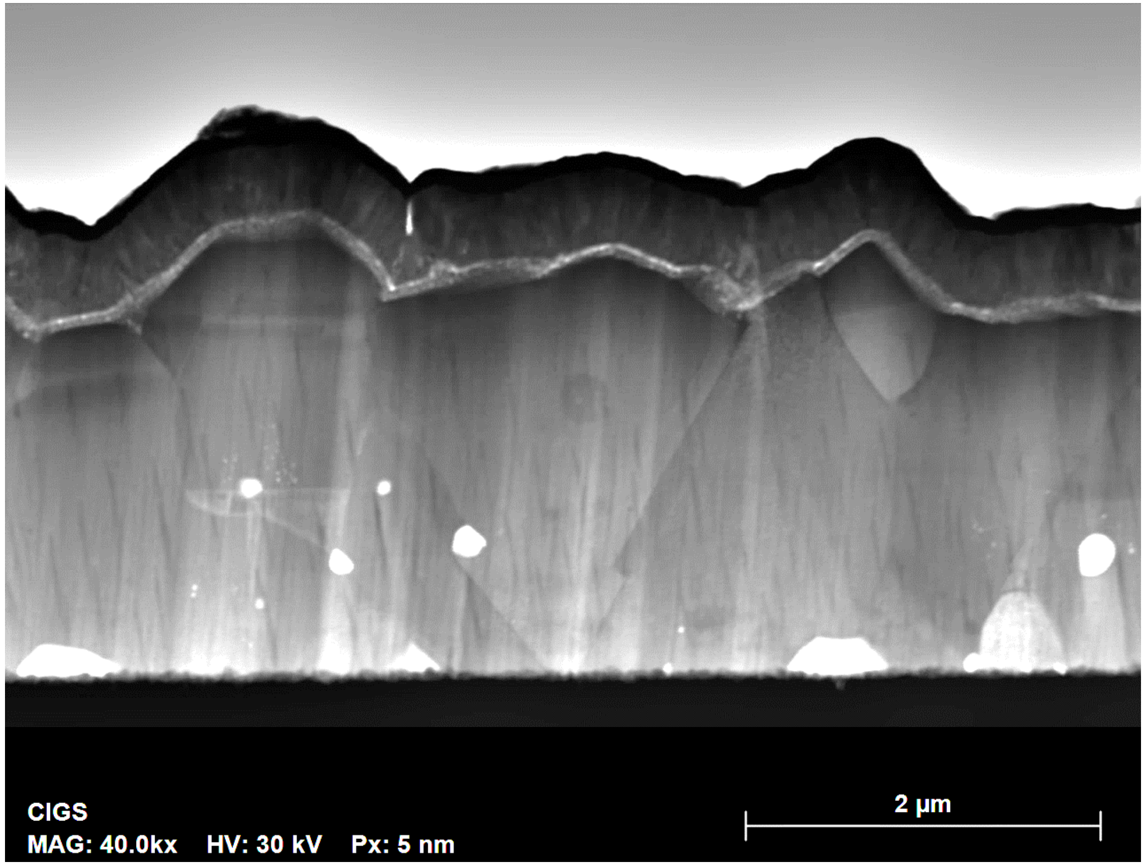
ARGUS imaging
Color coded orientation contrast imaging





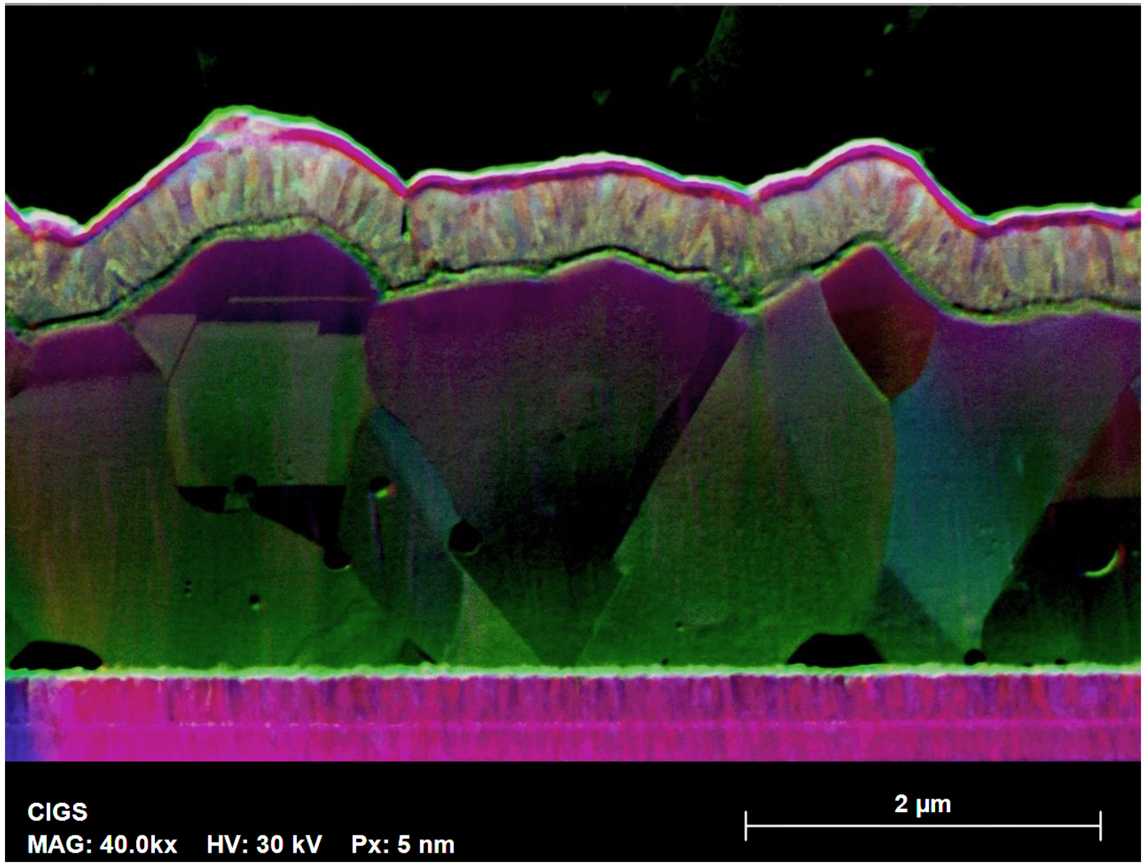
3x ARGUS diodes for STEM-like DF and BF imaging for e^- transparent sample

ARGUS imaging
Color coded orientation contrast imaging



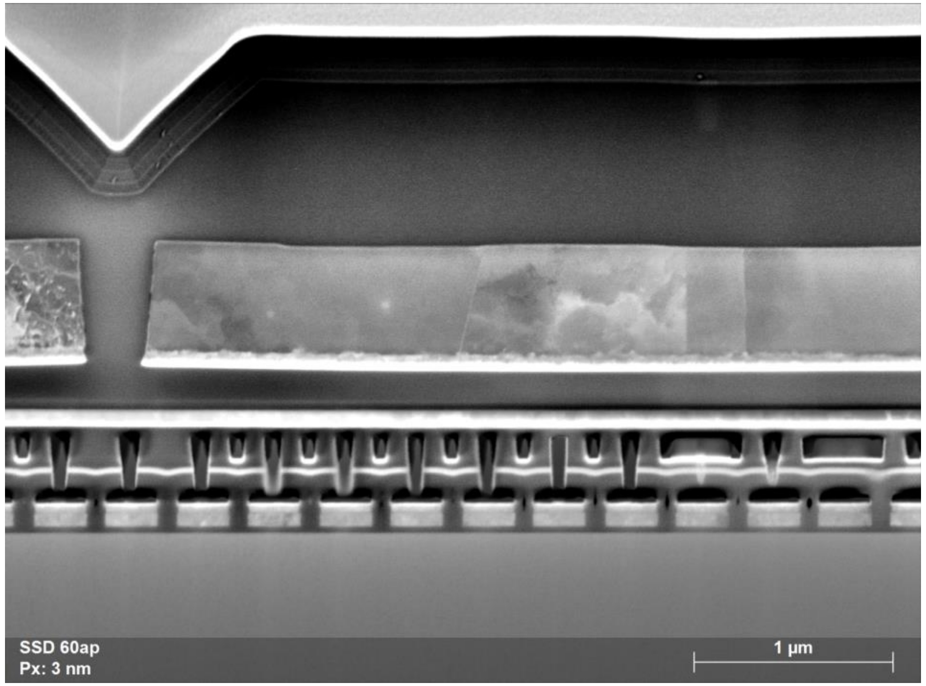
- Bright field mode imaging (direct detection)

ARGUS imaging
Color coded orientation contrast imaging



- Color coded dark field-like imaging (High angle scattered signal)

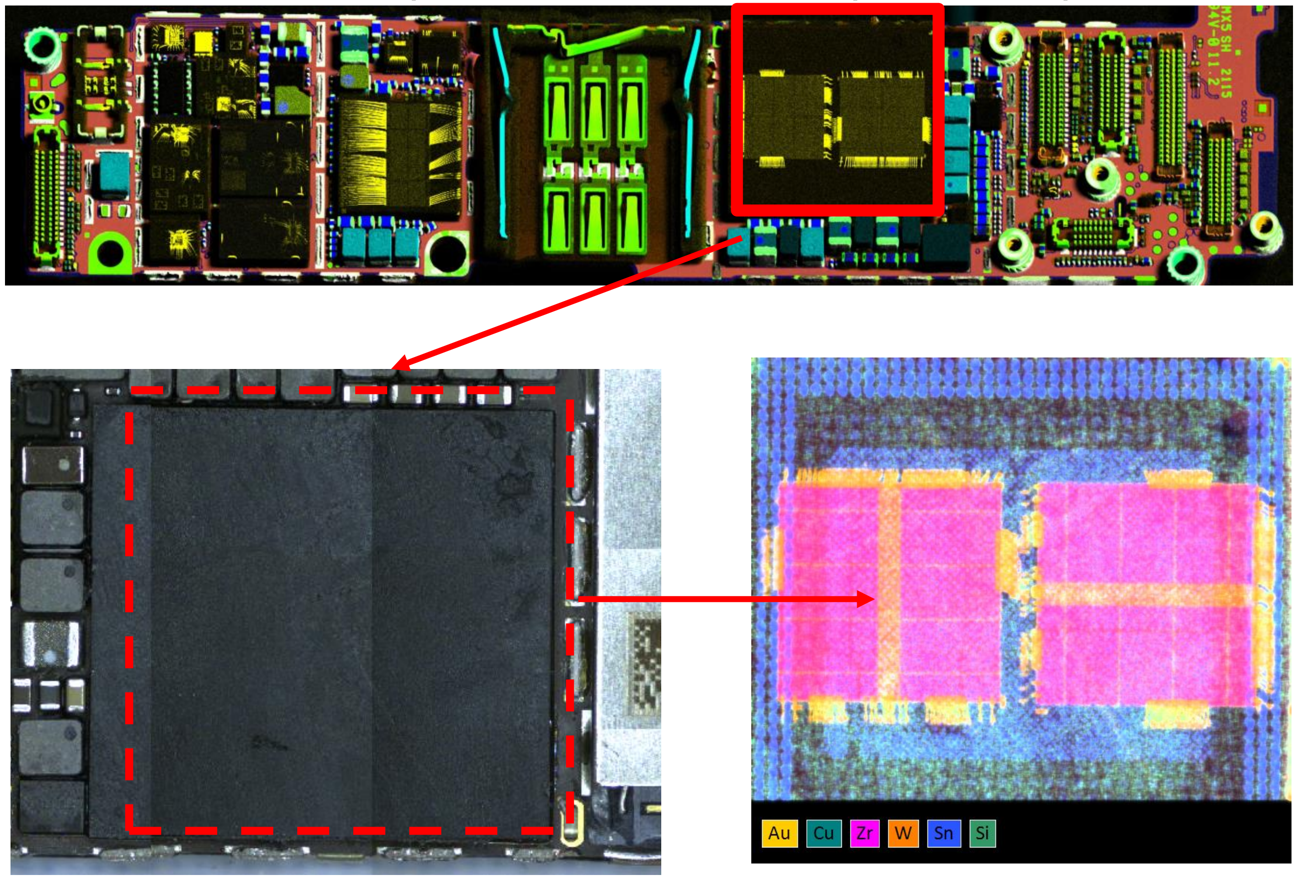
Application example: SSD



Large area mapping

Locating region of interest using µ-XRF

Smartphone motherboard µ-XRF map



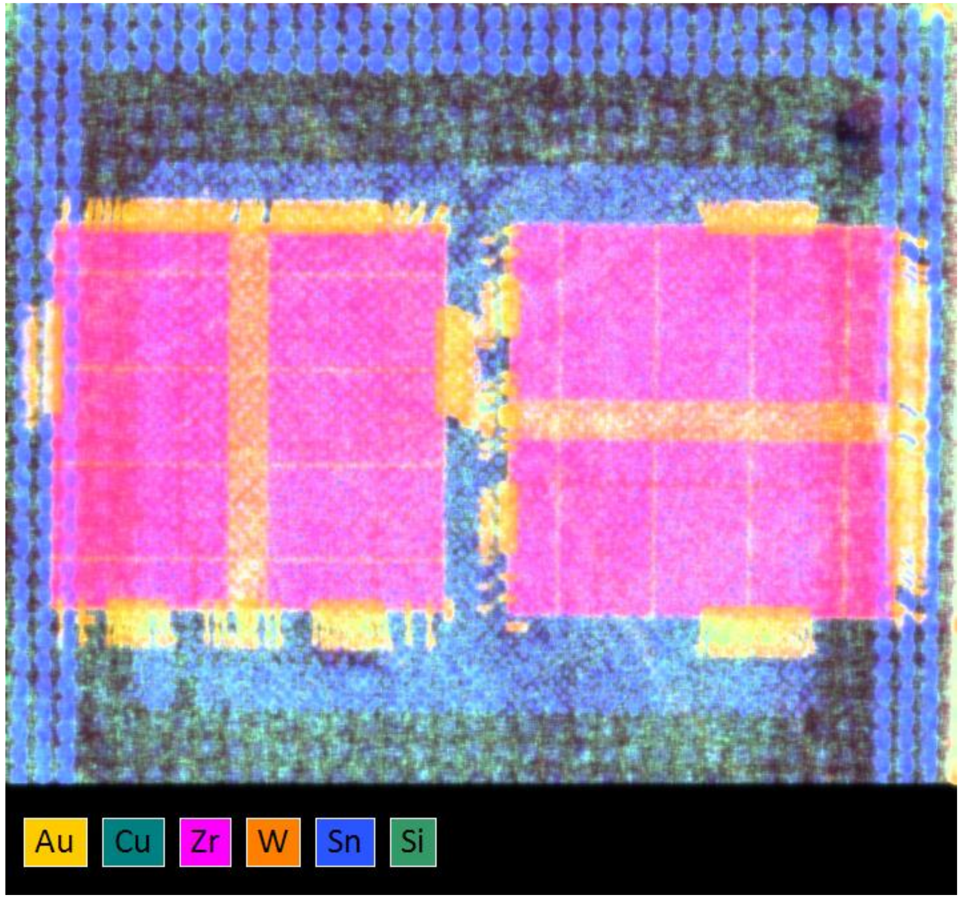
- Large area mapping using µ-XRF

- Locating the area for lift out on the sample

Large area mapping

Locating region of interest using µ-XRF

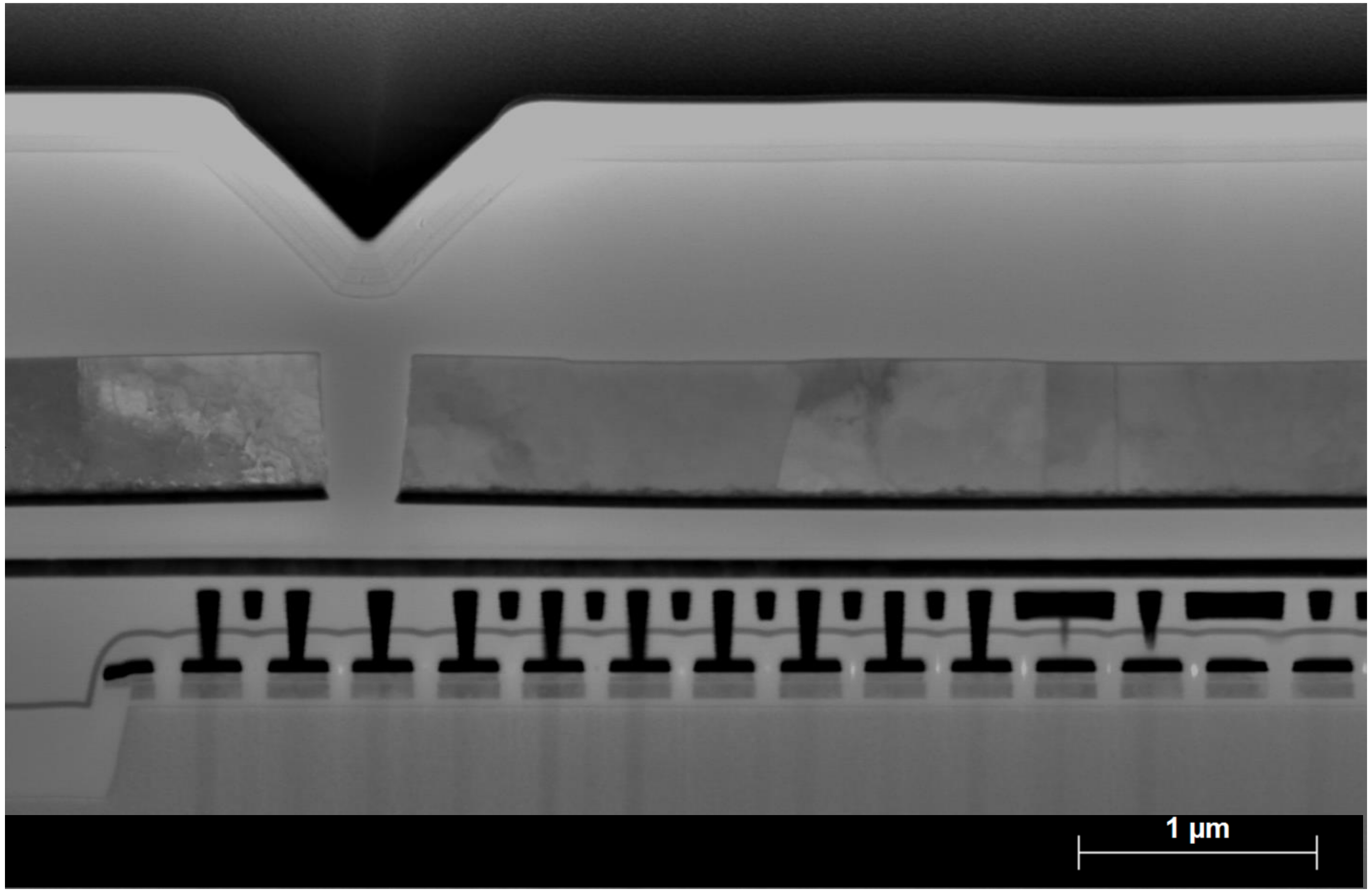
Smartphone motherboard µ-XRF map



- Large area mapping using µ-XRF

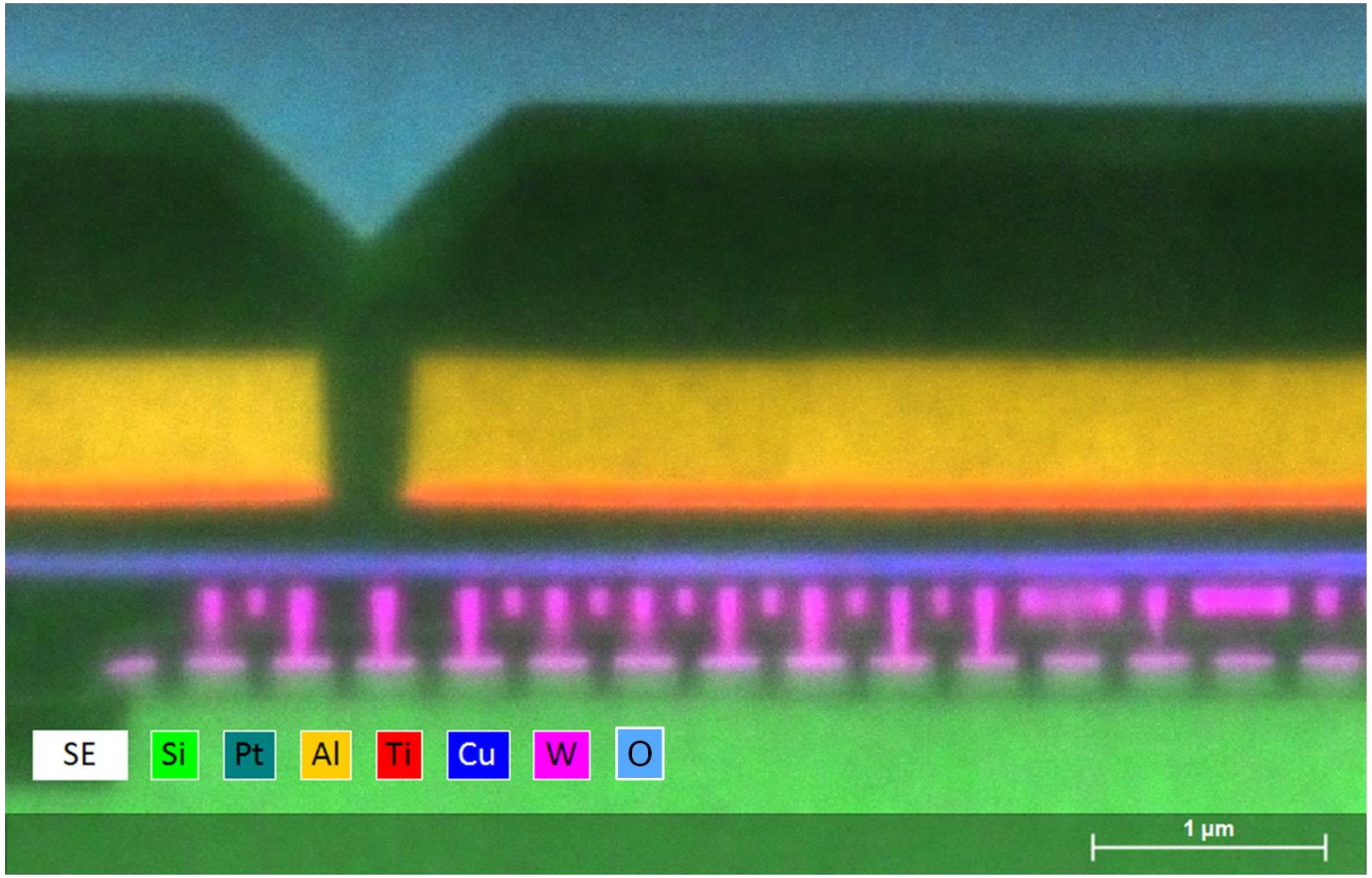
- Locating the area for lift out on the sample

Application example: SSD
EDS mapping – individual element profile



STEM Bright field

Application example: SSD
EDS mapping – individual element profile



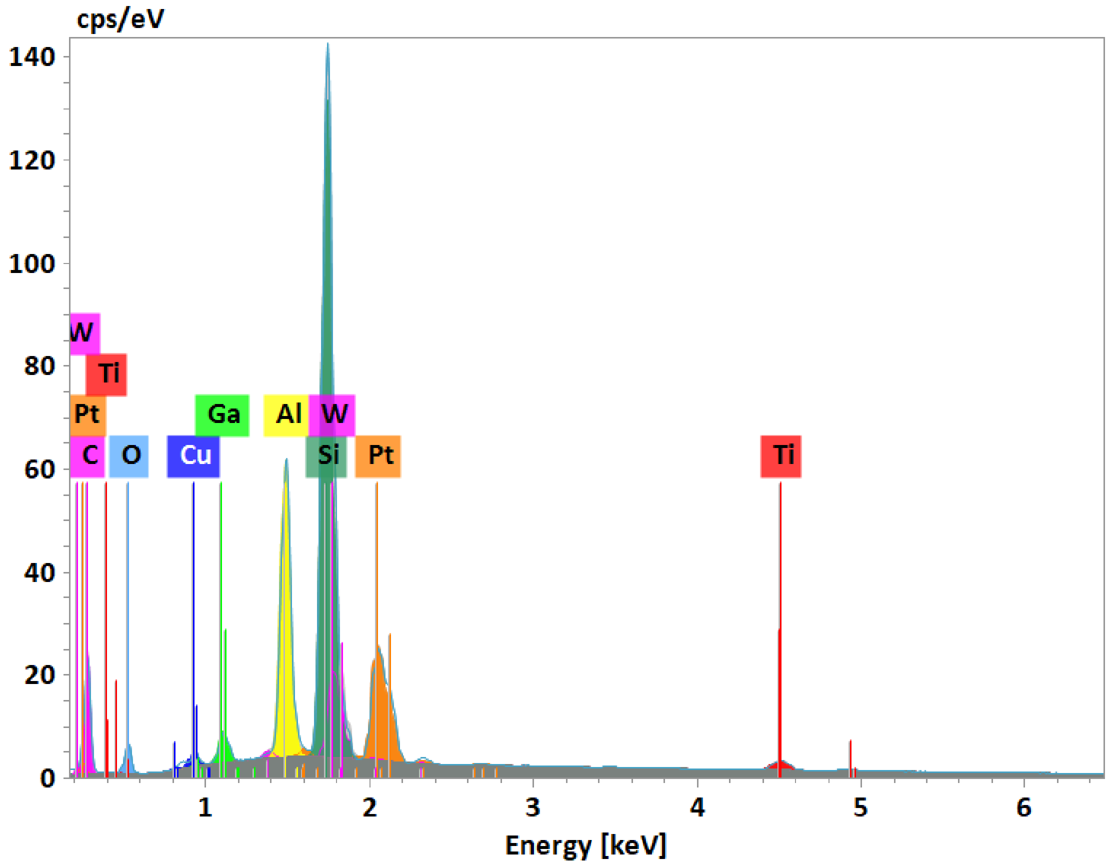
EDS map

EDS mapping parameters:

- High voltage: 10 kV
- Abs. current: 410 pA
- WD: 9 mm
- Mag: 35,000 x
- **Map time: 300 s**
- Map size: 800 × 533 px
- Input counts: 86.6 Kcps
- Output counts: 36.1 Kcps
- **Total counts: 1.08E+07 (10.8 M)**

Application example: SSD

EDS mapping – Spectrum and mapping details

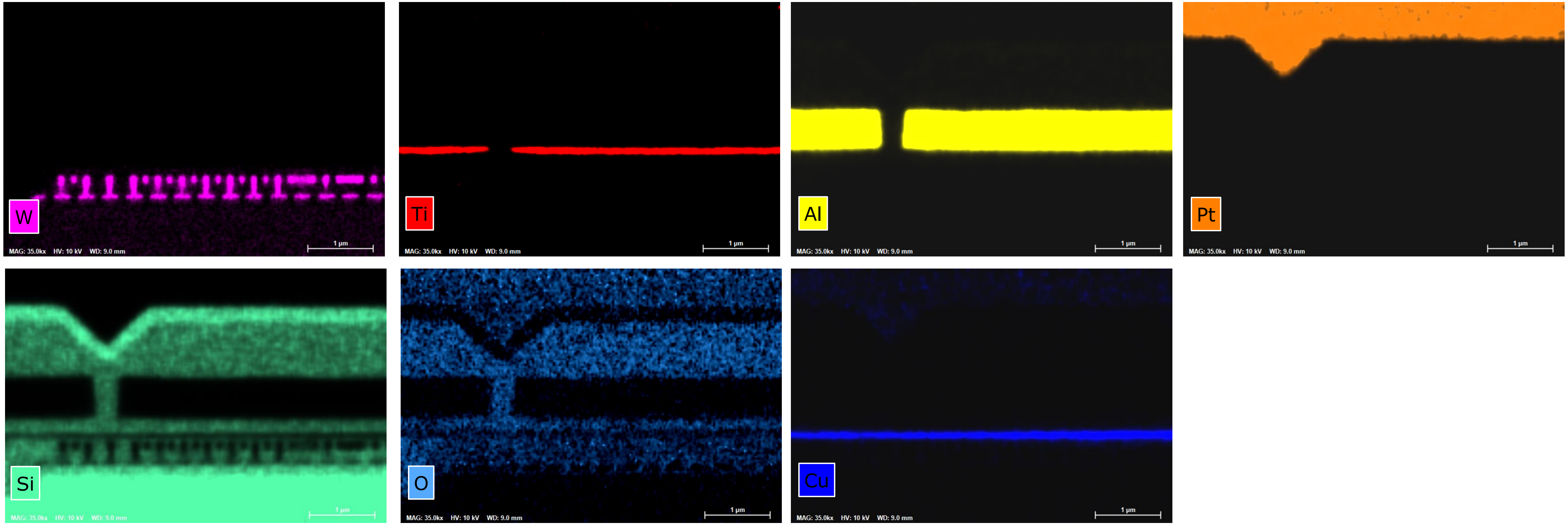


EDS mapping parameters:

- High voltage: 10 kV
- Abs. current: 410 pA
- WD: 9 mm
- Mag: 35,000 x
- **Map time: 300 s**
- Input counts: 86.6 Kcps
- Output counts: 36.1 Kcps
- **Total counts: 1.08E+07 (10.8 M)**

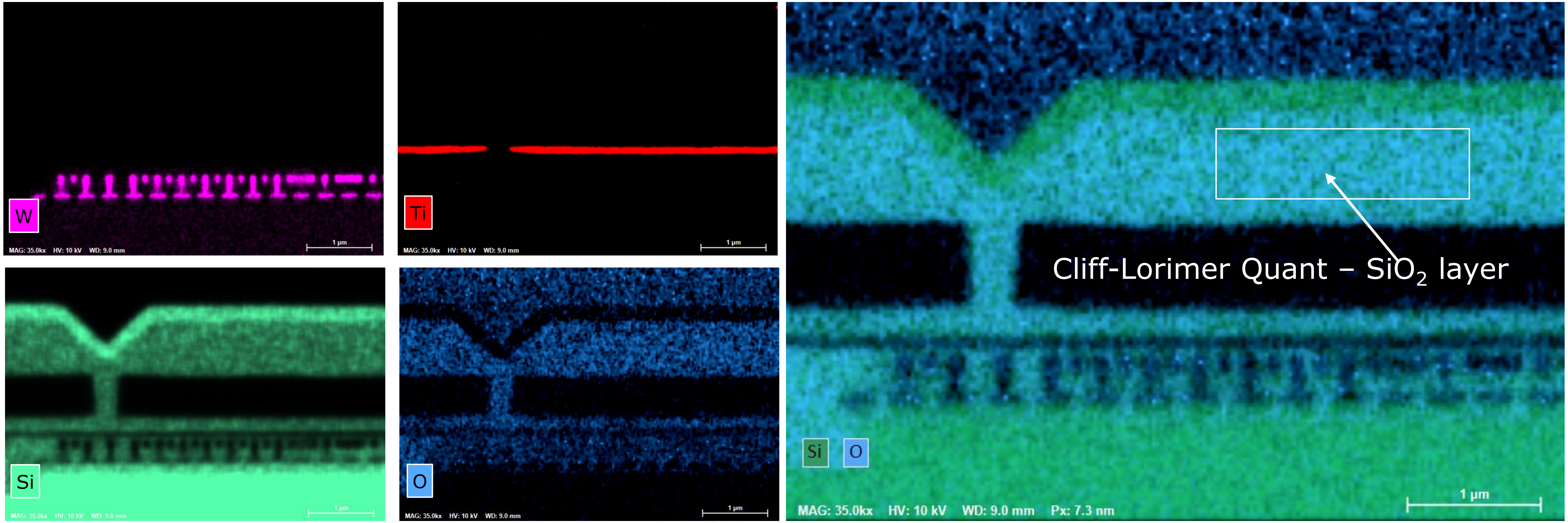
Application example: SSD

Quantified map in atomic %



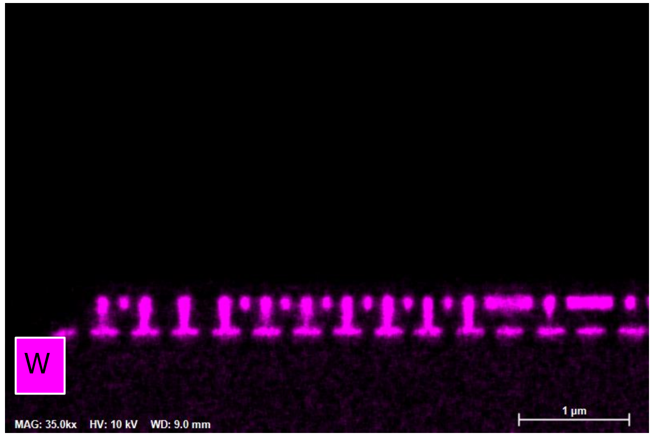
Application example: SSD

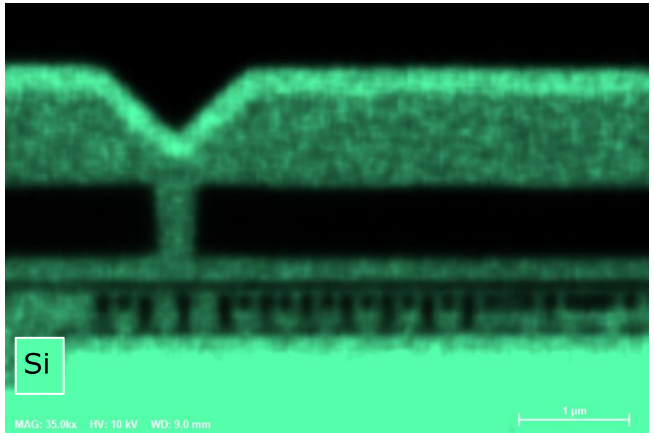
Quantified map in atomic %

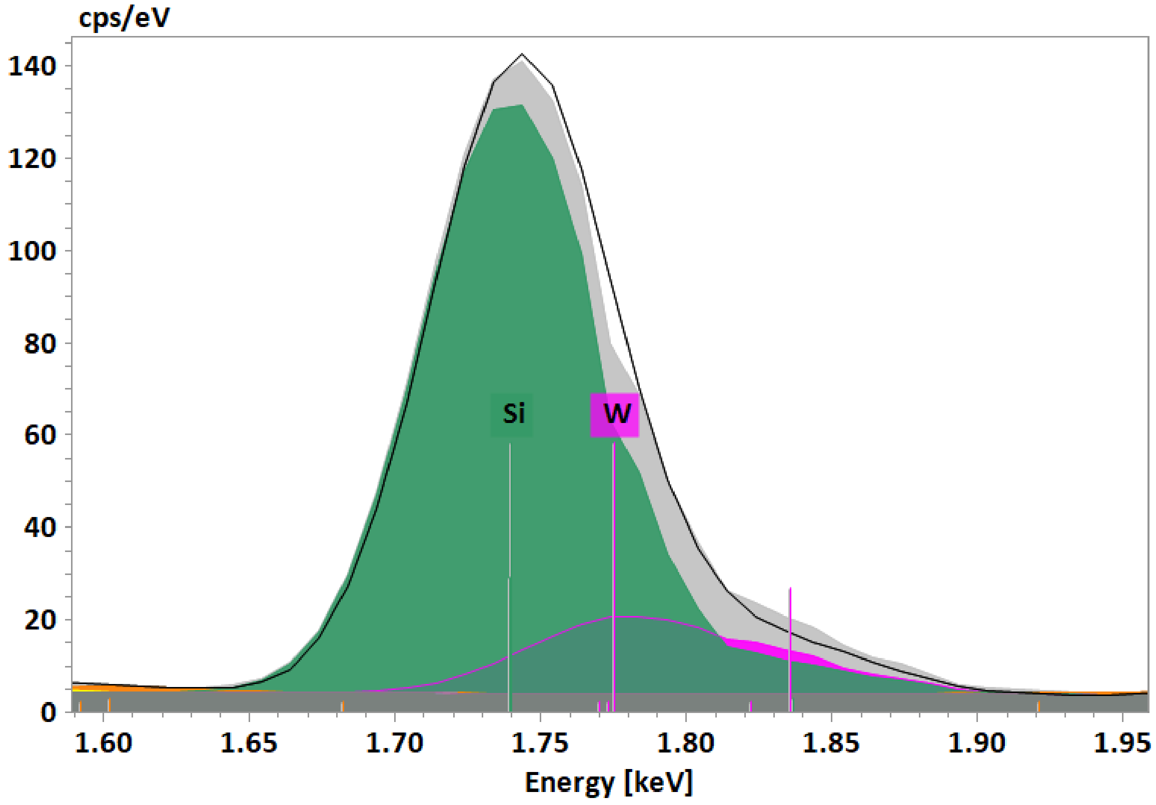


Application example: SSD

Quantified map in atomic %



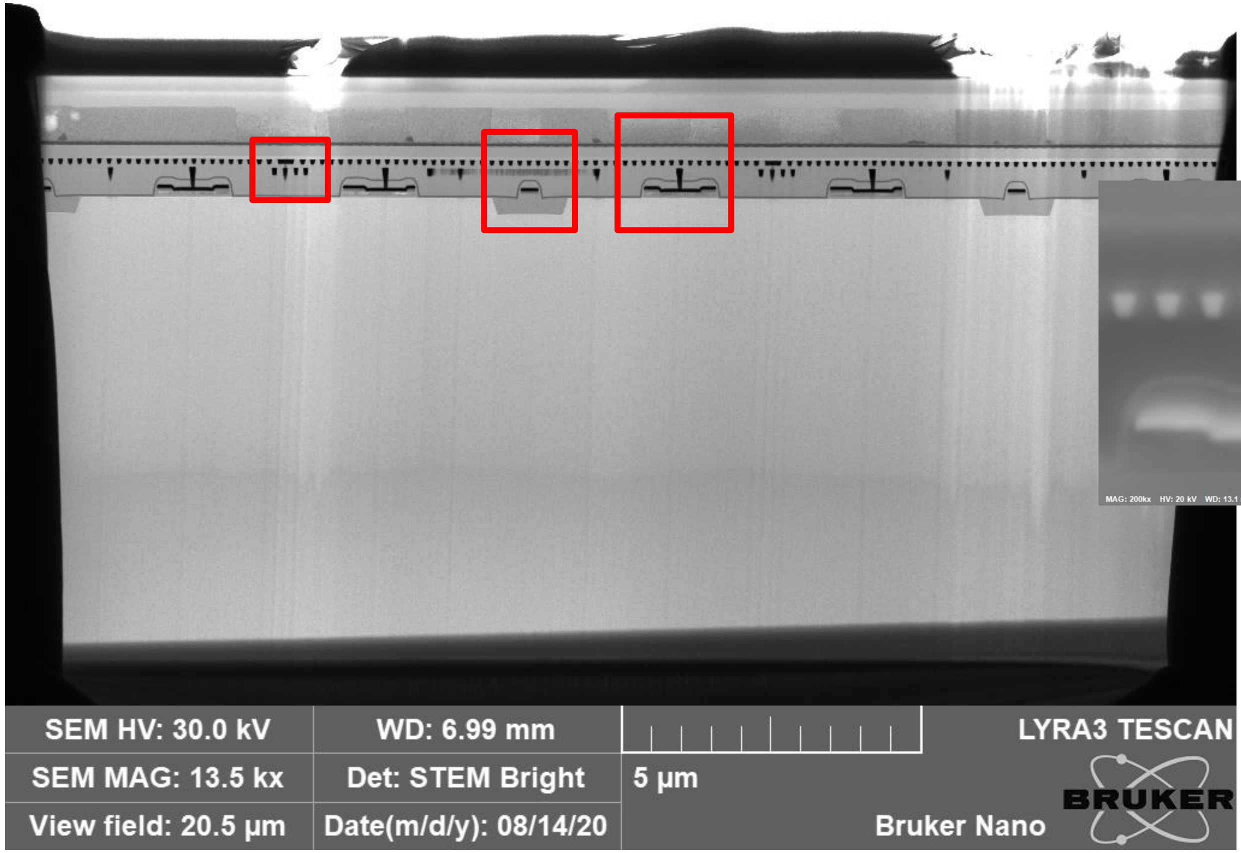


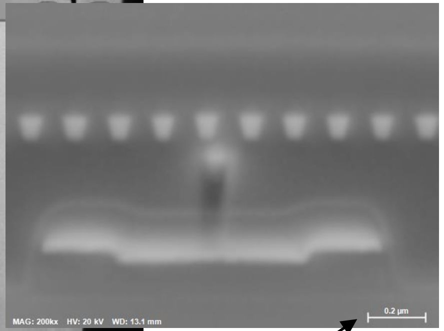


Si and W – automated deconvolution

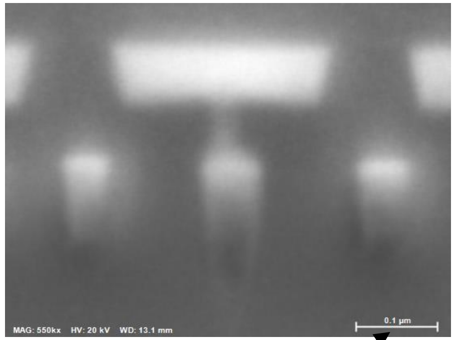
Application example: SSD-2

Lamella #2





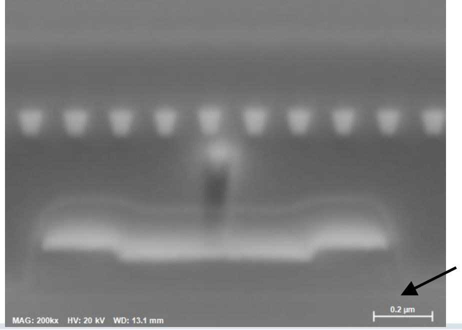
200 nm



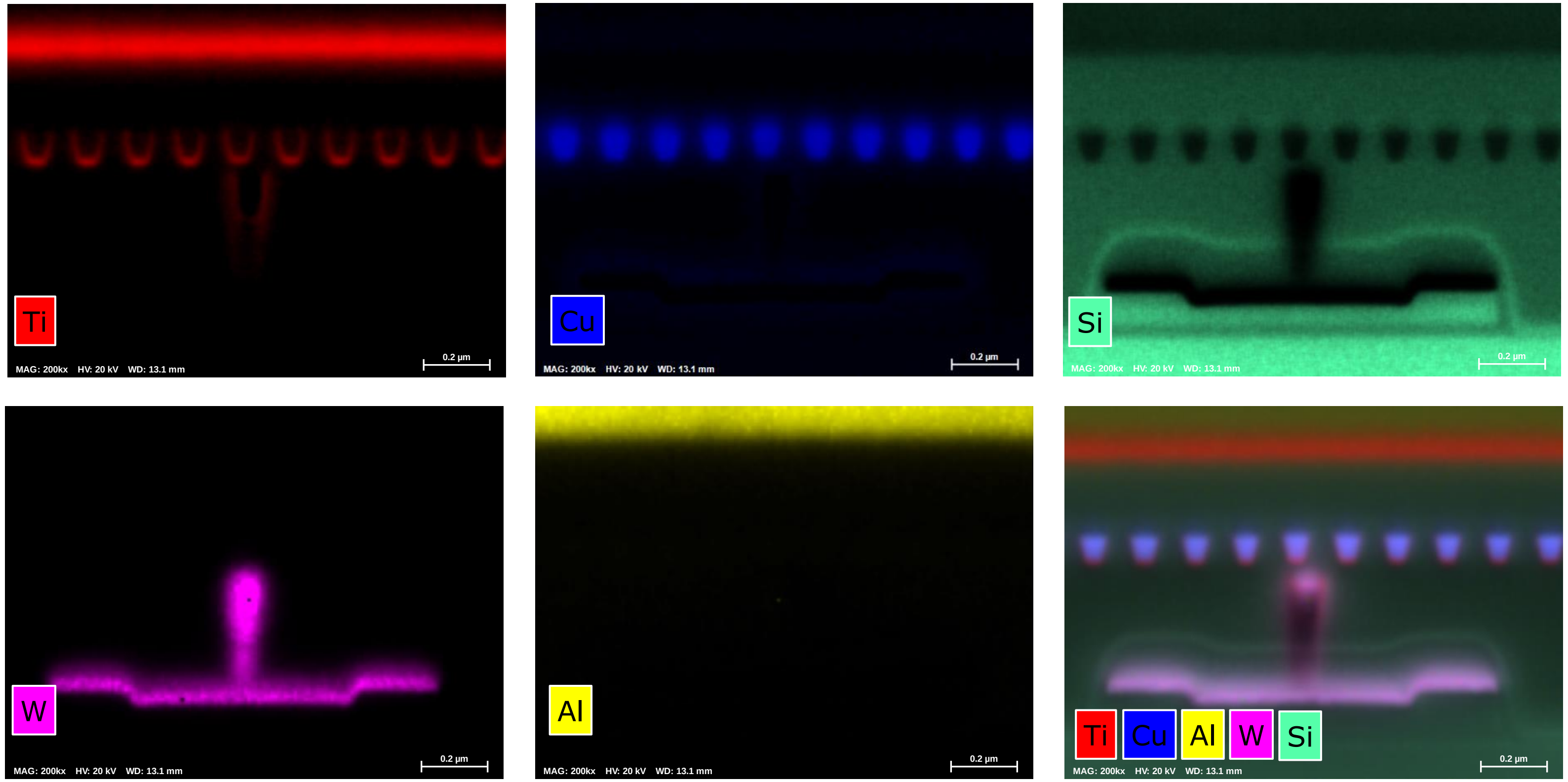
100 nm

200 nm

Application example: SSD-2
Quantified map in atomic %



200 nm



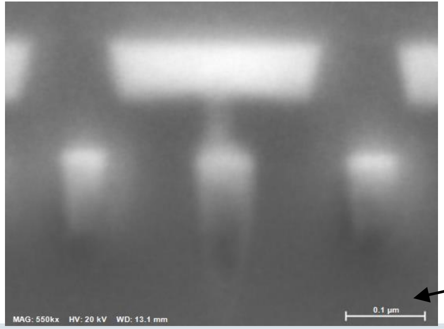
EDS mapping parameters:

- High voltage: 20 kV
- Abs. current: 350 pA
- WD: 13.1 mm
- **Mag: 200,000 X**
- Map time: 20 m
- Input counts: 298.2 Kcps
- Output counts: 170 Kcps
- **Total counts: 2.02E+08**

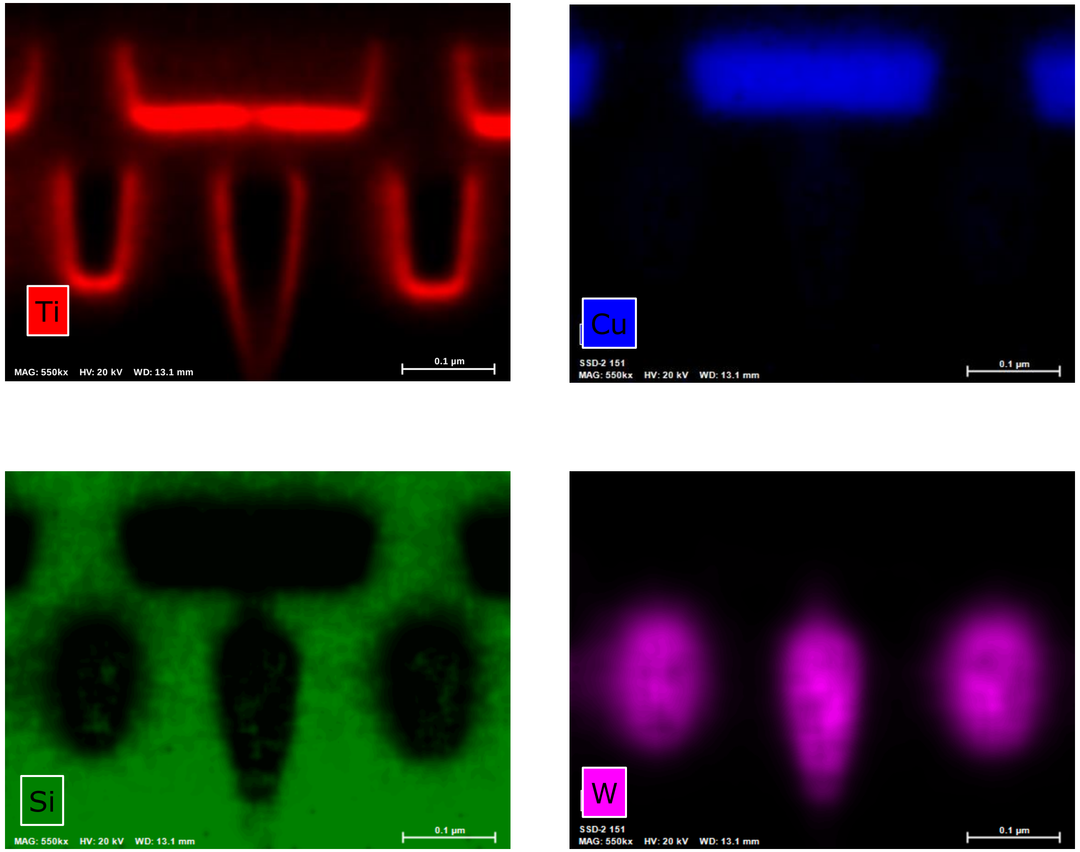
(0.2 B)

Application example: SSD-2

EDS mapping at high magnification in SEM



100 nm



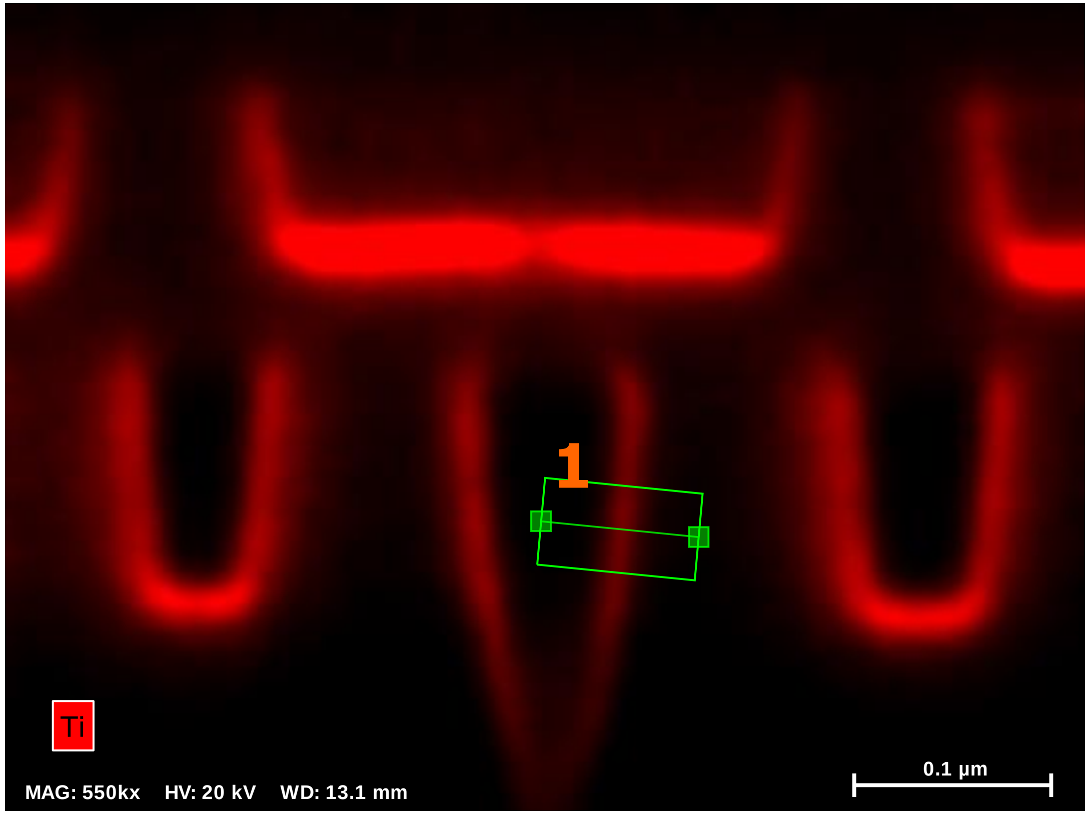
Quantified map in atomic %

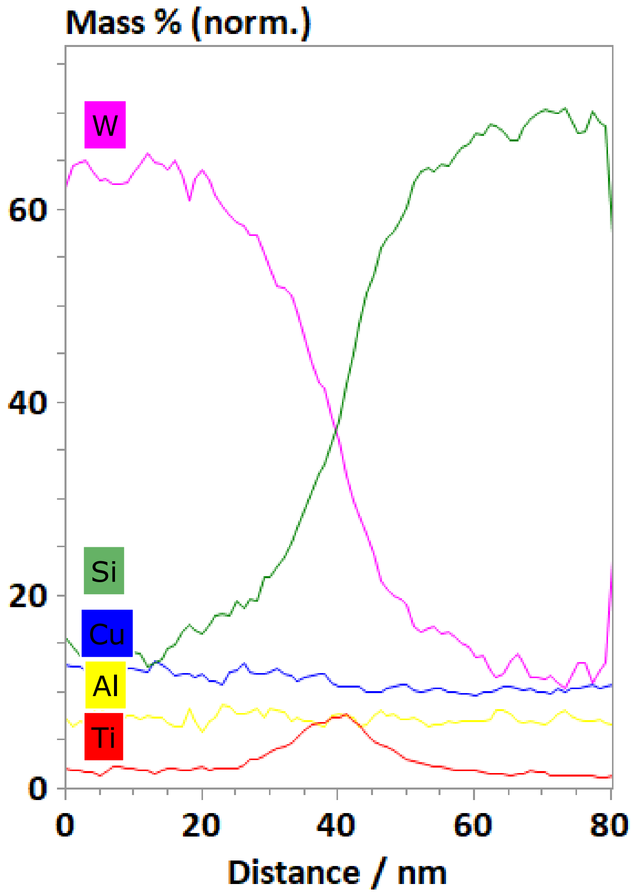
EDS mapping parameters:

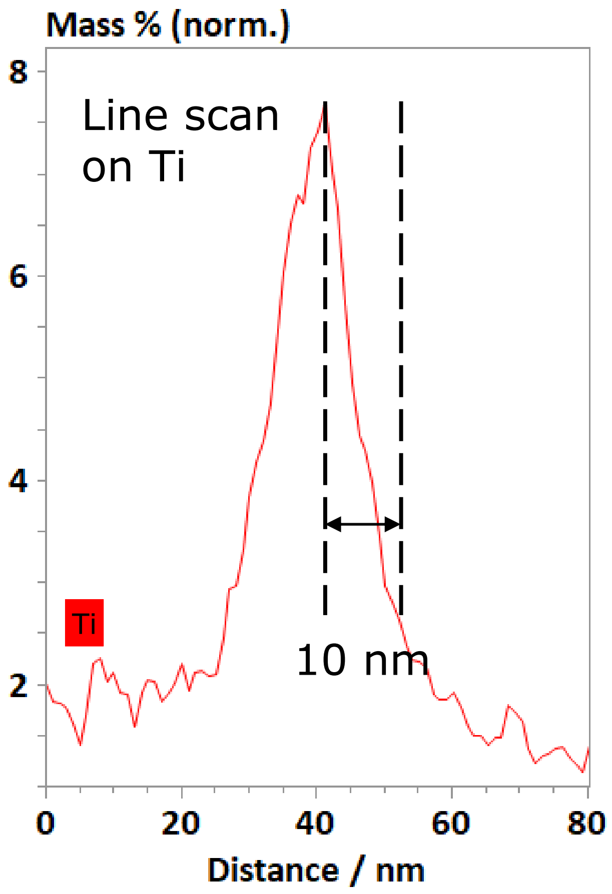
- High voltage: 20 kV
- Abs. current: 350 pA
- WD: 13.1 mm
- **Mag: 550,000 X**
- **Map time: 6 m**
- Input counts: 366.6 Kcps
- Output counts: 180.5 Kcps
- **Total counts: 7.28E+07 (70 M)**

Application example: SSD-2

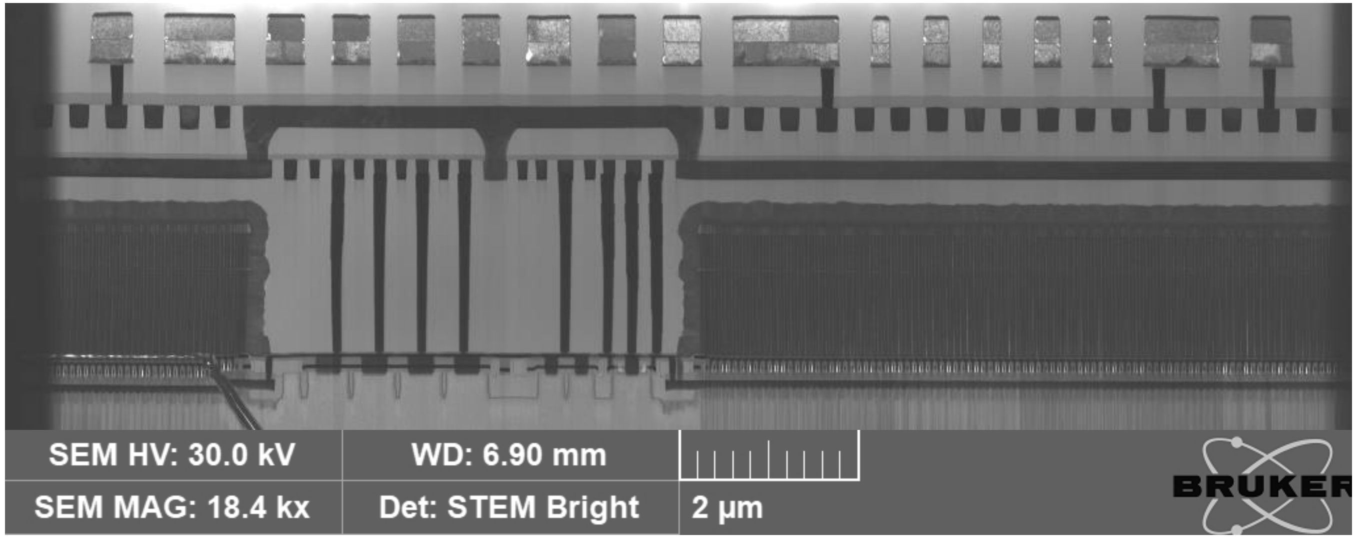
EDS mapping – Line scan



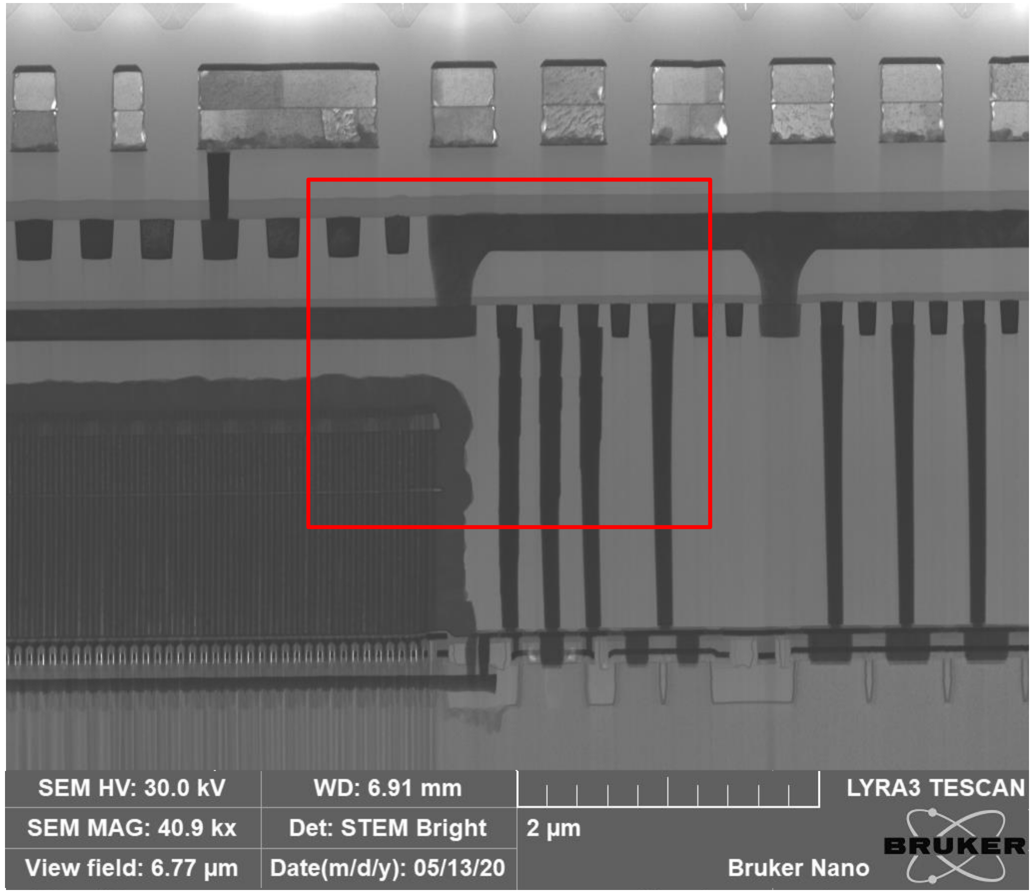


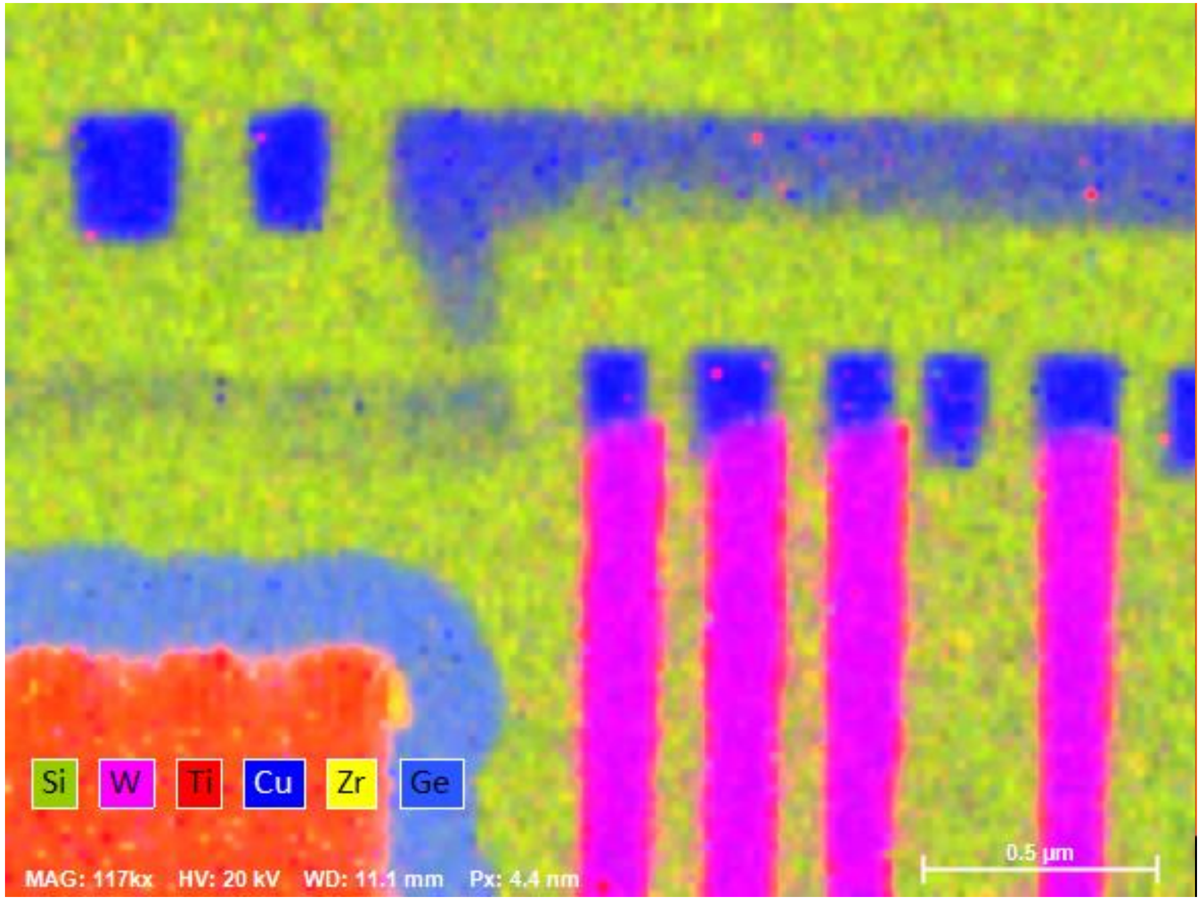


Application example: Microprocessor



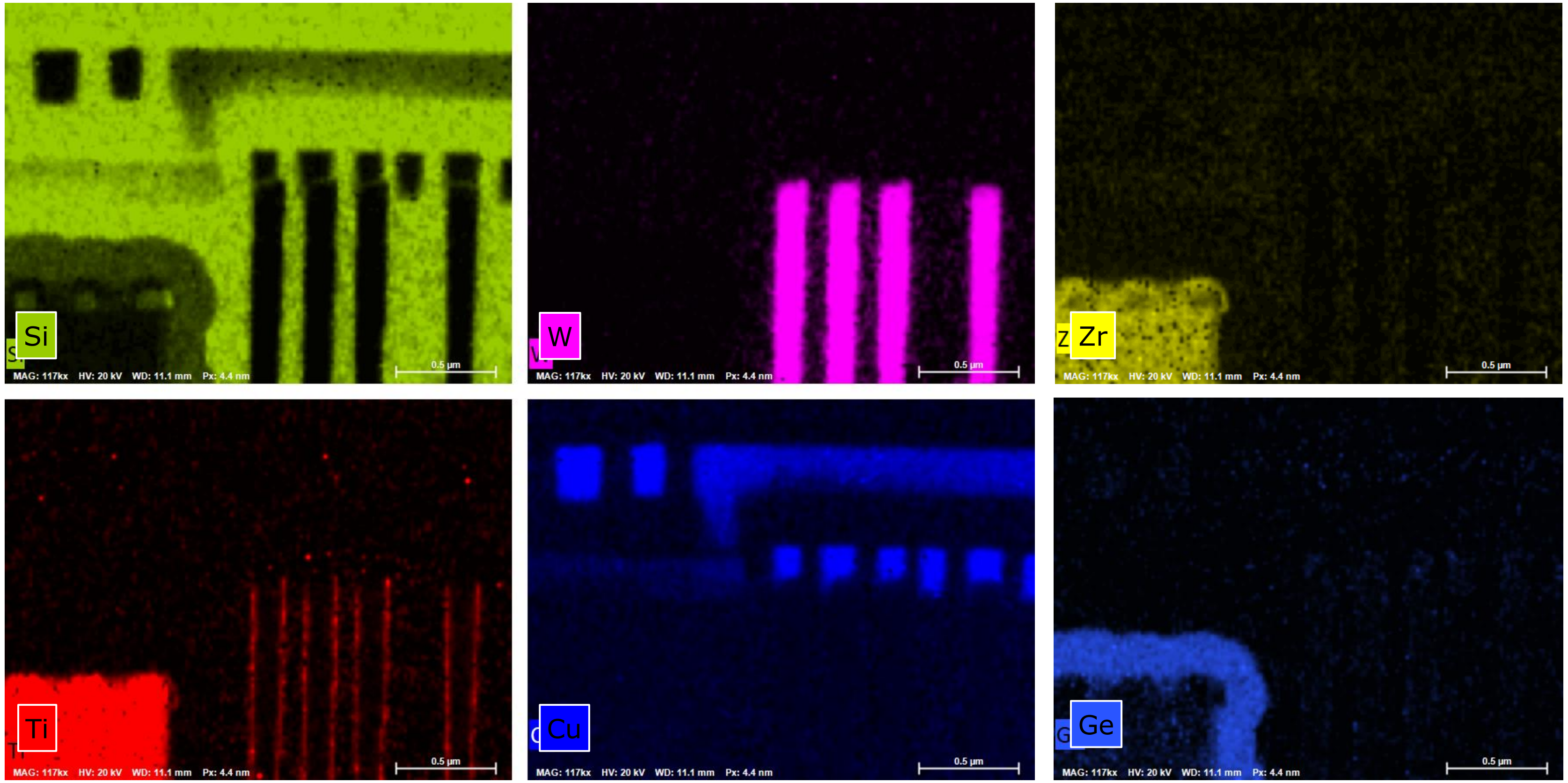
Application example: Microprocessor
EDS mapping – Quantified map in atomic %





Application example: Microprocessor

EDS mapping

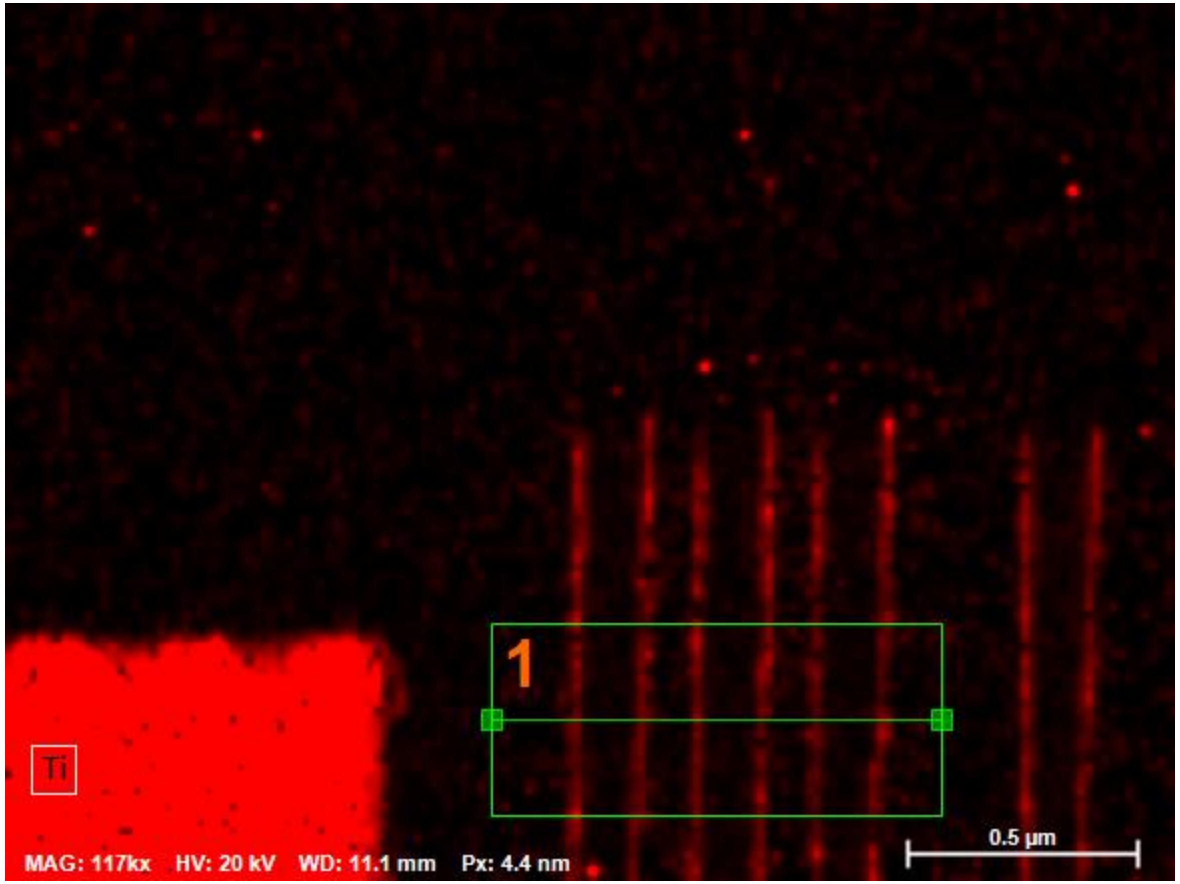


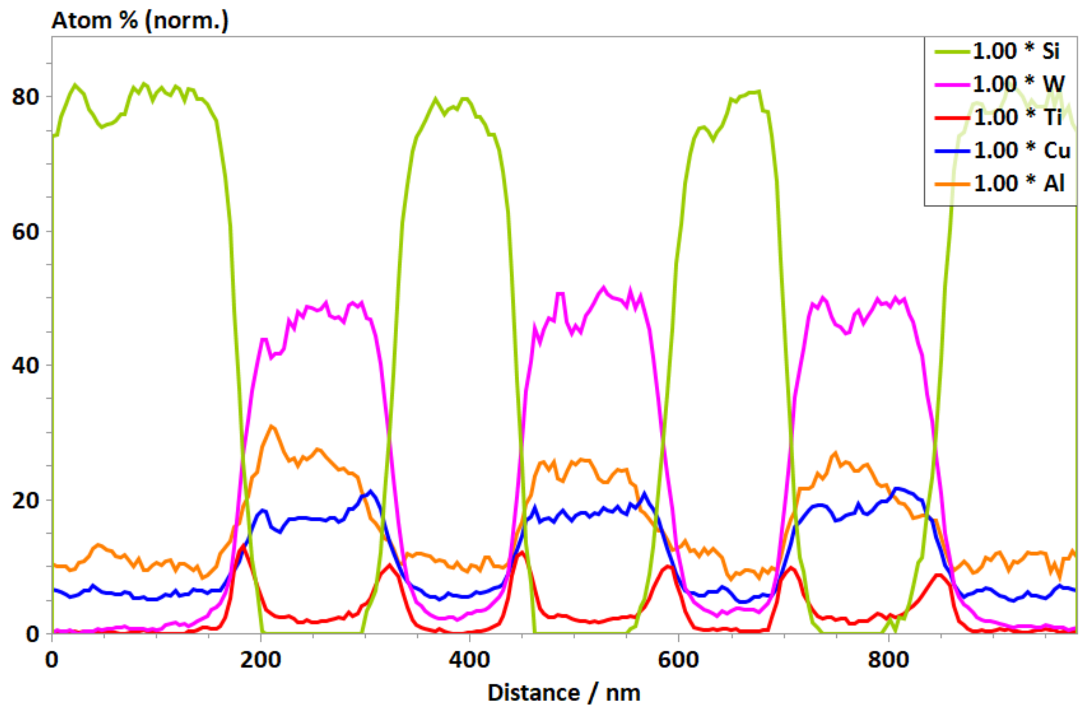
EDS mapping parameters:

- High voltage: 20 kV
- Abs. current: 350 pA
- WD: 11.1 mm
- Mag: 177,000 X
- Map time: 2 m
- Input counts: 64.0 Kcps
- Output counts: 53.2 Kcps
- Total counts: 6.838E+06 (6.8 M)

Application example: Microprocessor

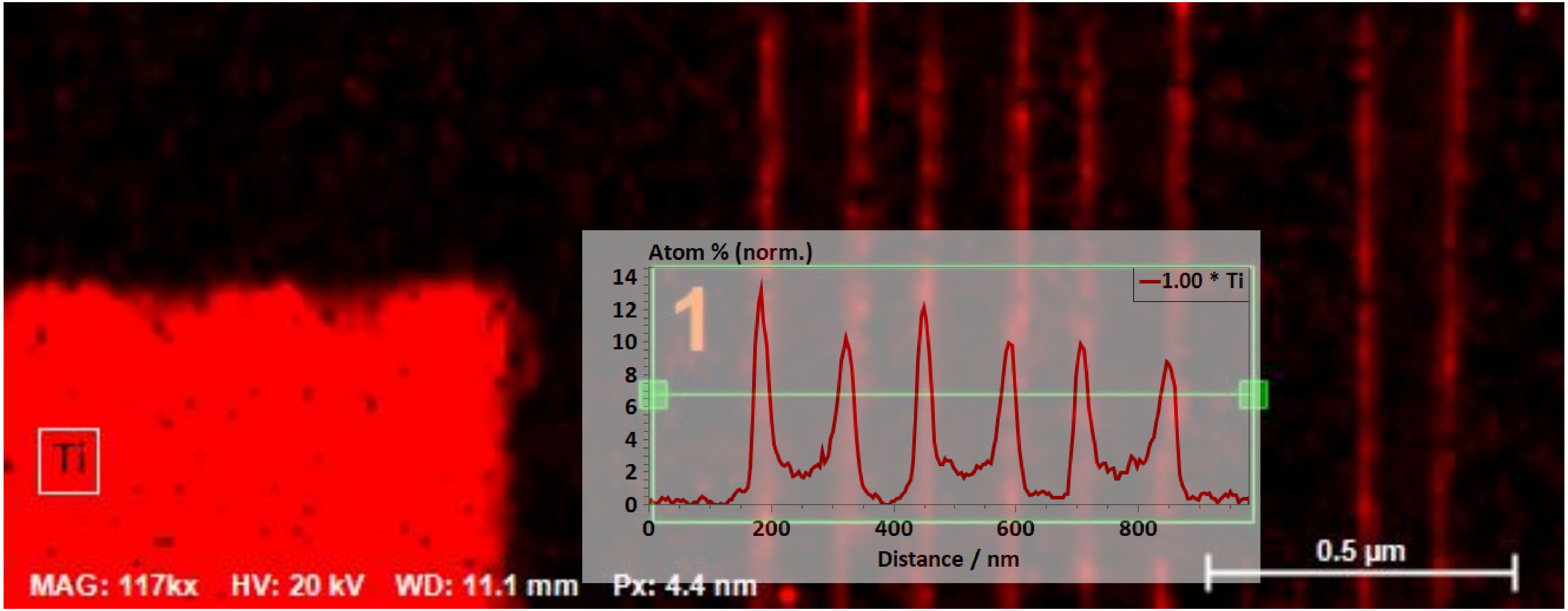
EDS Line scan – Quantified line scan in Atomic %





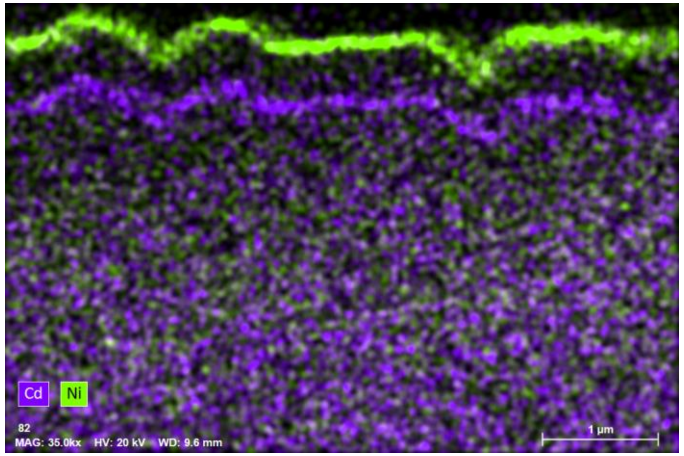
Application example: Microprocessor

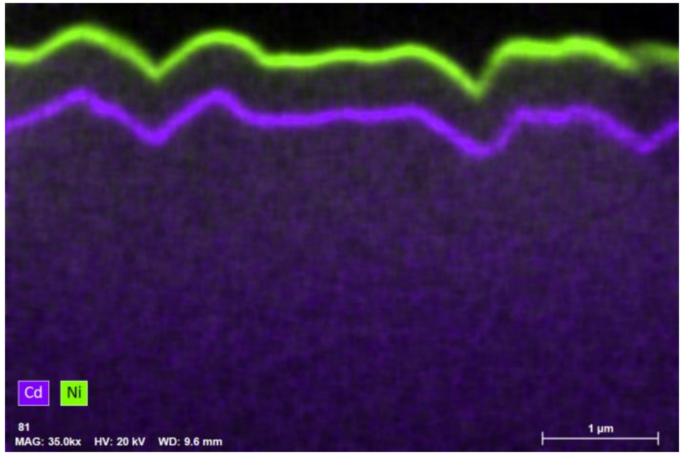
EDS Line scan - overlap



Conventional detector vs. FlatQUAD

FQ ideal for thin lamellae, nanoparticles and low x-ray yield samples
20 kV, 35000 X, WD 9.6 mm, 410 pA





Conventional EDS detector:

Size/solid angle: 60 mm², 0.0253 sr

Map time: 300 s

Total counts: 2.4E06 (2.4 M)

Input counts: 7.7 kcps

Output counts: 7.6 kcps

FlatQUAD:

Size/solid angle: 60 mm², 0.95 sr

Map time: 300 s

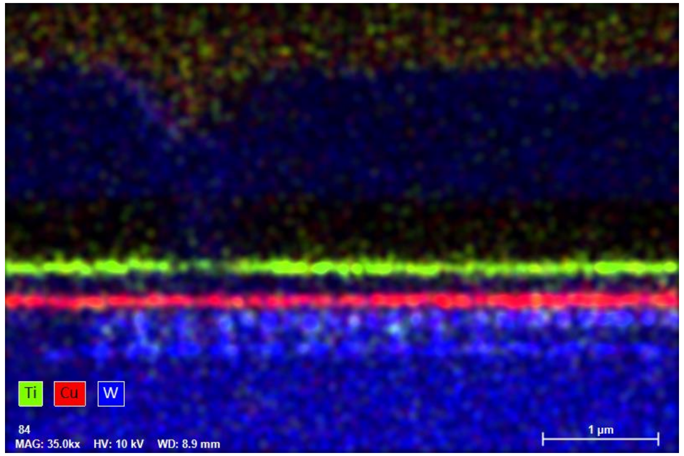
Total counts: 3.9E07 (39 M)

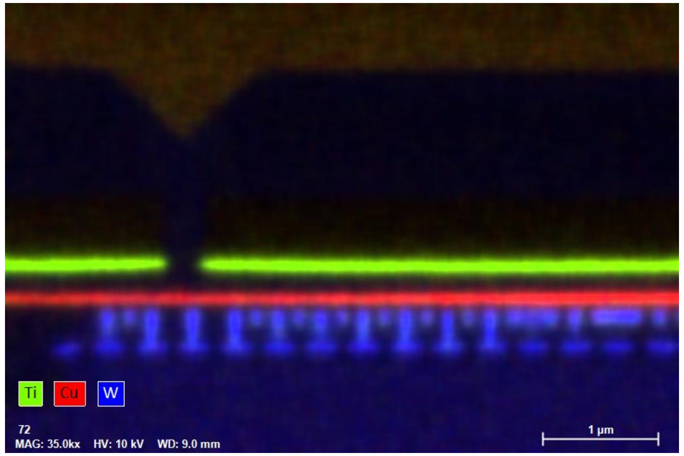
Input counts: 218.8 kcps

Output counts: 124.9 kcps

Conventional detector vs. FlatQUAD

FQ ideal for thin lamellae, nanoparticles and low x-ray yield samples
10 kV, 35000 X, WD 9.0 mm, 280 pA





Conventional EDS detector:

Size/solid angle: 60 mm², 0.0253 sr

Map time: 300 s

Total counts: 6.2E05 (0.62 M)

Input counts: 2.02 kcps

Output counts: 2.02 kcps

FlatQUAD:

Size/solid angle: 60 mm², 0.95 sr

Map time: 300 s

Total counts: 1.08E07 (10.8 M)

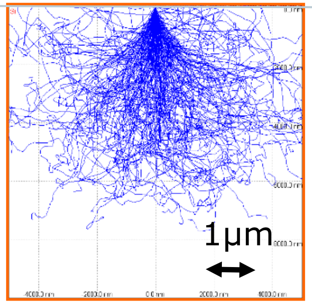
Input counts: 86.8 kcps

Output counts: 36.1 kcps

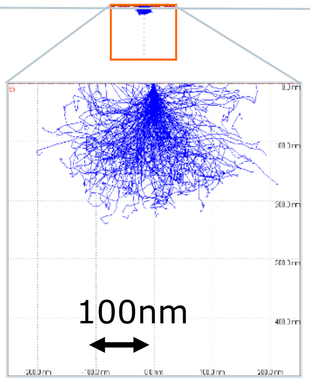
Spatial Resolution for Bulk and Electron Transparent Specimens

SEM: bulk

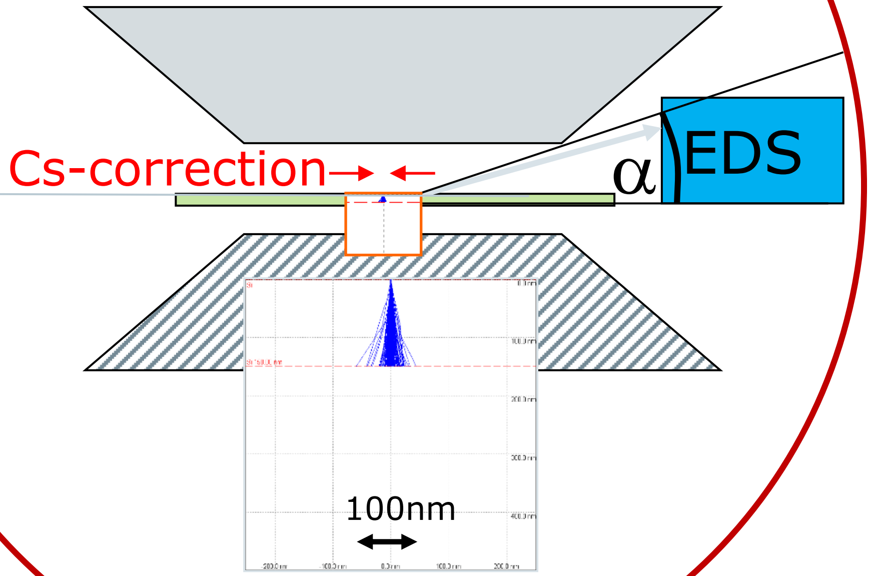
High voltage
30kV



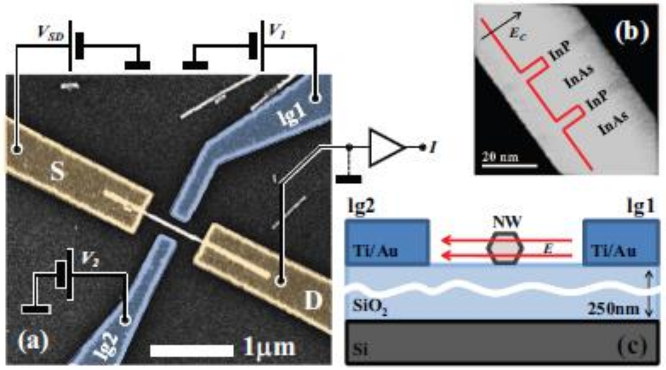
Lower voltage
4kV



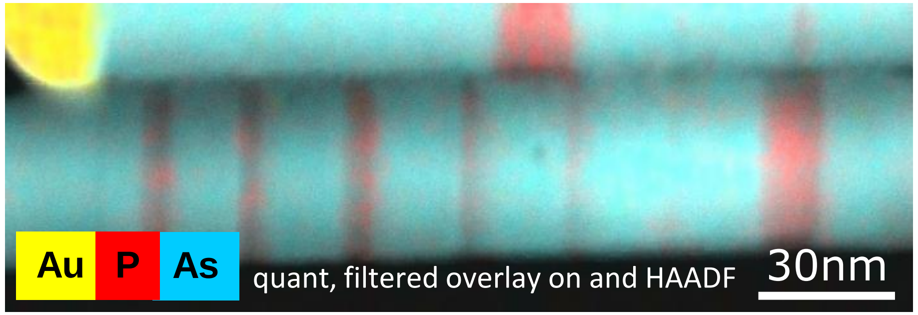
S/TEM, T-SEM: thin specimen,
small probe

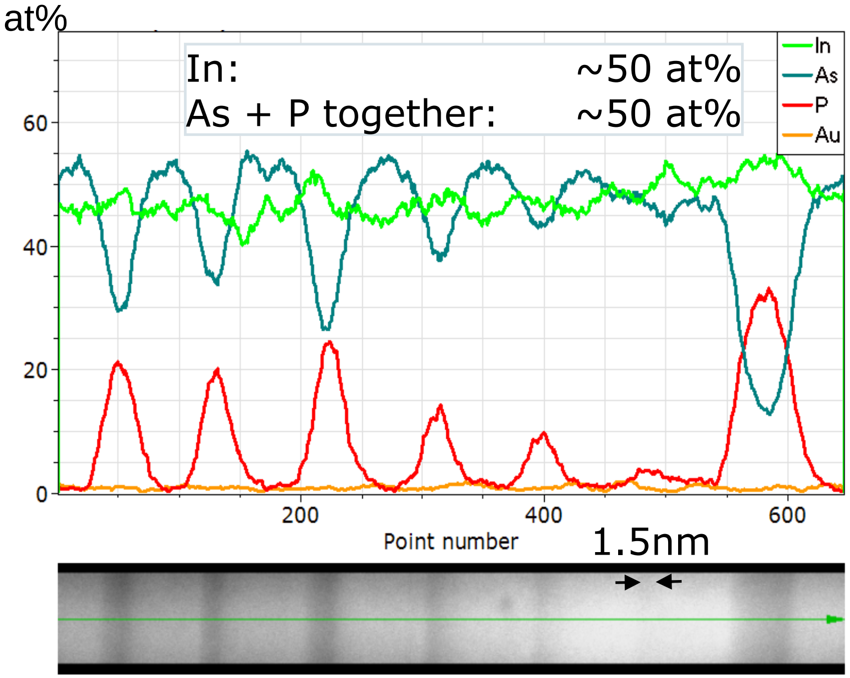


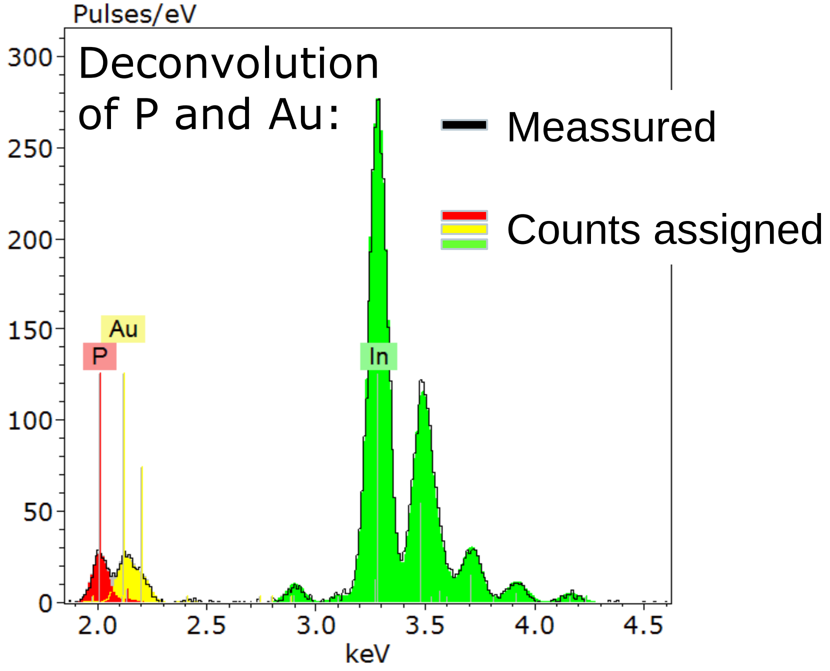
ED(X)S With 30mm² Standard EDS Detectors Using Standard STEM; Collection Angle: ~0.1sr; Cliff-Lorimer Quantification



Nanorods for Single e⁻ transistor:

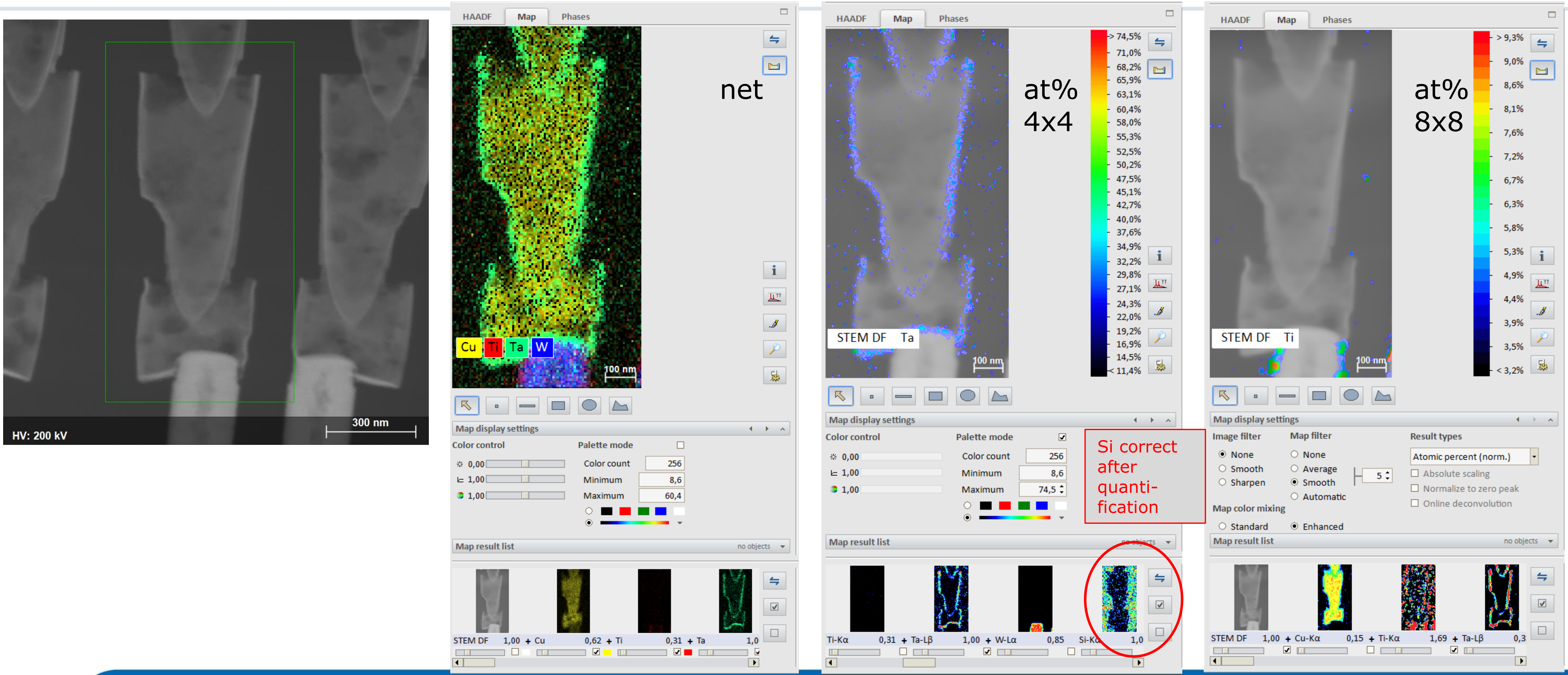






Courtesy: Specimen provided by Daniele Ercolani, Lucia Sorba et al.

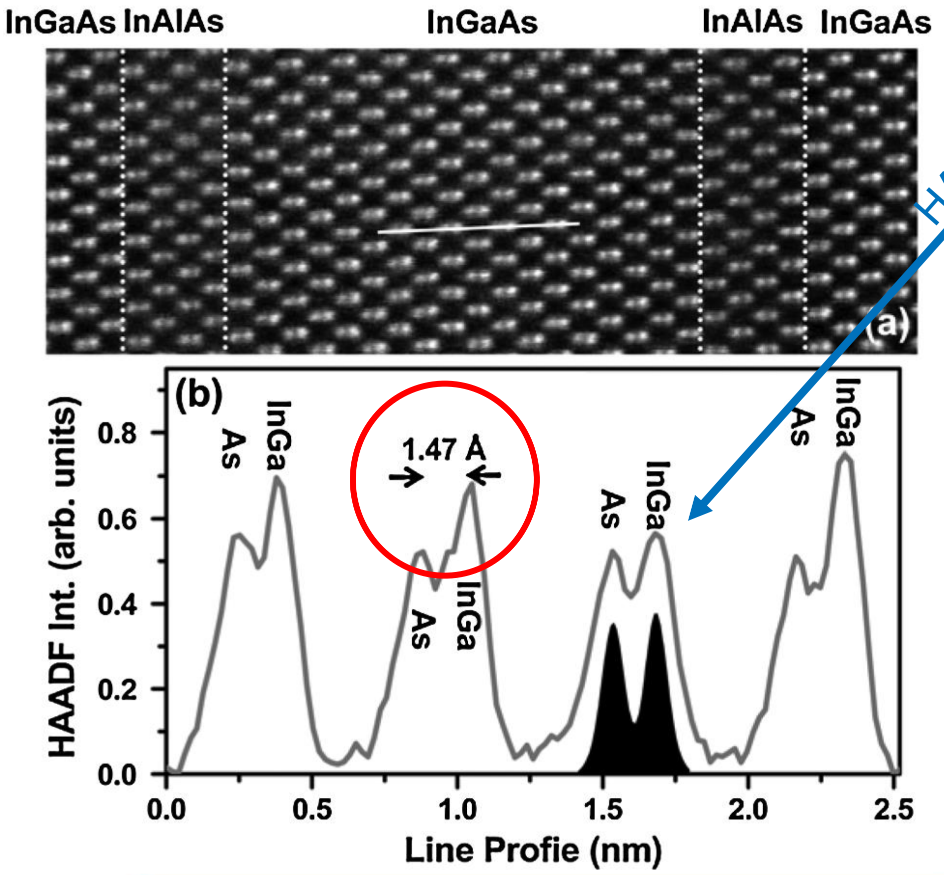
ED(X)S With 30mm² Standard EDS Detectors Using Standard STEM; Collection Angle: ~0.1sr; Cliff-Lorimer Quantification



Courtesy: Specimen provided by Synergie4

ED(X)S With 30mm² Standard EDS Detectors Using Cs-corrected STEM; Collection Angle: ~0.1sr

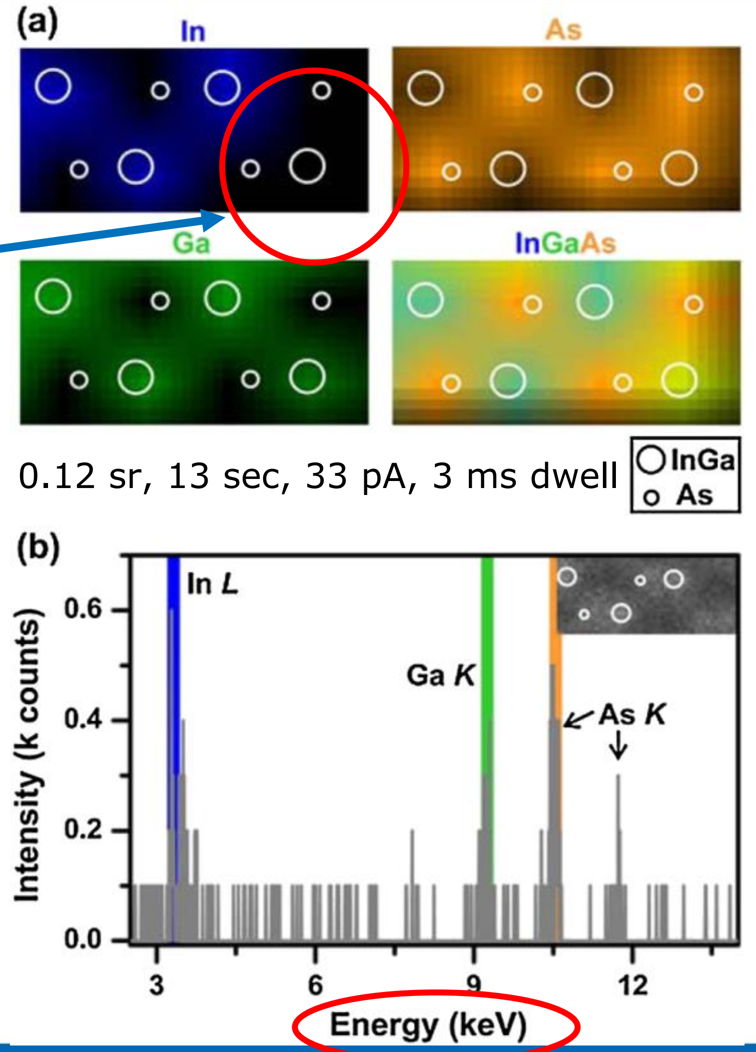
First atom column mapping using SDD



HAADF signal

Indium missing in one atomic column

EDS



Forbes, … Mc Comb et al.: Simulation needed, to correctly interpret atom column EDS!

M. W. Chu et al., Phys. Rev. Lett. 104, 196101 (2010)

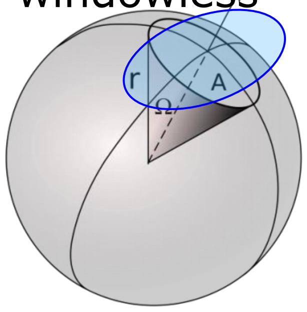
100 mm^2 Oval Detector Area, Windowless:
XFlash®T 100 oval

EDXS from 0.4 sr to 0.7 sr depending on geometry. This is the real solid angle for a flat SDD (see wiki below).

Wrong: $100mm^2 / (10.5mm)^2 = 0.91sr$; Correct: 0.65sr

TOA: ~ 13°

windowless



wikipedia

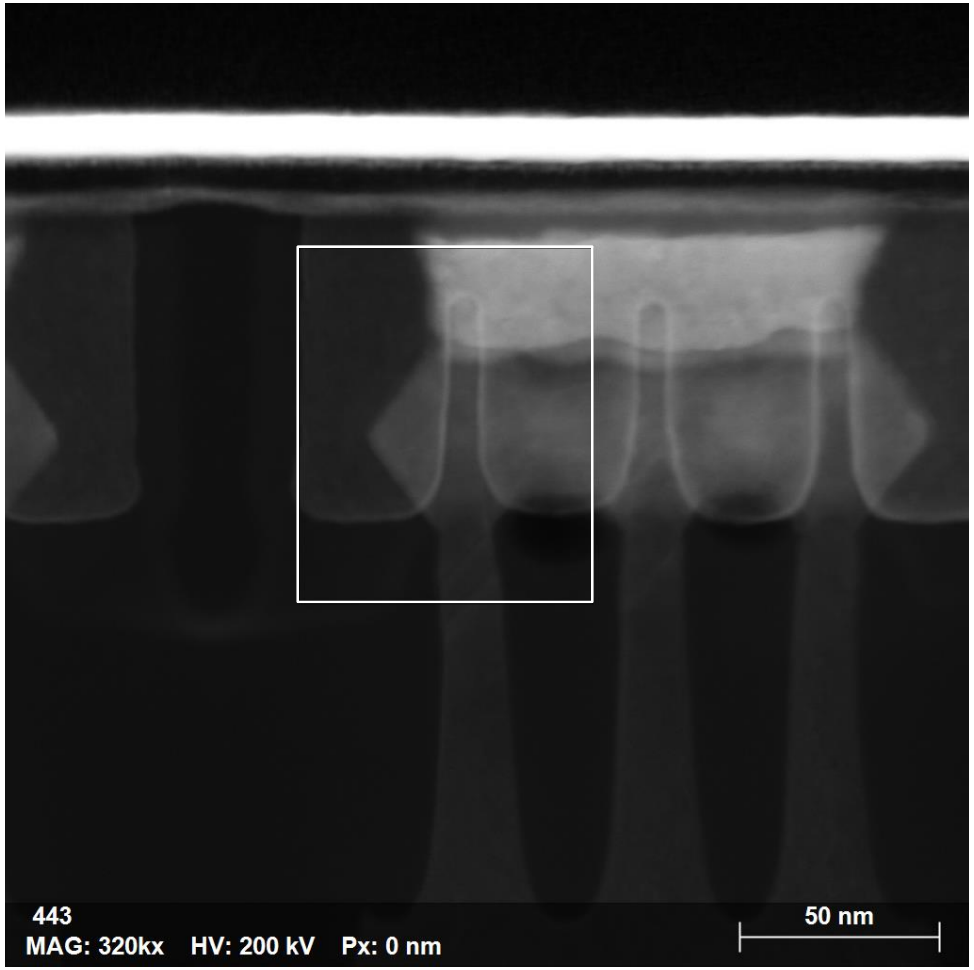
Single Atom ID:
*R. M. Stroud et al., APL **108**, 163101 (2016) open access*
*T. C. Lovejoy et al., APL **100**, 154101 (2012): 30mm², 0.1sr*

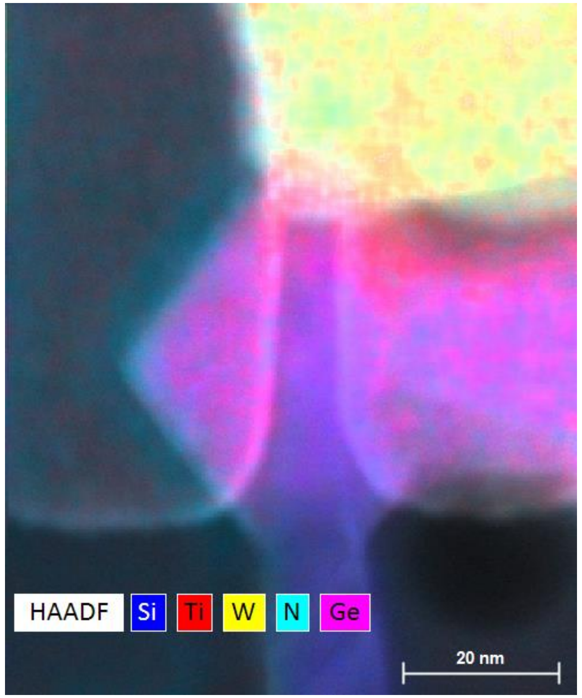
ED(X)S with 100mm² windowless oval detector area using Cs-corrected STEM; Collection angle: ~ 0.4sr

Raw data, acquisition time 24min

TFS Titan 80-300 with Bruker XFlash®T 100 oval EDS detector at ACE.

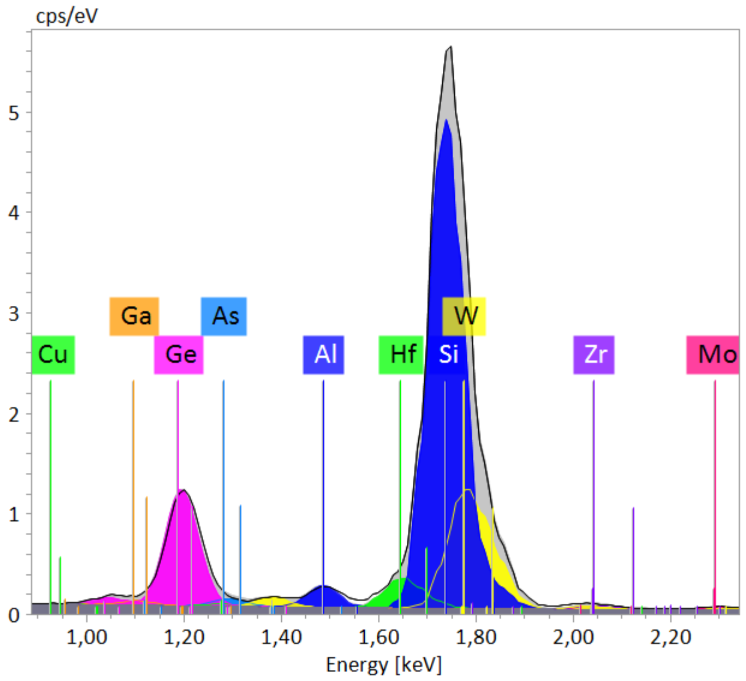
Quantitative element mapping of nanostructures.

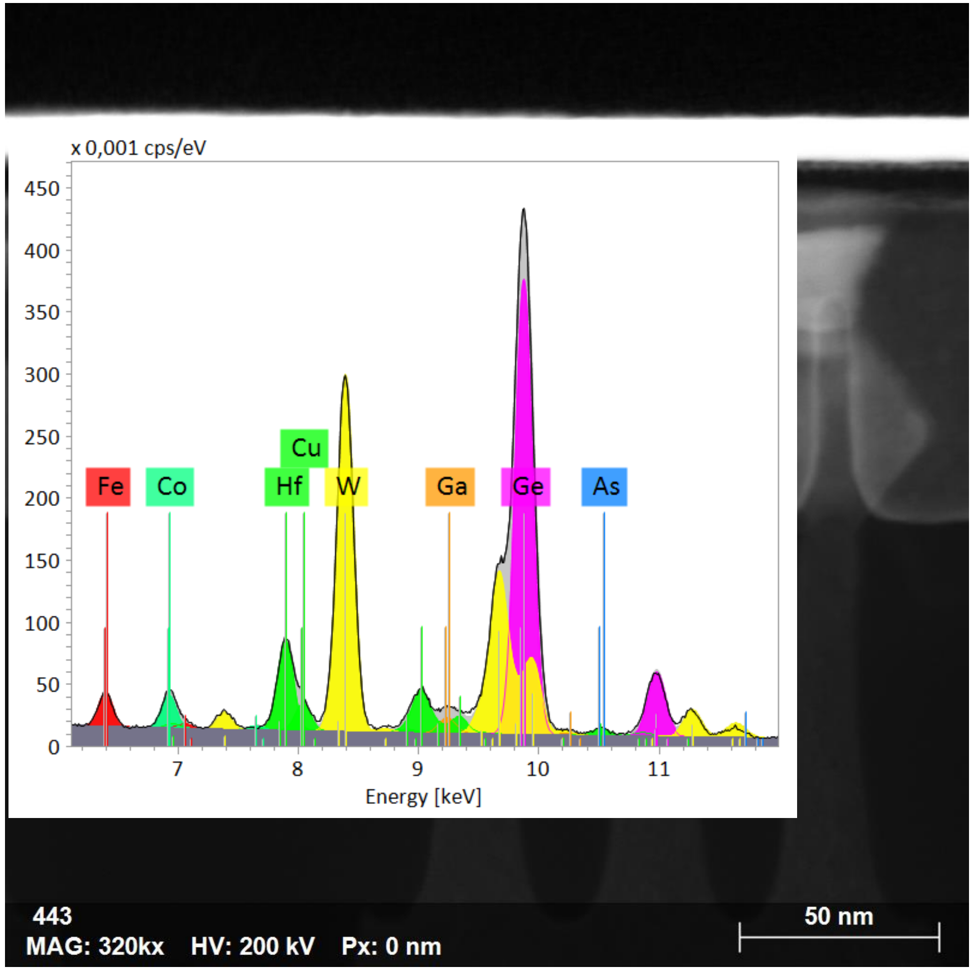


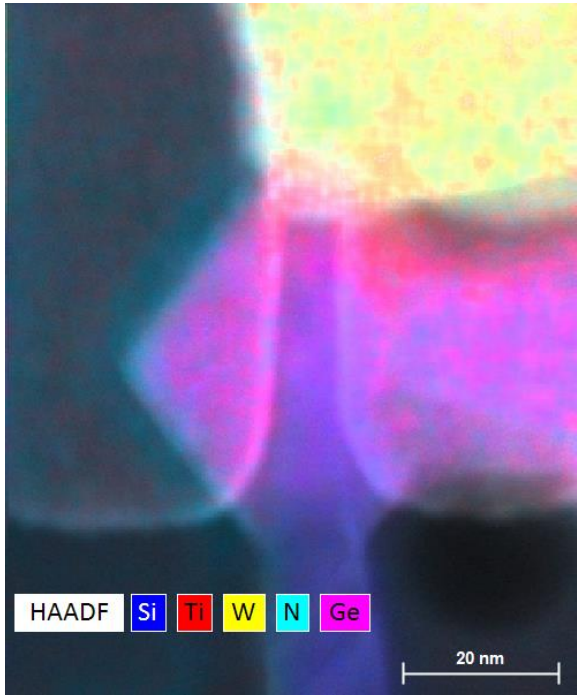


ED(X)S with 100mm² windowless oval detector area using Cs-corrected STEM; Collection angle: ~ 0.4sr

Raw data, acquisition time 24min





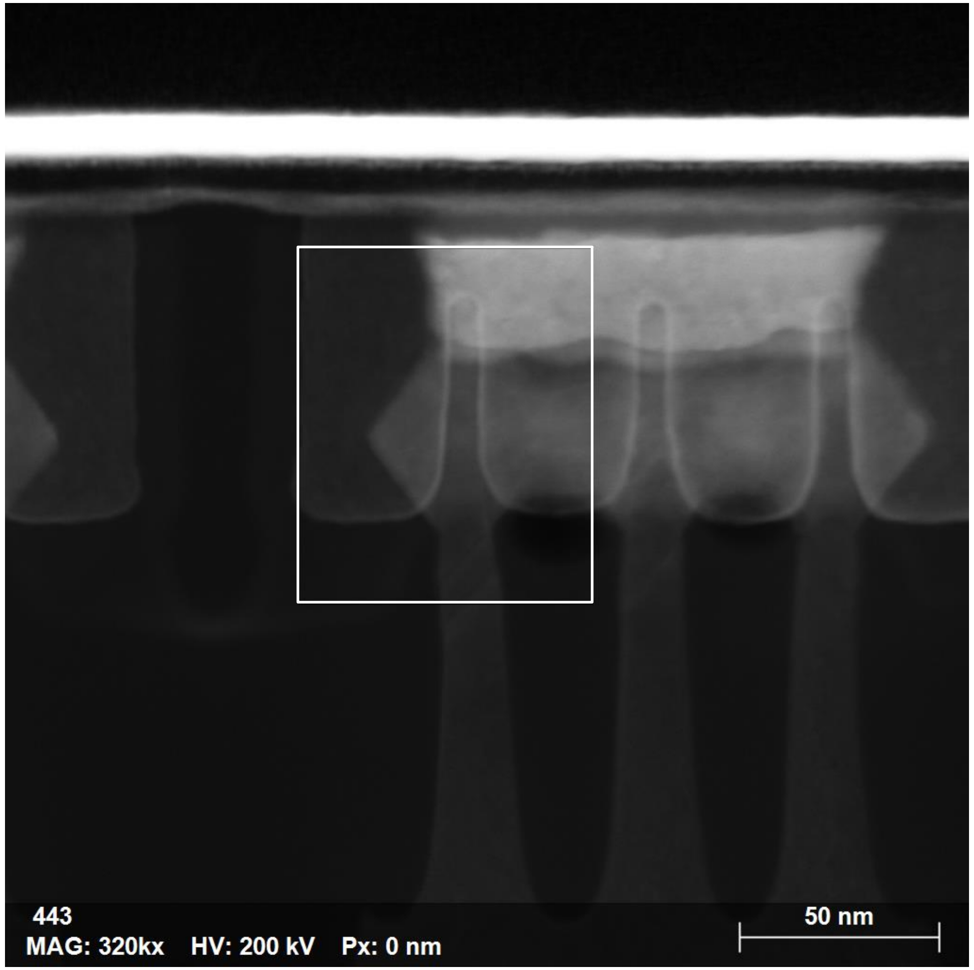


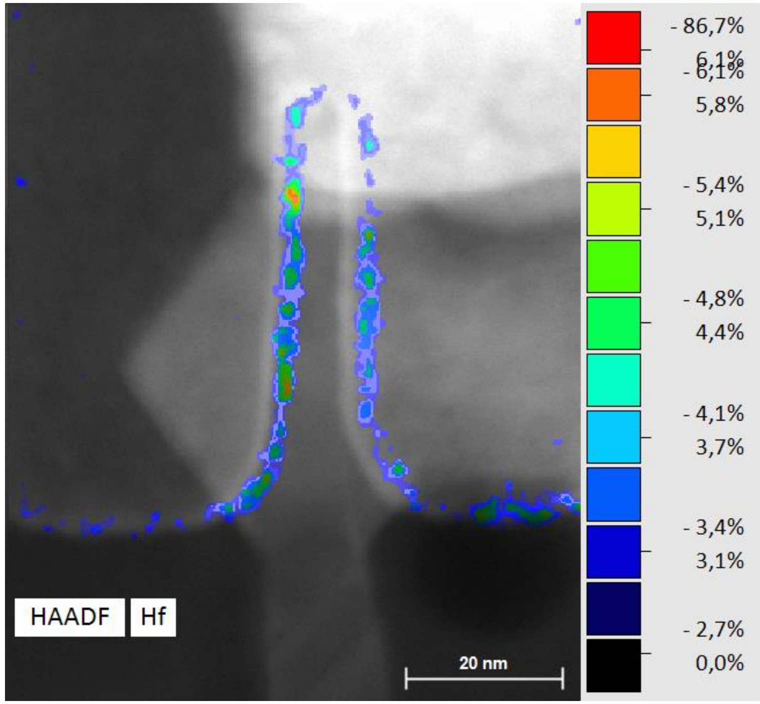
ED(X)S with 100mm² windowless oval detector area using Cs-corrected STEM; Collection angle: ~ 0.4sr

Cliff-Lorimer Quantification: at%

TFS Titan 80-300 with Bruker XFlash®T 100 oval EDS detector at ACE.

Quantitative element mapping of nanostructures.

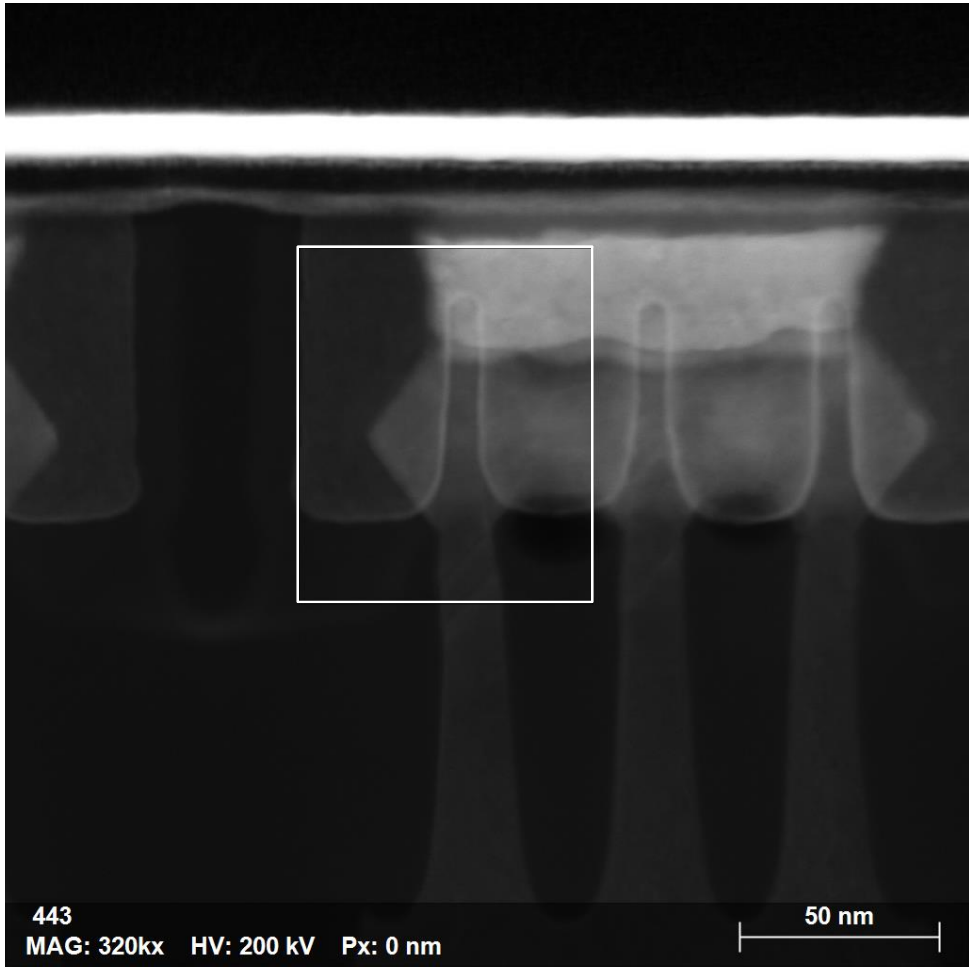


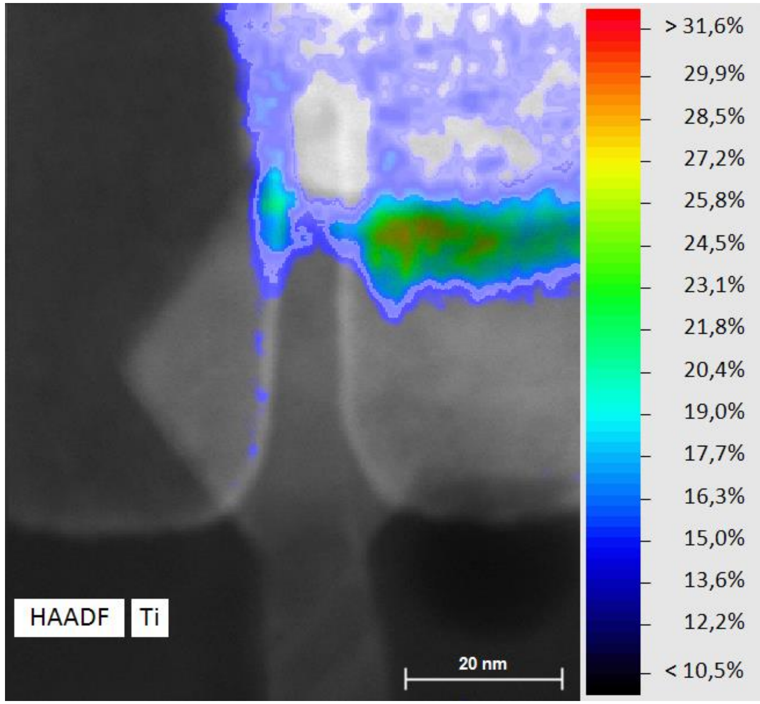


ED(X)S with 100mm² windowless oval detector area using Cs-corrected STEM; Collection angle: ~ 0.4sr
Cliff-Lorimer Quantification: at%

TFS Titan 80-300 with Bruker XFlash®T 100 oval EDS detector at ACE.

Quantitative element mapping of nanostructures.

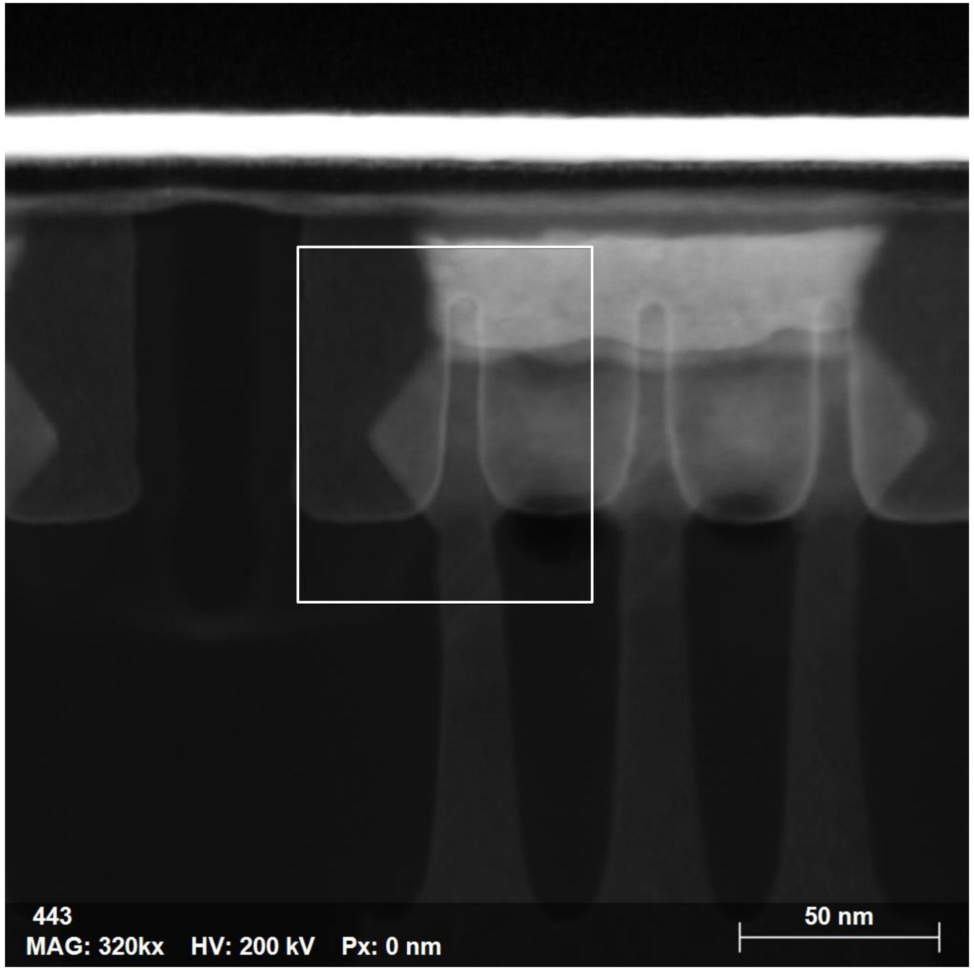


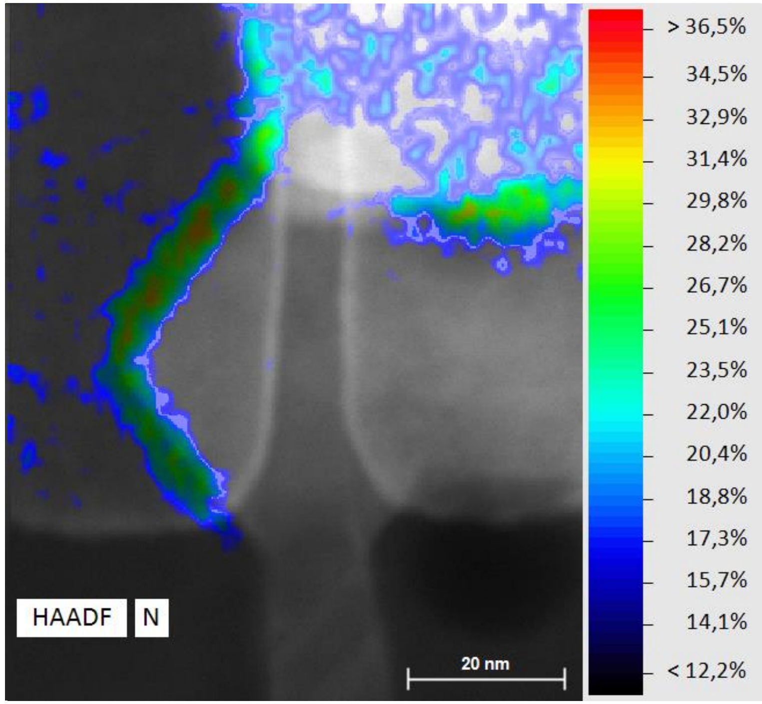


ED(X)S with 100mm² windowless oval detector area using Cs-corrected STEM; Collection angle: ~ 0.4sr
Cliff-Lorimer Quantification: at%

TFS Titan 80-300 with Bruker XFlash®T 100 oval EDS detector at ACE.

Quantitative element mapping of nanostructures.



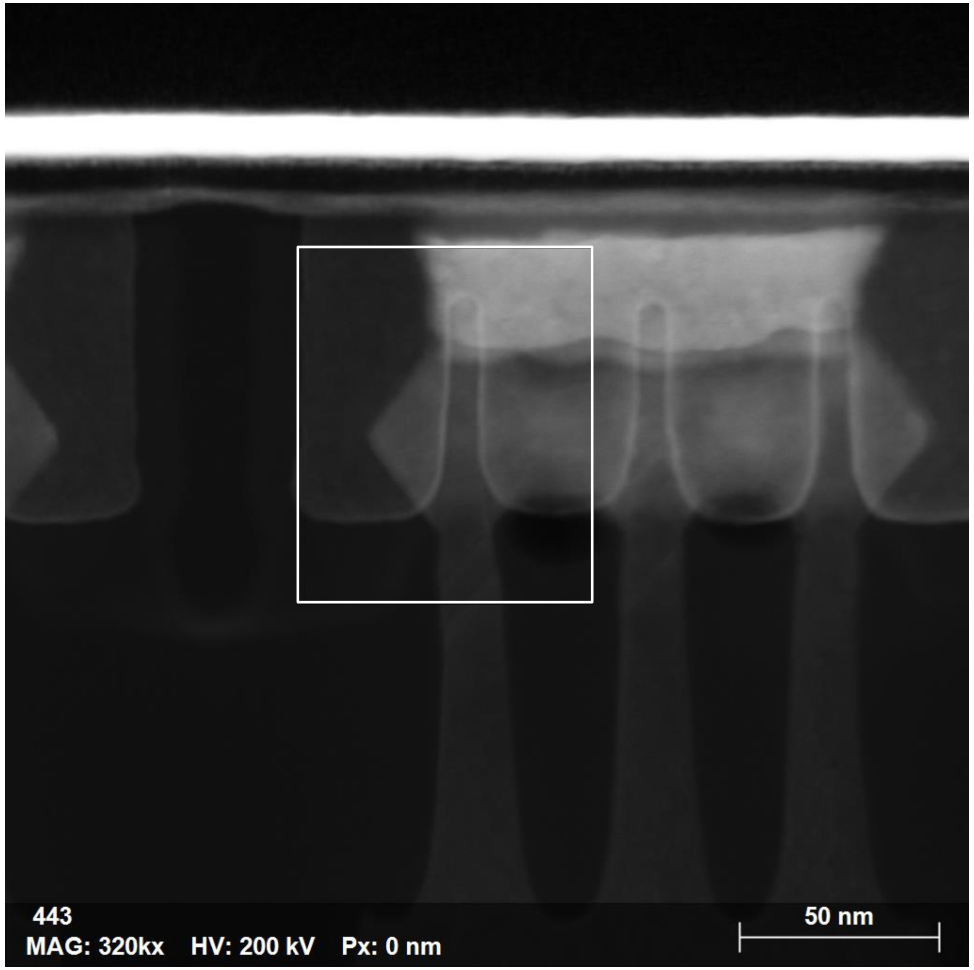


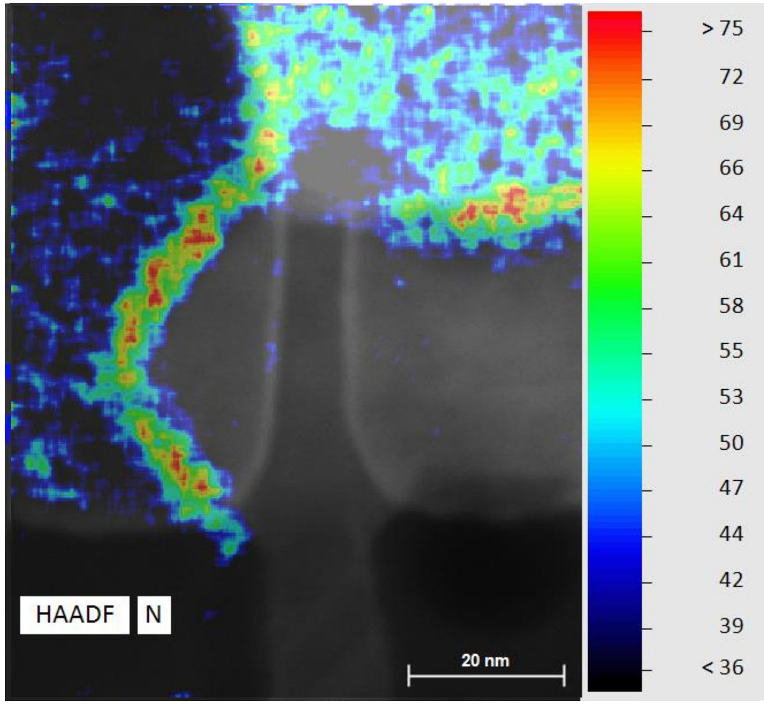
ED(X)S with 100mm² windowless oval detector area using Cs-corrected STEM; Collection angle: ~ 0.4sr

Raw data, acquisition time 24min, arbitr. units scaled to 100

TFS Titan 80-300 with Bruker XFlash®T 100 oval EDS detector at ACE.

Quantitative element mapping of nanostructures.





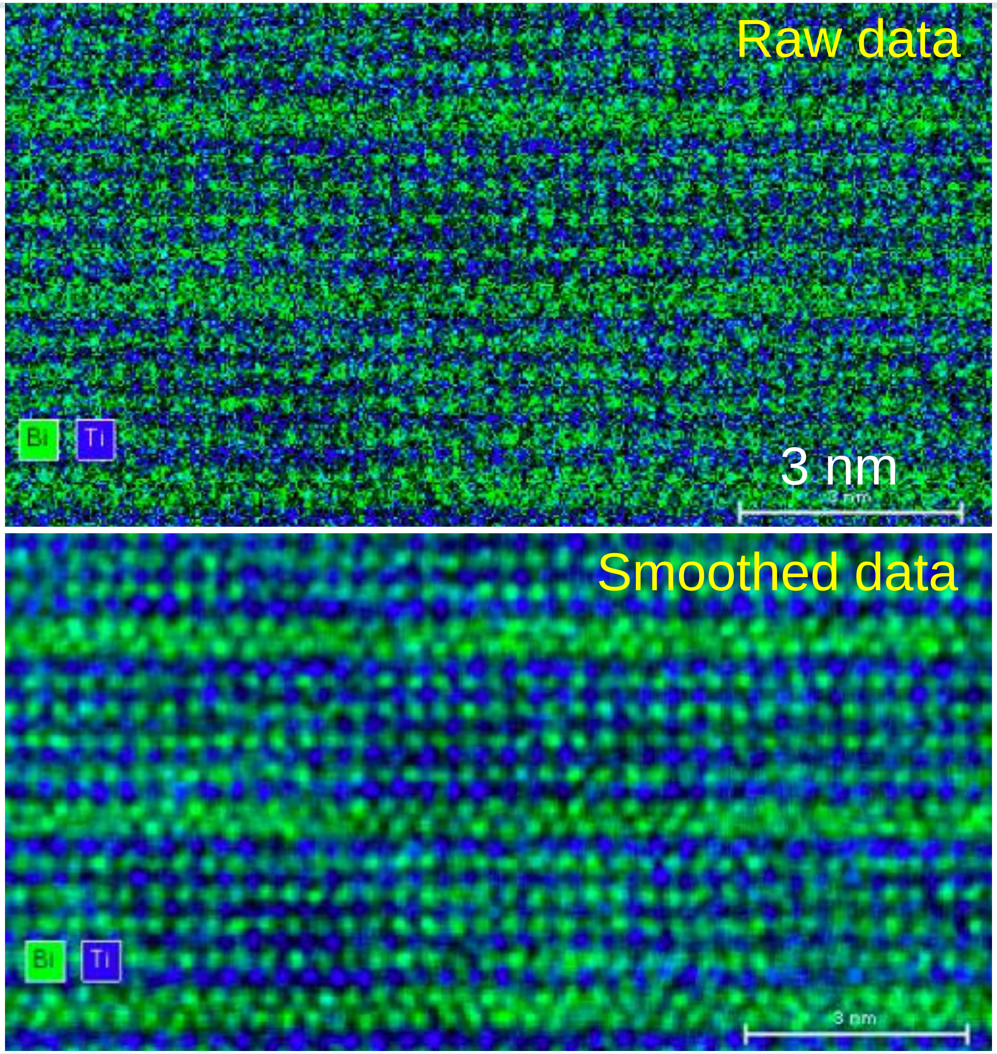
EDXS with 100 mm^2 windowless oval detector area;

Nion UltraSTEM, Cs-corrected, high brightness source (CFEG); ~0.7sr

NRL UltraSTEM200 with Bruker XFlash®T 100 oval EDS detector @ 200 kV.

Individua atom columns can be identified.

Specimen:
$Bi_6Ti_xFe_yMn_zO_{18}$
See:
"Direct atomic scale determination of magnetic ion partition in a room temperature multiferroic material", by L. Keeney et al.



TCD (Trinity College Dublin) Nion UltraSTEM200XE 200 kV with Bruker 100 mm^2 XFlash SD detector, 100 mm^2 windowless SDD; 0.7 sr collection angle.

432x225 pixels,
4.1 msec/pix => 400 sec for map.

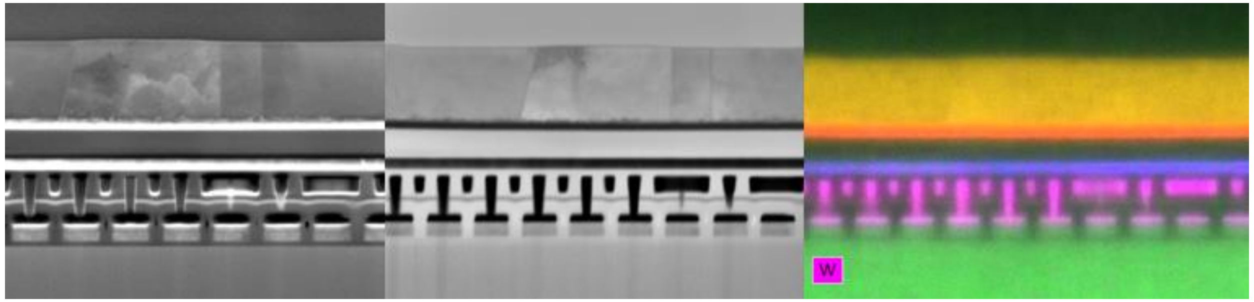
No drift correction.
Bi = green, Ti = blue.

courtesy Lynette Keeney, Clive Downing and Valeria Nicolosi. TCD, Ireland.

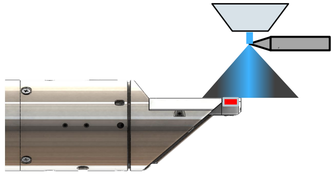
Atom column EDS needs simulation for correct interpretation
B. D. Forbes et al., PHYSICAL REVIEW B **86**, 024108 (2012)

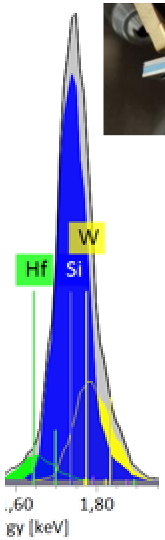
L. Keeney et al., Scientific Reports 7, Article number: 1737 (2017)

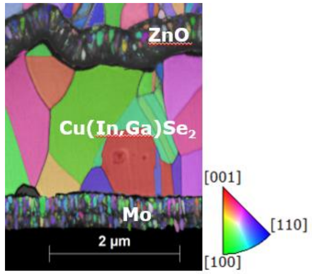
Summary

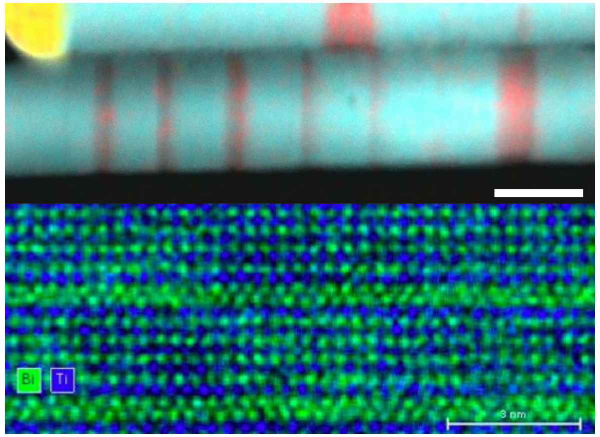


- SEM/FIB are valuable tools for the investigation of semiconductor lamellae using EDS > T-SEM.
- High collection angles and thus fast precise analysis can be achieved with the annular high solid angle XFlash® FlatQUAD detector.
- T-SEM invites for the combination with diffraction techniques, such as TKD, and with micro-XRF.
- Quantitative element analysis with EDS is possible via the relative Cliff-Lorimer and the absolute Zeta-factor method, also for SEM-typical voltages and specimens of considerable thickness.
- Few nm-resolution can be achieved routinely in standard STEM EDS, even with small detector areas (30mm2) at high collection angles.
- The combination of electron source type, Cs-correction and EDS geometry in STEM defines the availability of atomic resolution.









Q&A

Are there any questions?

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

Innovation with Integrity