Significance of STEM-EDXS Analysis in the Characterization of Rechargeable Battery Components

Guest speaker: Michael Malaki

Significance of STEM-EDXS Analysis in the Characterization of Rechargeable Battery Components

Dr. Igor Németh
Application Scientist EDS
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Outline

- EDS instrumentation for battery research

  Igor Németh
  Bruker Nano Analytics

- Significance of STEM-EDXS Analysis in the Characterization of Rechargeable Battery Components

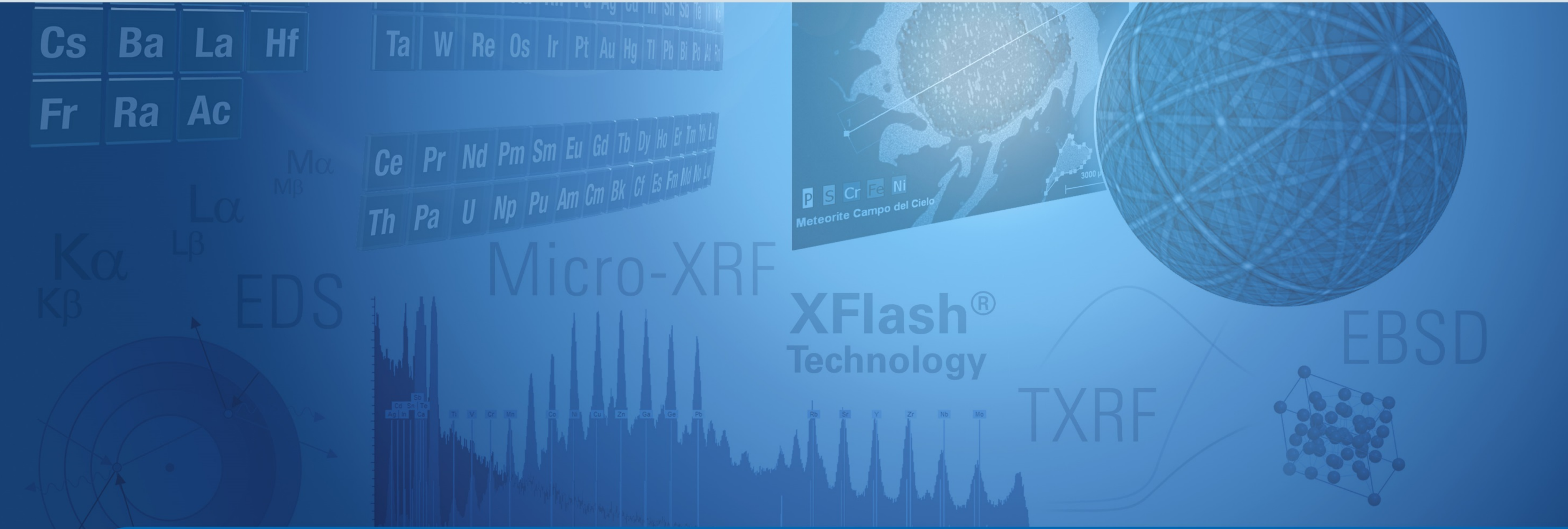
  Michael Malaki
  Phillips University Marburg

- Comparison of STEM-EDS and SEM-EDS

  Igor Németh
  Bruker Nano Analytics

Bruker Nano GmbH,
EDS instrumentation for battery research

Dr. Igor Németh



Requirements, tools and methods of EDS analysis for battery research

- High solid angle X-ray collection in SEM and in STEM

  -> sufficient data quantity for thin FIB lamellae samples
- Hypermap: measure data and process later

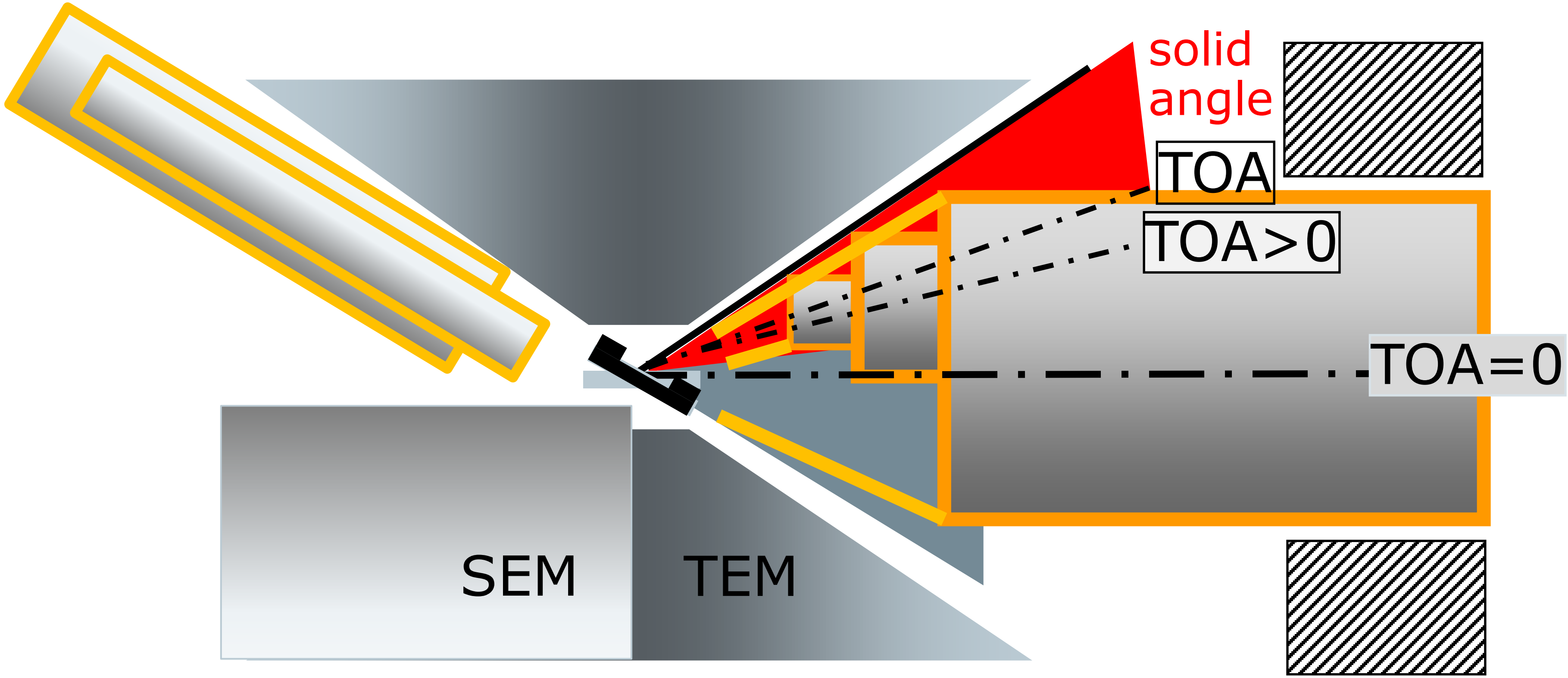
  -> element distribution maps, line profiles
- Deconvolution:

  -> Real distribution maps (also for overlapping peaks)

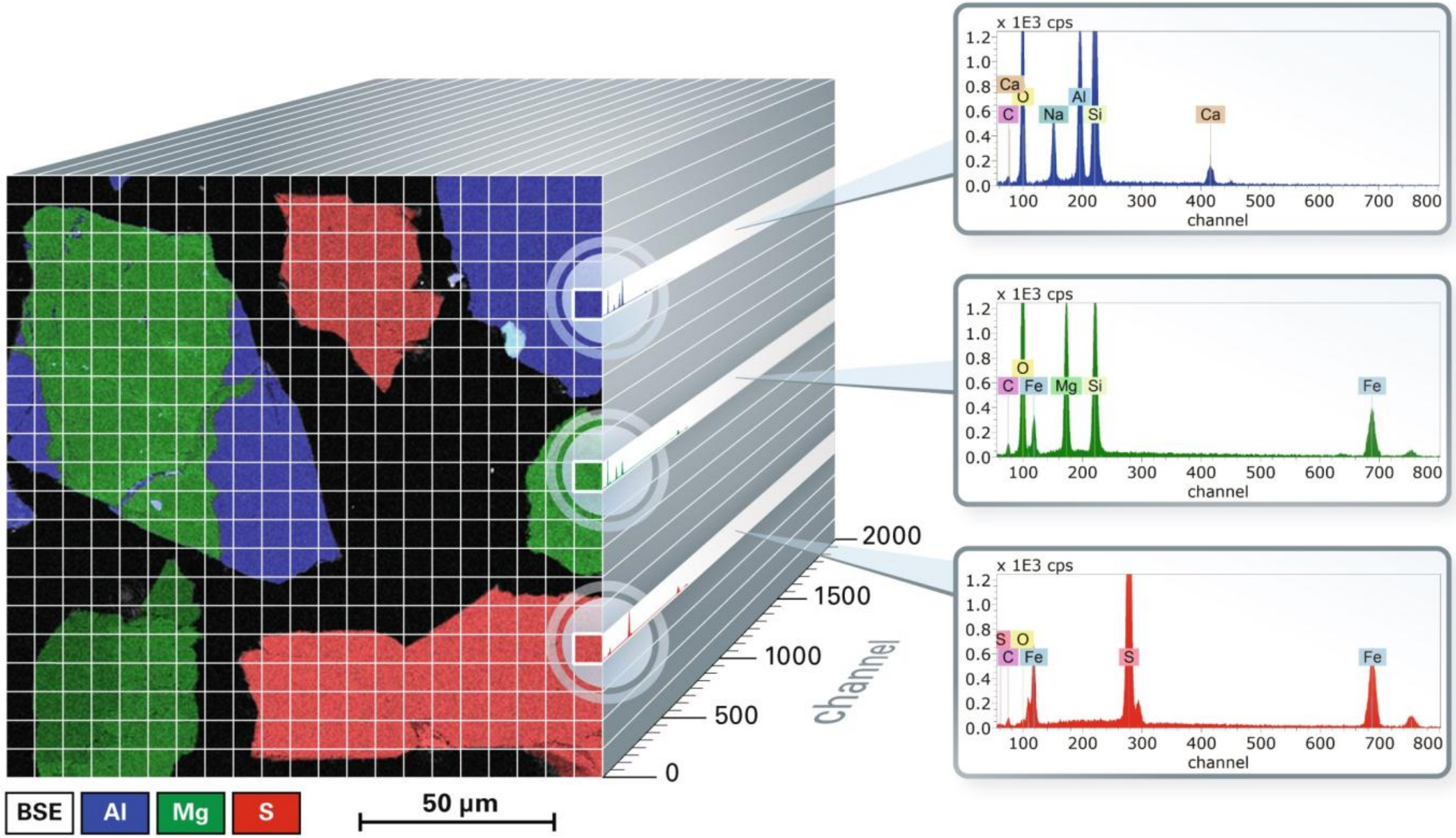
  -> Quantification of spectra and maps
- In situ measurements: EDS at elevated temperatures

Geometric constraints in SEM and STEM:

Solid and take-off angle are important to consider!



Tools of EDS analysis: Hypermap



Save data as **Hypermap** and **process later**:

Extract spectra:

-> prove presence/absence of elements

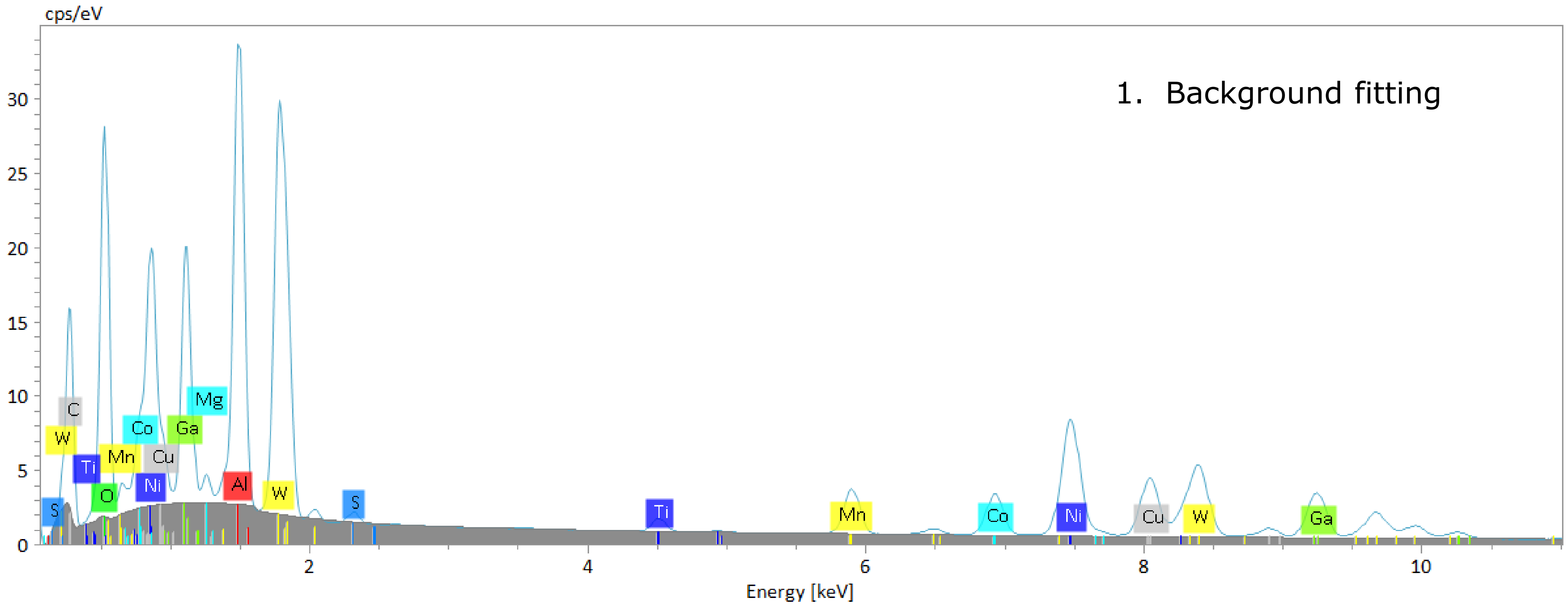
-> Calculate quantitative concentration values

Extract line profiles:

-> Quantitative line profiles

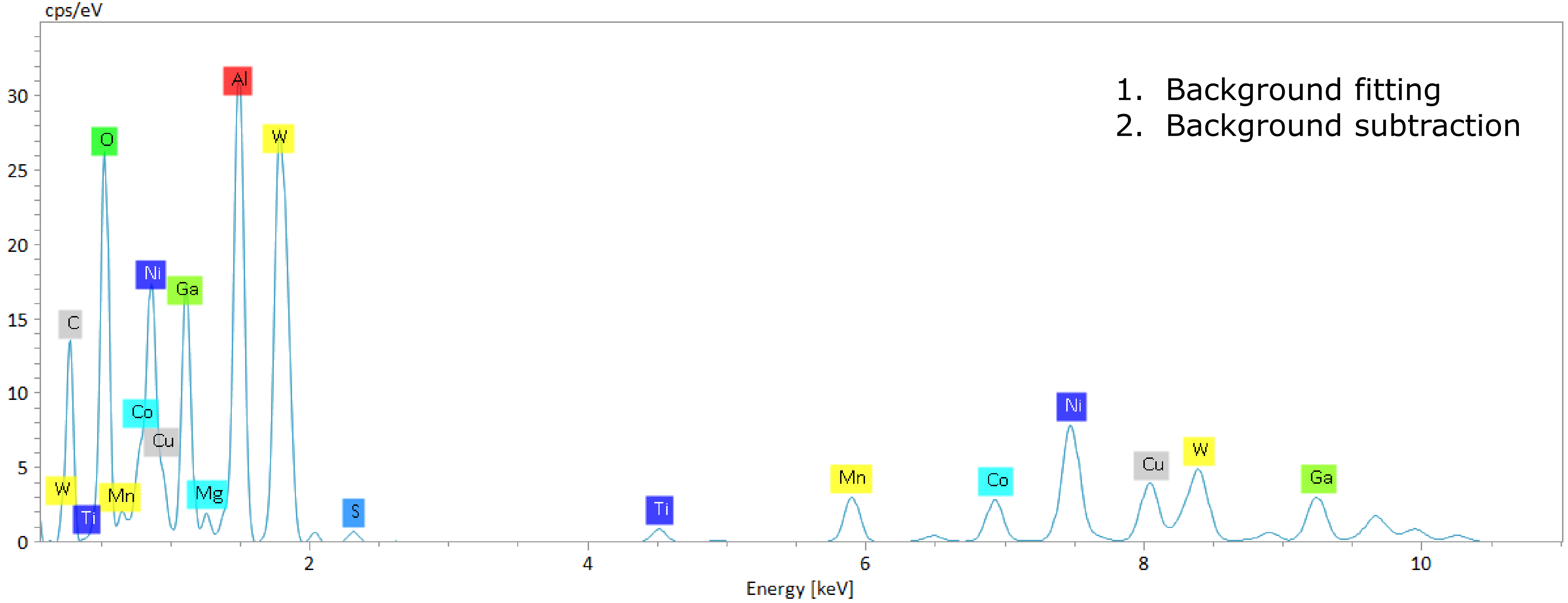
Quantitative element distribution maps

Tools of EDS analysis: Deconvolution



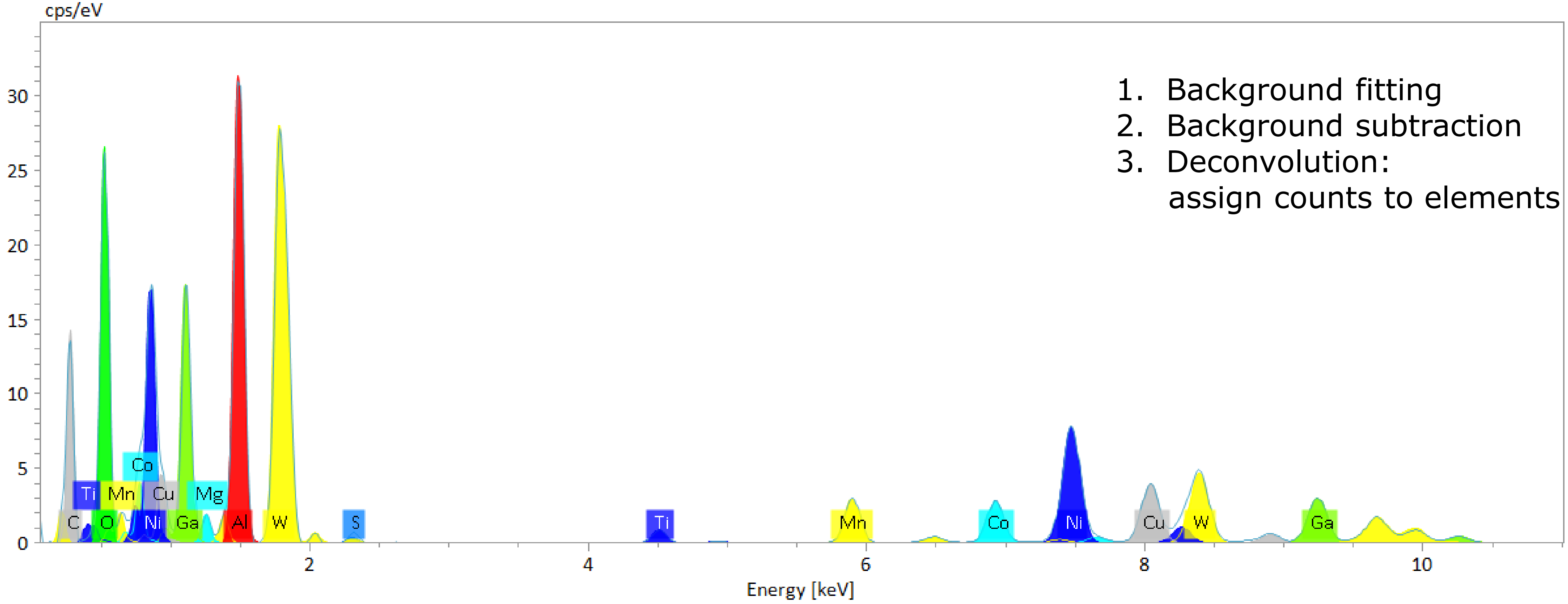
1. Background fitting

Tools of EDS analysis: Deconvolution



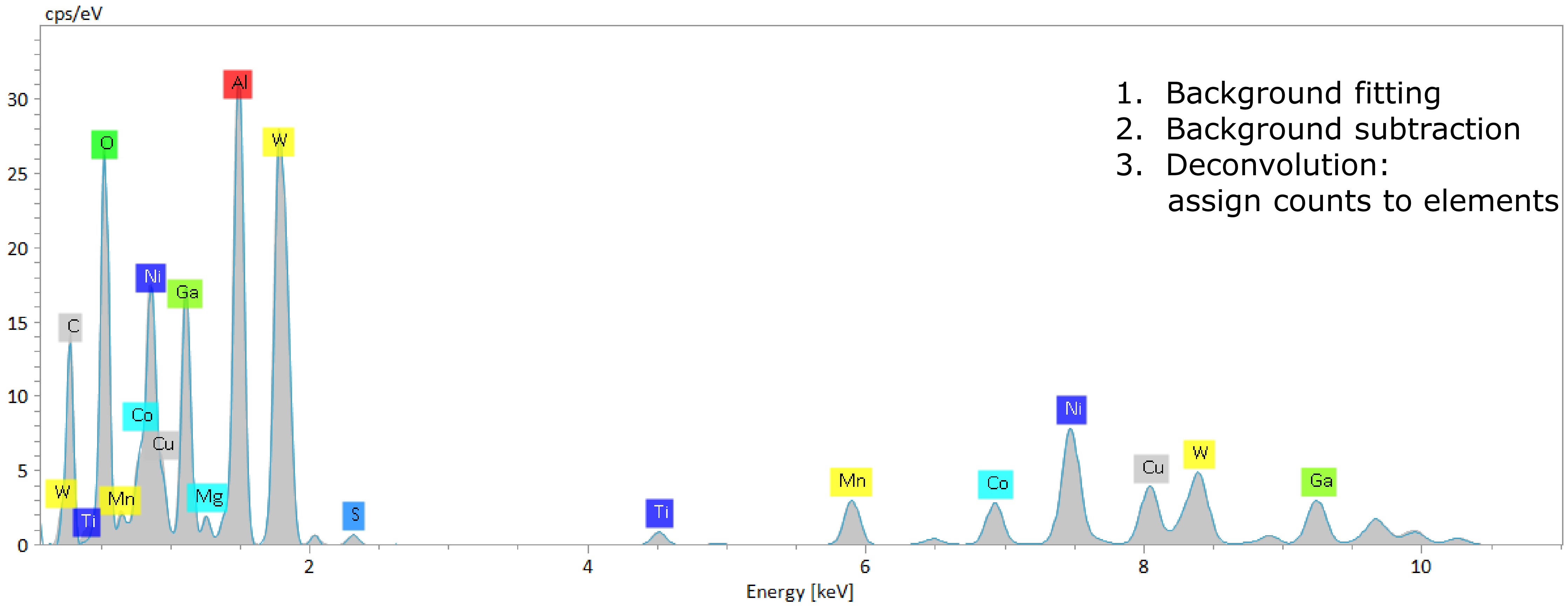
1. Background fitting
2. Background subtraction

Tools of EDS analysis: Deconvolution



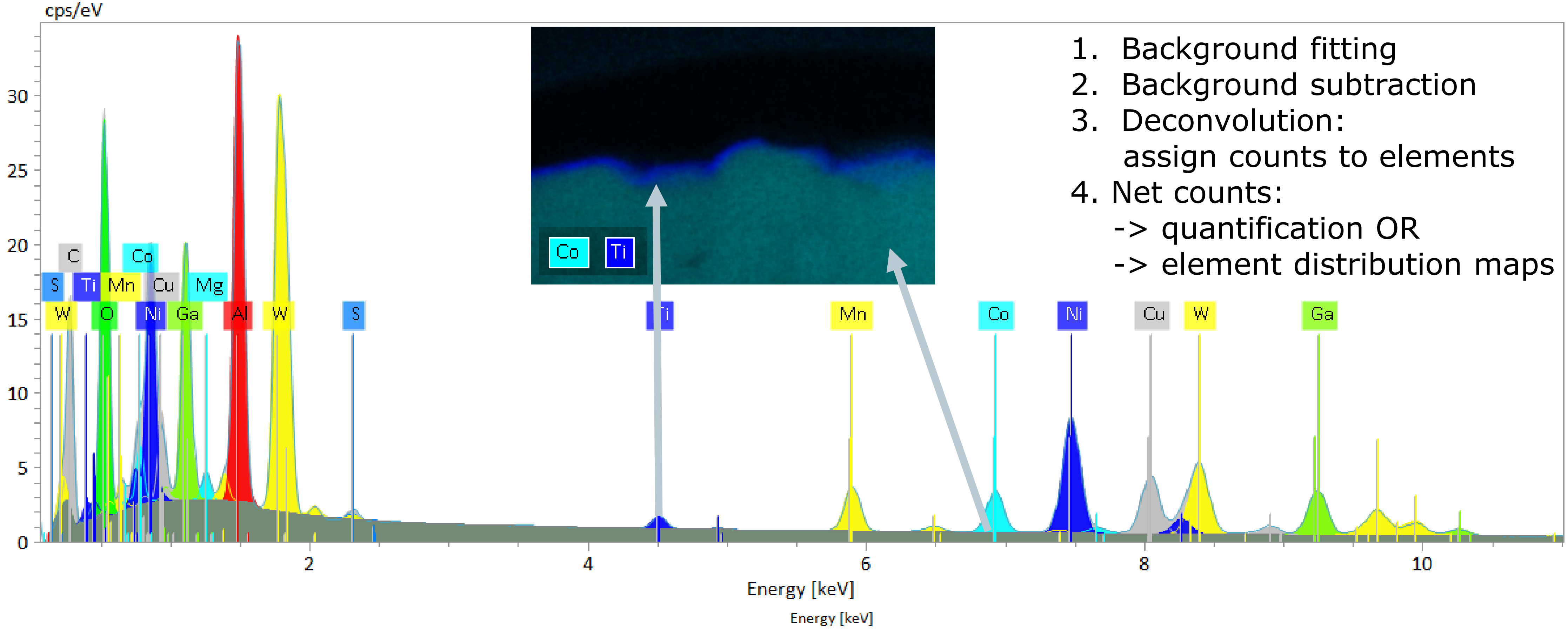
1. Background fitting
2. Background subtraction
3. Deconvolution:
   assign counts to elements

Tools of EDS analysis: Deconvolution



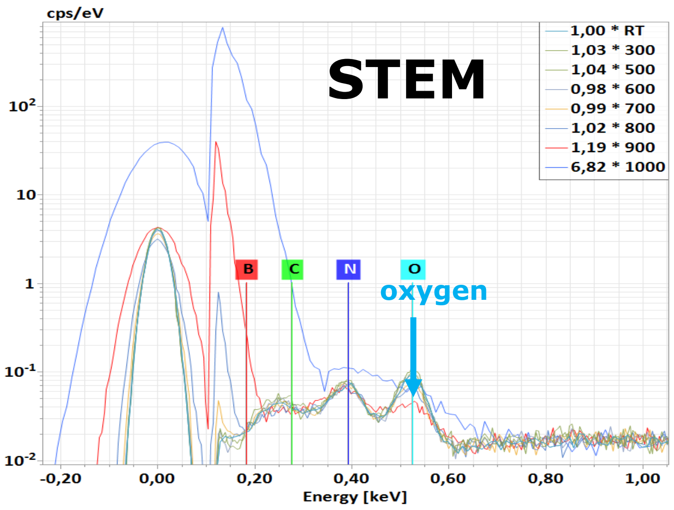
1. Background fitting
2. Background subtraction
3. Deconvolution:
   assign counts to elements

Tools of EDS analysis: Deconvolution



1. Background fitting
2. Background subtraction
3. Deconvolution:
   assign counts to elements
4. Net counts:
   -> quantification OR
   -> element distribution maps

EDS in situ / at elevated temperatures



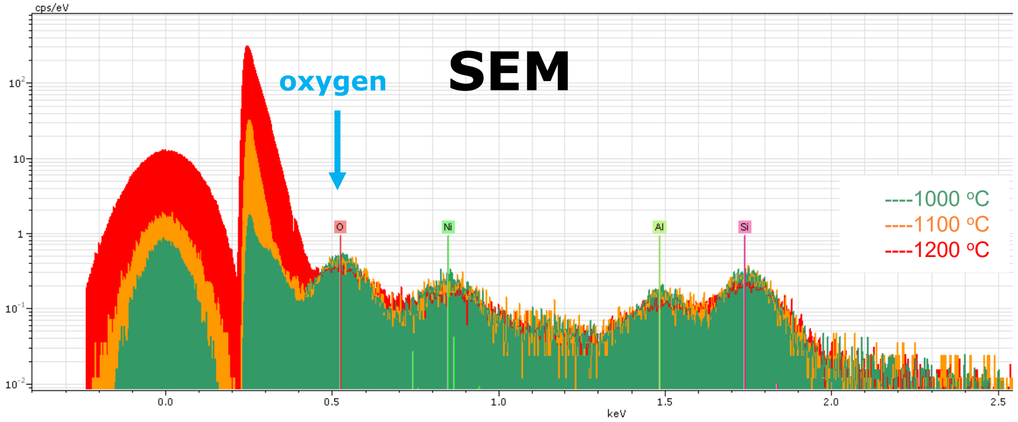
TEM: 11mm sample – detector distance

Challenges:

Thermal radiation -> noise -> high background below 2keV: detection of light elements affected

This effect depends on:

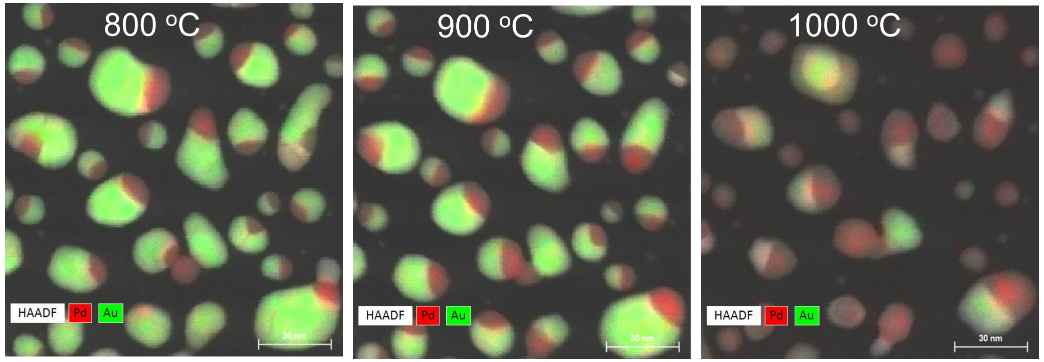
- sample-detector distance
- detector window material



SEM: 25mm sample – detector distance

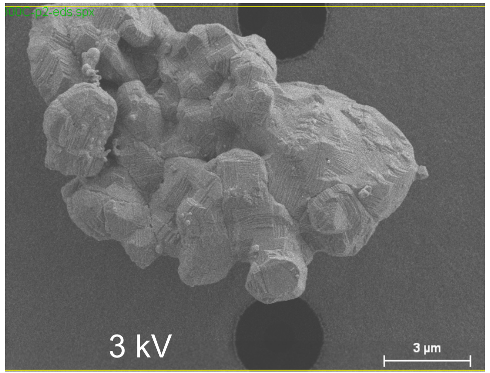
Possibilities:

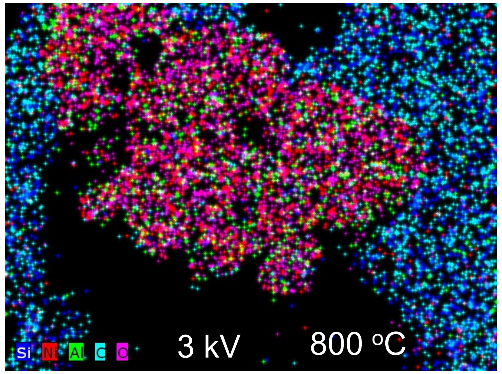
- Spectra: monitoring of element lines
- Mapping: Phase changes, segregations



J. T. van Omme et al., Ultramicroscopy 192 (2018) 14–20

DENS solutions





Jane Y. Howe (ORNL), Christianne Beekman (Florida St. Uni)

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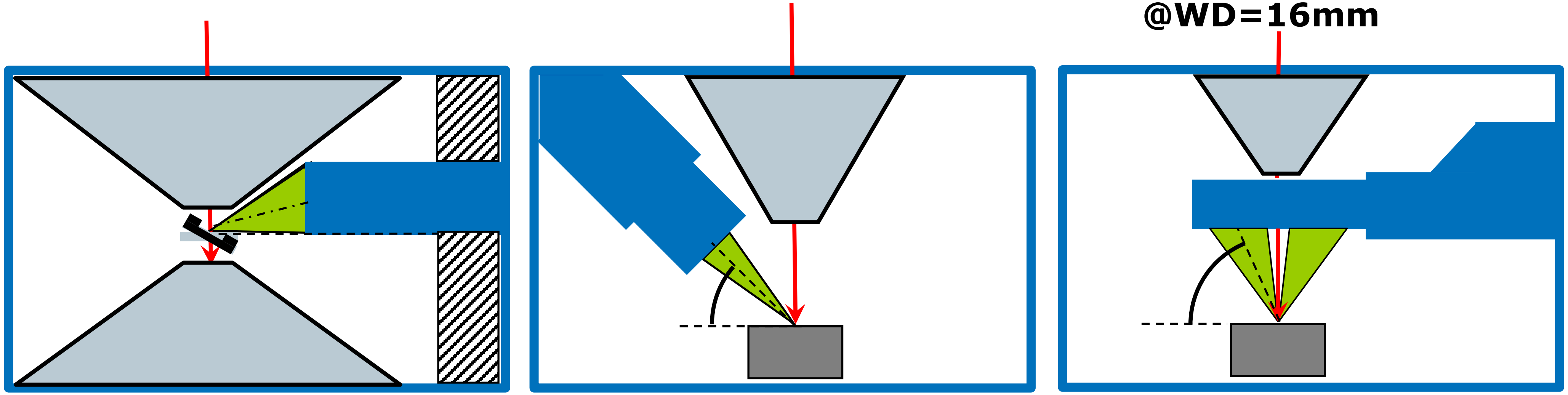
Comparison of the same sample STEM - SEM

STEM 200kV
60 mm^2 EDS detector

SEM 20kV
60 mm^2 EDS detector
@WD= 8mm

SEM 20kV
60 mm^2 4x15mm^2
FlatQUAD detector
@WD=16mm



take off angle=22°
solid angle~0.26sr
beam current~0.05 nA →

take off angle=35°
solid angle=0.043 sr →
beam current~ 2nA

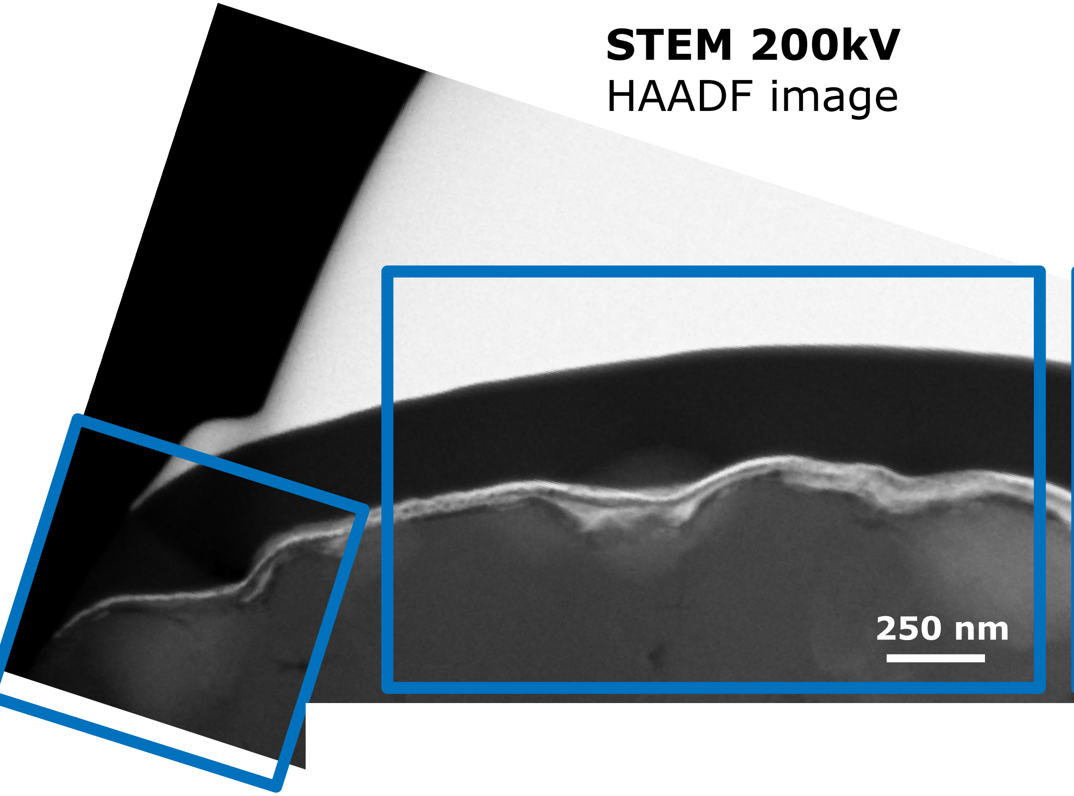
take off angle=60-70°
solid angle= 0.7-1 sr
beam current~ 2nA

x7 more X-ray signal

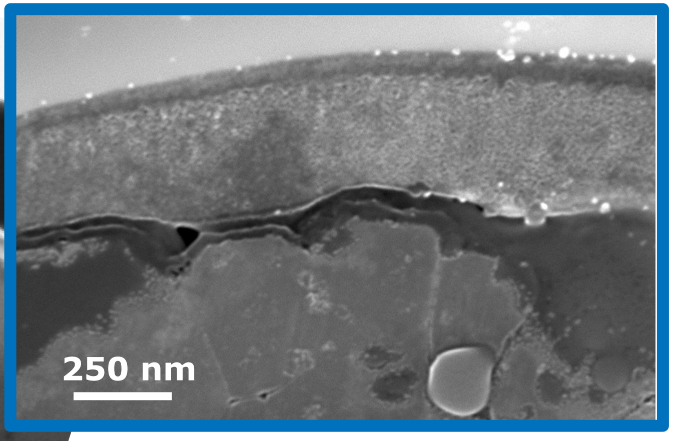
x15-20 more X-ray signal

Comparison of the same sample STEM - SEM

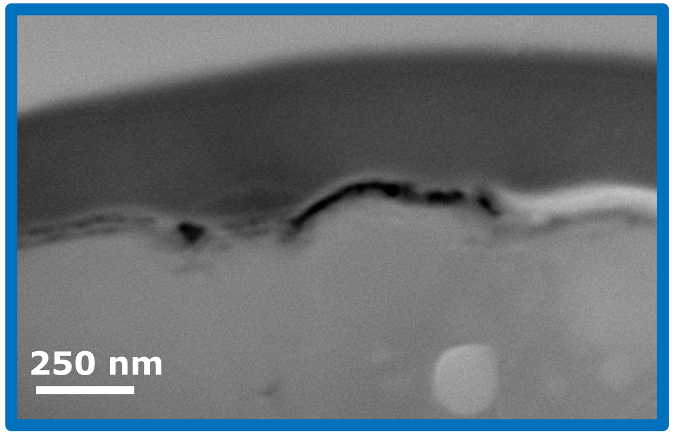
STEM 200kV
HAADF image



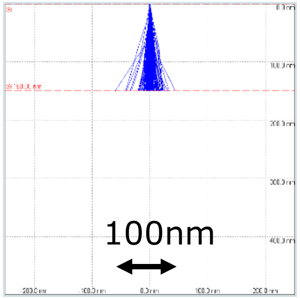
SEM 20kV
In-lens image
WD=8mm



SEM 20kV
SE image with
inserted FlatQUAD
WD=16mm



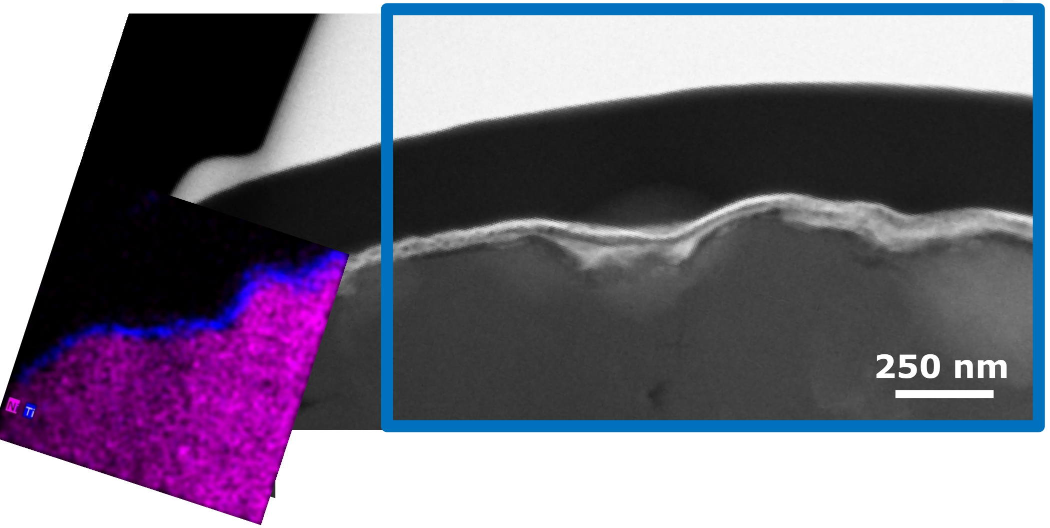
Images taken under measurement conditions optimized for EDS analysis
Image quality does not affect EDS resolution on this scale!



Sample and data courtesy: Michael Malaki, Shamail Ahmed, Materials Sciences Center, Philipps University Marburg

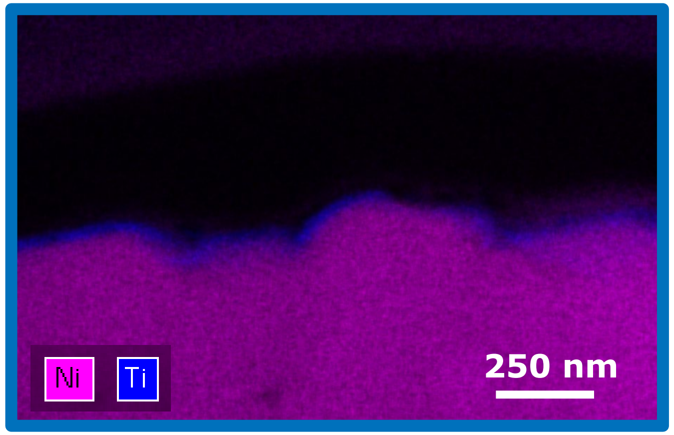
Comparison of the same sample STEM - SEM

STEM 200kV
60 mm² EDS detector



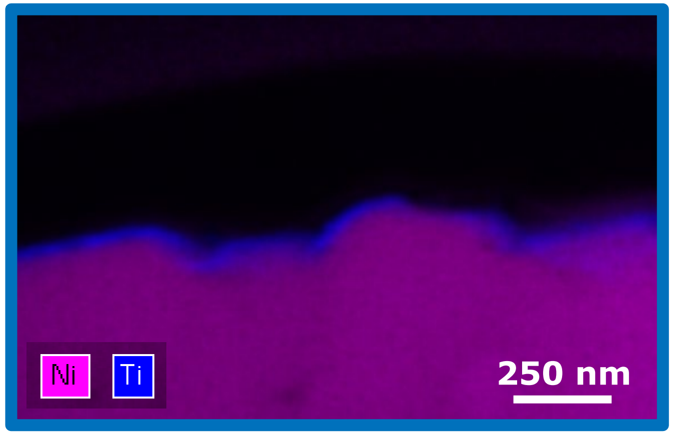
Total measurement time= 8 mins
Beam current= 0.2 nA
Input count rate ~ 1 kcps

SEM 20kV
60 mm² EDS detector



Total measurement time= 34 mins
Beam current=2 nA
Input count rate ~ 30 kcps

SEM 20kV
FlatQuad detector

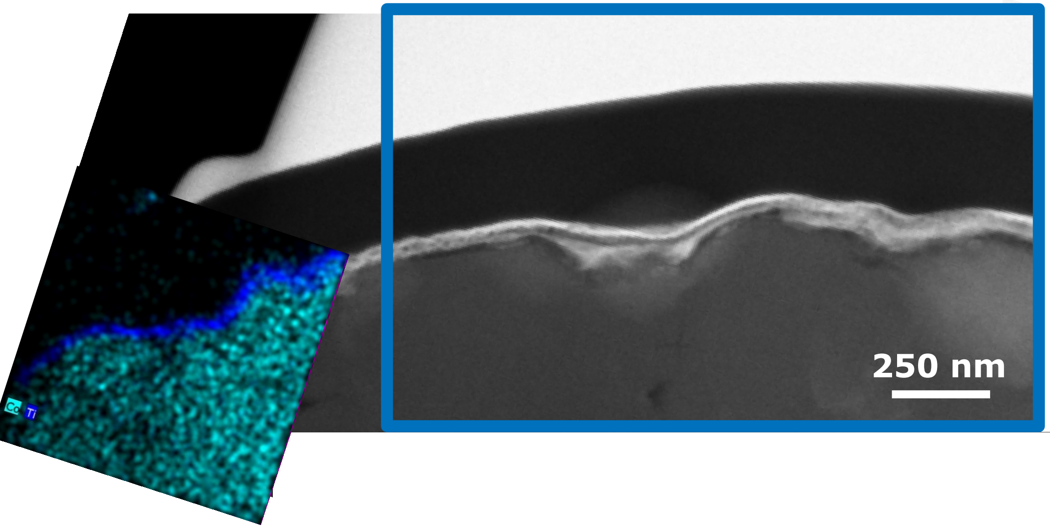


Total measurement time= 34 mins
Beam current= 2 nA
Input count rate ~ 460 kcps

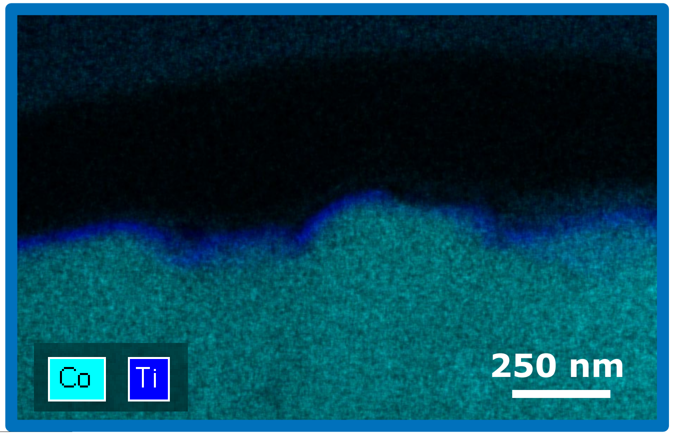
Sample and data courtesy: Michael Malaki, Shamail Ahmed, Materials Sciences Center, Philipps University Marburg

Comparison of the same sample STEM - SEM

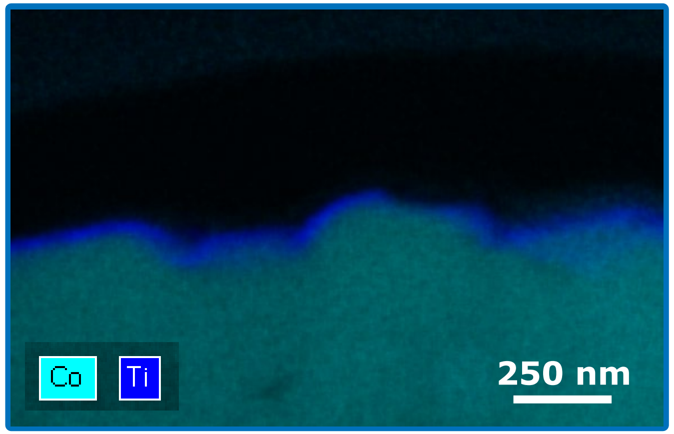
STEM 200kV
60 mm² EDS detector



SEM 20kV
60 mm² EDS detector



SEM 20kV
FlatQuad detector



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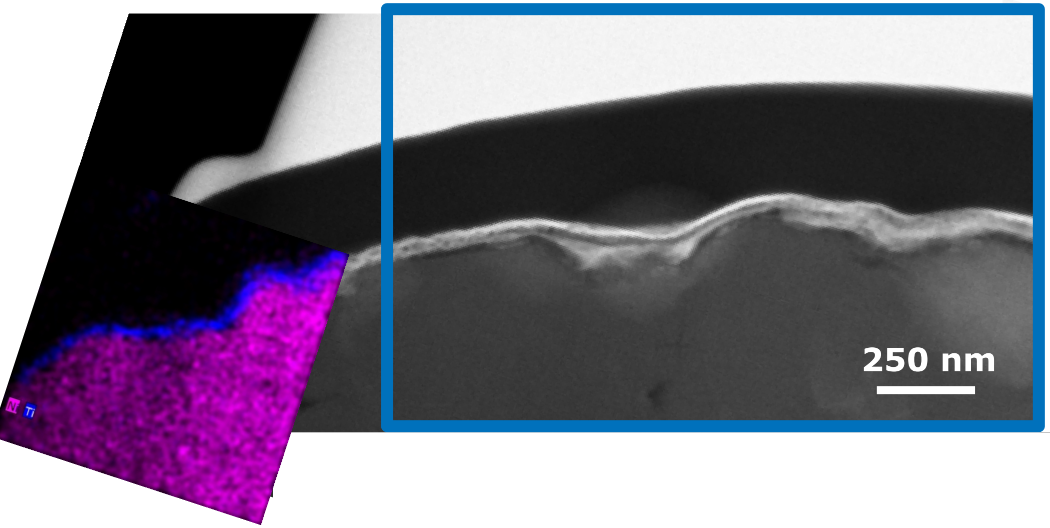
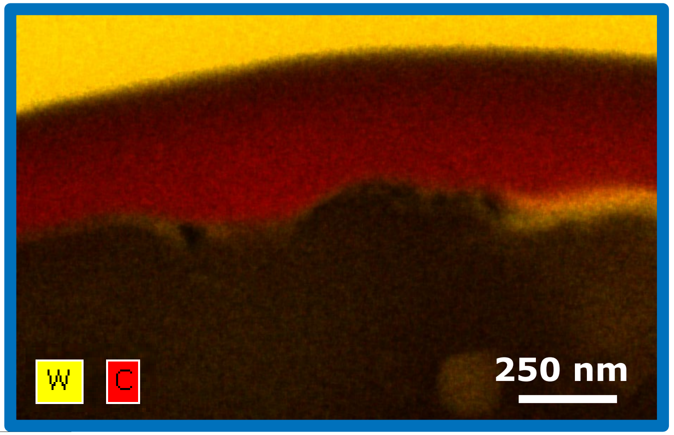
Comparison of the same sample STEM - SEM

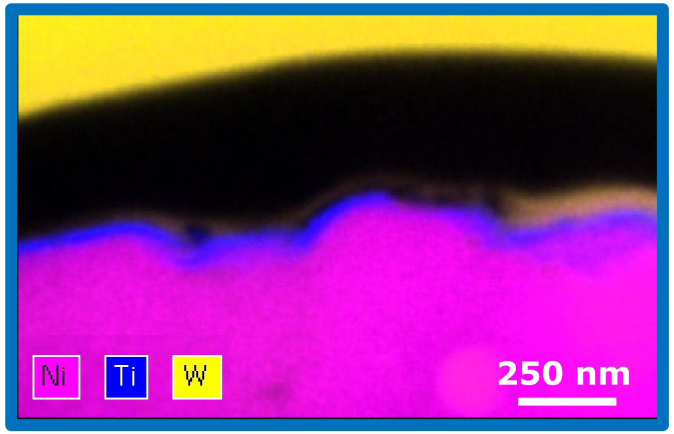
STEM 200kV
60 mm² EDS detector



SEM 20kV
60 mm² EDS detector



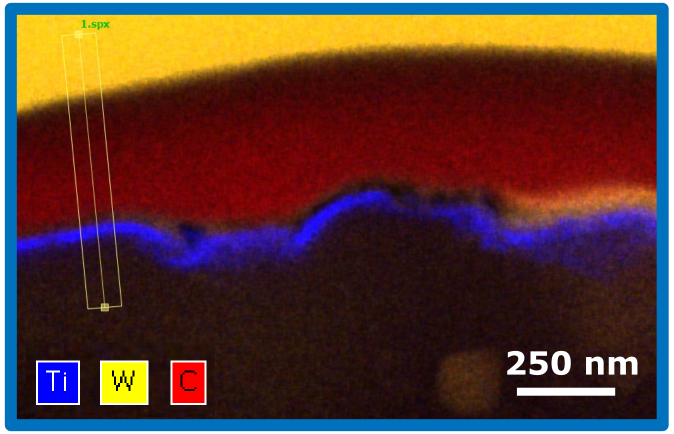
SEM 20kV
FlatQuad detector

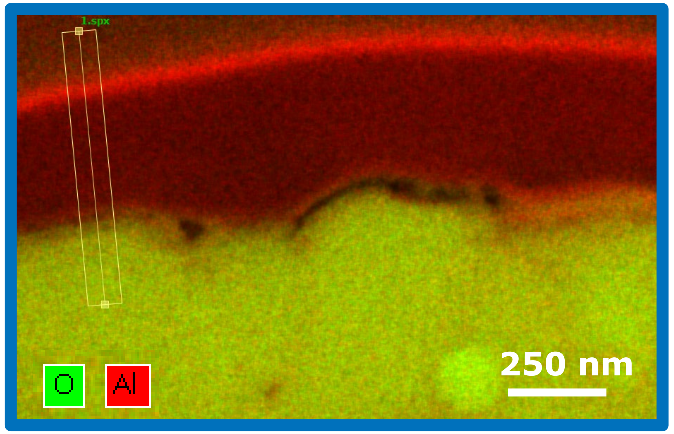


Sample and data courtesy: Michael Malaki, Shamail Ahmed, Materials Sciences Center, Philipps University Marburg

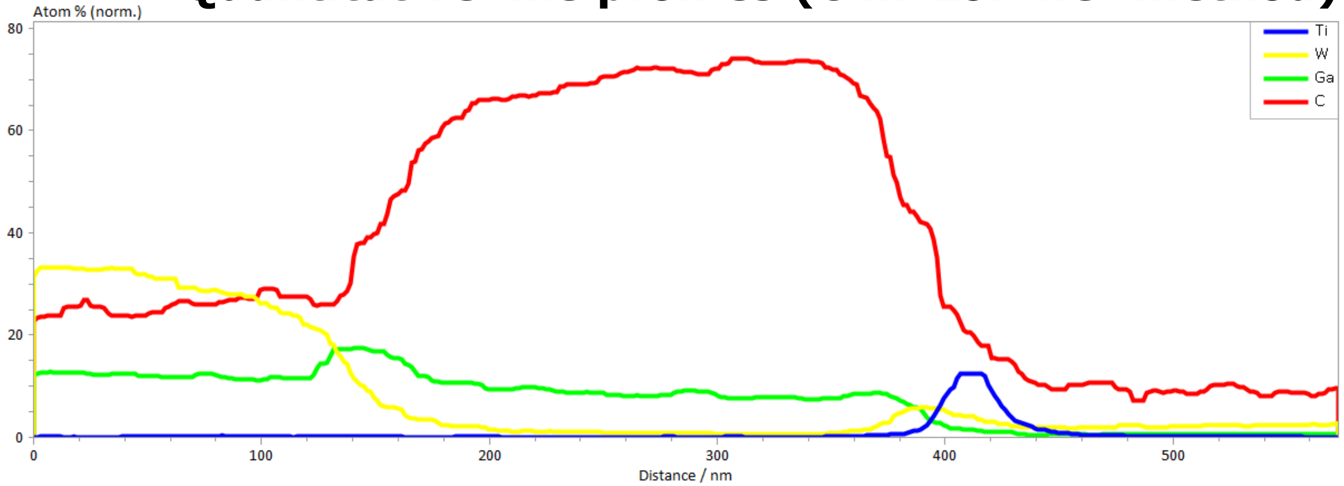
What additional information EDS reveals

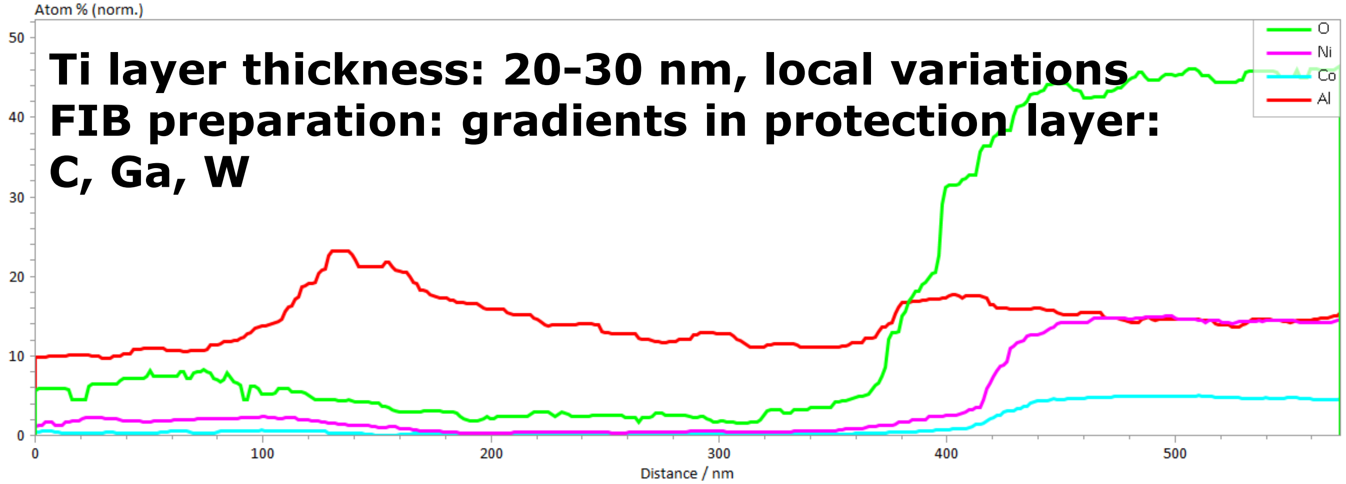
SEM 20kV
60 mm^2 EDS detector





Quantitative line profiles (Cliff-Lorimer method)



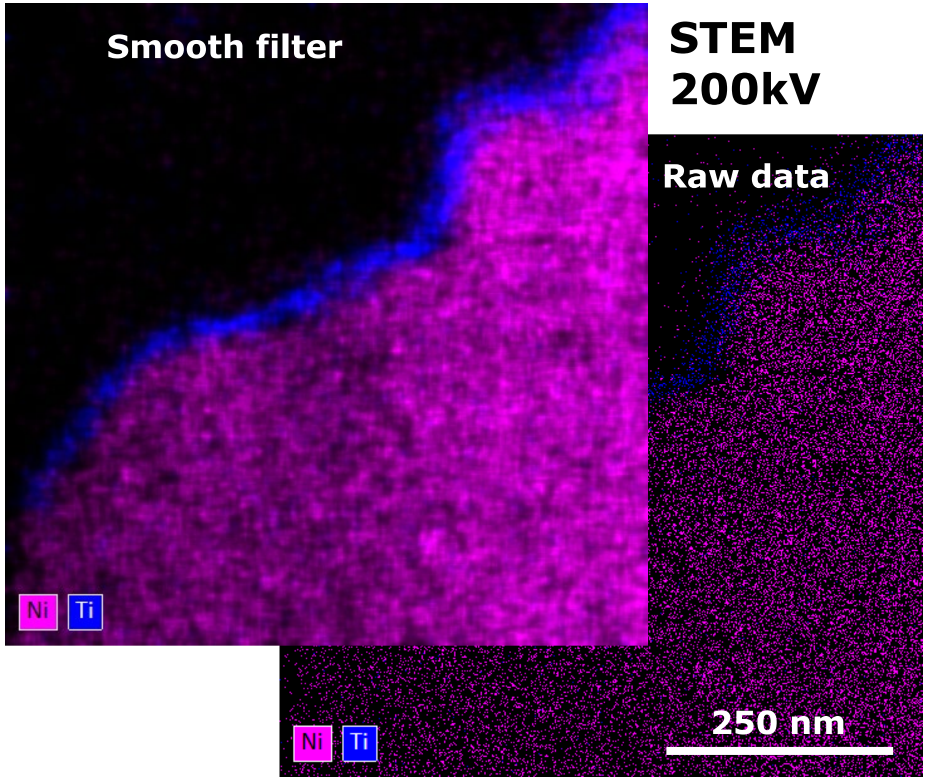


Ti layer thickness: 20-30 nm, local variations
FIB preparation: gradients in protection layer: C, Ga, W

Sample and data courtesy: Michael Malaki, Shamail Ahmed, Materials Sciences Center, Philipps University Marburg

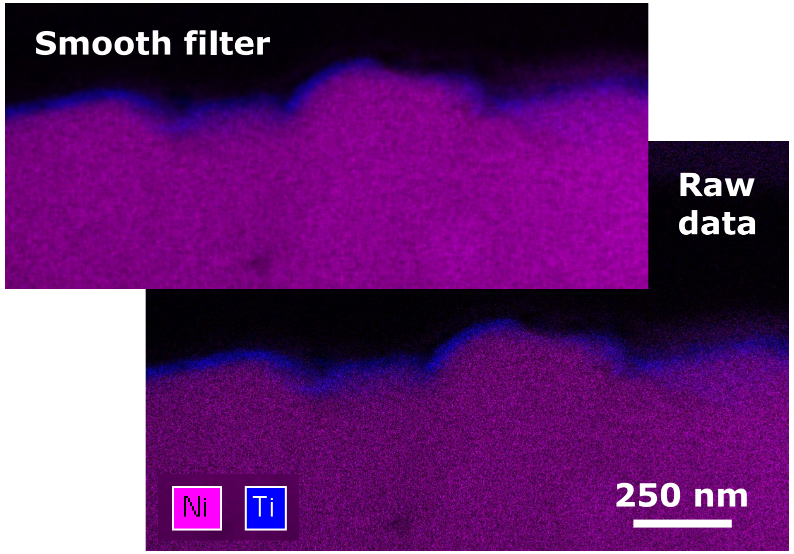
STEM-EDS vs. SEM-EDS vs. SEM-FlatQuad EDS

STEM 200kV



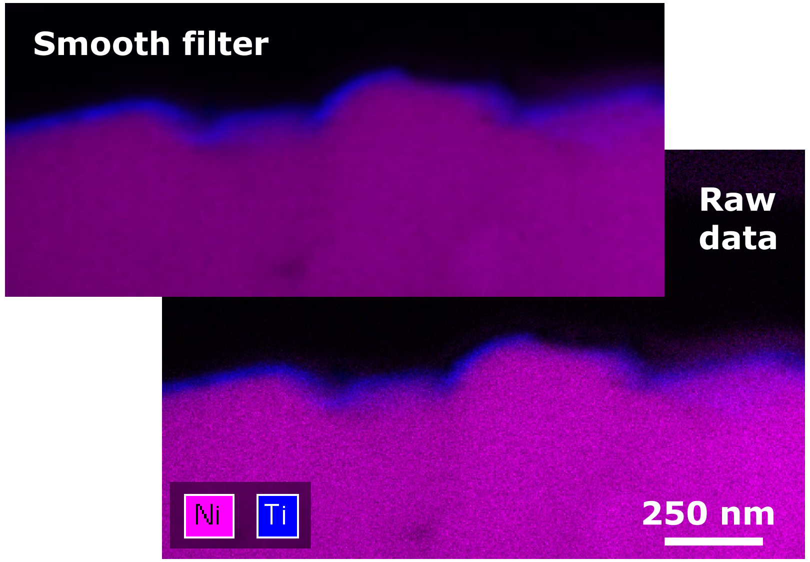
Higher spatial resolution
Lower beam currents
-> less signal (filtering needed) or longer measurements

SEM 20kV conventional EDS



Lower spatial resolution due to lower kV
Lower solid angle due to larger sample-detector distance
Higher beam currents -> more signal or shorter measurements

SEM 20kV FlatQUAD EDS



Maps with very high statistics
EDS spatial resolution not affected due to longer WD

Sample and data courtesy: Michael Malaki, Shamail Ahmed, Materials Sciences Center, Philipps University Marburg

Innovation with Integrity