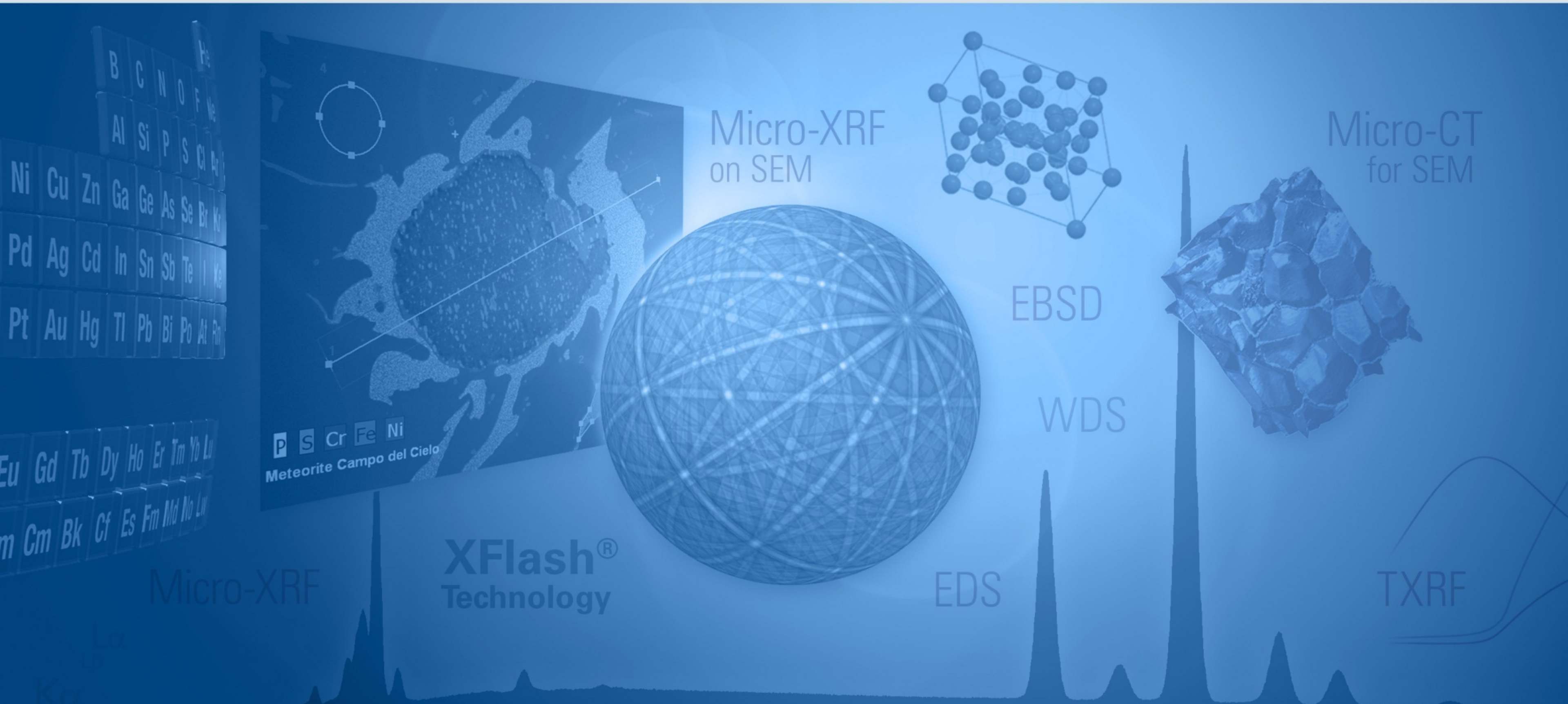
Analytical SEM Solutions for Geology – Part I



Webinar, August 13, 2019

Presenters in Part I

SEM for Geological Applications - important aspects to consider

Mats Eriksson

Department Manager

Hitachi High-Technologies

Advanced Microanalysis with the QUANTAX System

Max Patzschke

Application Specialist

Bruker Nano Analytics

Macro-Micro-Nanoscale SEM/EDS of Earth and Planetary Materials

Dr. Tobias Salge

Electron Probe Microanalyst

Imaging and Analysis Centre, Natural History Museum, London, UK

SEM(s) for Geological Applications

- important aspects to consider

Mats Eriksson
EM Department Manager
Hitachi High-Technologies Europe GmbH

Outline

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- Introduction to SEM
- Signals detected
- Electron sources
- Chamber and stage
- Variable pressure / low vacuum
- Imaging requirements in geology
- Finding the area of interest

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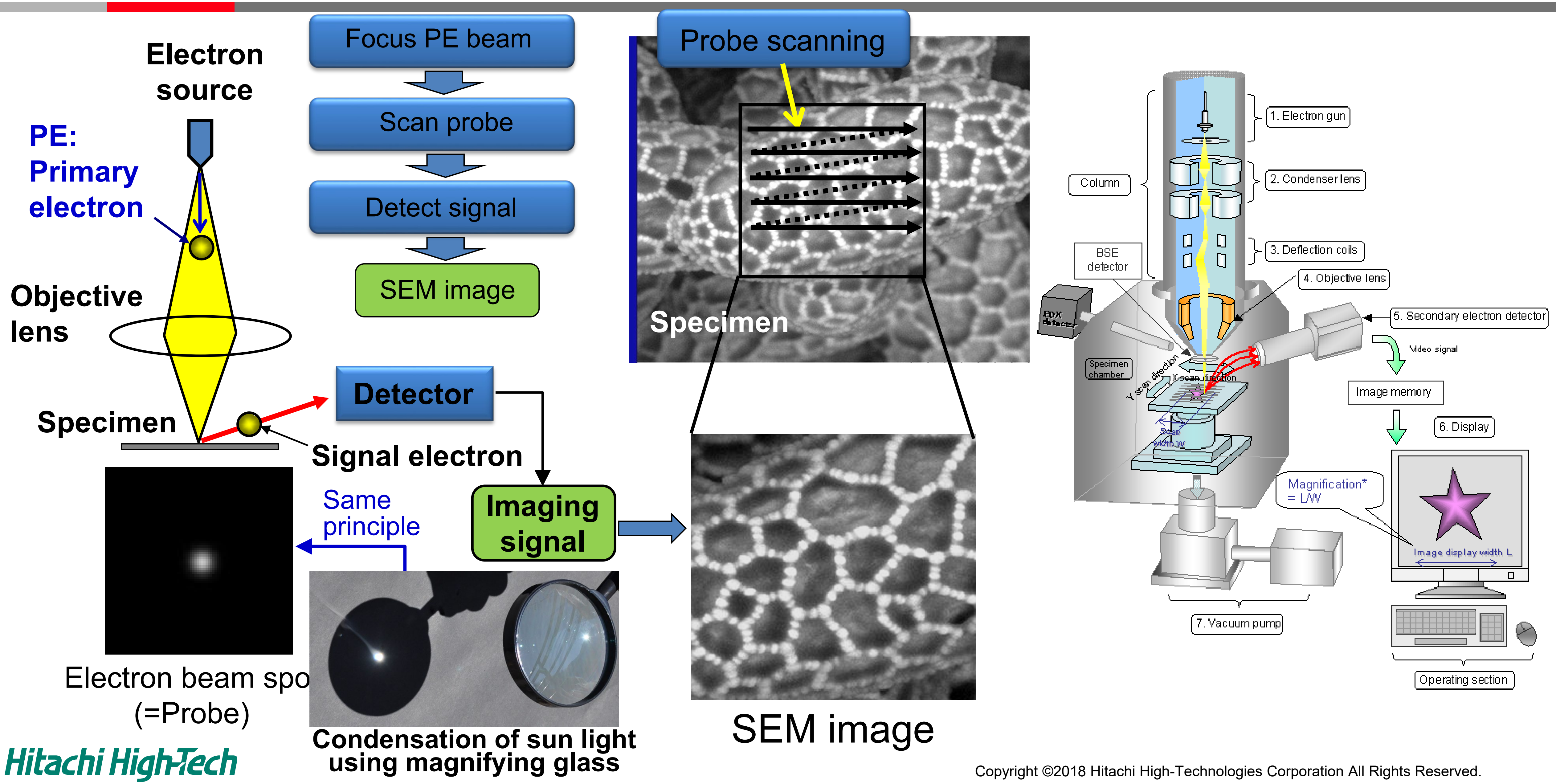
Basic operation of the SEM



Imaging signals in the SEM, SE/BSE emission

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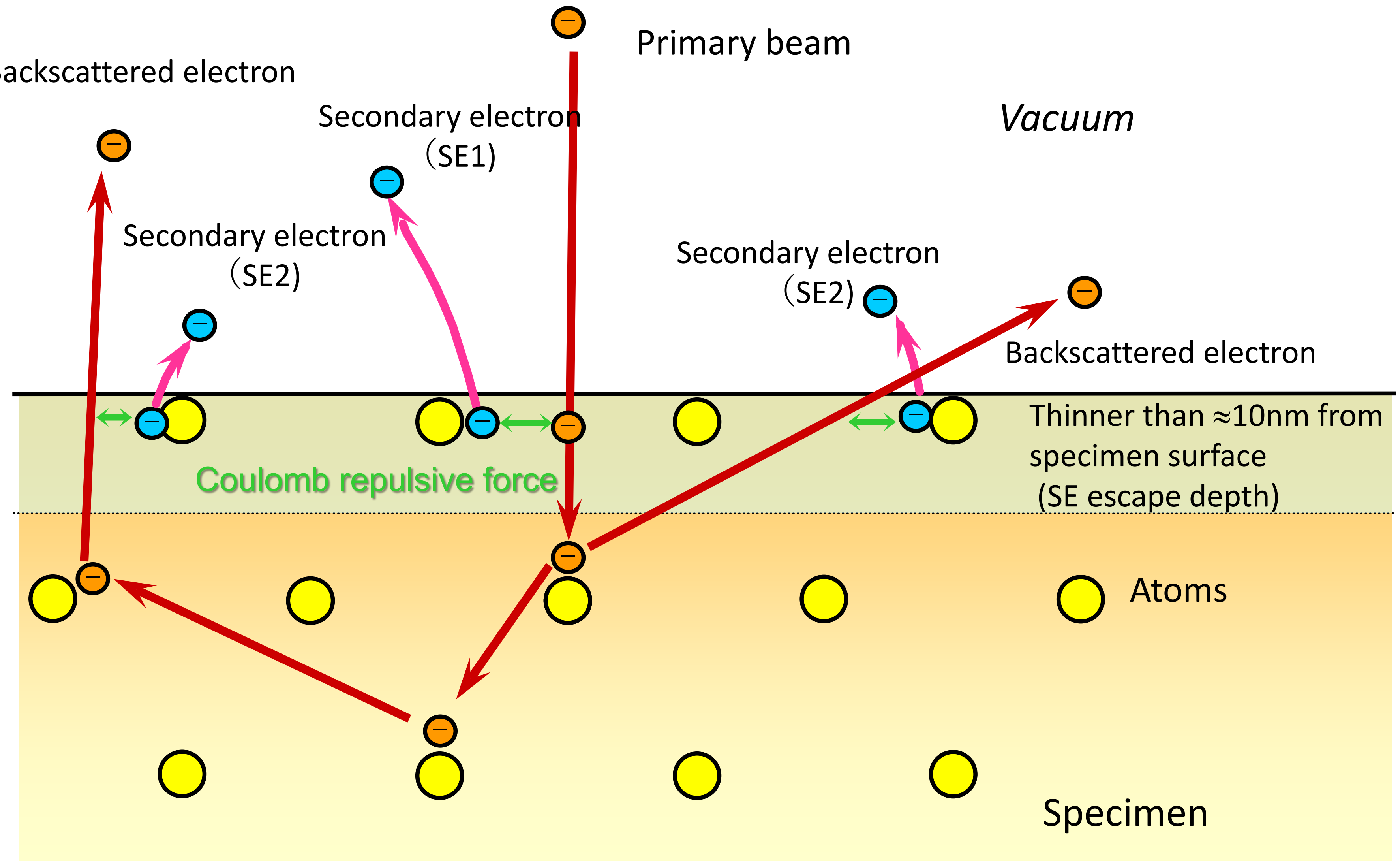

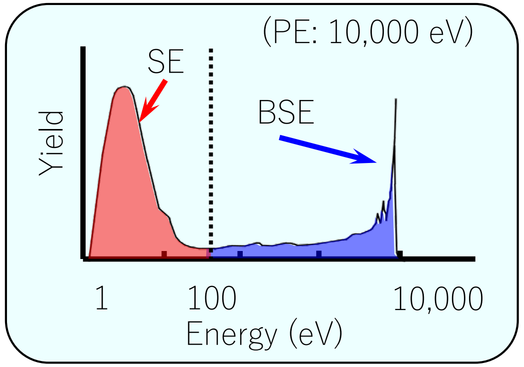

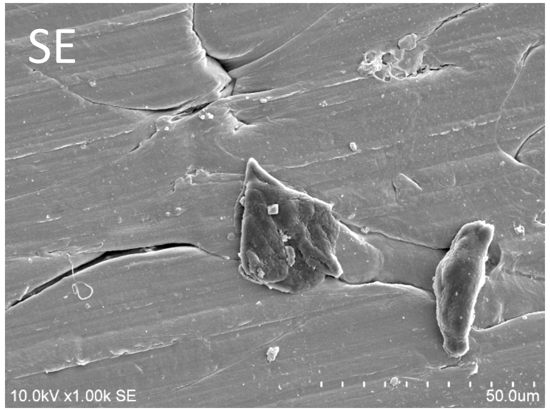

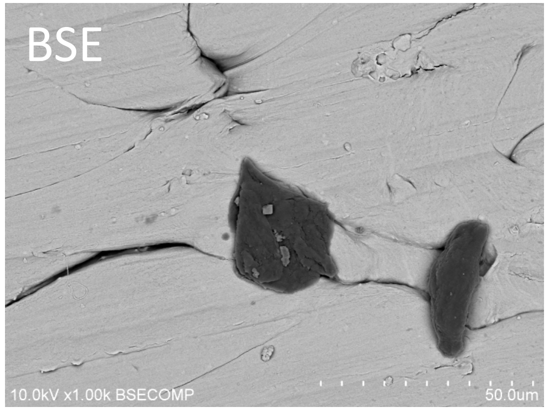

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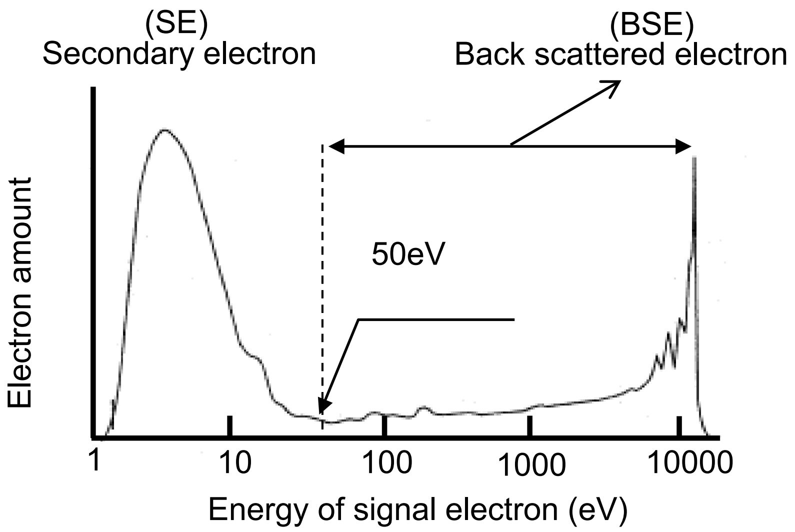
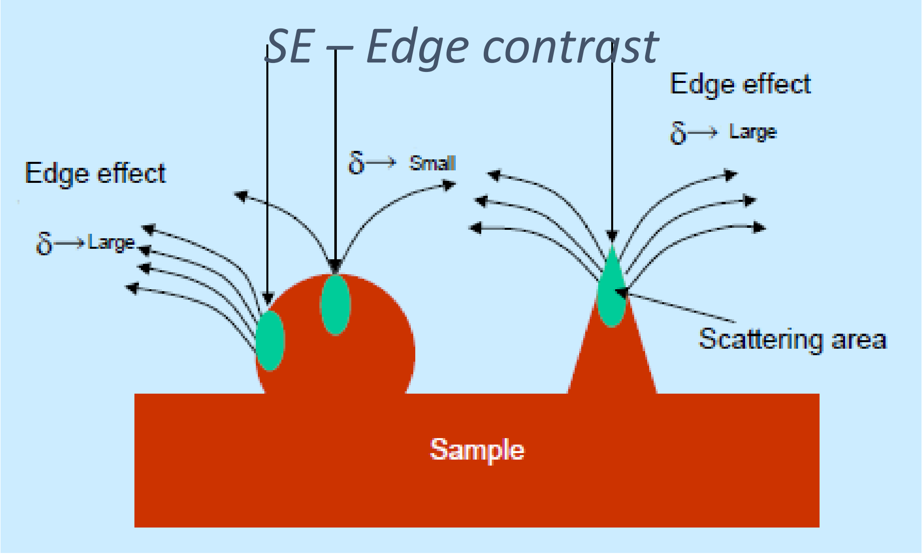
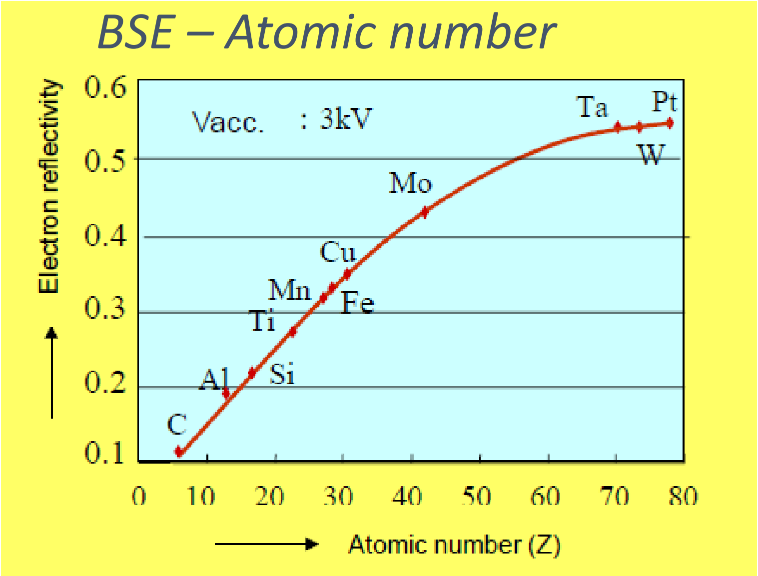
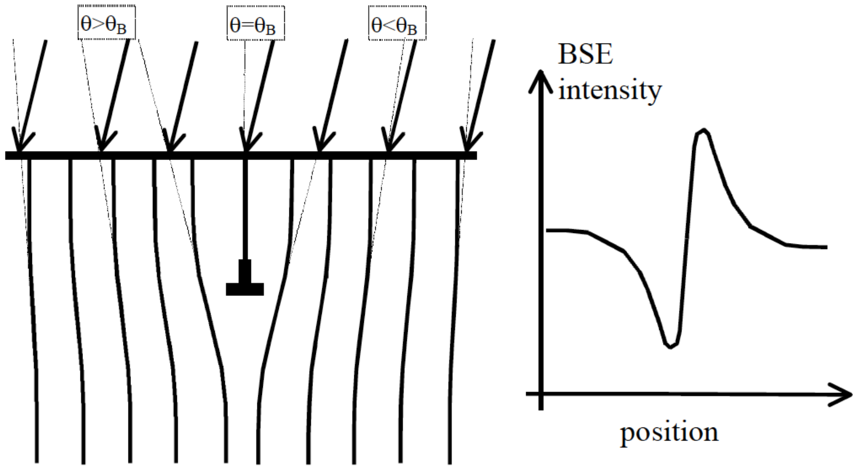
Imaging signals in the SEM, SE/BSE emission





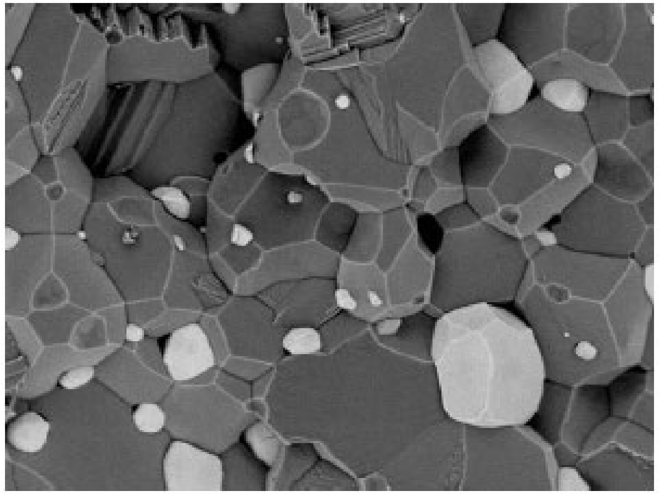




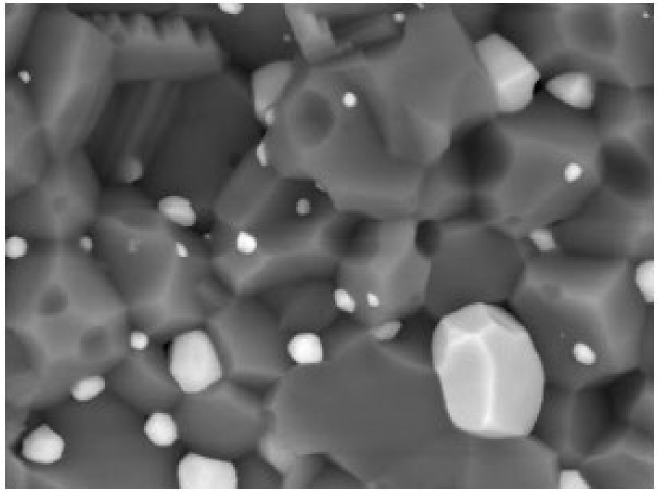
BSE – Channeling contrast
ECCI

Beam Excitation Volumes

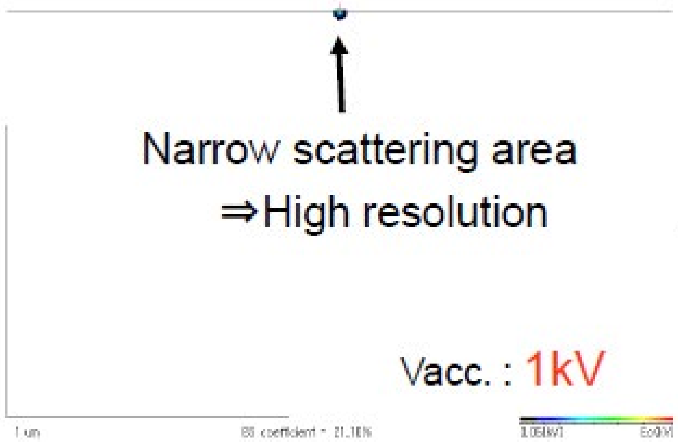
Vacc. : 1kV



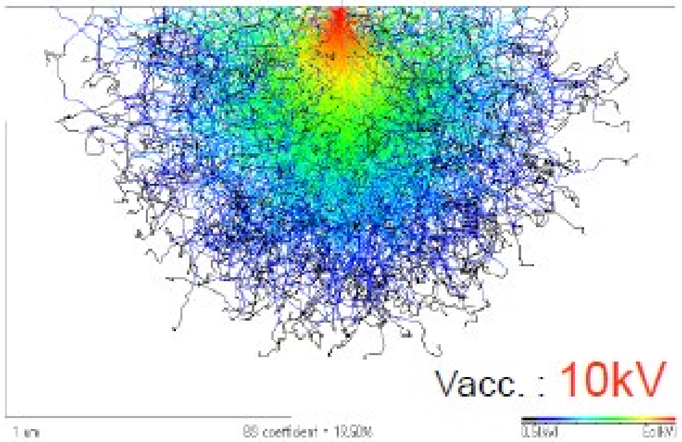
Vacc. : 10kV



Specimen
Ni-Al_2O_3



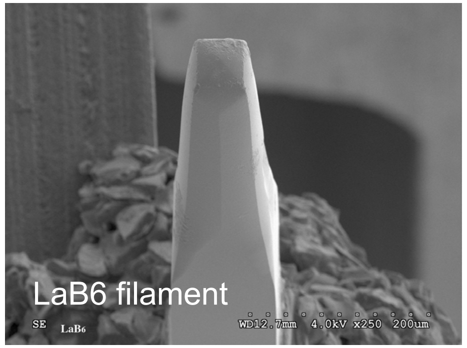
1μm

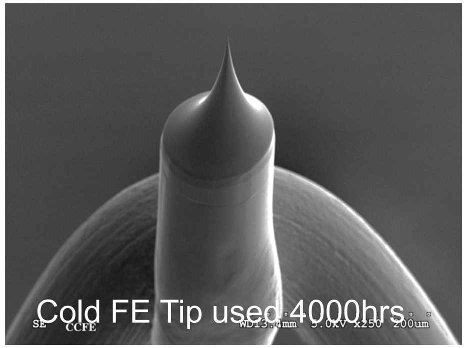


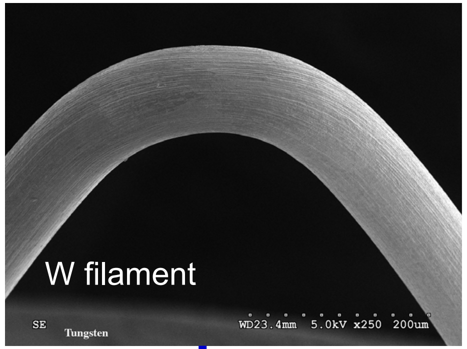
Monte Carlo simulation
Comparison of scattering region of incident electrons (Sample : Si)

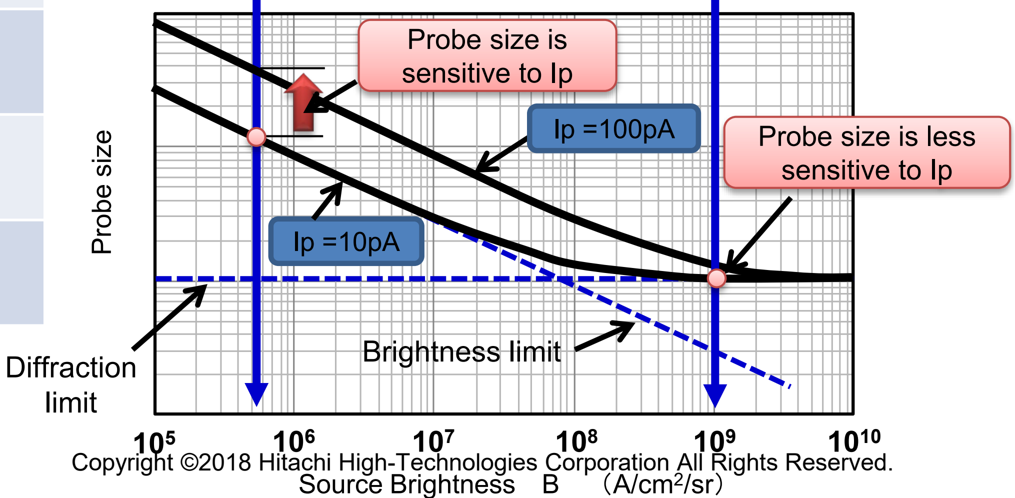
Electron Source Comparison

Emitter	W	LaB6	Schottky FE	Cold FE	New Cold FE
Source size	1 – 2 μm	1 – 2 μm	10-25 nm	3-5 nm	3-5 nm
Temperature	2300°C	1500°C	1500°C	R.T.	R.T.
Brightness	1	10	500	1000	1500
[A/cm²sr]	10^6	10^7	$5x10^8$	$1x10^9$	$2x10^9$
Energy spread	2.0 eV	1.5 eV	0.5 eV	0.2 eV	0.2 eV
Stability [%/hr]	< 1%	< 2 %	0.2%	3-5%	0.8%
Emission	200 μA	100 μA	100 μA	10 μA	20 μA
Probe current	1 μA	1 μA	200 nA	20 nA	20 nA
Life time	1 month	1 year	2 years	>5 years	>5 years

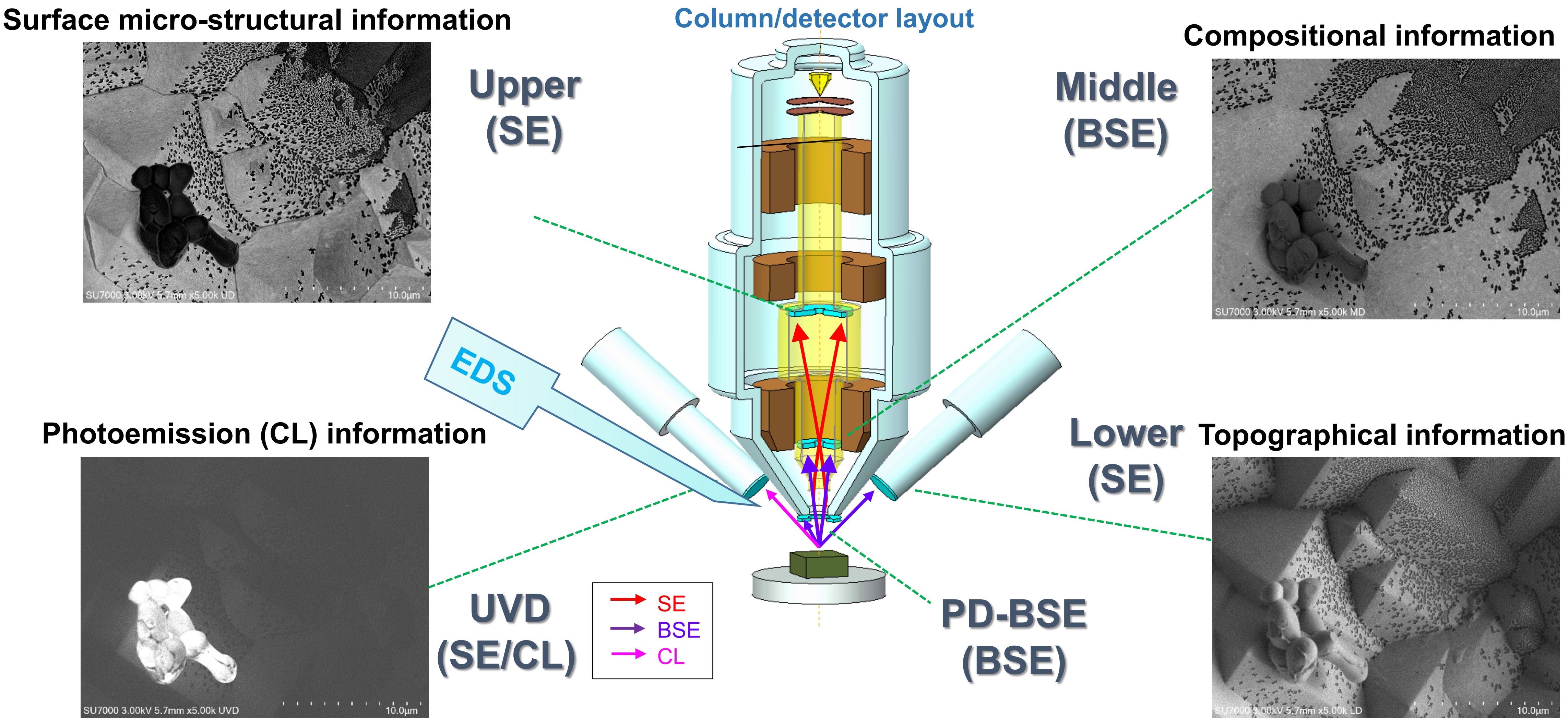








Detection system for various signals



Specimen: Solar Cell incl. fluorescent material, V_{acc}*: 3kV*

Cathodoluminescence using the UVD detector

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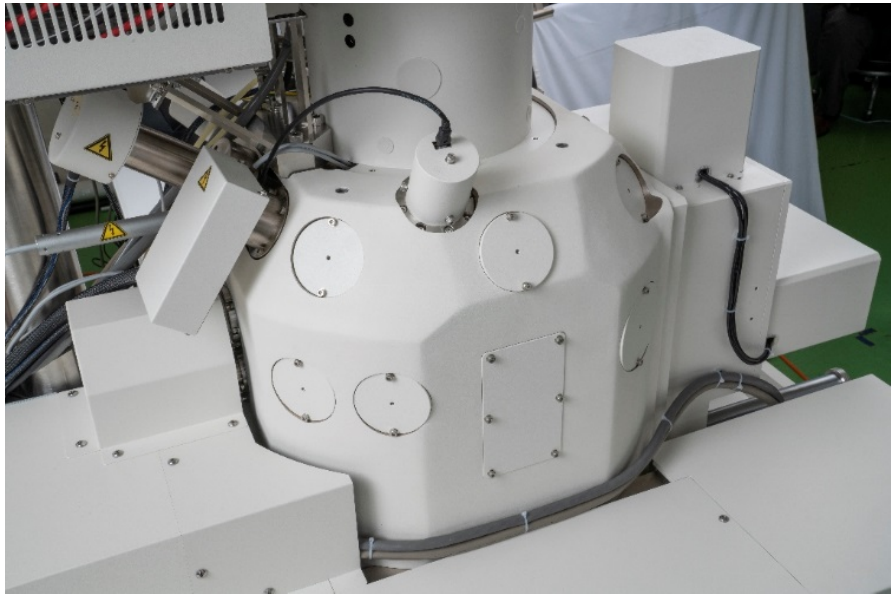
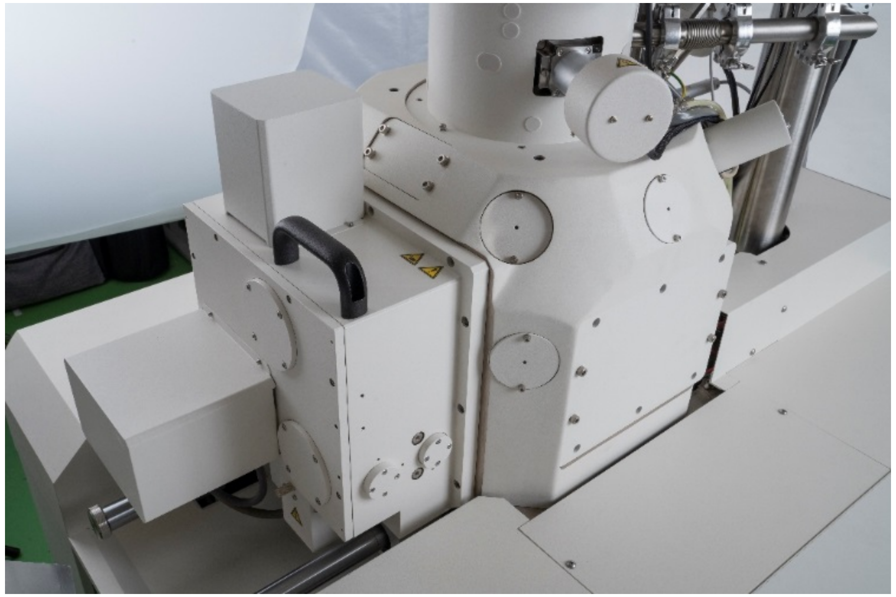
BSE

UVD (CL)

10 kV, 350 X

Flexible chamber, ports and stage



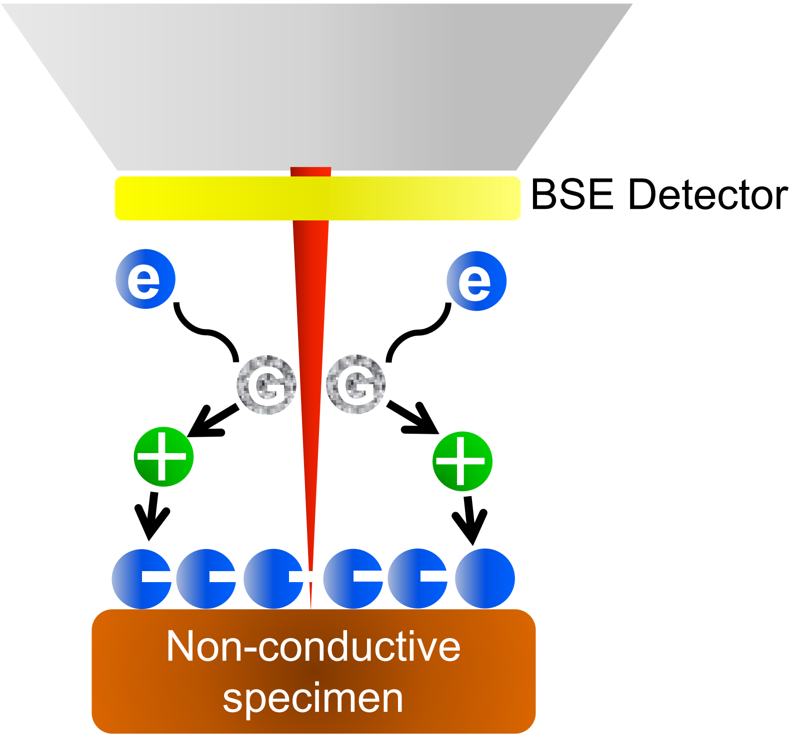


- Enough ports for potential accessories EDS(s), WDS, EBSD, CL(s), µXRF, substages, …
- Geometry where EBSD sits 90 degrees from tilt axis
- True eucentric stage, so that you move (XY) in the tilted sample plane
- Space for several EDS detectors from different directions (topography, speed)

- XY travel to be able to cover you largest sample or sample holder
- Ability to handle high samples
- Stable enough for heavy samples
- Sample holder to allow stable mounting

Low vacuum / Variable pressure operation

Utilizing a low vacuum environment can allow observation of water or oil based specimens in a natural state. The positively charged ions originated from the residual gas molecules generated by electron beam neutralize negatively charged electrons impinged on the specimen surface. Low vacuum observation eliminates traditional sample preparation requirements such as specimen dehydration or metal coating.



Neutralization of specimen surface by positive ions → Prevention of specimen surface charging

Low pressure environment → Wet, oily specimens can be observed with less evaporation

- G: Residual gas molecules
- e: Negative ion originated from residual gas molecule
- +: Positive ion originated from residual gas molecule
- ●: Negative ion on the surface

Low vacuum / Variable pressure operation

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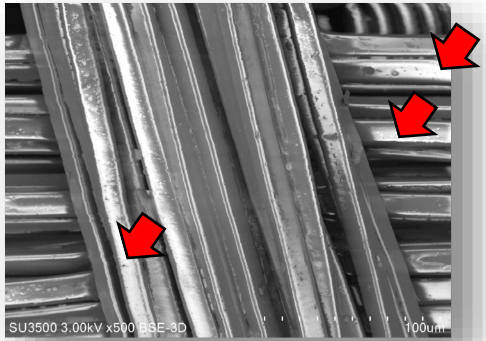
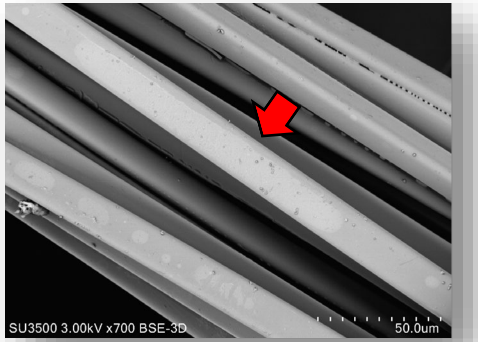
Charge free observation without metal coating of non-conductive specimen possible.

Metal coating, such as Au or Pd absorbs SE, BSE, and X-ray signals from the specimen and weaken SEM detectable signals.

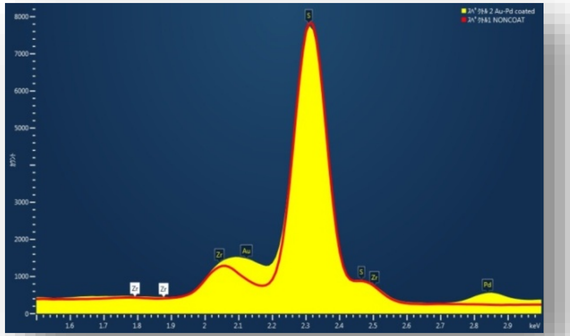
No peak overlapping X-ray analysis is possible without metal coating.



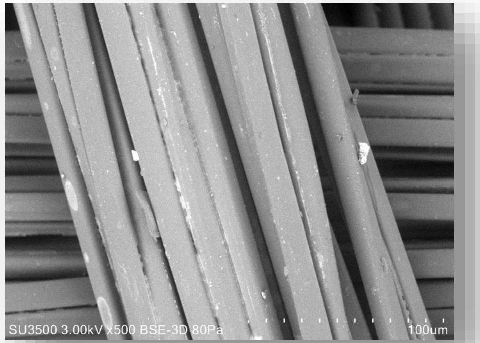
High Vacuum mode without metal coating :
Image distortion due to surface charging



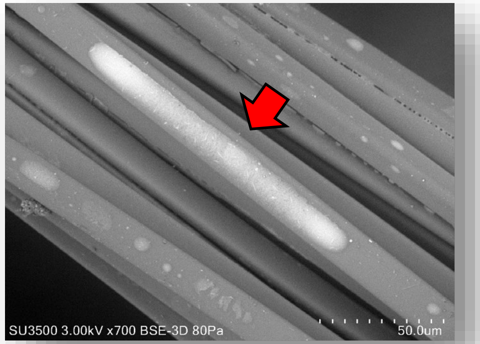
Observation with metal coating :
Material contrast of Ti (arrowed) is reduced by metal coating.



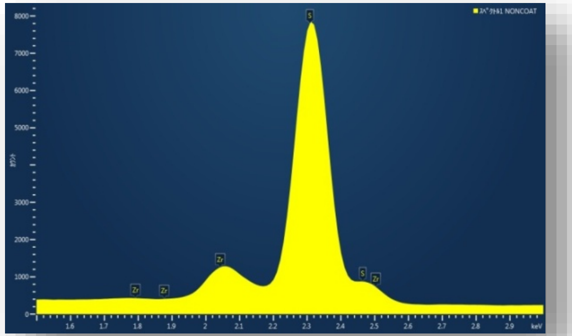
EDS Spectrums with metal coating :
Spectrums of Zr and Pt (coating material) are overlapped.



Low Vacuum mode without metal coating :
Less specimen surface charging.



Observation without metal coating :
Clearer material contrast of Ti (arrowed) at low vacuum mode



EDS Spectrums without metal coating :
Spectrums of Zr can be clearly identified

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Low-vacuum versus high-vacuum: No difference in FoV and current

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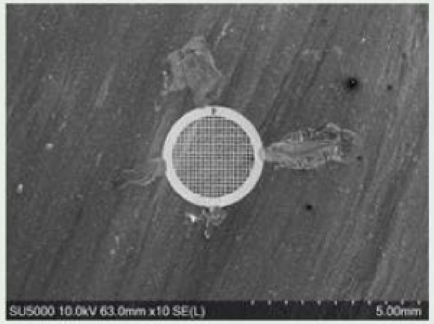
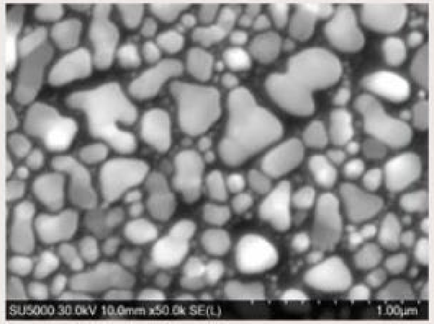
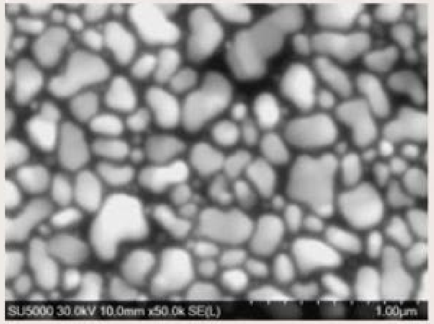
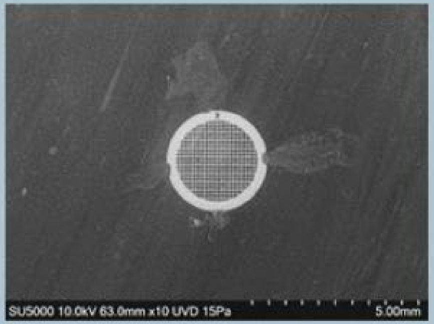
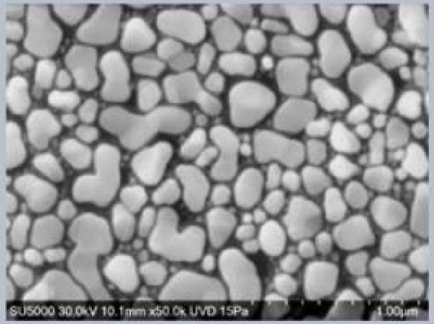
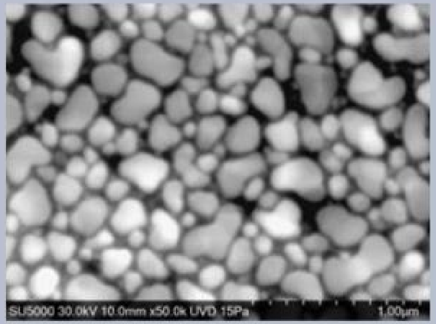
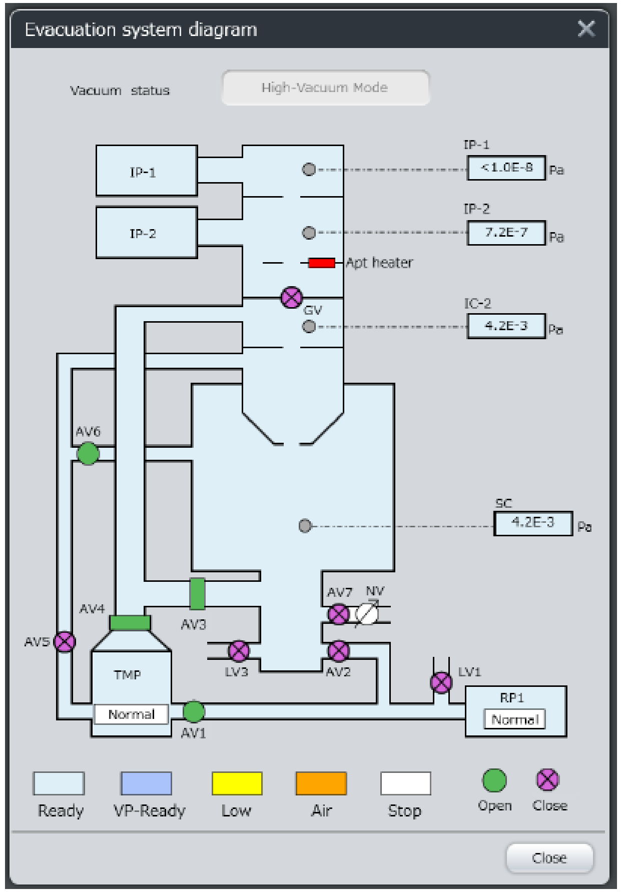
	Low magnification : 10x	Probe current: 50 nA Magnification : 50,000x	Probe current: 200 nA Magnification : 50,000x
HV High Vacuum < 7 x 10^{-4} Pa			
LV Low Vacuum 15 Pa			
	No restriction reduce field of view by aperture between HV and LV	High probe current utilize up to 200 nA for analytical between HV and LV	



No need to insert pressure limiting aperture for VP mode.

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Finding the area of interest

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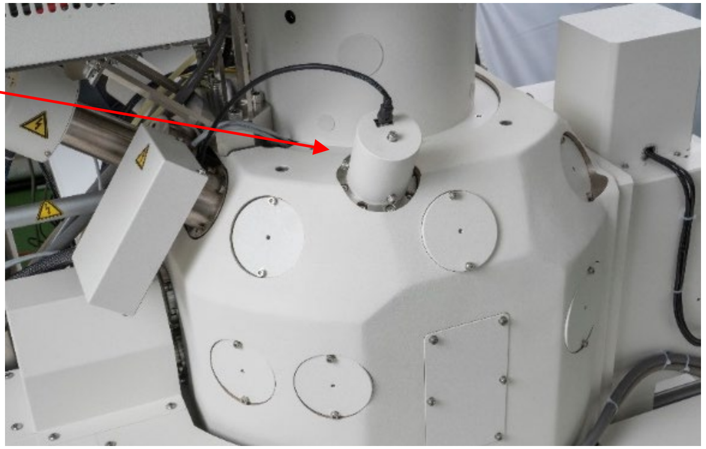
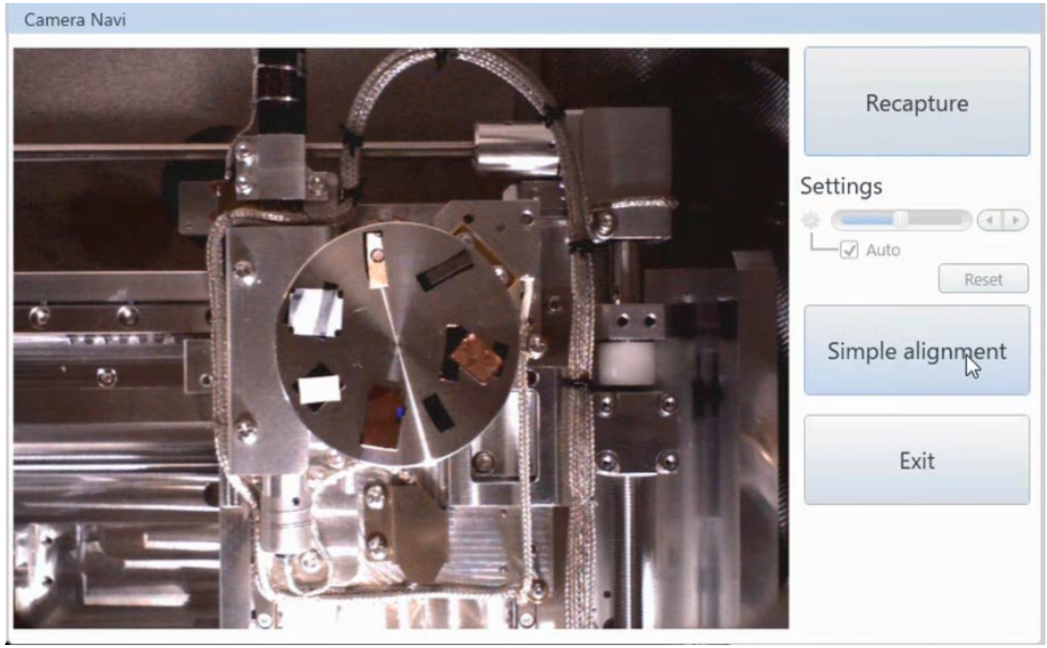
Navicam built into dedicated chamber port.



Automatic image acquisition during chamber evacuation



Automatically acquired picture of the sample holder and samples

Camera image transferred to the SEM-MAP for navigation

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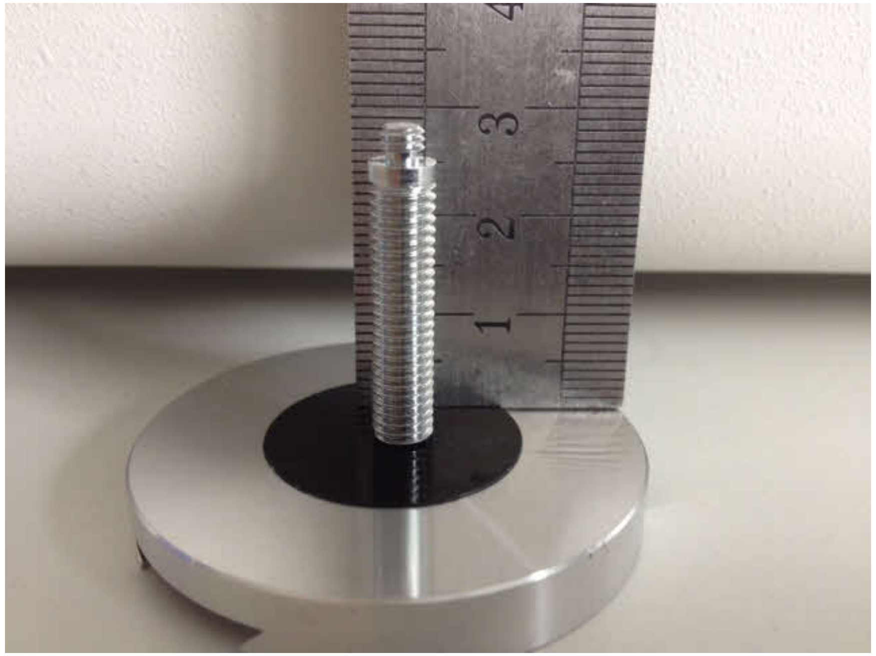
Low mag - Depth of focus

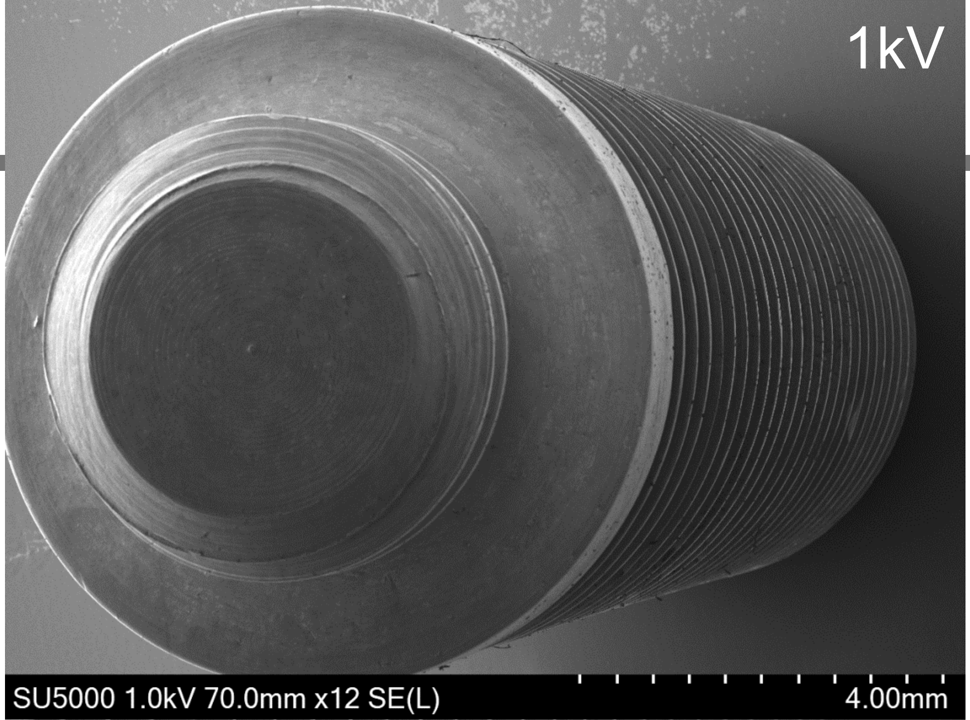
For many applications low magnification is more important than resolution.

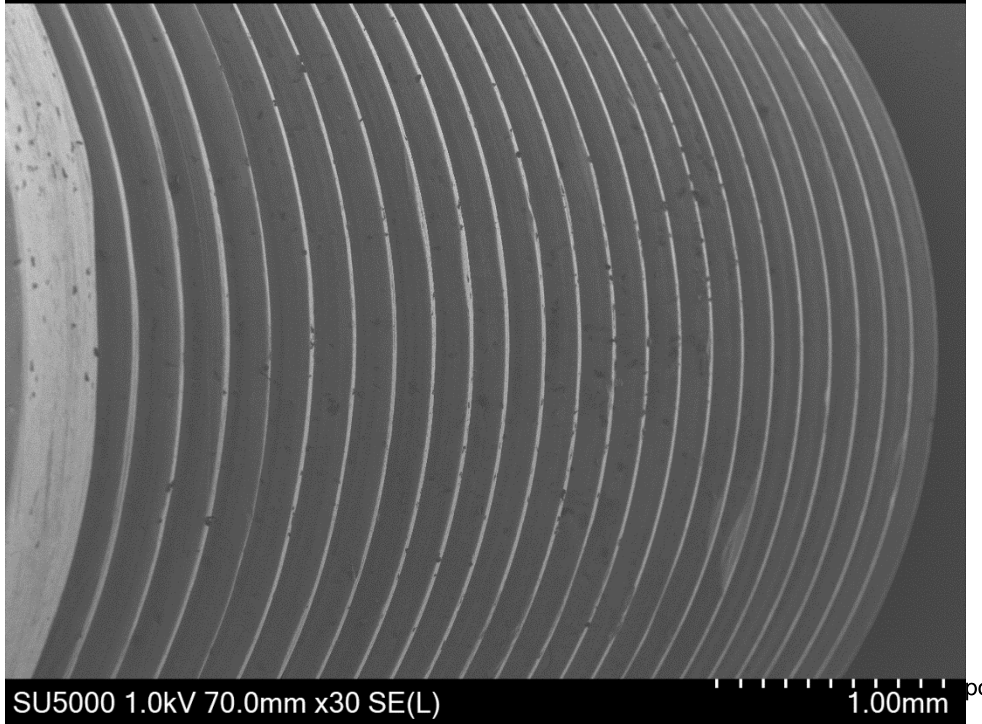
- To easily find where you are on the sample
- Easy correlative microscopy
- Taking overview images and EDS-maps

When you have rough or tilted samples (EBSD) depth of focus and potential distortions becomes critical



30mm long screw on SU5000 sample holder





Low distortion at low mag on tilted samples

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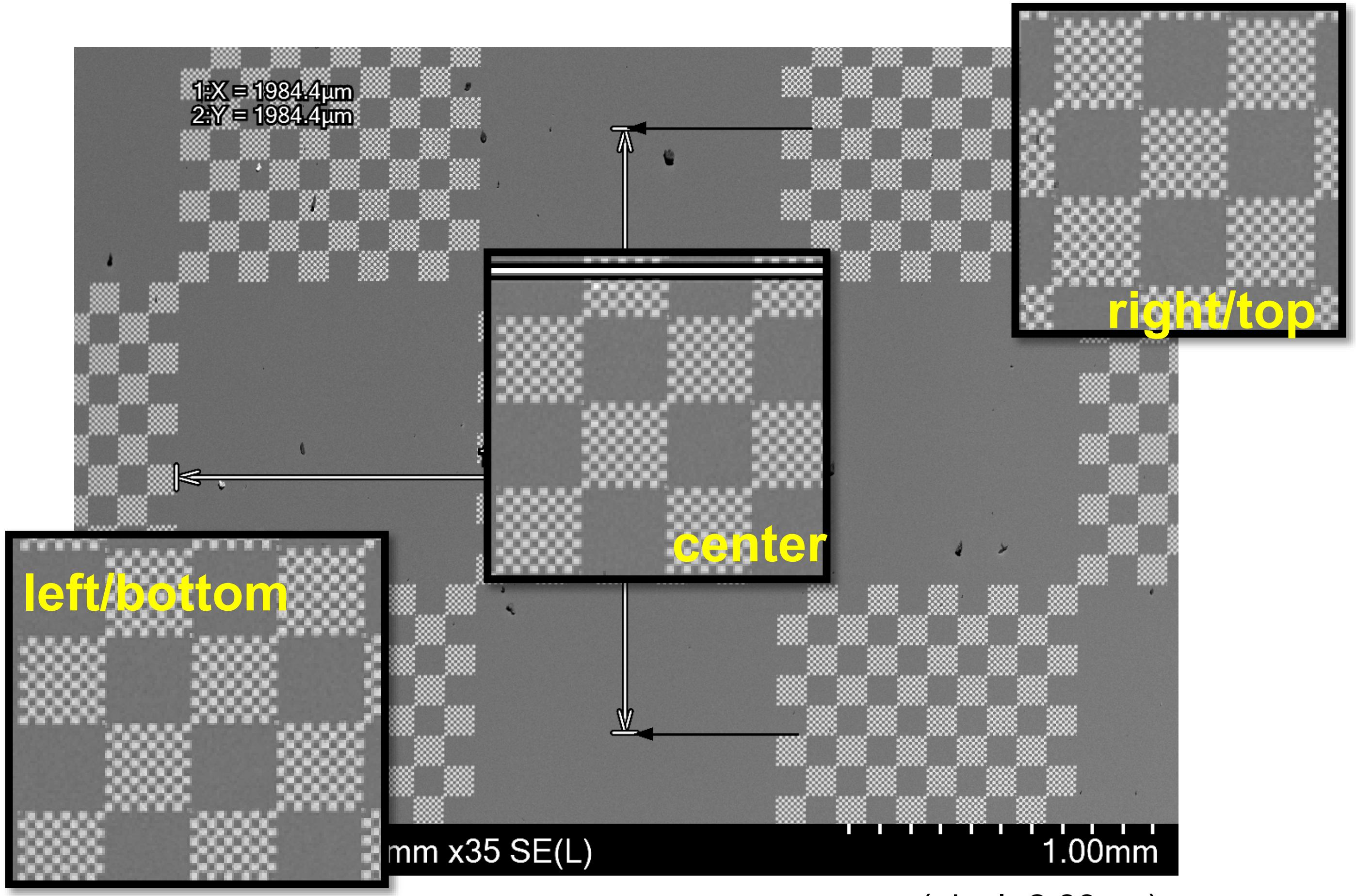
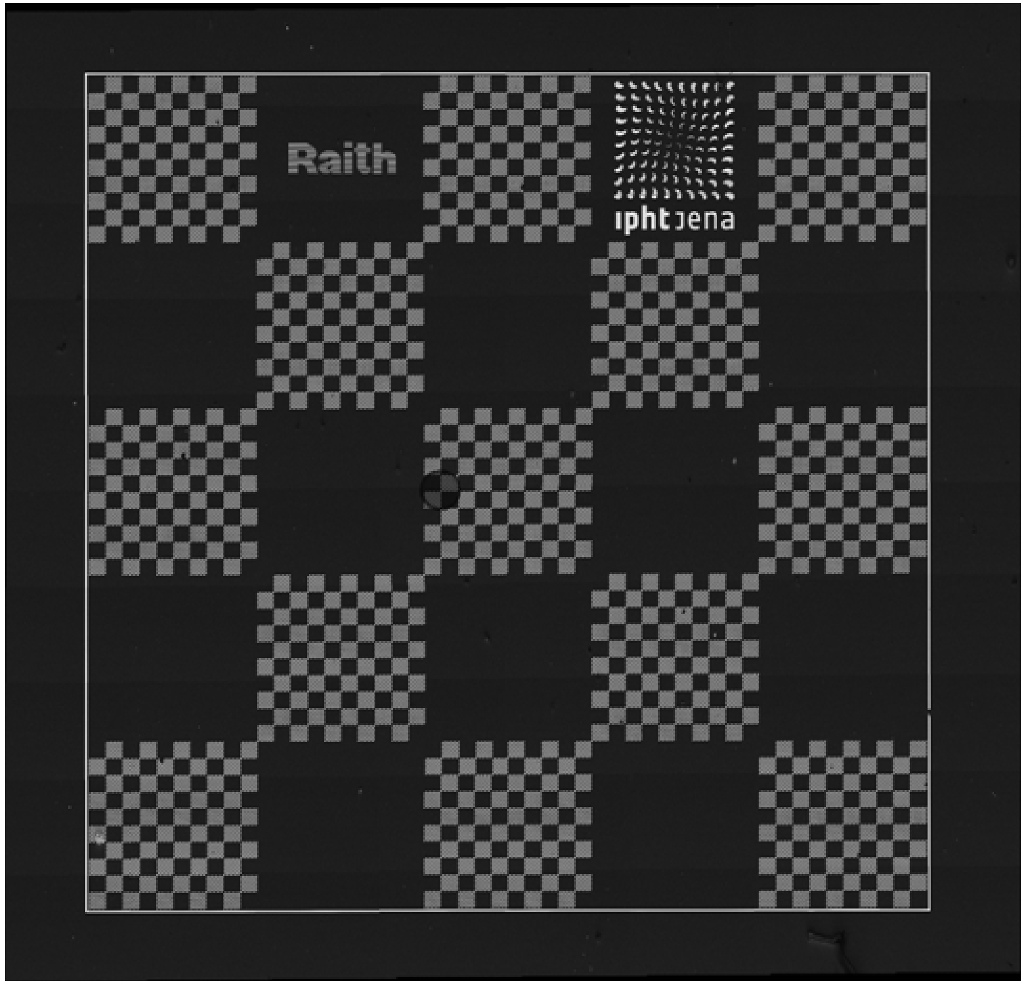
10μm lattices are in focus through 2.7mm DoF



(pixel=2.83um)

Critical for large area EBSD measurements



6x6 mm area, 8x10 images stitched, at 70 degrees tilt

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Easy imaging and analysis at the same WD and settings

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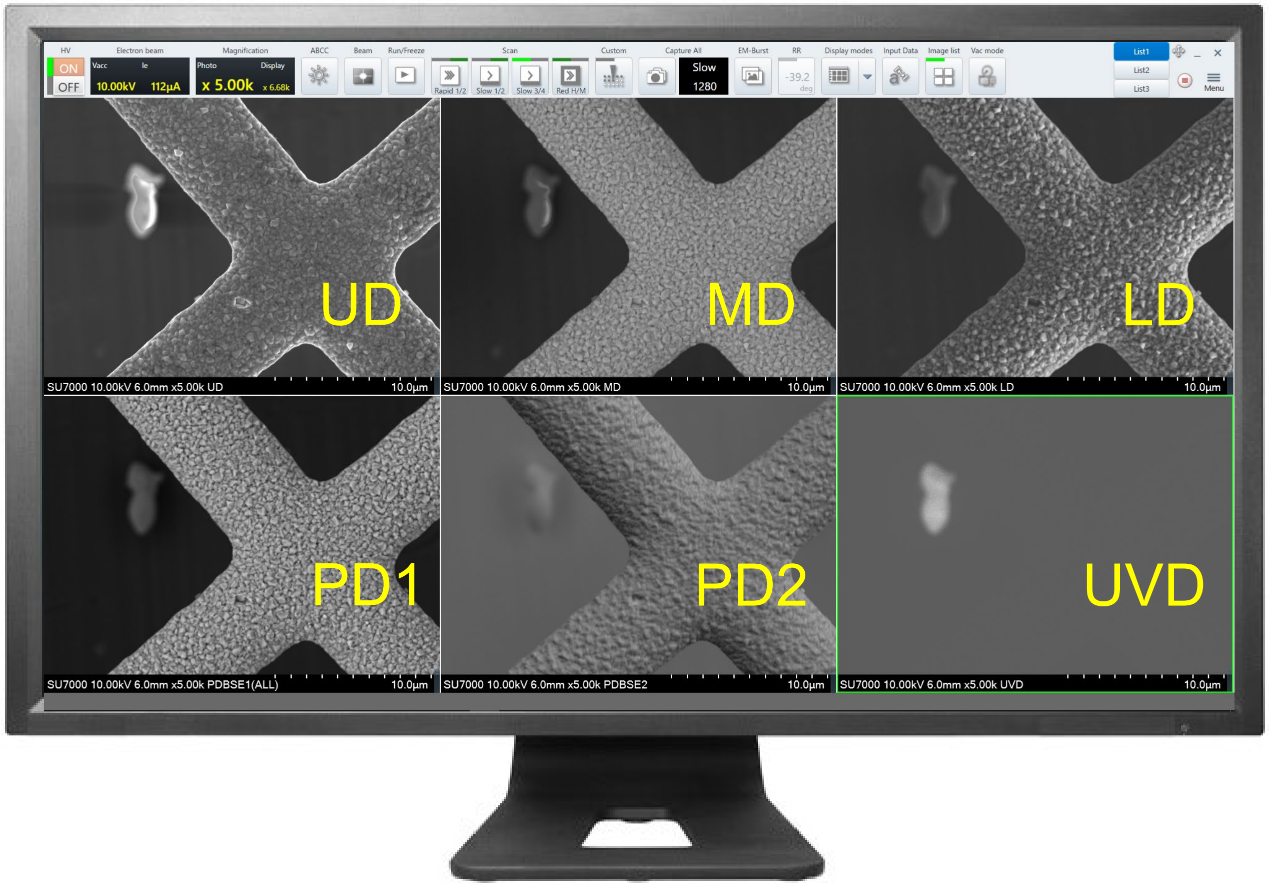

Dedicated use of left screen for image display
Easy image comparison by signal type
6 x identical picture size

Operation related windows are moved to the right screen for improved usability.
SEM-MAP and chamber scope images can also be displayed.

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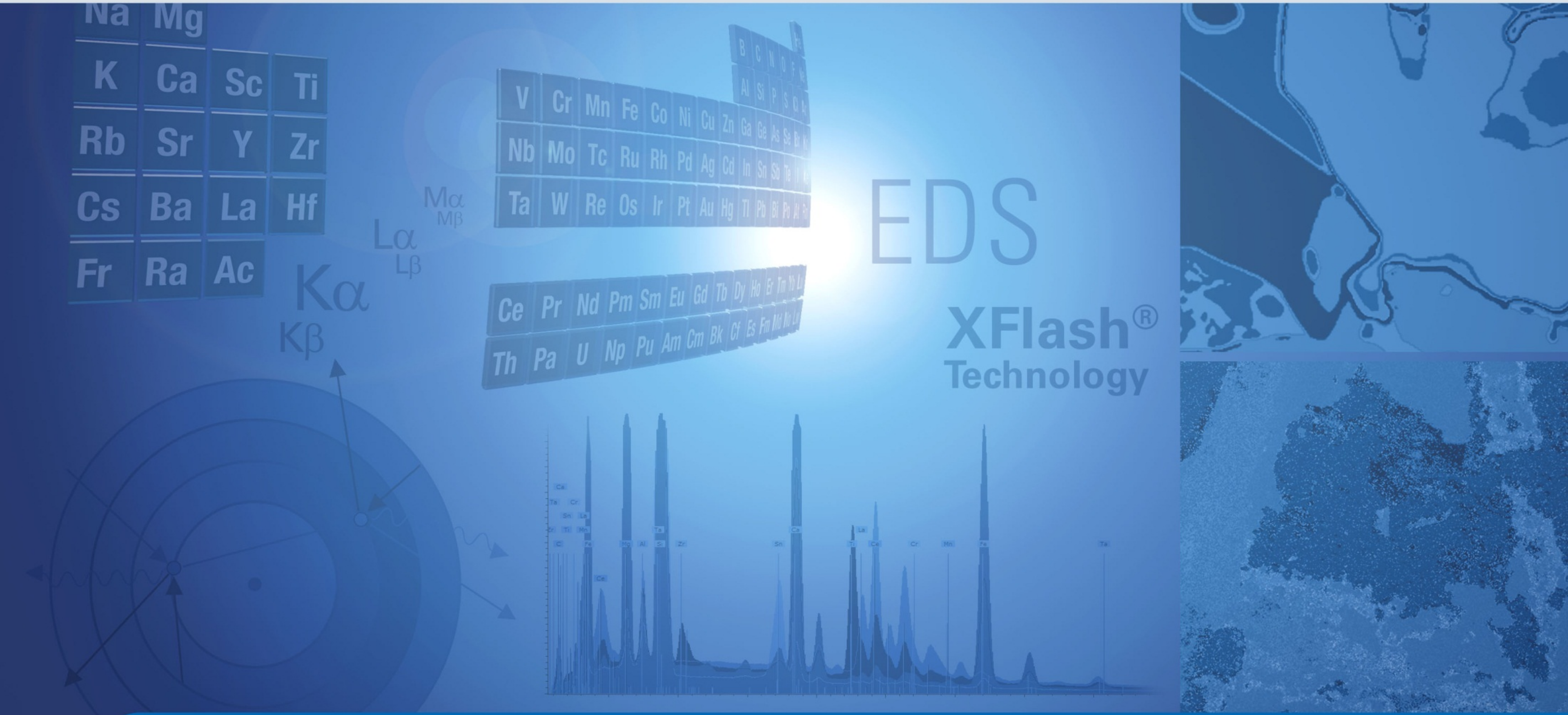
Thank you !

Advanced Microanalysis With The QUANTAX System

Max Patzschke



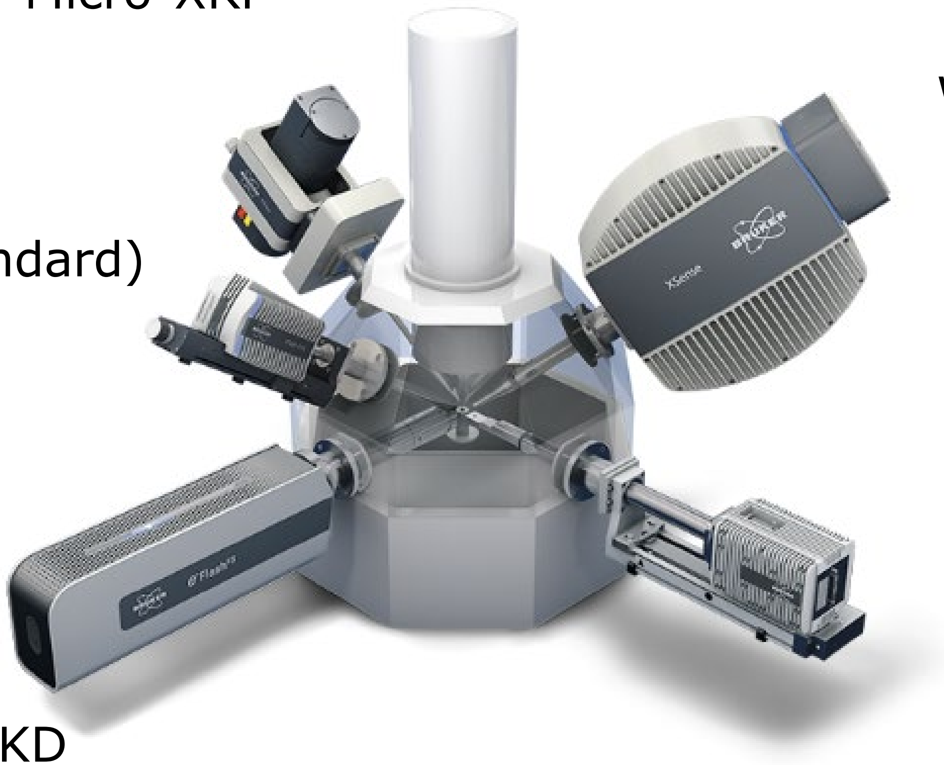
Innovation with Integrity

Overview

- Introduction to QUANTAX Electron Microscope Analyzers
- EDS maps with overlapping lines (S, Pb and Mo)
- Annular FlatQUAD detector for EDS
- Fast maps using the FlatQUAD detector
- AMICS – automated mineral classification and identification software
- Summary

Bruker Nano Analytics Division

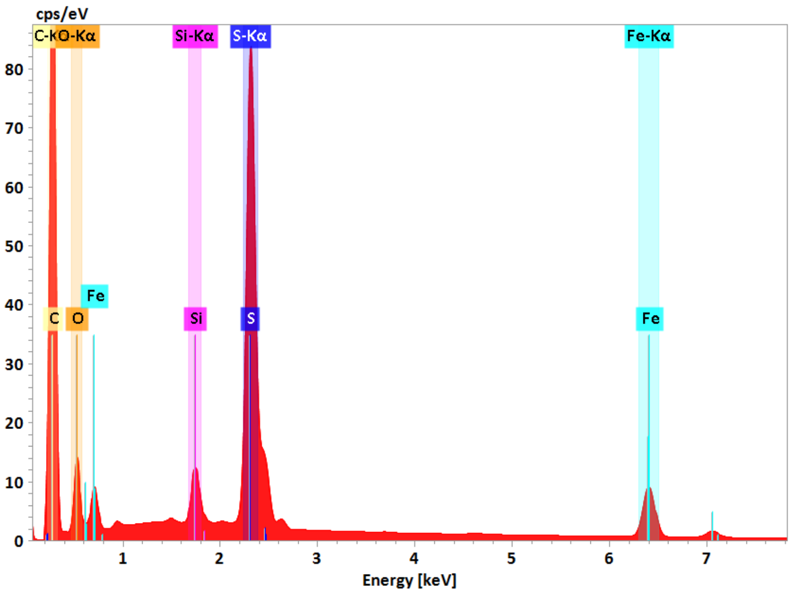
EM Analyzers

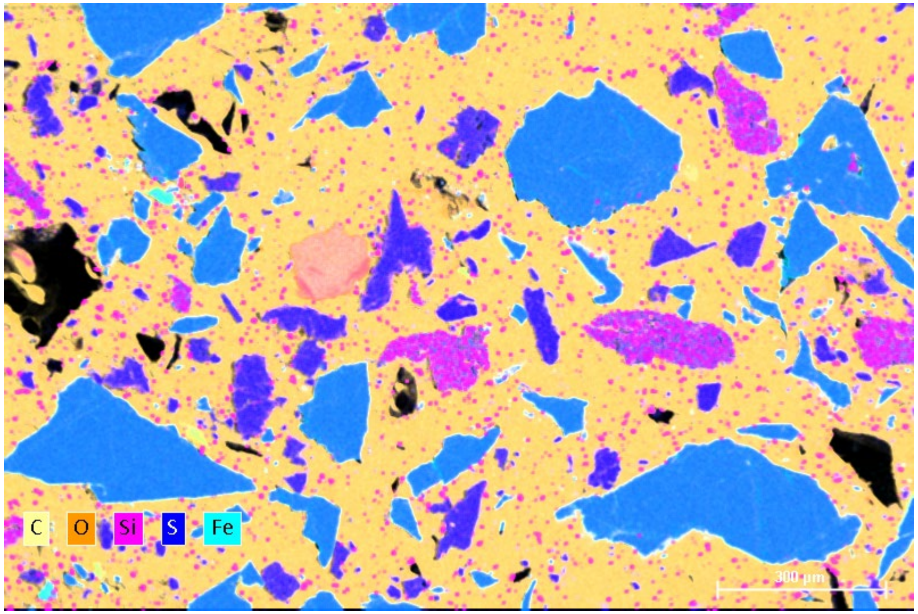


Bruker Nano Analytics Division
EM Analyzers

ESPRIT for EDS

Peak intensity (ROI) maps





Peak intensity maps: an element is displayed by the number of total counts in a predifined energy region around the peak.

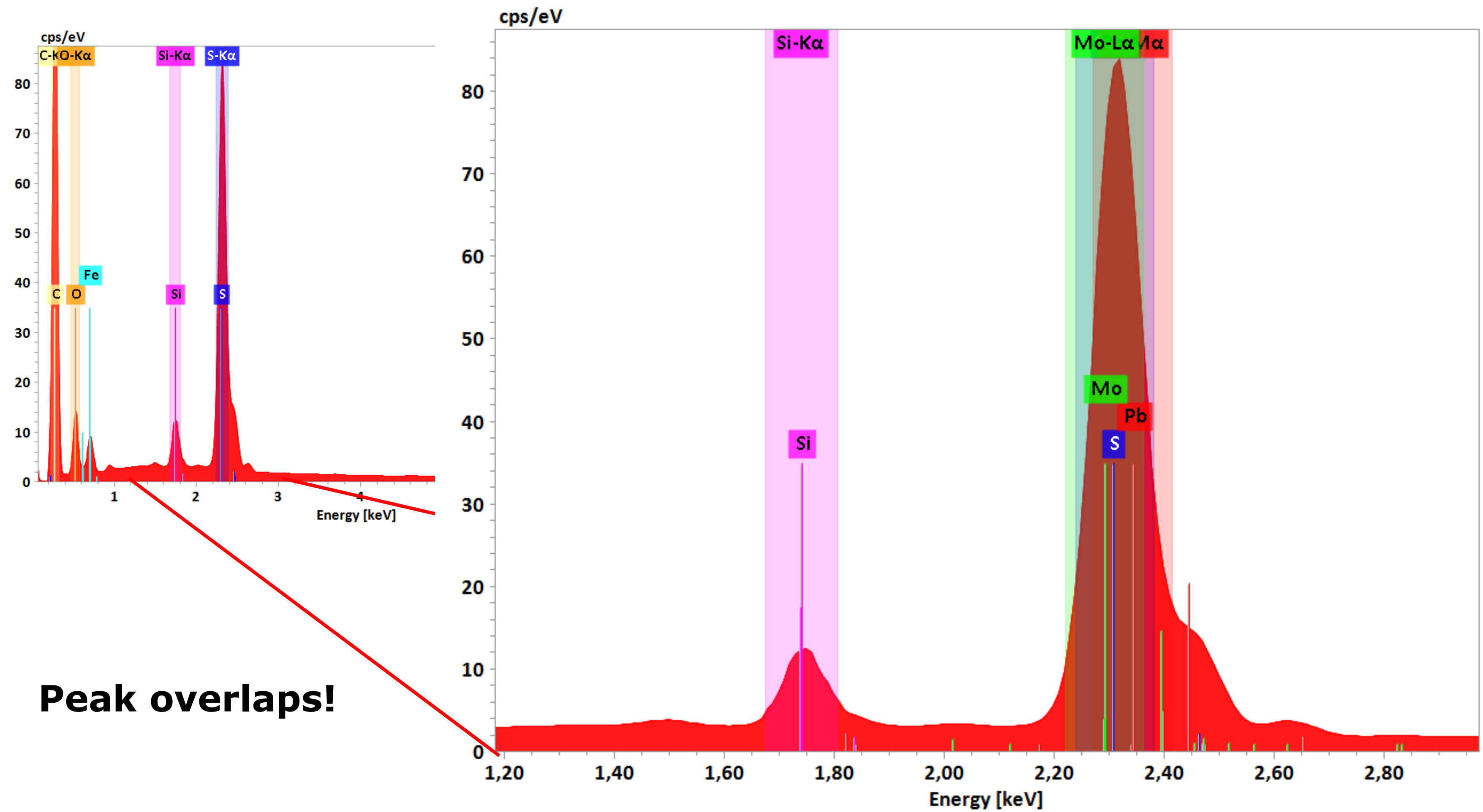
Peak overlaps of different elements

Element and line	Interferences with	Δ eV	Samples or applications where the overlaps are found
Cu-L	Na-Kα	18	Biological samples (grid)
As-L	Na-Kα	79	Biological samples (stain or fixative)
Ag-L	Cl-Kα	10	Biological samples (stain or fixative)
Ru-L	S-Kα	54	Biological samples (stain or fixative)
Os-M	Al-Kα	5	Biological samples (stain or fixative)
U-M	K-Kα	22	Biological samples (stain or fixative)
Sr-Lα	Si-Kα	31	Silicates (feldspars in particular)
Y-Lβ	P-Kα	18	Phosphates
Y-Lβ	Zr-Lα	46	Silicates (zircon), oxides (zirconia)
S-Kα,β	Mo-Lα; Pb-Mα	14; 38	Minerals, lubricants, sulfides, sulfates
Ti-Kβ	V-Kα	20	Steels, Fe-Ti oxides
V-Kβ	Cr-Kα	13	Steels
Cr-Kβ	Mn-Kα	47	Steels
Mn-Kβ	Fe-Kα	87	Steels
Fe-Kβ	Co-Kα	128	Steels, magnetic alloys
Co-Kβ	Ni-Kα	169	Steels, hard surfacing alloys
W-Mα,β	Si-Kα,β	35	Semiconductor processing
Ta-Mα,β	Si-Kα,β	27	Semiconductor processing
Ti-Kα	Ba-Lα	45	Optoelectronics, silicates
As-Kα	Pb-Lα	8	Pigments

Overlaps known from biological, geological and material sciences and industries

Modified after Goldstein et al. (2007). Scanning Electron Microscopy and X-Ray Microanalysis. Springer

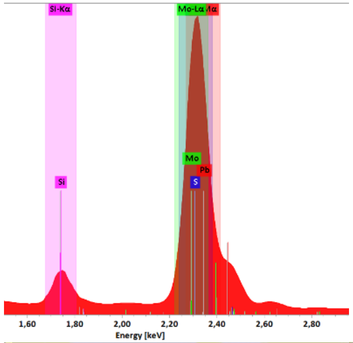
ESPRIT for EDS
Peak intensity (ROI) maps

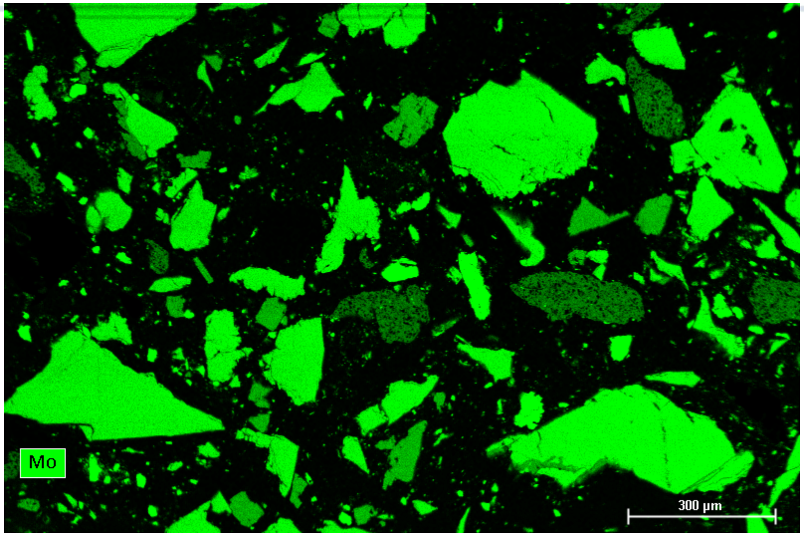


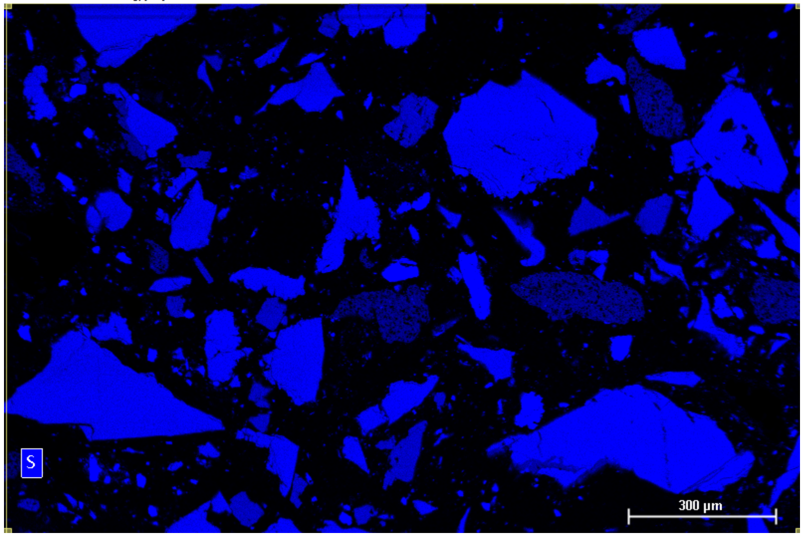
Peak overlaps!

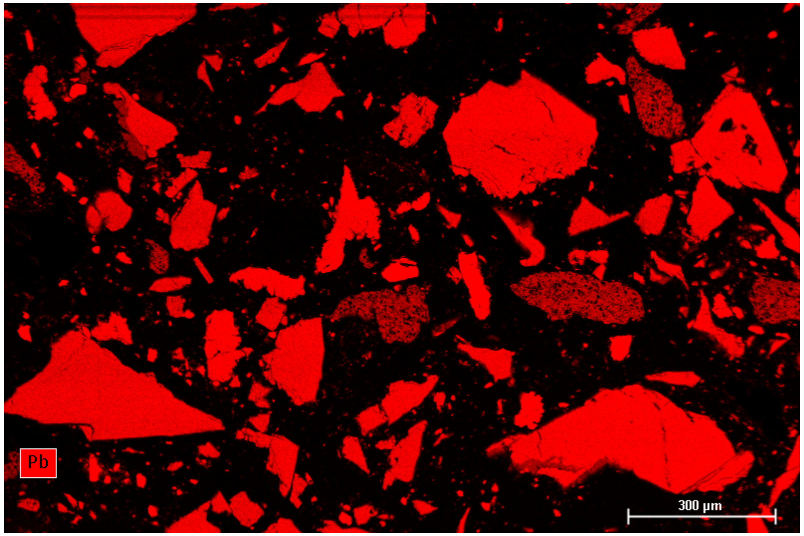
ESPRIT for EDS
Peak intensity (ROI) maps

Peak intensity maps show same distribution for elements with overlapping energies!





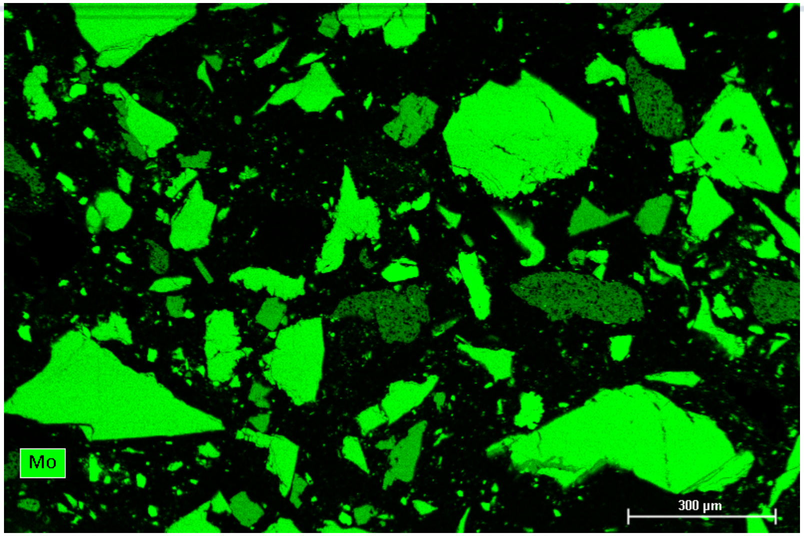


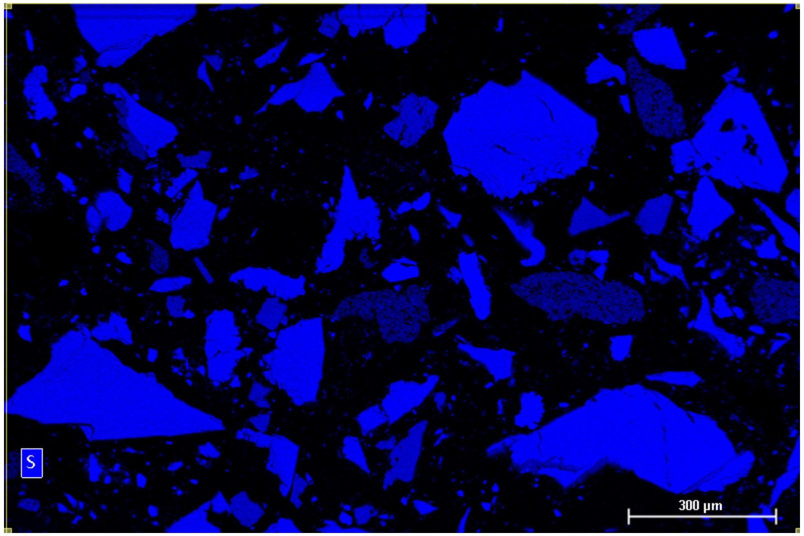


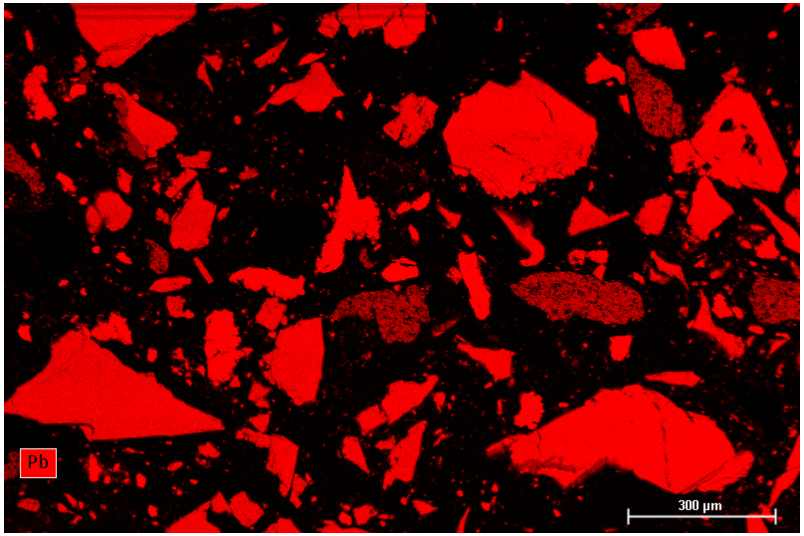
ESPRIT for EDS
Peak intensity (ROI) maps

Peak intensity maps show same distribution for elements with overlapping energies!

What to do?





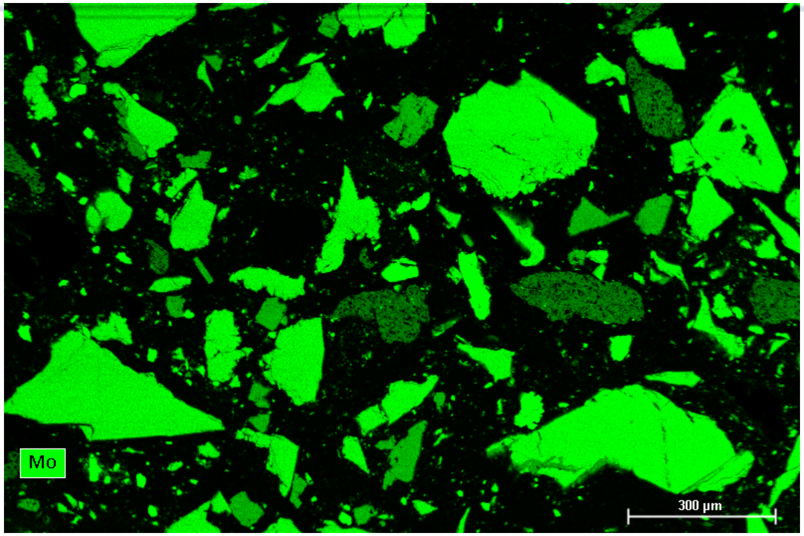


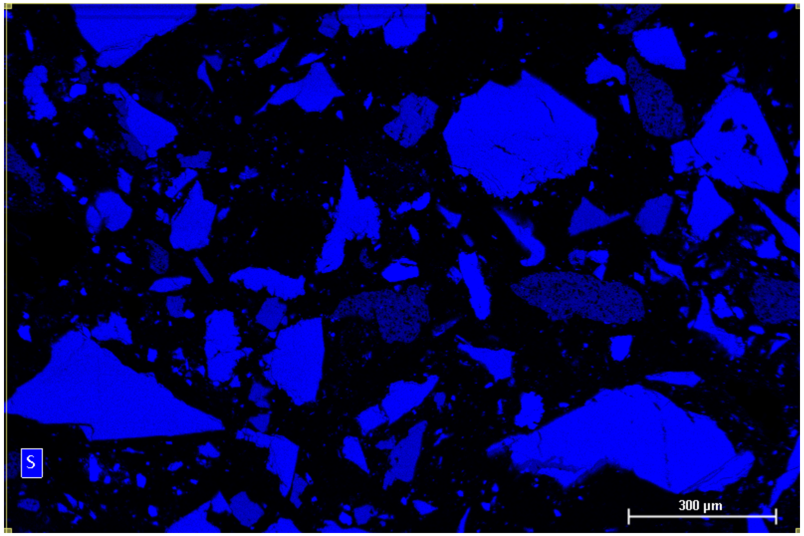
ESPRIT for EDS
Peak intensity (ROI) maps

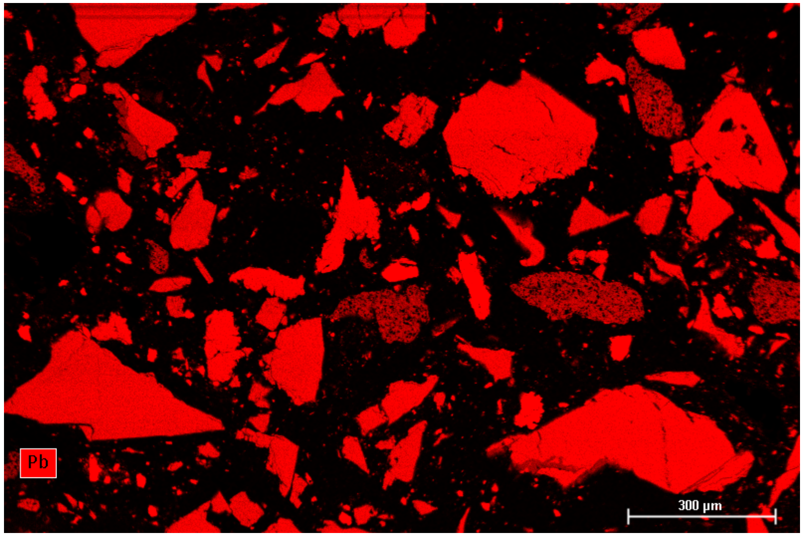
Peak intensity maps show same distribution for elements with overlapping energies!

What to do?

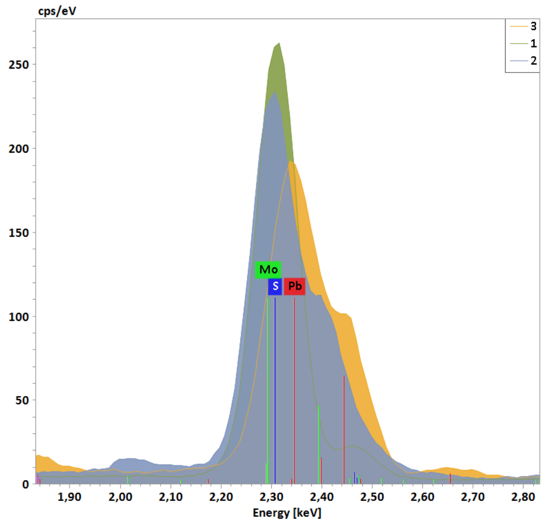
Need Deconvolution of peak intensities of the map!



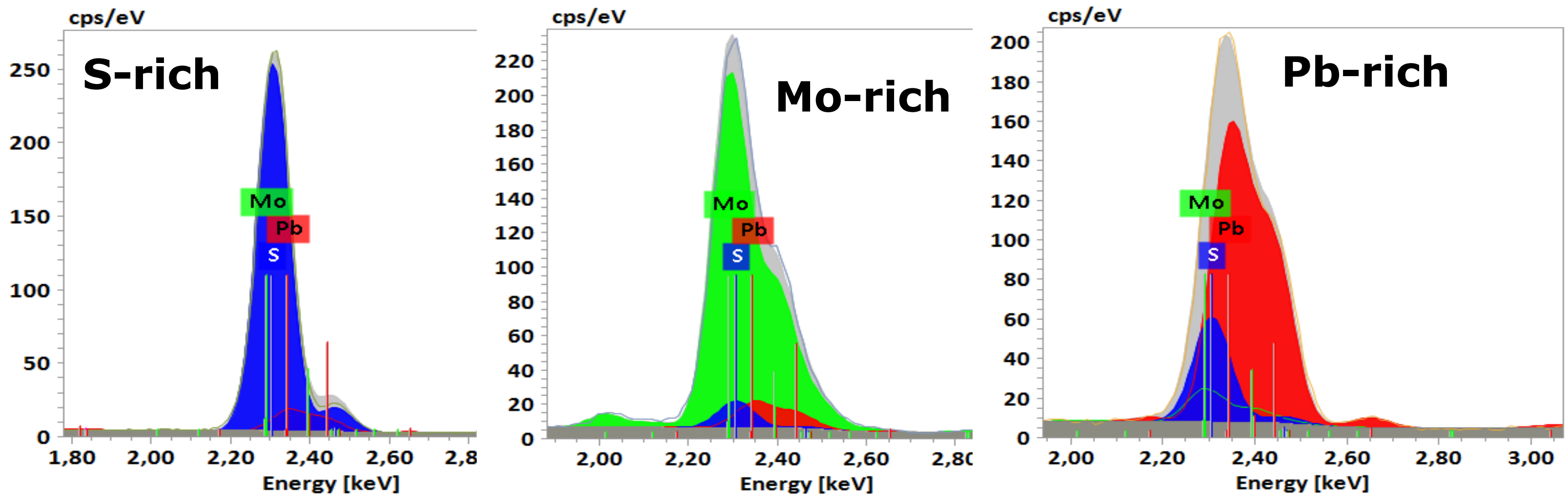




ESPRIT for EDS
Deconvolution of peak intensities



Impulses of the spectra are correlated to identified elements.
An „experimental" spectrum is compiled and compared with the acquired spectrum.

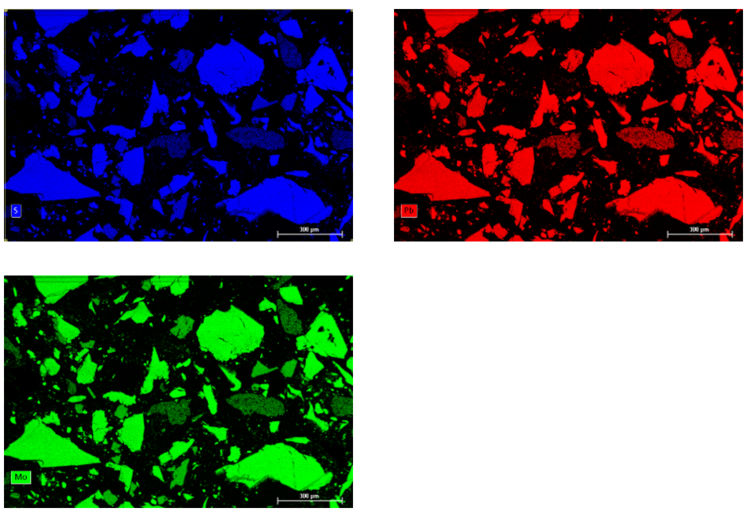


ESPRIT for EDS
Peak intensity maps vs. net intensity map

Same map data of elements with **overlapping peaks**.
Different interpretations:

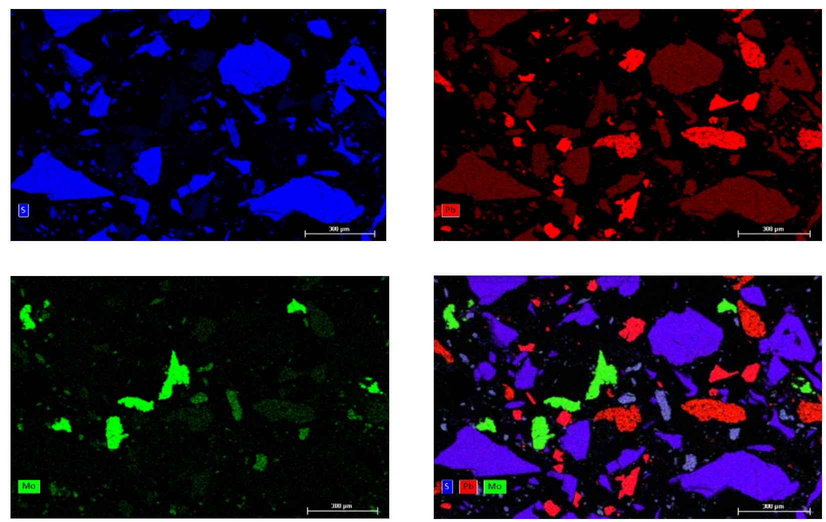
Peak intensity map:

Fake element distribution!

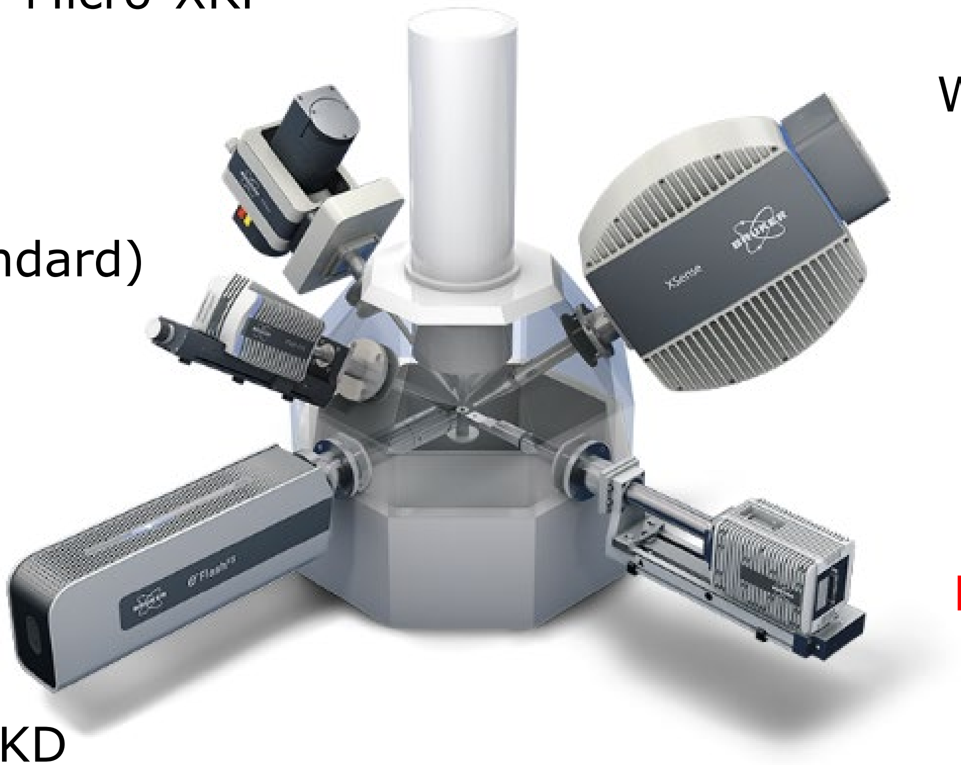


Net intensity (deconvolved) map:

Correct distribution of elements



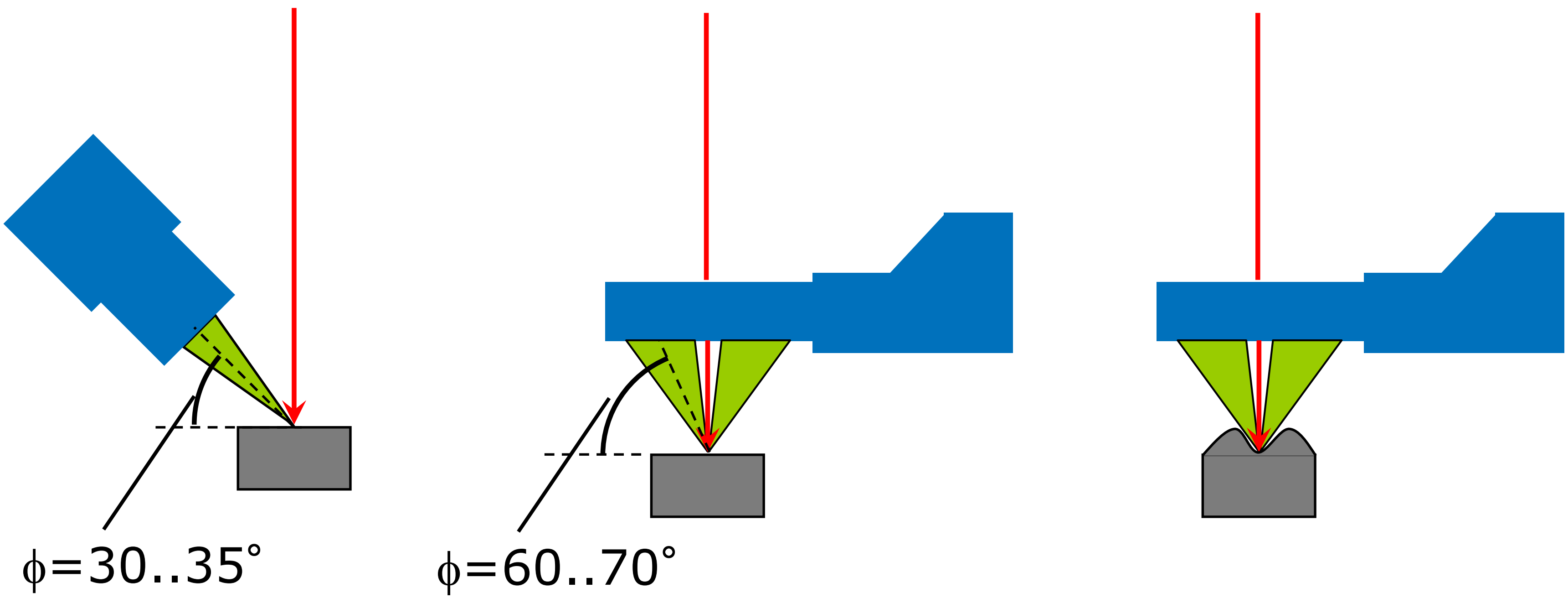
Bruker Nano Analytics Division
EM Analyzers



ESPRIT for EDS with the FlatQUAD

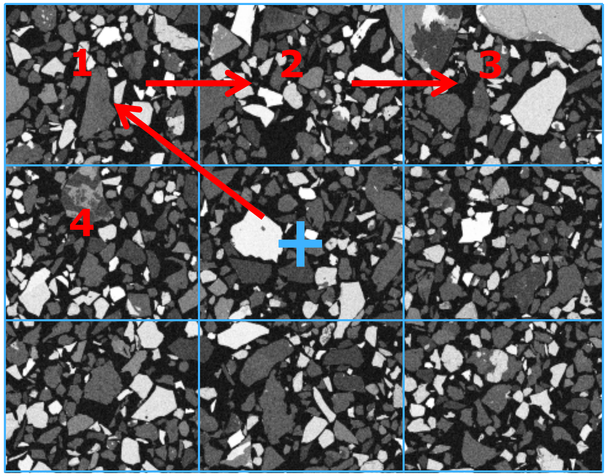
Advantage of high take-off angle and annular design

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



max solid angle at
d = 2.5mm:
Ω > 1.1 sr

ESPRIT for SEM-EDS
Hypermapping: Image Extension



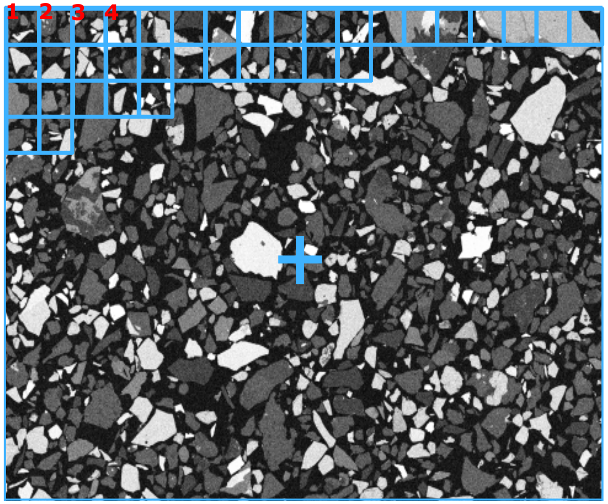
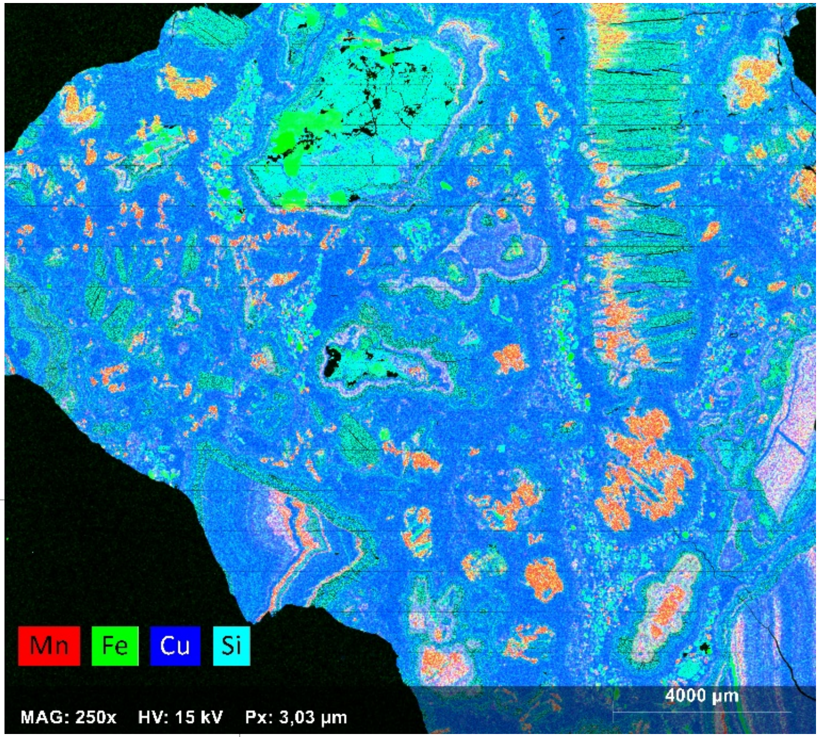


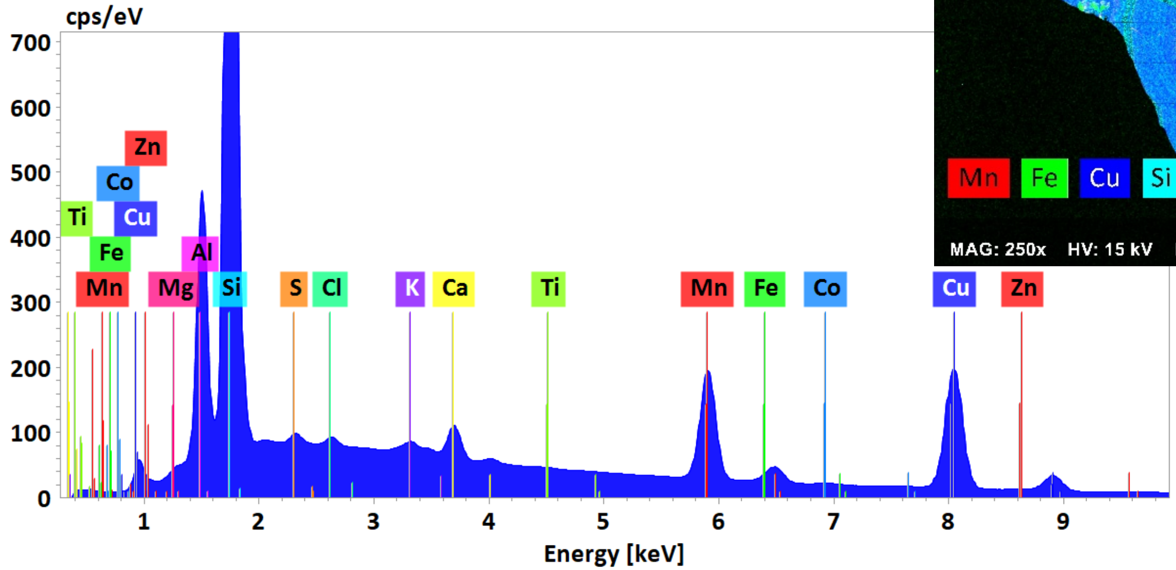
Image Extension

- Use actual sample position as central Mapping position and define number of x/y frames around
- Result: **one** Hypermap file
- Image extension can be enlarged for a full sample map with more than 20,000 x 15,000 pixels

ESPRIT for EDS with the FlatQUAD
Large area Hypermap

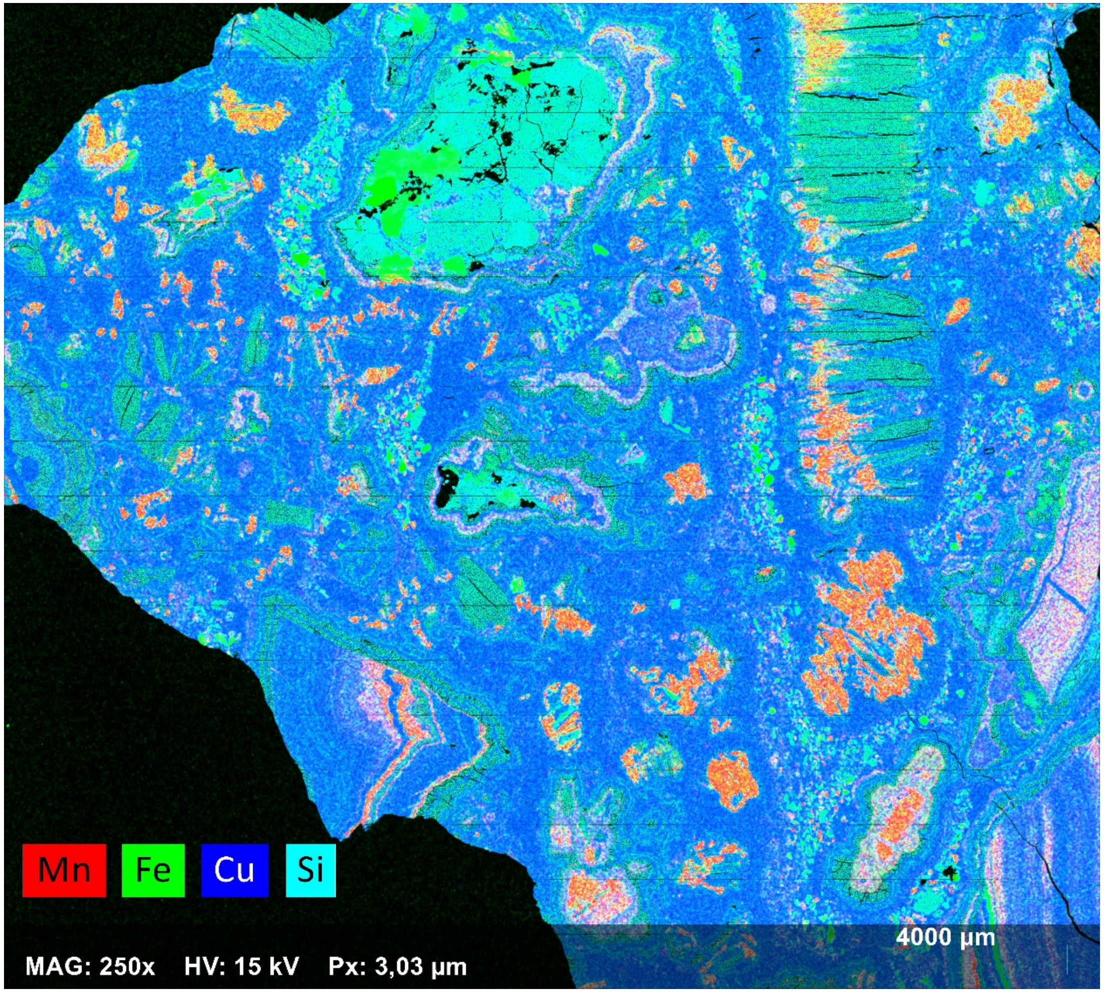
- Image Extension Map with 10x10 fields stitched automatic together
- FOV 18.1mm
- Aquired at 15kV,





ESPRIT for EDS with the FlatQUAD
Large area Hypermap

- Image Extension Map with 10x10 fields stitched automatic together
- FOV 18.1mm
- Aquired at 15kV,
- **3 s per field (406 s total mapping time)**
- **ICR: 880 kcps**



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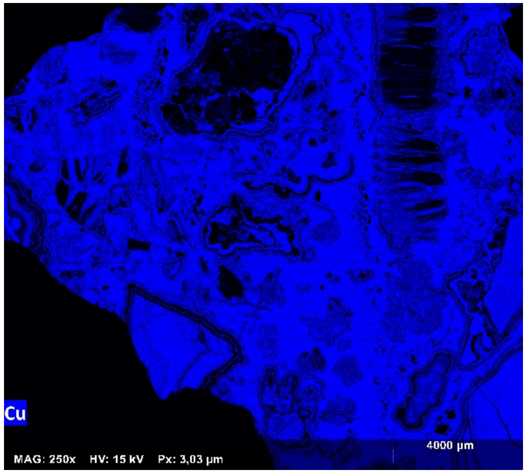
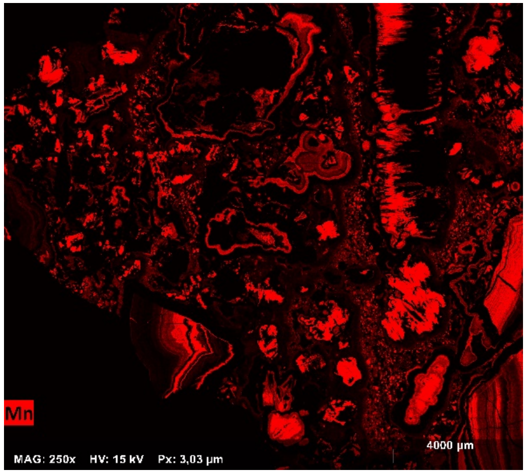
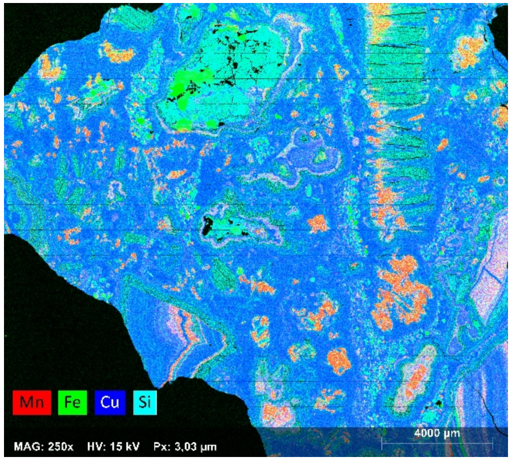
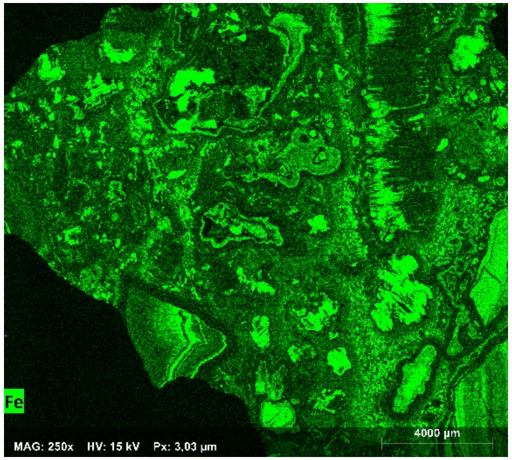
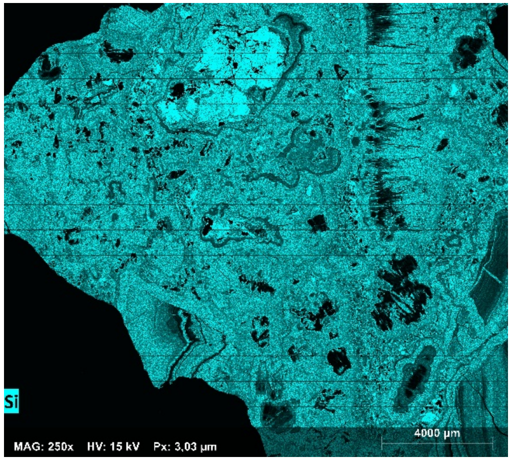






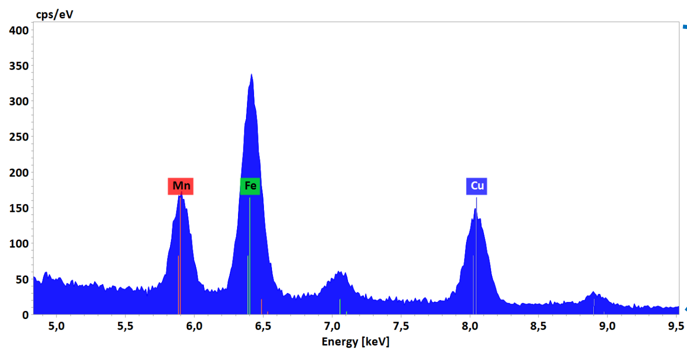


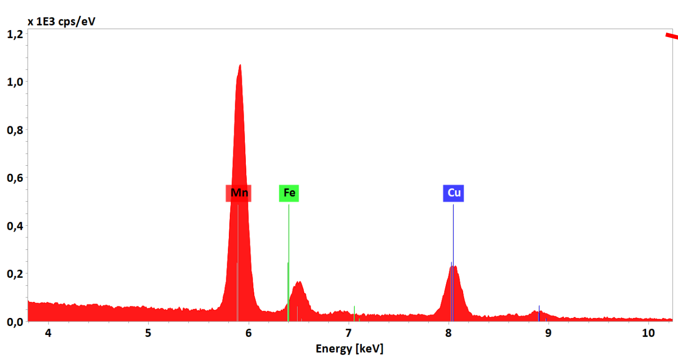


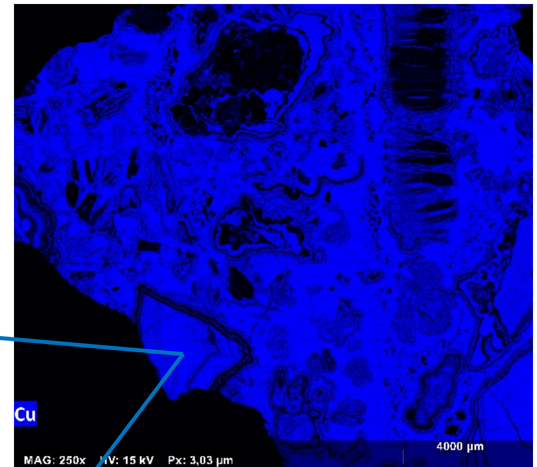
ESPRIT for EDS with the FlatQUAD

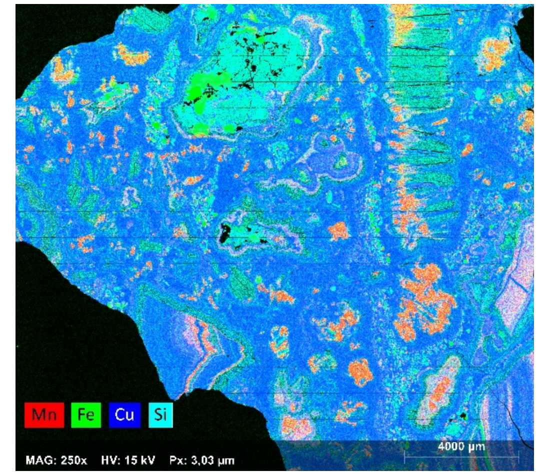
Large area Hypermap

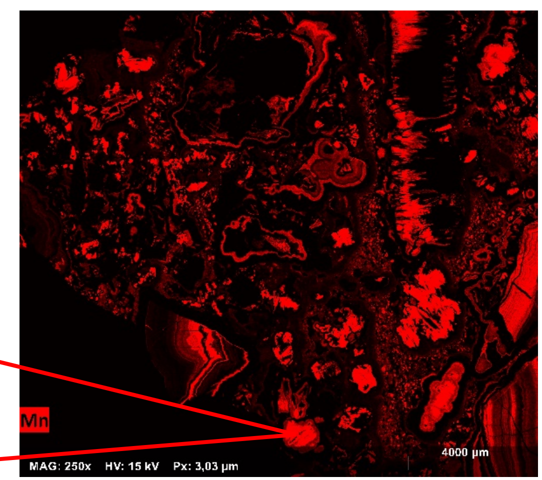
- Complete offline data processing
- Spectrum storage for each pixel







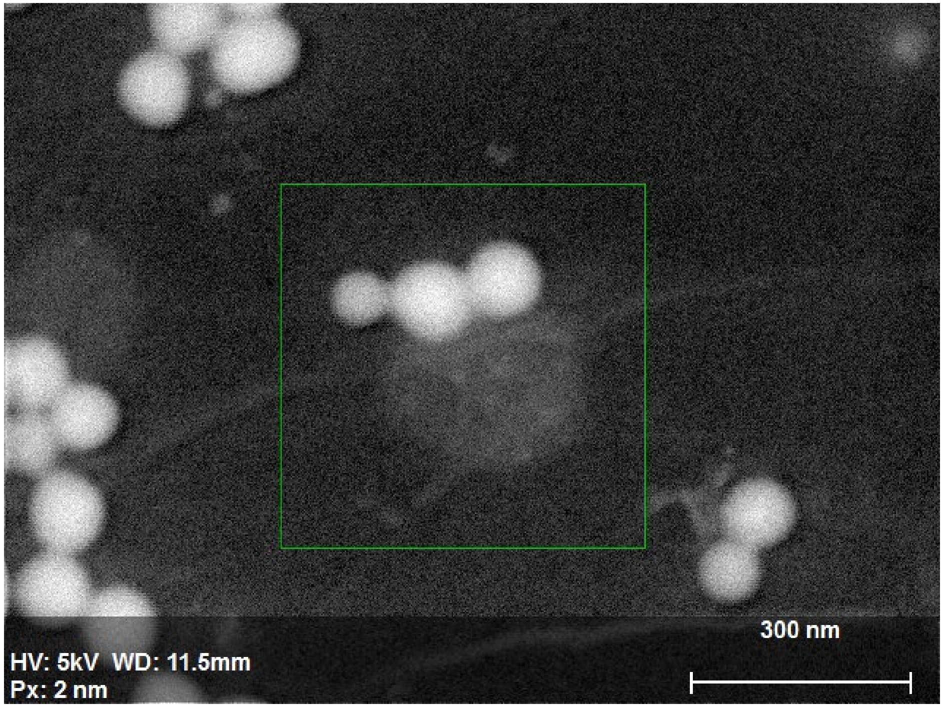


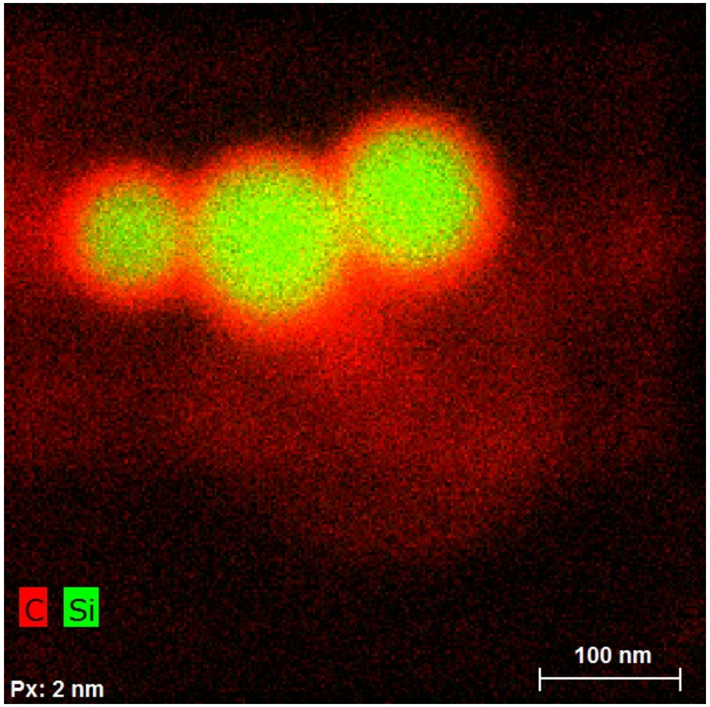


ESPRIT for EDS with the FlatQUAD

Silica nanoparticles

XFlash FlatQUAD, 5 kV, 520 pA , 22.5 kcps, 250x250 pixel, 2 nm pixel size, 377 s

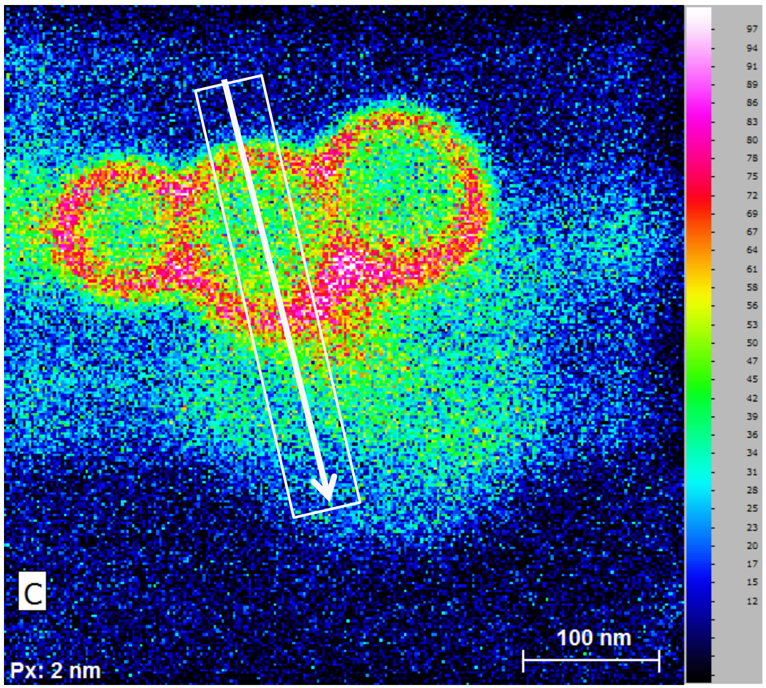




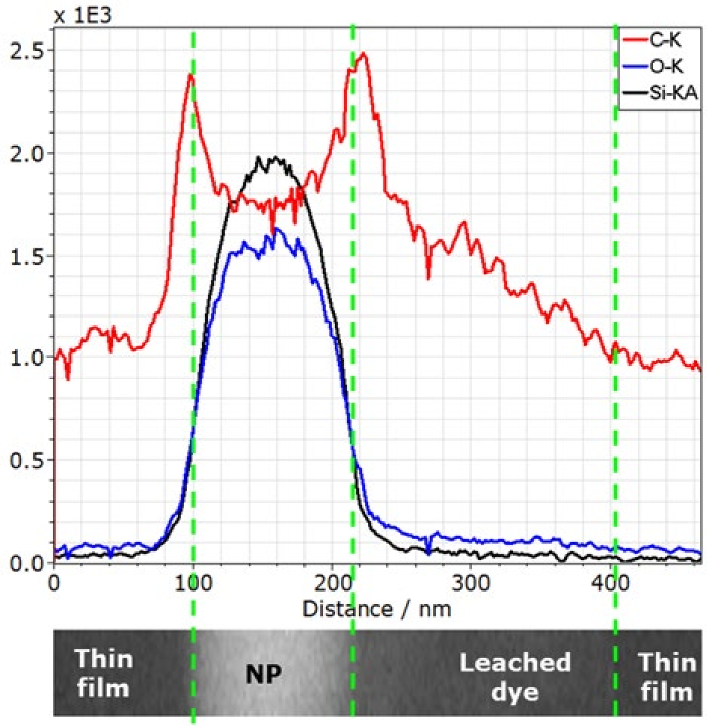
K. Natte, T. Behnke, G. Orts-Gil, C. Würth, J. F. Friedrich, W. Österle and U. Resch-Genger, J Nanopart Res, 2012, 14, 680

ESPRIT for EDS with the FlatQUAD
Silica nanoparticles

XFlash FlatQUAD, 5 kV, 520 pA , 22.5 kcps, 250x250 pixel, 2 nm pixel size, 377 s



Rades et al



Nano Valid

AMICS for QUANTAX – EDS on SEM

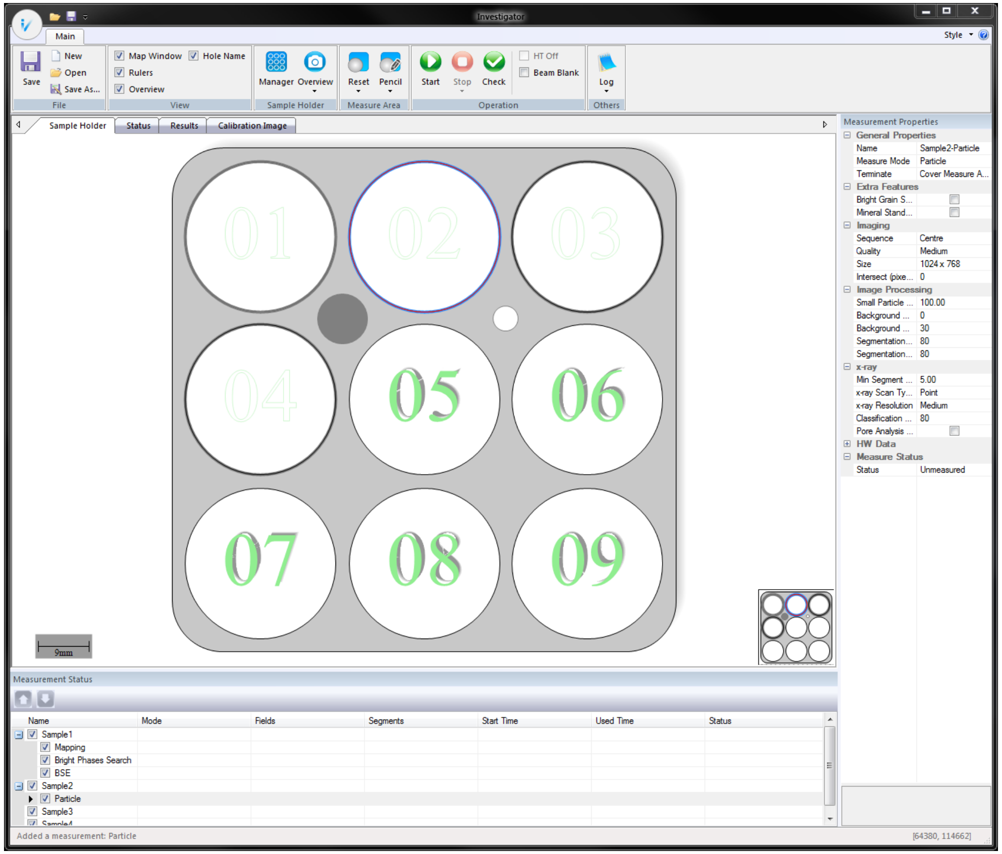
AMICS for QUANTAX EDS
How does AMICS work

AMICS - easy to configure measurement area by definable sample block holders

And can perform the following

- Particle
- Mapping
- BSE Image acquisition
- Standards acquisition
- Bright Phase Search

Controls SEM settings, moves stage to each position and acquires images through the QUANTAX system.



AMICS for QUANTAX EDS
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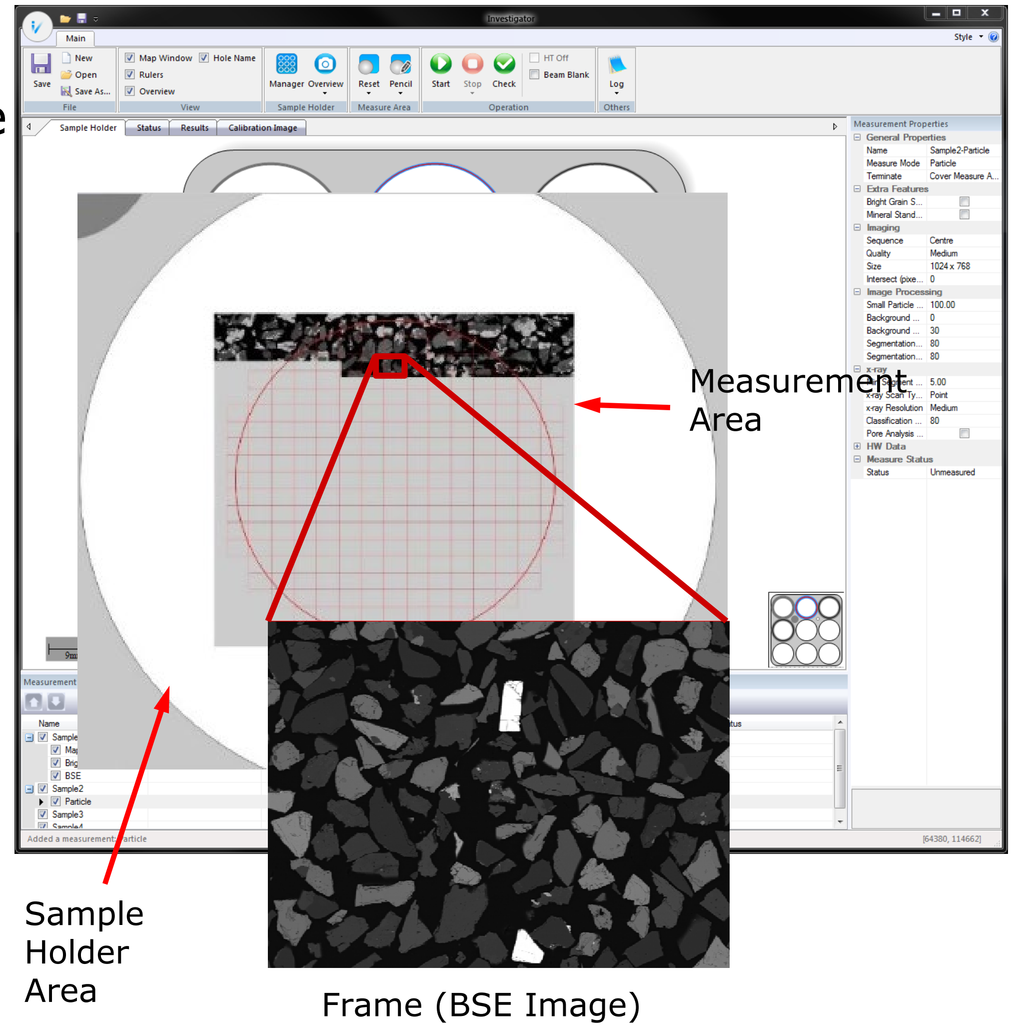


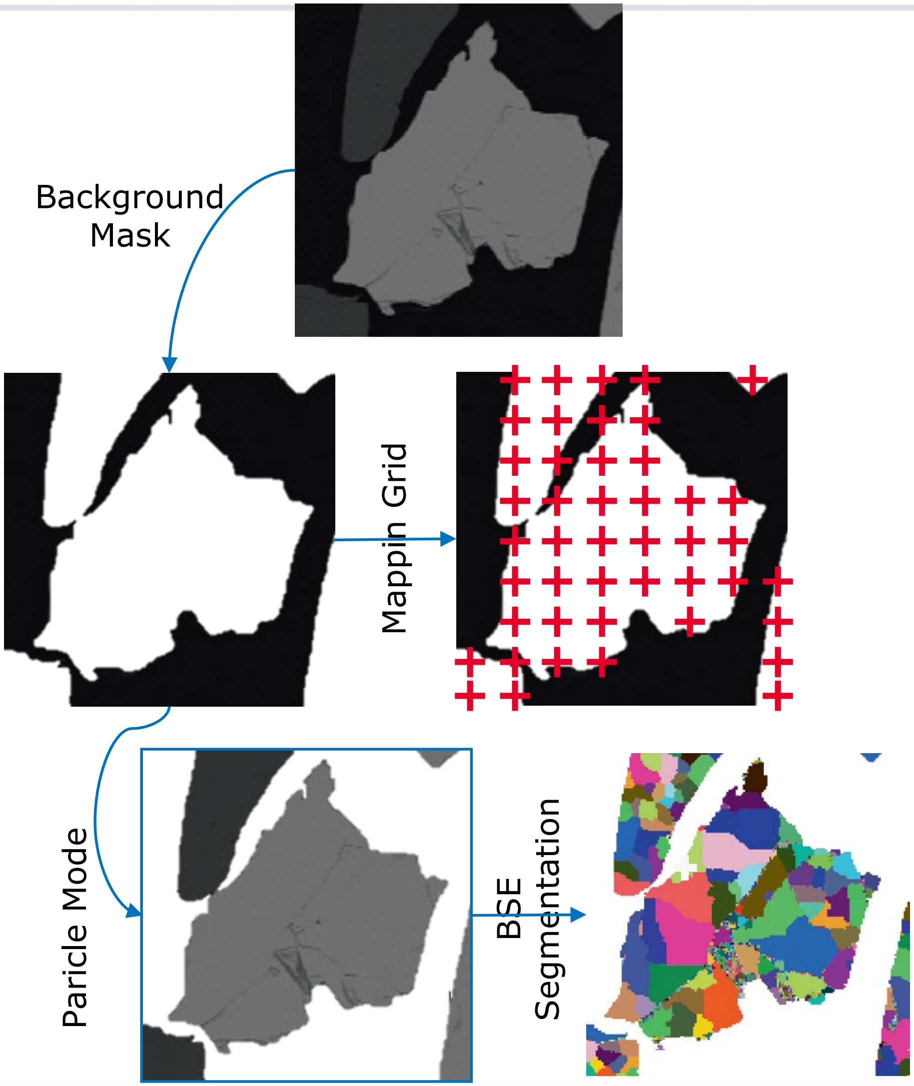
AMICS for QUANTAX EDS
How does AMICS work

AMICS will allow setting background thresholds and will create a mask for the particles.

When combined with mapping, particle can be mapped, e.g. with a step size of 5 µm.

In particle mode, computer vision techniques allow grey level variation and segment size to be adjusted to segment particles. Each segment (above a set size) will be analyzed by a single X-ray spot

X-ray spectra acquisition is via the QUANTAX system.



AMICS for QUANTAX EDS
How does AMICS work

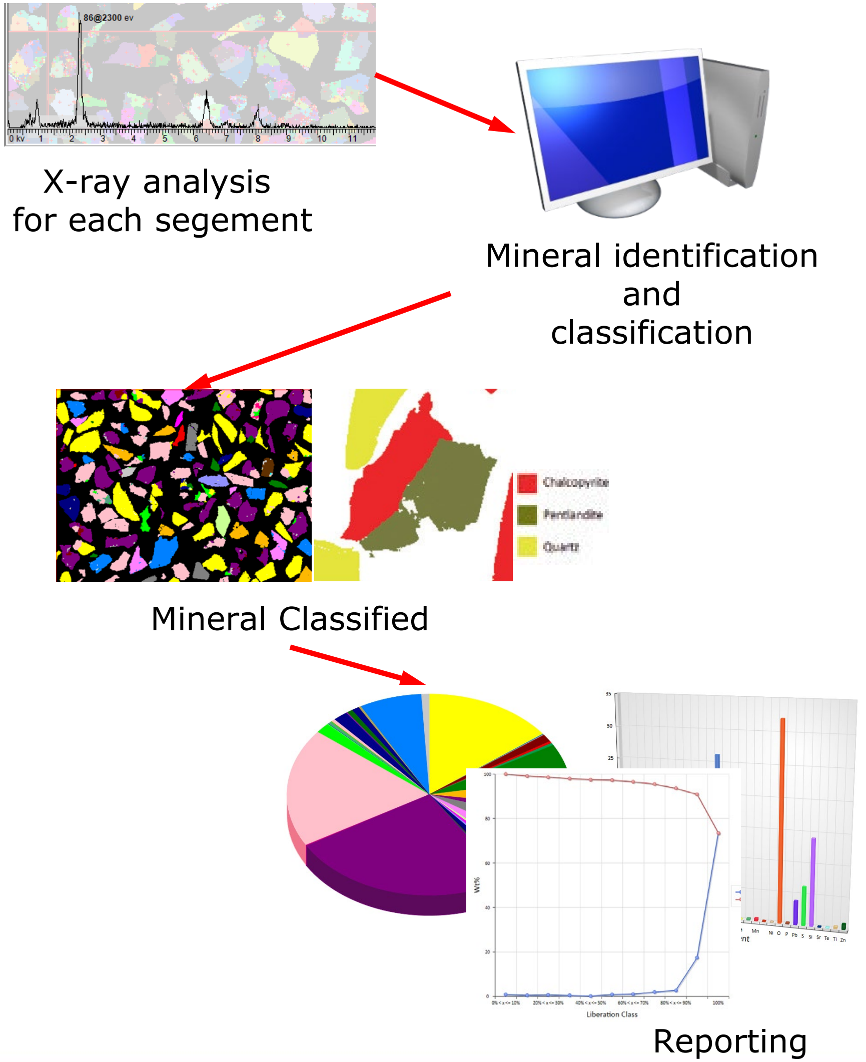
Mineral identification:

For each point or segment a spectrum is acquired, analyzed and classified live – using the specified species list.

A live distribution of minerals can be seen during measurement.

All the data is saved progressively and modal data by area and wt % and segments measured is updated after each completed frame.

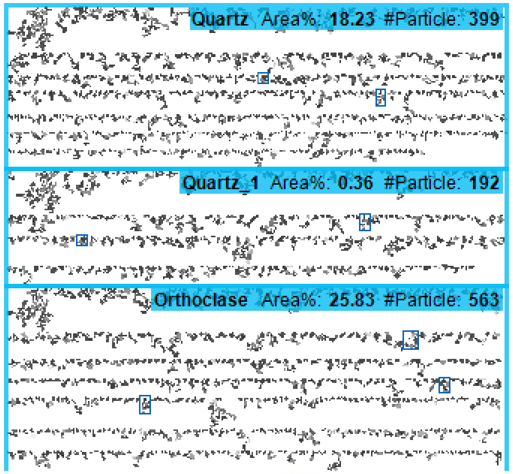
Processing and reclassification of data is done post measurement in order to create images, charts and tables for reports.



AMICS for QUANTAX EDS
Advantage of AMICS

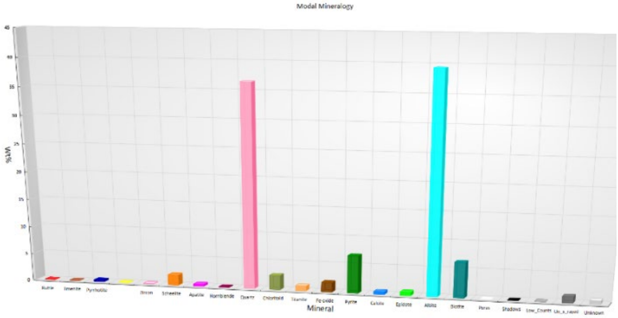
One-click built-in calculations

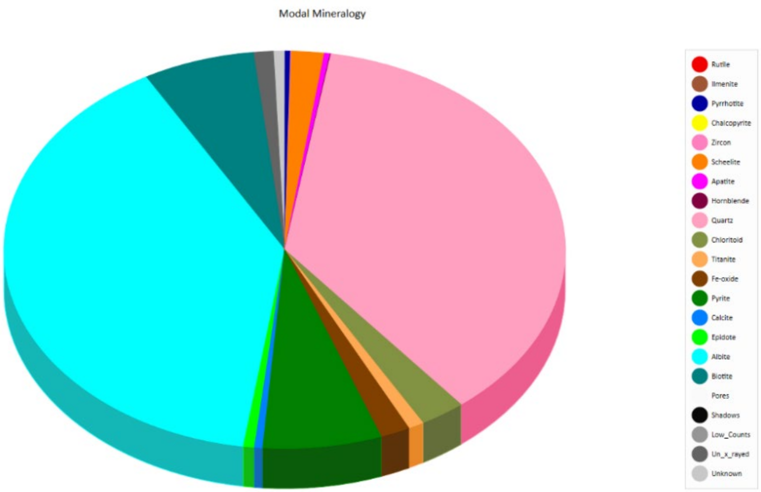
- Can view as data table
- Or chart
- Or image grid



Modal Mineralogy

	Name	Wt%	Area%	Area (µ2)	Particle Num...	Grain Number	Relative Error
	<all>	<all>	<all>	<all>	<all>	<all>	<all>
1	Quartz	6.20	7.32	784575.10	1	931	1.87
2	Andesine	21.93	25.37	2721339.45	1	1067	1.87
3	Ferrohornble...	48.03	46.09	4942962.19	1	666	1.87
4	Biotite	9.97	9.74	1044473.43	1	2051	1.87
5	Ilmenite	7.24	4.74	508612.79	1	467	1.87
6	Apatite	4.58	4.44	476034.98	1	205	1.87
7	Epidote	0.66	0.60	63998.02	1	403	1.87
8	Chlorite	0.39	0.38	40686.61	1	547	1.87
9	Other	0.61	0.58	62555.11	1	712	1.87
10	Unknown	0.39	0.74	79395.40	8	2864	0.00





QUANTAX
Summary

- With the Bruker QUANTAX System: Samples can be investigated using different techniques at the same SEM, with the same software without changing the sample
- Combining WDS, EBSD and Micro-XRF with EDS - overcomes the physical limit for EDS
- Enhanced atomic library from Bruker allows deconvolution and quantification of strong overlapping elements
- FlatQUAD detector allows measurements on beam sensitive samples, with topography, as well as high speed mappings for big areas and nanoparticles
- AMICS - Minerals with similar BSE intensities can be effectively distinguished with AMICS's unique advanced segmentation
- AMICS - A number of predefined calculations are available, which can be displayed in tabular, image grid or chart formats

Innovation with Integrity

Macro-Micro-Nanoscale SEM/EDS of Earth and Planetary Materials

T. Salge, J. Spratt, S. Russell, R. Neumann, T. Mohr-Westheide, A. Greshake, W. U. Reimold, L. Ferrière

museum für naturkunde berlin

CETEM CENTRO DE TECNOLOGIA MINERAL

UnB

naturhistorisches museum wien nhm

NATURAL HISTORY MUSEUM

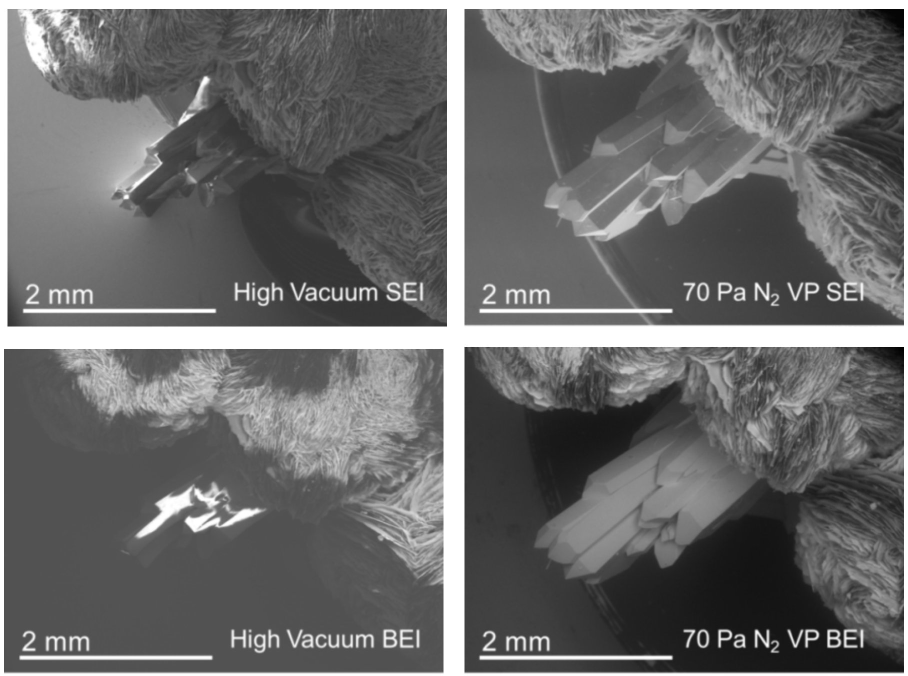
SEM and EDS
Introduction

- SEM imaging
  - ➔ Secondary and back-scattered electrons
  - ➔ Variable pressure mode
- EDS microanalysis
  - ➔ Standards-based quantification
  - ➔ Advanced hyperspectral imaging techniques
- Novel analytical approaches
  - ➔ Macroscale analysis using automatic stage control
  - ➔ Sub-micron spatial resolution by low energy analysis
  - ➔ Non-destructive analysis using ultra-low beam currents

Sample preparation
High vaccuum and VP mode SEM analysis

- To inhibit charging, non-conductive samples are usually coated.
- Gold or palladium improves the SE signal for topographic imaging.
- Carbon coating is preferred for X-ray analysis.

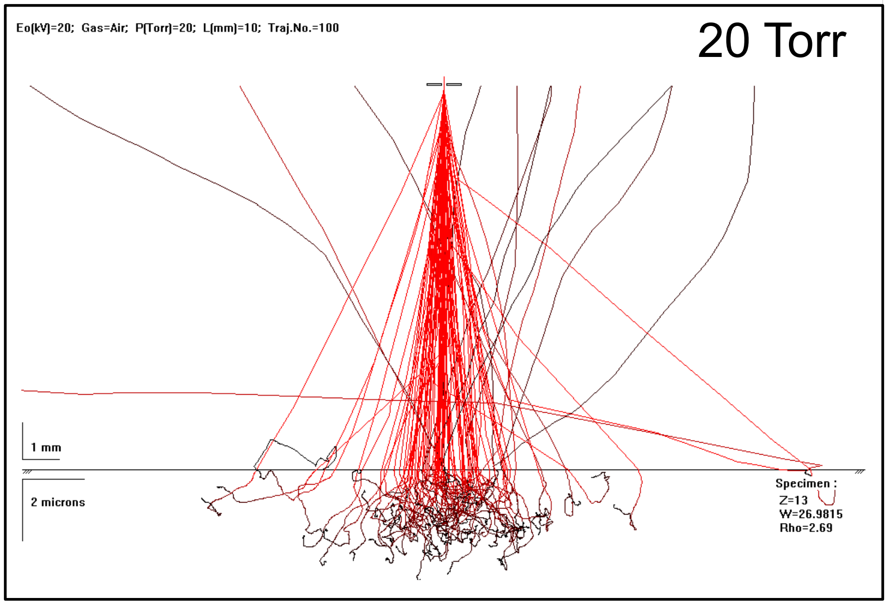
High vacuum 70 Pa



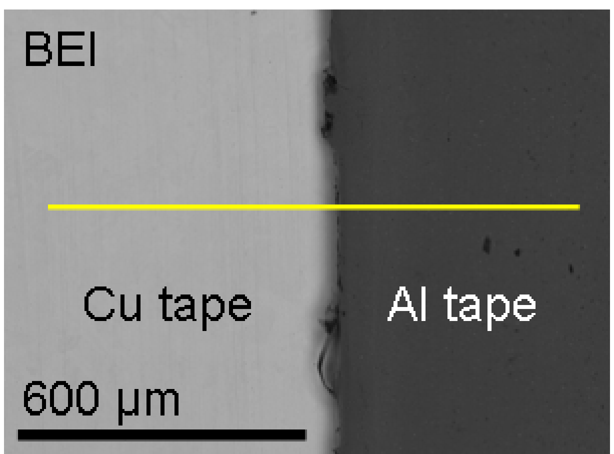
- Uncoated samples can be analysed at ~10 Pa to 400 Pa.
- Collisions of the electron beam with gas molecules create positive ions.
- These migrate to negatively charged areas of the specimen and neutralise the surface charge.

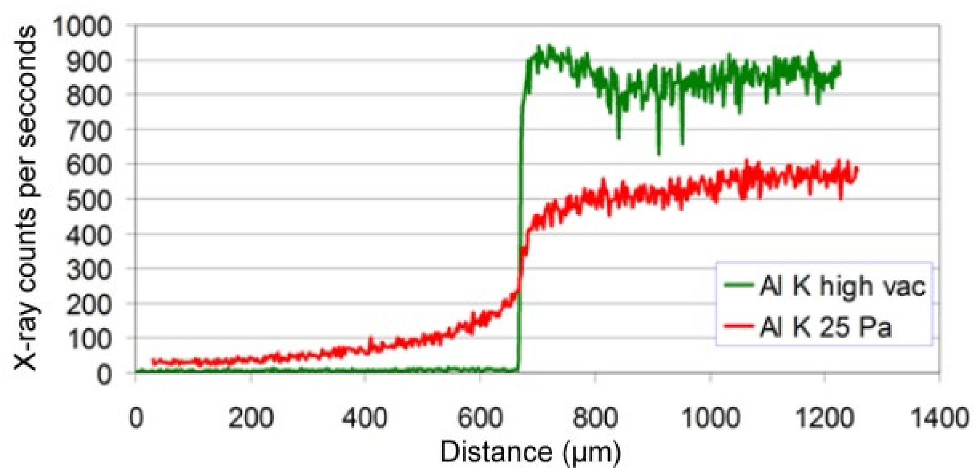
Salge et al. (2018)

VP-SEM Micronalysis
Beam skirting



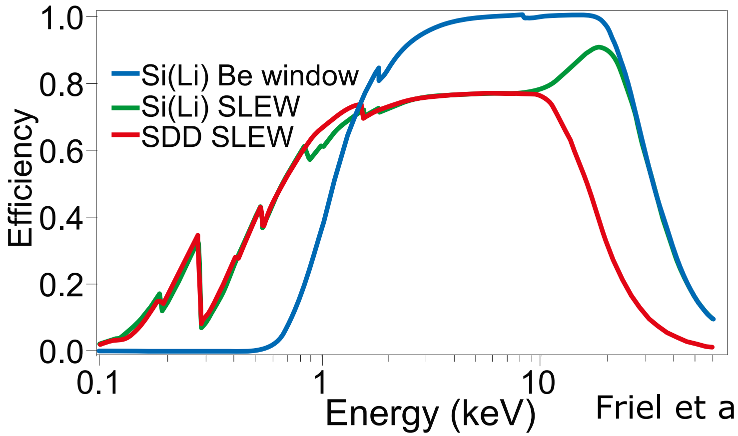
- Remote generation of X-rays by electrons scattered out of the focussed beam.
- Reduces the spatial resolution for X-ray microanalysis.
- Contributions from the environmental gas.





Salge et al. (2018)

EDS



Friel et al. (2017)

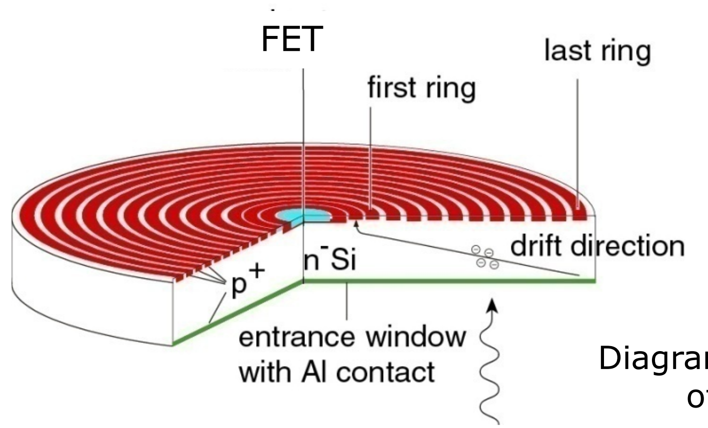
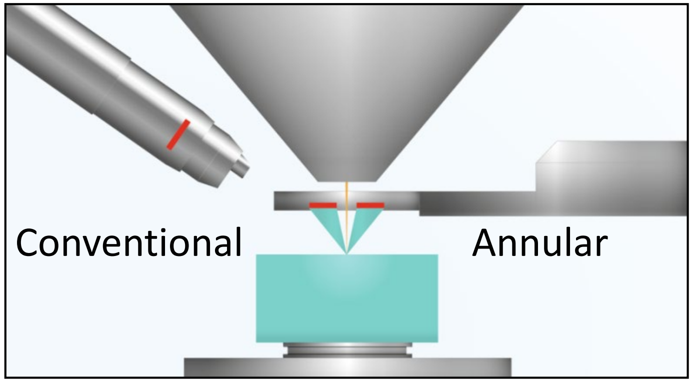
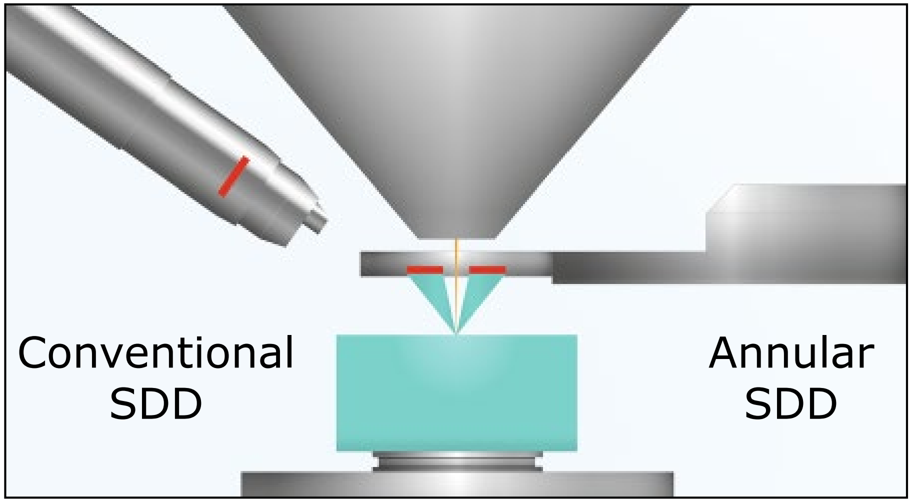


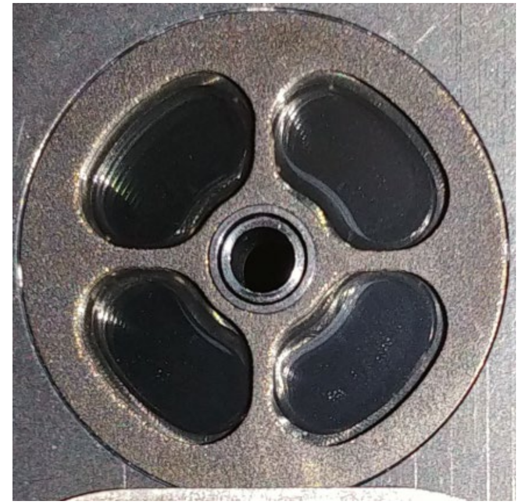
Diagram courtesy of PNSensor

- Polymer window
  - ➔ Analysis of low energy X-ray lines
- SDD
  - ➔ No liquid N_2 cooling
  - ➔ High process count rates (up to 600 kcps)
- Conventional geometry
  - ➔ Shadowing effects
  - ➔ Multi detector option
- Annular SDD
  - ➔ No shadowing effects
  - ➔ 10-100 times higher sensitivity

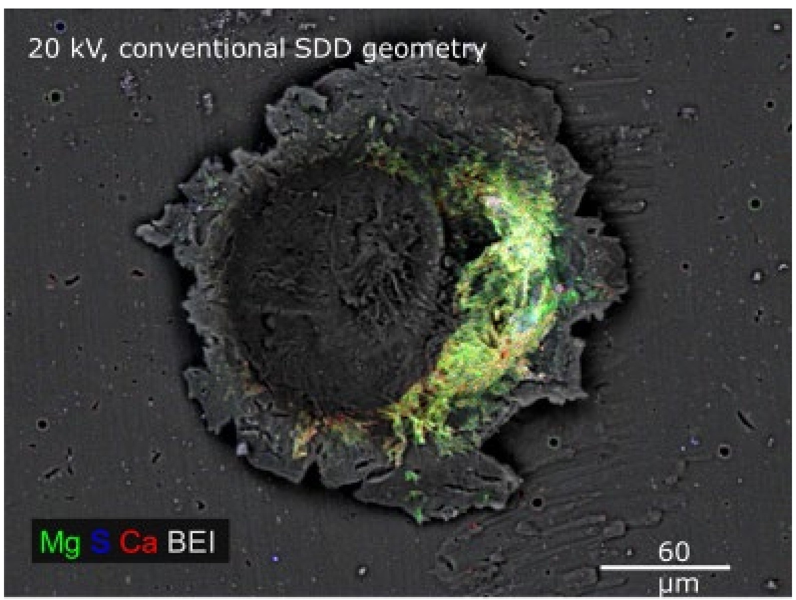


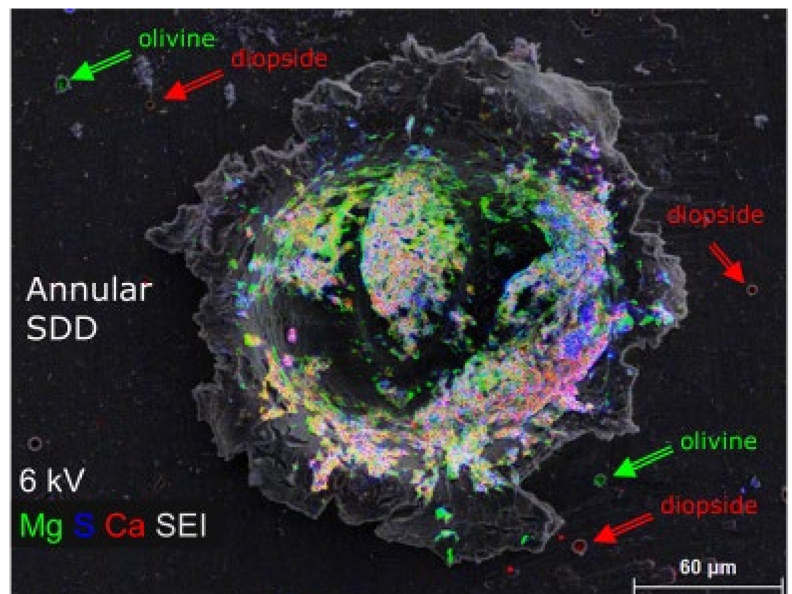
Annular SDD – comparison of crater area coverage





- Annular design, ~1sr, 4 detection units, 2.4 Mcps OCR

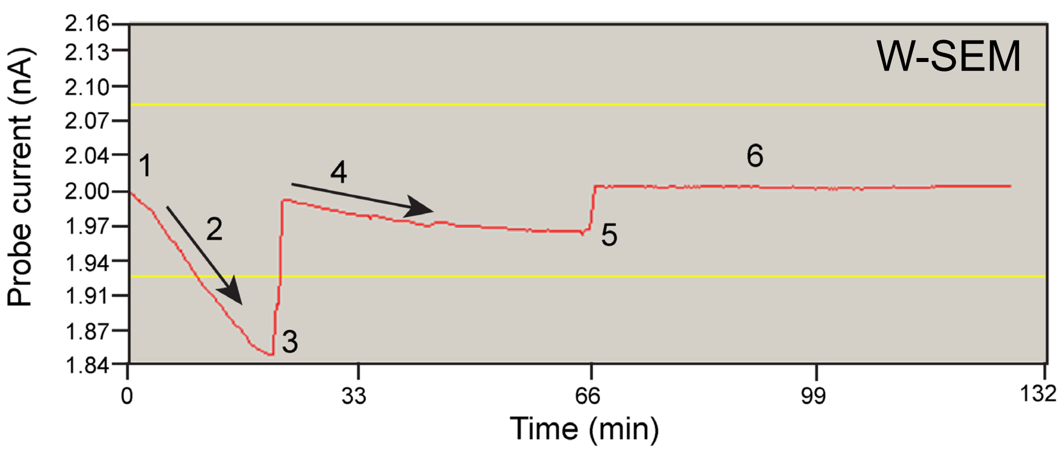




Kearsley et al. (2013), Terborg et al. (2017)

Standards-based Quantification
Requirements

- Samples: polished, perpendicular aligned, carbon coated
- References: similar matrix, high element concentration
- Same operating conditions (HV, beam current, WD)
- Beam current must be monitored (SEM has no current regulator)
- Vacuum pump for the electron gun improves beam stability and operating time (1000’s of hours).



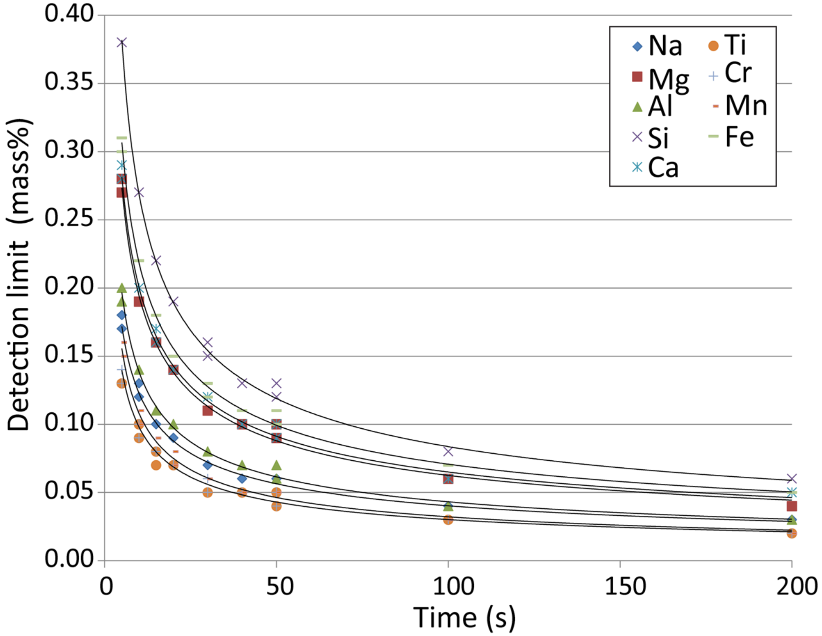
1 Filament and accelerating voltage on
2 Beam current quickly drops
3 Beam alignment using shift and tilt coils
4 Beam current slowly drops
5 Beam alignment using shift and tilt coils
6 Beam stabilises

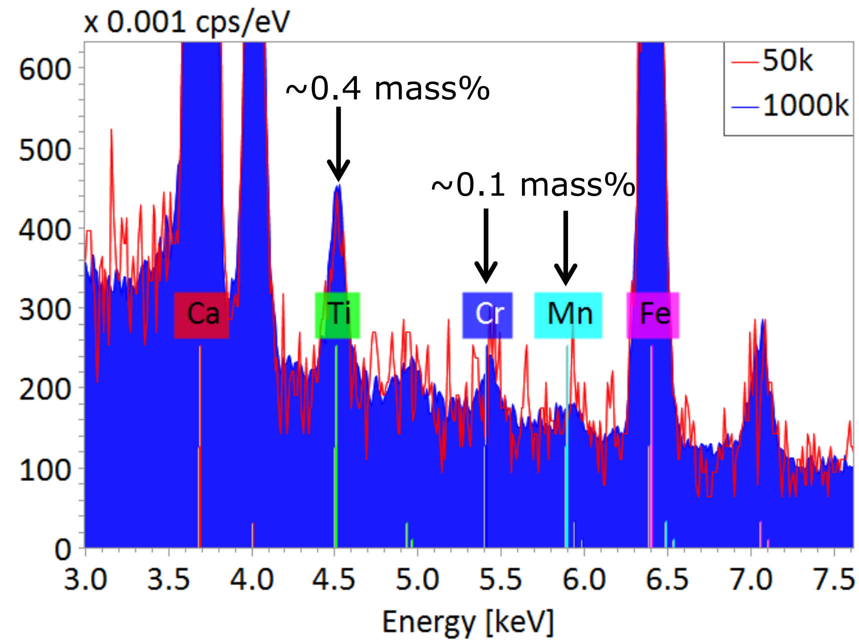
Salge et al. (2018)

Standards-based Quantification
Limit of Detection (LOD)

- LOD depends on the counts in the peak above the background.
- Typical LOD EDS ~0.1 mass% (WDS: ~0.02 mass%)

Reference material NMNH 122142 Augite, Kakanui, New Zealand
20 kV, 8 kcps output count rate





Standards-based Quantification
Impulse statistics

NMNH 122142 Augite, Kakanui, NZ, 20 kV, ~8 kcps, SDD, W-SEM

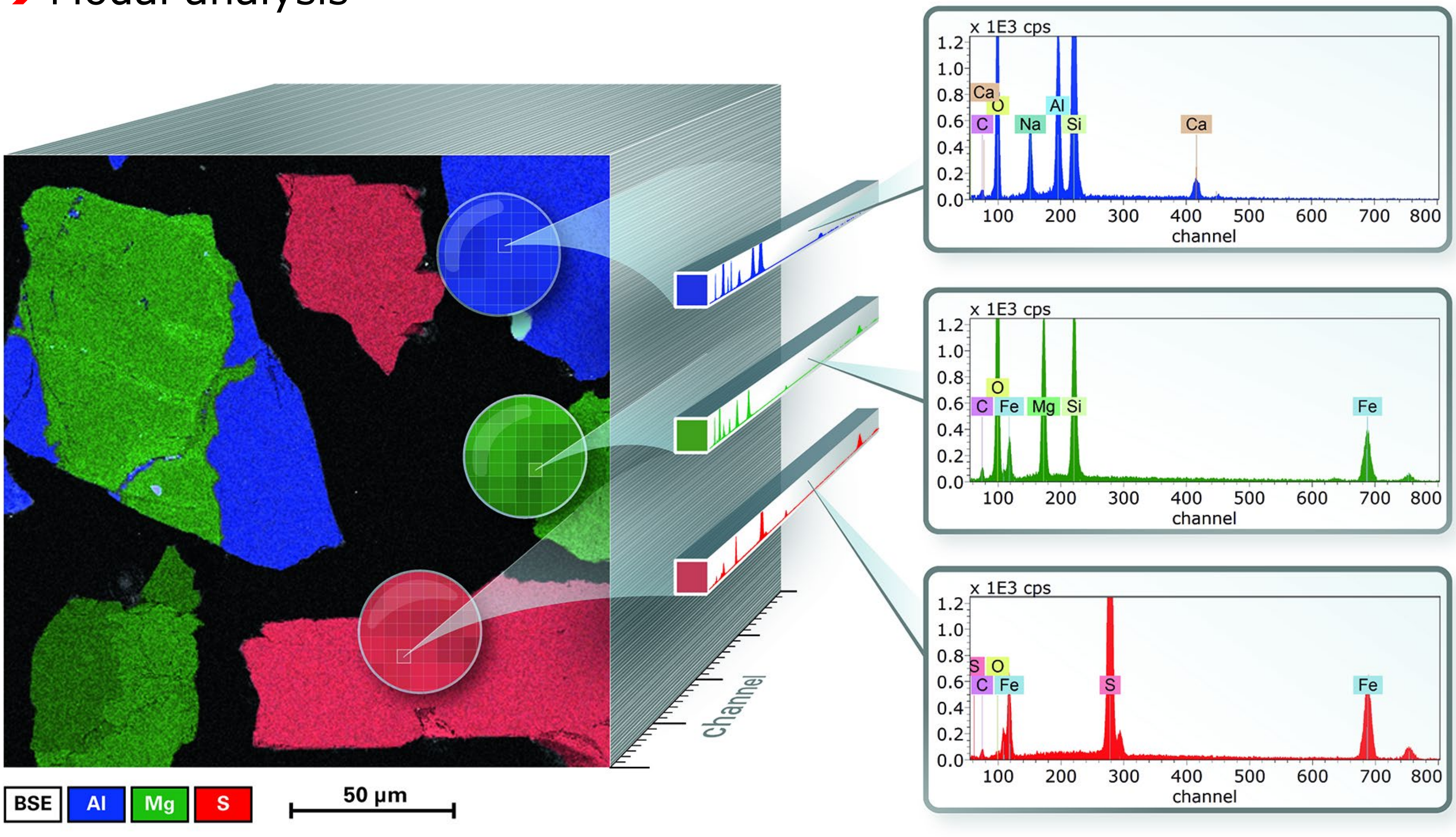
	n	Na	Mg	Al	Si	Ca	Ti	Cr	Mn	Fe	O	Total
WD-EPMA	21	1.01	9.88	4.17	23.7	11.5	0.51	0.11	0.11	4.91	44.0	99.9
s (± mass%)		0.02	0.07	0.07	0.06	0.07	0.01	0.01	0.01	0.03	0.11	
EDS 50 k	12	0.92	9.93	4.29	23.5	11.8	0.58	(0.17)	(0.12)	4.89	44.1	100.3
s (± mass%)		0.10	0.20	0.14	0.30	0.29	0.10	0.07	0.09	0.29	0.41	
EDS 1000 k	12	0.89	9.89	4.21	23.5	11.6	0.50	0.17	0.09	4.99	43.8	99.6
s (± mass%)		0.04	0.04	0.03	0.07	0.05	0.02	0.02	0.03	0.04	0.15	

- Better impulse statistics results to a lower deviation of mean values and improved precision.
- EDS can achieve precise and accurate results (4 year old calibration).
- For elements with low concentration and significant peak overlaps in EDS spectra, WDS can obtain better results (better spectral resolution, superiour signal to noise ratio).

Salge et al. (2018)

Spectrum Imaging
Advanced EDS

- Improved element ID
- Modal analysis

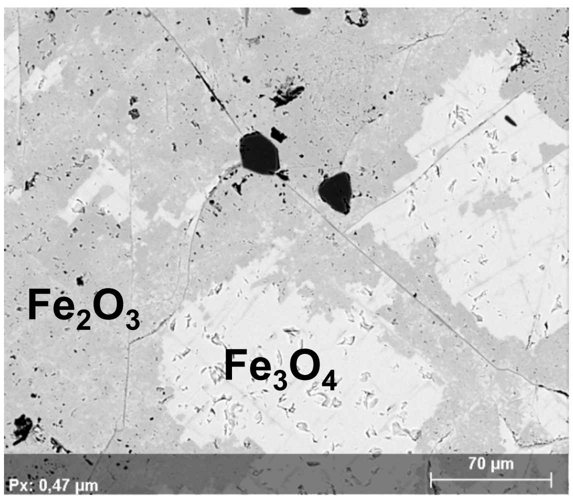


Friel et al. (2017)

Polymetallic iron ore

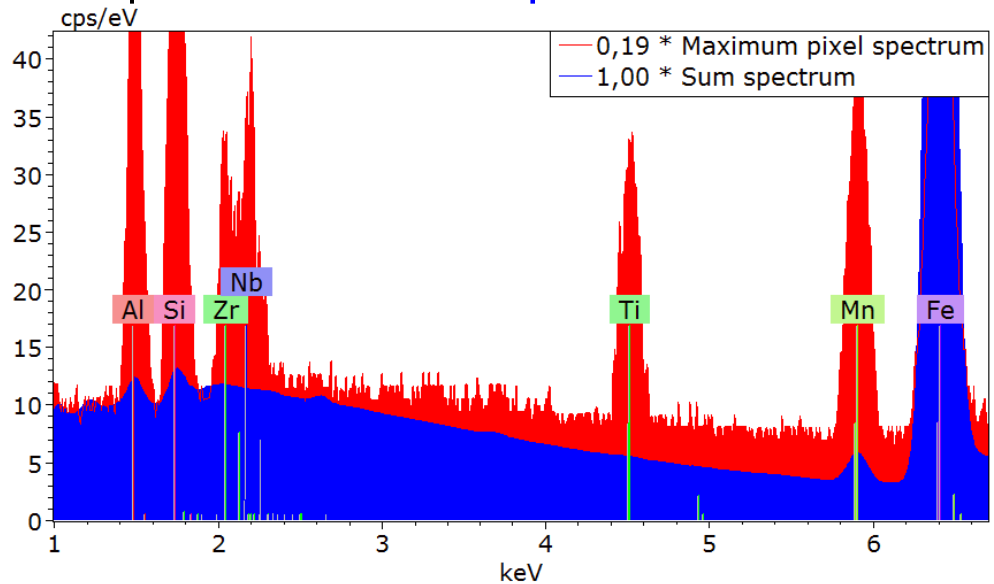
Maximum Pixel Spectrum

- Synthetic spectrum of highest count level found in each spectrum channel
- Detection of elements present in some pixel



20 kV, 220 kcps, 132 min
0.47 µm pixel size, 700x604 pixels
30 mm² SDD, FE-SEM

Comparison of sum spectrum and MaxPixSpec



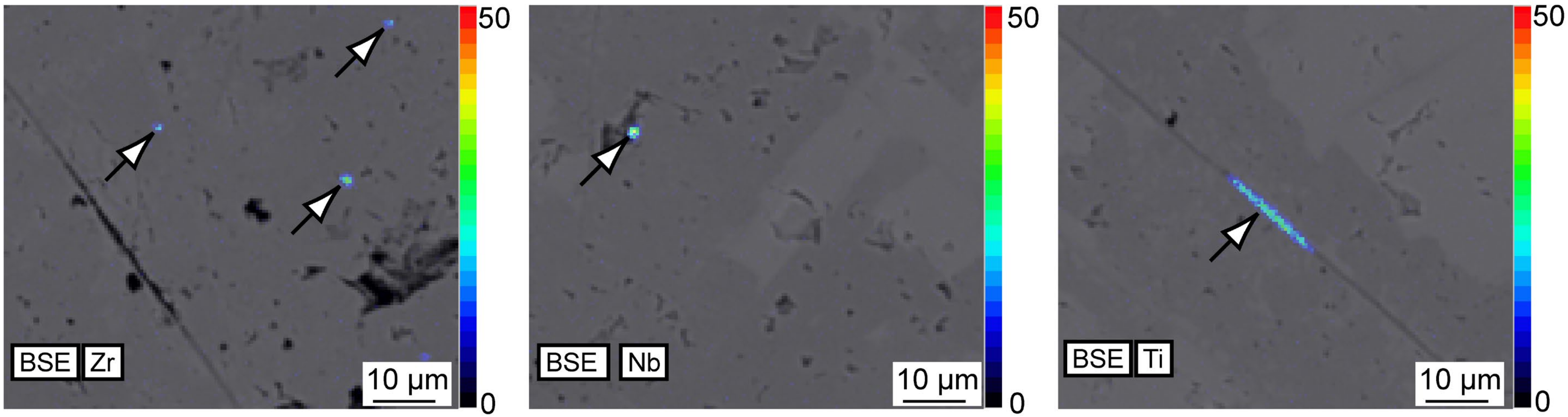
Niobium, zirconium and titanium had not been targeted by conventional wet-chemical analysis, but were identified by the Maximum Pixel Spectrum function.

Salge et al. (2013)

Polymetallic iron ore

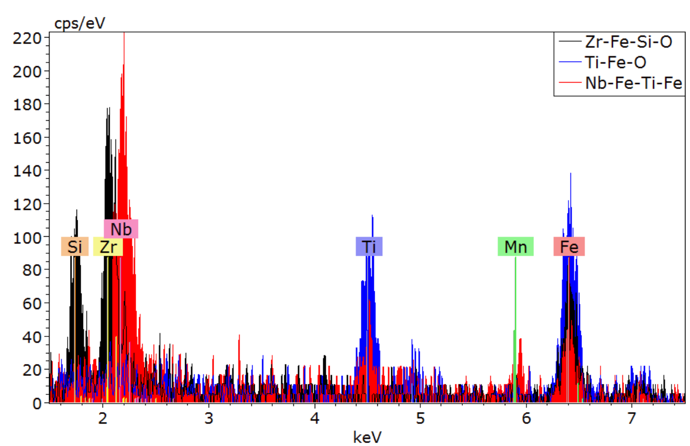
Pixel spectra and quantitative mapping

BSE overlain with quantified element map (mass%)



Zircon inclusions

Fe(Mn) columbite



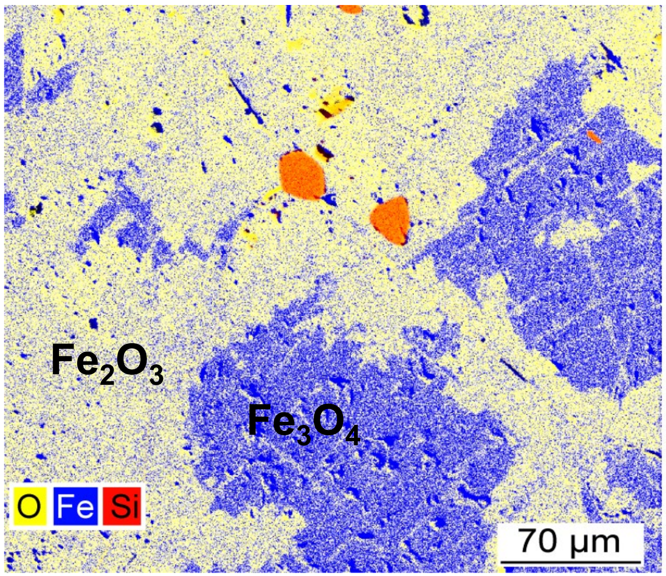
One pixel spectra (~2,000 counts in the complete spectrum)

Salge et al. (2013)

Mapping options

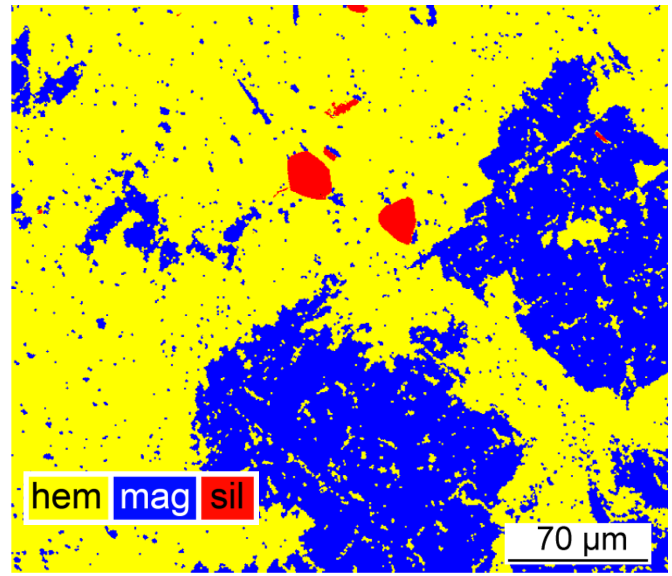
Intensity and chemical phase map

Composite intensity map



- Iron oxides can be recognized by composite intensity maps of oxygen and iron.

Chemical phase map



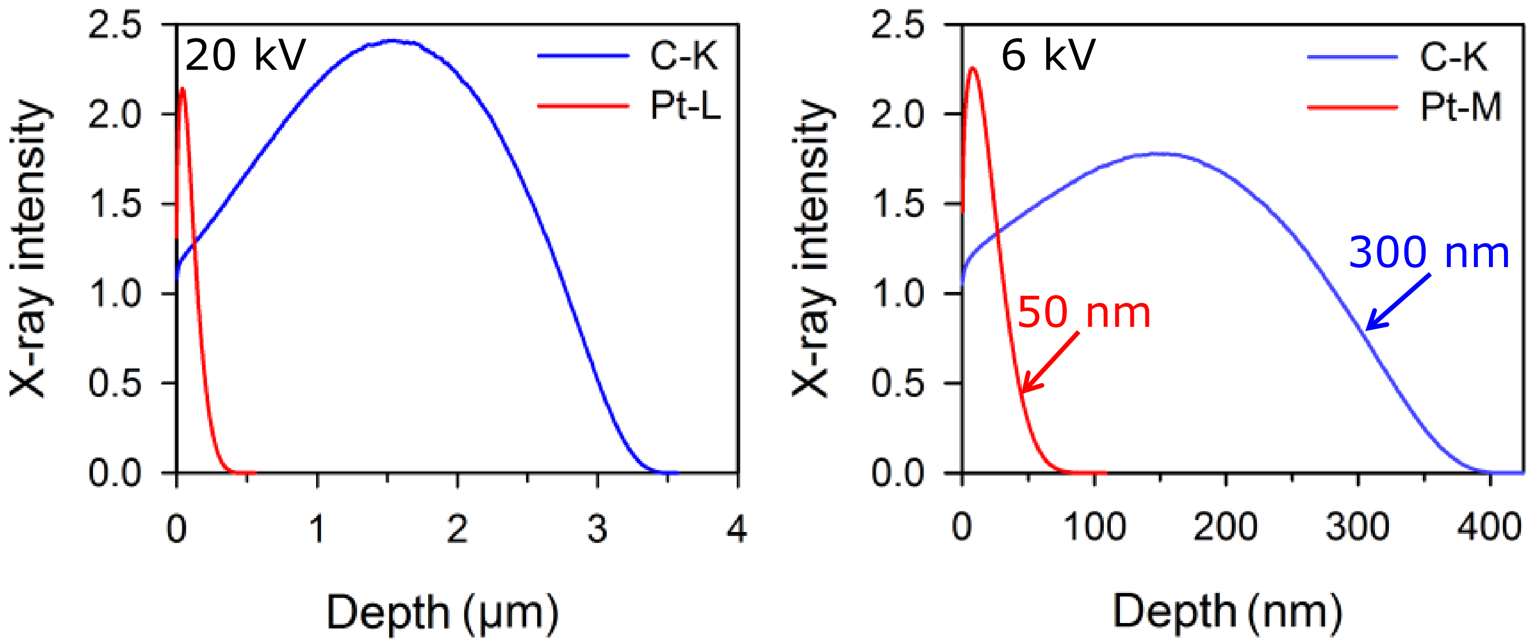
- Autophase result with consideration of oxygen, iron and silicon.

	Area [µm²]	Area [Pixels)	Area [%]
Hematite	61360	277004	65.5
Magnetite	31418	141834	33.5
Silicate	877	3960	0.9
Unassigned	<1	2	<0.1

Salge et al. (2013)

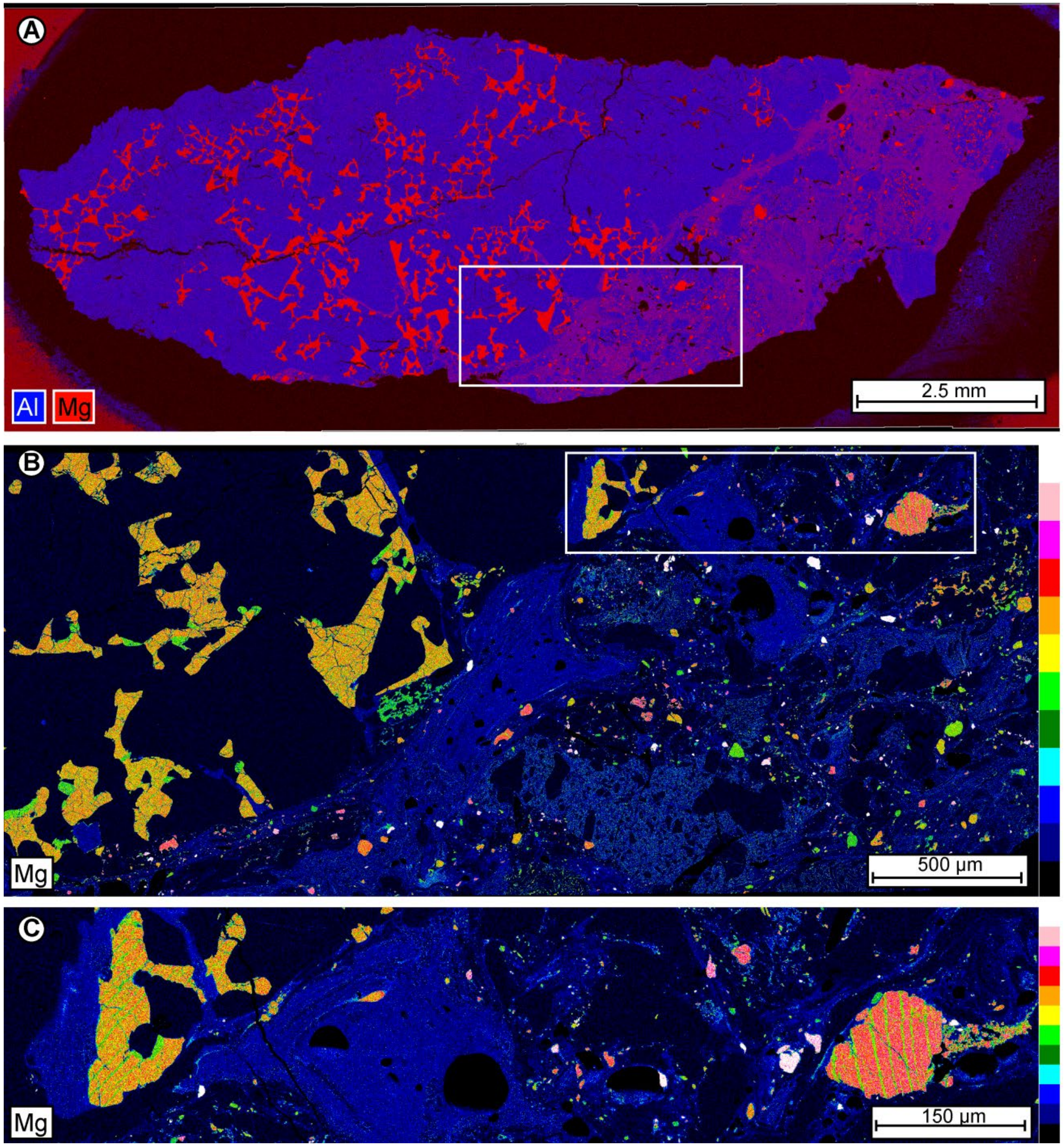
High Resolution Imaging at the Macroscale
Automatic stage control

- Acquisition of several datasets over a larger area
- Montaging of individual datasets to one file at high pixel resolution
- SEM: Giga pixels / EDS: 10’s of mega pixels
- Sub-micron spatial resolution by low energy analysis



Salge et al. (2018)

Lunar meteorite Dhofar081



20 kV, 2 µm pixel size,
22 MP, 7295 x 2986 pixels,
480 fields

10 kV, 0.5 µm pixel size,
21 MP, 6851 x 3058 pixels,
28 fields

Salge et al. (2018)

Too much

- Information?
- Measurement Time?
- Evaluation Time!

Automated feature analysis
Image Analysis and Chemical Classification

Particle sample (BSE Image)

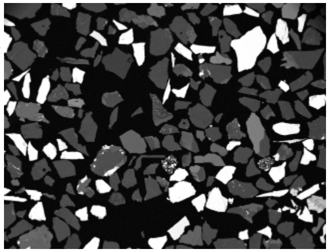
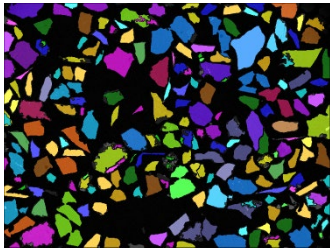
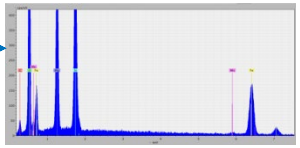


Image binarization

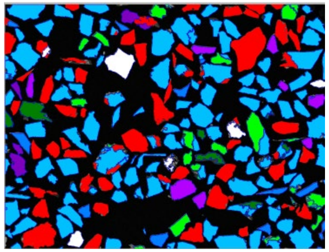
Automatic detection of particles and image analysis → particle morphology (area, length, width, aspect ratio, diameter, …)



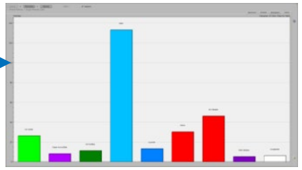
Automated collection of EDS spectra (each particle) → quantification of EDS spectra



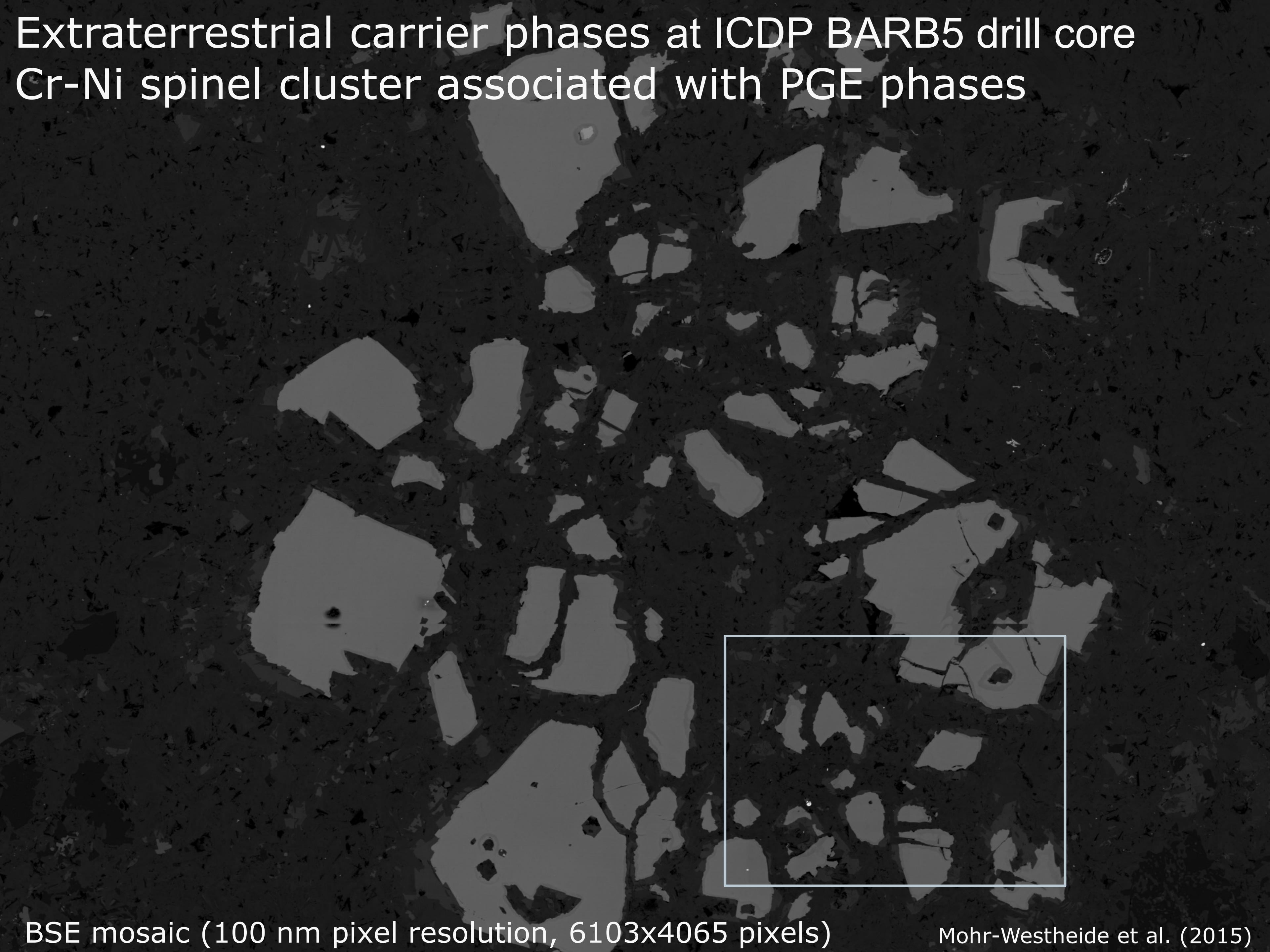
Classification of particles based on pre-defined chemical groups



Review, data analysis and reporting



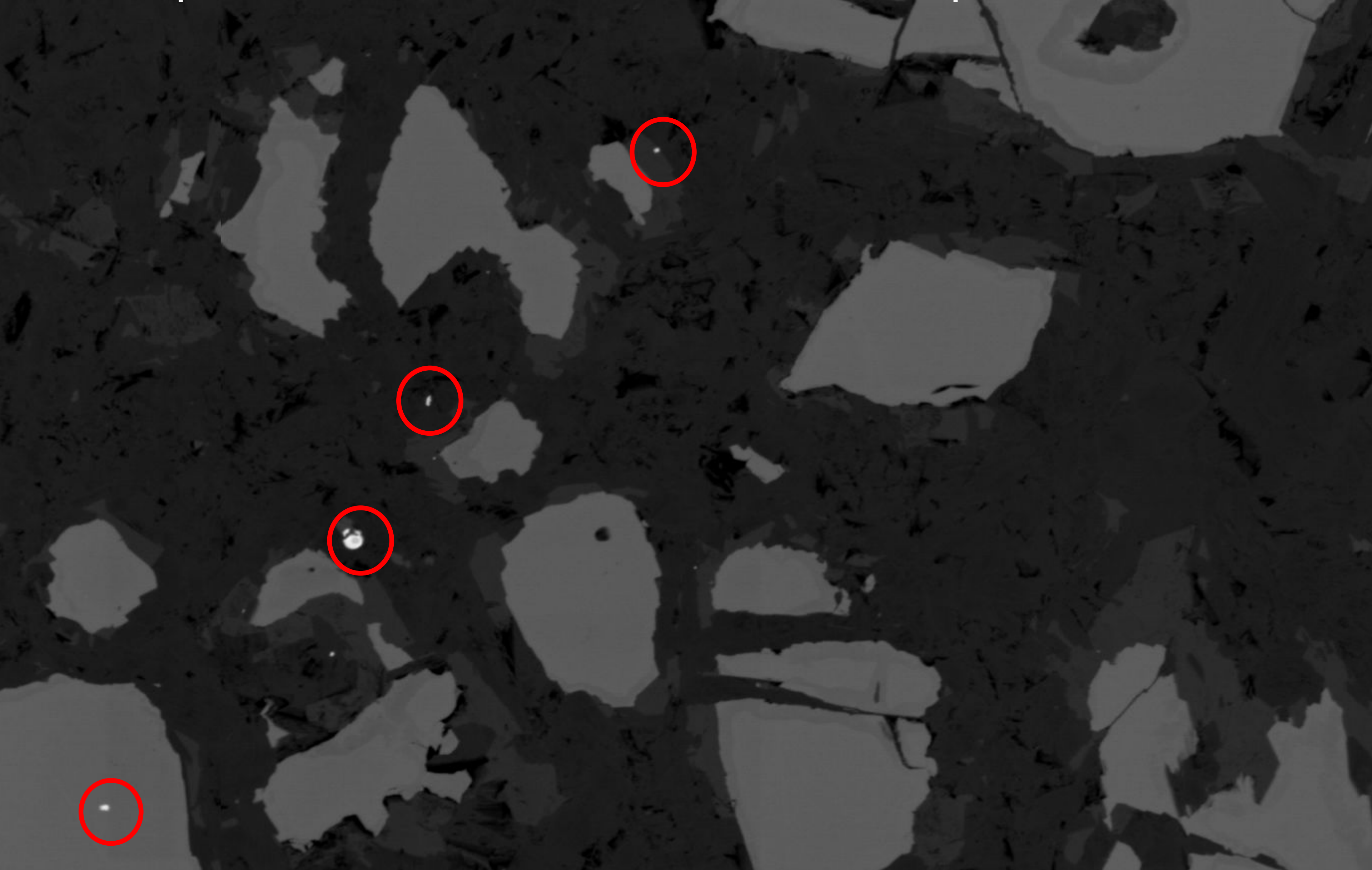
Extraterrestrial carrier phases at ICDP BARB5 drill core
Cr-Ni spinel cluster associated with PGE phases



BSE mosaic (100 nm pixel resolution, 6103x4065 pixels)

Mohr-Westheide et al. (2015)

Extraterrestrial carrier phases at ICDP BARB5 drill core
Cr-Ni spinel cluster associated with PGE phases

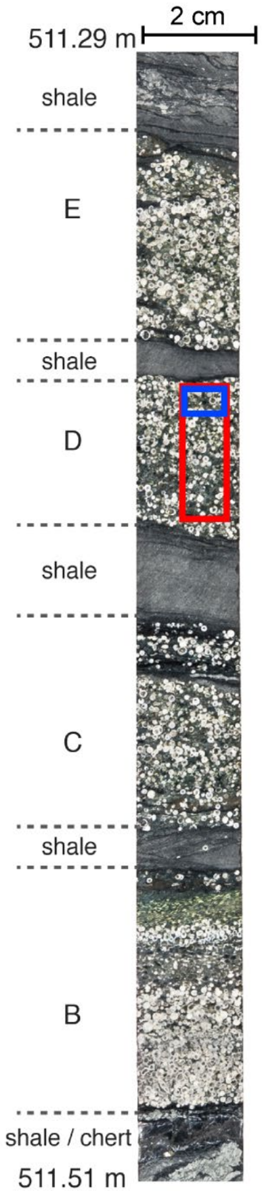


BSE mosaic (100 nm pixel resolution, 6103x4065 pixels)

Mohr-Westheide et al. (2015)

Automated feature analysis
Cr-Ni spinel and PGE phases

museum für naturkunde berlin



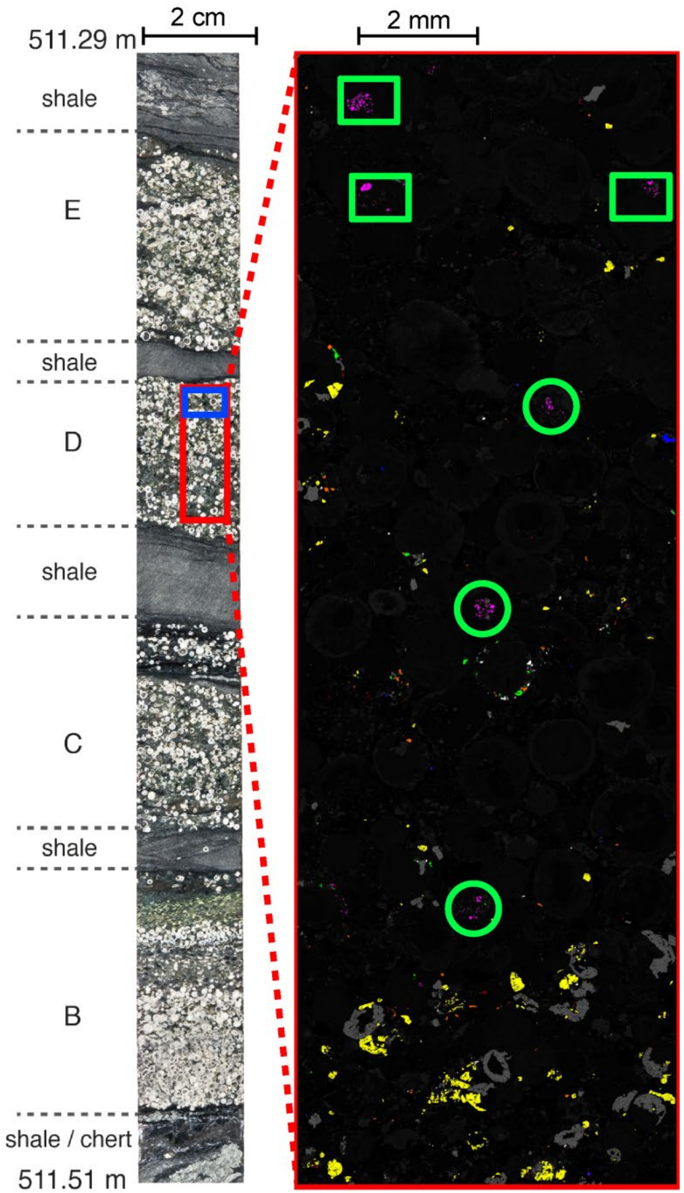
- Impact spherule layers at the ICDP BARB5 drill core.
- Ni-Cr spinel cluster are associated with sub-micron-sized PGE-rich metal alloy and PGE sulpharsenides

Analysis	Cr-Ni spinel	PGE
BSE threshold	Intermediate to bright	bright
Pixel resolution	**~2 µm**	**~100 nm**
Accepted particles	**6 µm radius**	**250 nm radius**
HV	**20 kV**	**6 kV**
Input count rate	~90 kcps	~70 kcps
Spectrum acquisition time	0.5 s	3 s
Fields	288 (400x266 pixels)	170 (3600x2397 pixels)
Count	707	38
Total time	**60 min**	**90 min**

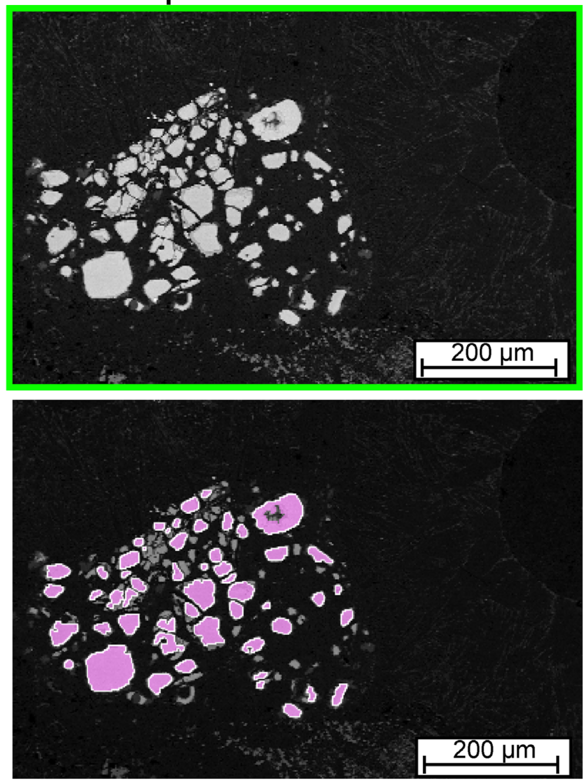
Mohr-Westheide et al. (2015)
Salge et al. (2018)

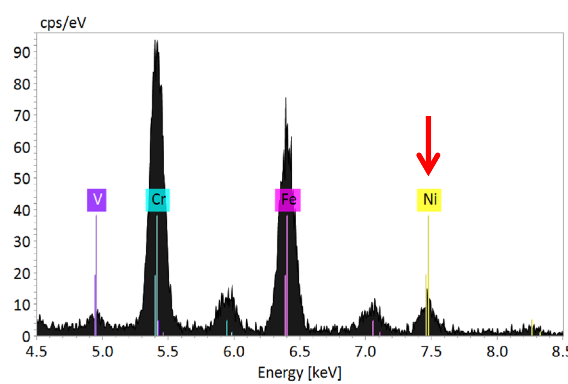
Automated feature analysis
Cr-Ni spinel and PGE phases

museum für naturkunde berlin

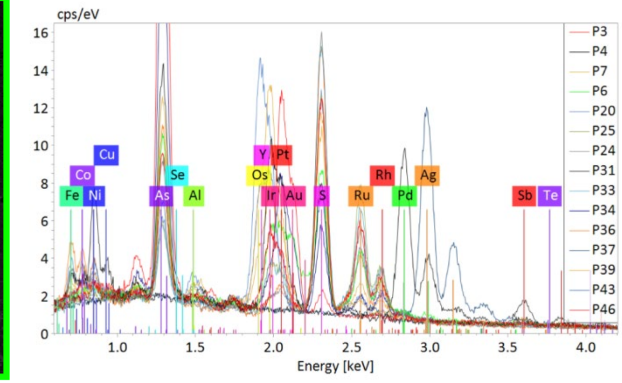


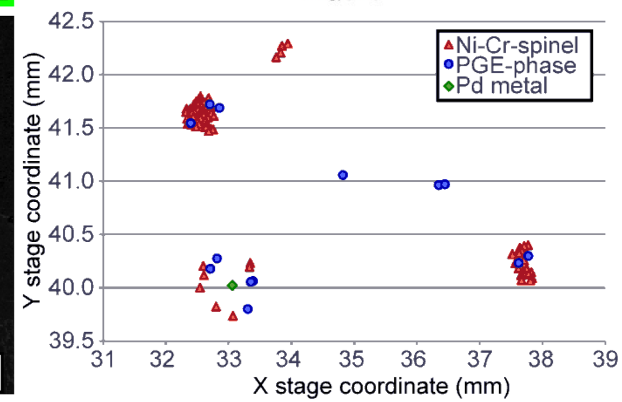
NiCr spinel cluster





PGE-phases





- 17 PGE phases
- Average Ø 0.6-1.4 µm
- Association of both phases

Mohr-Westheide et al. (2015)
Salge et al. (2018)

Low kV analysis at the sub-micron scale
Challenges

museum für naturkunde berlin

Peak overlaps

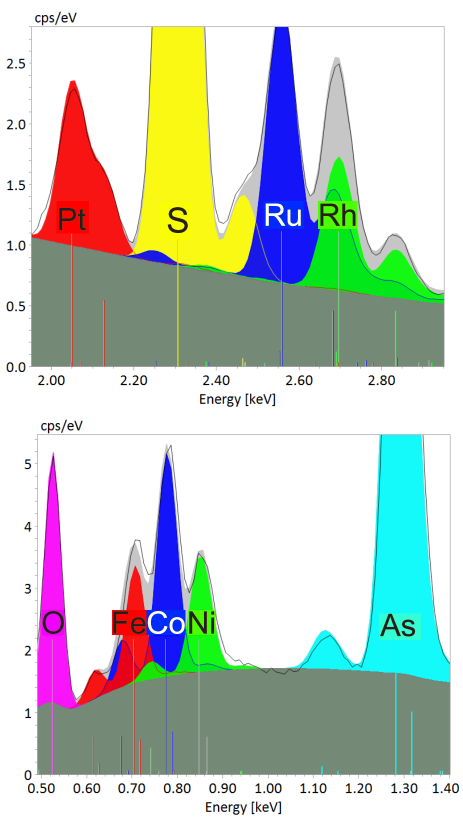
- Separation by peak deconvolution
- Net intensities

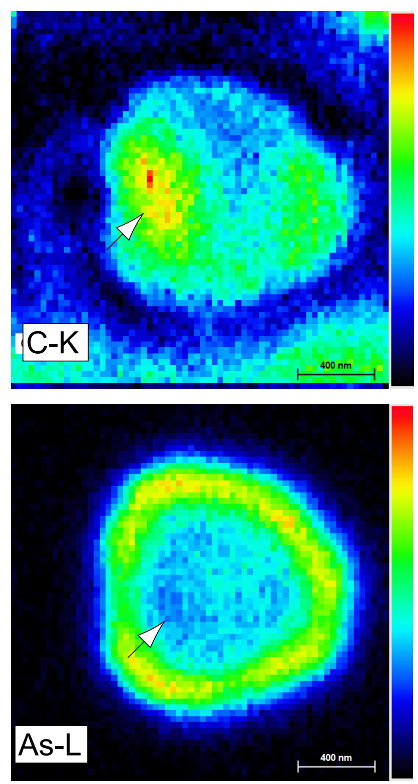
Contamination

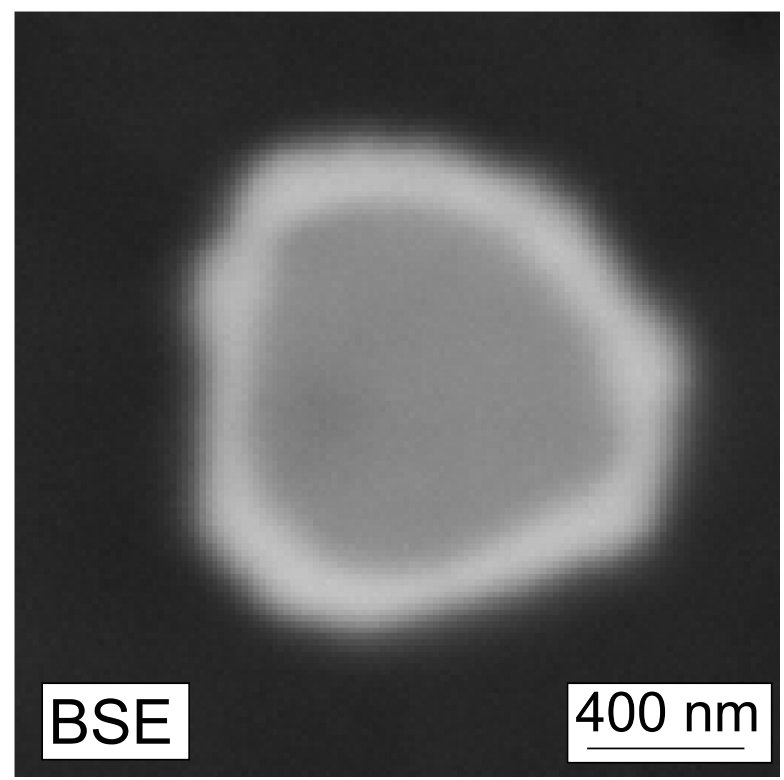
- Deposition of hydrocarbon
- Preventing by cold trap

Samples drift

- Sample heating
- Drift compensation by SEM image comparison







Salge et al. (2018)

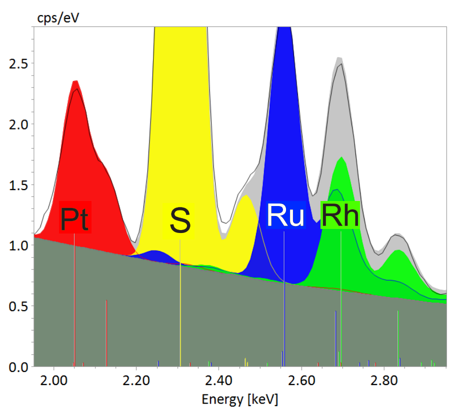
Low kV analysis at the sub-micron scale

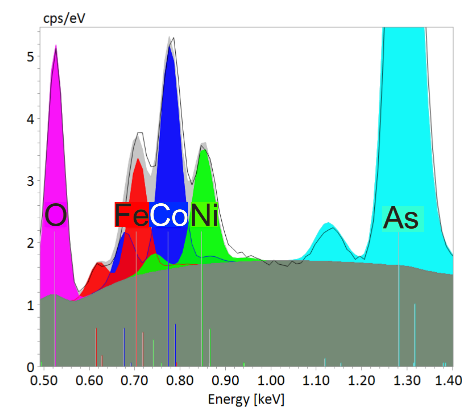
Net intensity map

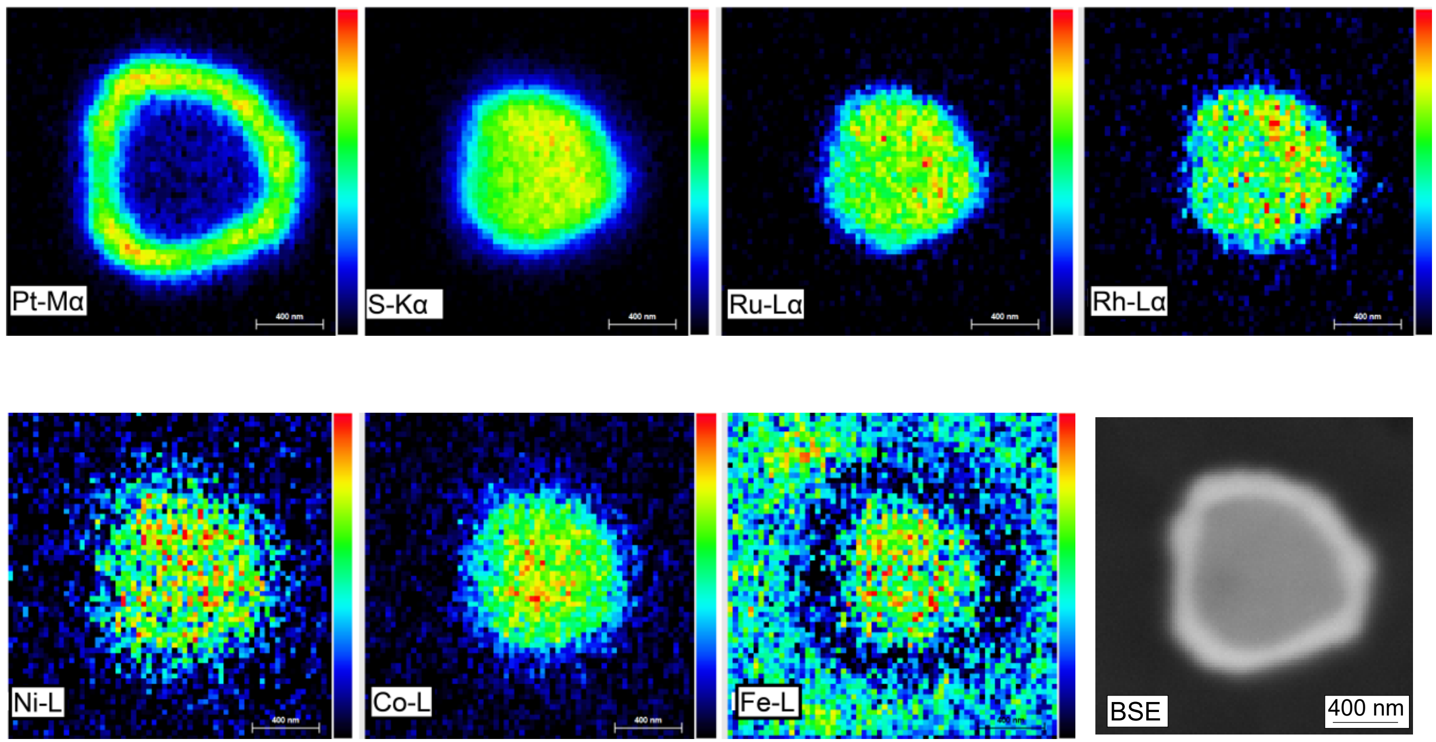
museum für naturkunde berlin

EDS deconvolution algorithms allows the analysis of overlapping peak in the low and intermediate energy range.

6 kV, 52 min, 8 kcps, 30.6 nm pixels, 10 mm^2 SDD, FE-SEM







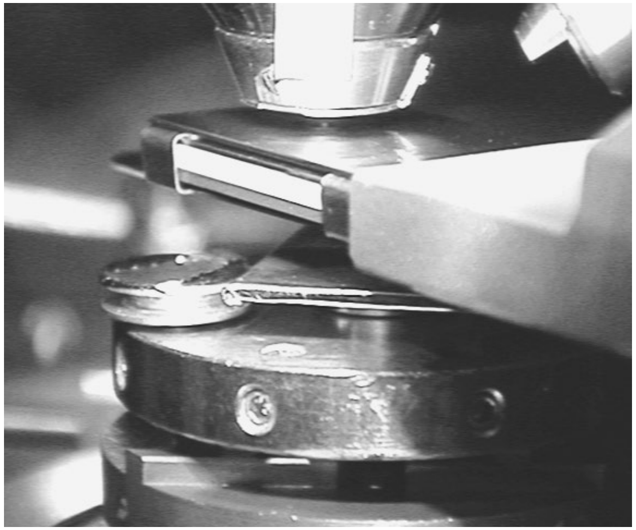
Salge et al. (2018)

Microanalyst's Dream

- Non-destructive, non-invasive analysis
  (no polishing, no coating, reducing carbon contamination)
- High sensitivity of the annnular SDD
- Ultra-low beam current (~10 pA) at high vacuum

Planetologist's Dream

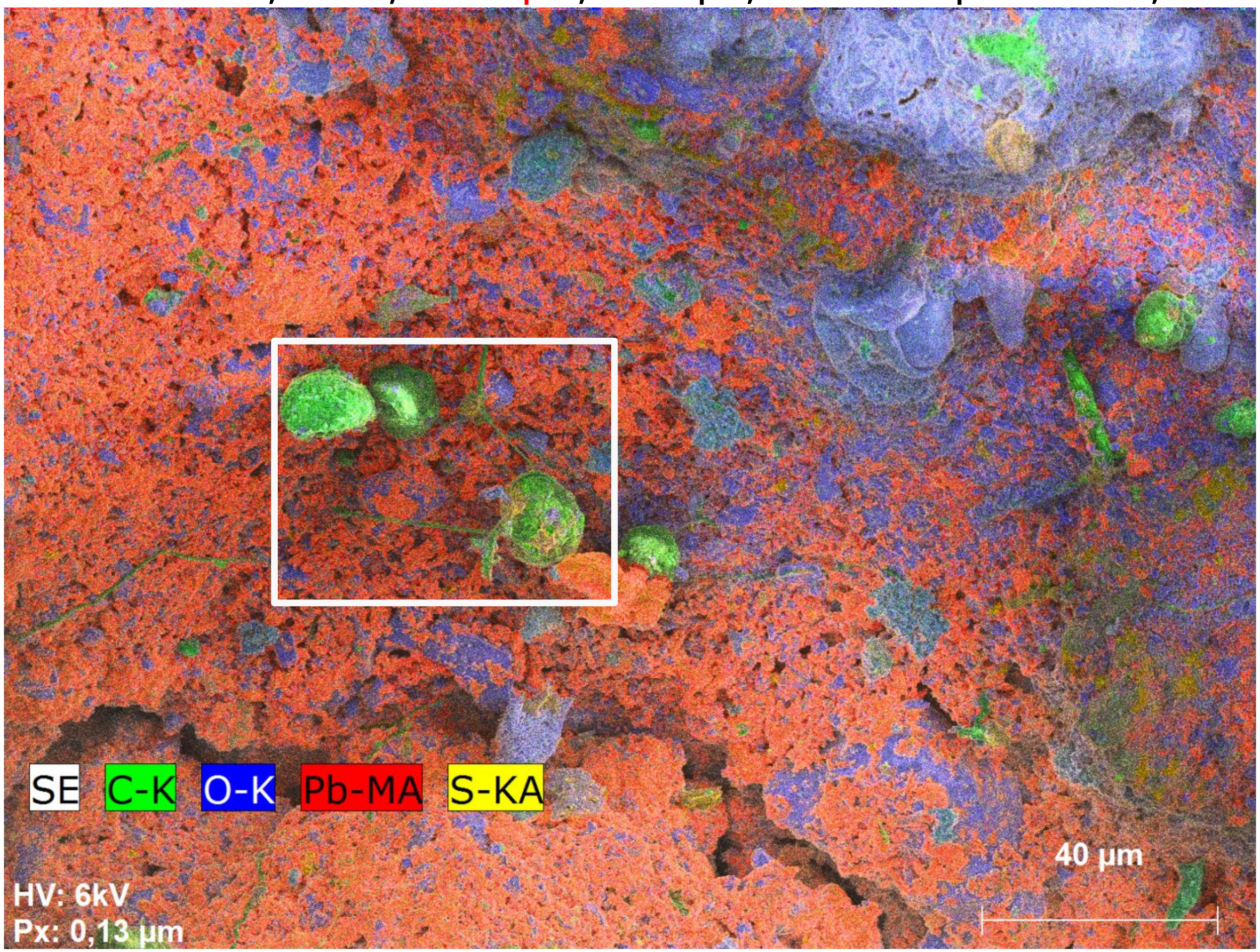
- Samples "Out of Space"



High vacuum analysis at low beam current
Historic stony meteorite (“Mocs”)

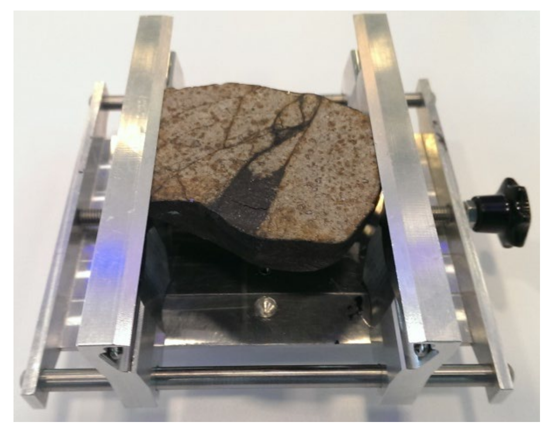
naturhistorisches museum wien

Annular SDD, 6 kV, <10 pA, 2 kcps, 130 nm pixel size, 15 h



- Fell 3rd February 1882
- Sample preparation (coating) exluded

1. Lead (old polishing) is deposited on top of silicates
2. Contamination with soot by heating with coal-fired furnaces

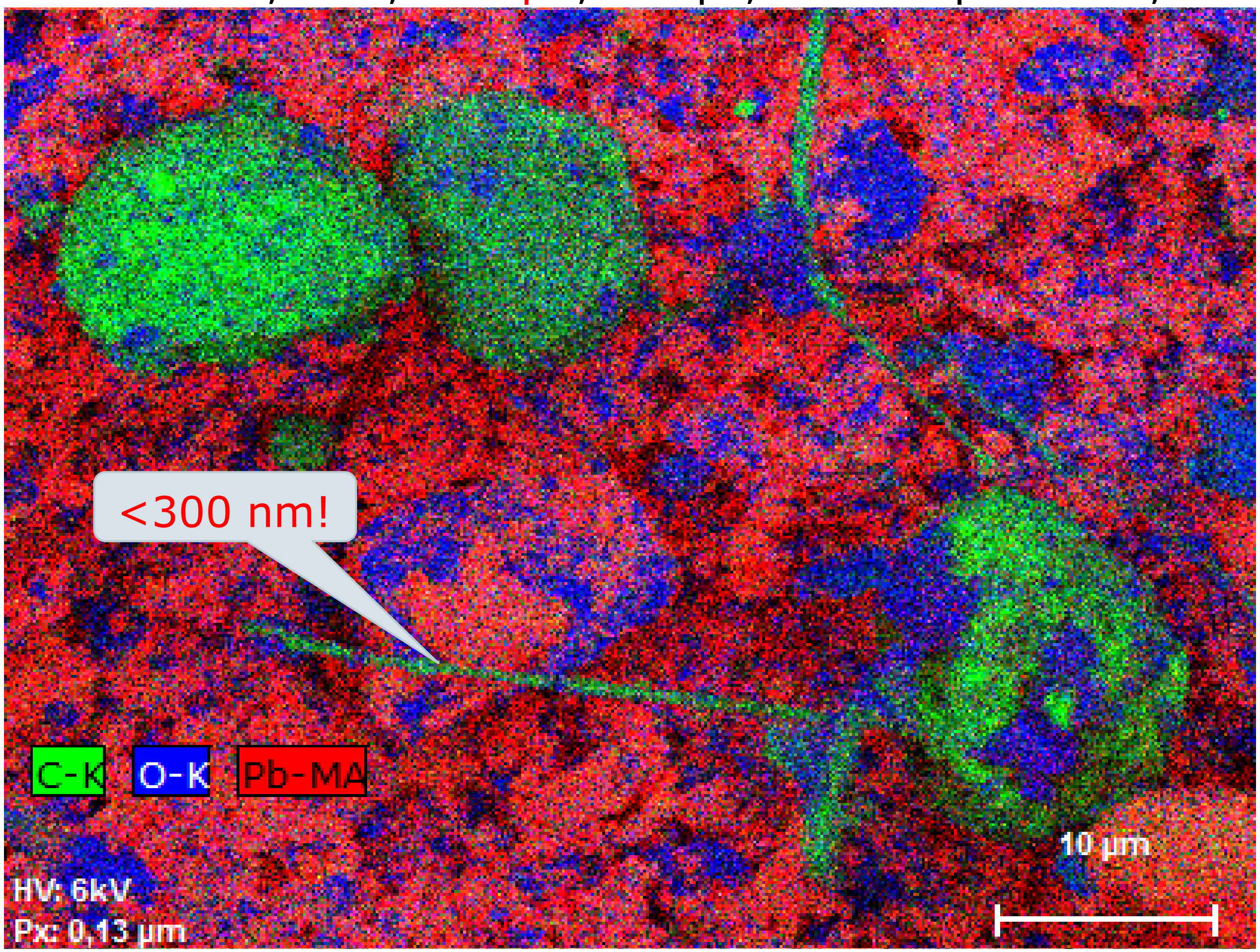


Terborg et al. (2017)

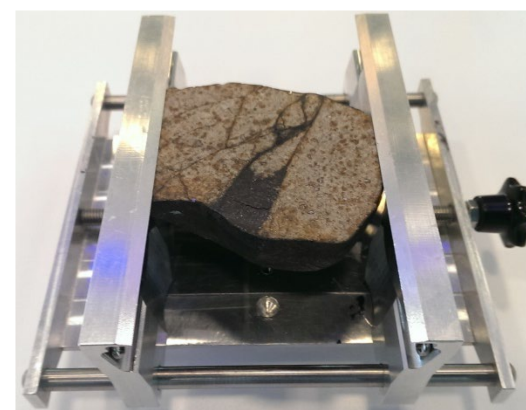
High vacuum analysis at low beam current
Historic stony meteorite ("Mocs")

naturhistorisches museum wien

Annular SDD, 6 kV, <10 pA, 2 kcps, 130 nm pixel size, 15 h



- EDS at high vacuum offers better spatial resolution compared to low vacuum



Terborg et al. (2017)

Summary
Advanced SEM-EDS

Hyperspectral imaging techniques

➔ Enhance element ID and chemical phase analysis.

Low voltage EDS

➔ Enhances the spatial resolution to the sub-micron scale.

Automatic stage control

➔ Allows to acquire datasets at the macroscale.

➔ Classification of minerals over short measurement times by feature analysis.

Annular SDD

➔ Provides high spatial resolution and high detection sensitivity without the necessity of applying a conductive coating or working in low vacuum.

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