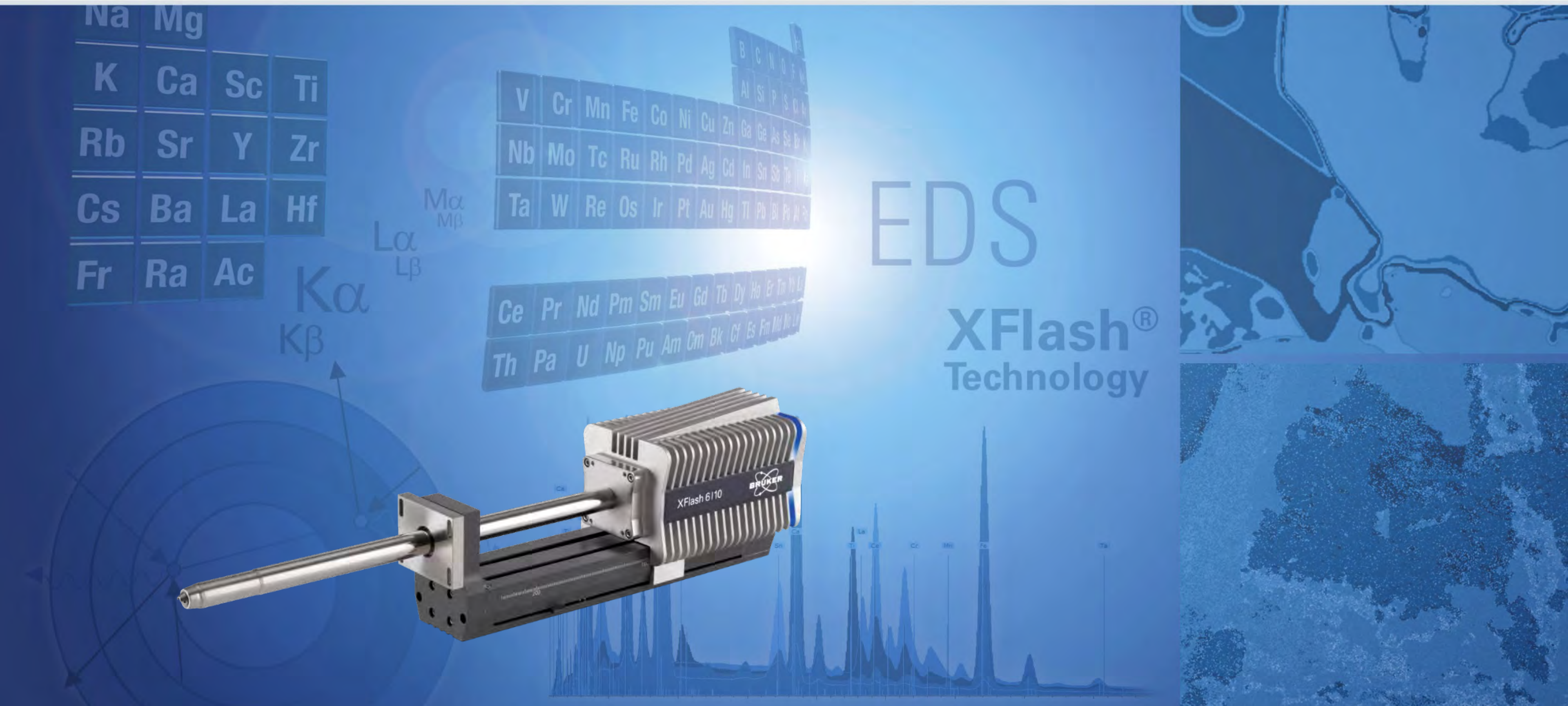
Microanalysis: SEM-EDS

Large Area High Resolution Maps of Geological Samples

Bruker Nano Analytics, Berlin, Germany
Webinar, March, 2019



Presenters

Max Patzschke

Application Scientist
Bruker Nano Analytics, Berlin, Germany

Andrew Menzies, PhD

Applications Scientist,
Bruker Nano Analytics, Berlin, Germany

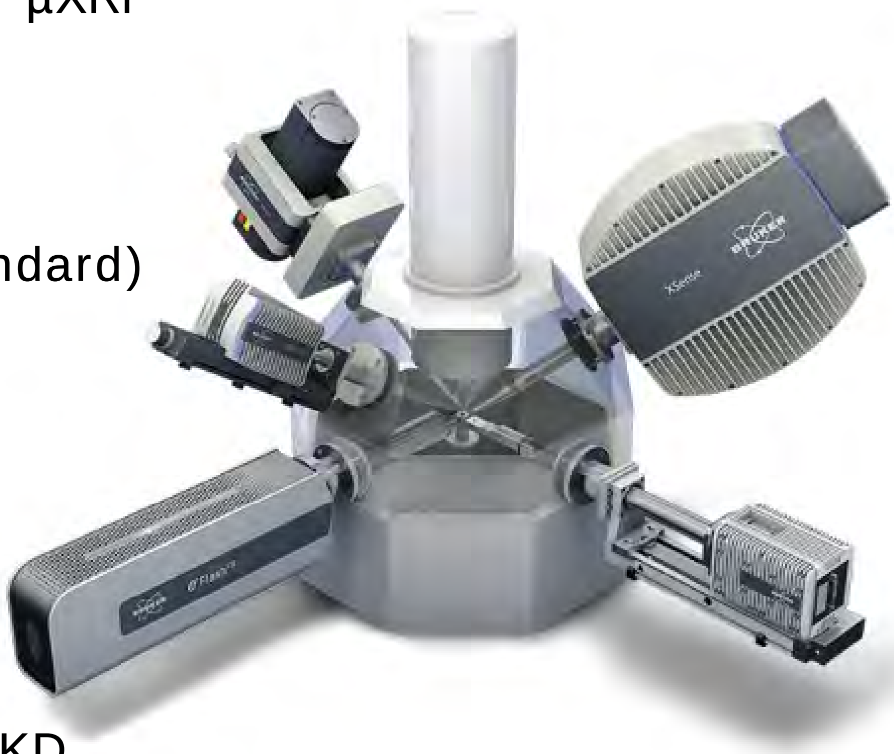
Overview

- Introduction / Presenters
- Webinar Overview – Microanalysis
- Workspaces: Esprit Software
  - SEM-EDS: Elemental Hypermapping
- Examples: Geological Applications:
  - Economic Geology - Exotic Cu Deposits
  - Igneous Petrology - Volcanic Extrusives
  - Metamorphic Petrology
- Analytical Considerations, Summary and Conclusions

Microanalysis: SEM-EDS

Introduction Esprit Software Workspaces

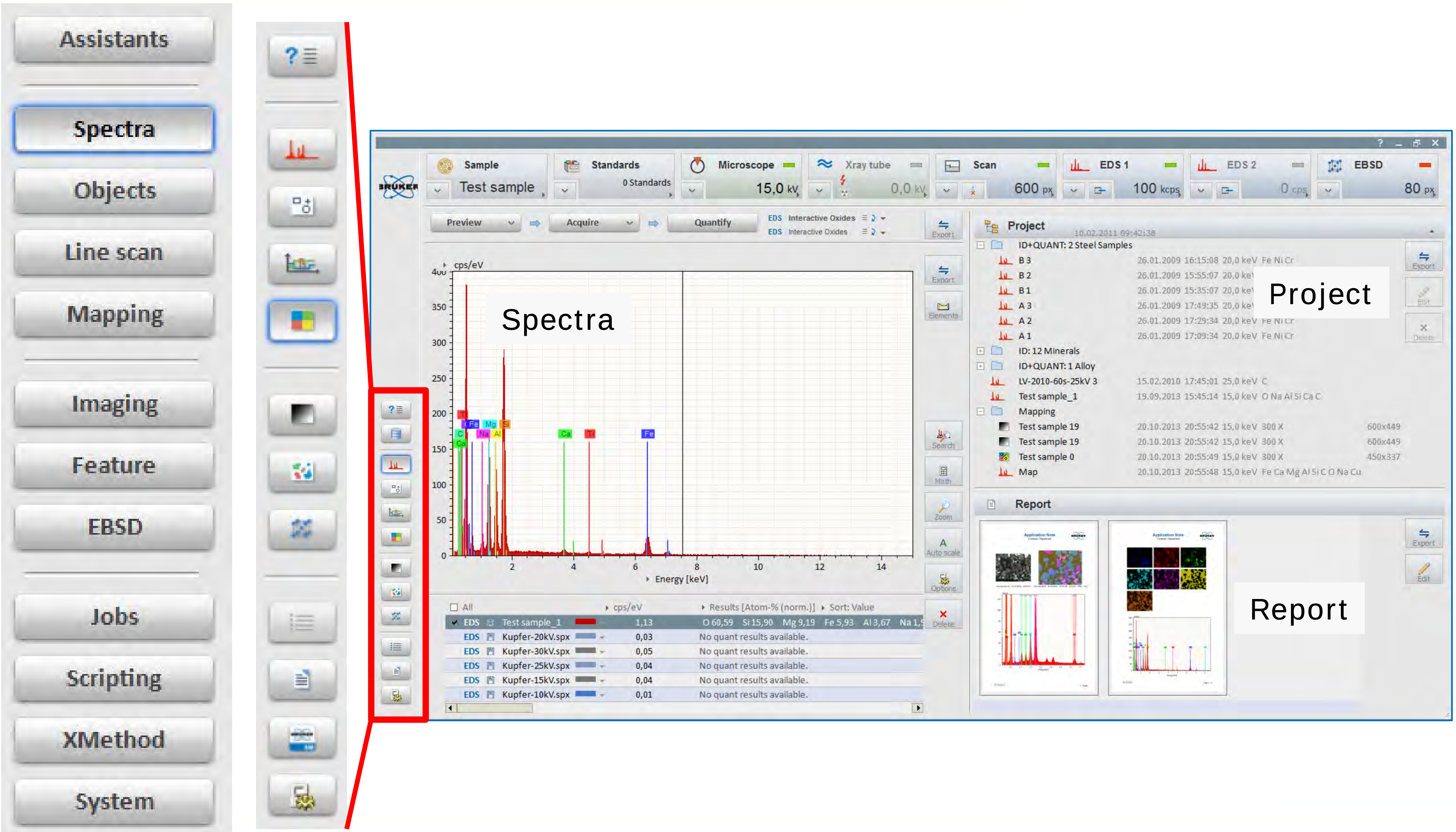
Bruker SEM Analyzers
Our “evolving eyes”



SEM-EDS: Overview
Software – Esprit 2.1

ESPRIT for SEM-EDS

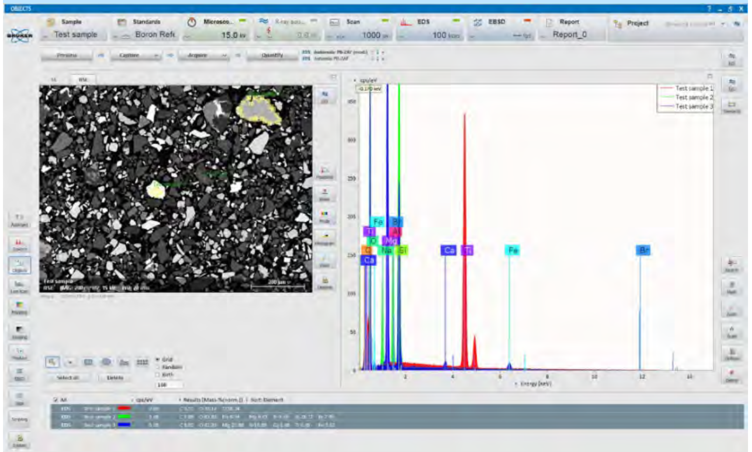
Spectra, Project Management and Report



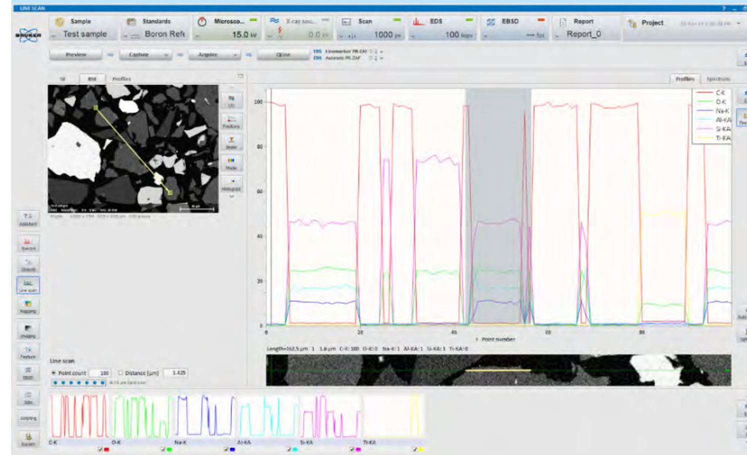
ESPRIT for SEM-EDS
Workspaces

- Assistants
- Spectra
- Objects
- Line scan
- **Mapping**
- Imaging
- Feature
- EBSD
- Jobs
- Scripting
- XMethod
- System

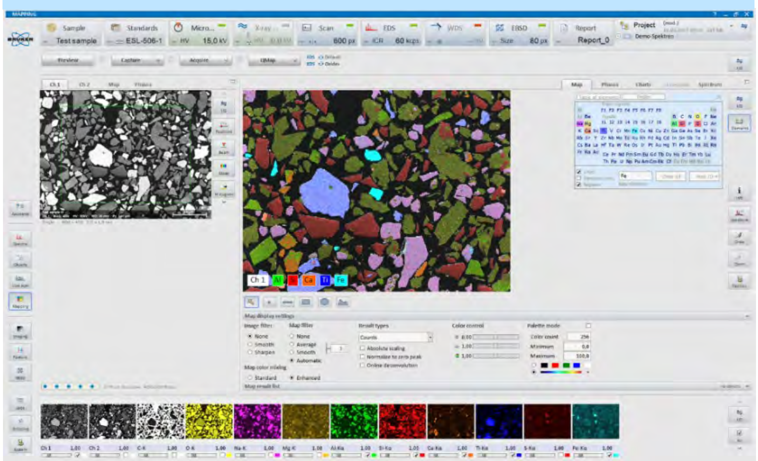
Objects



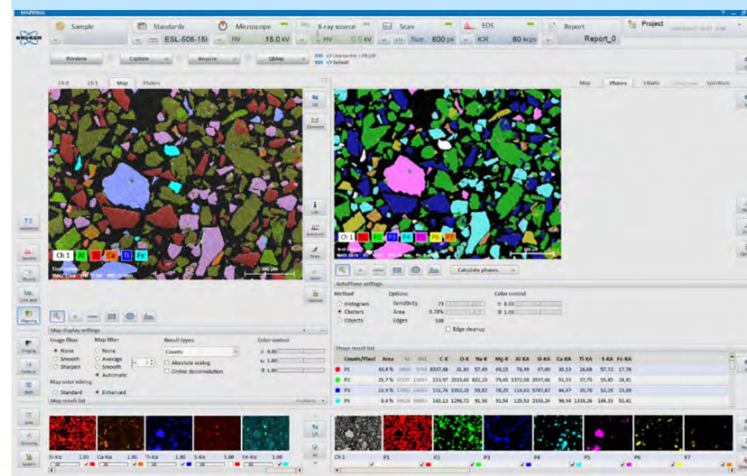
Line Scan



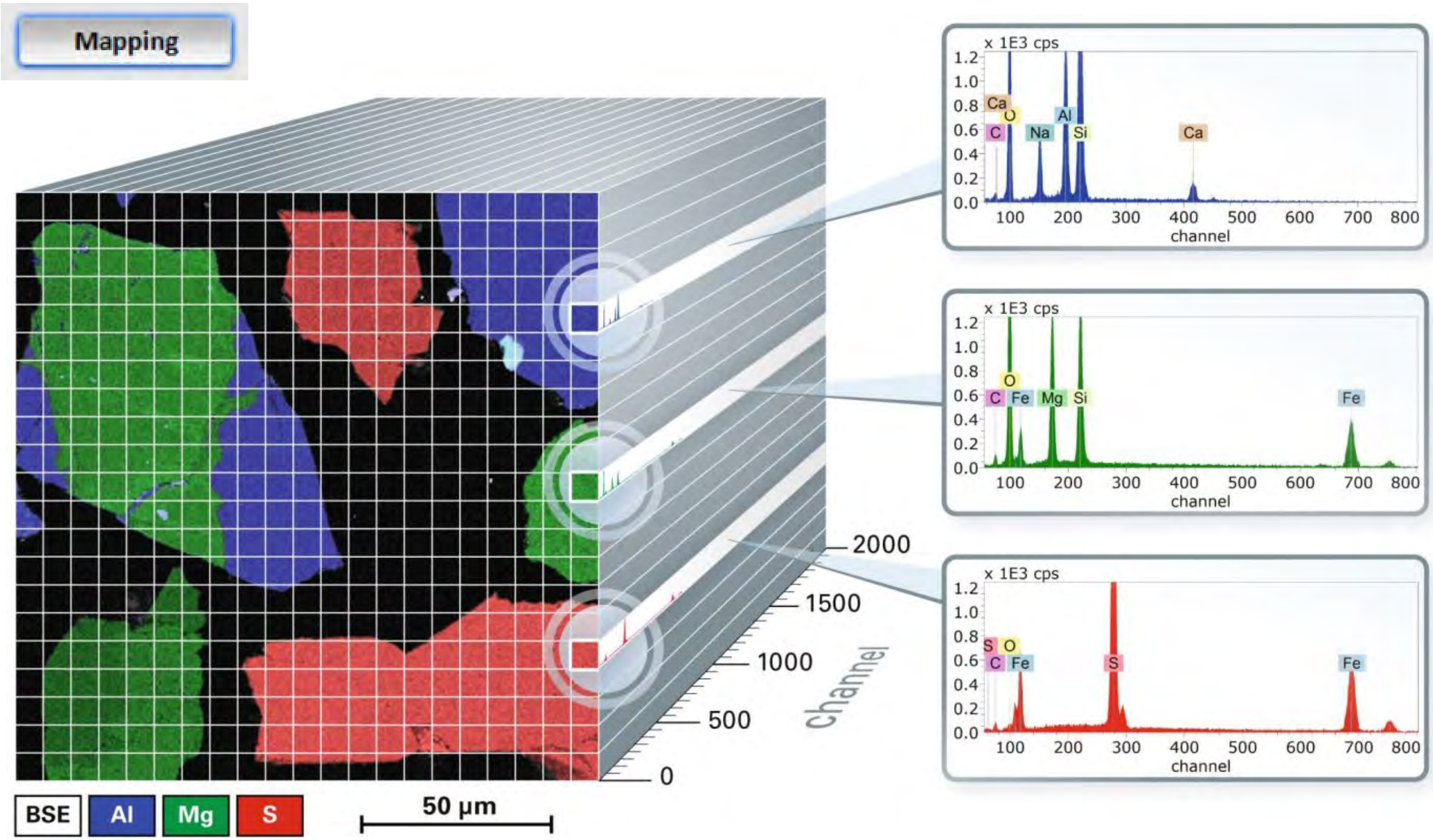
Mapping



Phase Analysis

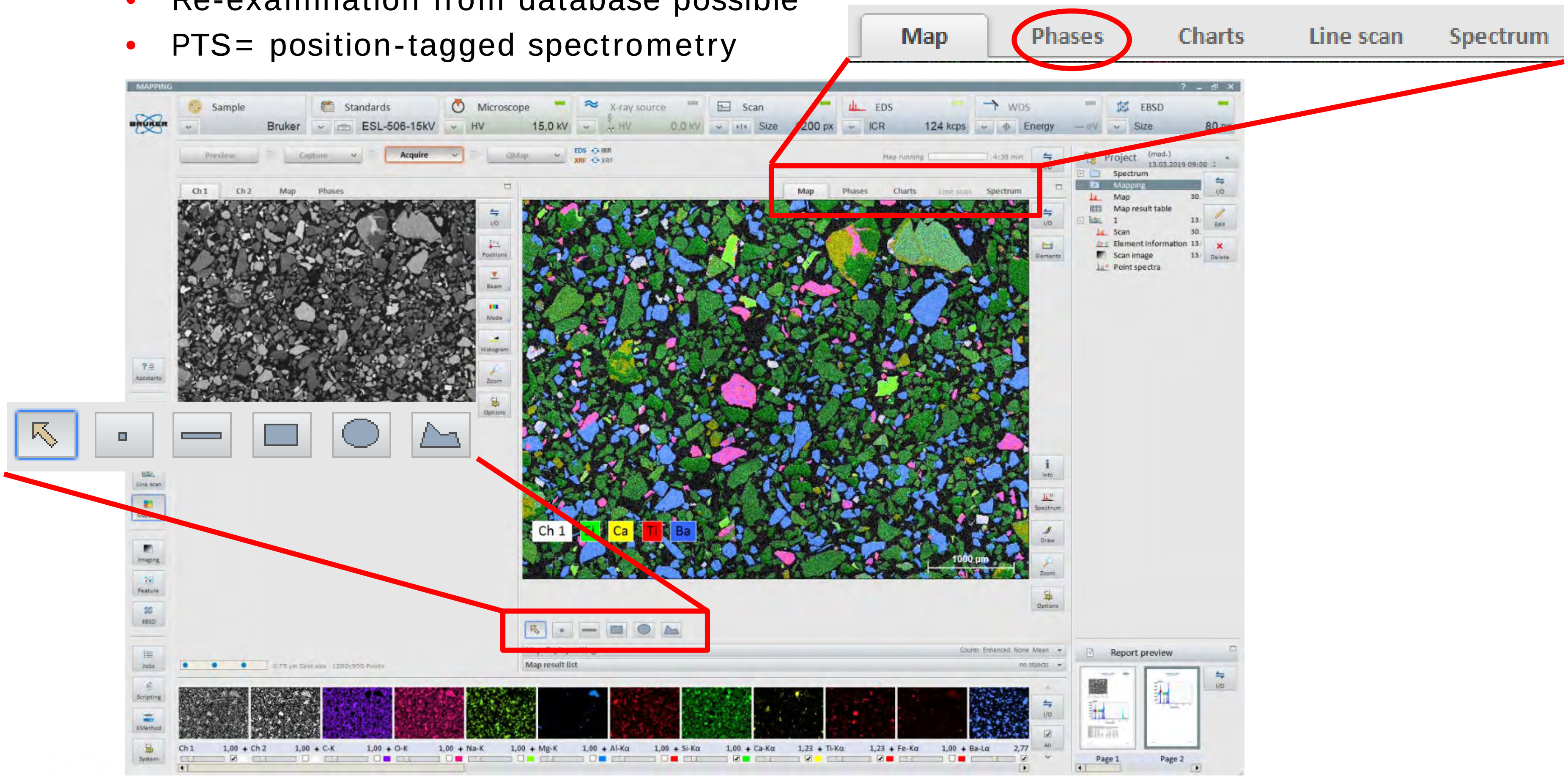


ESPRIT for SEM-EDS
HyperMapping

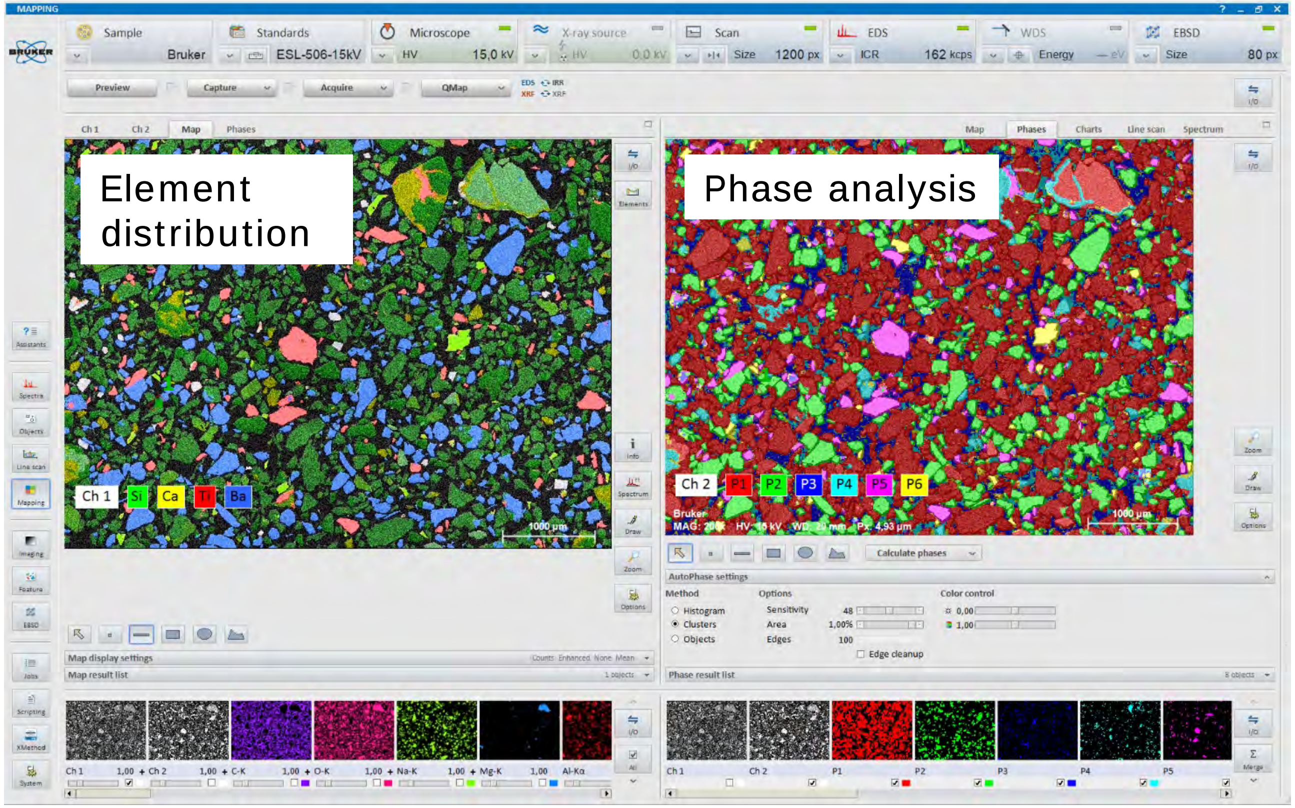


ESPRIT for SEM-EDS
Workspace: HyperMapping

- Spectra storage per pixel
- Internal storage of all element signals
- Re-examination from database possible
- PTS= position-tagged spectrometry



ESPRIT for SEM-EDS
Workspace: HyperMapping



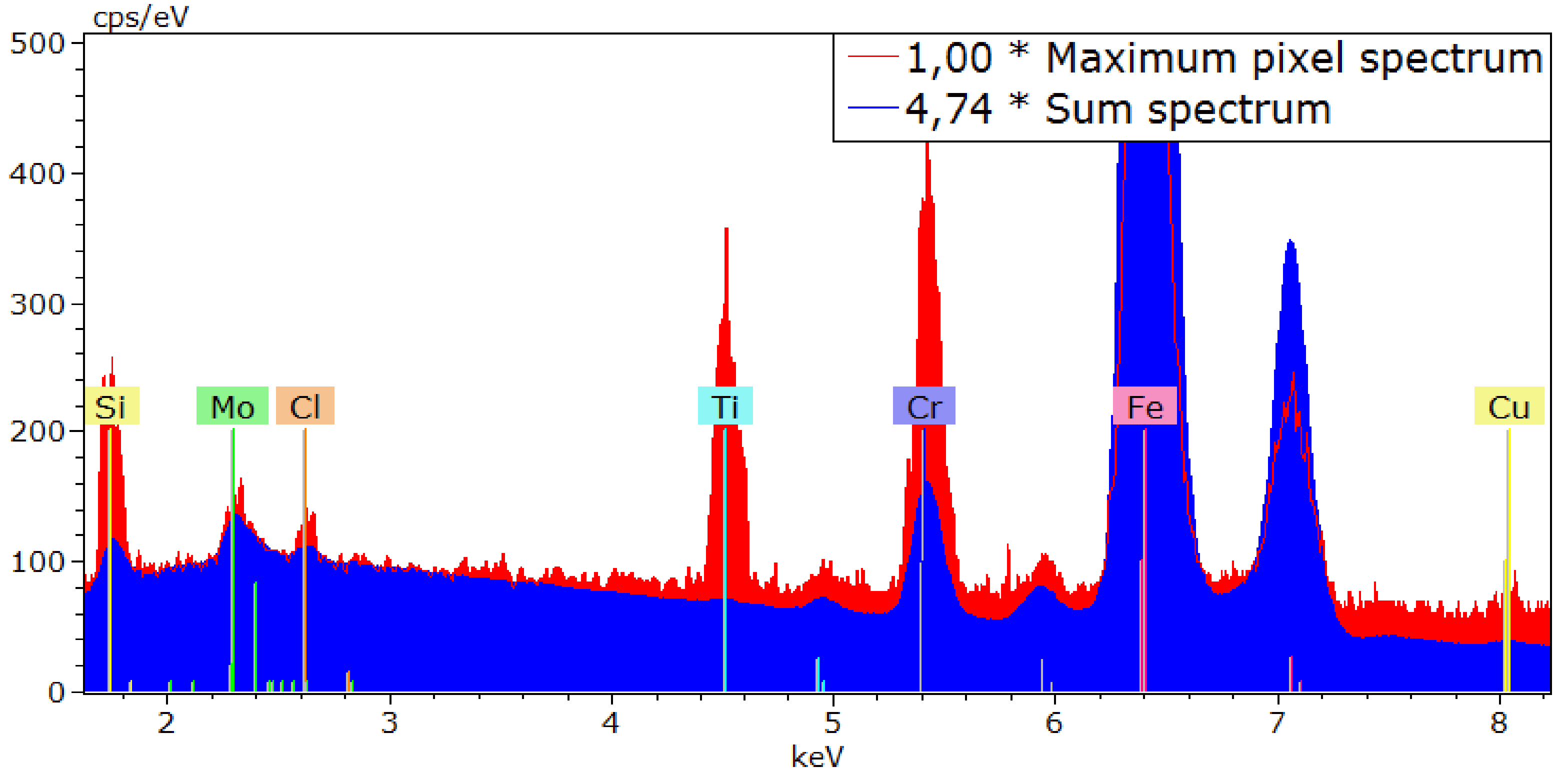
Phases calculated automatically by the software using:

- Histogram,
- Cluster or
- pre defined objects

Sum spectrum and area % are calculated from each phase

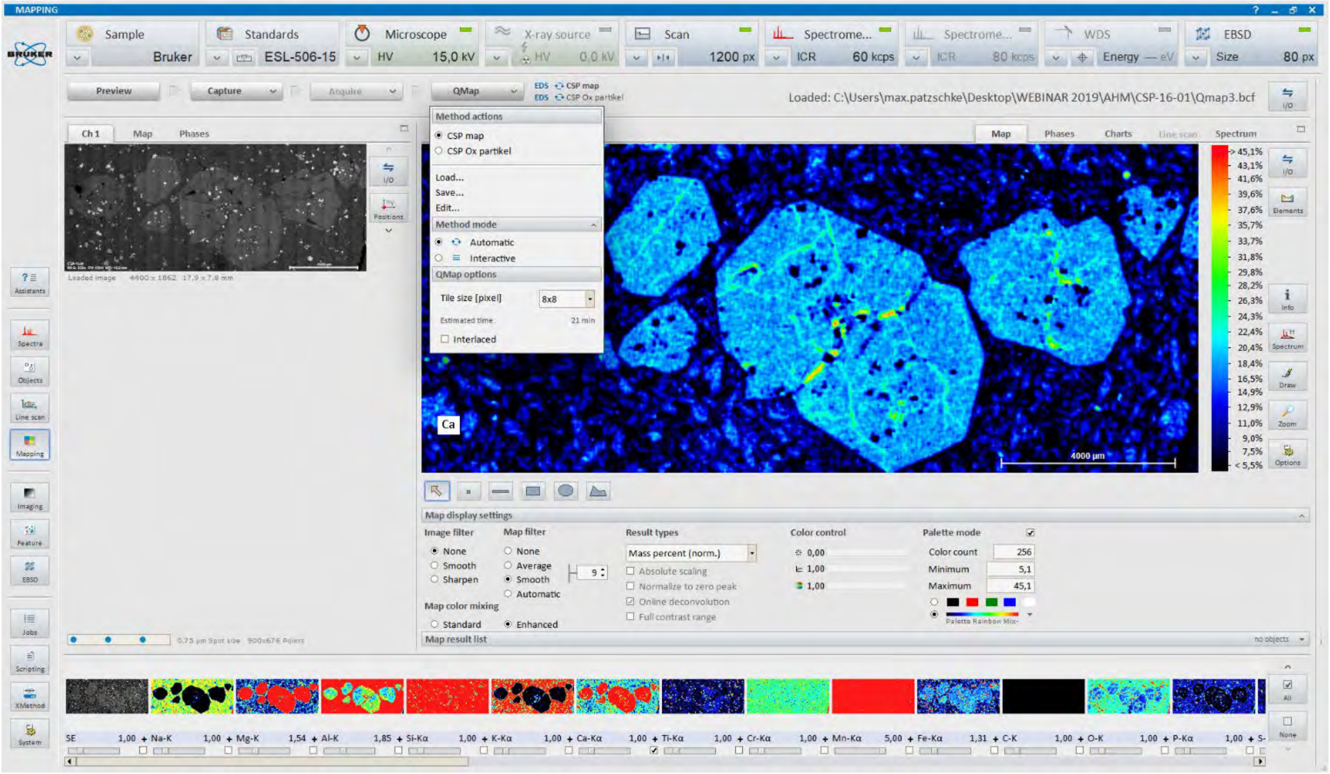
ESPRIT for SEM-EDS
Maximum Pixel Spectrum

Element Identification: MaximumPixelSpectrum



- Maximum Pixel Spectrum for identification of elements that are locally enriched in only a few pixels.

ESPRIT for SEM-EDS
Quantified Map (Qmap)



Maps can be quantified with binning of:

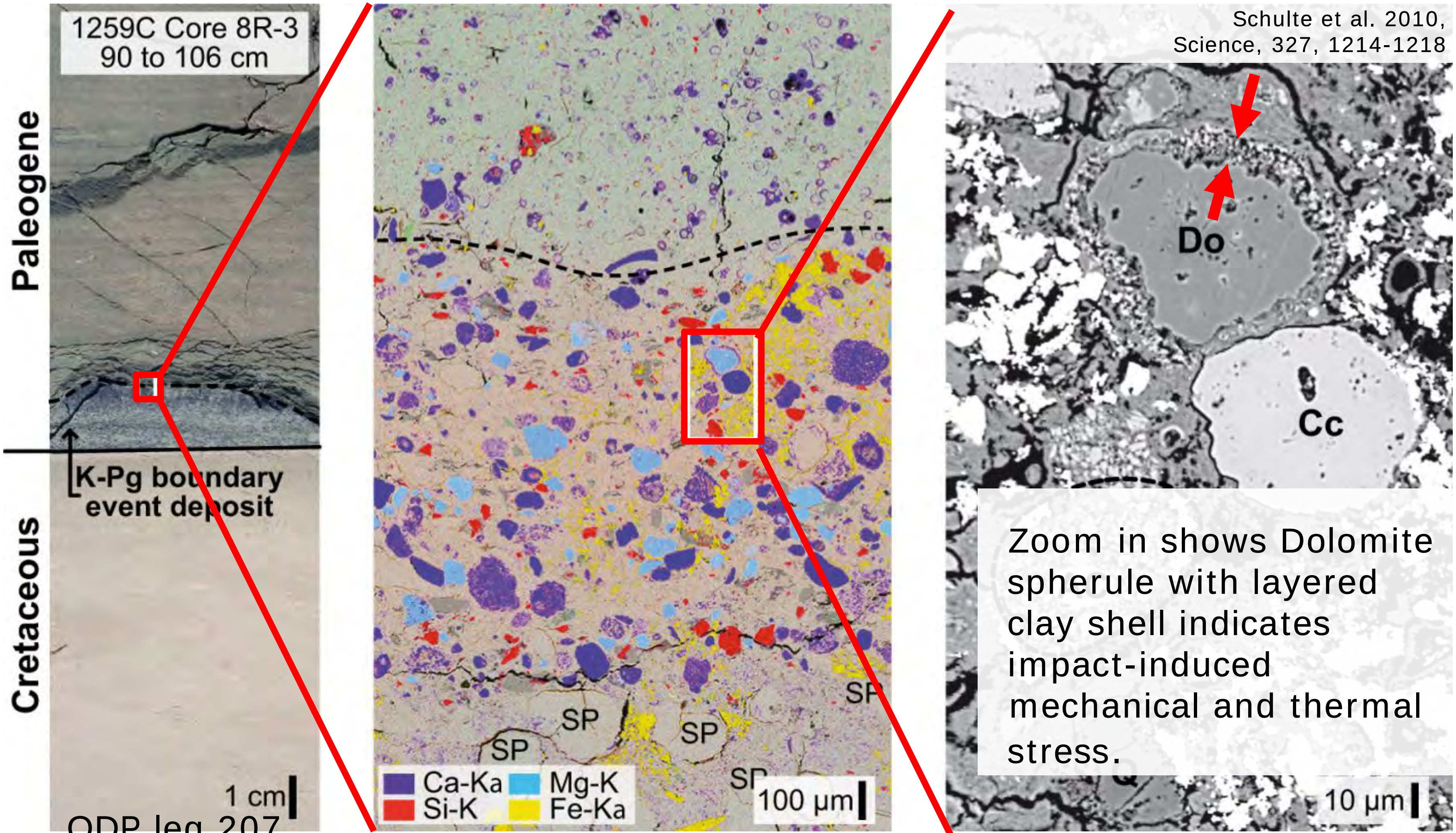
- 1x1 pixel to
- 64x64 pixel

and displayed in:

- counts,
- net sum,
- mass%, or
- atomic%

ESPRIT for SEM-EDS

High-resolution Map with 4072x3072 pixel and 500 kcps

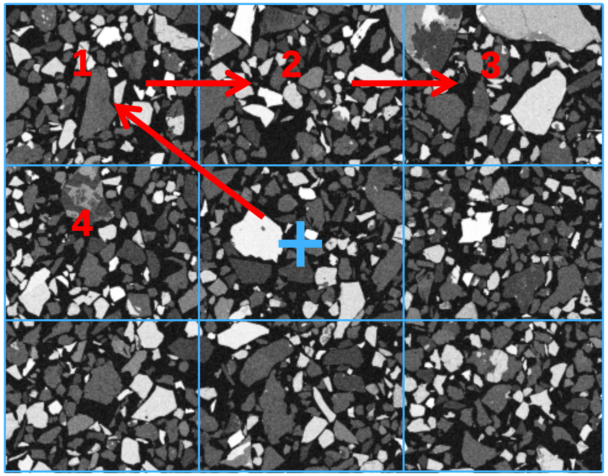


ODP leg 207

Zoom in shows Dolomite spherule with layered clay shell indicates impact-induced mechanical and thermal stress.

ESPRIT for SEM-EDS

Hypermapping: Image Extension



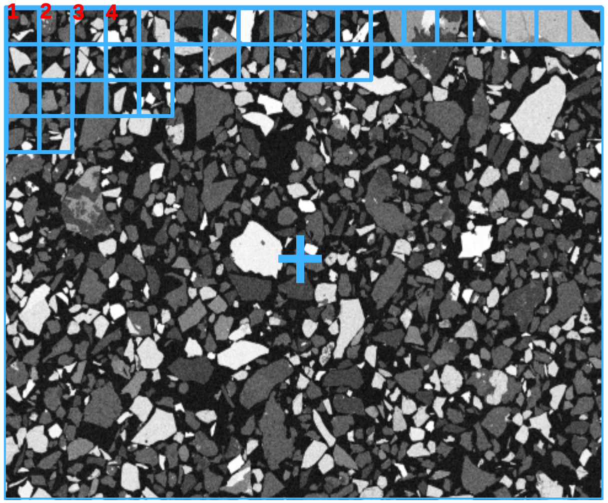
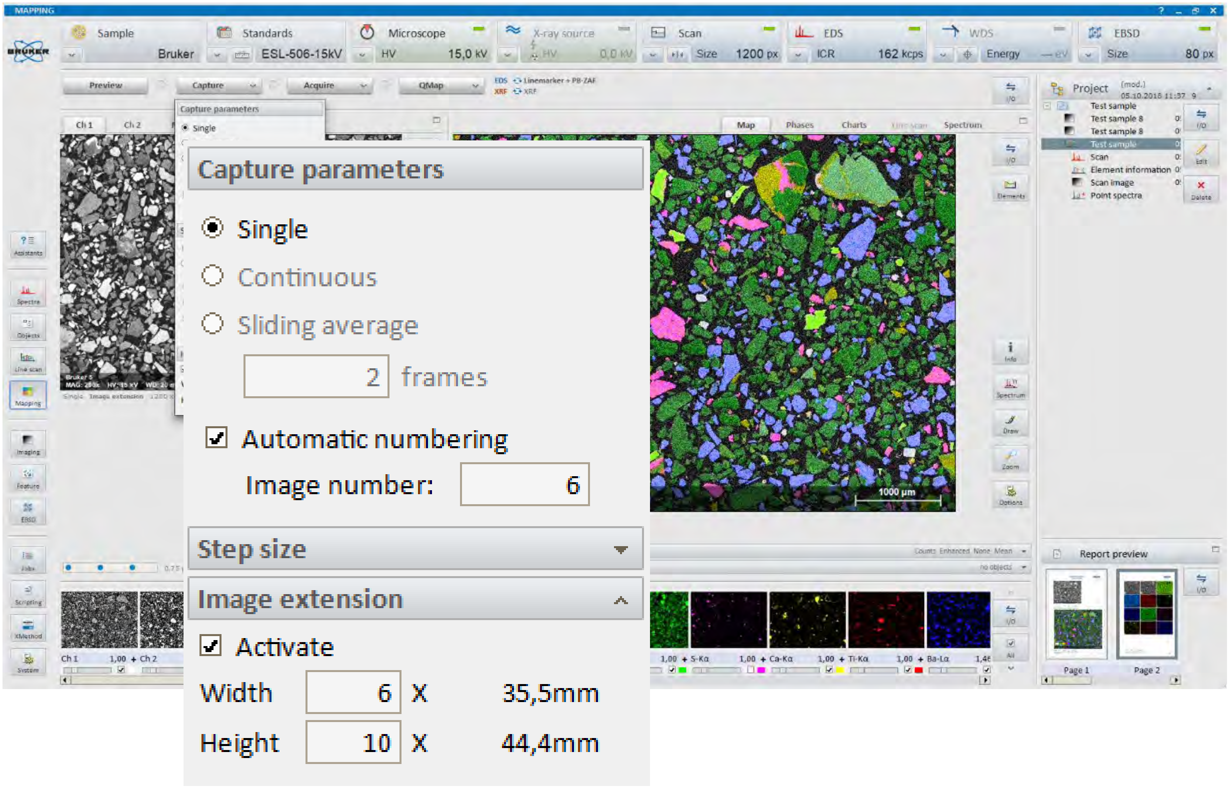


Image Extension

- 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 SEM-EDS

Hypermapping: Image Extension

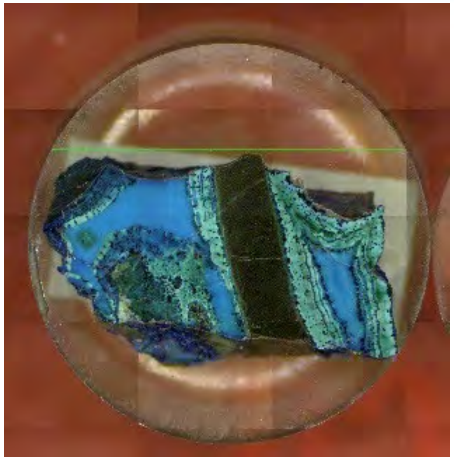


Easy setup for large area mappings

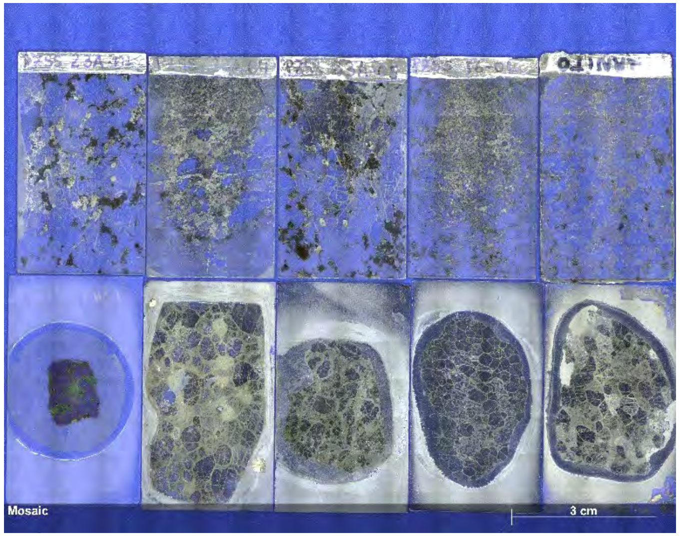
- HyperMaps and images of all kind can be enlarged by activating image extension
- Software uses central mapping position, and build automatic number of desired x/y fields around
- One HyperMap file as result for entire on- and offline data processing
- HyperMaps with more than 20.000x15.000 pixels can be acquired

SEM-EDS: Hypermap
Results - Examples

SEM-EDS: Hypermapping (Large Area Maps)

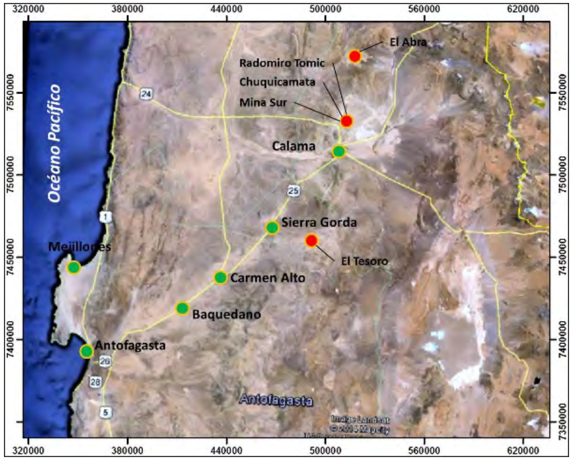


- Peak Intensity maps
  - Deconvolution of Peak Intensities
- Objects:
  - Line Scans
  - Area Spectra and Quantification
- Maximum Pixel Spectrum
  - Identifying trace phases and trace elements
- Qmap (Quantification Maps)
- Autophase ID: Mineralogy

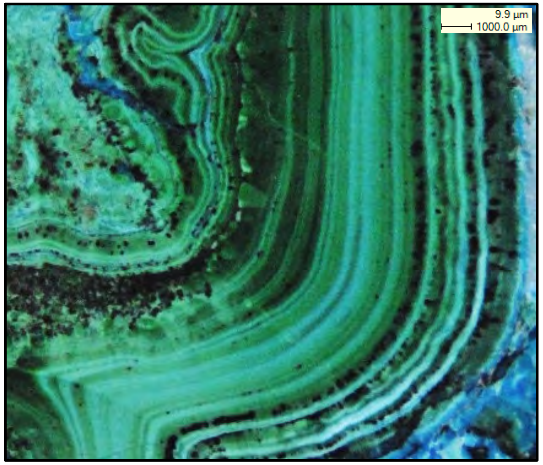


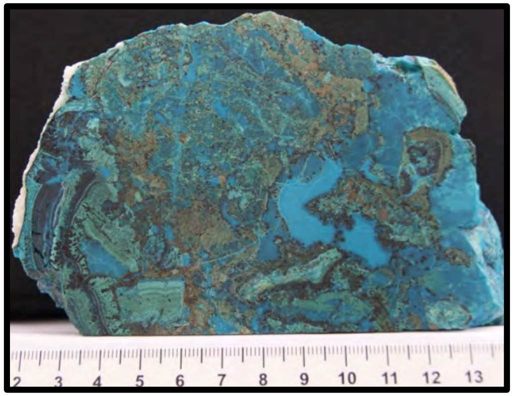
Microanalysis: SEM-EDS
Hypermap - Examples

Geological Applications:
Exotic-Cu Deposits



- Exotic-Cu deposits often form in the vicinity of the parental porphyry system due to the lateral migration of Cu-bearing fluids.
- Mineralisation in this type of deposit comprises different species of copper minerals and mineraloids broadly defined as green-copper (*cobre-verde*) and black-copper (*cobre-negro*) ores.
- The analysis and subsequent definition of Cu-bearing minerals from exotic-Cu deposits is extremely complex due to the fine scaled textures and compositional variation.





Chrysocola

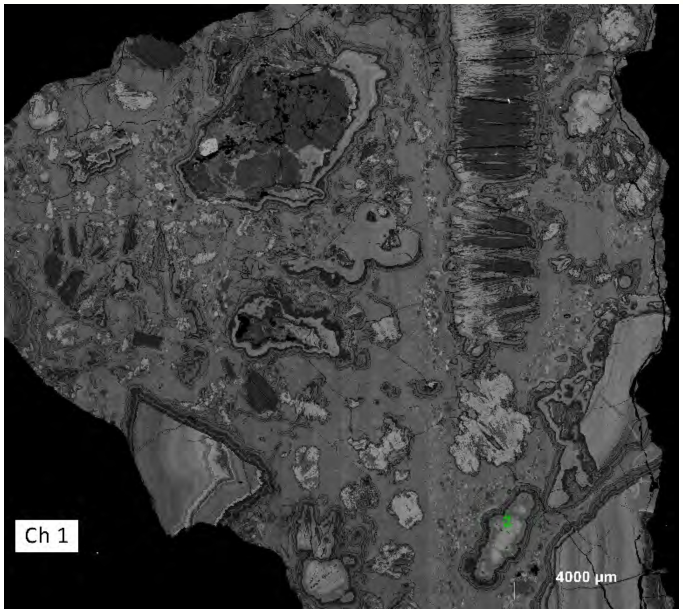
$$Cu_{2-x}(Al,Fe^{3+})_xH_{2-x}[Si_2O_5](OH)_4*nH_2O$$

Geological Applications:
Exotic-Cu Deposits

Cu-Sulphides	Cu-bearing Sulphides, including Covellite, Diginite, Chalcopyrite, Bornite, Tennantite, Tetrahedrite and Enargite
Cu-Oxides	Cu-bearing Oxides, including Tennorite, Cuprite, Delafossite, and Cuprospinel
Cu-Halides	Cu-bearing Halides, including Atacamite
Cu-Carbonates	Cu-bearing Carbonates, including Malachite and Azurite
Cu-Sulphates	Cu-bearing Sulphates, including Brochantite
Cu-Phosphates	Cu-bearing Phosphates, including Turquoise, Pseudomalachite, Libethenite, Sampleite and Chalcosiderite
Cu-Silicates	Cu-bearing Silicates, including Chrysocolla, Plancheite, Shattuckite and closely related Cu-Si-O phases
Cu-Mn Oxides	Cu-bearing mineral phases incorporating Cu-Mn-O with other minor elemental components
Silicates+Cu	Various Silicates that incorporate an additional minor Cu component, including Chlorite, Biotite, Kaolinite, Illite, Montmorillonite and Nontronite
Fe-Oxides+Cu	Various Fe-Oxides that incorporate an additional minor Cu component, including Goethite, Limonite, Hematite, and Magnetite

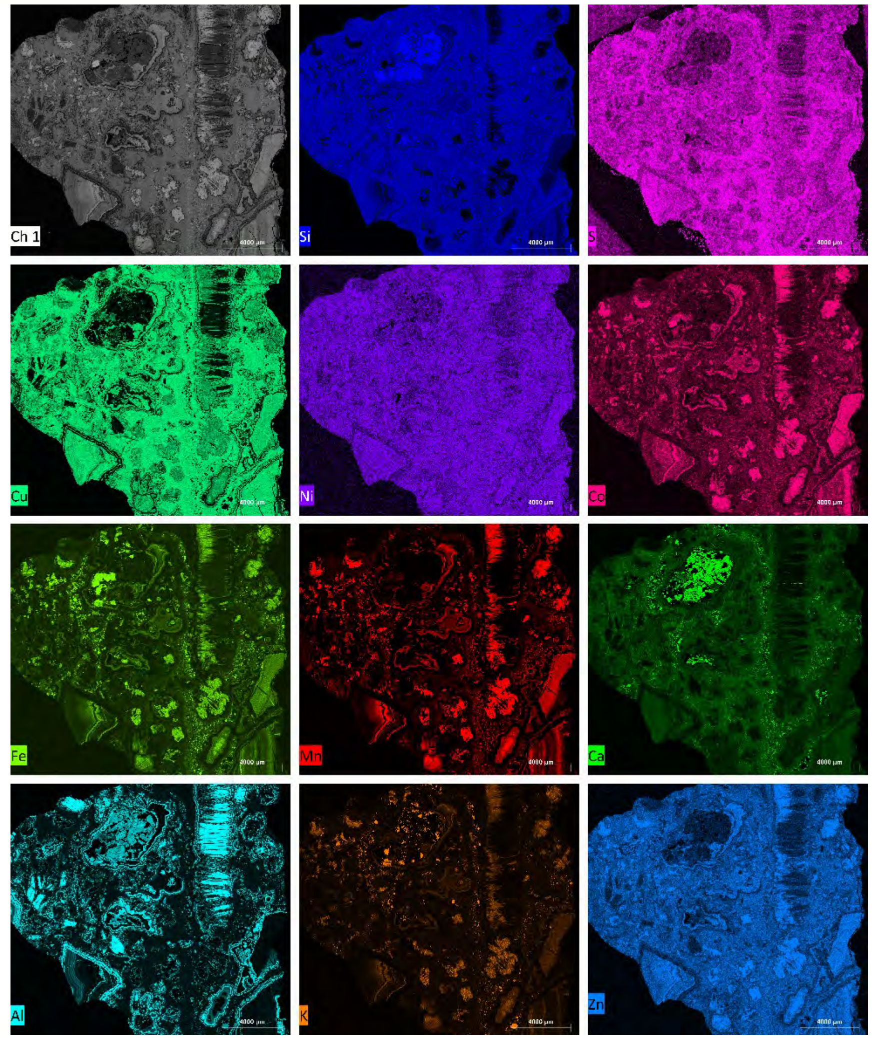
SEM-EDS: Hypermap Results

Image Extension

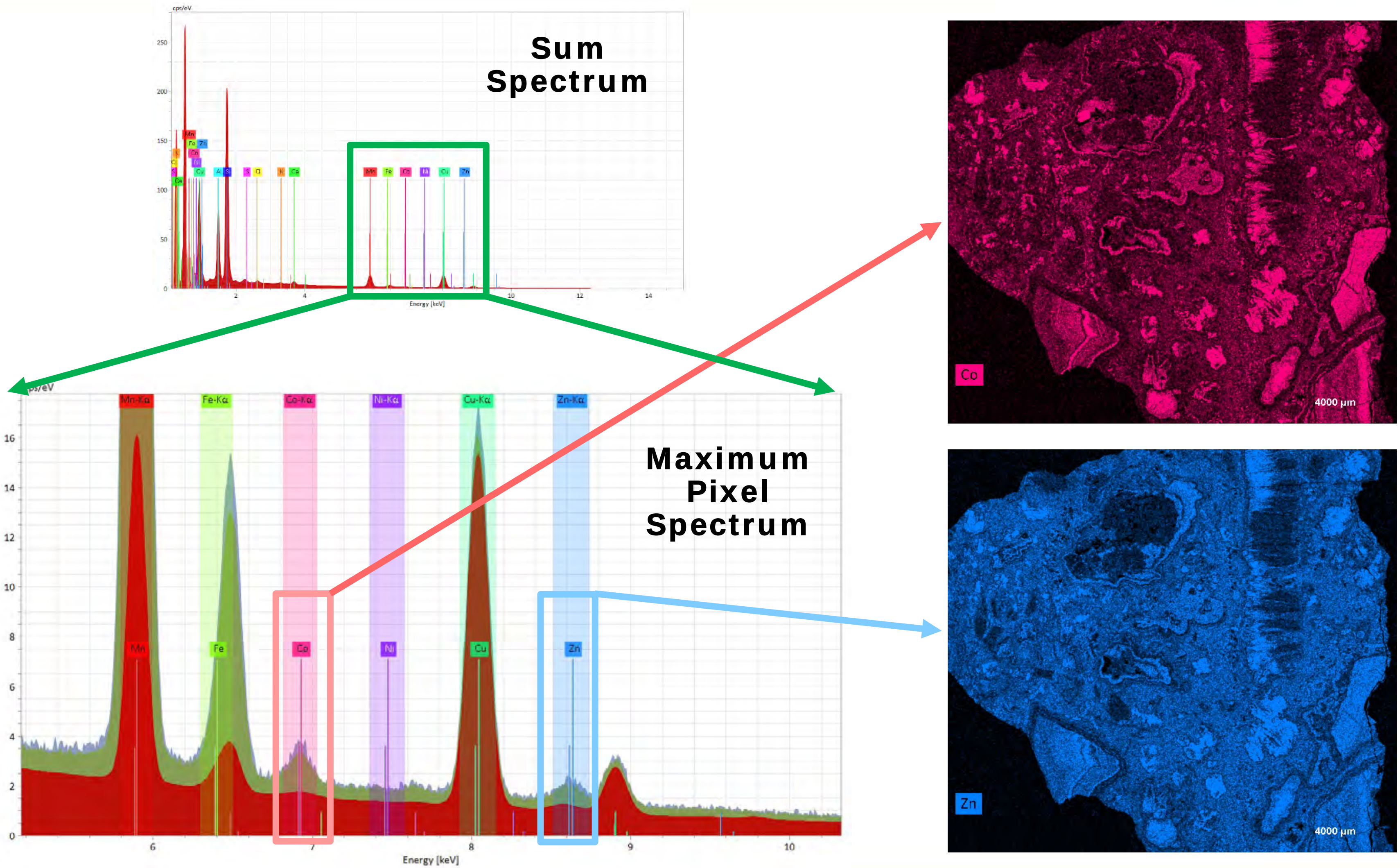


Measurement Conditions

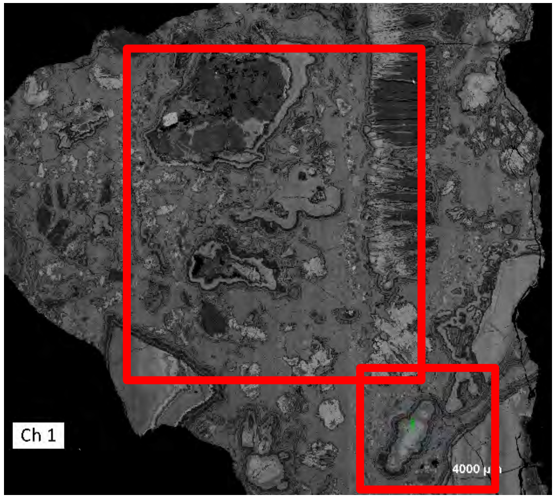
High Voltage:	15 kV
Pixels:	4800x4788
Measurment Time:	1568 min
SDD:	10 mm^2
Dwell time:	4096 µs
FOV:	19.0 mm
Pixel size:	4.1 µm
Fields:	308 (30x40)
Magnification:	250x

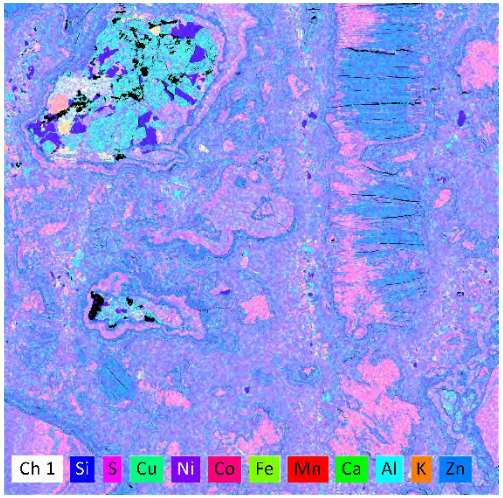


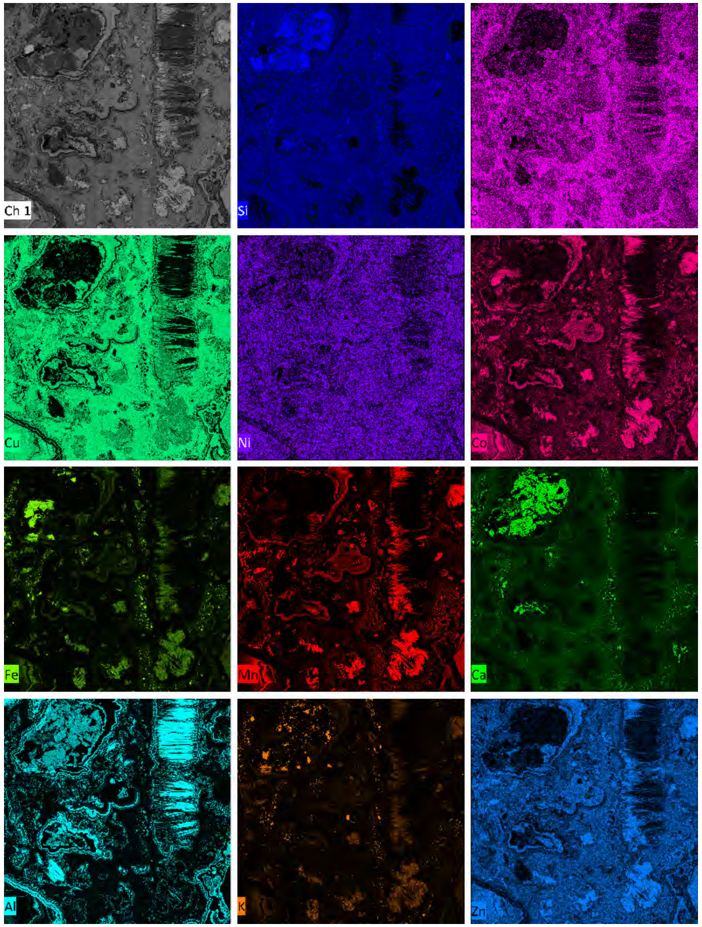
SEM-EDS: Hypermap Results
Minor Elements – Maximum Pixel Spectrum



SEM-EDS: Hypermap Results
Image Extension



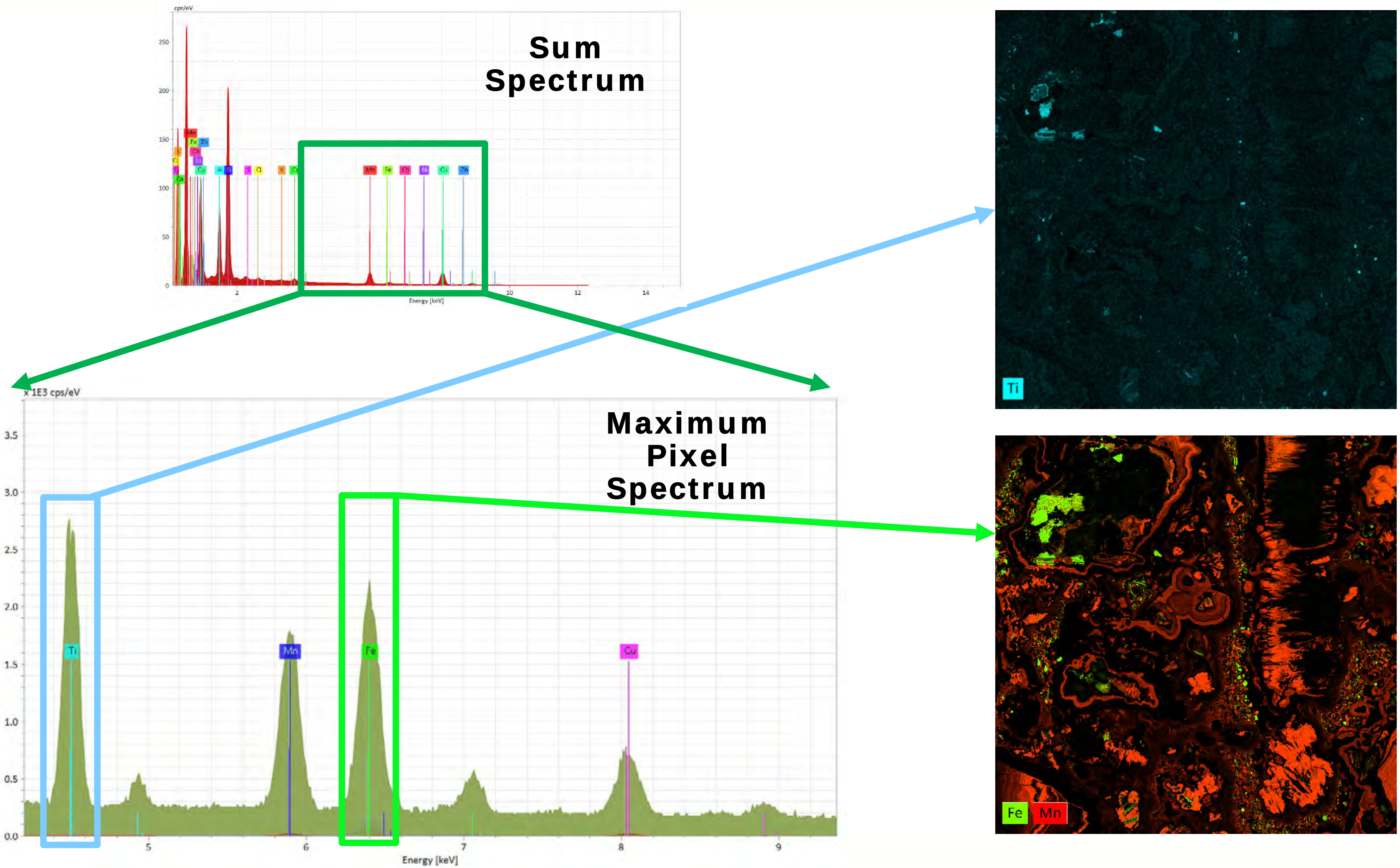




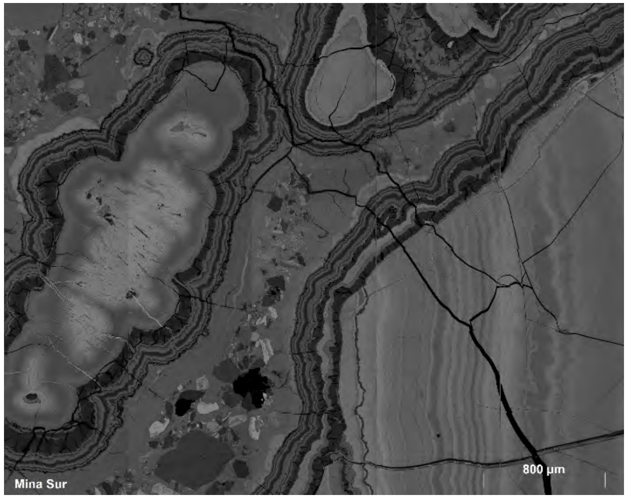
Measurement Conditions

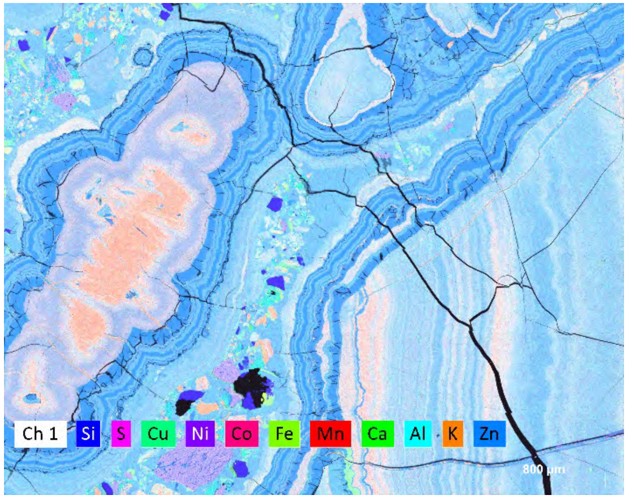
High Voltage:	15 kV
Pixels:	4800x4788
Measurment Time:	1568 min
SDD:	10 mm^2
Dwell time:	4096 µs
FOV:	19.0 mm
Pixel size:	4.1 µm
Fields:	308 (30x40)
Magnification:	250x

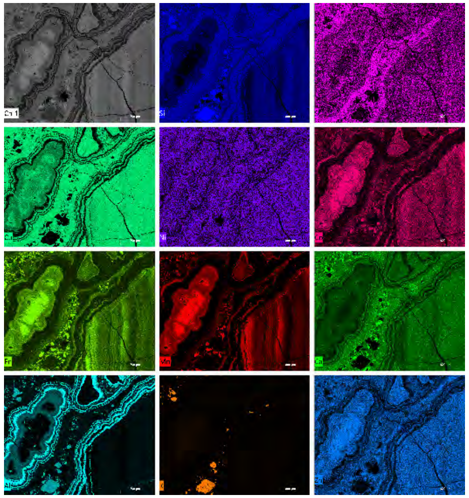
SEM-EDS: Hypermap Results
Minor Elements – Maximum Pixel Spectrum



SEM-EDS: Hypermap Results
Image Extension

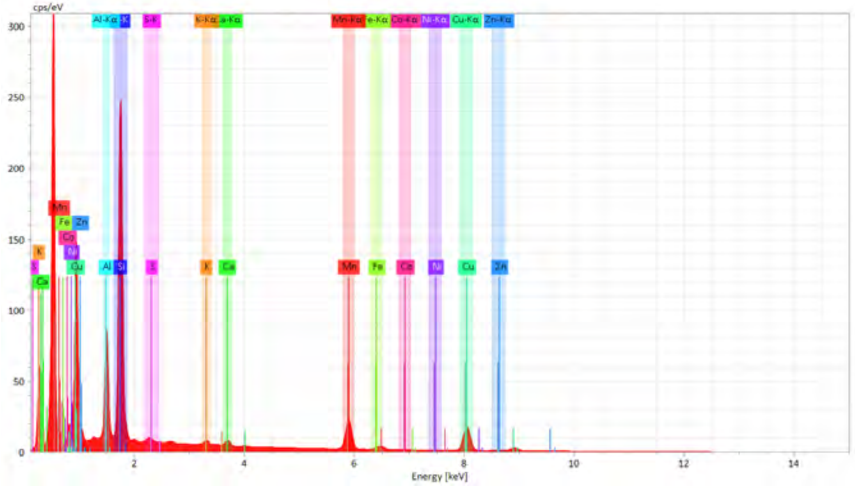






Measurement Conditions

High Voltage:	15 kV
Pixels:	3000x2400
Measurment Time:	490 min
SDD:	10 mm²
Dwell time:	4096 µs
FOV:	4.1 mm
Pixel size:	1.4 µm
Fields:	560 (20x28)
Magnification:	250x

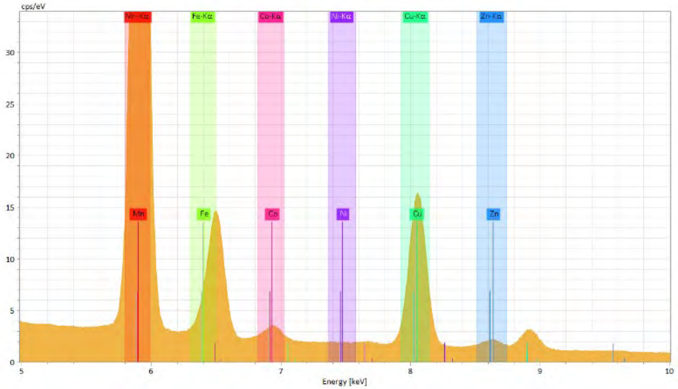


SEM-EDS: Hypermap Results

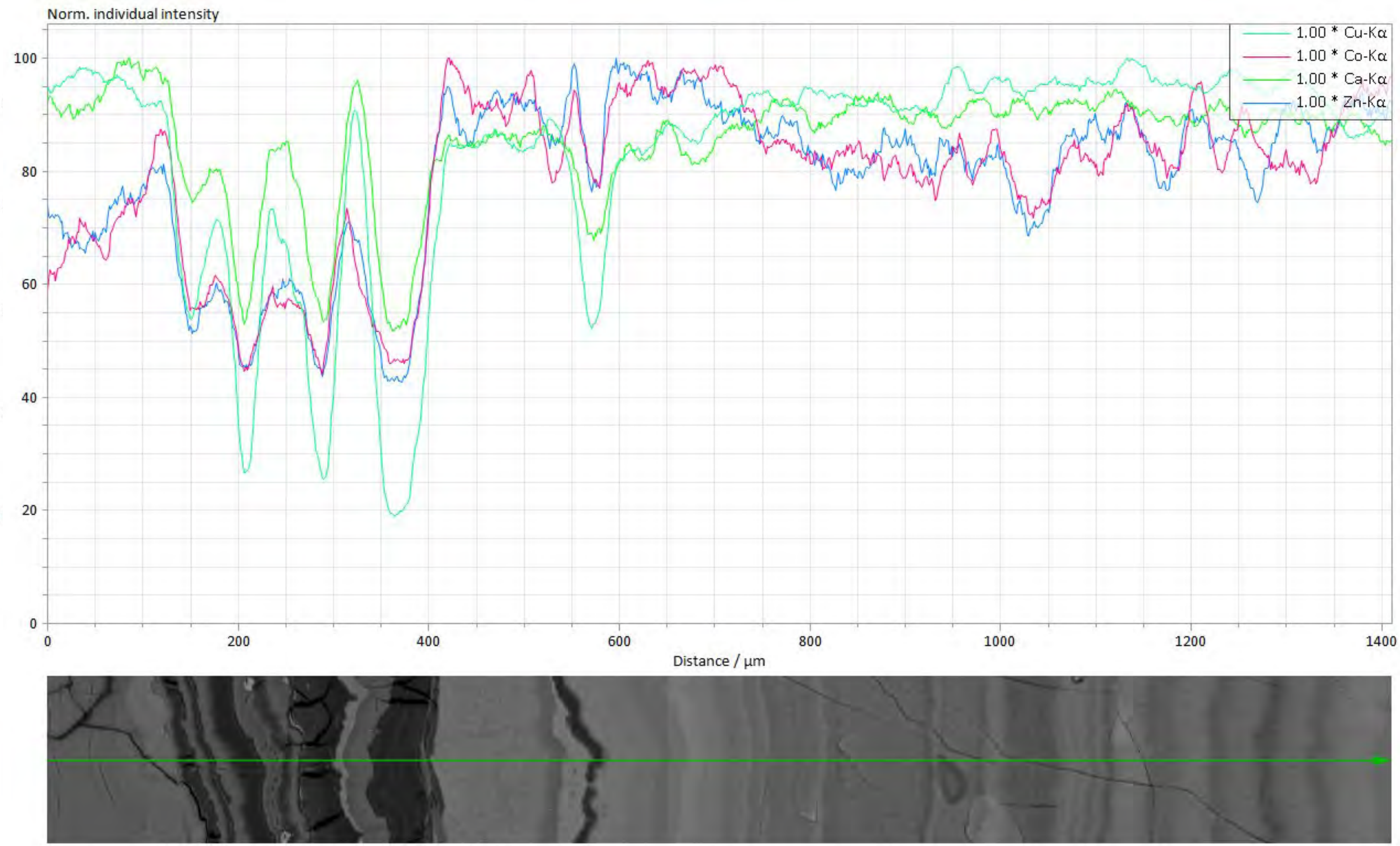
Line Scans

Measurement Conditions

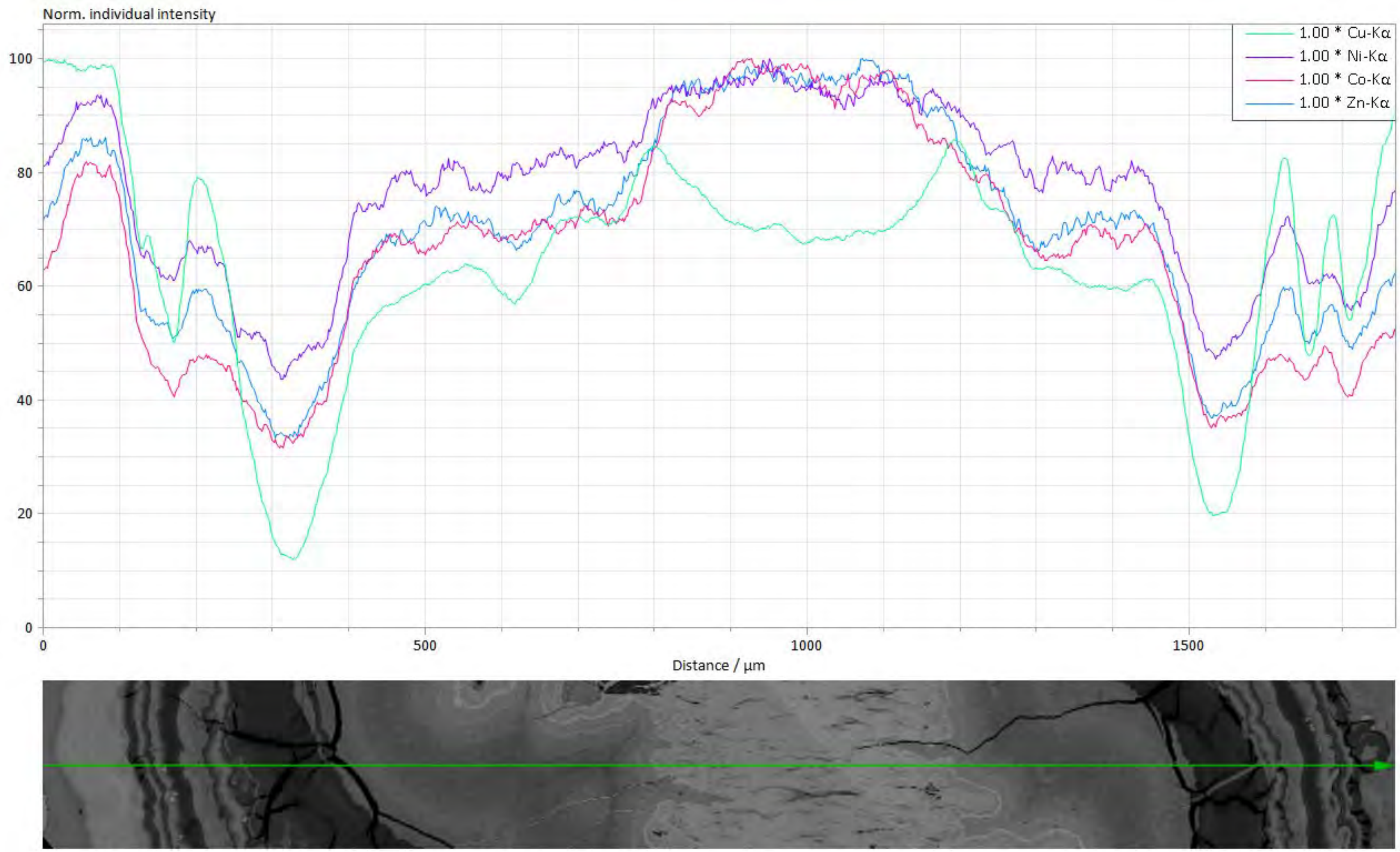
High Voltage:	15 kV
Pixels:	3000x2400
Measurment Time:	490 min
SDD:	10 mm^2
Dwell time:	4096 µs
FOV:	4.1 mm
Pixel size:	1.4 µm
Fields:	560 (20x28)
Magnification:	250x



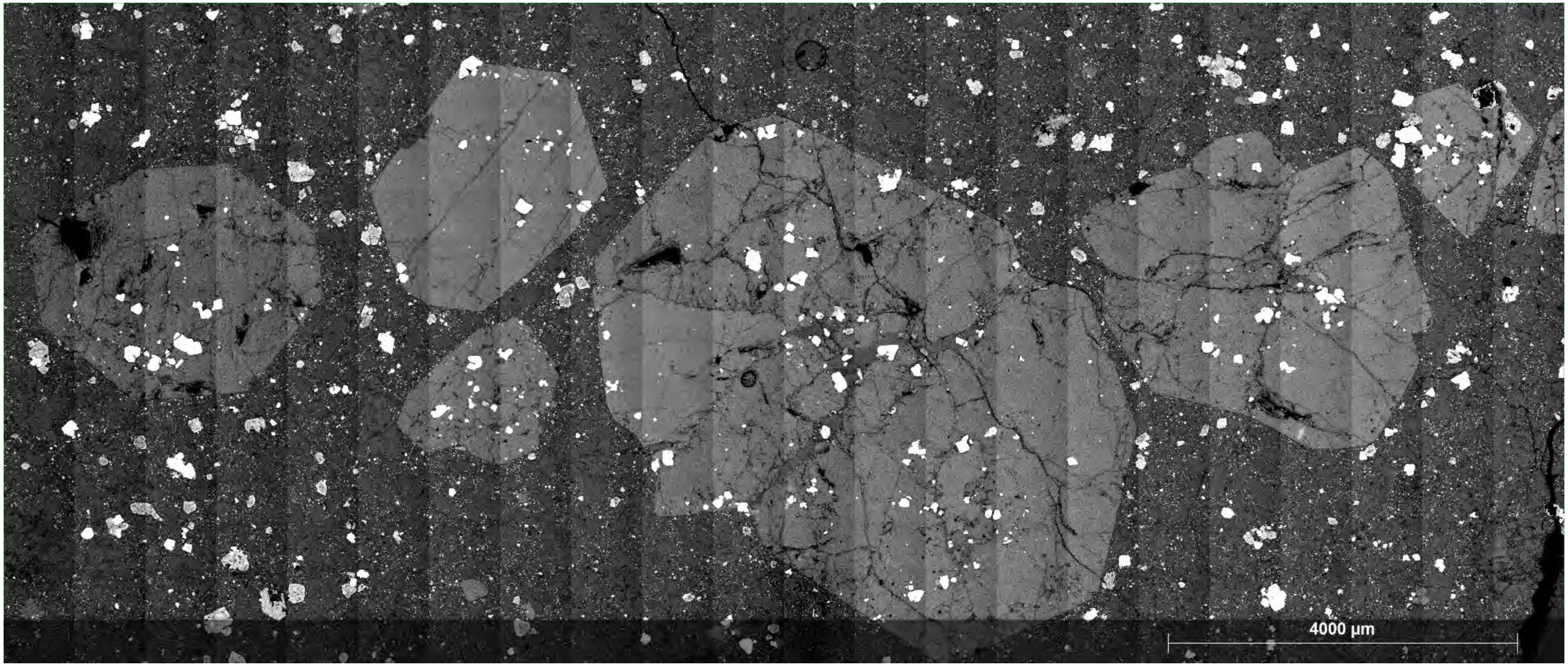
SEM-EDS: Hypermap Results
Line Scans



SEM-EDS: Hypermap Results
Line Scans



Geological Applications:
Igneous Volcanic Extrusive

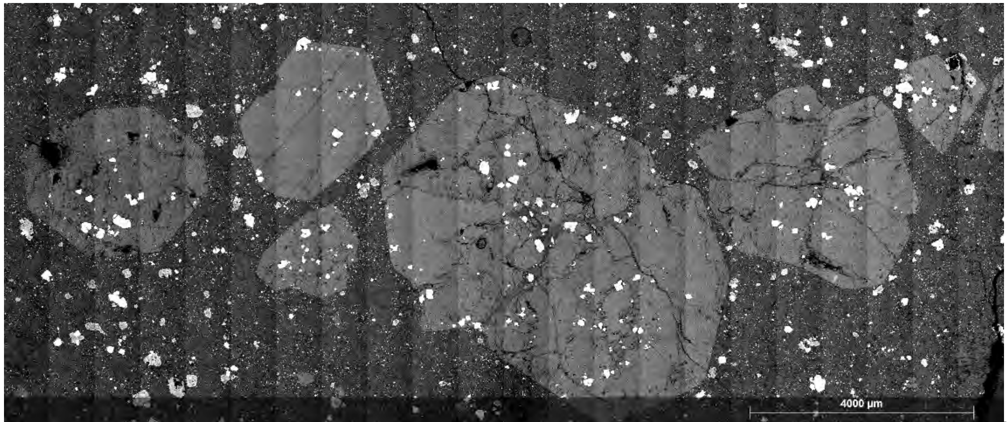


Measurement Conditions:

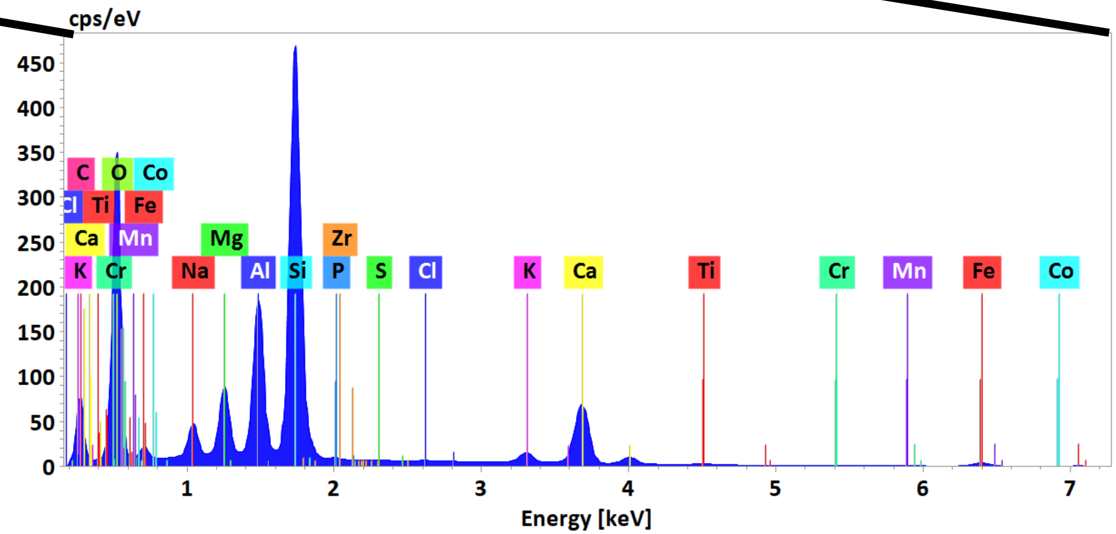
High Voltage:	15 kV
Pixels:	8800x5600
Measurment Time:	616 min
SDD:	10 mm^2

Dwell time:	23 µs
FOV:	17.8 mm
Pixel size:	4.1 µm
Fields:	308 (22x14)
Magnification:	250x

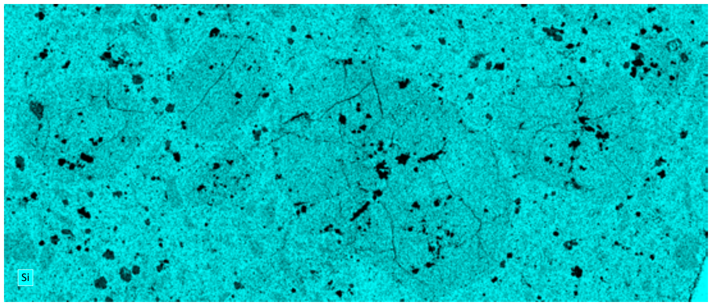
SEM-EDS: Hypermap Results
Image Extension

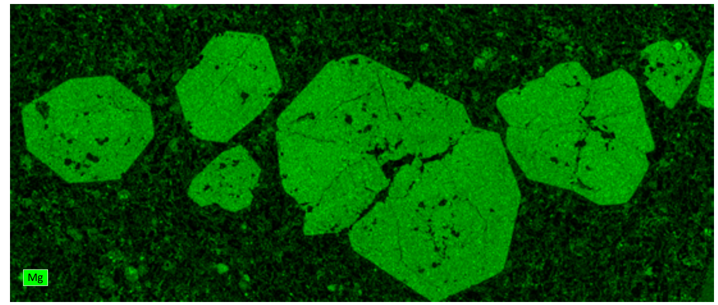


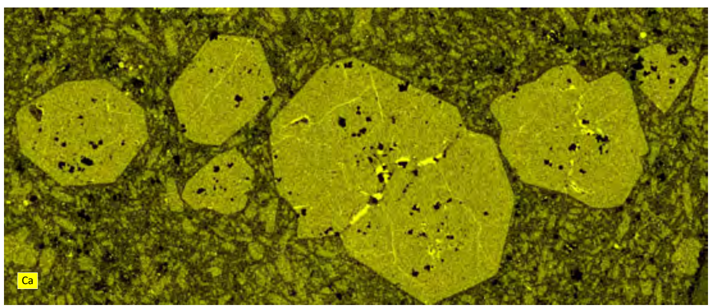
Hypermap:
- Database contains a spectrum for each pixel
- Sum spectrum reveals major and minor elements in the map
- Full offline post proccessing possible

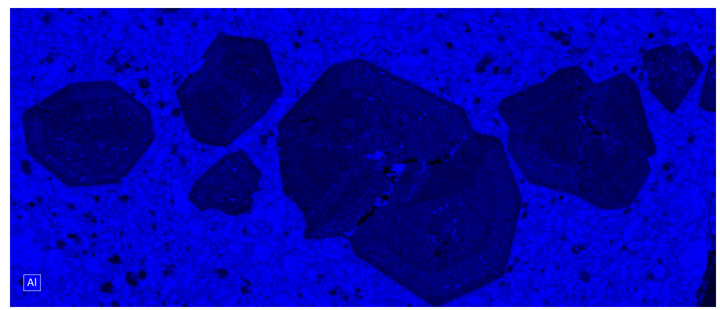


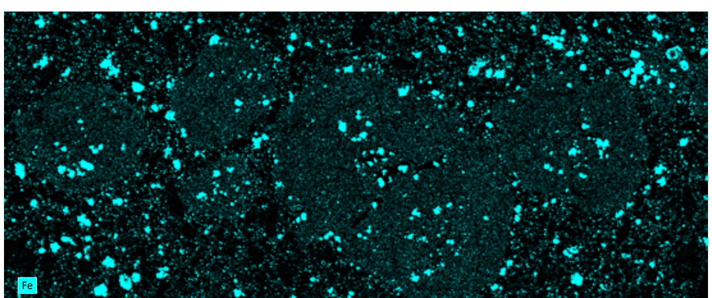
SEM-EDS: Hypermap Results
Elemental Peak Intensity Maps

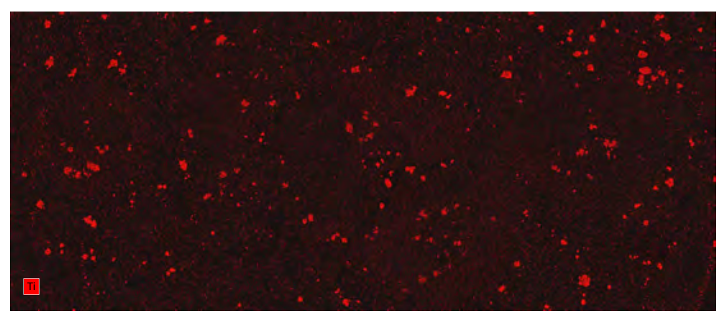






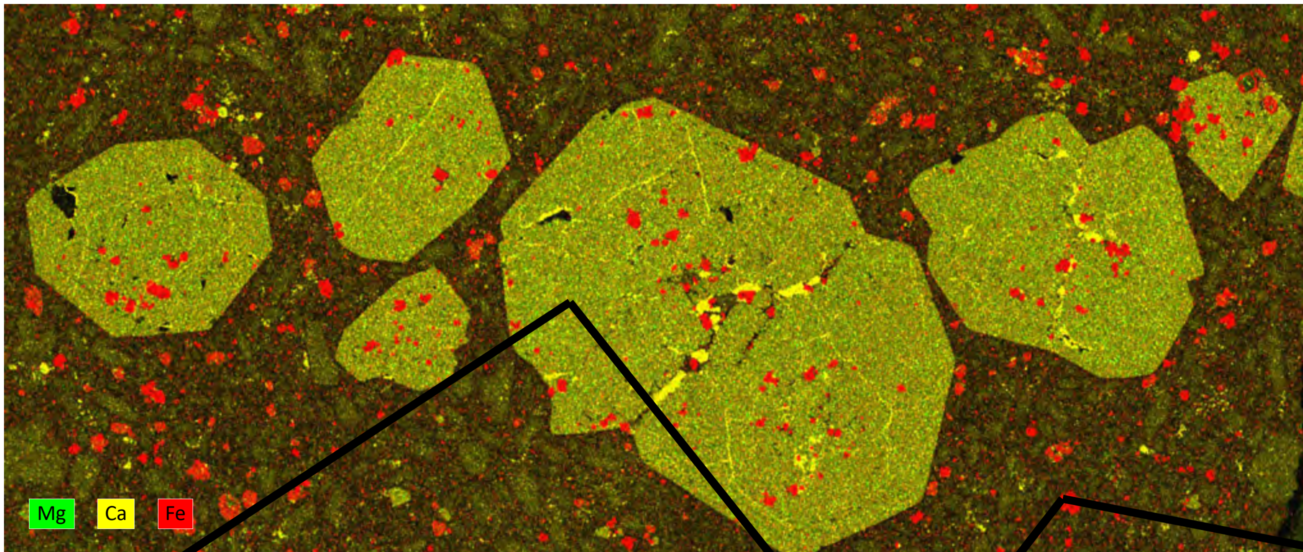


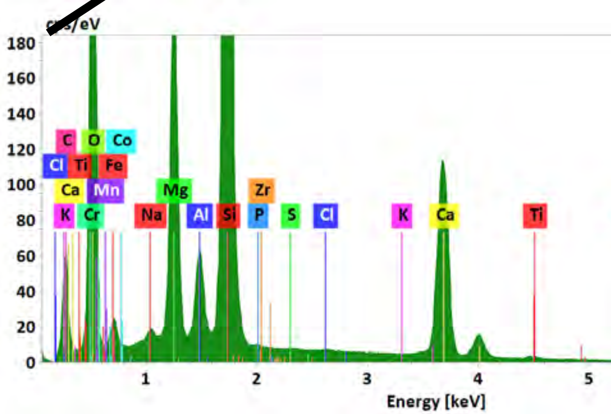




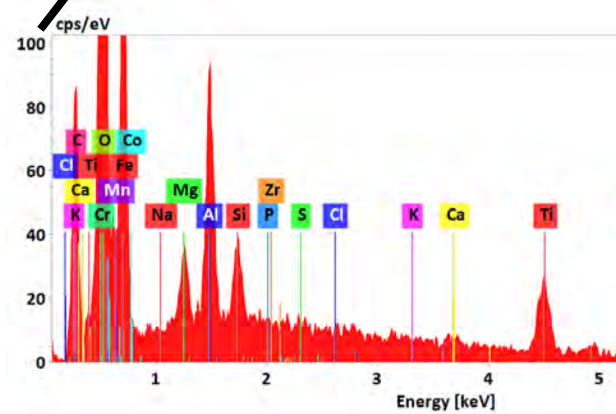
SEM-EDS: Hypermap Results

Each pixel can be quantified



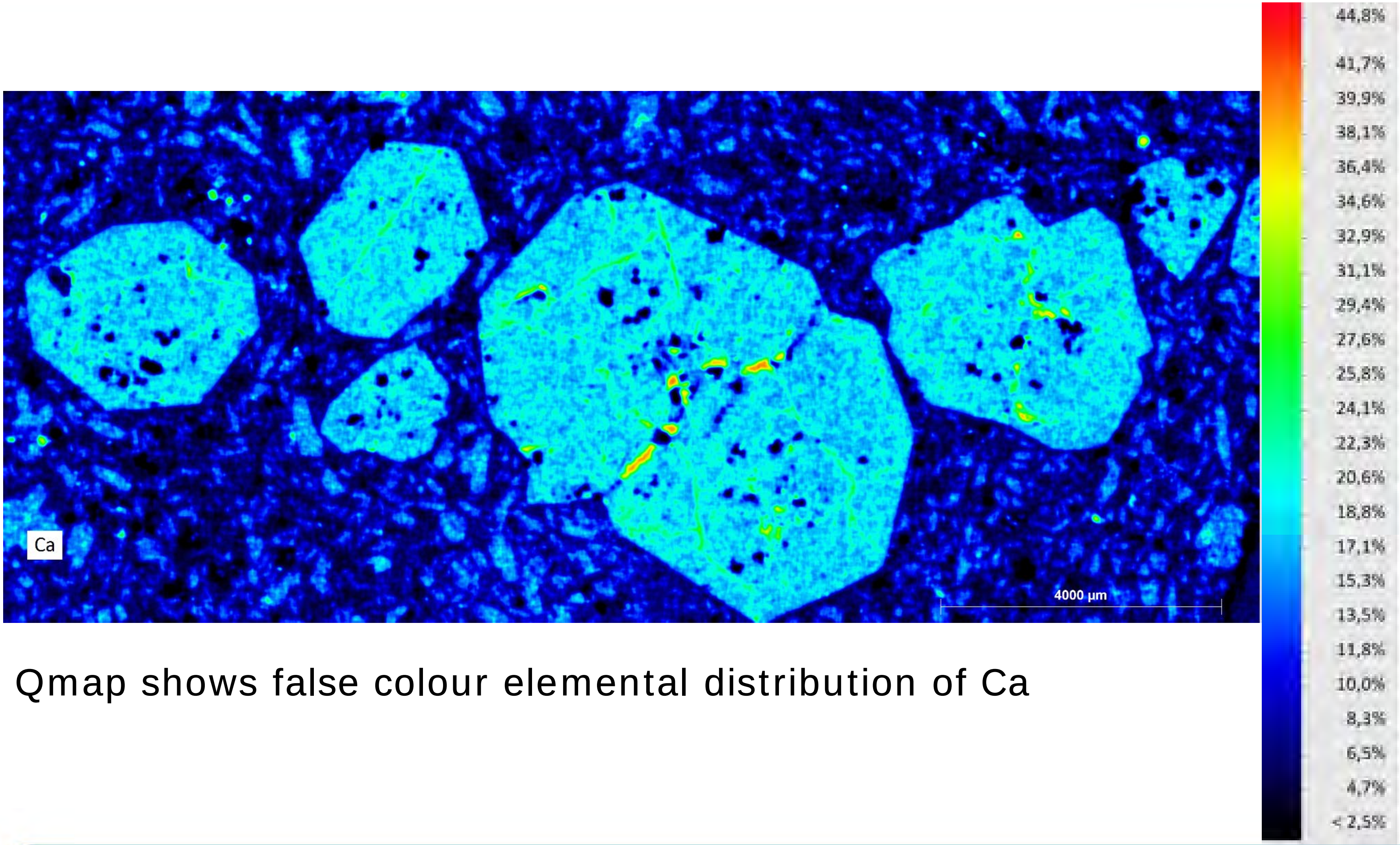


Comp.	Sto. Norm. [%]
	0,00
	0,00
MgO	14,95
Al2O3	4,84
SiO2	50,24
CaO	19,68
FeO	10,28
	100,00



Comp.	Sto. Norm. [%]
	0,00
	0,00
MgO	2,13
Al2O3	7,37
SiO2	2,61
TiO2	10,61
FeO	77,29
	100,00

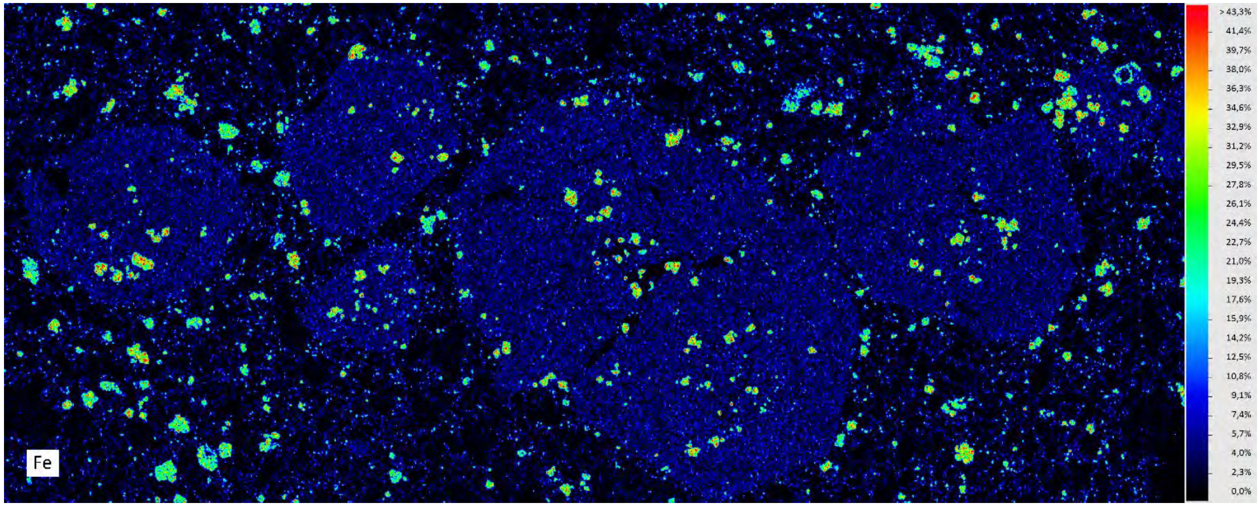
SEM-EDS: Hypermap Results
Quantified Map (Qmap)

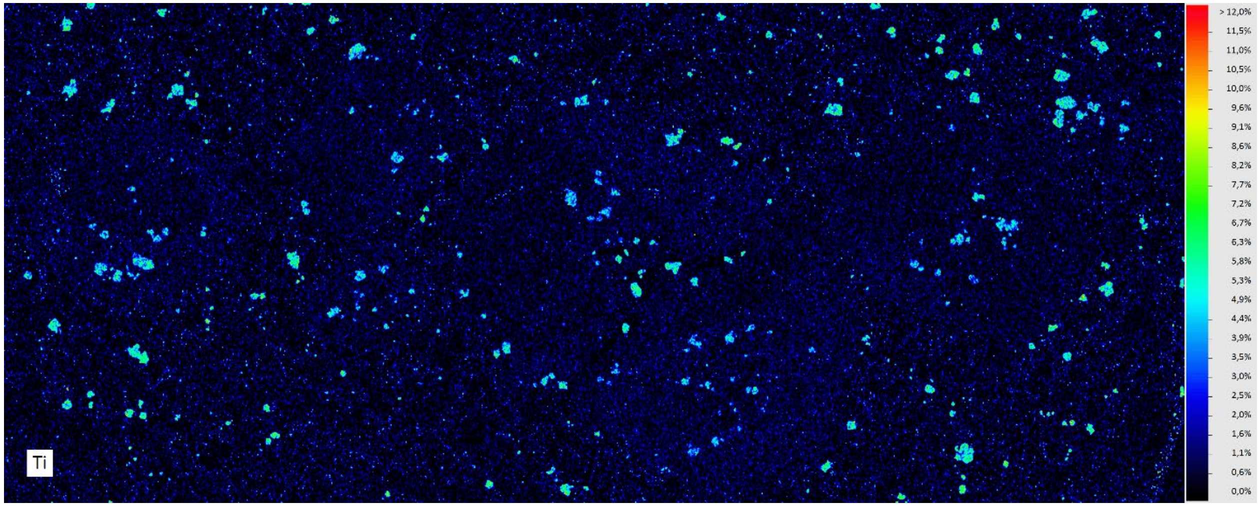


Qmap shows false colour elemental distribution of Ca

SEM-EDS: Hypermap Results

Quantified Map (Qmap)





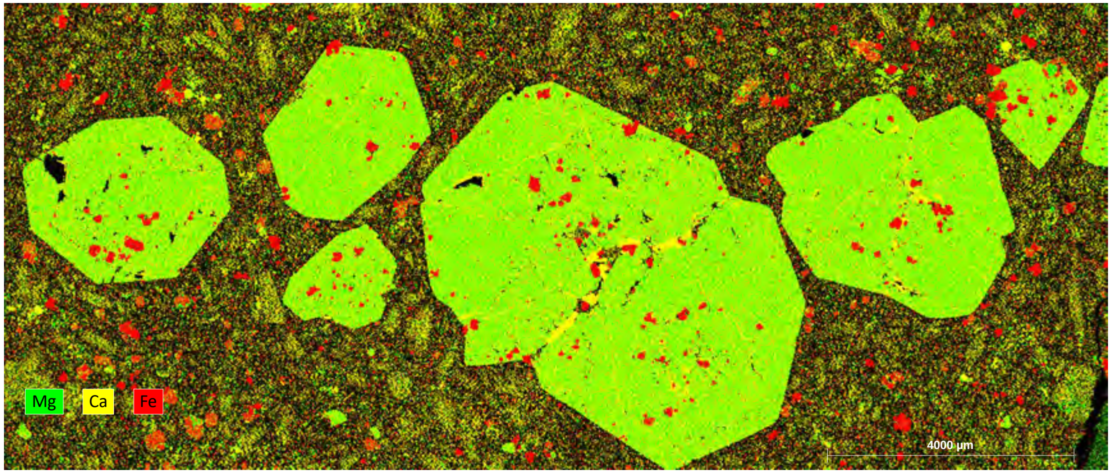
Qmap displays elemental mass% distribution over the entire sample

Top: Fe

Bottom: Ti

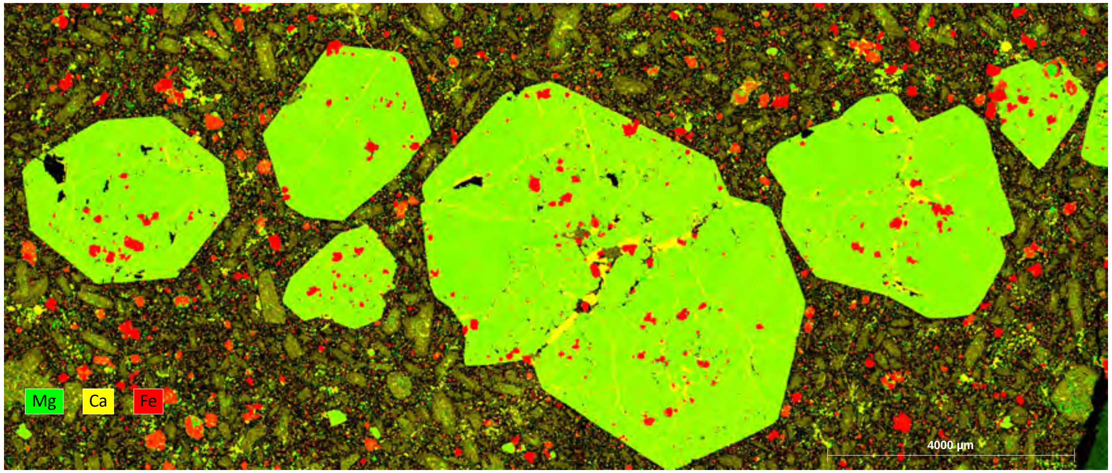
SEM-EDS: Hypermap Results

Elemental Peak Intensity Maps



Measurment Time: 77 min

SDD:	**10 mm²**
Dwell time:	64 µs
FOV:	17.8 mm
Pixel size:	4.1 µm
Fields:	308 (22x14)
Magnification:	250x

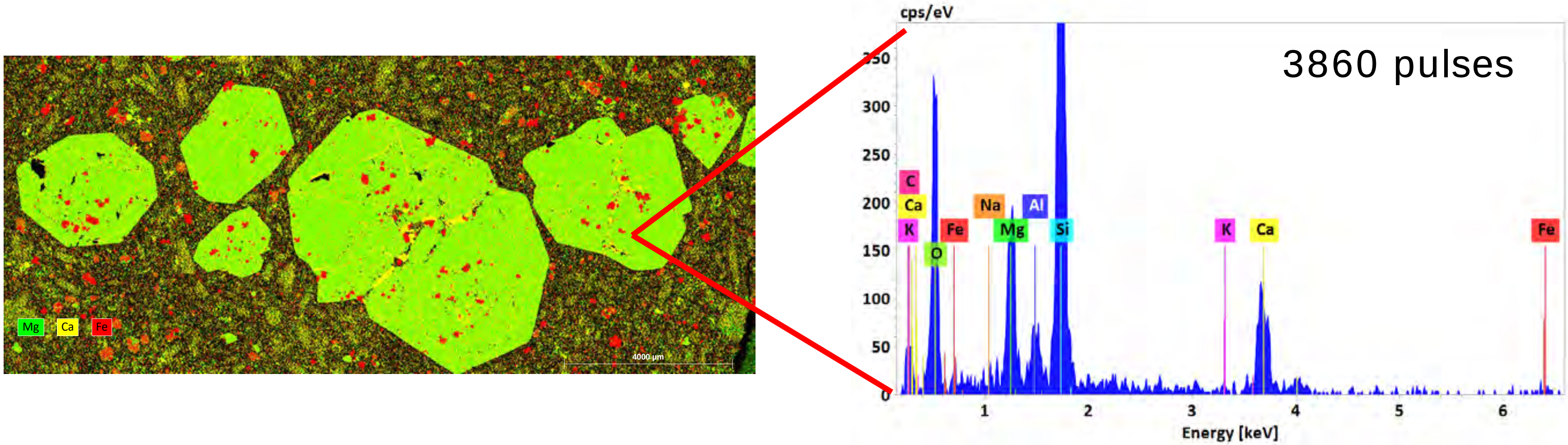


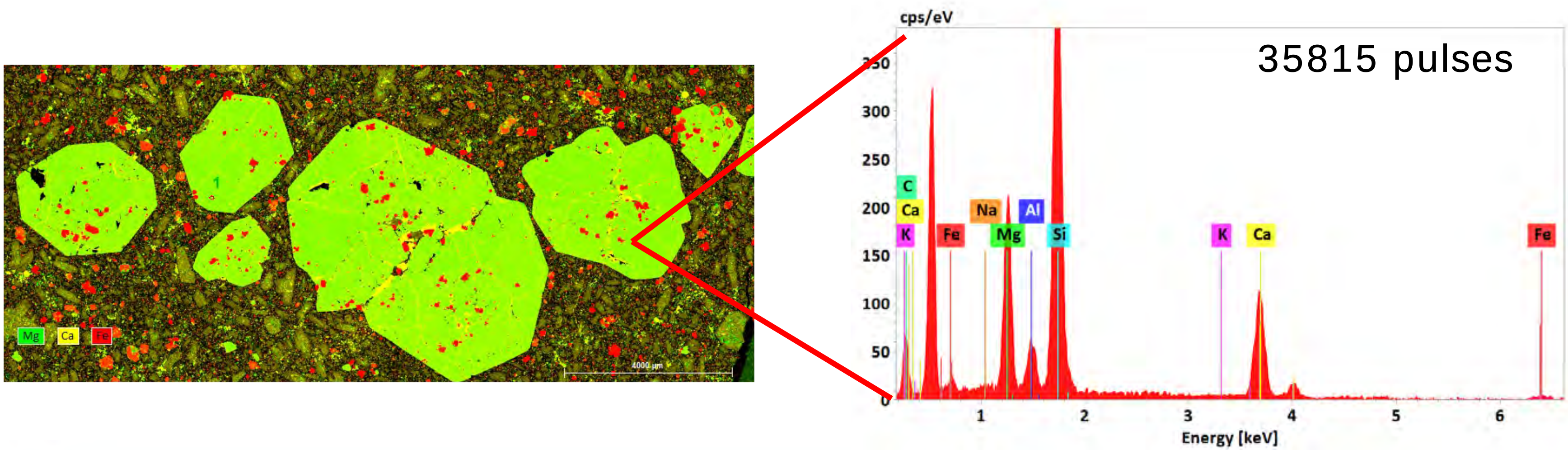
Measurment Time: 616 min

SDD:	**10 mm²**
Dwell time:	512 µs
FOV:	17.8 mm
Pixel size:	4.1 µm
Fields:	308 (22x14)
Magnification:	250x

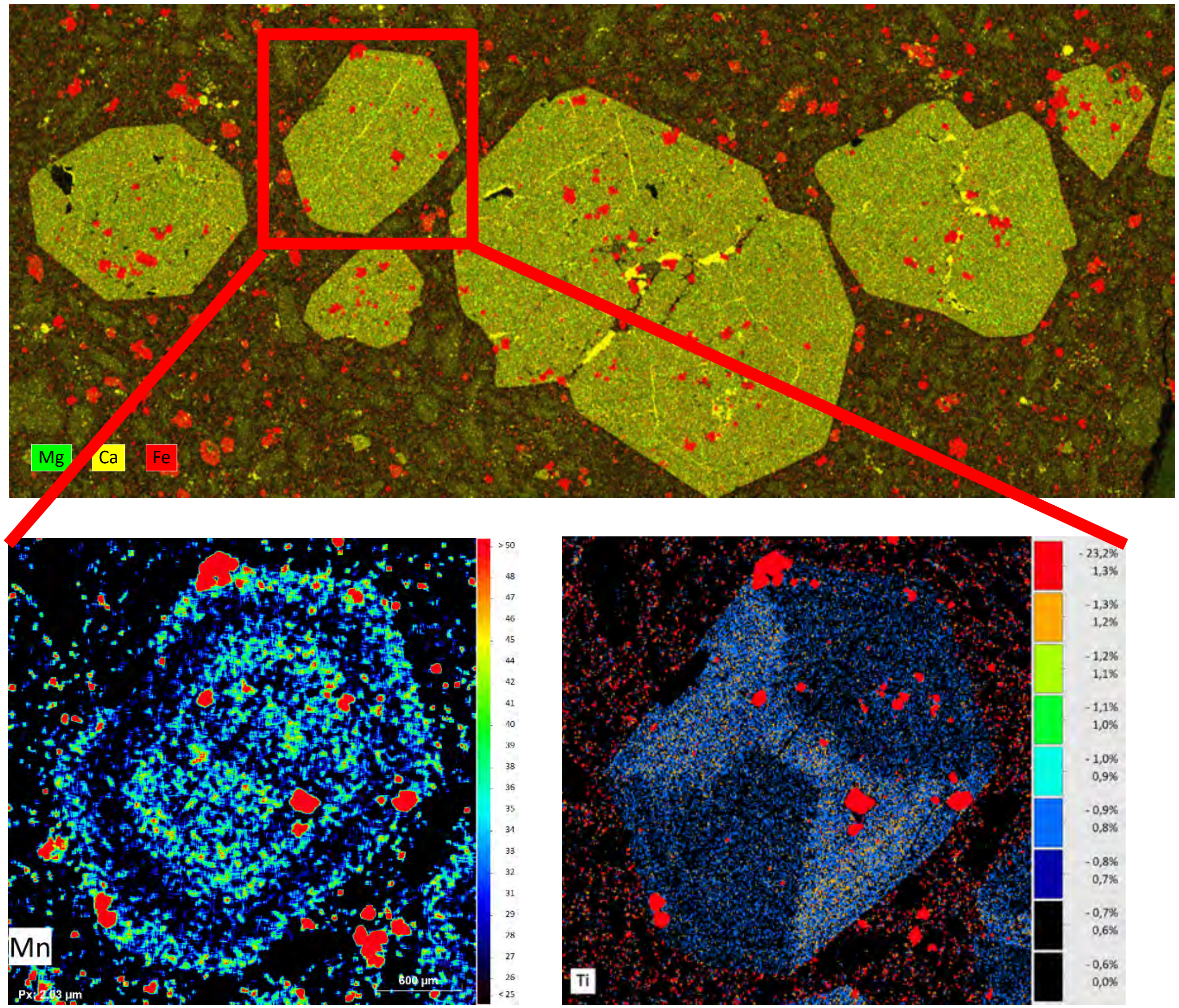
SEM-EDS: Hypermap Results

Elemental Peak Intensity Maps





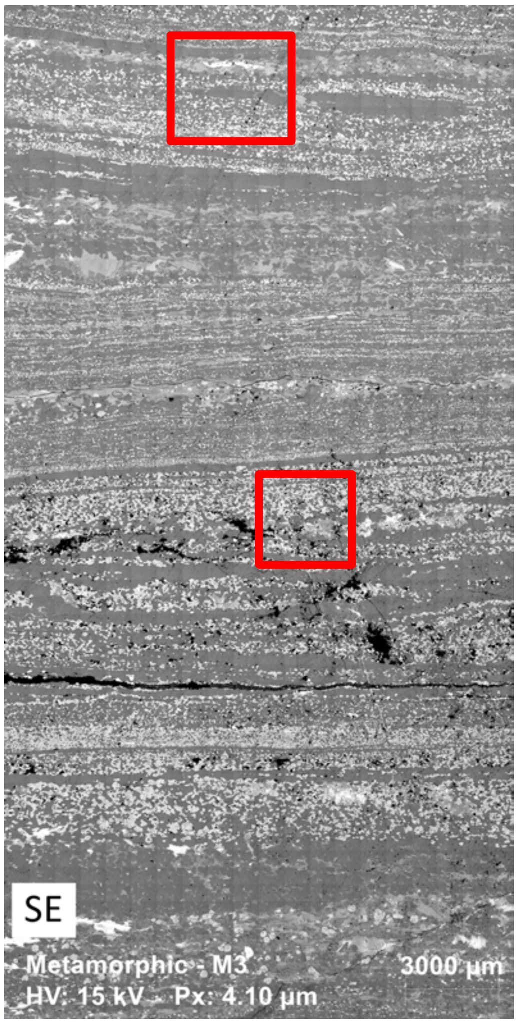
SEM-EDS: Hypermap Results
Elemental Peak Intensity Maps

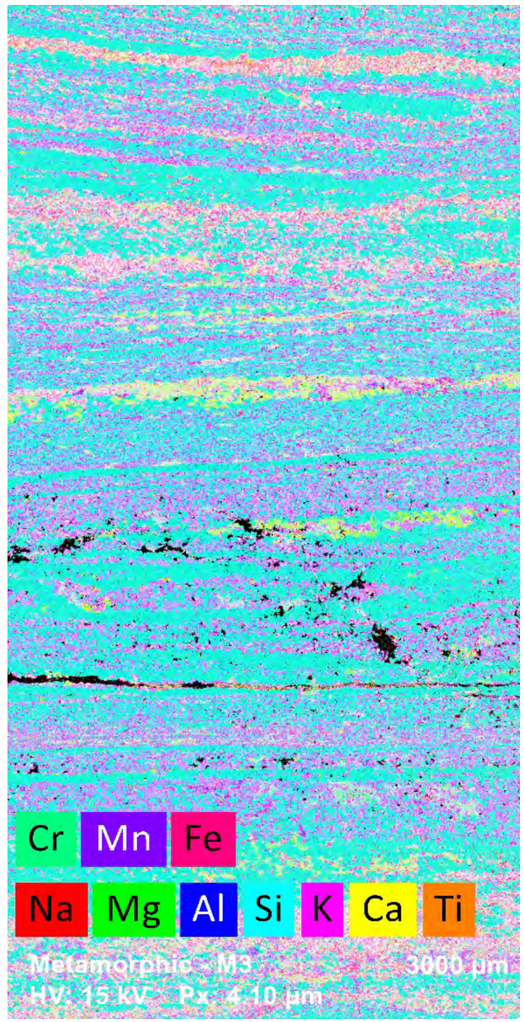


Zonation of Mn and Ti are visible in the pyroxene grains using a longer measurment times

SEM-EDS: Hypermap Results

Image Extension

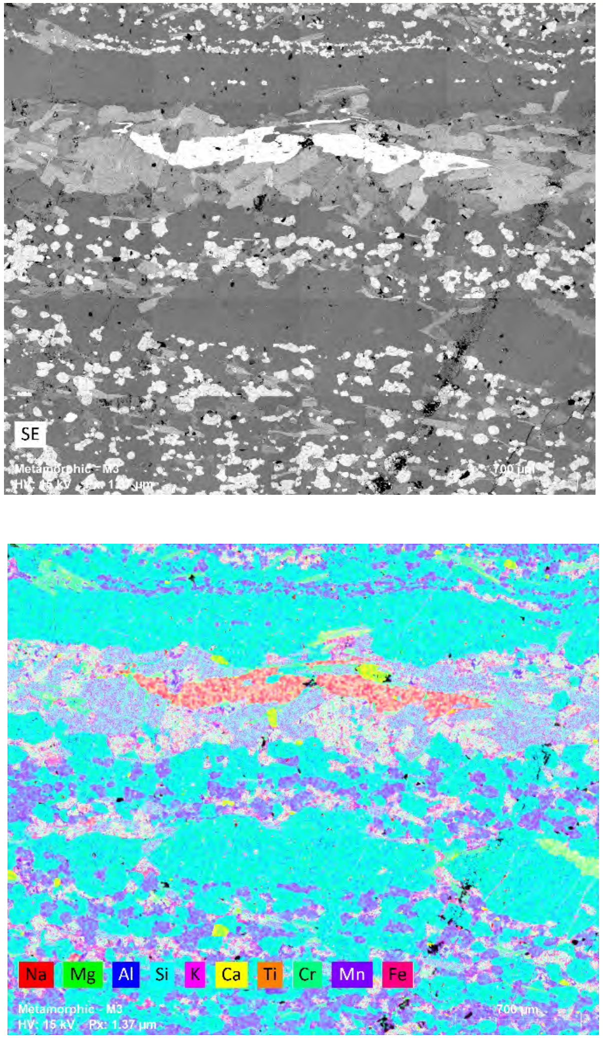


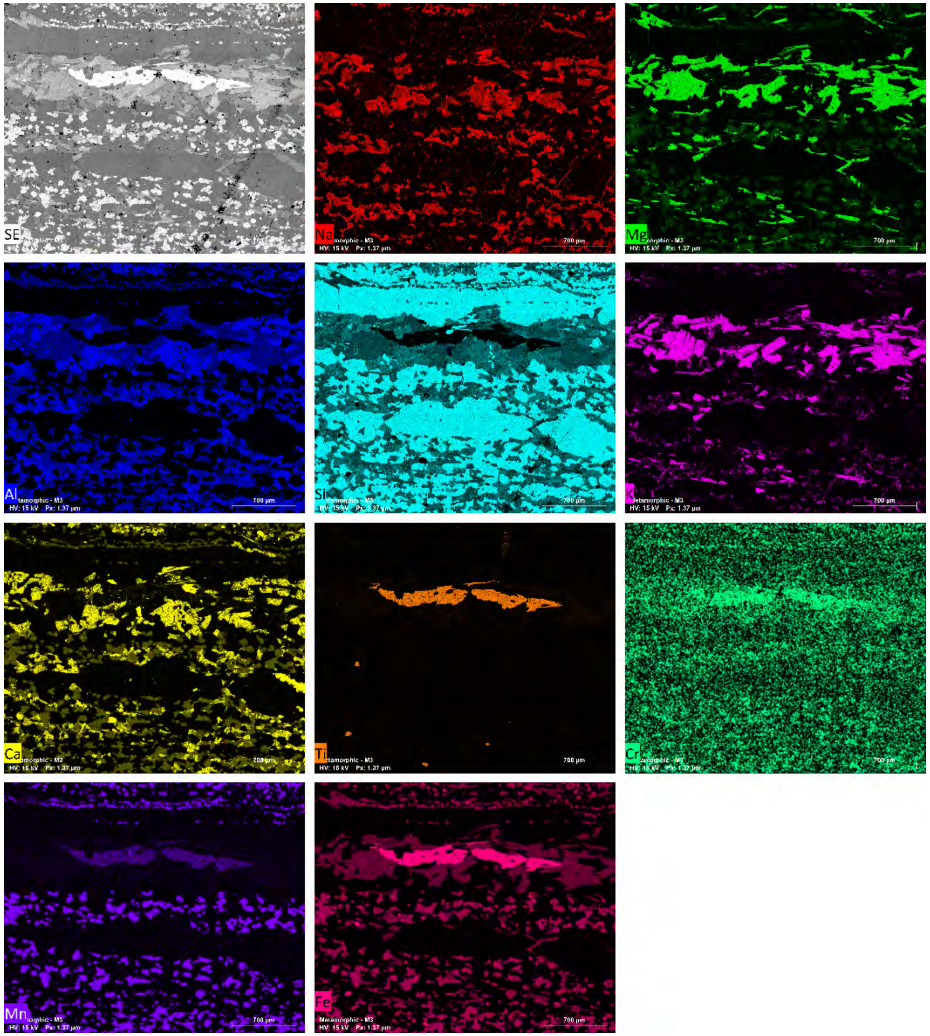


Measurement Conditions

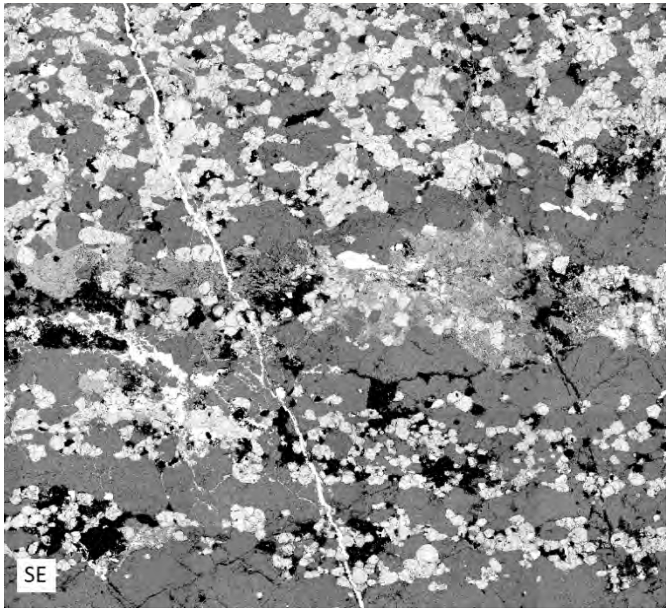
High Voltage:	15 kV
Pixels:	4000x7980
Measurment Time:	545 min
SDD:	10 mm^2
Dwell time:	1024 µs
FOV:	16 mm
Pixel size:	4 µm
Fields:	1200 (20x60)
Magnification:	250x

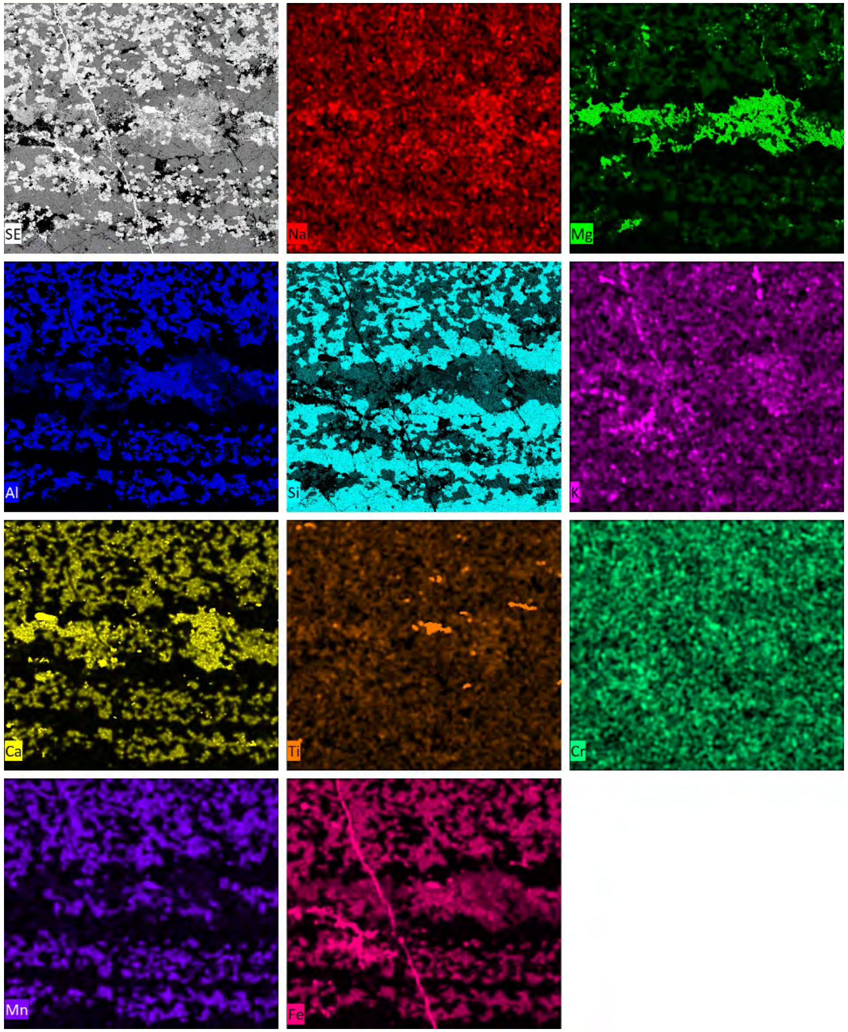
SEM-EDS: Hypermap Results
Elemental Peak Intensity Maps





SEM-EDS: Hypermap Results
Elemental Peak Intensity Maps



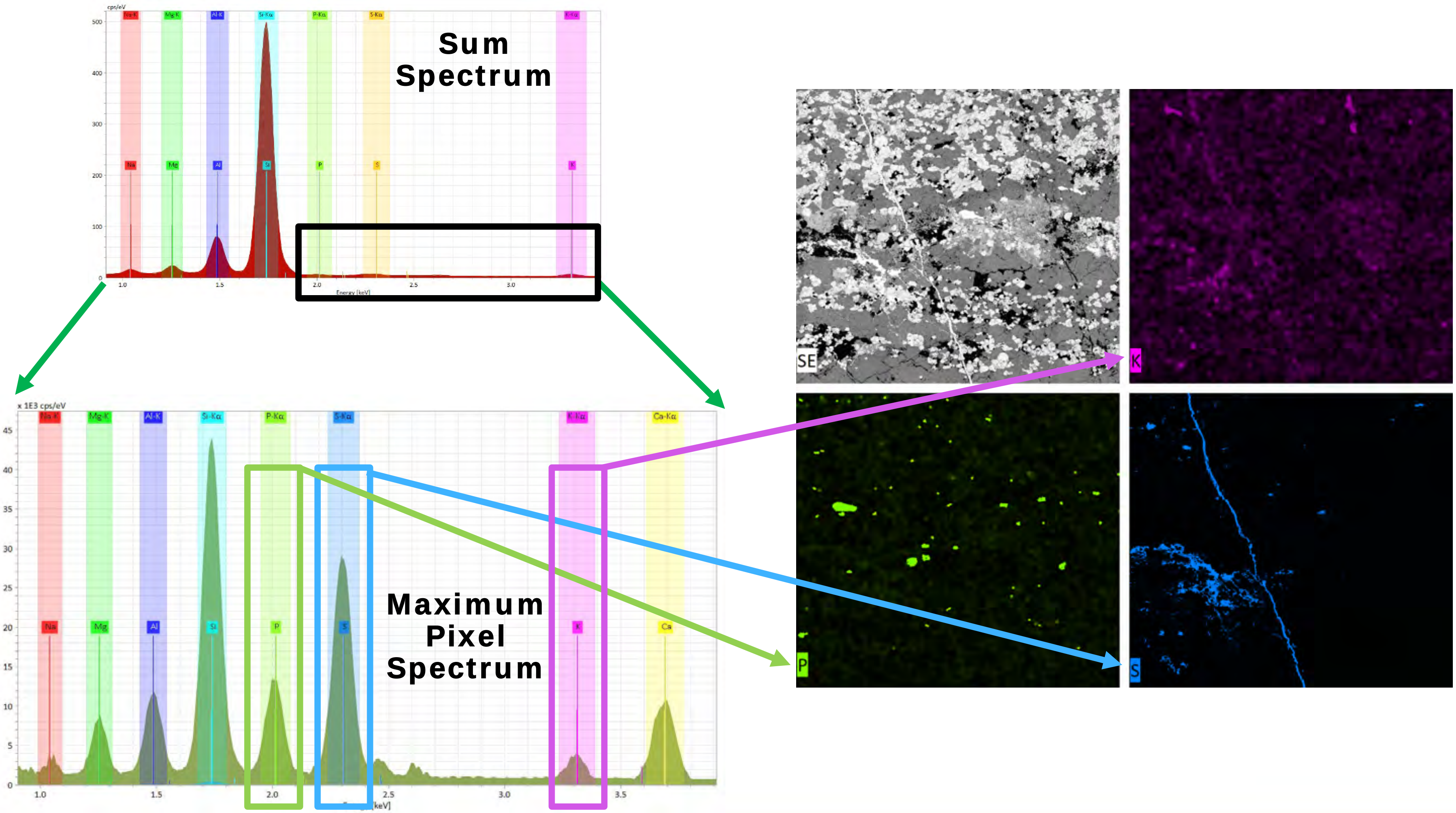


Measurement Conditions

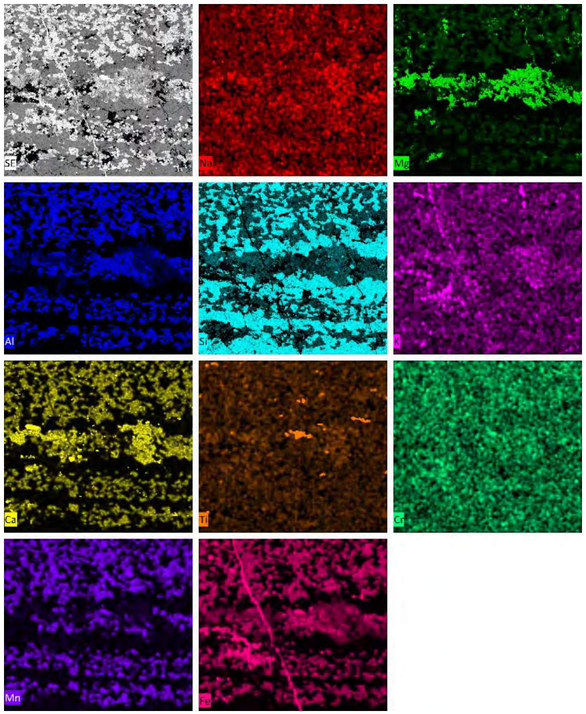
High Voltage:	15 kV
Pixels:	4000x7980
Measurment Time:	545 min
SDD:	10 mm^2
Dwell time:	1024 µs
FOV:	16 mm
Pixel size:	4 µm
Fields:	1200 (20x60)
Magnification:	250x

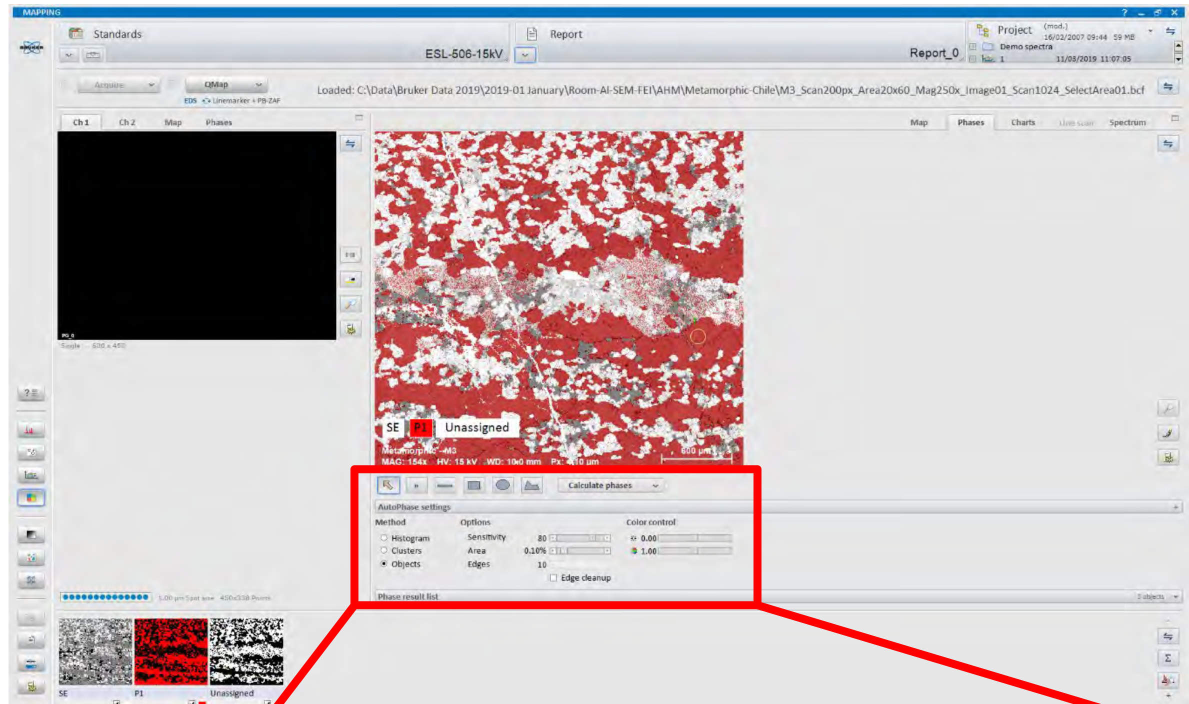
SEM-EDS: Hypermap Results

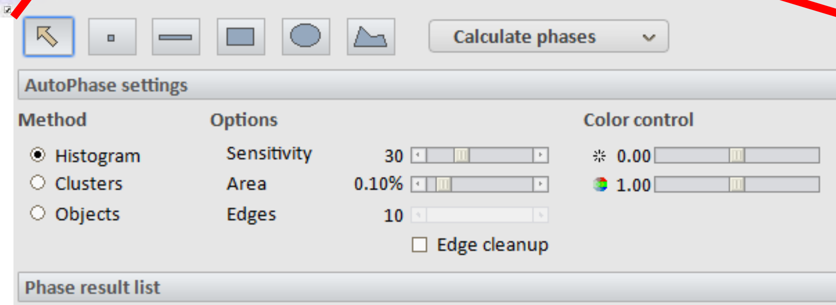
Minor Elements – Maximum Pixel Spectrum



SEM-EDS: Hypermap Results
Autophase

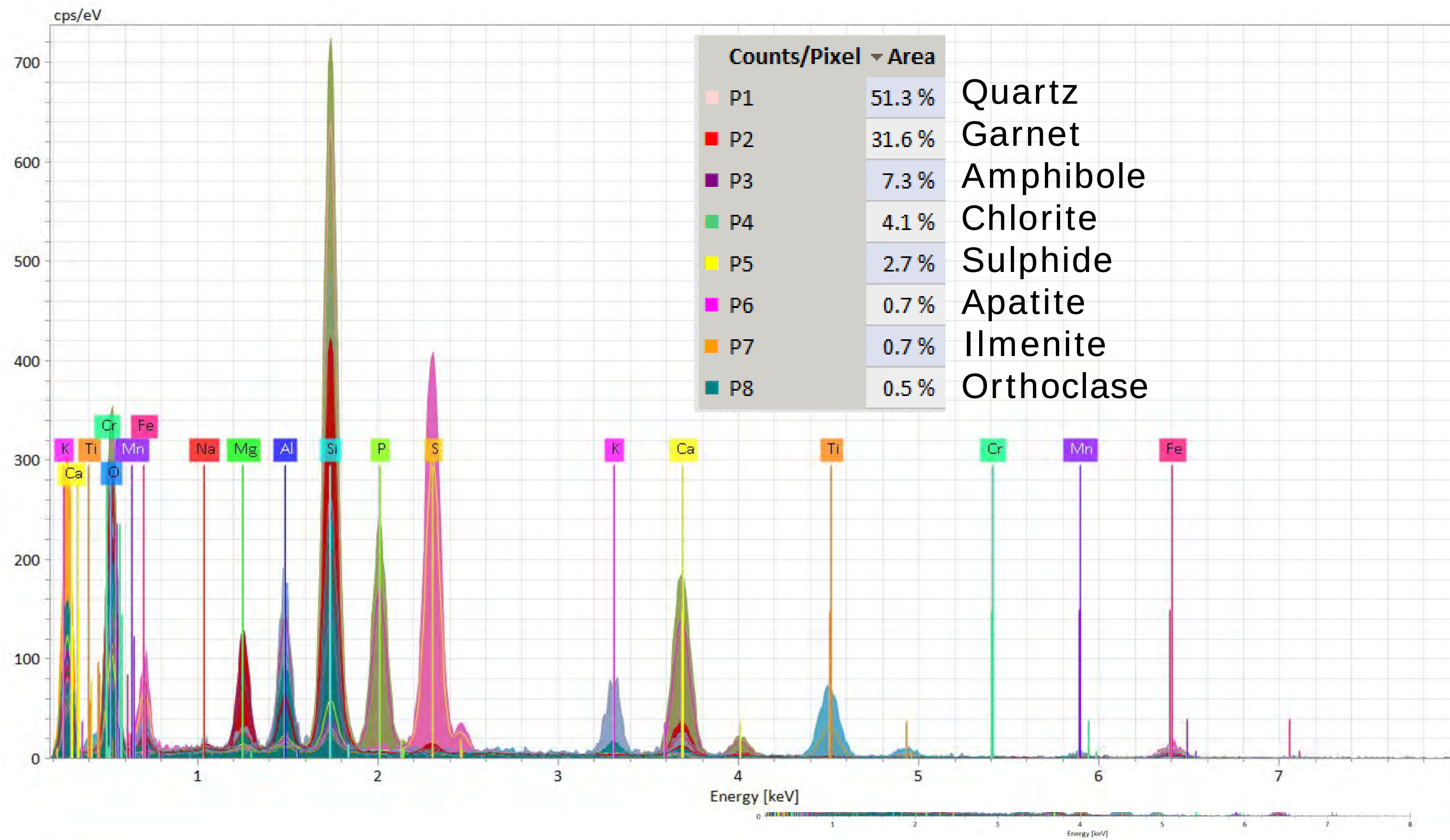






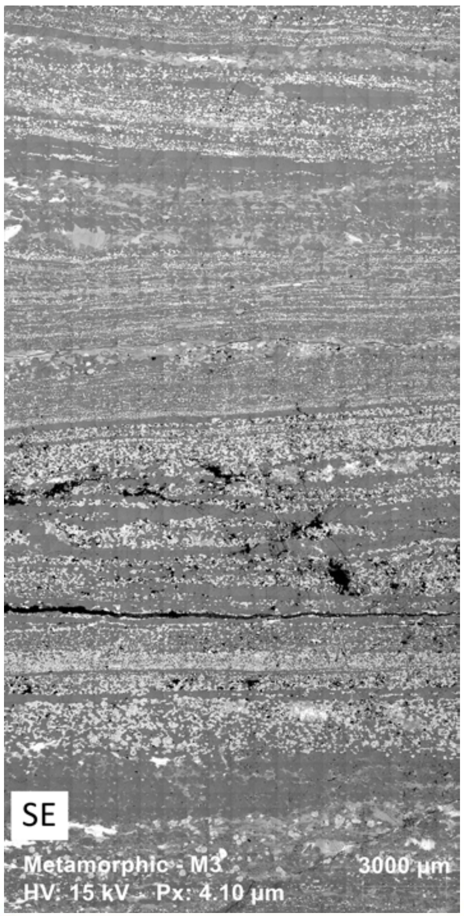
- Automatic and user-controlled phase analysis options
- Phase ratio and compositional information

SEM-EDS: Hypermap Results
Autophase

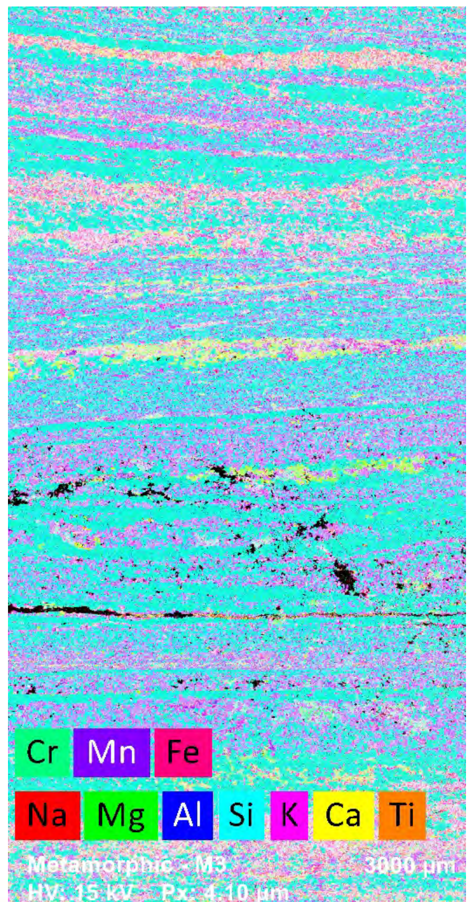


SEM-EDS: Hypermap Results
Autophase

Hypermap

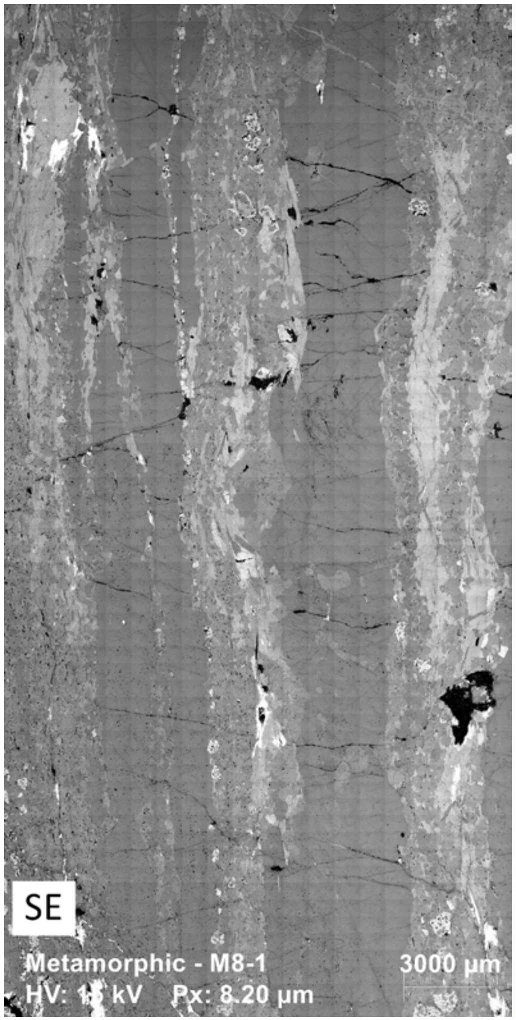


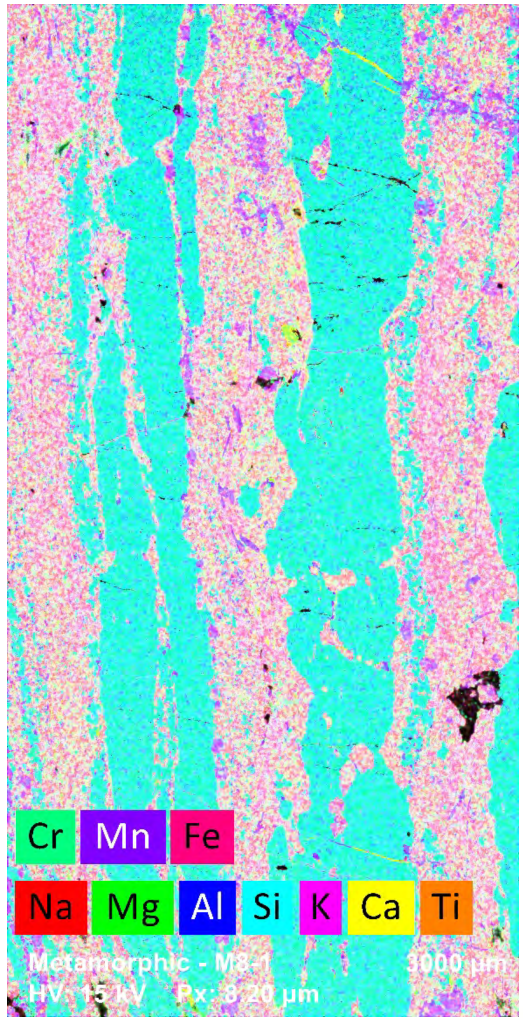
Elemental map



Phasemap

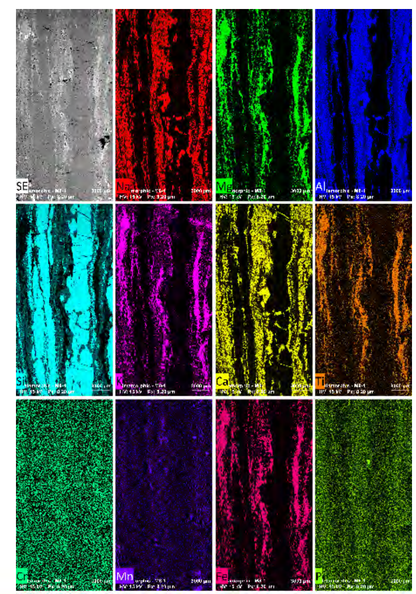
SEM-EDS: Hypermap Results
Image Extension





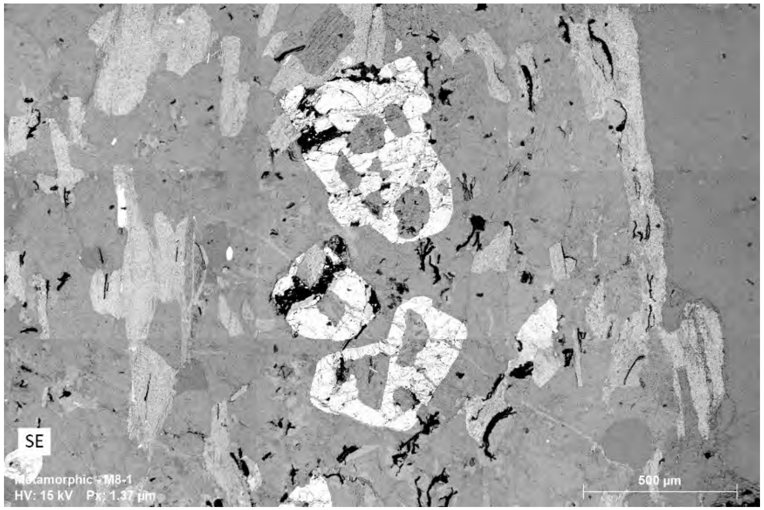
Measurement Conditions

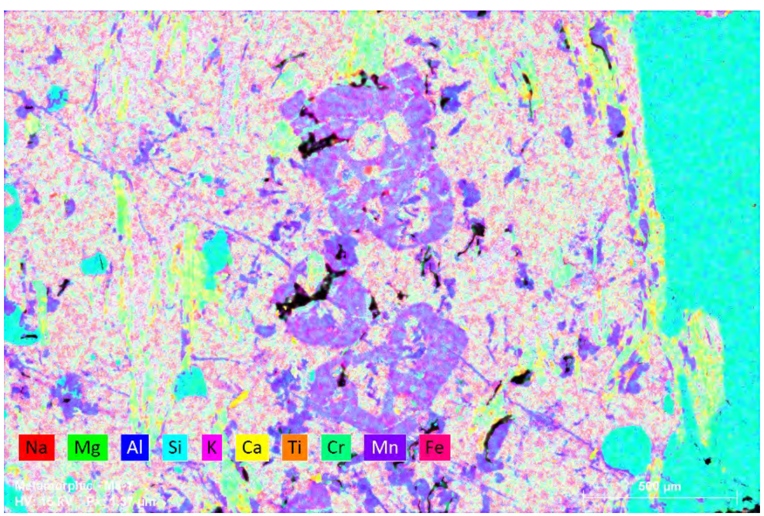
High Voltage:	15 kV
Pixels:	2000x3960
Measurment Time:	134 min
SDD:	10 mm^2
Dwell time:	512 µs
FOV:	16 mm
Pixel size:	8 µm
Fields:	1200 (20x60)
Magnification:	250x



SEM-EDS: Hypermap Results

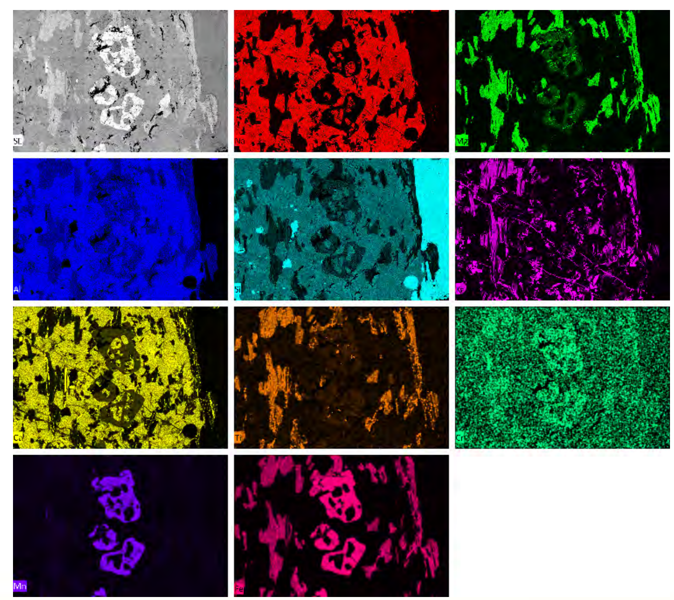
Image Extension



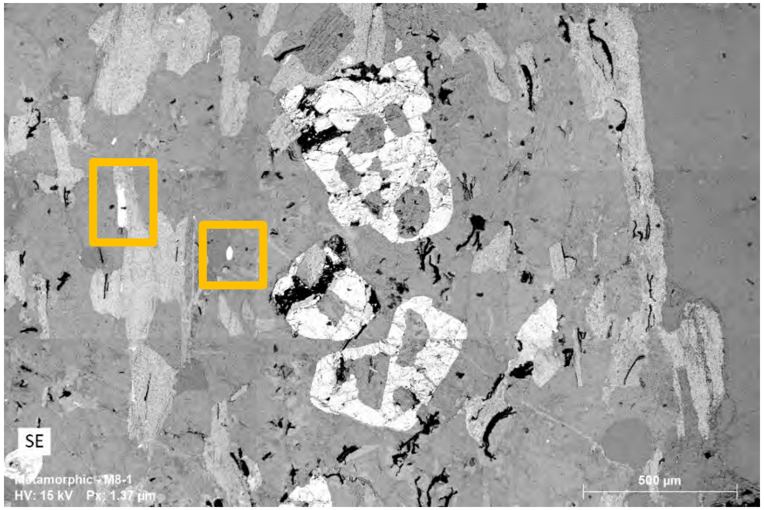


Measurement Conditions

High Voltage:	15 kV
Pixels:	1800x1200
Measurment Time:	10 min
SDD:	10 mm^2
Dwell time:	1024 µs
FOV:	2.4 mm
Pixel size:	1.4 µm
Fields:	9 (3x3)
Magnification:	250x



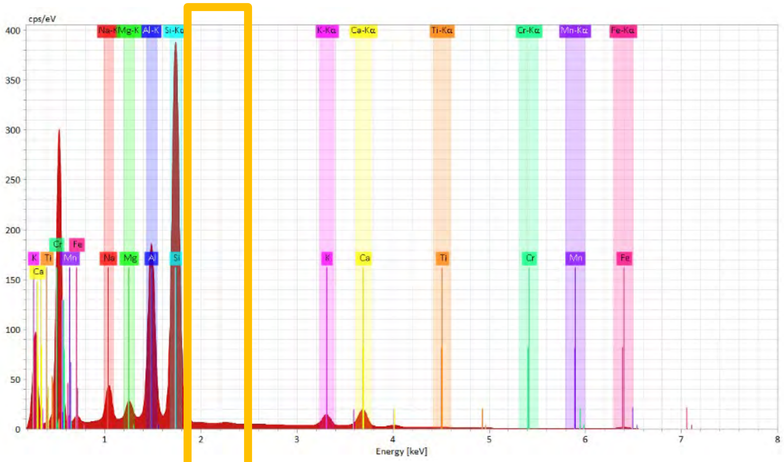
SEM-EDS: Hypermap Results
Maximum Pixel Spectrum

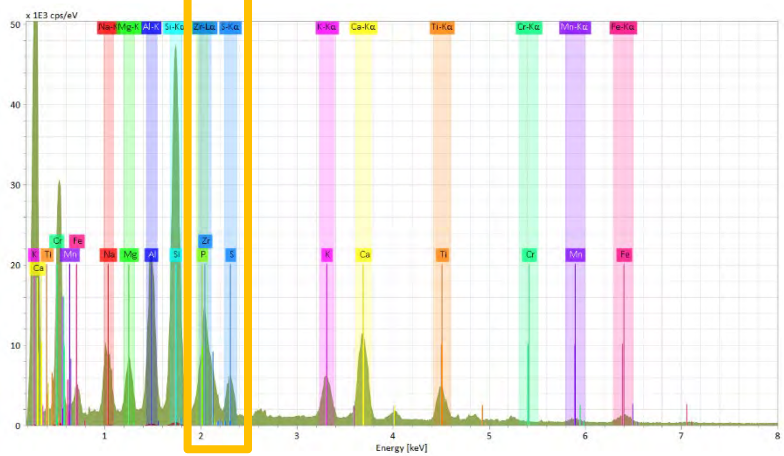


Measurement Conditions

High Voltage:	15 kV
Pixels:	1800x1200
Measurment Time:	10 min
SDD:	10 mm^2
Dwell time:	1024 µs
FOV:	2.4 mm
Pixel size:	1.4 µm
Fields:	9 (3x3)
Magnification:	250x

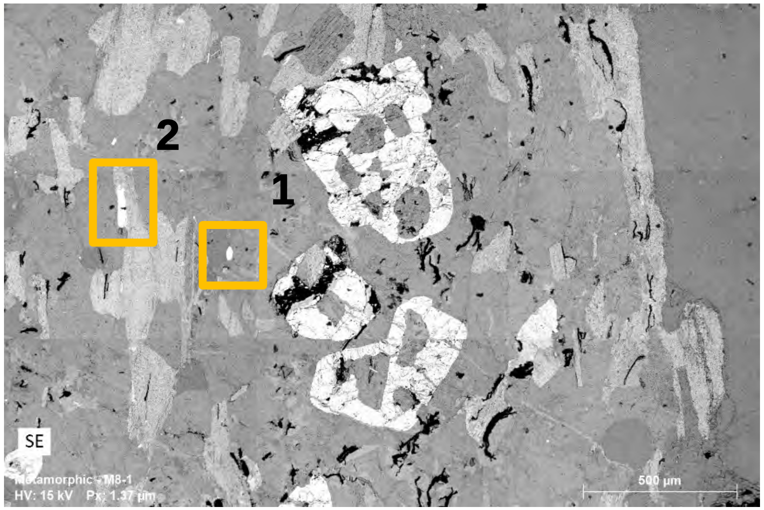
Sum Spectrum



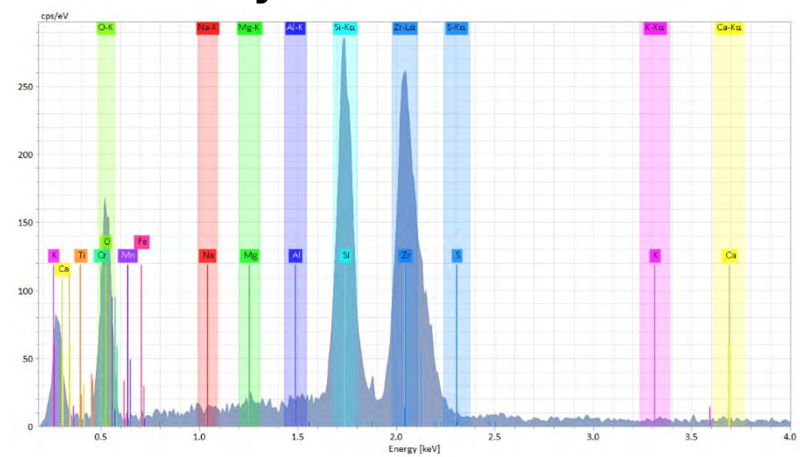


Maximum Pixel Spectrum

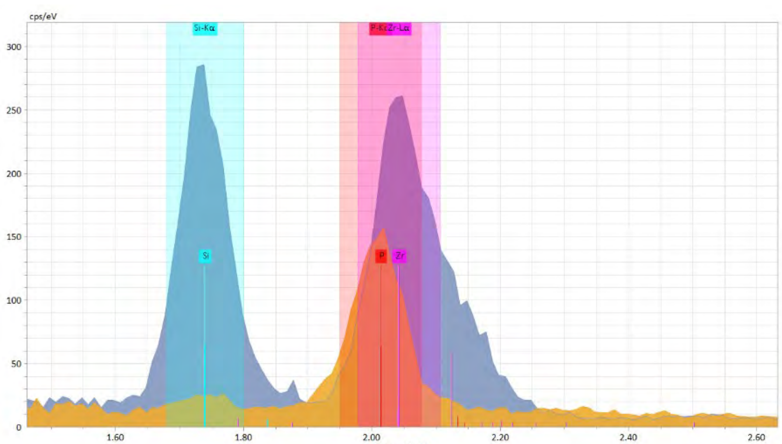
SEM-EDS: Hypermap Results
Maximum Pixel Spectrum

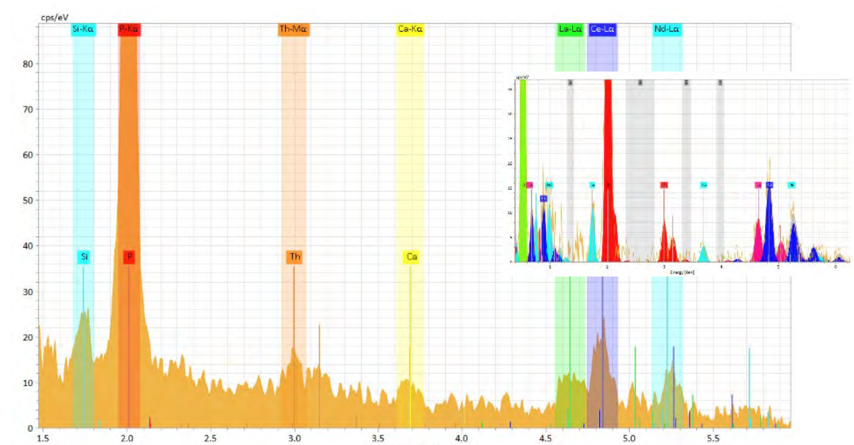


Object 1 - Zircon



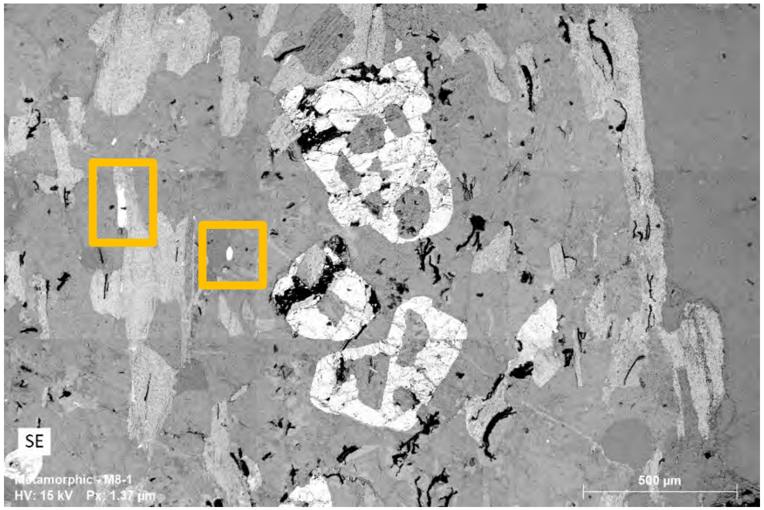
Deconvolution: Zr and P



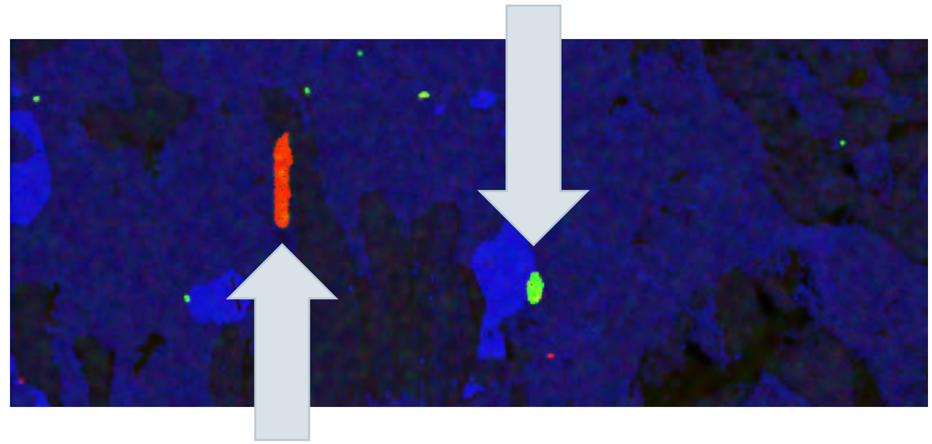


Object 2 - Monazite

SEM-EDS: Hypermap Results
Deconvolution

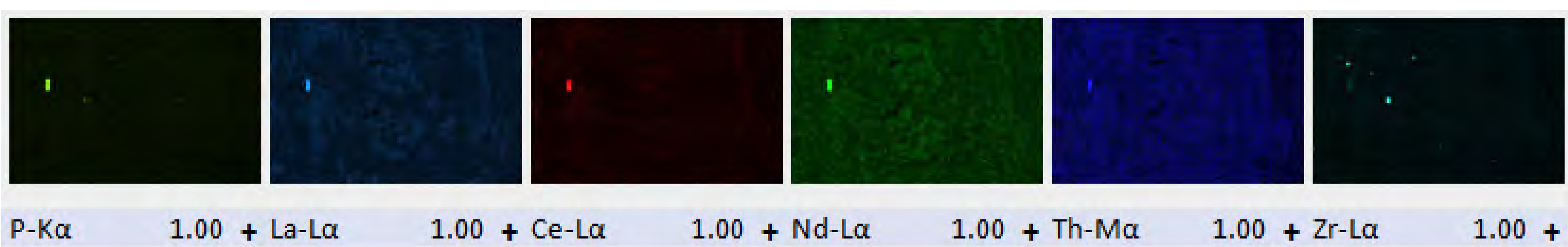


Object 1 - Zircon



Object 2 - Monazite

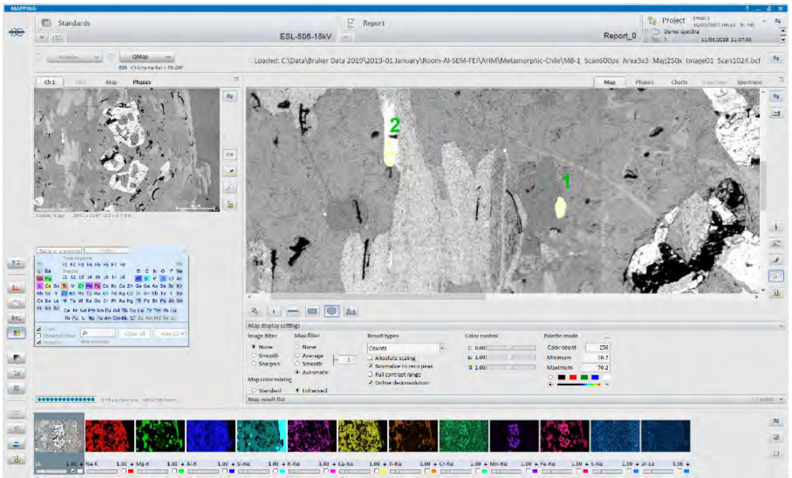
Deconvolution: Zr and P

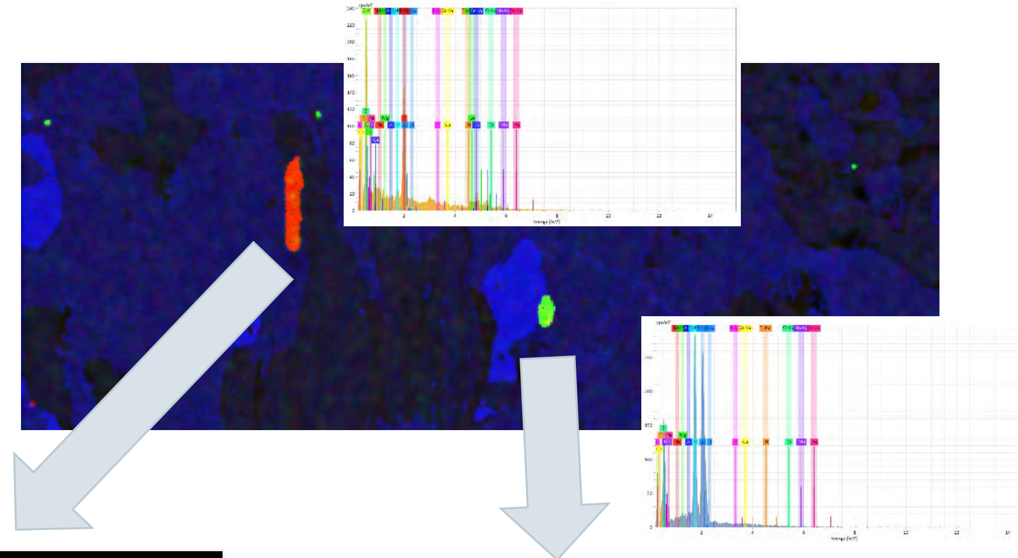


SEM-EDS: Hypermap Results

Quantification

BRUKER





Object 2 Monazite	Concentration (wt%)
P_2O_5	33.35
CaO	4.57
La_2O_3	16,61
Ce_2O_3	34.02
Nd_2O_3	5.61
ThO	5.85

Object 1 Zircon	Concentration (wt%)
SiO_2	31.94
ZrO_2	68.06

SEM-EDS: Hypermapping
Summary and conclusions

- Hypermapping is capable of capturing detailed high resolution data over large areas.
- The data can be postprocessed and investigated in a variety of forms with the Esprit software including point spectra, line scans, quantification, and autophase.
- There are a variety of parameters that are important in the set-up of the Hypermap. These will affect the analytical time taken and the quality of the information within the hypermap. Such parameters include: High Voltage, Beam Current, SDD Type, Dwell time, Pixel size, Magnification, Working Distance.
- Hypermaps and image extension are compatiable with SEM-EDS, but can also be used with other Bruker SEM products such as the microXRF and the WDS. In addition, the maps can be imported in AMICS, an automated mineralogy software with expanded mineralogical capability.

Micro XRF Webinar in near Future:

Exotic Cu Deposits

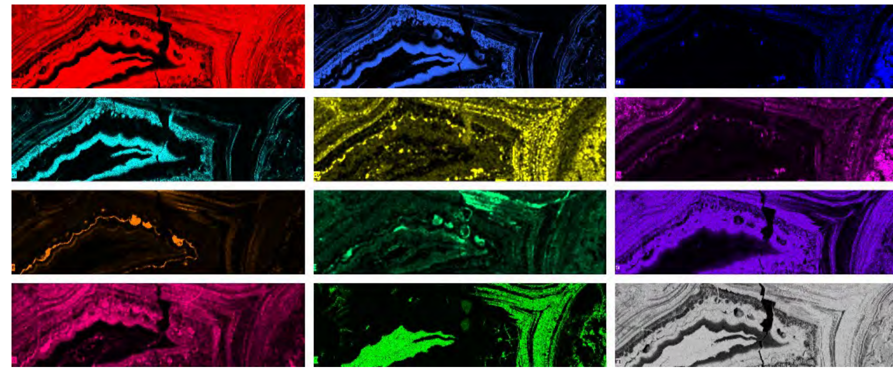
Large Area Map

Sample Size: Polished Section: 45 x 30 mm

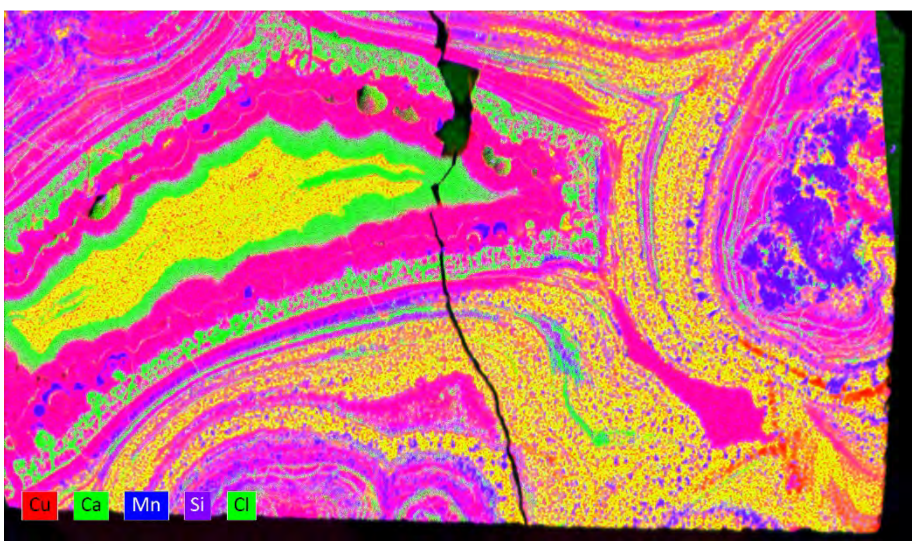
Sample from El Tesoro, Chile.
Clearly Defined Elemental and Mineralogical Phases
Can identify the presence of trace elements, in this case, Cobalt (Co), Manganese (Mn), Strontium (Sr)

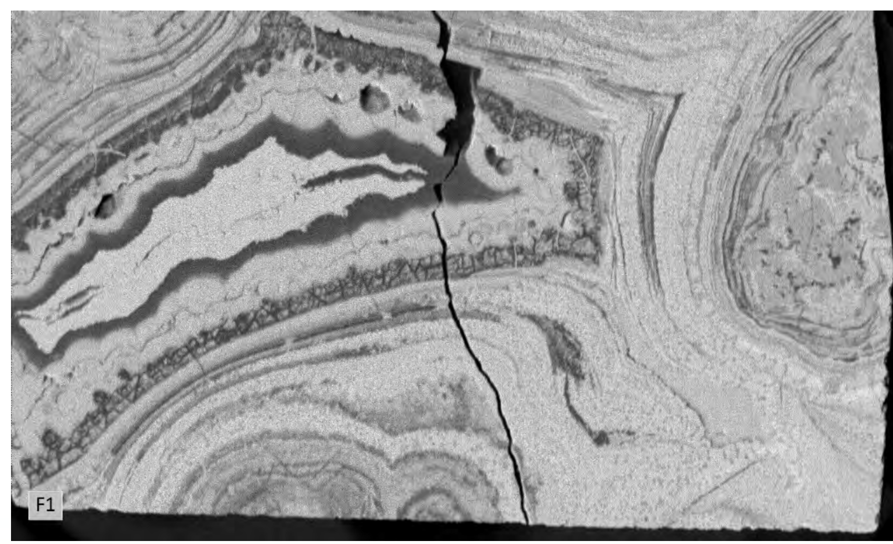
Analytical Parameters:

Tube Voltage: Rh at 50 kV
Anode Current: 600 uA
Pixel Spacing: 25 um
Analytical Time: 101 mins



Top: Elemental Maps; Bottom Left: Mixed Elemental Map; Bottom, Right: X-Ray Intensity Map.





Acknowledgements

GeoZentrum Nordbayern

IfP

BRUKER

Dr. Tobias Salge

Dr. Eduardo Campos

Dr. Ivan Soto

Sebastian Eade

Javiera Errazuriz

Mauricio Galarce

Victor Hernández

Sebastian Sola

Patricia Muñoz

Rocio Ibaceta

UNIVERSIDAD CATOLICA DEL NORTE CHILE

UNIVERSIDAD CATOLICA DEL NORTE GEOLOGIA

maini

Unidad de Equipamiento Científico

Are There Any Questions?

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

More Information

For more information, please contact us:

info.bna@bruker.com

Innovation with Integrity