

Microanalysis: SEM-EDS

Large Area High Resolution Maps of Geological Samples



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

This complex graphic features a central image of the Bruker XFlash 6110 detector, a cylindrical device with a long probe and a multi-fingered silicon drift chamber. The background is a blue gradient with several elements: a periodic table of elements, a large "EDS" text, the "XFlash® Technology" logo, a spectral plot with peaks labeled "Cu", "Ni", "Mn", and "Fe", and two high-resolution maps of a geological sample. The maps show detailed elemental distribution in a cross-section of a material. Various chemical symbols like Na, Mg, K, Ca, Sc, Ti, Rb, Sr, Y, Zr, Cs, Ba, La, Hf, Fr, Ra, Ac, and others are scattered across the background, along with labels for X-ray lines such as K α , K β , L α , L β , M α , and M β .

Presenters



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Application Scientist
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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



A composite image illustrating SEM-EDS technology. On the left, a Bruker XFlash 6110 detector is shown, a cylindrical metal probe with a long, thin tip. The background is a blue-tinted collage. It includes a periodic table of elements, with various elements highlighted in different colors. Text labels like "Kα", "Kβ", "Lα", "Lβ", "Mα", and "Mβ" are scattered around the table. In the center, the text "EDS" is written in large, light blue letters, with "XFlash® Technology" below it. At the bottom, there is a spectral graph showing several sharp peaks of varying heights. On the right side, there are two grayscale images: the top one shows a cross-section of a material with a crack, and the bottom one shows a textured surface.

Bruker SEM Analyzers

Our "evolving eyes"



SEM-EDS: Overview Software – Esprit 2.1



SEM EDS

Microscope: HV 15.0 kV, Magn: 200.0 x, Stage: 0.000 mm, Stage Y: 0.000 mm, Stage Z: 0.000 mm, X-ray source: 10 kV, Max throughput: 180 kcps, Temp: 19.2 °C, Load time: 1 s, Last time: 1 s, Size: 1200 px, ICR 182 kcps, Energy: 0 V, Size: 80 px

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

Ch 1 Ch 2

cps/eV

Energy [keV]

Results [Mass-%(norm.)]	Sort: Element
EDS Bruker 4 6.68 C 7.64 O 46.14 Na 6.98 Al 5.16 Si 30.15	
EDS Bruker 1 4.07 C 3.62 O 37.99 Ti 58.39	
EDS Bruker 2 8.72 C 6.18 O 33.99 Si 17.73 Ti 13.12 Ba 28.98	
EDS Bruker 3 13.00 C 6.09 O 34.27 Si 19.00 Ti 12.94 Ba 27.69	

Report preview

Page 1 Page 2

ESPRIT for SEM-EDS

Spectra, Project Management and Report



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

- ? [Menu]
- [Spectra Icon]
- [Objects Icon]
- [Line scan Icon]
- [Mapping Icon]
- [Imaging Icon]
- [Feature Icon]
- [EBSD Icon]
- [Jobs Icon]
- [Scripting Icon]
- [XMethod Icon]
- [System Icon]

Sample: Test sample
Standards: 0 Standards
Microscope: 15.0 kV
X-ray tube: 0.0 kV
Scan: 600 px
EDS 1: 100 kcps
EDS 2: 0 cps
EBSD: 80 px

Preview
Acquire
Quantify
EDS Interactive Oxides
EDS Interactive Oxides
Export

Spectra

Project

10.02.2011 09:42:36

- ID+QUANT: 2 Steel Samples
 - B 3 26.01.2009 16:15:08 20.0 keV Fe NiCr
 - B 2 26.01.2009 15:55:07 20.0 keV
 - B 1 26.01.2009 15:35:07 20.0 keV
 - A 3 26.01.2009 17:49:35 20.0 keV
 - A 2 26.01.2009 17:29:34 20.0 keV Fe NiCr
 - A 1 26.01.2009 17:09:34 20.0 keV Fe NiCr
- ID: 12 Minerals
- ID+QUANT: 1 Alloy
 - LV-2010-60s-25kV 3 15.02.2010 17:45:01 25.0 keV C
 - Test sample_1 19.09.2013 15:45:14 15.0 keV O Na Al Si Ca C
- Mapping
 - Test sample 19 20.10.2013 20:55:42 15.0 keV 300 X 600x449
 - Test sample 19 20.10.2013 20:55:42 15.0 keV 300 X 600x449
 - Test sample 0 20.10.2013 20:55:49 15.0 keV 300 X 450x337
 - Map 20.10.2013 20:55:48 15.0 keV Fe Ca Mg Al Si C O Na Cu

Report

	cps/eV	Results [Atom-% (norm.)]	Sort: Value
EDS Test sample_1	1,13	O 60,59 Si 15,90 Mg 9,19 Fe 5,93 Al 3,67 Na 1,5	
EDS Kupfer-20kV.spx	0,03	No quant results available.	
EDS Kupfer-30kV.spx	0,05	No quant results available.	
EDS Kupfer-25kV.spx	0,04	No quant results available.	
EDS Kupfer-15kV.spx	0,04	No quant results available.	
EDS Kupfer-10kV.spx	0,01	No quant results available.	

ESPRIT for SEM-EDS Workspaces



Assistants

Spectra

Objects

Line scan

Mapping

Imaging

Feature

EBS

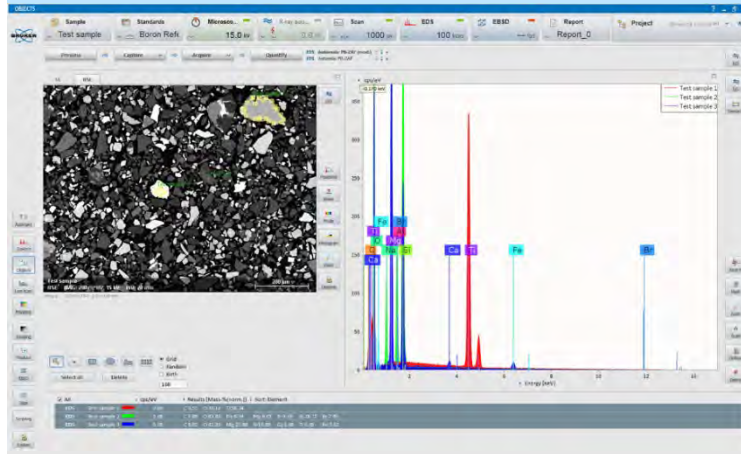
Jobs

Scripting

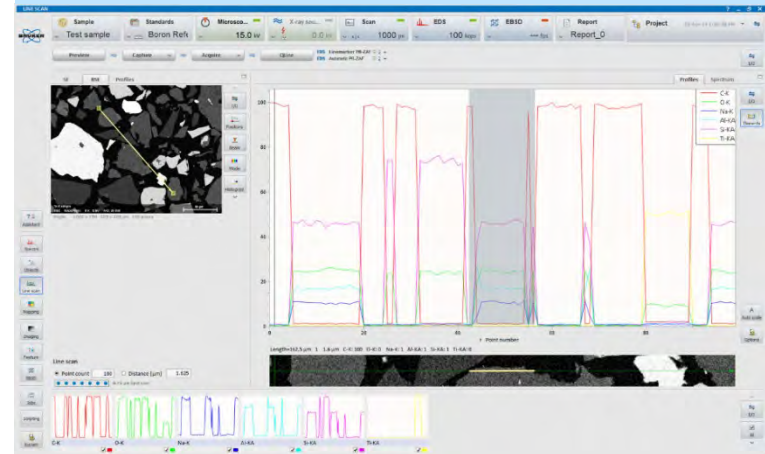
XMethod

System

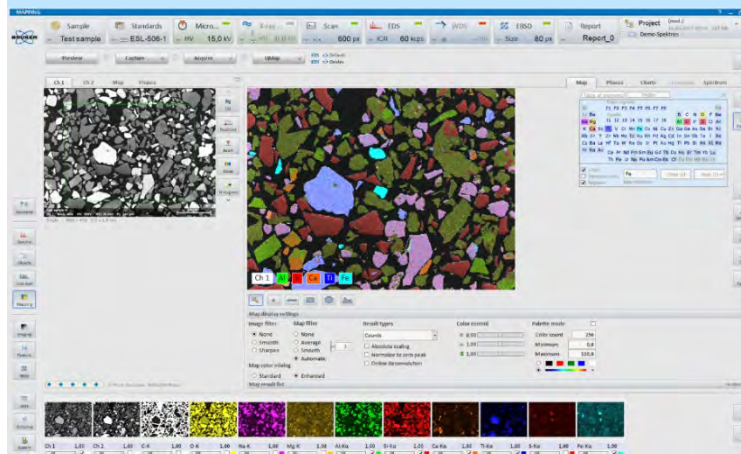
Objects



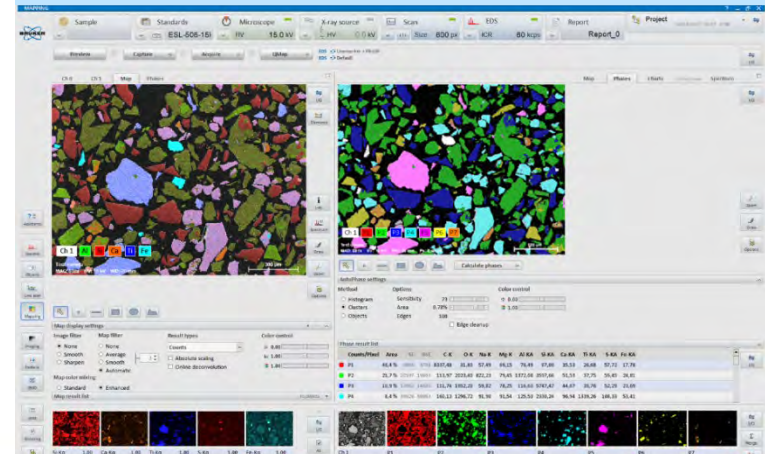
Line Scan



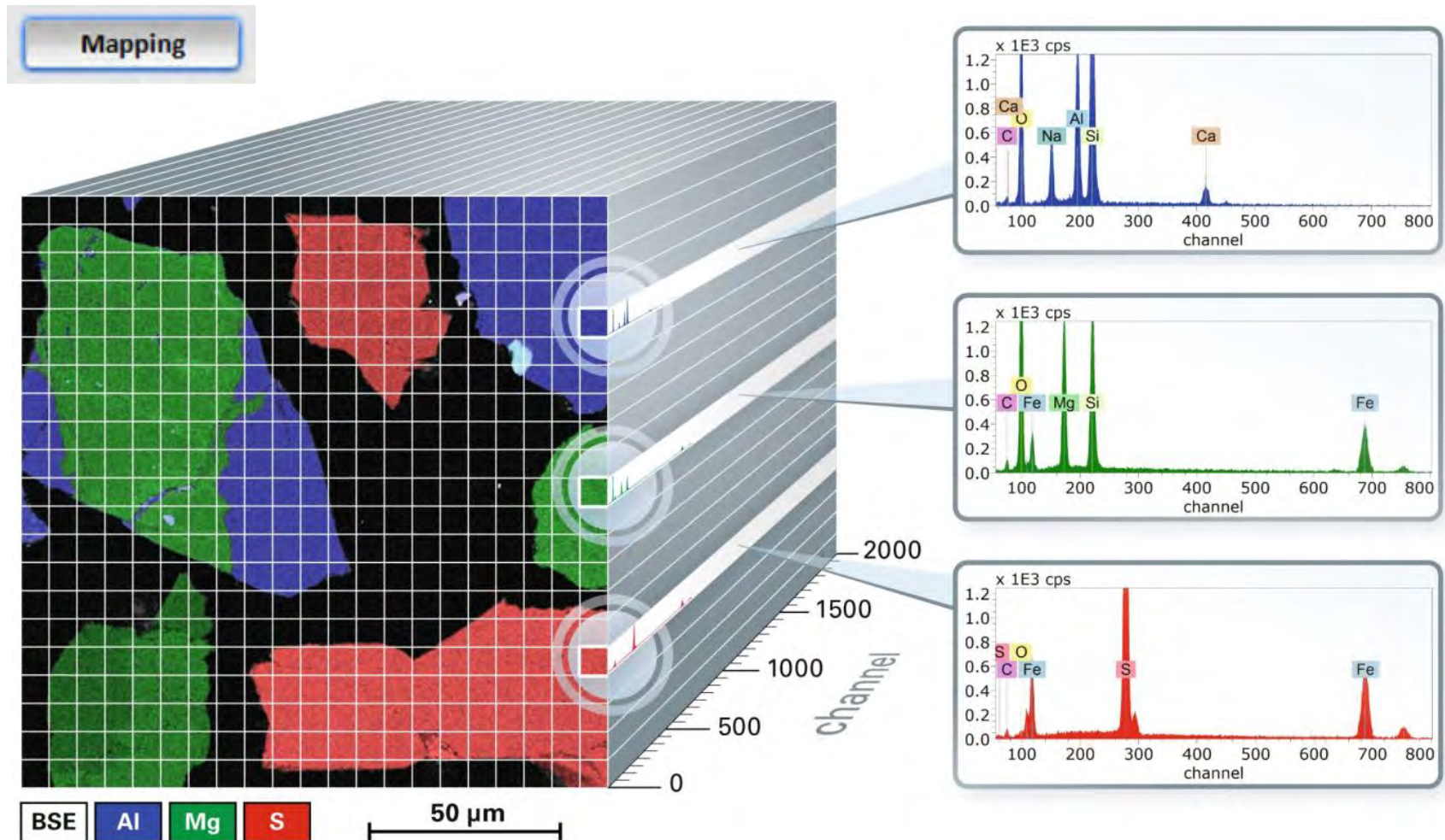
Mapping



Phase Analysis



ESPRIT for SEM-EDS HyperMapping

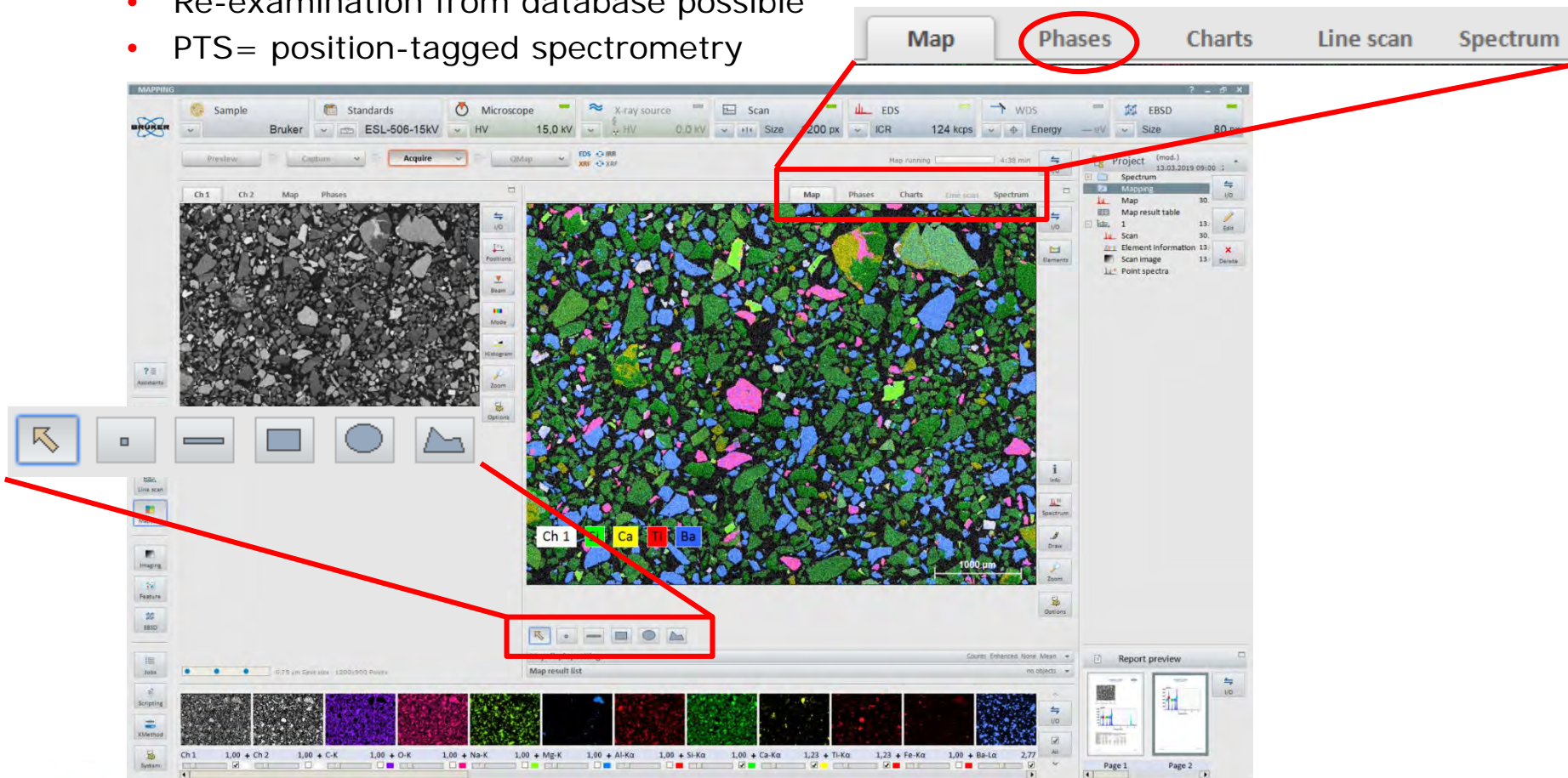


ESPRIT for SEM-EDS

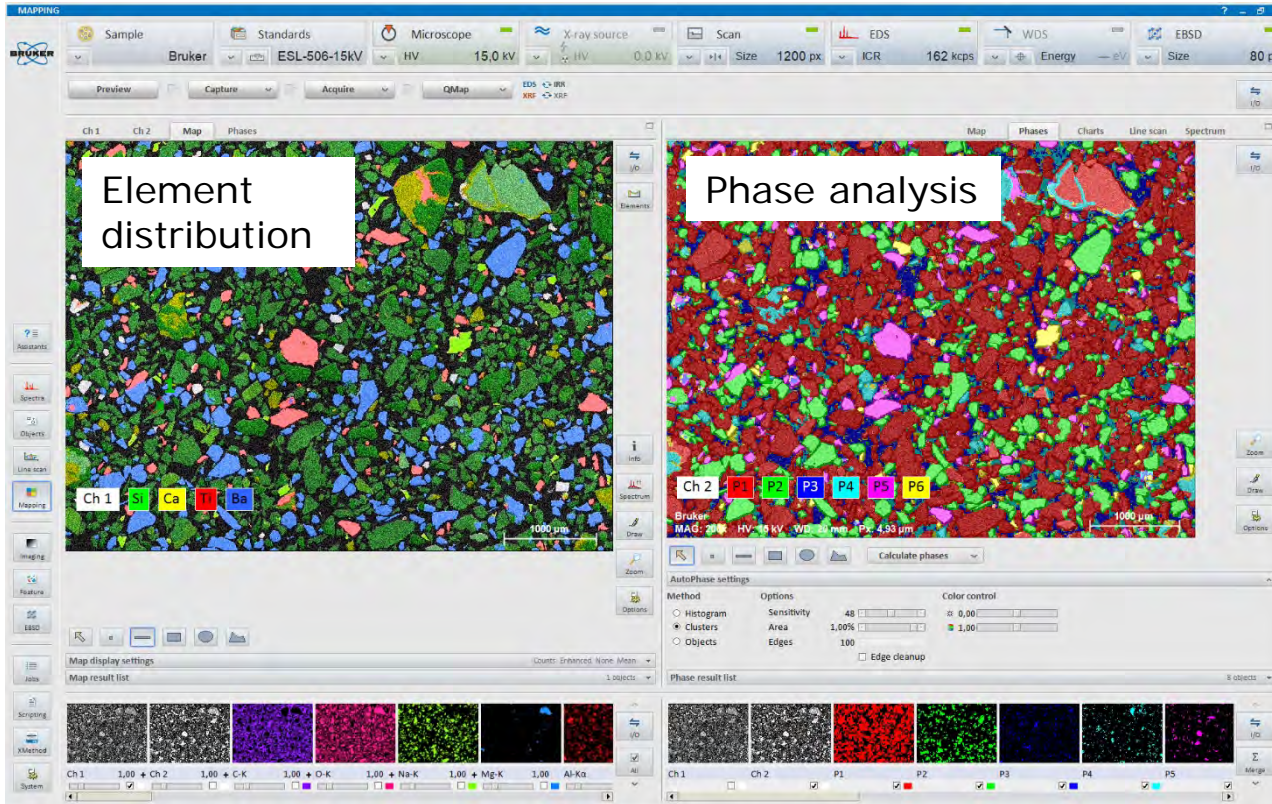
Workspace: HyperMapping



- 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

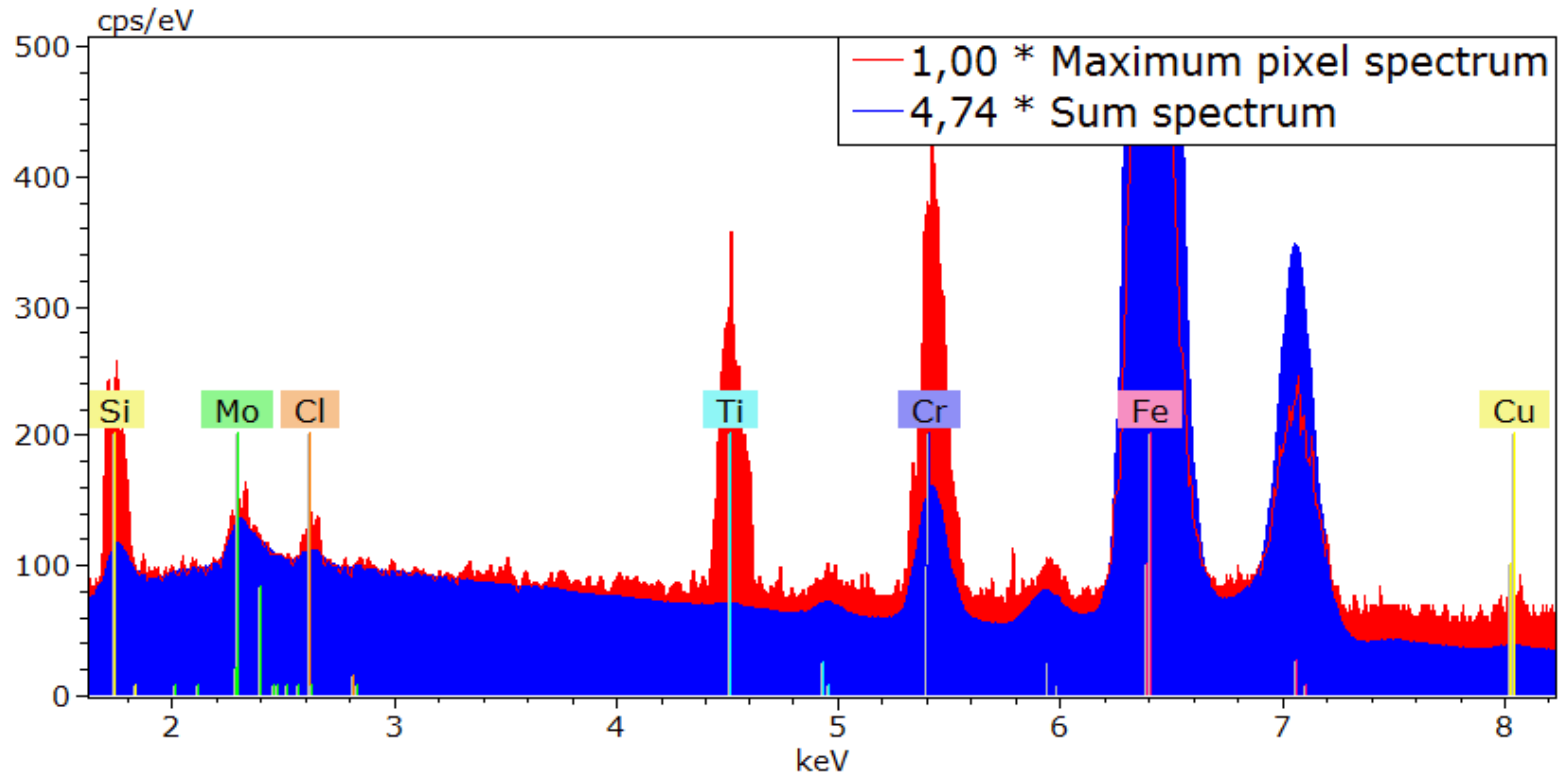
Sum spectrum and area % are calculated from each phase

ESPRIT for SEM-EDS

Maximum Pixel Spectrum

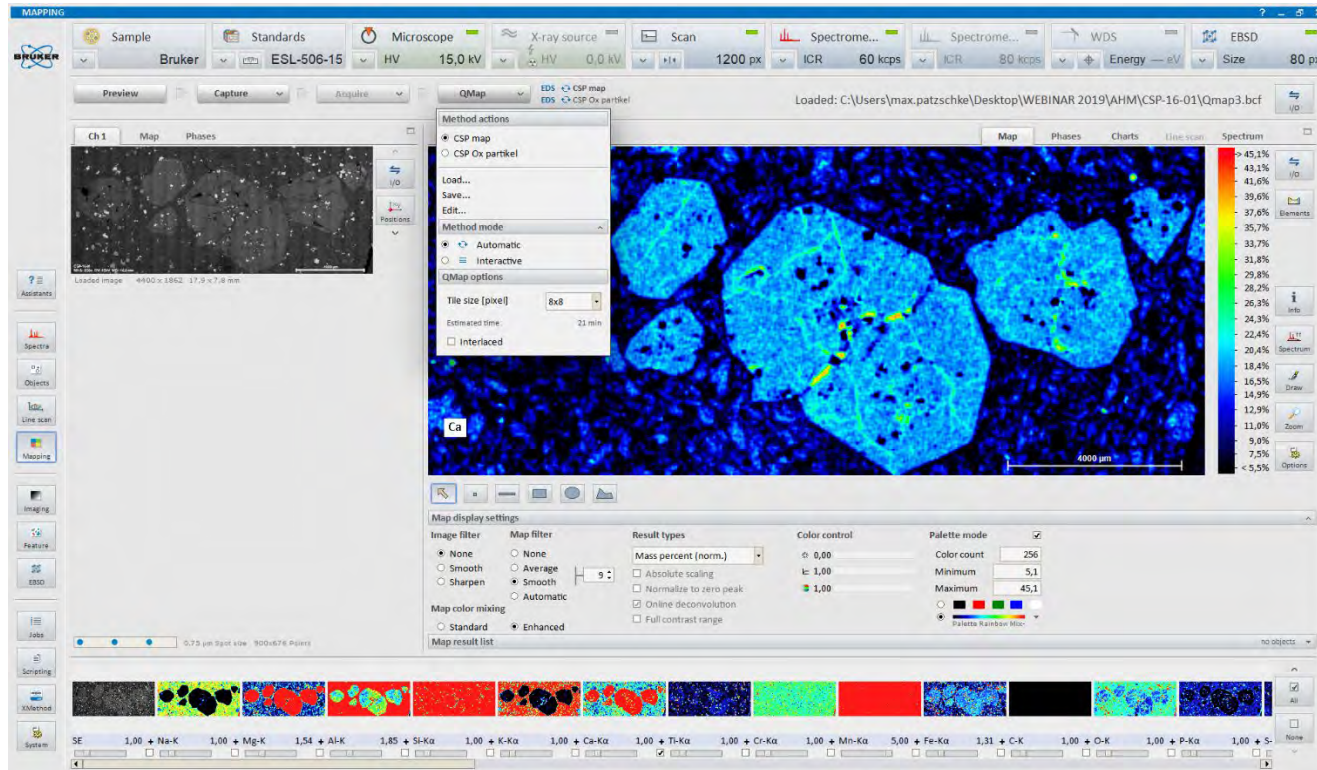


Element Identification: MaximumPixelSpectrum



- 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:

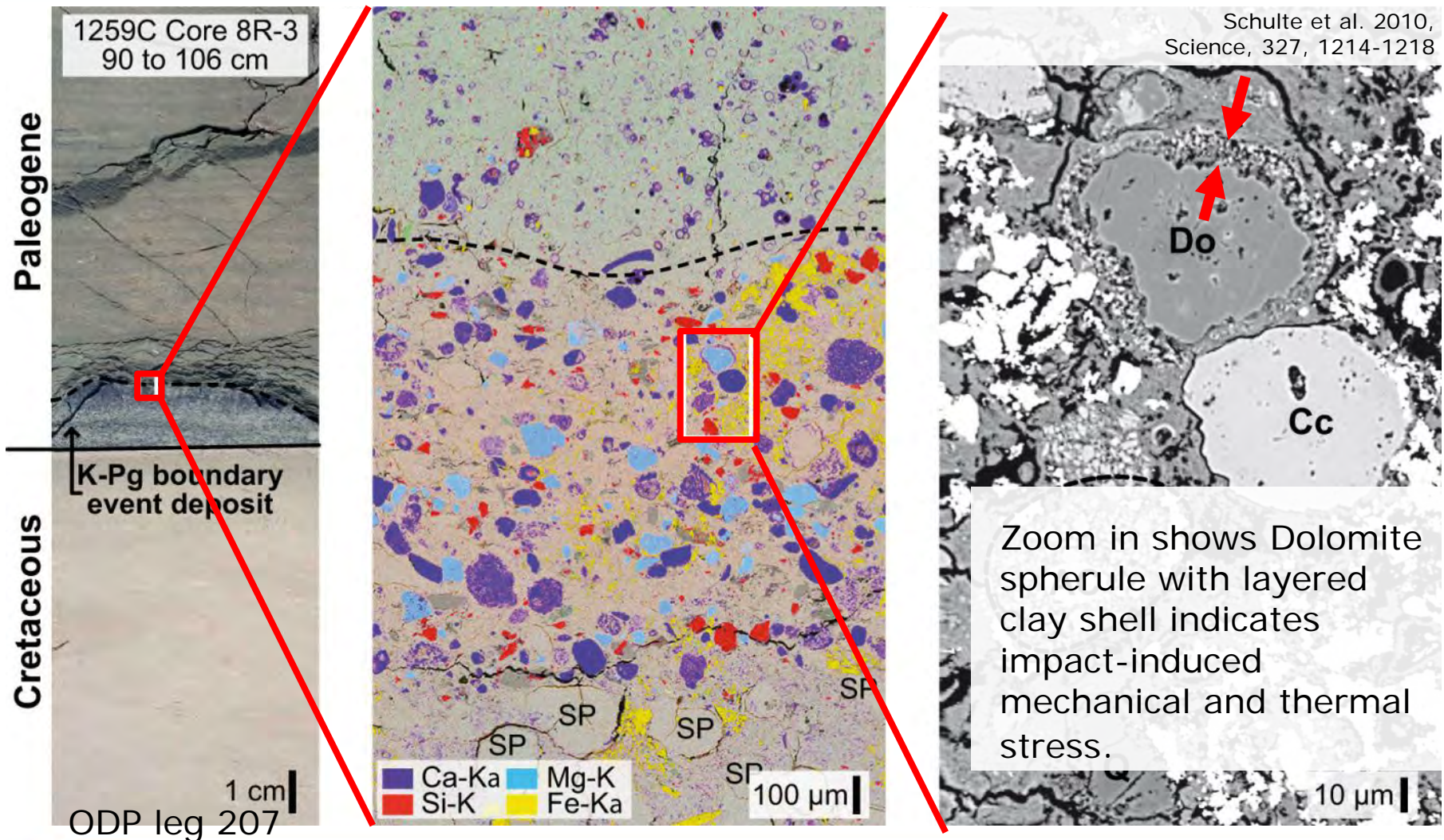
- 1x1 pixel to
- 64x64 pixel

and displayed in:

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

ESPRIT for SEM-EDS

High-resolution Map with 4072x3072 pixel and 500 kcps



ESPRIT for SEM-EDS

Hypermapping: Image Extension

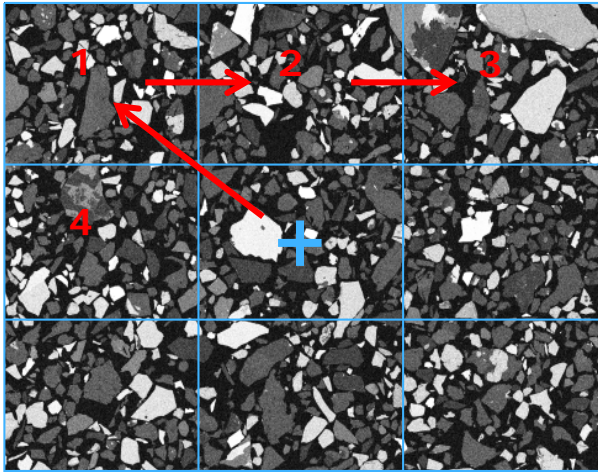
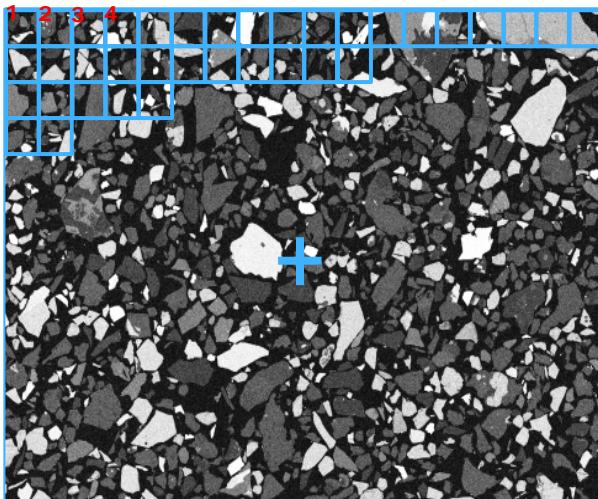


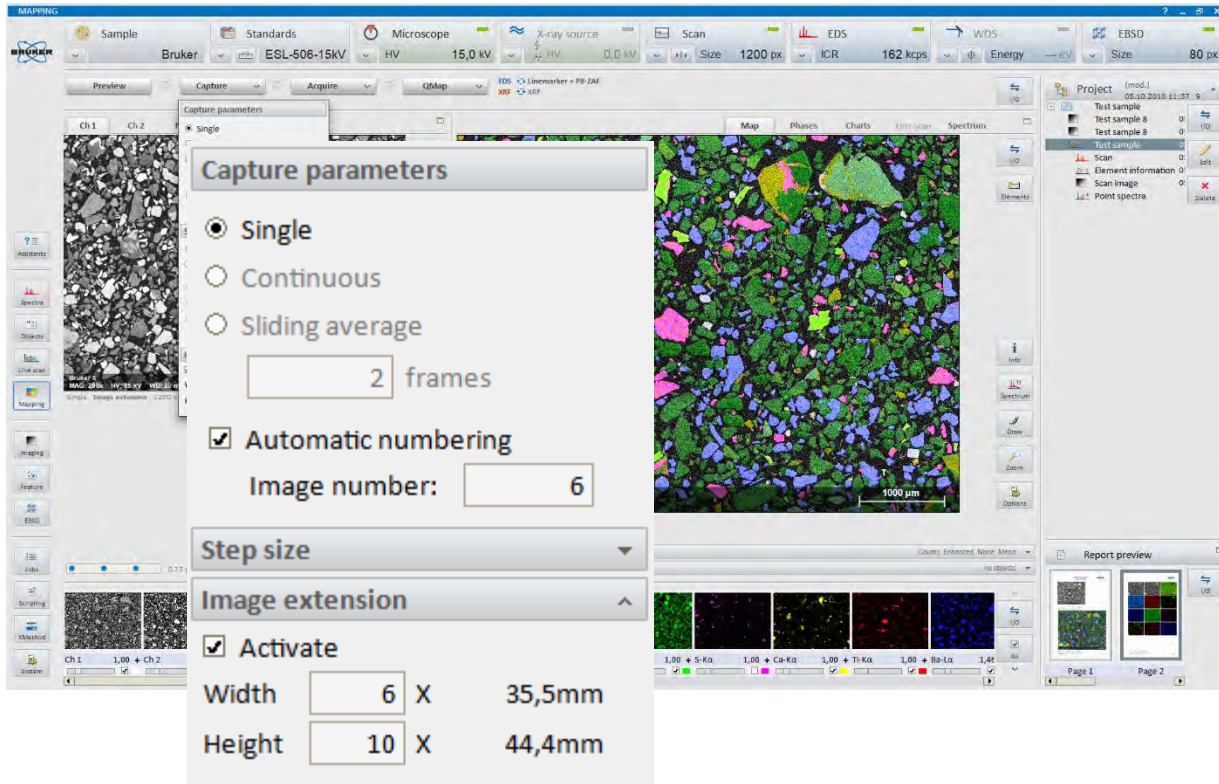
Image Extension

- 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

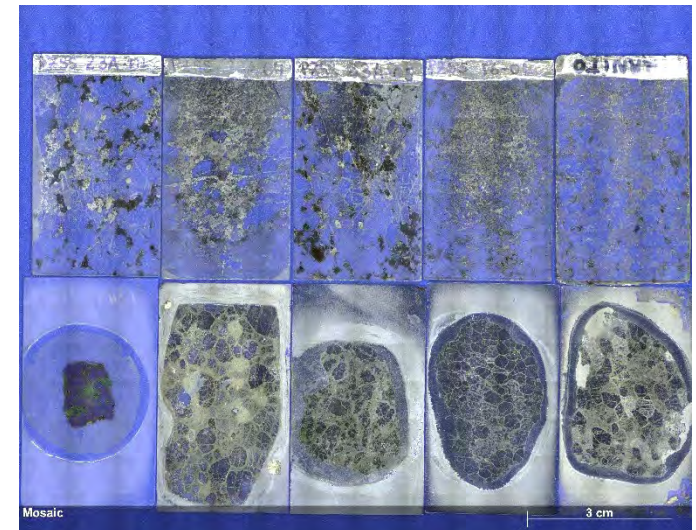
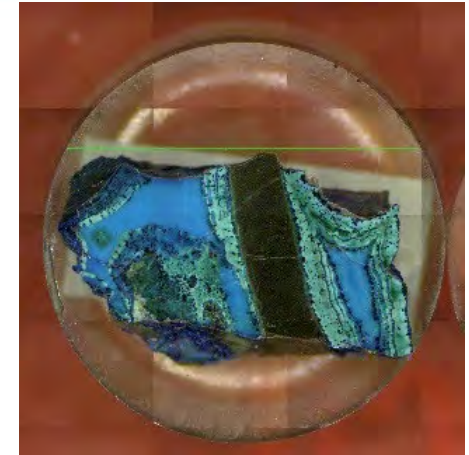
- HyperMaps and images of all kind can be enlarged by activating image extension
- Software uses central mapping position, and build automatic number of desired x/y fields around
- One HyperMap file as result for entire on- and offline data processing
- HyperMaps with more than 20.000x15.000 pixels can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

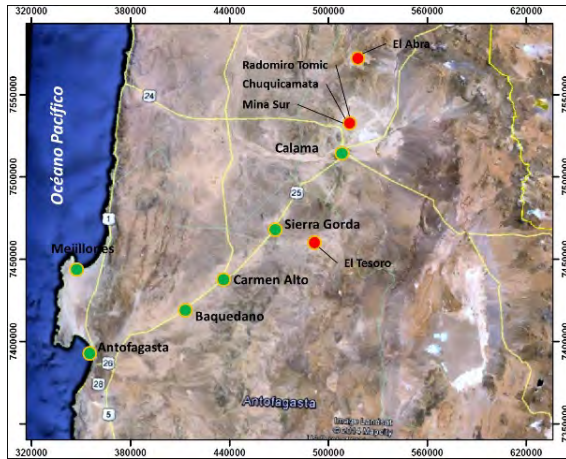


Microanalysis: SEM-EDS Hypermap - Examples

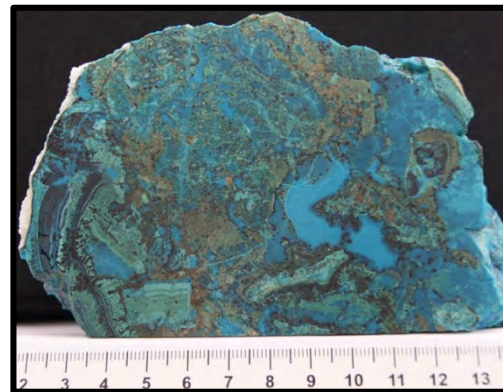
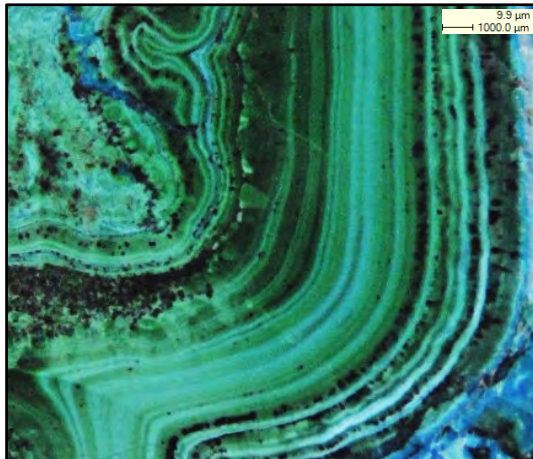


This composite image illustrates SEM-EDS capabilities. On the left, a periodic table of elements is shown in a blue, semi-transparent style. In the center, a detailed view of the Bruker XFlash 6110 detector is shown, a compact, multi-layered silicon drift chamber. To the right, the text "EDS" and "XFlash® Technology" is displayed in a large, light blue font. Below the text, a series of sharp peaks from an EDS spectrum are visible. On the far right, two examples of elemental maps (hypermaps) are shown, displaying the spatial distribution of different elements in a sample.

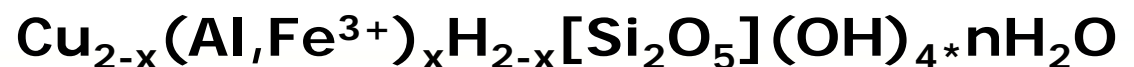
Geological Applications: Exotic-Cu Deposits



- 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.



Chrysocolla

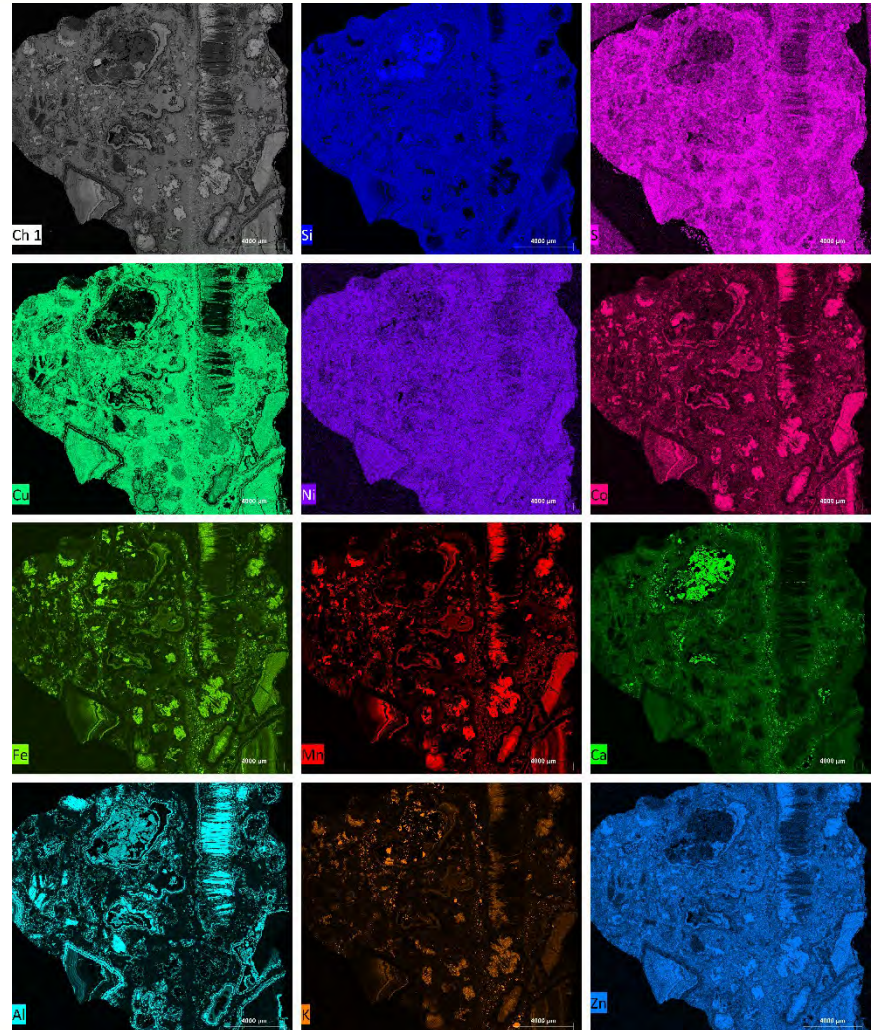
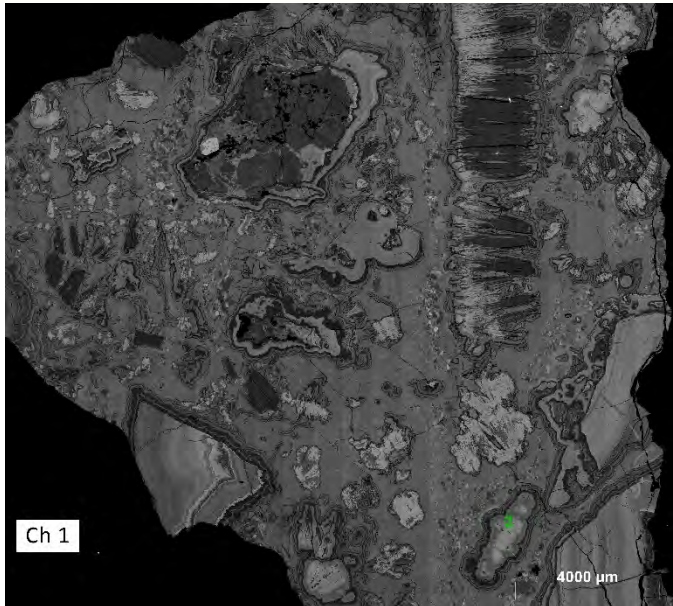


Geological Applications: Exotic-Cu Deposits



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

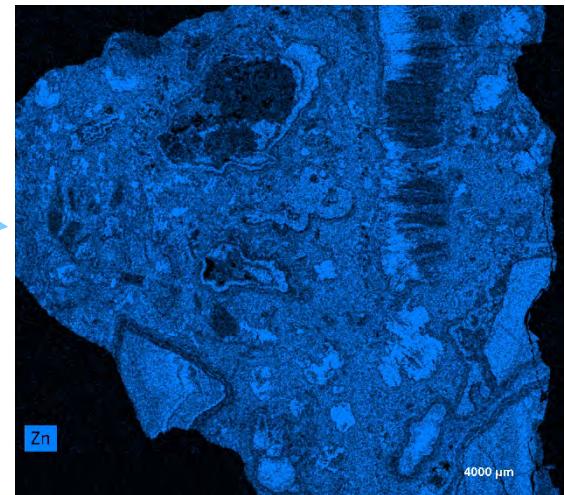
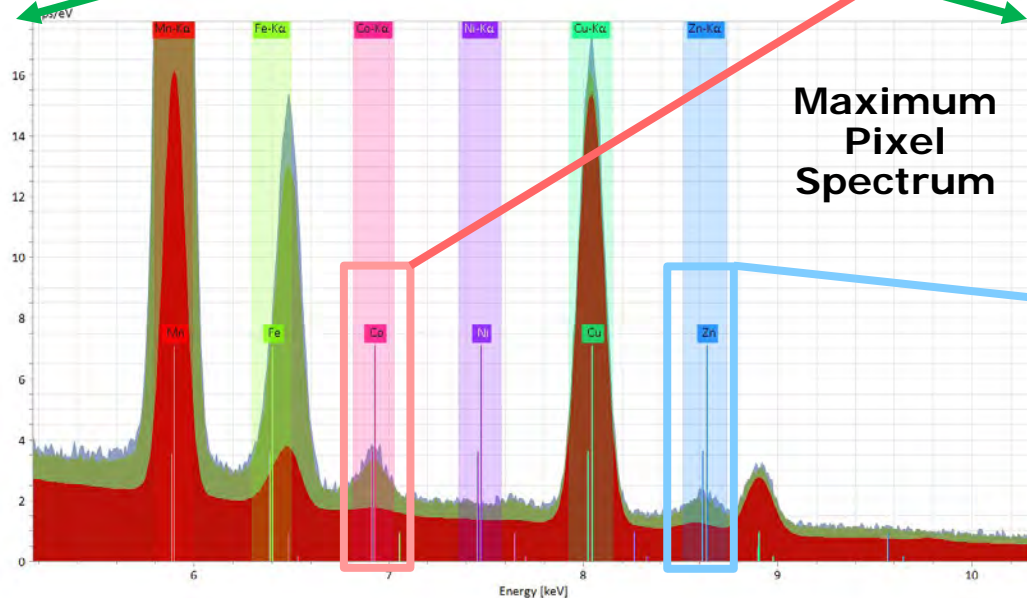
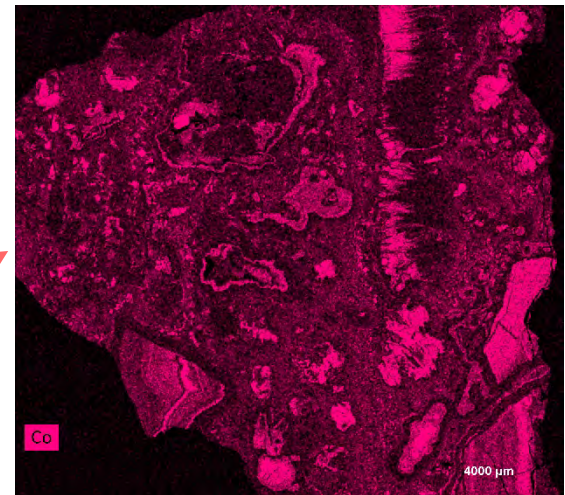
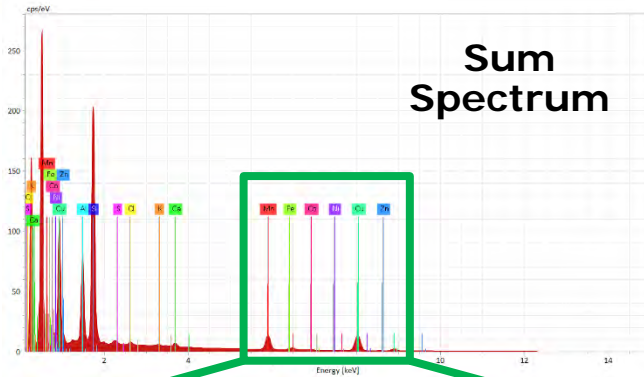


Measurement Conditions

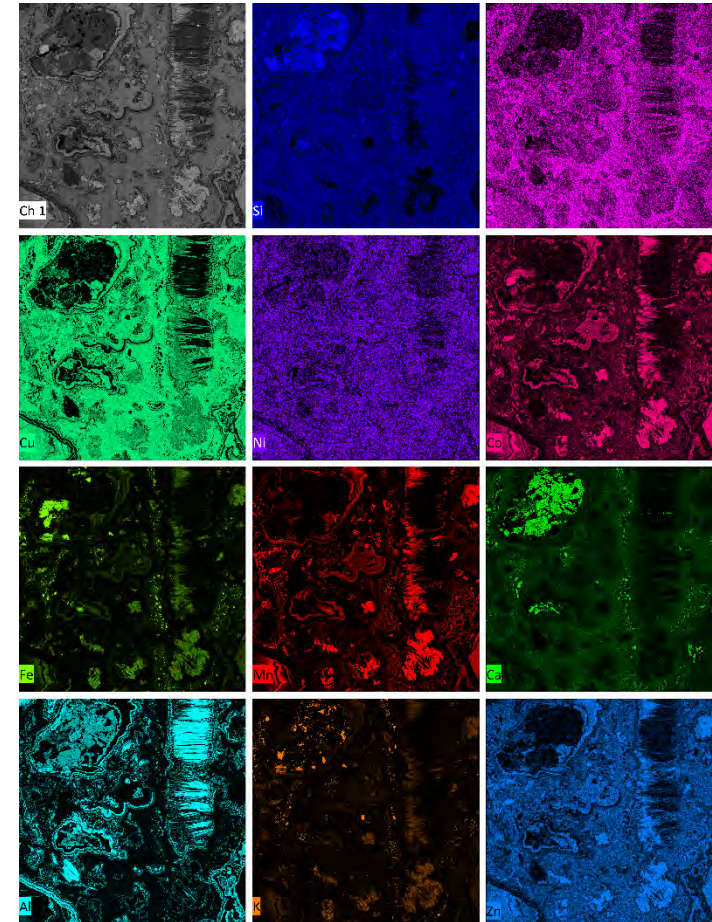
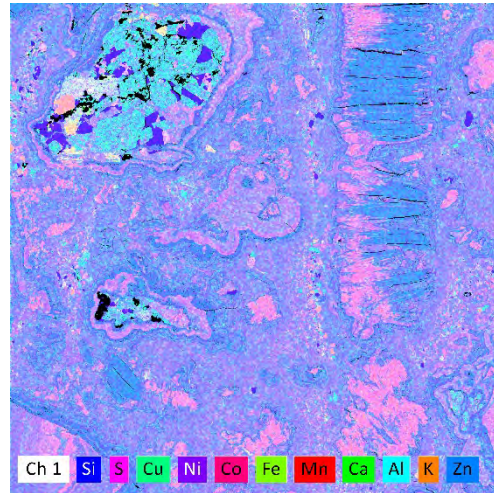
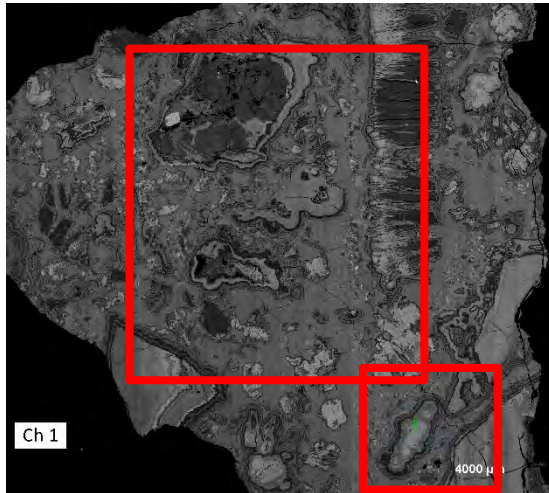
High Voltage: 15 kV
Pixels: 4800x4788
Measurement Time: 1568 min
SDD: 10 mm²
Dwell time: 4096 µs
FOV: 19.0 mm
Pixel size: 4.1 µm
Fields: 308 (30x40)
Magnification: 250x

SEM-EDS: Hypermap Results

Minor Elements – Maximum Pixel Spectrum



SEM-EDS: Hypermap Results Image Extension

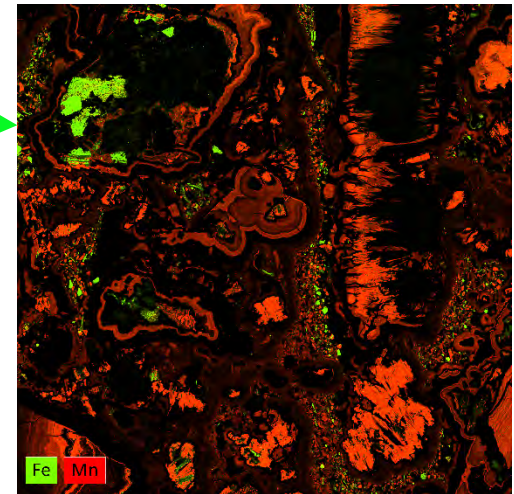
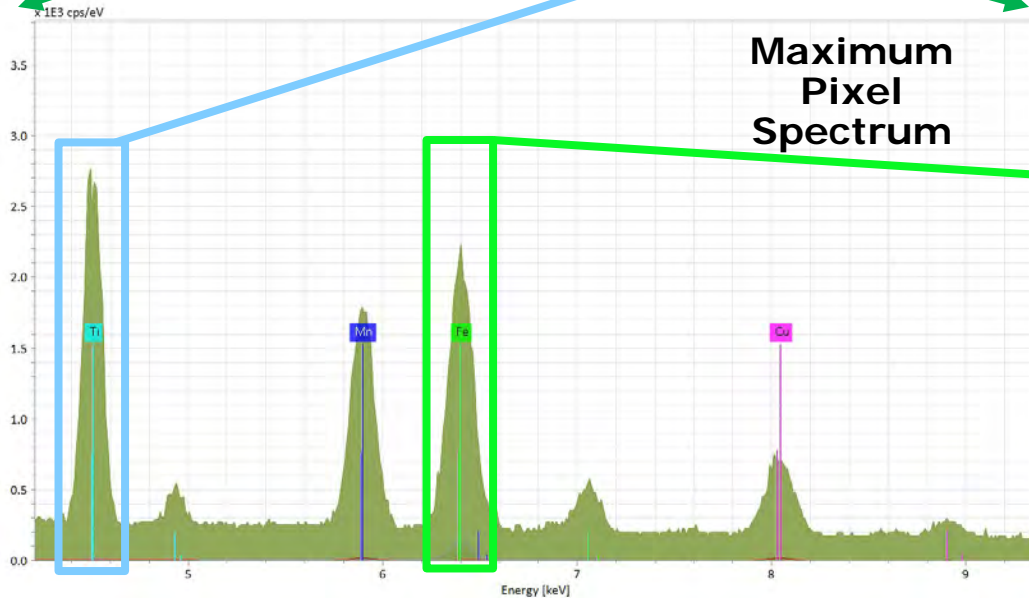
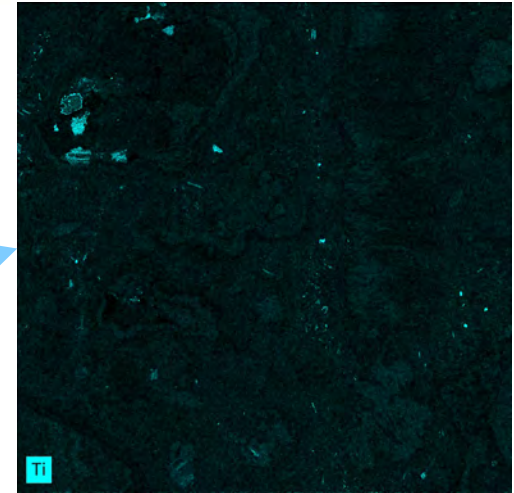
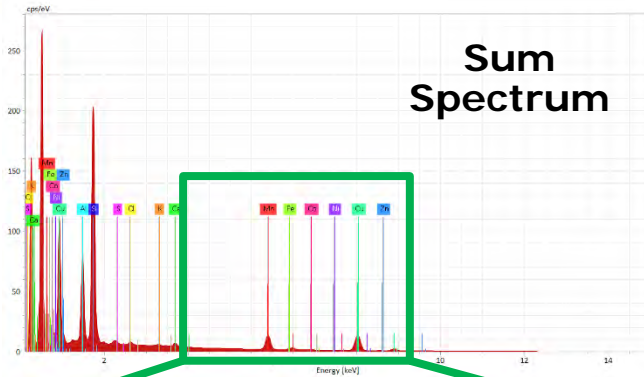


Measurement Conditions

High Voltage:	15 kV
Pixels:	4800x4788
Measurement 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

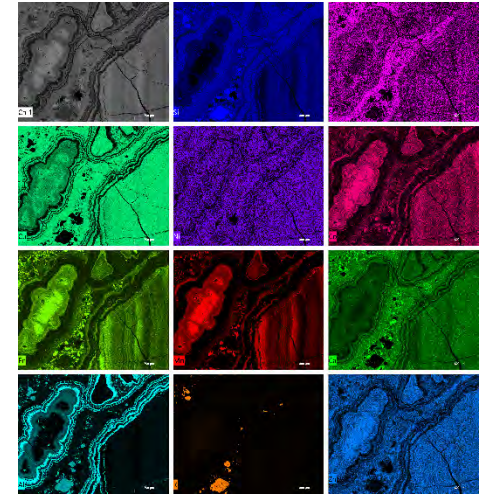
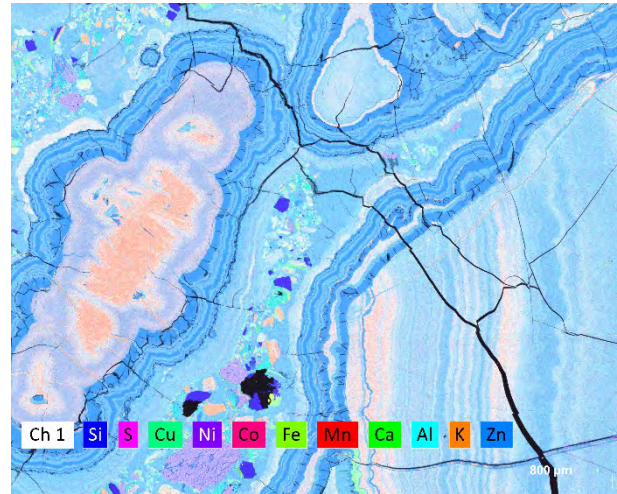
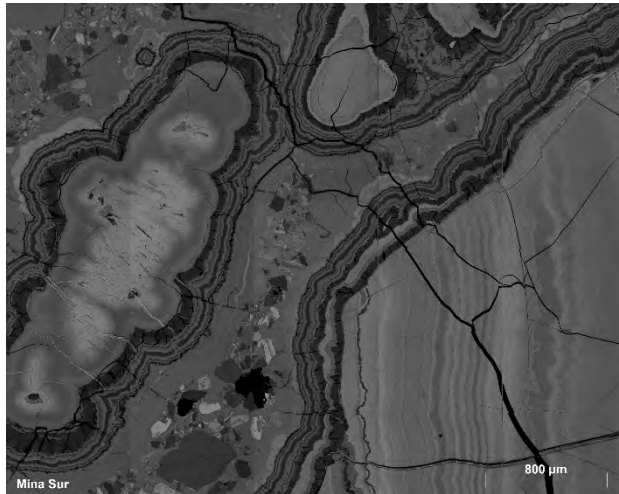
SEM-EDS: Hypermap Results

Minor Elements – Maximum Pixel Spectrum



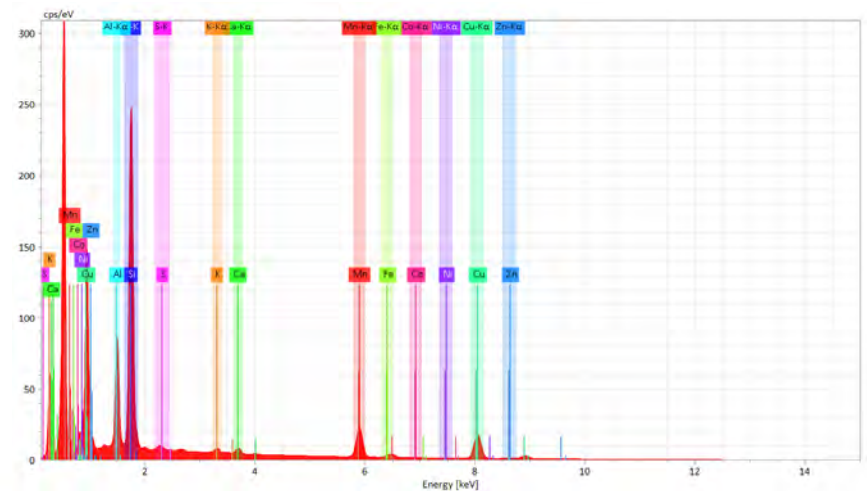
SEM-EDS: Hypermap Results

Image Extension



Measurement Conditions

High Voltage:	15 kV
Pixels:	3000x2400
Measurement 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



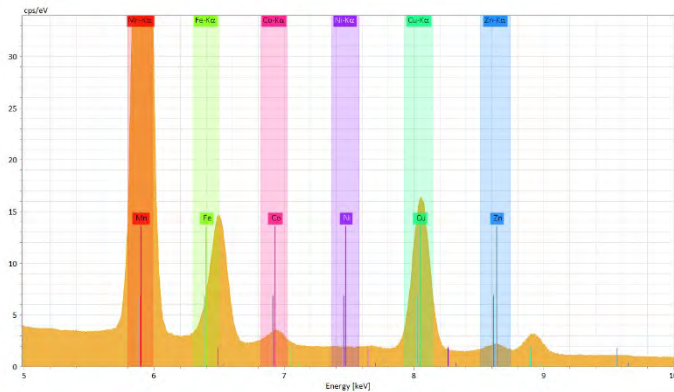
SEM-EDS: Hypermap Results

Line Scans



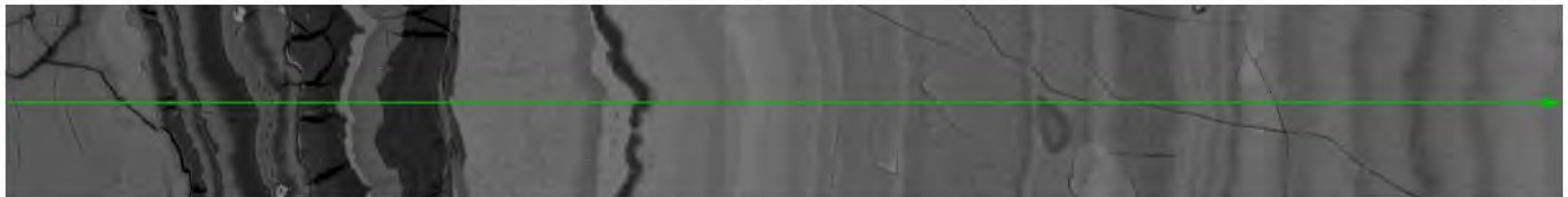
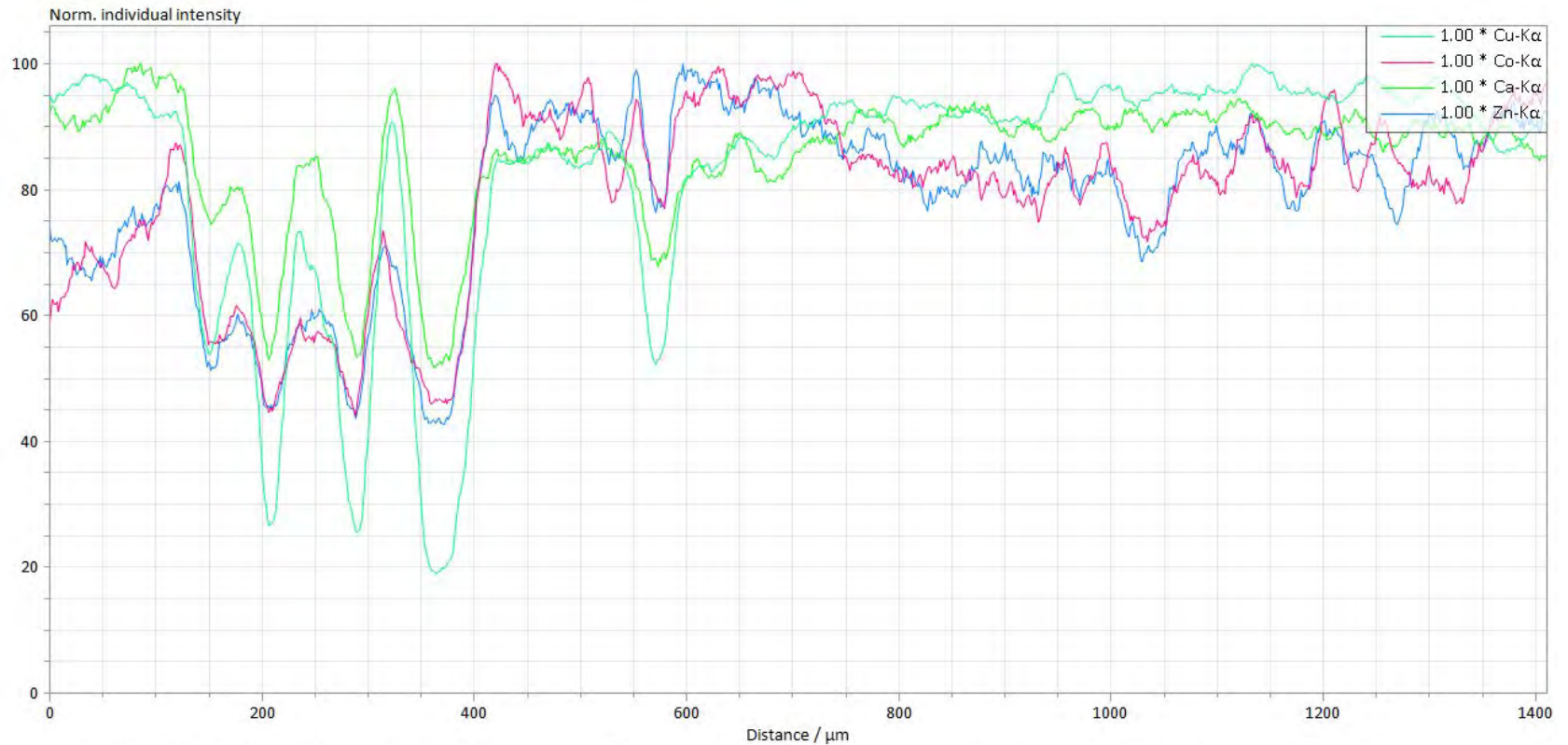
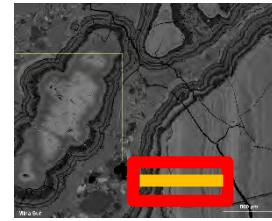
Measurement Conditions

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



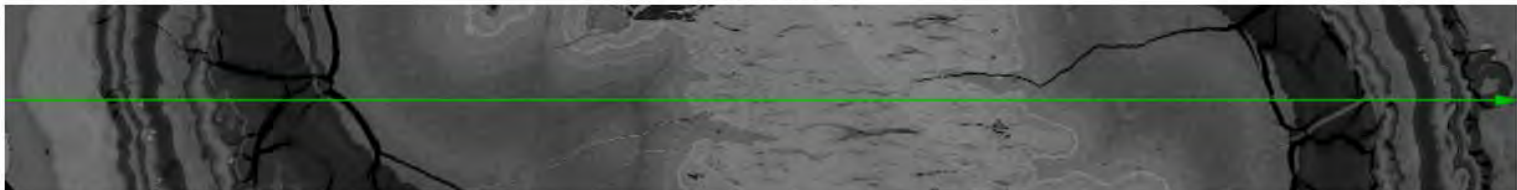
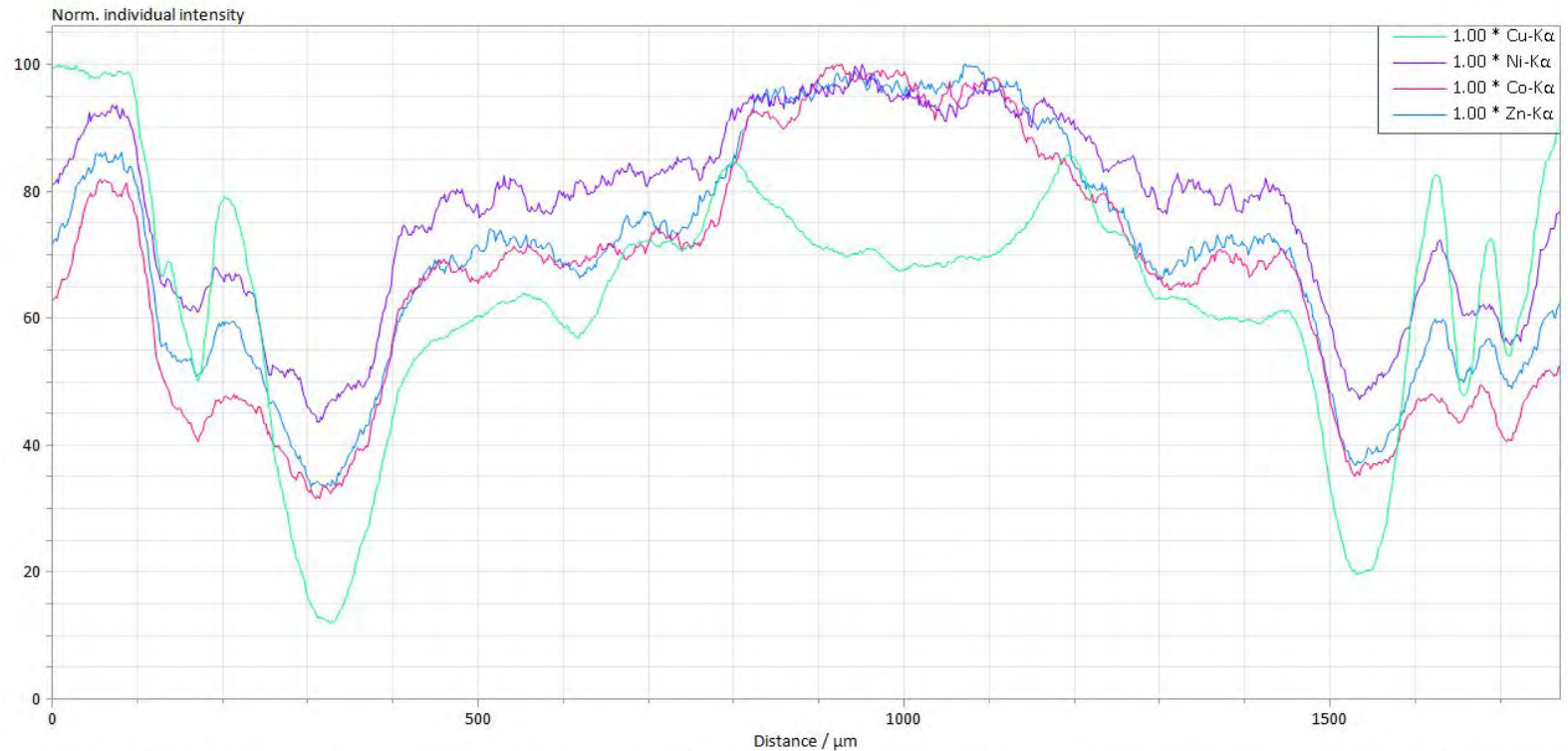
SEM-EDS: Hypermap Results

Line Scans

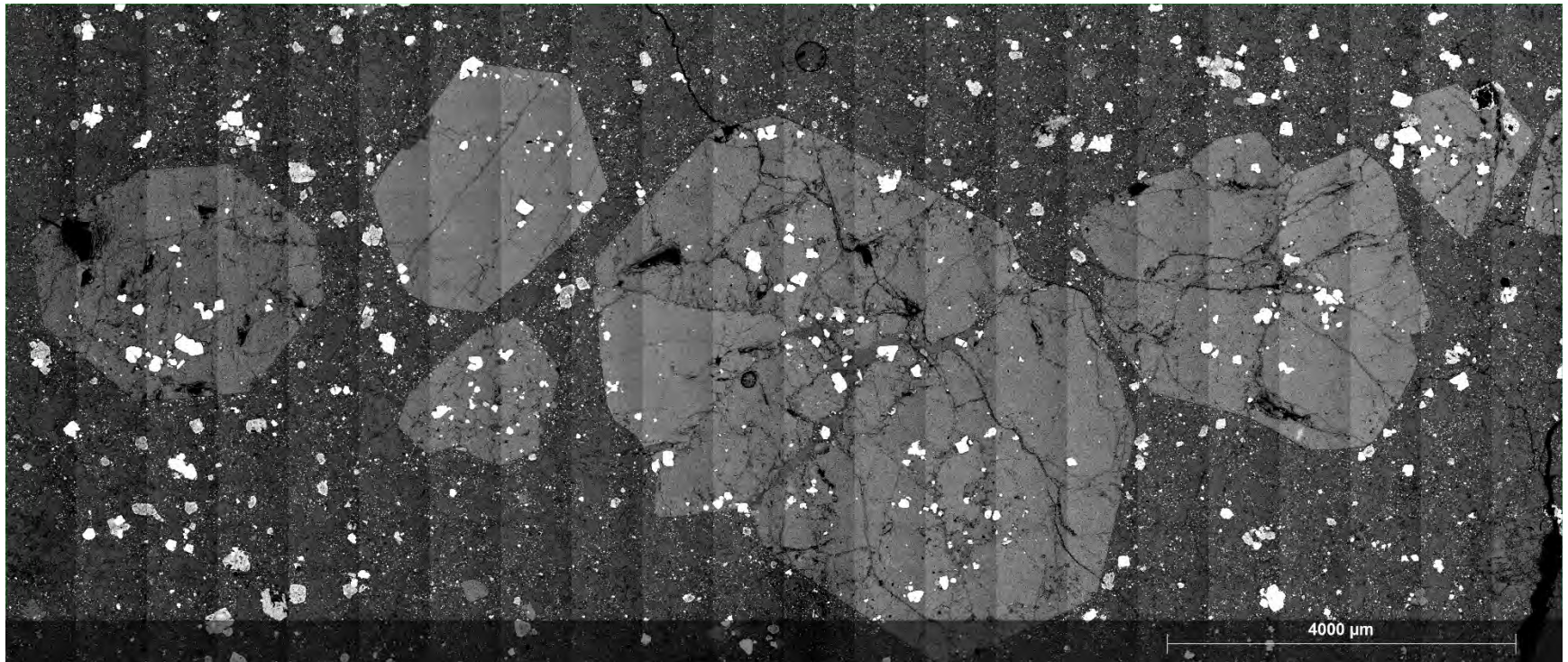


SEM-EDS: Hypermap Results

Line Scans



Geological Applications: Igneous Volcanic Extrusive

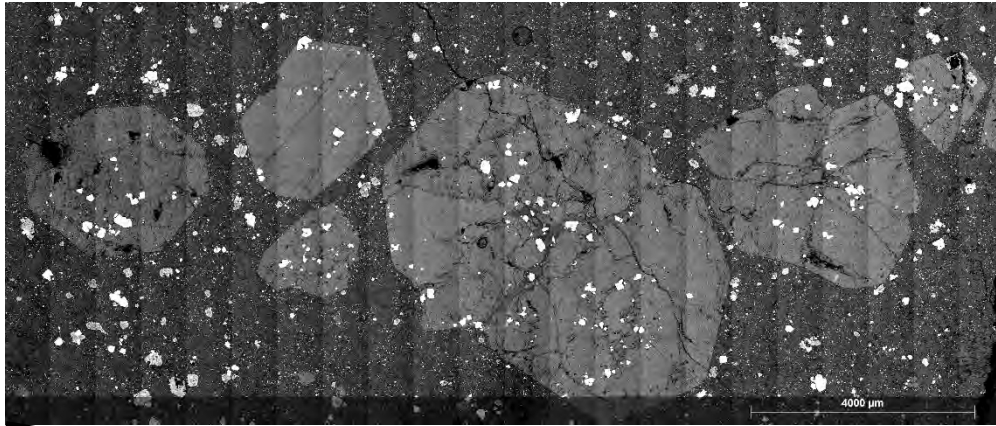


Measurement Conditions:

High Voltage:	15 kV	Dwell time:	23 μs
Pixels:	8800x5600	FOV:	17.8 mm
Measurement Time:	616 min	Pixel size:	4.1 μm
SDD:	10 mm ²	Fields:	308 (22x14)
		Magnification:	250x

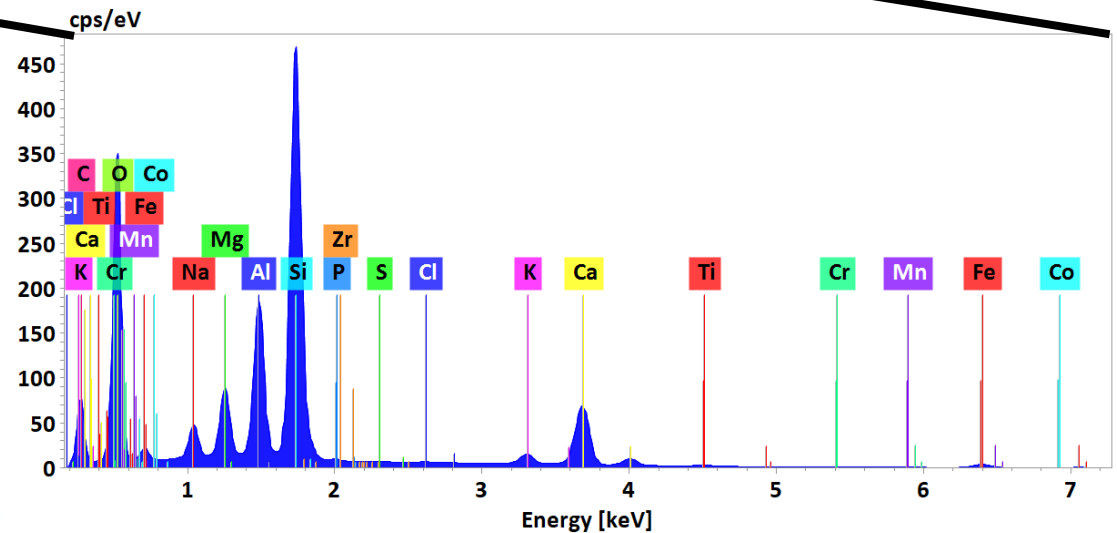
SEM-EDS: Hypermap Results

Image Extension



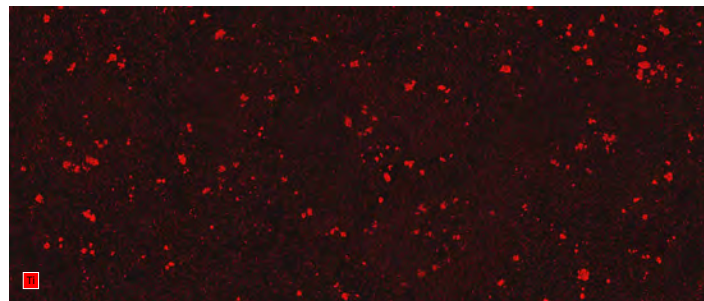
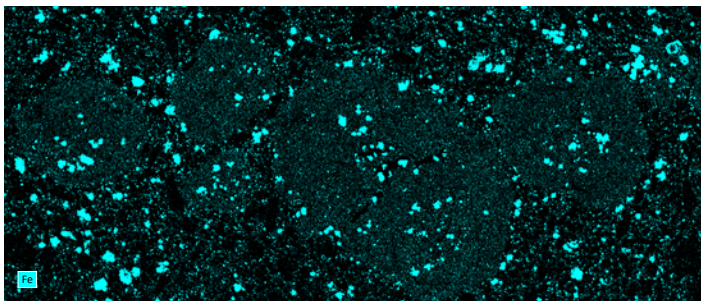
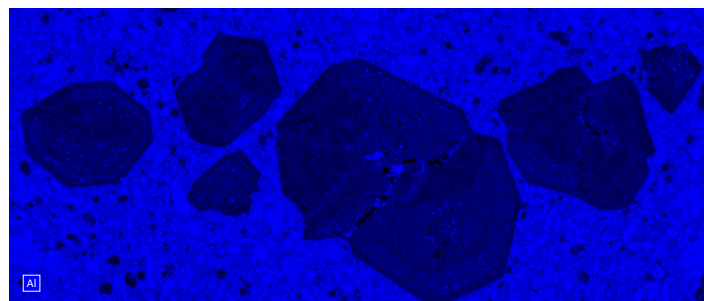
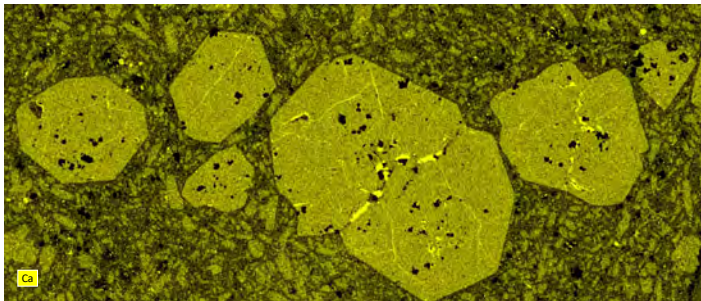
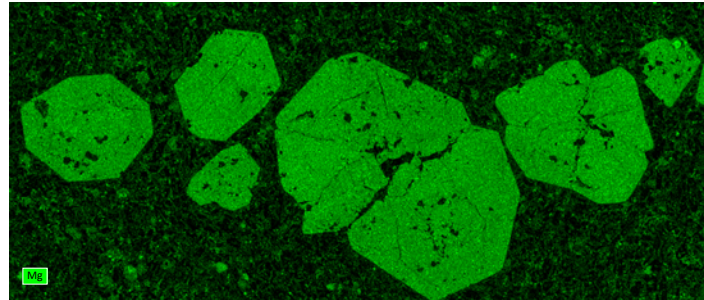
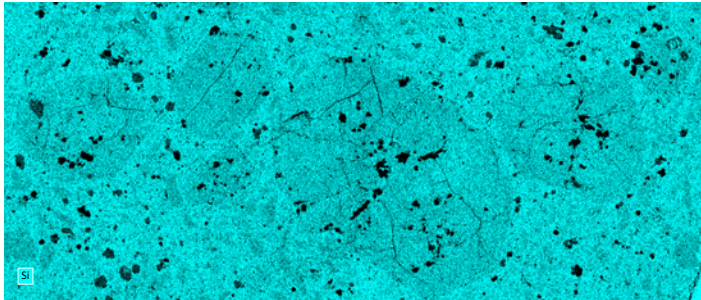
Hypermap:

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



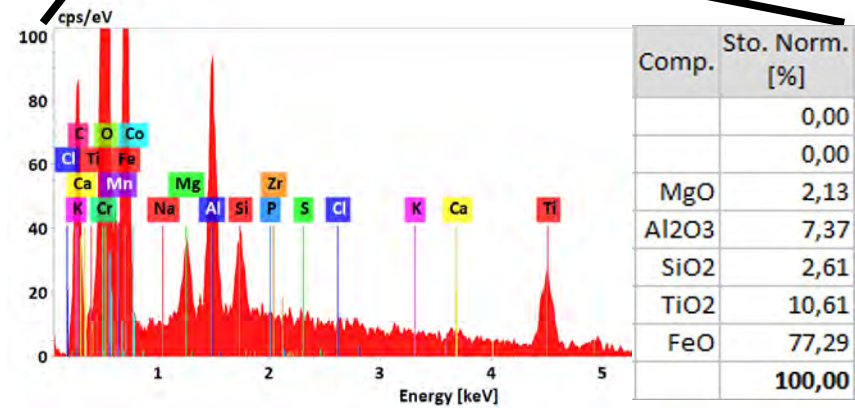
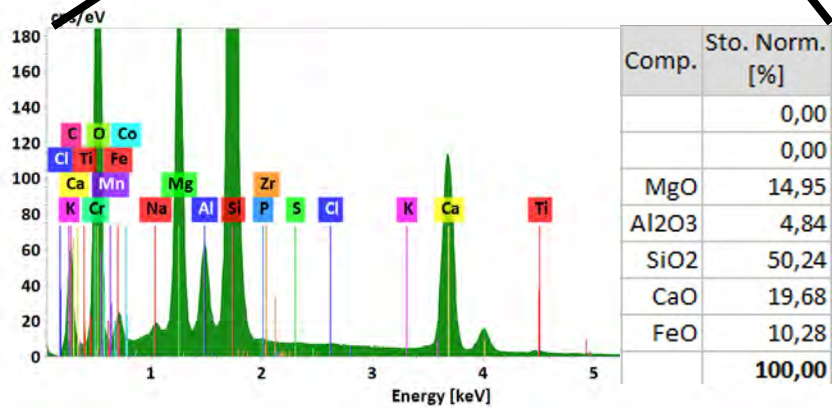
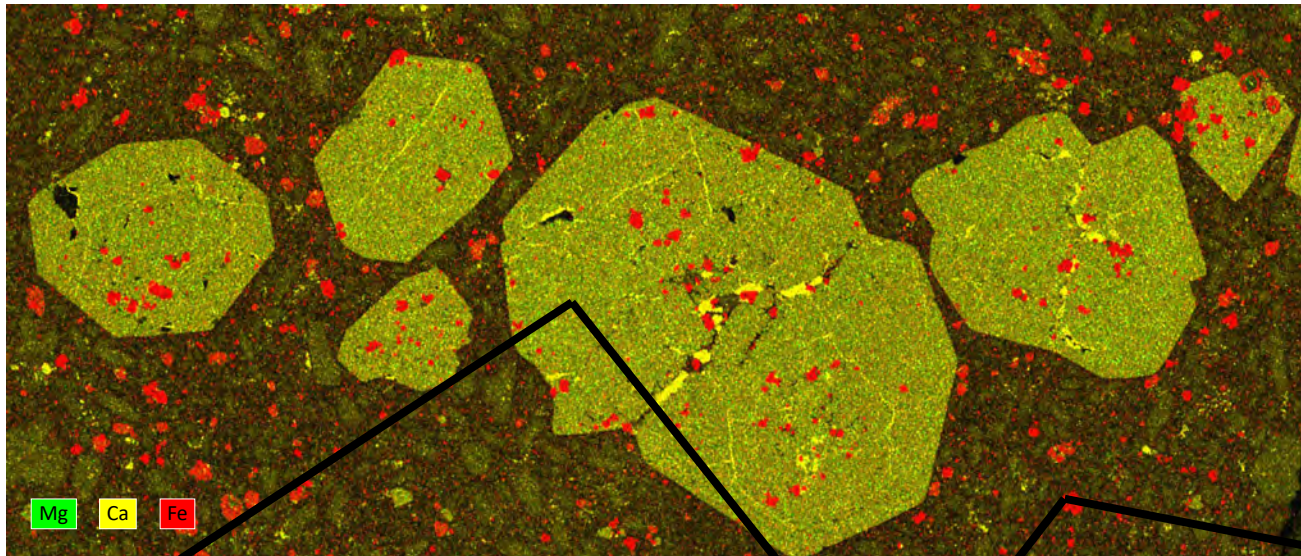
SEM-EDS: Hypermap Results

Elemental Peak Intensity Maps



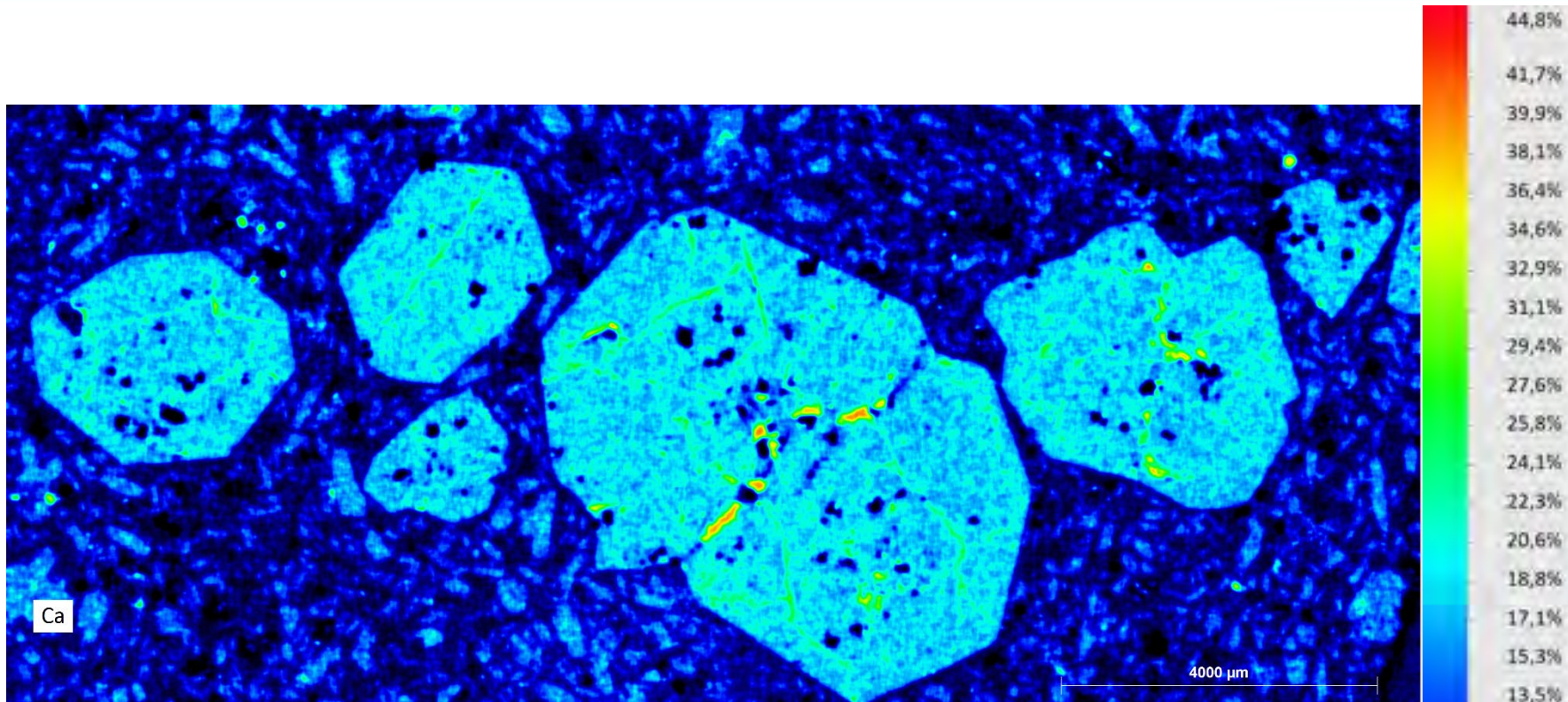
SEM-EDS: Hypermap Results

Each pixel can be quantified



SEM-EDS: Hypermap Results

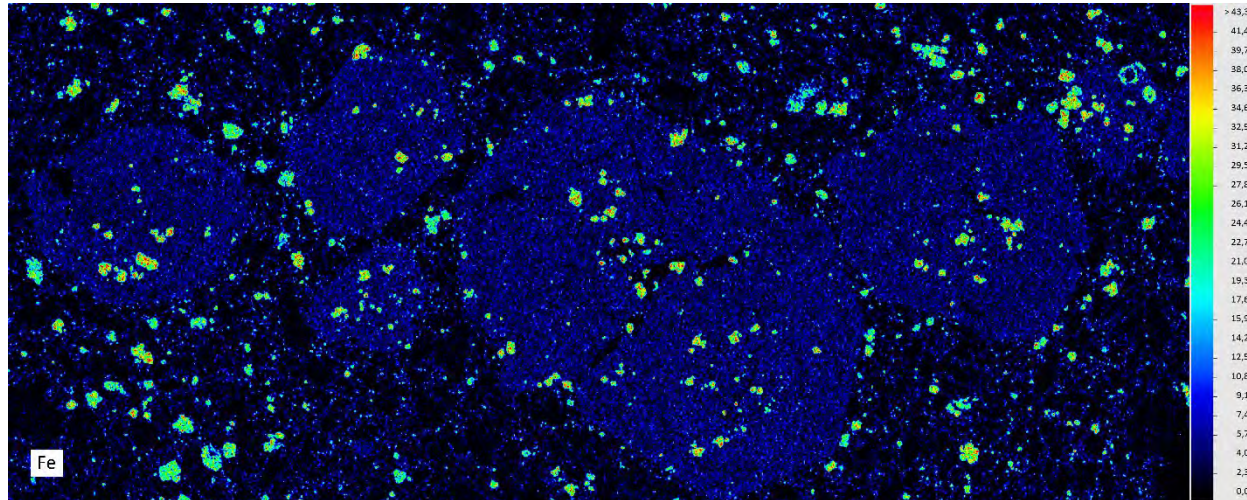
Quantified Map (Qmap)



Qmap shows false colour elemental distribution of Ca

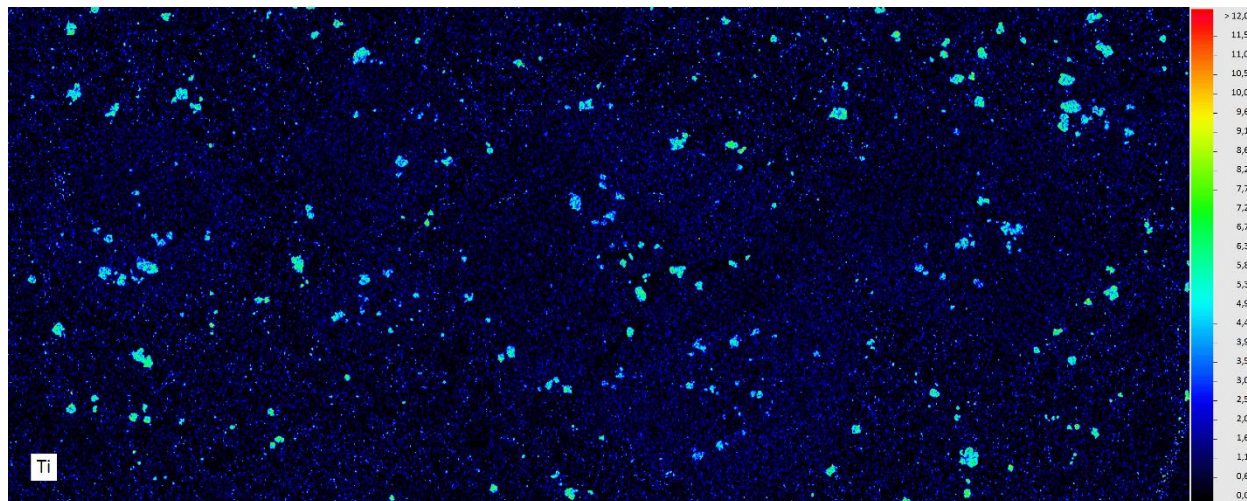
SEM-EDS: Hypermap Results

Quantified Map (Qmap)



Qmap displays elemental mass% distribution over the entire sample

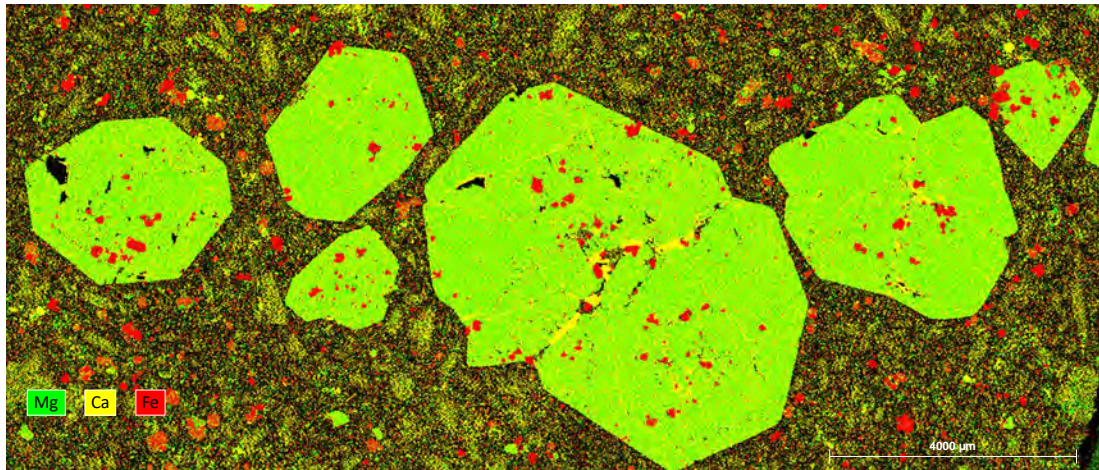
Top: Fe



Bottom: Ti

SEM-EDS: Hypermap Results

Elemental Peak Intensity Maps



Measurement Time: 77 min

SDD: 10 mm²

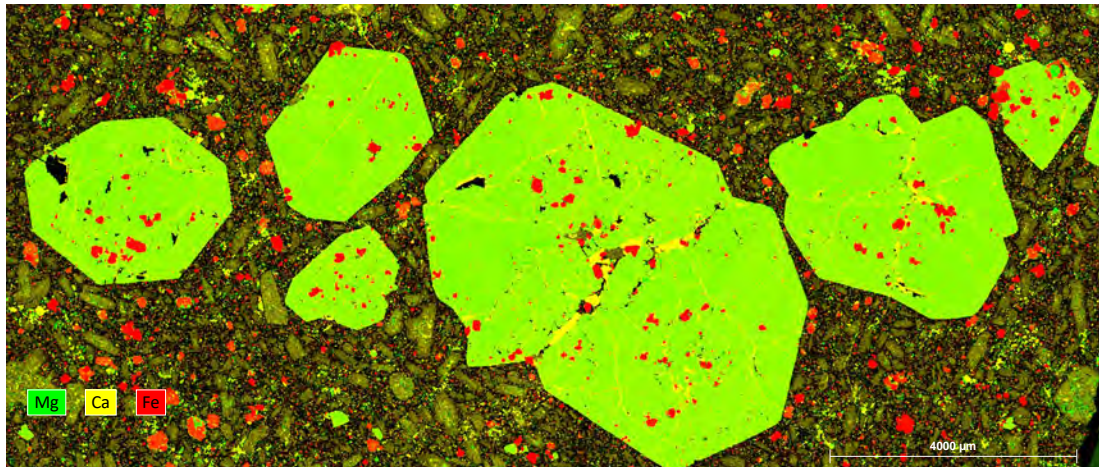
Dwell time: 64 μs

FOV: 17.8 mm

Pixel size: 4.1 μm

Fields: 308 (22x14)

Magnification: 250x



Measurement Time: 616 min

SDD: 10 mm²

Dwell time: 512 μs

FOV: 17.8 mm

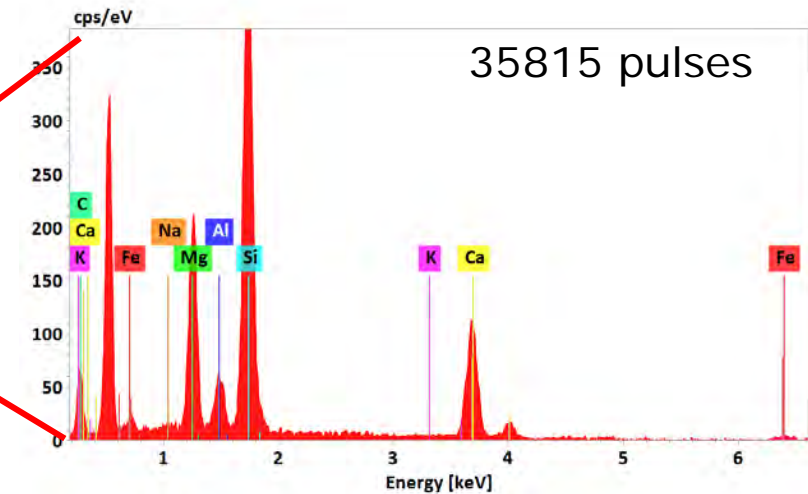
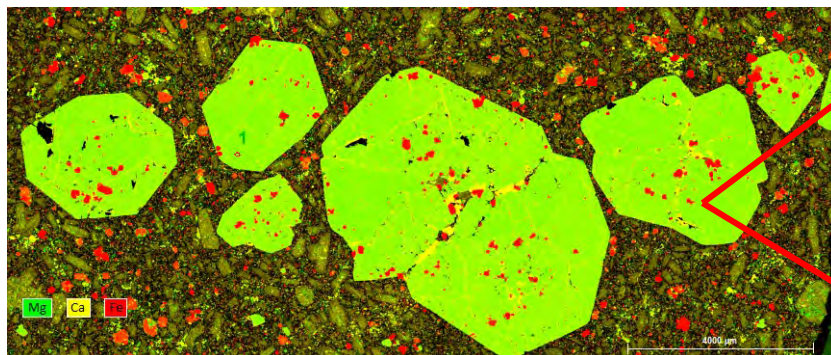
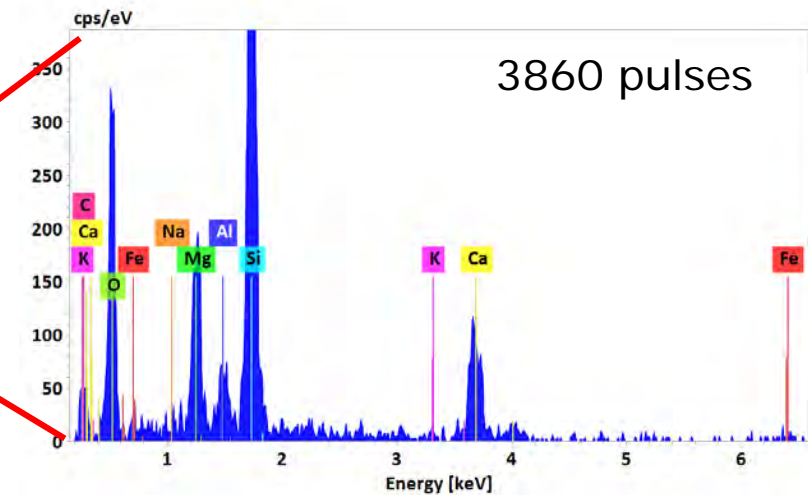
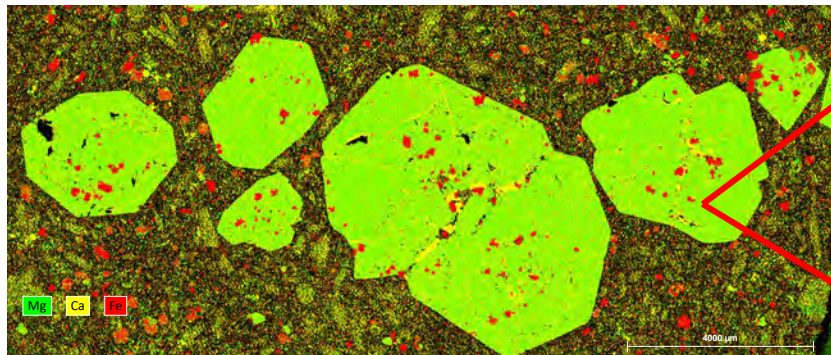
Pixel size: 4.1 μm

Fields: 308 (22x14)

Magnification: 250x

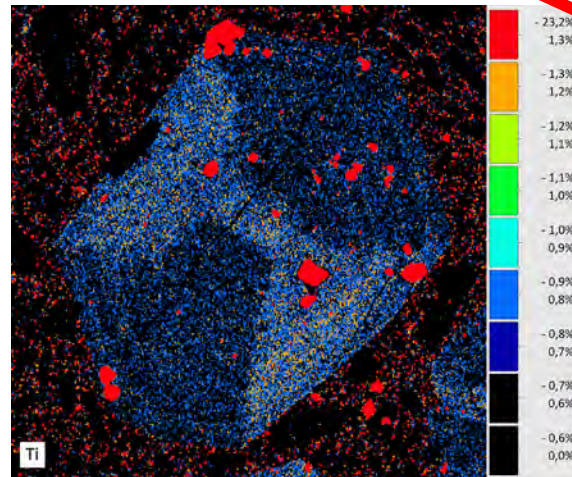
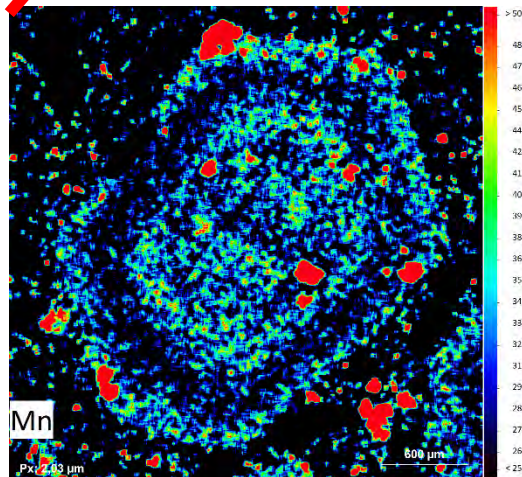
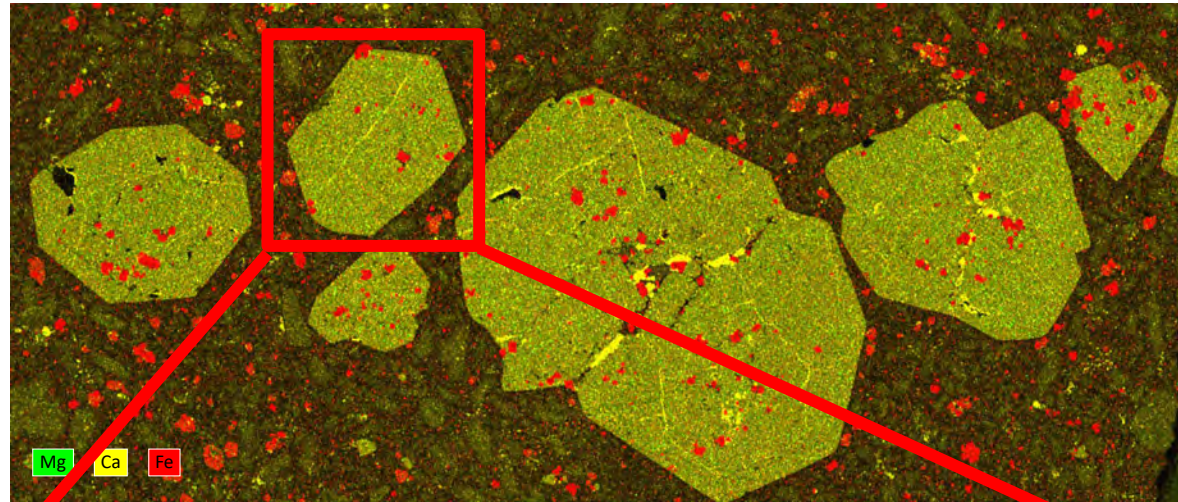
SEM-EDS: Hypermap Results

Elemental Peak Intensity Maps



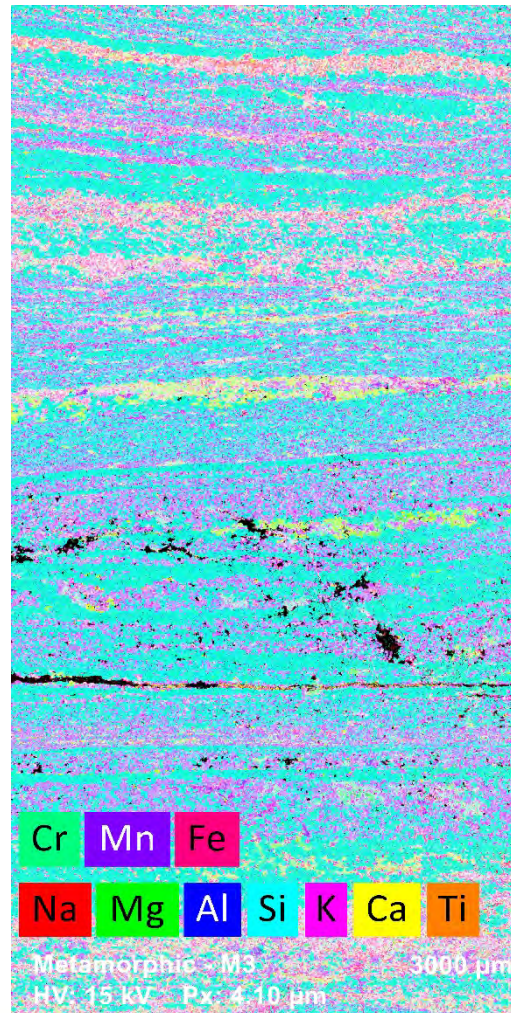
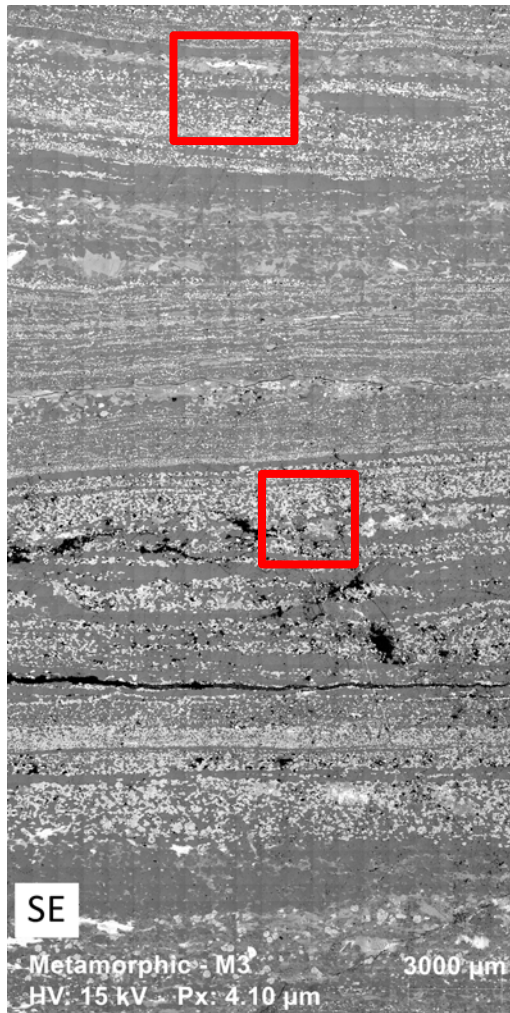
SEM-EDS: Hypermap Results

Elemental Peak Intensity Maps



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

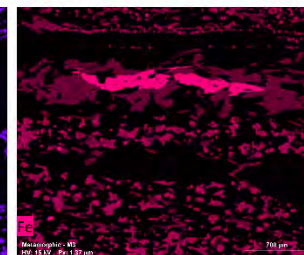
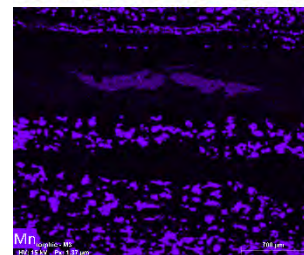
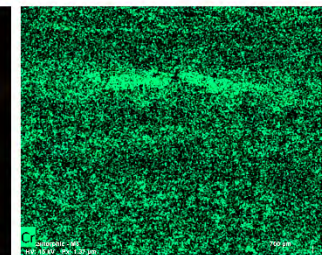
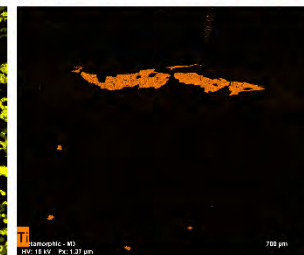
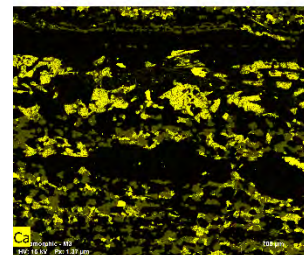
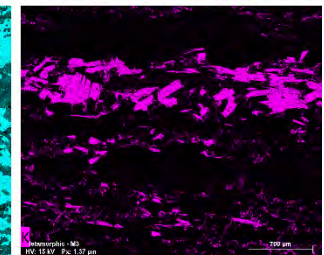
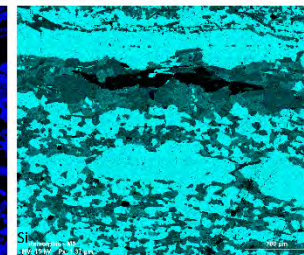
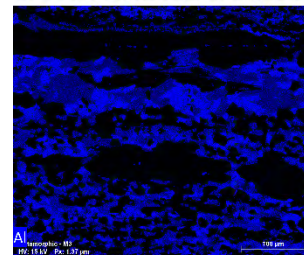
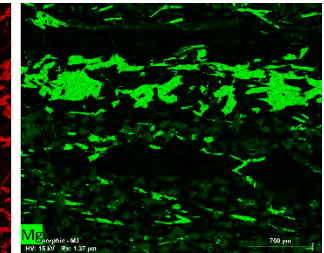
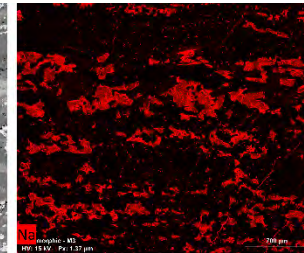
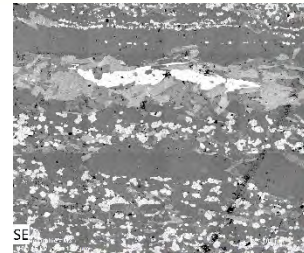
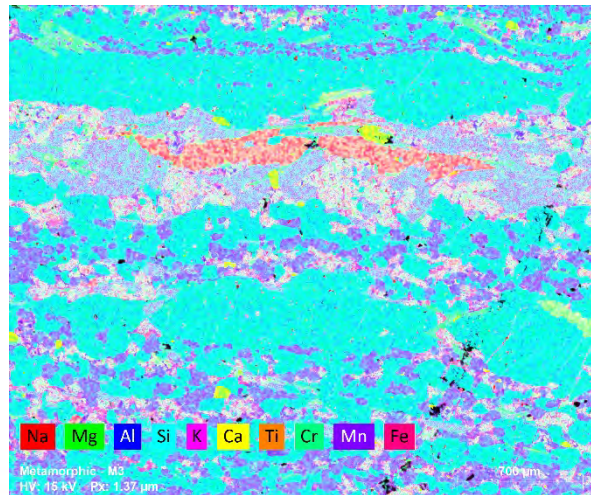
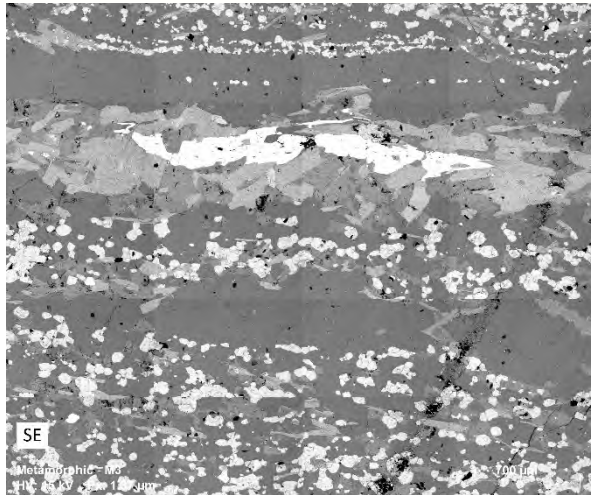
SEM-EDS: Hypermap Results Image Extension



Measurement Conditions

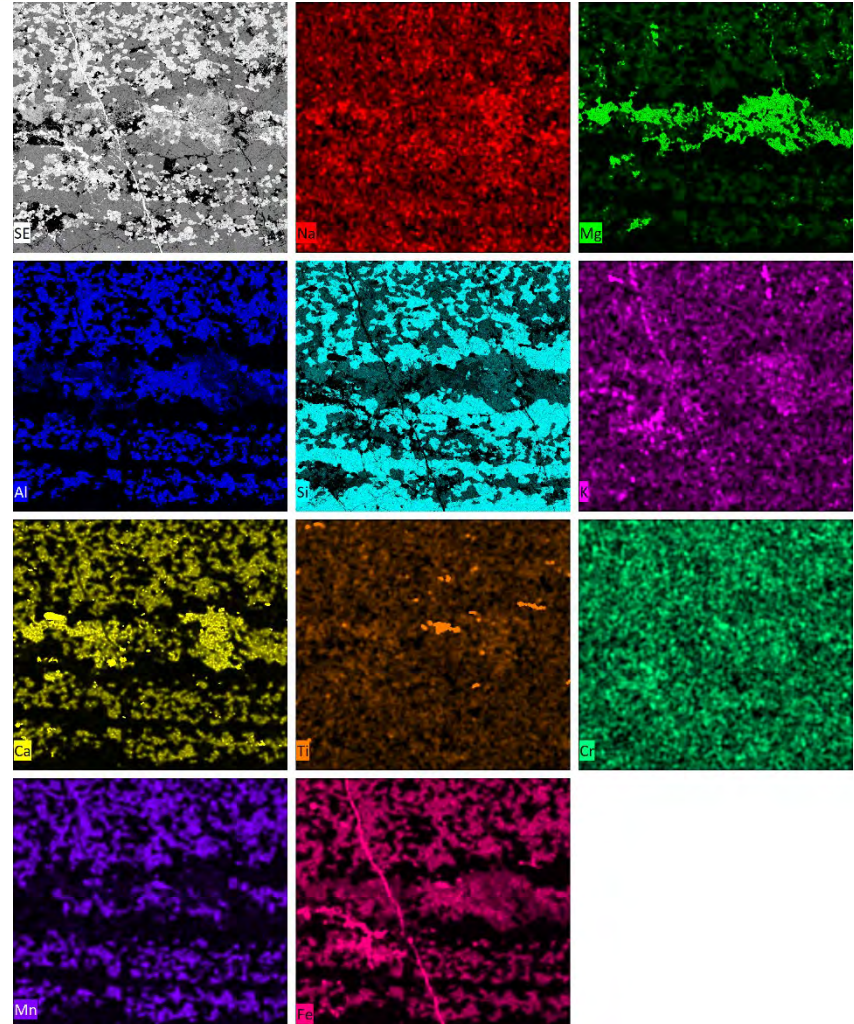
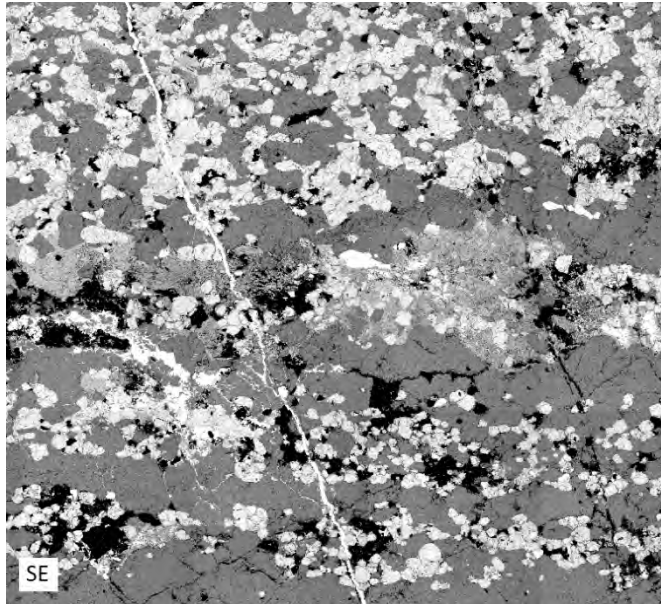
High Voltage:	15 kV
Pixels:	4000x7980
Measurement Time:	545 min
SDD:	10 mm ²
Dwell time:	1024 μs
FOV:	16 mm
Pixel size:	4 μm
Fields:	1200 (20x60)
Magnification:	250x

SEM-EDS: Hypermap Results Elemental Peak Intensity Maps



SEM-EDS: Hypermap Results

Elemental Peak Intensity Maps

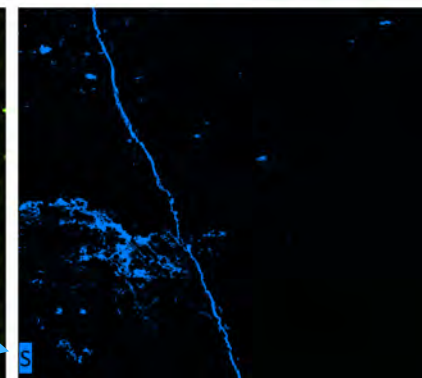
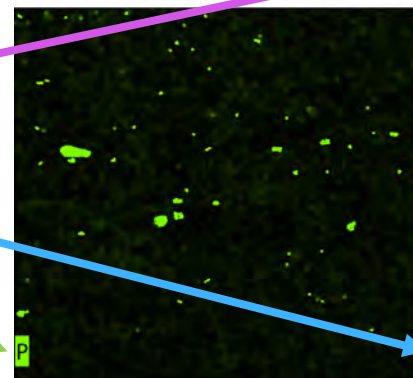
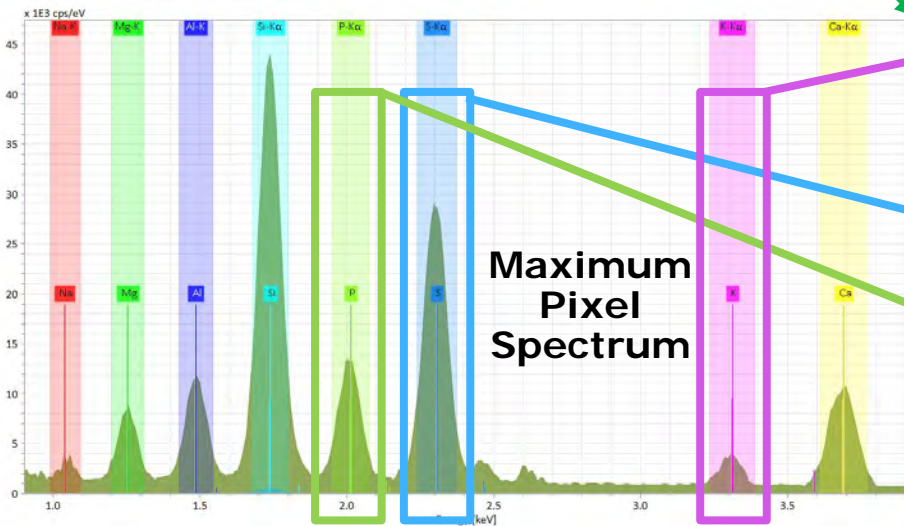
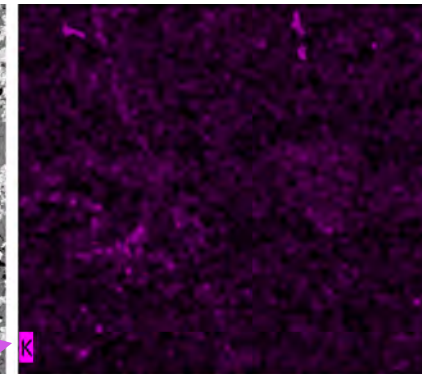
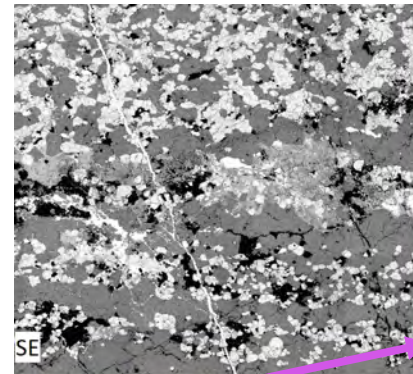
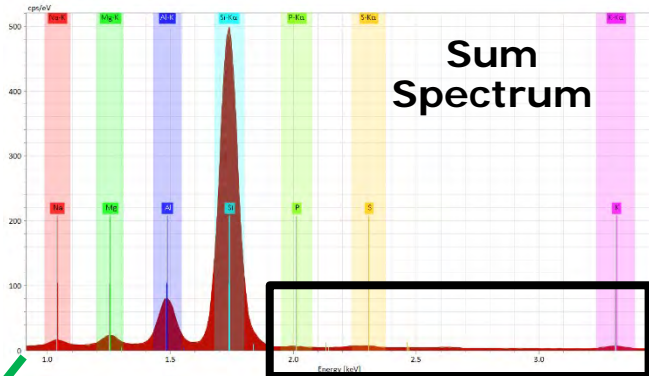


Measurement Conditions

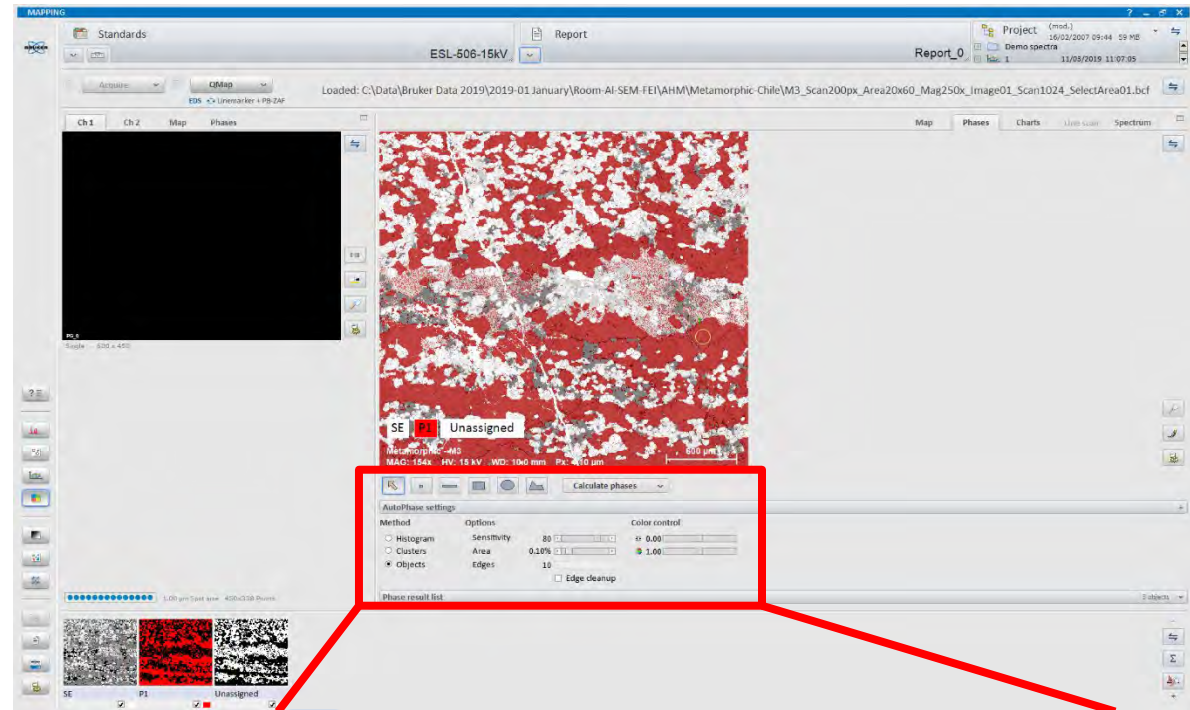
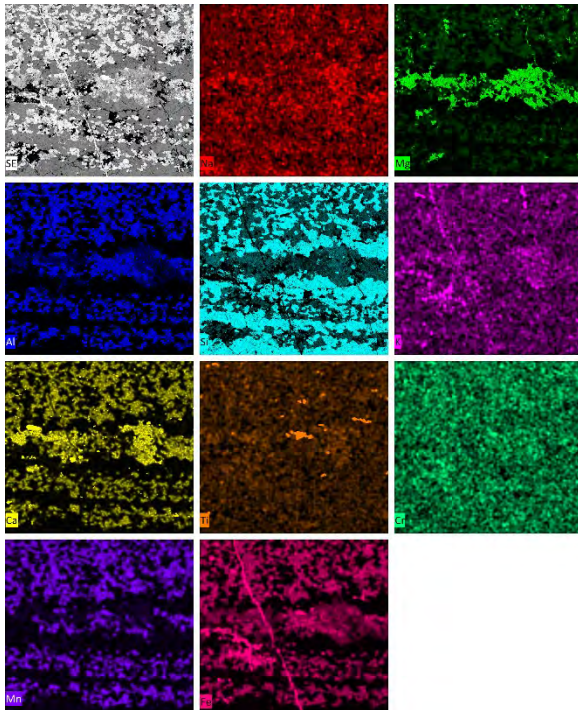
High Voltage:	15 kV
Pixels:	4000x7980
Measurement Time:	545 min
SDD:	10 mm ²
Dwell time:	1024 μ s
FOV:	16 mm
Pixel size:	4 μ m
Fields:	1200 (20x60)
Magnification:	250x

SEM-EDS: Hypermap Results

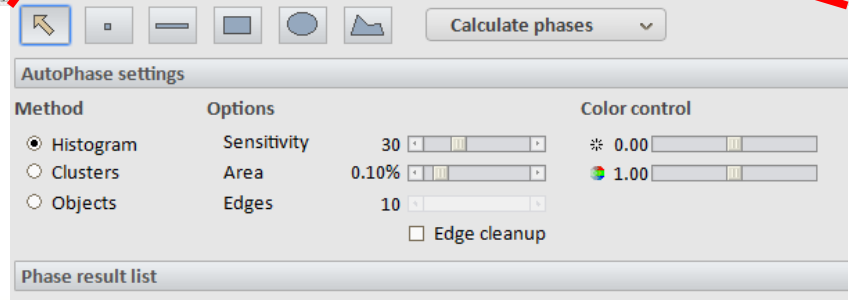
Minor Elements – Maximum Pixel Spectrum



SEM-EDS: Hypermap Results Autophase

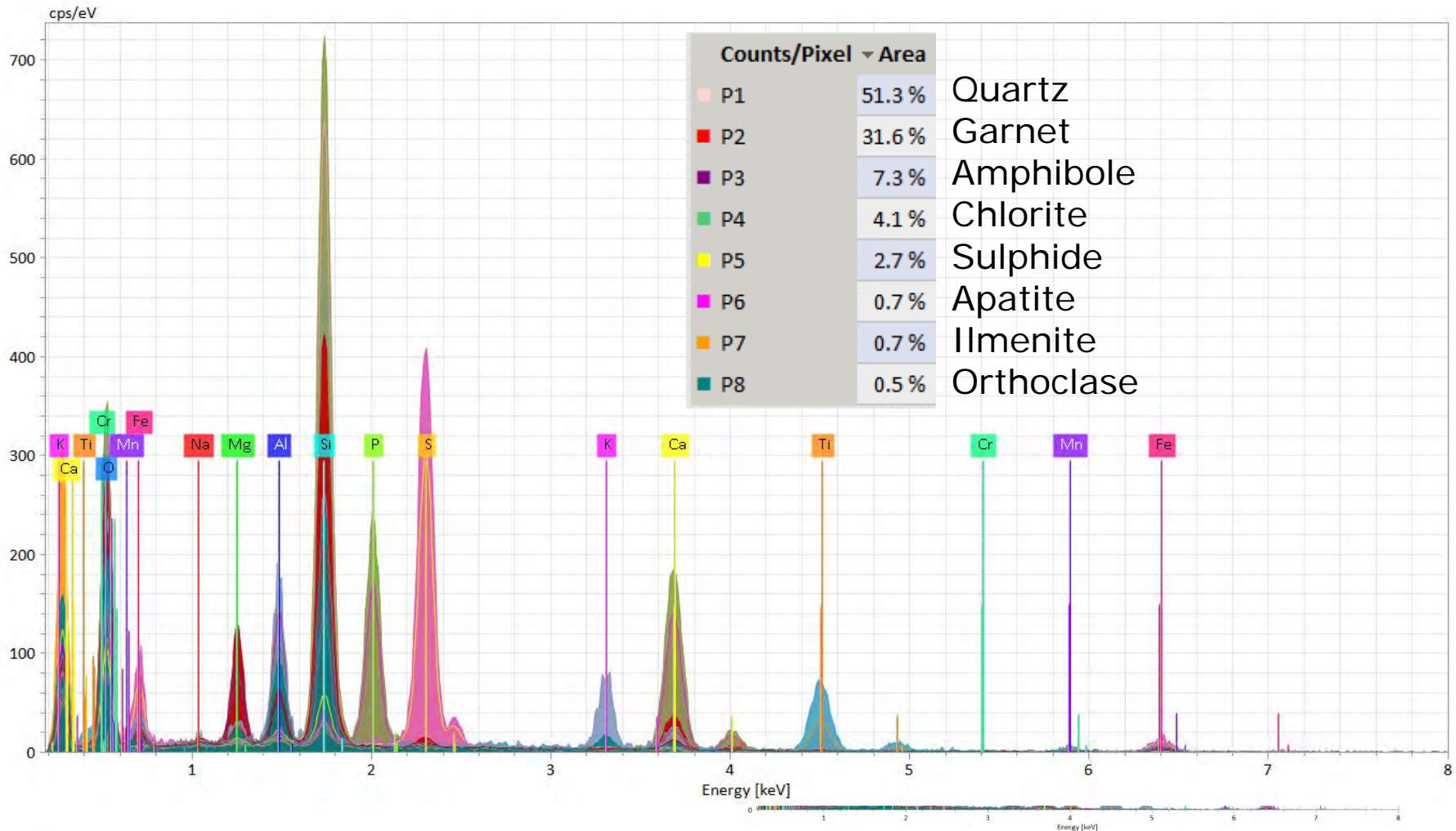


- Automatic and user-controlled phase analysis options
- Phase ratio and compositional information



SEM-EDS: Hypermap Results

Autophase



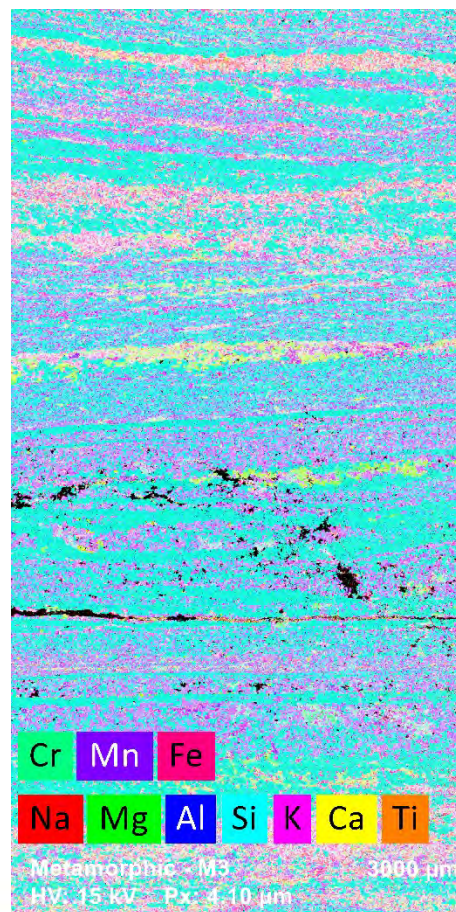
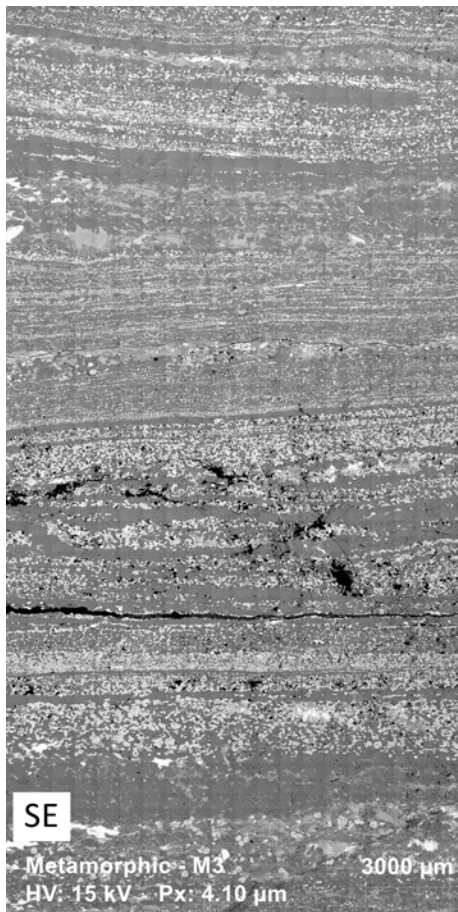
SEM-EDS: Hypermap Results Autophase



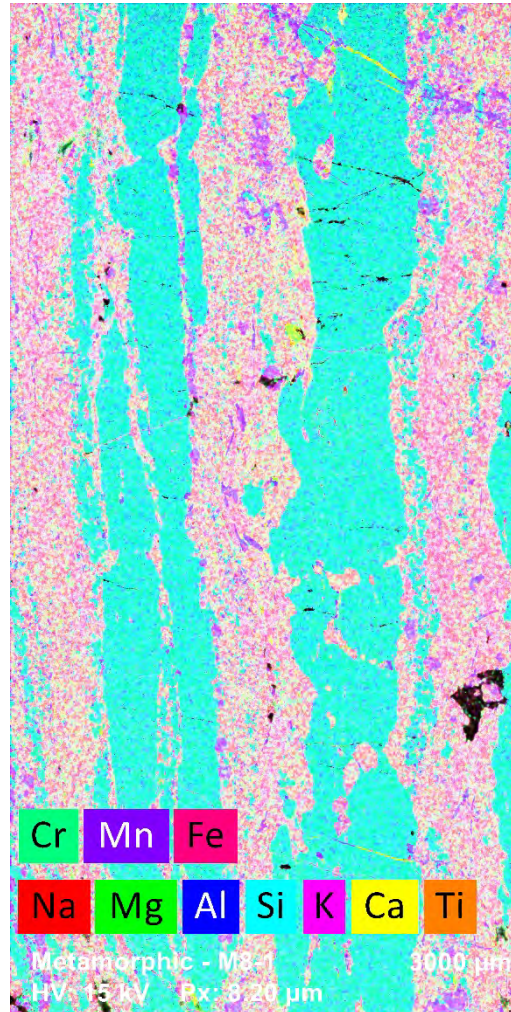
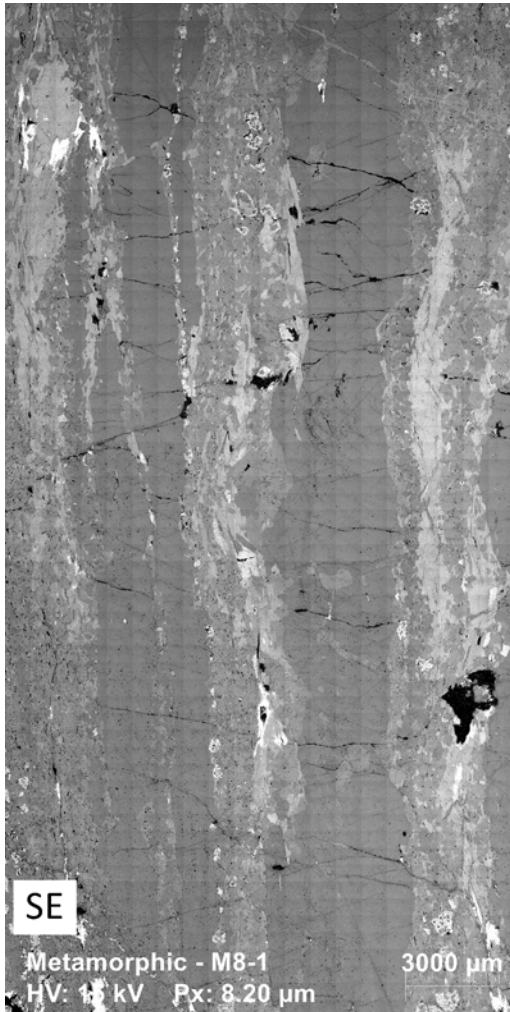
Hypermap

Elemental map

Phasemap

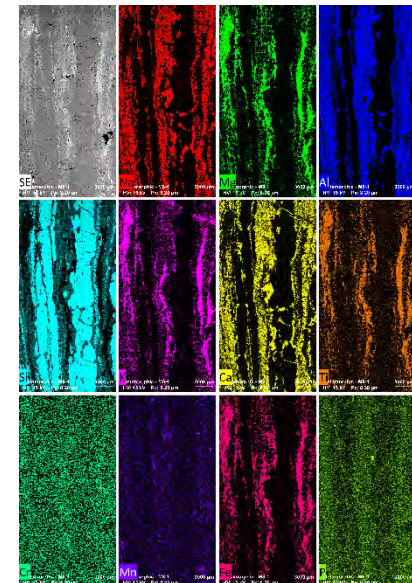


SEM-EDS: Hypermap Results Image Extension

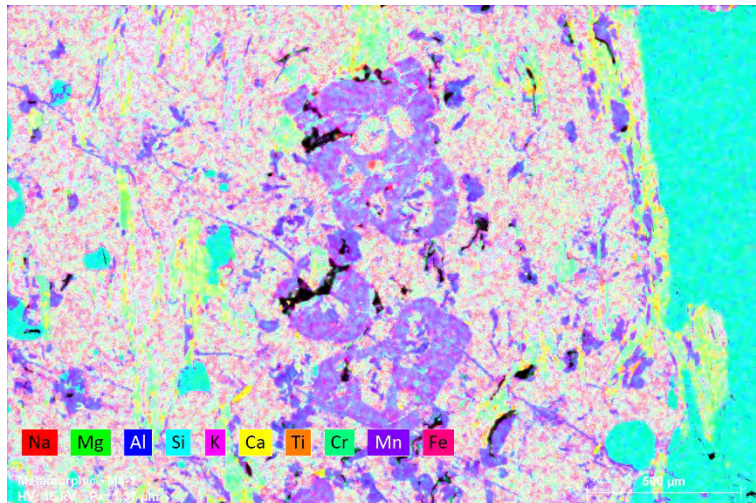
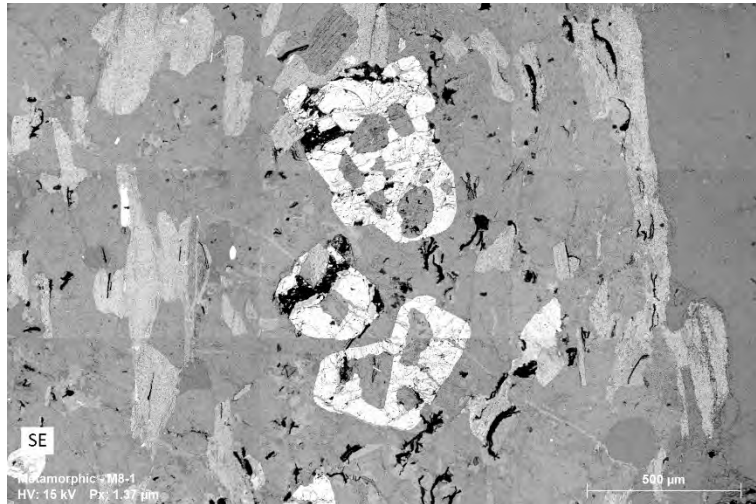


Measurement Conditions

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

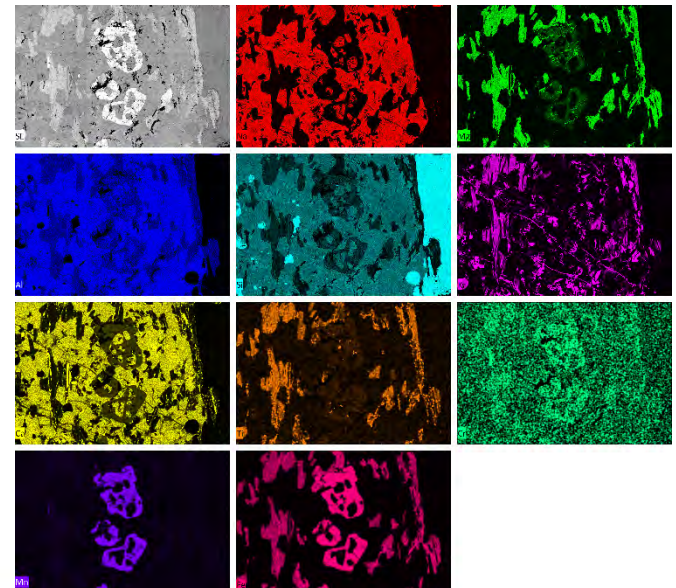


SEM-EDS: Hypermap Results Image Extension



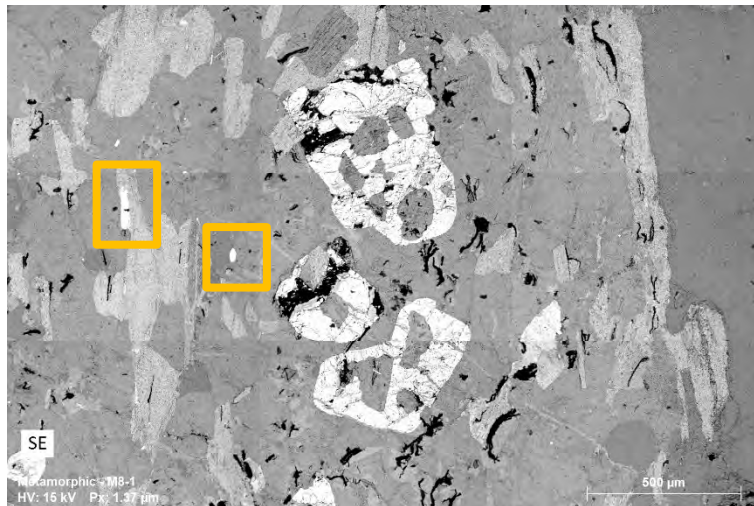
Measurement Conditions

High Voltage:	15 kV
Pixels:	1800x1200
Measurement 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

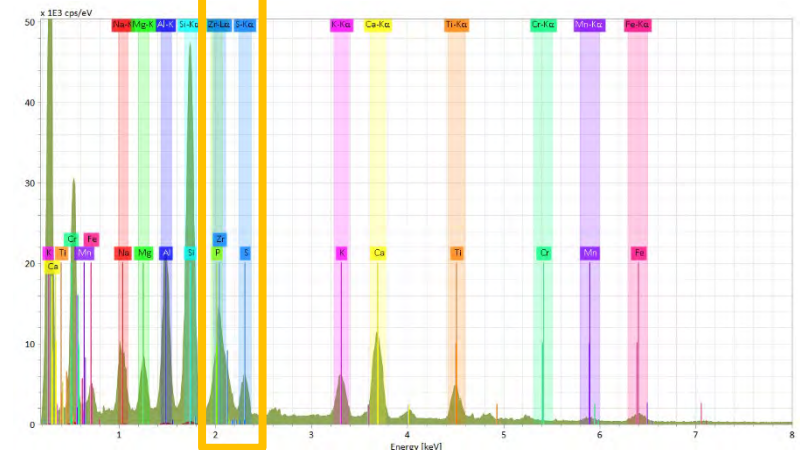
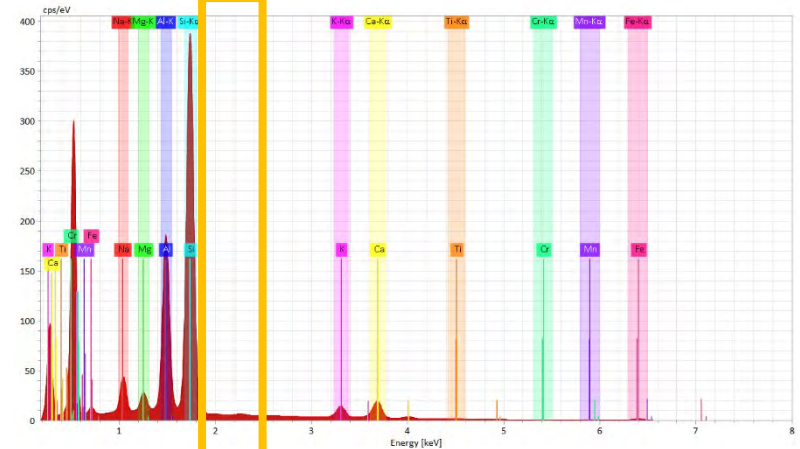
Maximum Pixel Spectrum



Measurement Conditions

High Voltage: 15 kV
Pixels: 1800x1200
Measurement Time: 10 min
SDD: 10 mm²
Dwell time: 1024 μs
FOV: 2.4 mm
Pixel size: 1.4 μm
Fields: 9 (3x3)
Magnification: 250x

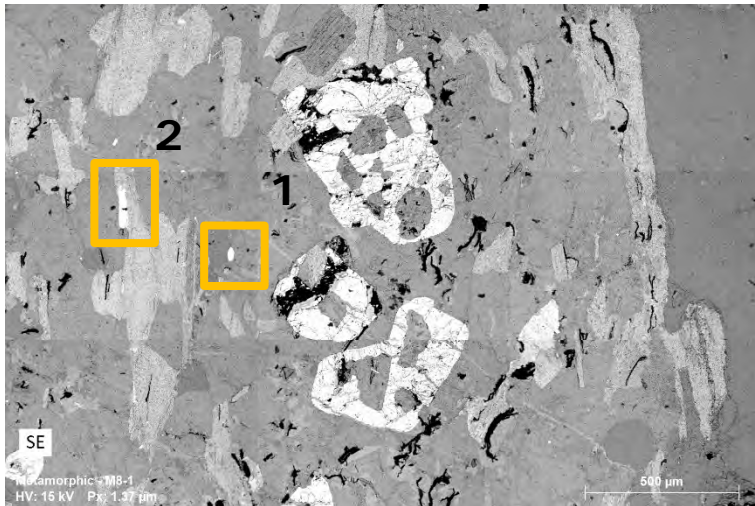
Sum Spectrum



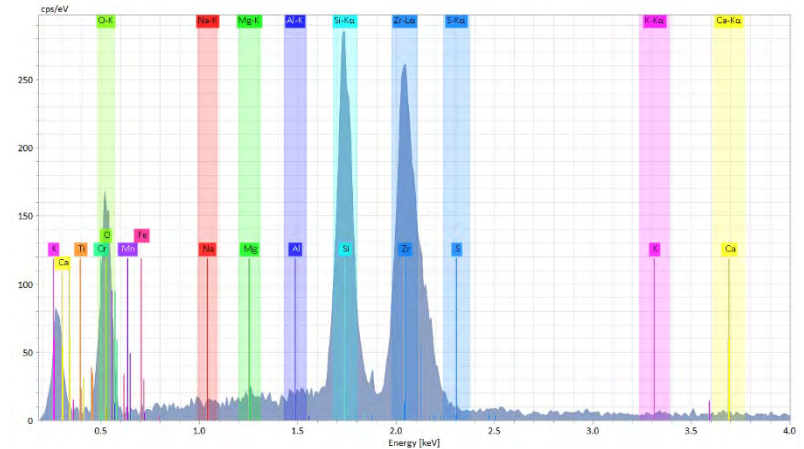
Maximum Pixel Spectrum

SEM-EDS: Hypermap Results

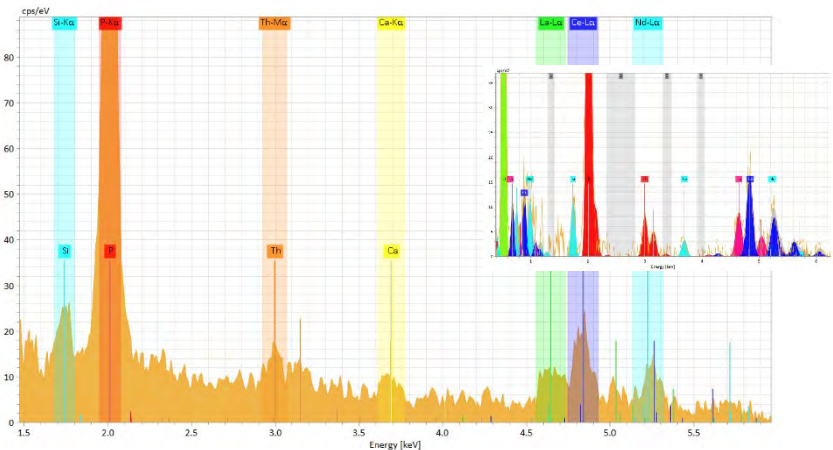
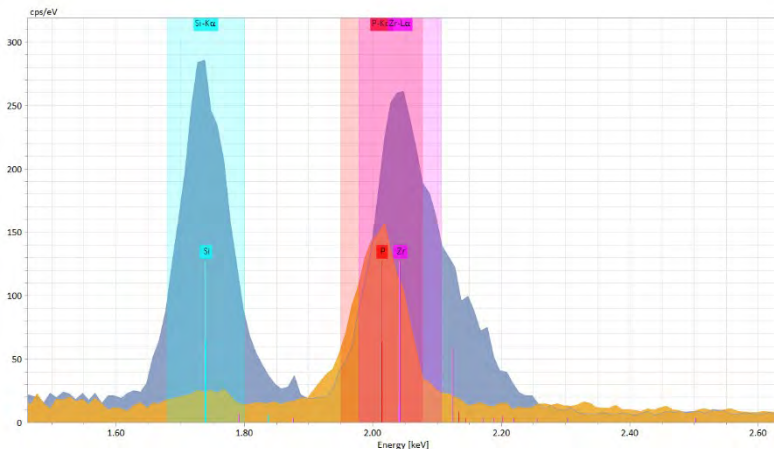
Maximum Pixel Spectrum



Object 1 - Zircon

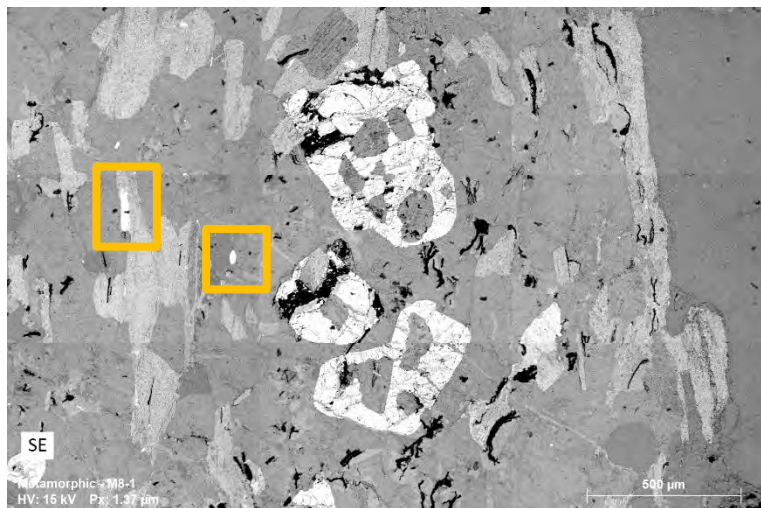


Deconvolution: Zr and P

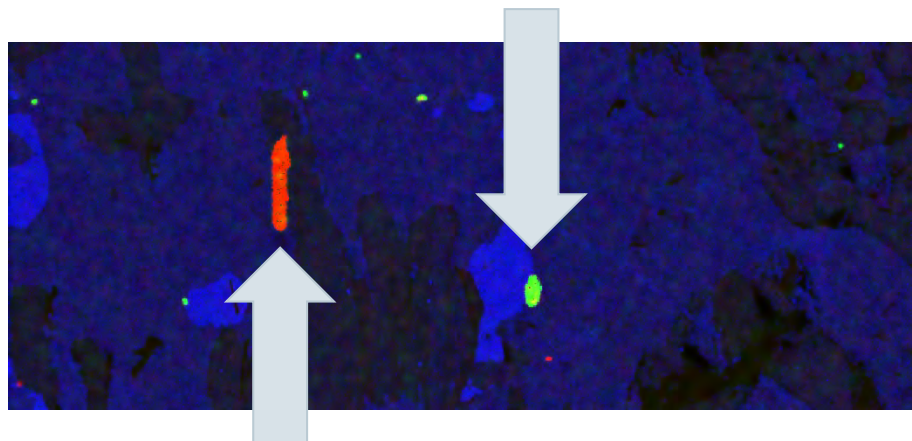


Object 2 - Monazite

SEM-EDS: Hypermap Results Deconvolution

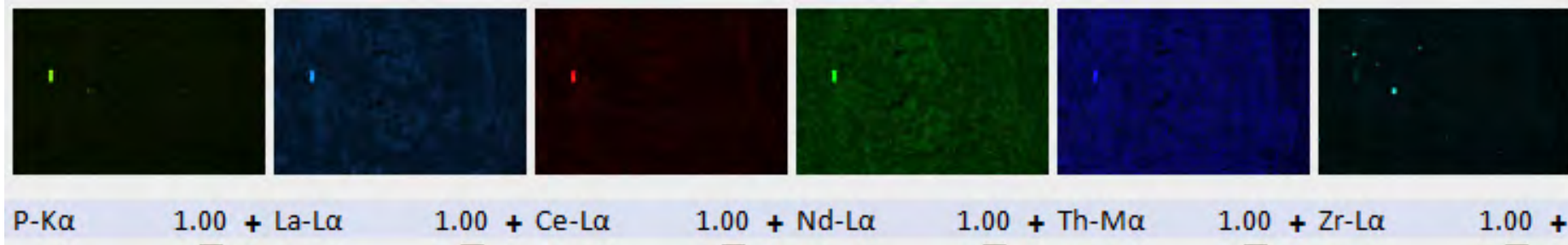


Object 1 - Zircon

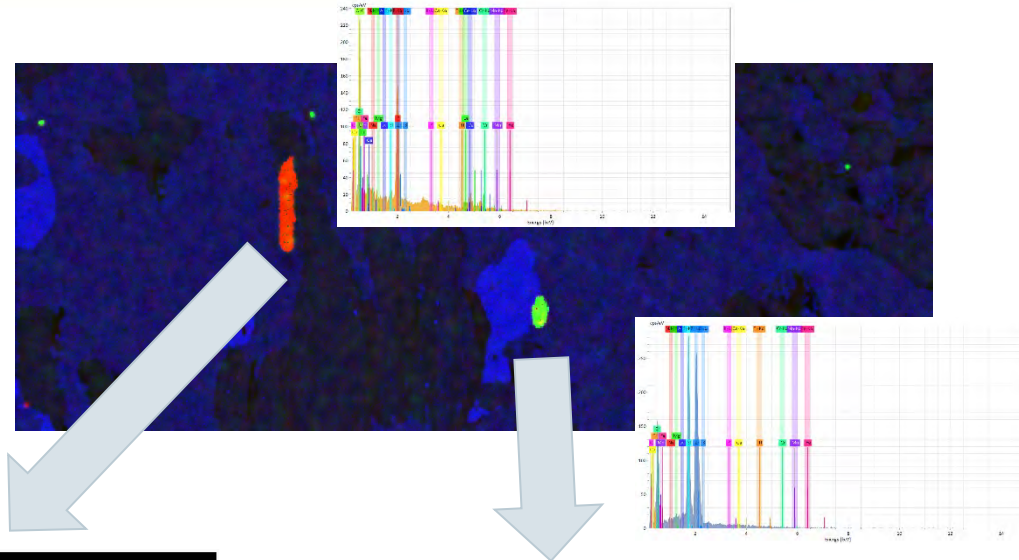
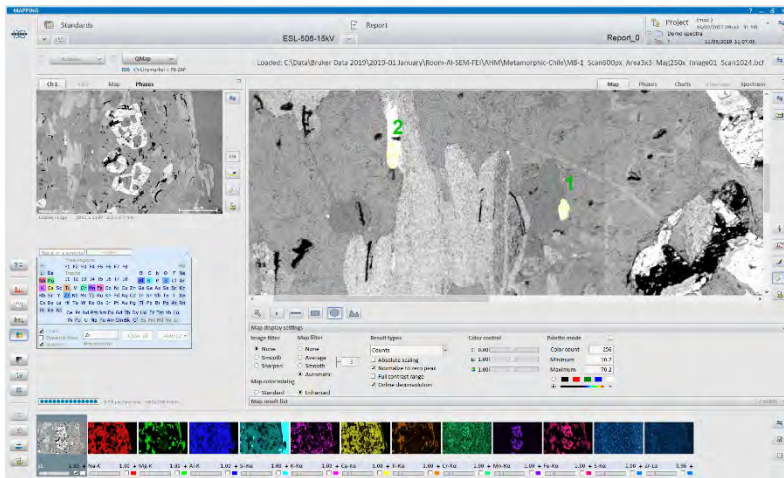


Object 2 - Monazite

Deconvolution: Zr and P



SEM-EDS: Hypermap Results Quantification



Object 2 Monazite	Concentration (wt%)
P ₂ O ₅	33.35
CaO	4.57
La ₂ O ₃	16,61
Ce ₂ O ₃	34.02
Nd ₂ O 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.
- Hypermapping and image extension are compatible 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)

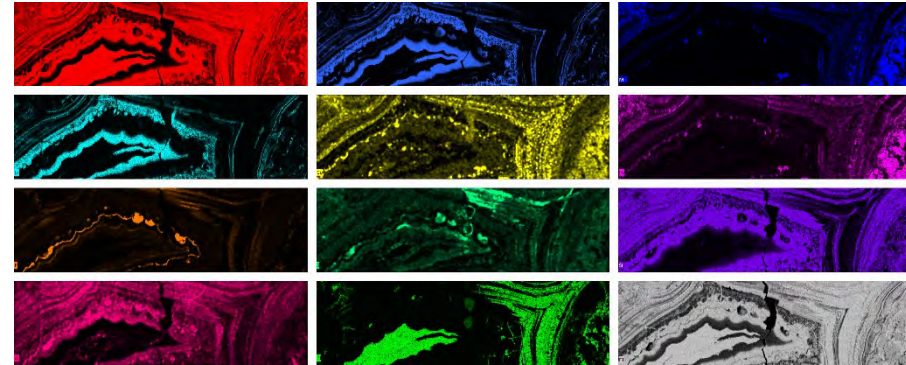
Analytical Parameters:

Tube Voltage: Rh at 50 kV

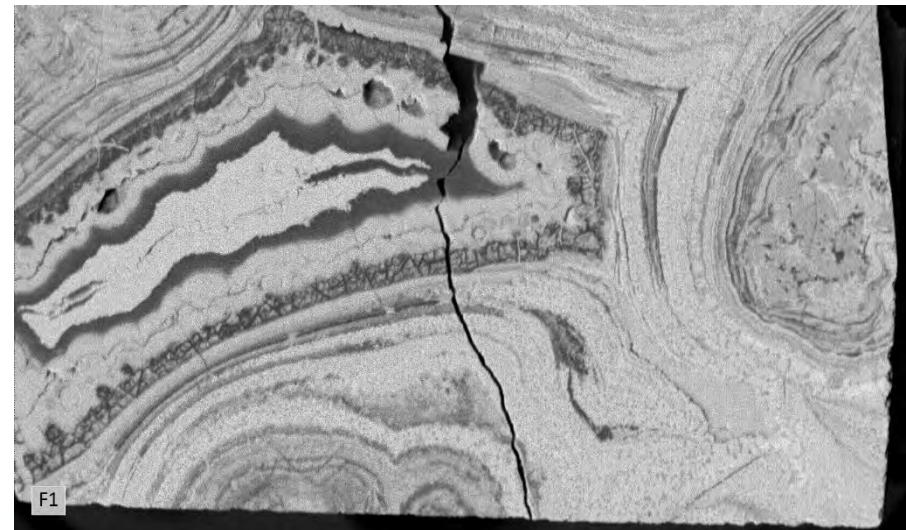
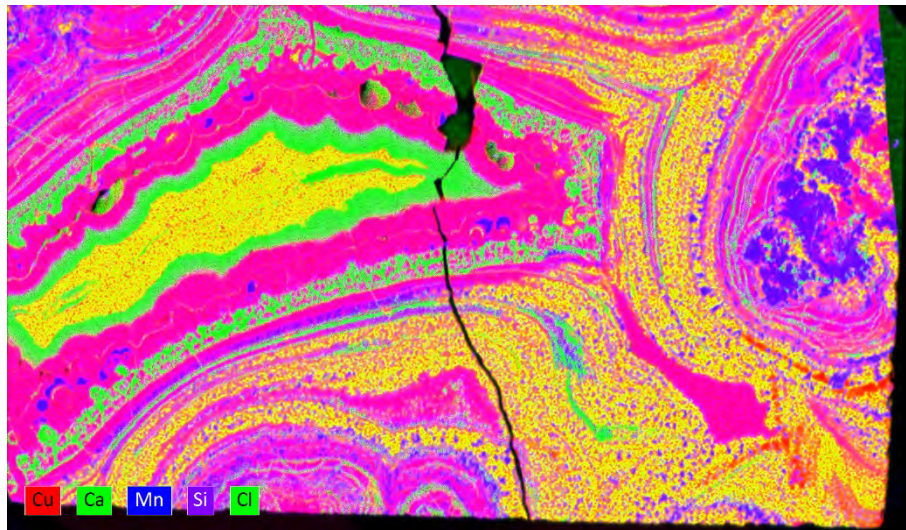
Anode Current: 600 uA

Pixel Spacing: 25 um

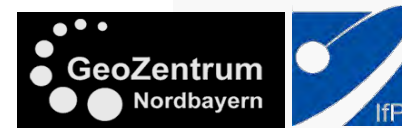
Analytical Time: 101 mins



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



Acknowledgements



Dr. Tobias Salge

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Sebastian Eade

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Mauricio Galarce

Victor Hernández

Sebastian Sola

Patricia Muñoz

Rocio Ibaceta



maini

Unidad de Equipamiento Científico



Are There Any Questions?

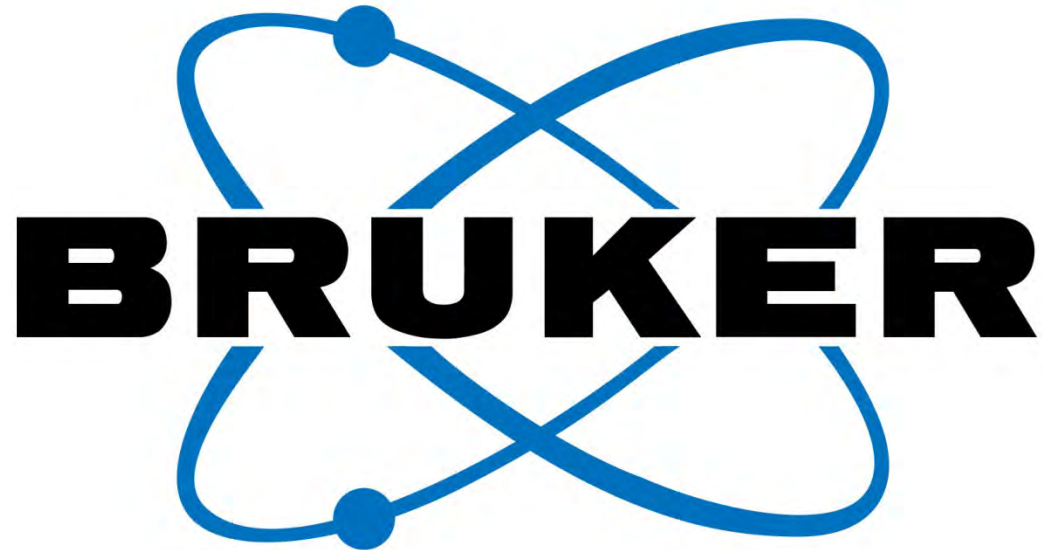
Please type in the questions you might have
in the Q&A box and press *Send*.

More Information



For more information, please contact us:

info.bna@bruker.com



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