New Possibilities: Full Range EDS Analysis with SEM-XRF

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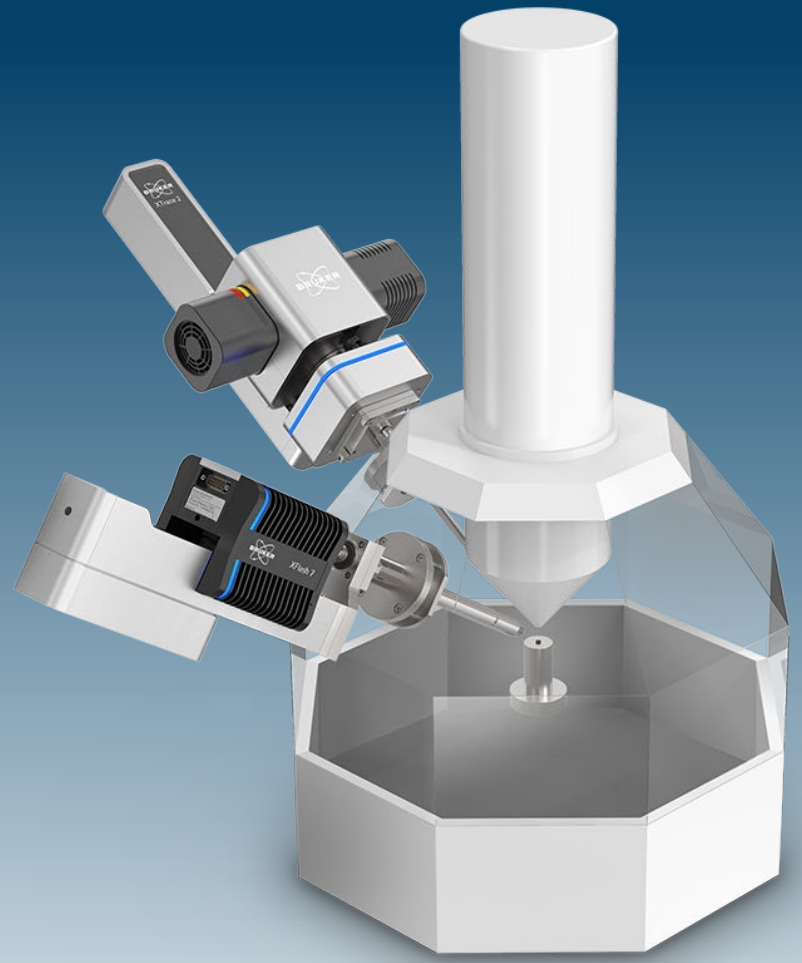

Presenters

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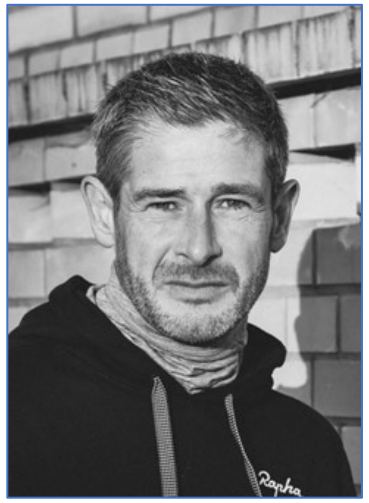

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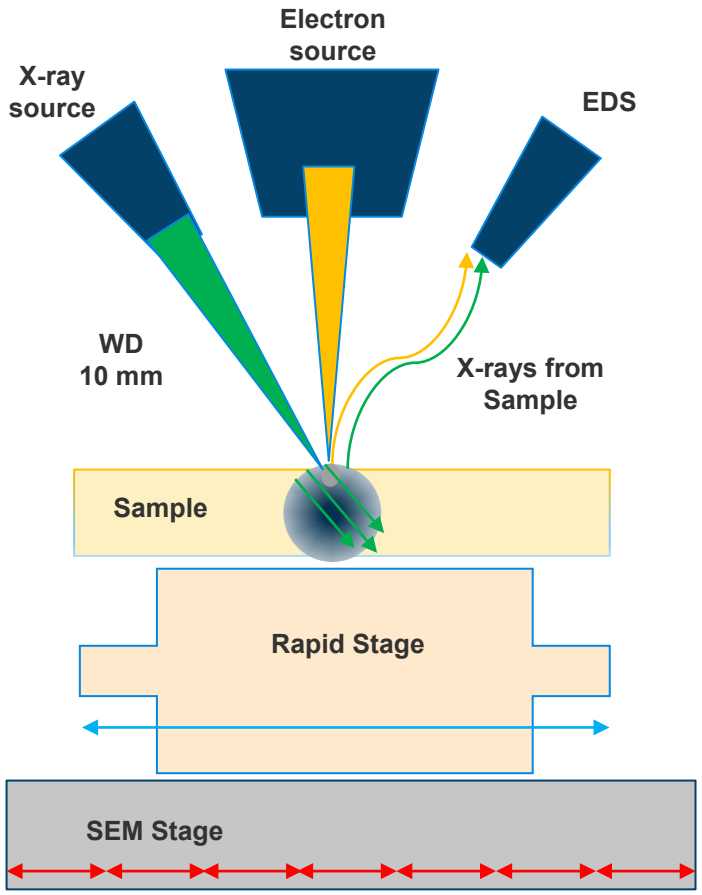
Bruker´s Full Range EDS Analysis

Bruker´s Full Range EDS Analysis

Obtain additional sample information with your Bruker EDS detector

- Full Range EDS is Bruker's name for analysis using dual excitation of micro-XRF and electron beam sources combined with an EDS detector.
- Converts the SEM to a dual-excitation system, where samples interact with either the electron beam of the SEM or the X-ray photons from the micro-XRF source, or both.
- The EDS can benefit from the advantages of each individual source and can now see sample information which cannot be obtained by conventional electron beam EDS analysis
- Full Range EDS is an analytical solution for providing a more complete elemental composition with a high dynamic range from major to trace elements



2 Excitation Sources:

Electron Beam (e-beam)

Micro-XRF (X-ray beam)

1 Detector:

Energy Dispersive Spectrometer (EDS)

2 Stages:

SEM Stage

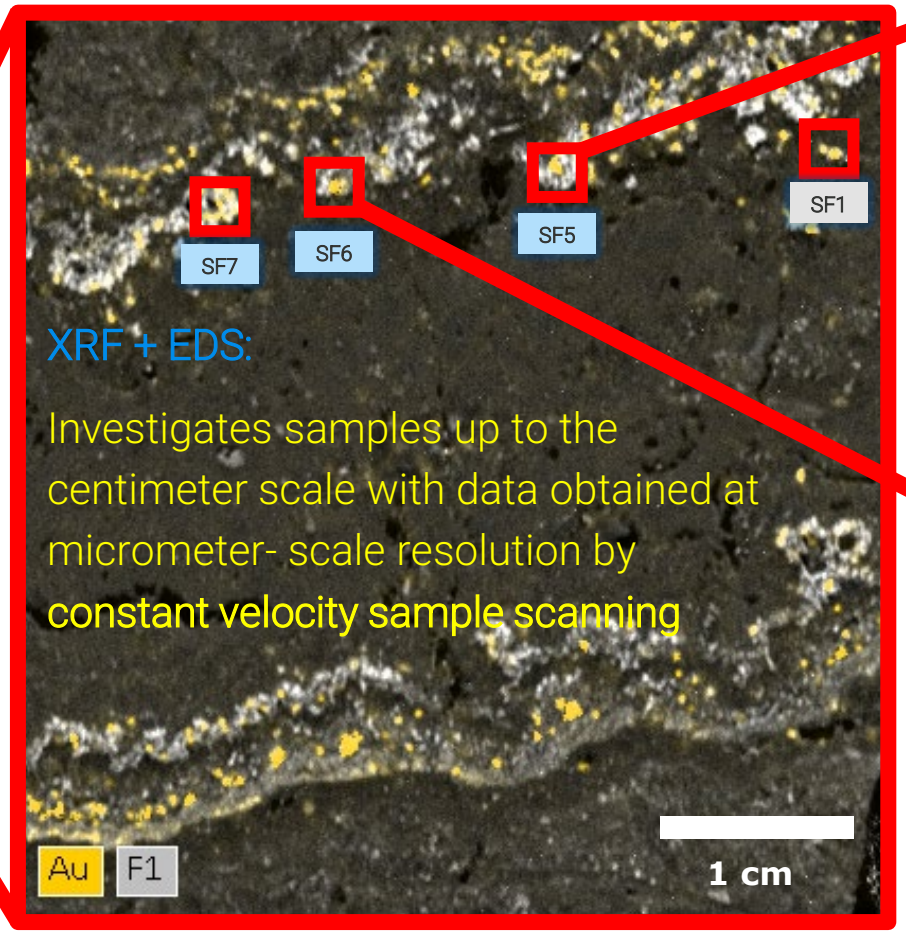
Rapid Stage

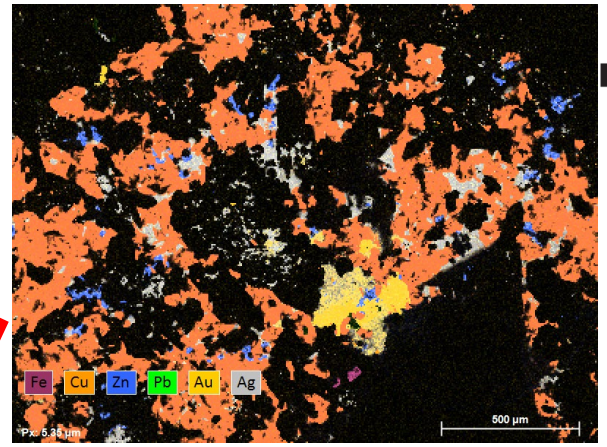
Full Range EDS
Builds Bridges Across Analytical Scales

Sample dimension:
4.5 x 4.5 cm

Gold-bearing sample from Karangahake Gold Mine, New Zealand.

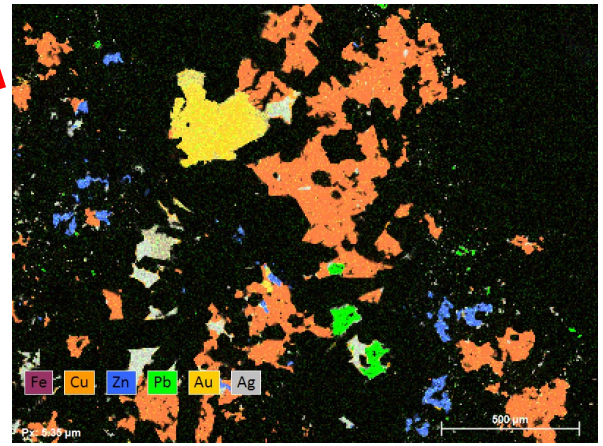
Sample courtesy of Jeff Mauk



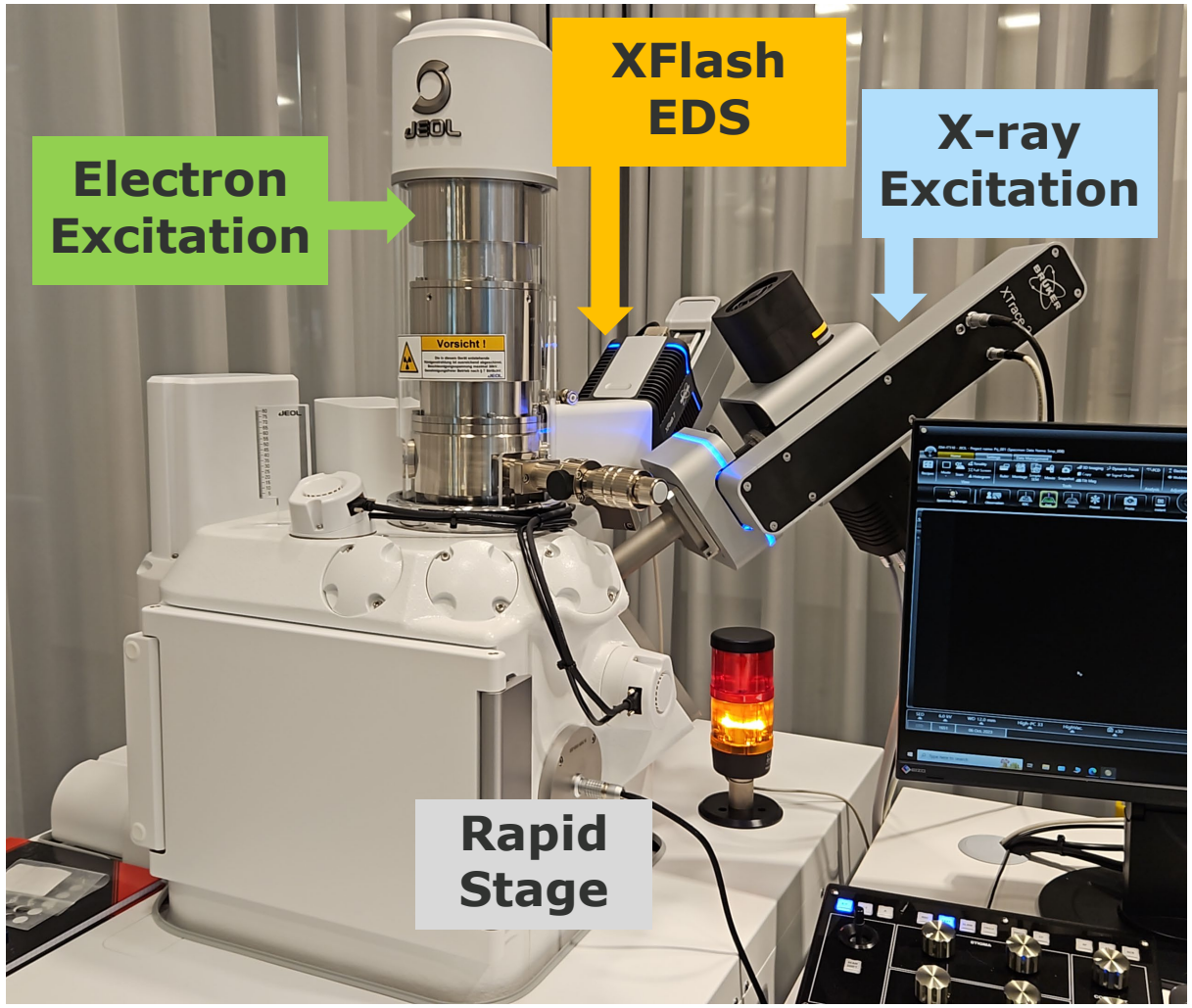


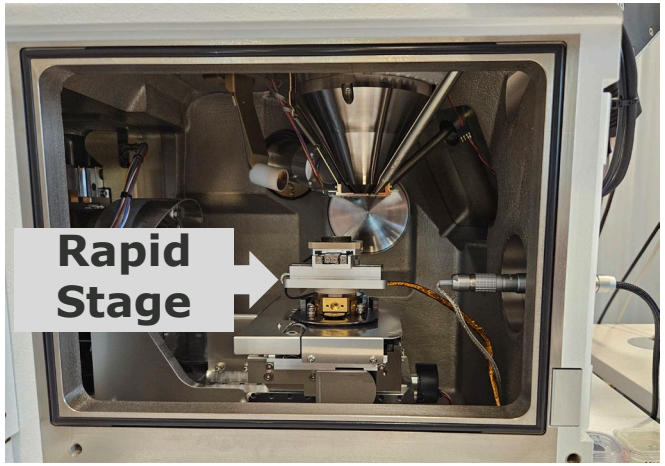
Electron beam + EDS:

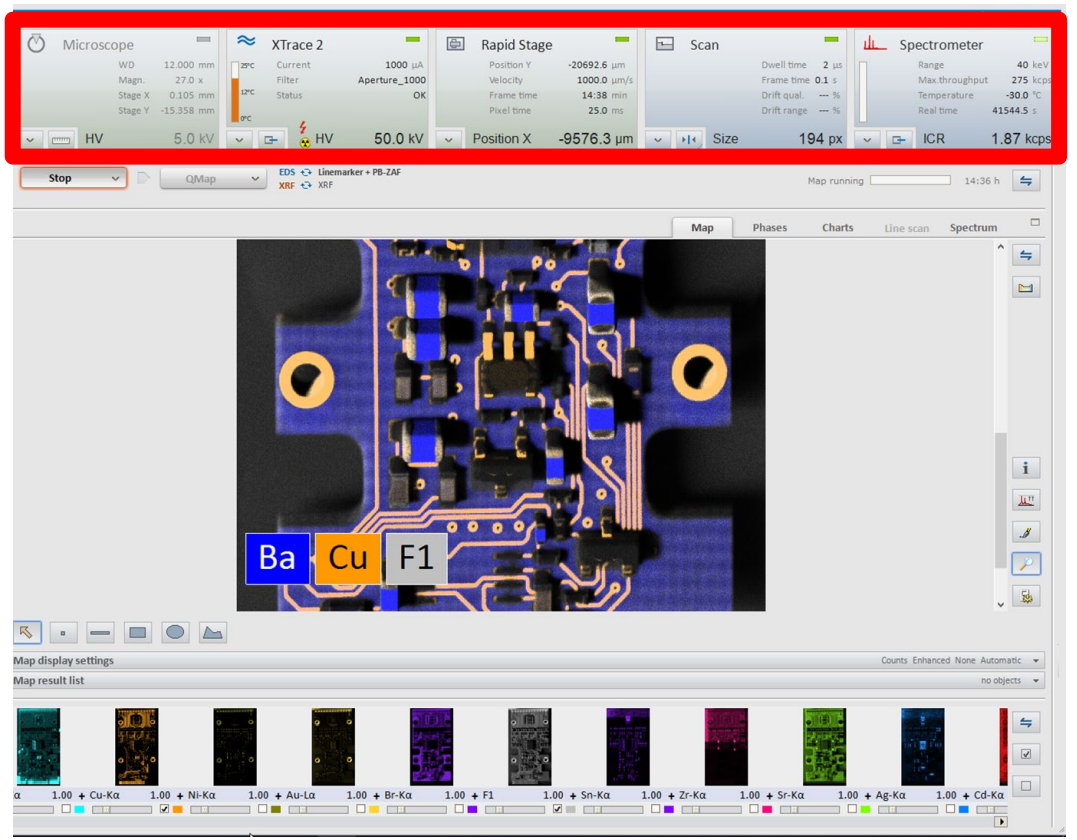
High spatial resolution to resolve small sample structures in the lower micrometer down to nanometer range by **e-beam rastering**



Full Range EDS
SEM setup and Analysis Software







Full Range EDS Enables the EDS detector to…

See much lower detection limits

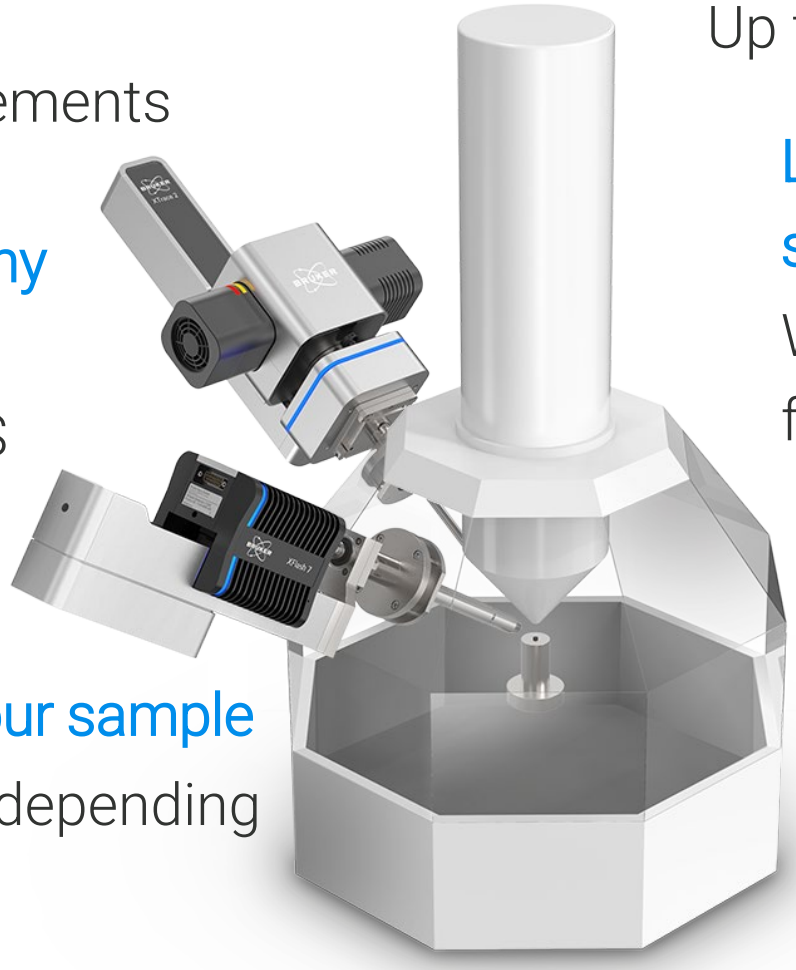
Down to 10 ppm for specific elements

Visualize samples with topography

By changing the focal range to several millimeters with the AMS system

Obtain Information from within your sample

From millimeters to centimeters depending on the sample matrix



See High Energy X-ray line series

Up to K-lines of 40 keV

Large area maps acquired in a single image

Without image mosaicing effects for both e-beam and XRF maps

Analyze thin films and multi-layered structures

Ranging from 1 nm up to 40 µm (no cross-section view)

Full Range EDS

All those additional sample information are accessible without time consuming sample preparation work, since:

- Micro-XRF is non-destructive
- No sample charging effects at any XRF High Voltage
- Micro-XRF provides both sample and volume sample information, hence samples doesn't need to be polished
- Works even on samples with rough surfaces

It works on a variety of samples types, this also includes samples that can be very challenging for SEM´s, such as:

- Light atomic number materials (powders, plastics which could be damaged by the e-beam)
- Large area samples with rough surfaces or topography
- Beam sensitive samples
- No sample coating required!

Full Range EDS Analysis with SEM-XRF: Background

Full Range EDS Analysis
Introduction

2 Excitation Sources:

Electron Beam (e-beam)

Micro-XRF (X-ray beam)

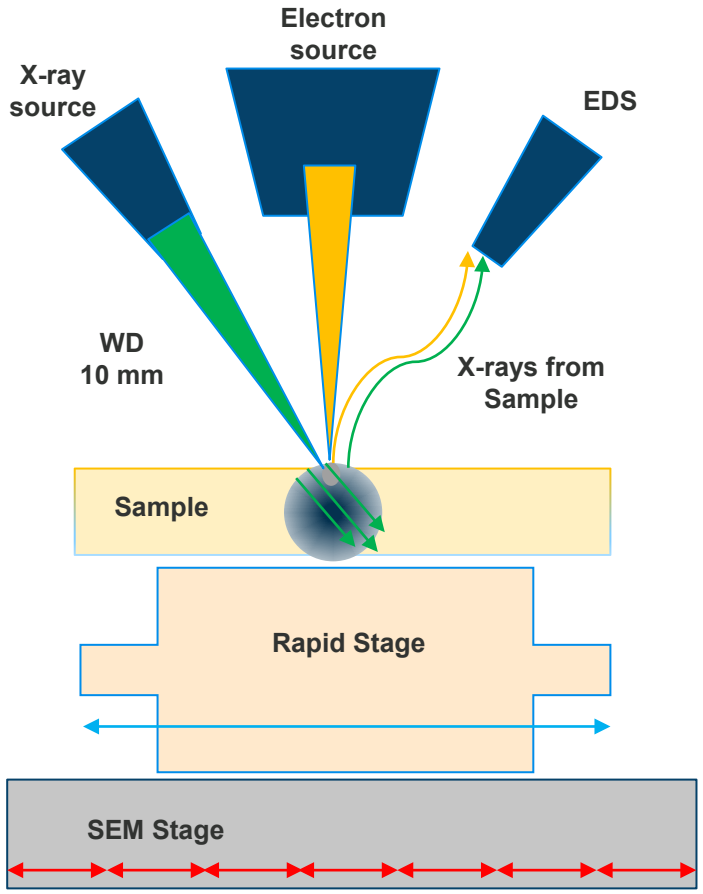
1 Detector:

Energy Dispersive Spectrometer (EDS)

2 Stages:

SEM Stage

Rapid Stage



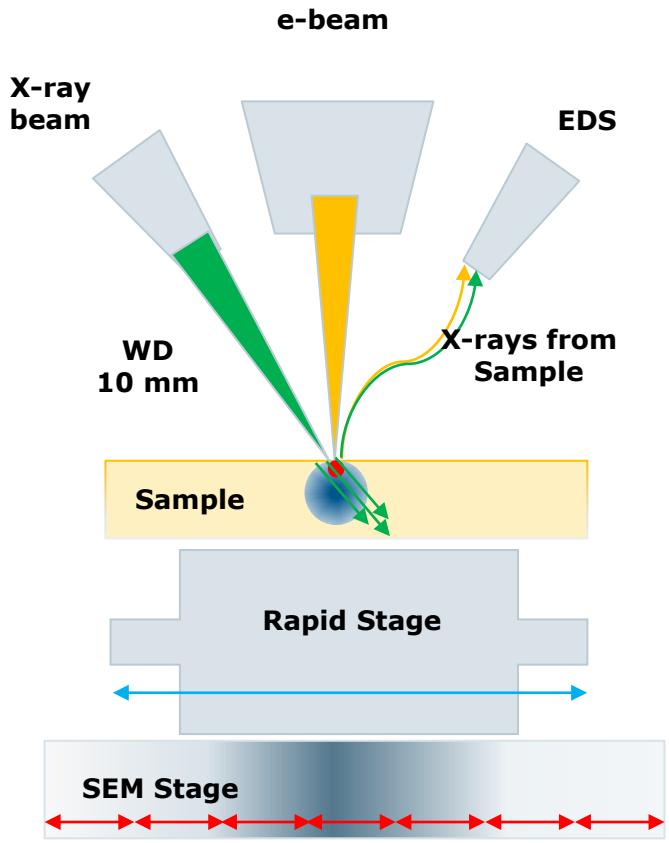
Micro-XRF Benefits:

- Non-destructive analytical technique
- No charging effects
- Minimal Sample Preparation Required
- Lower detection limits (down to 5 ppm)
- High Energy Lines Detection (Full Spectrum Range up to 40 keV)
- Ideal for Low kV or Beam sensitive samples
- Fast Large Area Mapping
- Micrometer scale measurement over cm

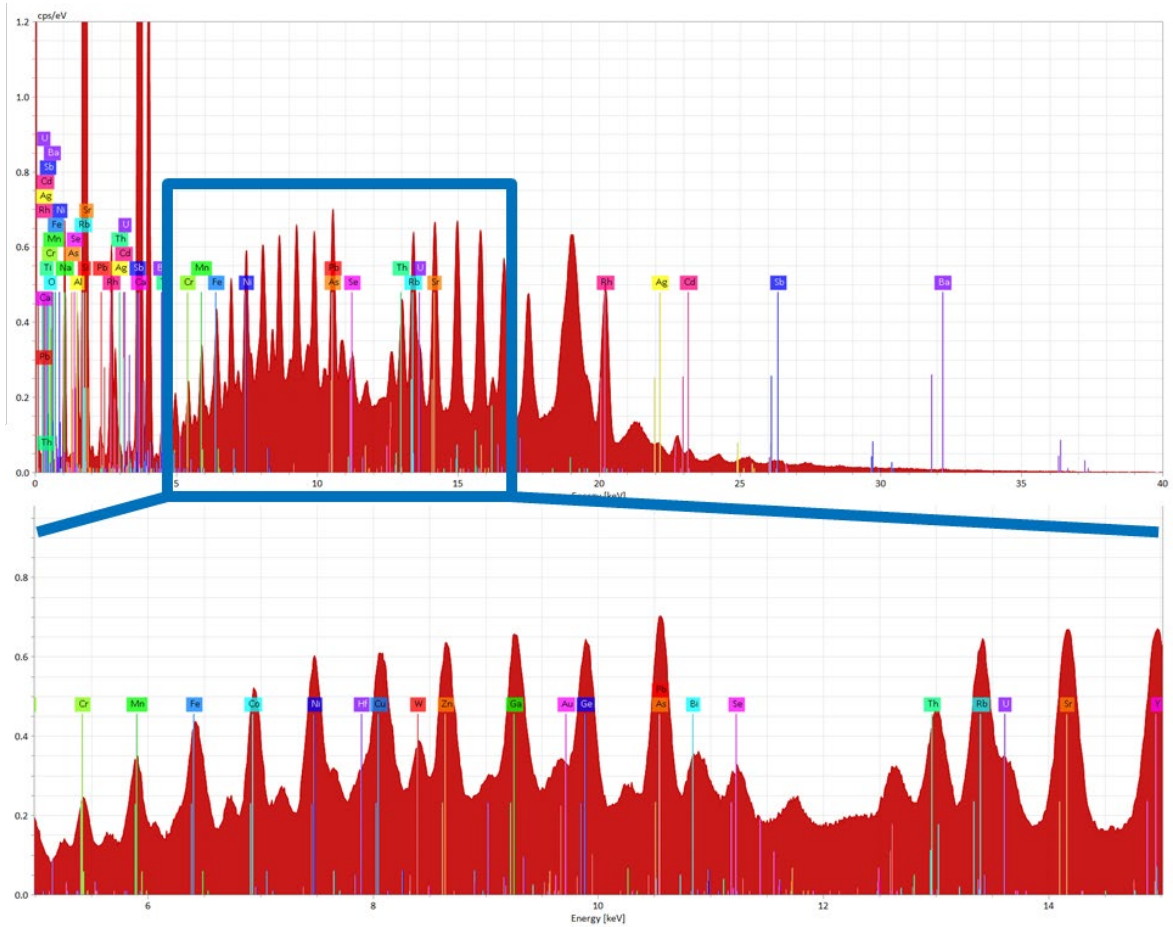
Full Range EDS Analysis

Analytical Parameters and Conditions: SEM-EDS vs SEM-XRF

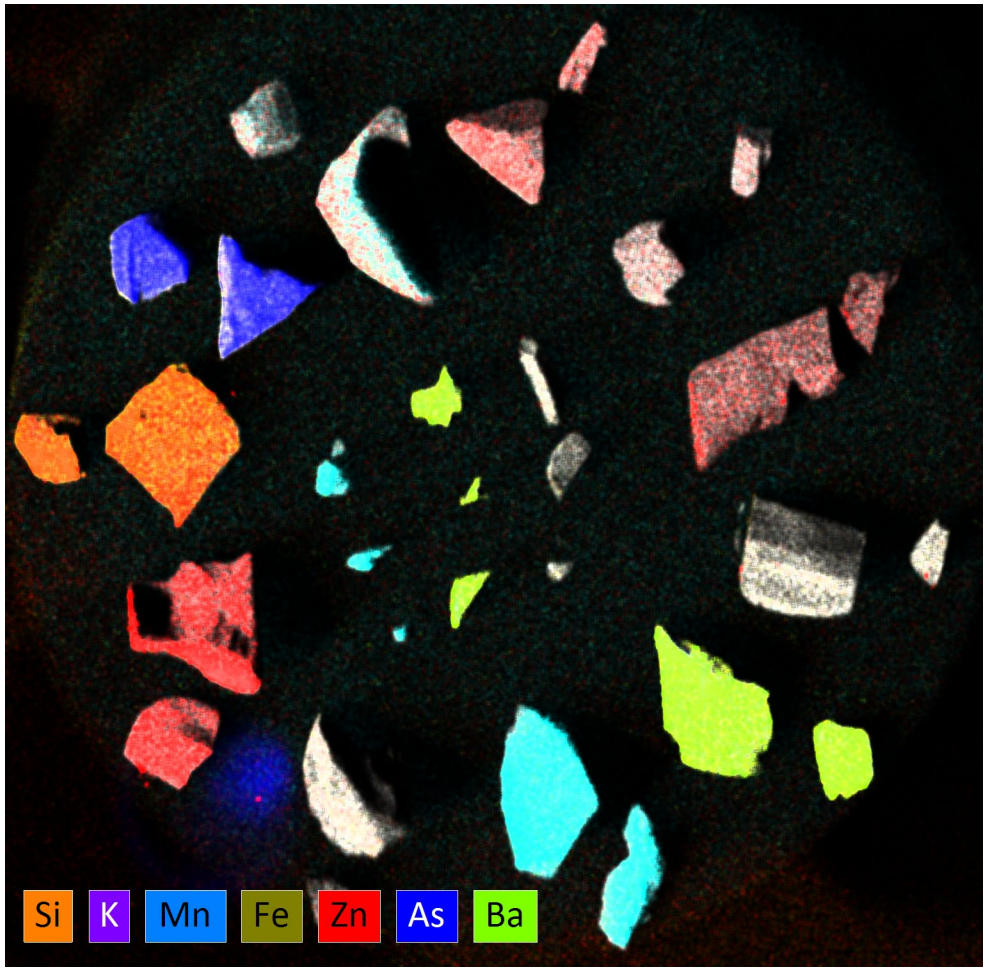
Parameter	E-beam (SEM-EDS)	Micro-XRF (SEM-XRF-EDS)
Spatial Resolution & Analyzed Volume	Ø: few µm Information depth: µm; (depending primarily on electron energy)	Ø: 15-30 µm Information depth: µm to mm; (depending on analysed element and matrix)
Detectable Elements	Atomic number Z ≥ 4 (beryllium)	Atomic number Z ≥ 6 (carbon)
Energy range	K- L –M – Lines (up to 20 keV)	K- L –M – Lines (up to 40 keV)
Concentration Range	Down to 1000 ppm	Down to 5 ppm
Quantification	Standard less and Standard based	Standard less and standard based
Data collection	Simultaneously	Simultaneously
Sample Preparation	Sample needs to be electrically conductive (commonly carbon-coated), polishing required	Electrical Conductivity not required, samples doesn´t need to be polished
Sample stress	Heated due to absorbed electrons	minimal
Spectroscopic resolution	Down to 121 eV for Mn Ka	Down to 121 eV for Mn Ka
Distribution Measurements	By rastering e-beam	By continuously (Rapid) Stage movement since the X-ray optic is fixed in space



Trace Elements



Glass analysis: NIST 610



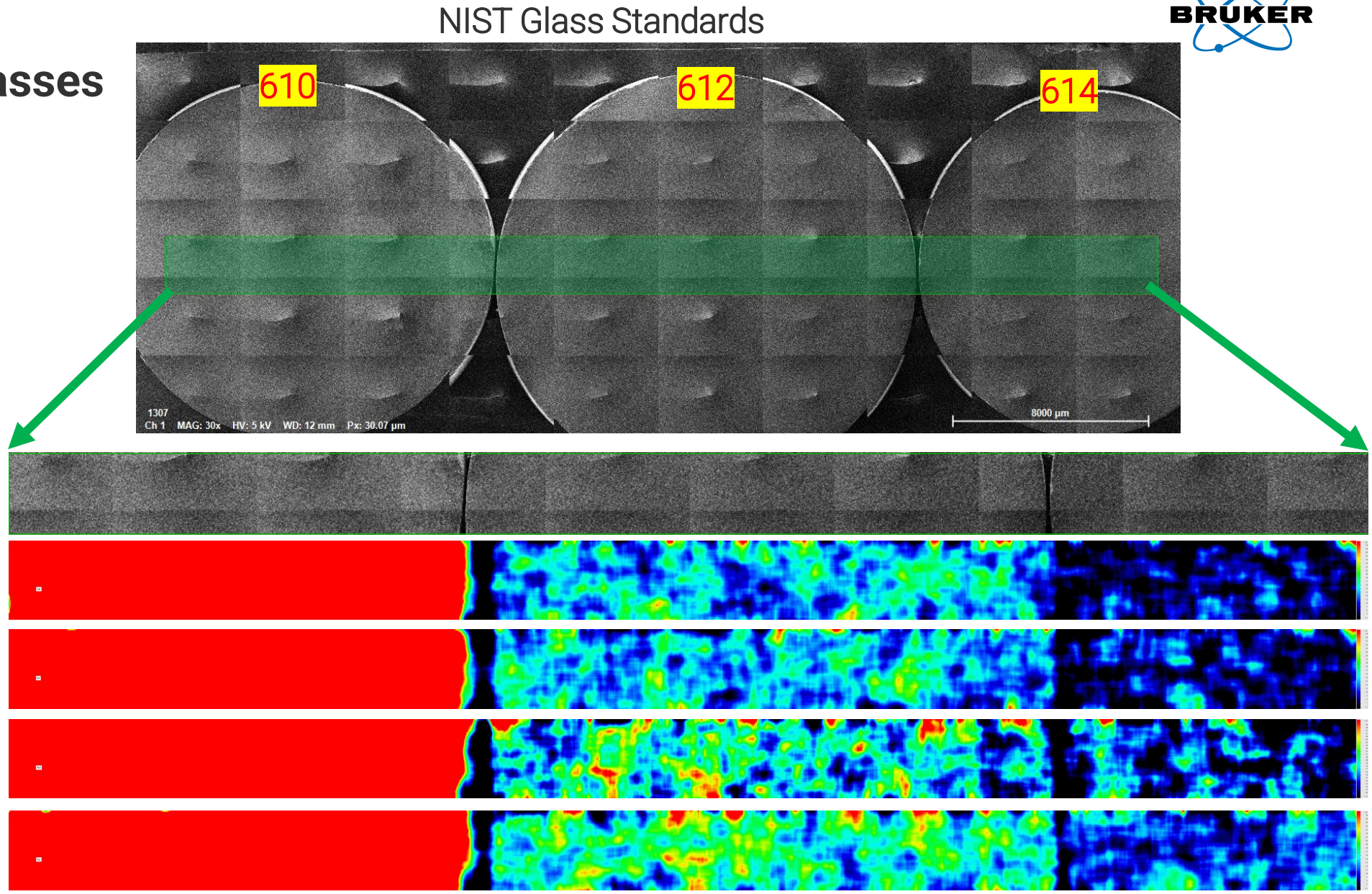
Forensics: Glass fragment analysis

NIST Standard Glasses

Analysis of NIST Standard Glasses with dopped concentrations in the approximate range of:

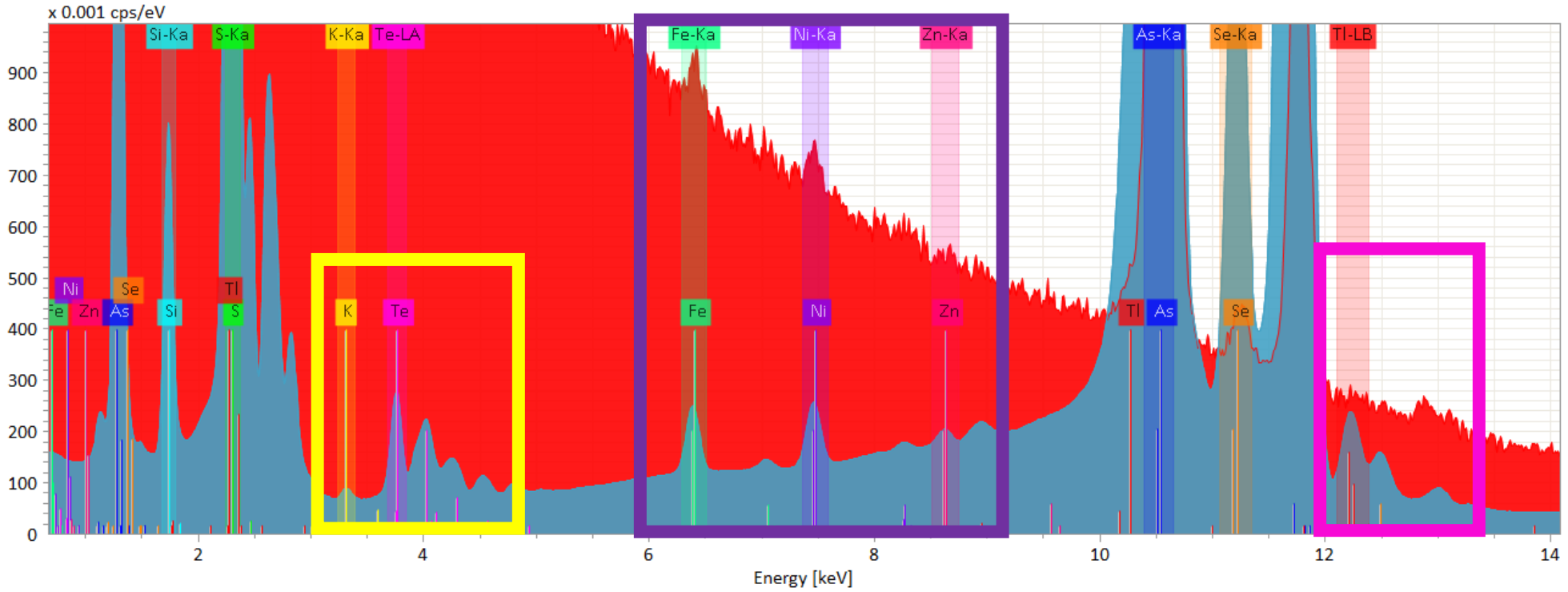
NIST 610: 500 ppm
NIST 612: 50 ppm
NIST 614: 5 ppm

Easily identify trace element concentrations

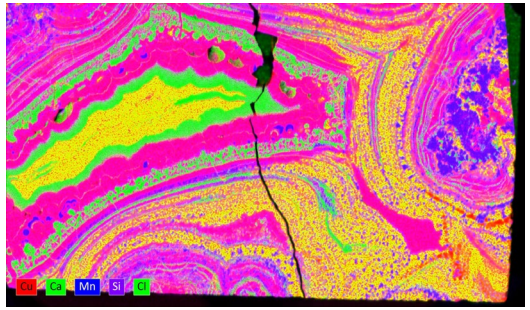


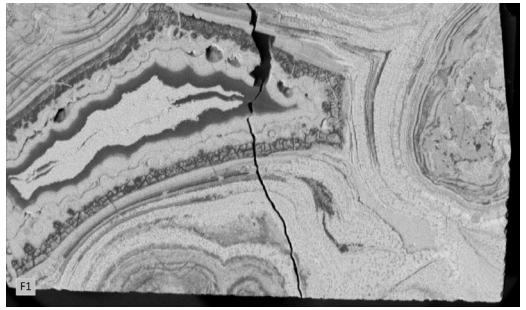
Geological Applications: Volcanic Fumeroles

- Comparison between XRF-EDS (XTrace) and SEM-EDS (e-beam) total map spectra, which highlights the capabilities of the XRF-EDS to detect trace concentrations of elements not visible in the SEM-EDS spectrum.
- Note the distinct peaks observable in the X-Ray beam image (blue) at various energies that are not present in the e-beam spectrum (red), and the significantly lower noise in the x-beam spectra.

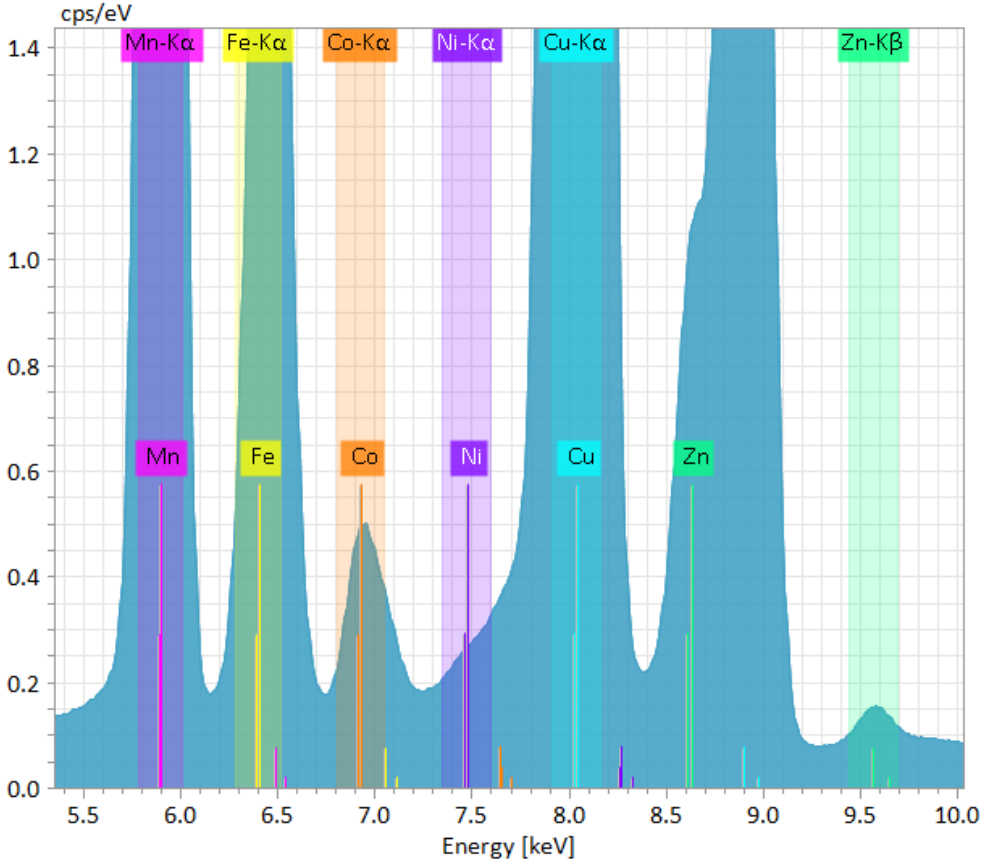


Geological Applications: Exotic-Cu Deposits

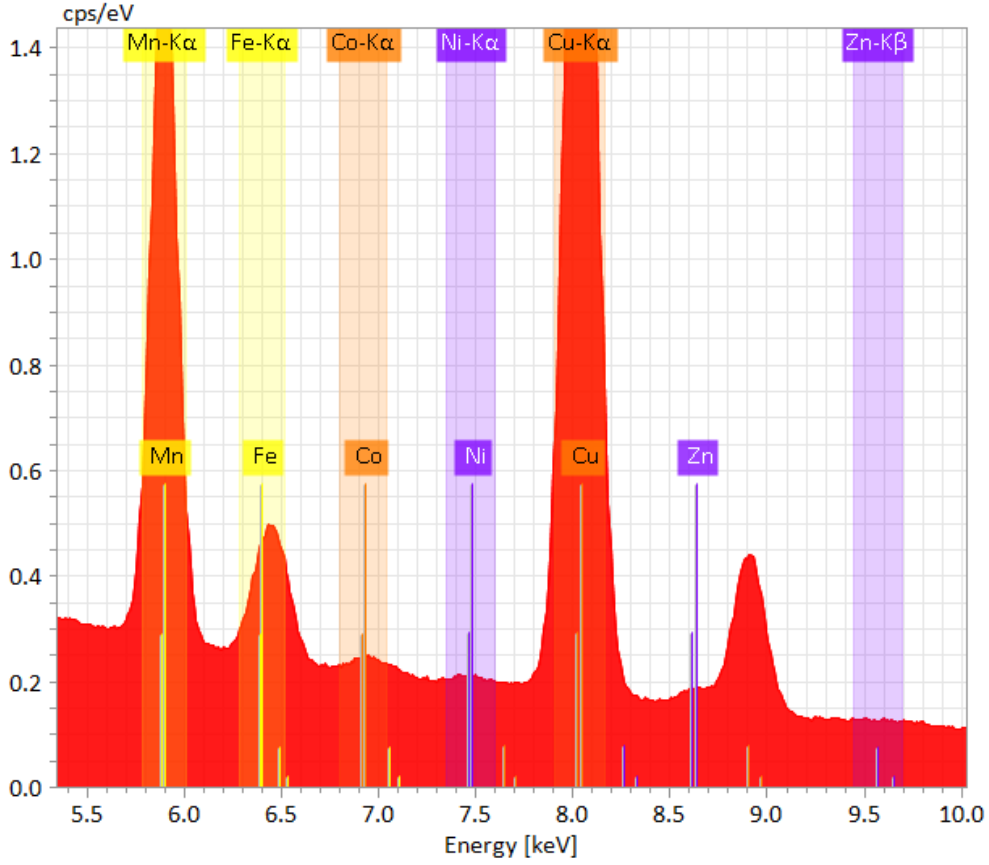




XRF-EDS (XTrace)

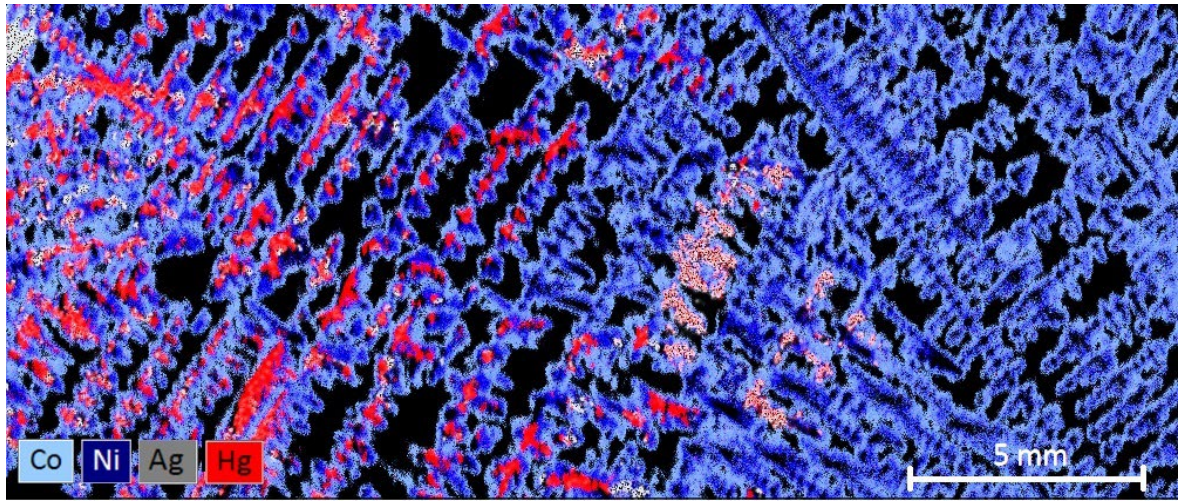


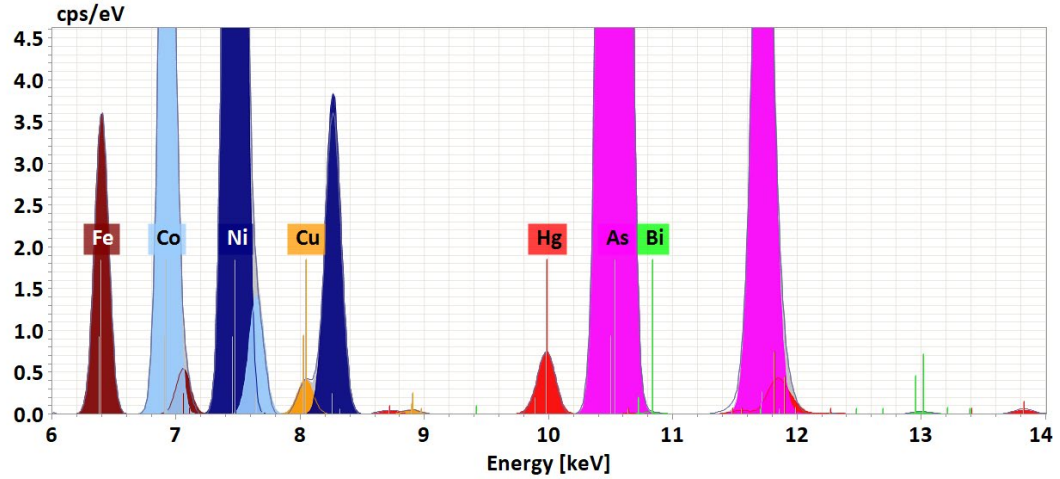
SEM-EDS (e-beam)



Identification and determination of the elements in ore at trace levels

- Micro-XRF on SEM allows for the direct analysis of a cut rock sample with minimal sample preparation. Detailed mapping helps to quickly identify the elements present in the sample and their distribution.
- In addition to identifying and determining the distribution of valuable elements, such as silver (Ag), copper (Cu), nickel (Ni), and cobalt (Co), toxic elements and those that can complicate mineral processing, such as arsenic (As) and mercury (Hg), can also be identified. Due to the low background and high signal-to-noise ratio even elements present at trace levels can be detected, in this example, mercury (Hg), bismuth (Bi), and copper (Cu).

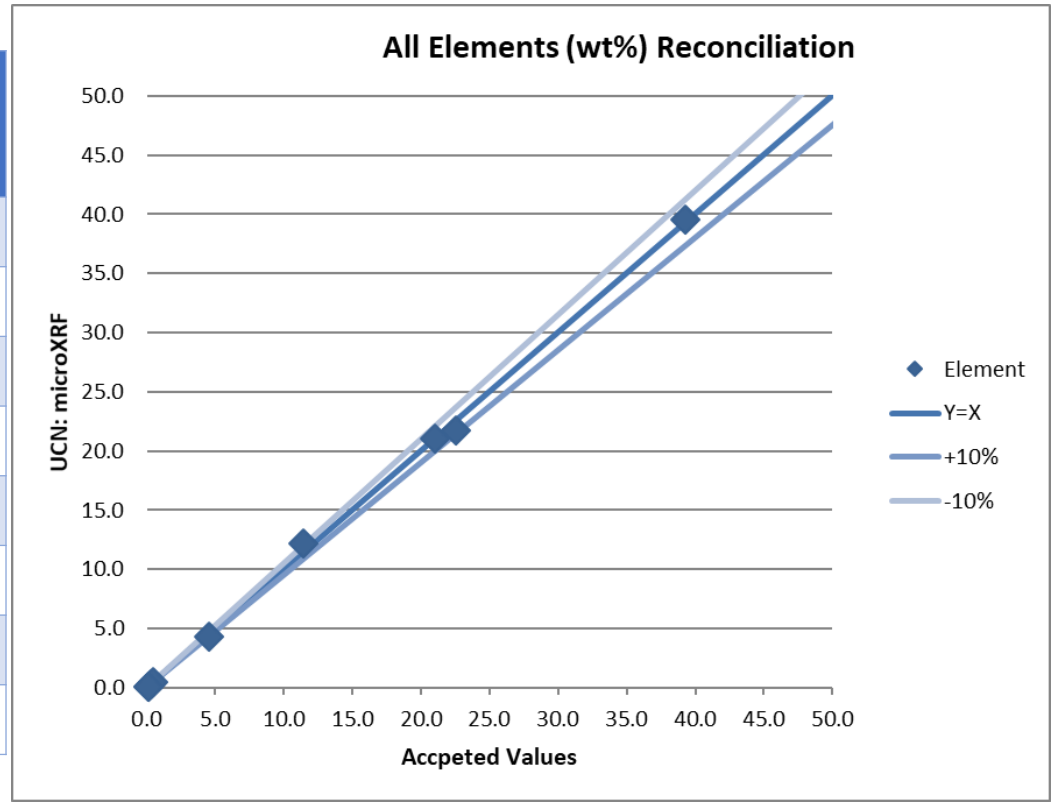




Quantitative Microanalysis with SEM-XRF
Point Analysis - major elements

Comparison XRF-EDS spectra for the same point: analytical precision

Element	Unit	EPMA Values	XRF-EDS Values	Std. Dev.	Maximum	Minimum	Range
SiO2	(%)	39.28	39.22	0.18	39.48	38.89	0.58
TiO2	(%)	0.28	0.30	0.02	0.33	0.26	0.07
Al2O3	(%)	22.51	21.68	0.31	22.21	21.23	0.98
Cr2O3	(%)	0.12	0.11	0.01	0.11	0.09	0.02
FeO	(%)	21.00	21.41	0.20	21.85	21.18	0.67
MnO	(%)	0.47	0.47	0.01	0.50	0.46	0.04
MgO	(%)	11.44	12.22	0.32	12.62	11.57	1.05
CaO	(%)	4.57	4.36	0.05	4.45	4.29	0.16

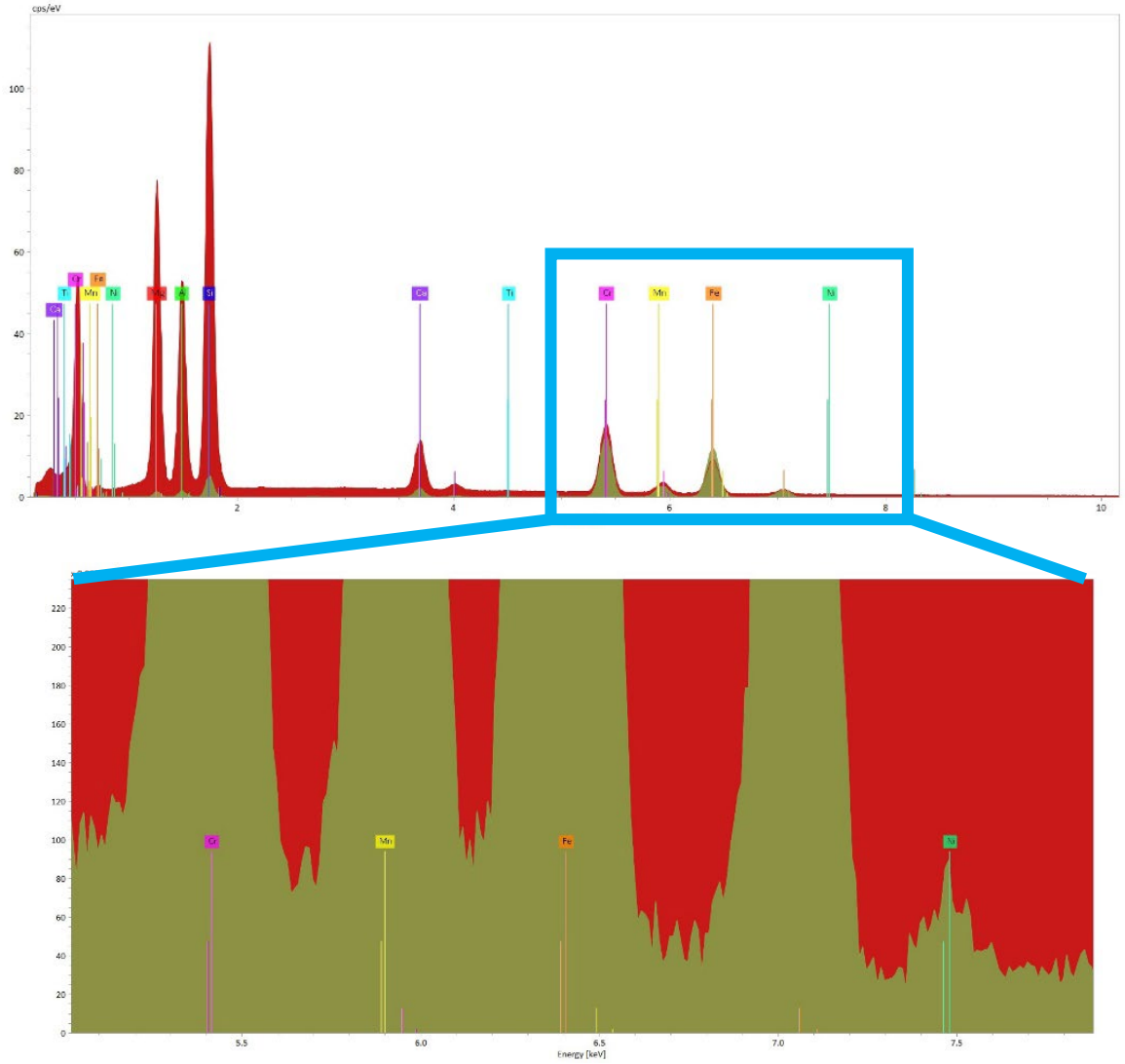


- Results for the analysis of the same point on a Mantle Eclogitic Garnet
- Silicate Mineral: 8 Elements of Interest: Si, Ti, Al, Cr, Fe, Mn, Mg, Ca.
- Presented as oxides based on stoichiometry

Quantitative Microanalysis with SEM-XRF
Point Analysis – major and trace elements

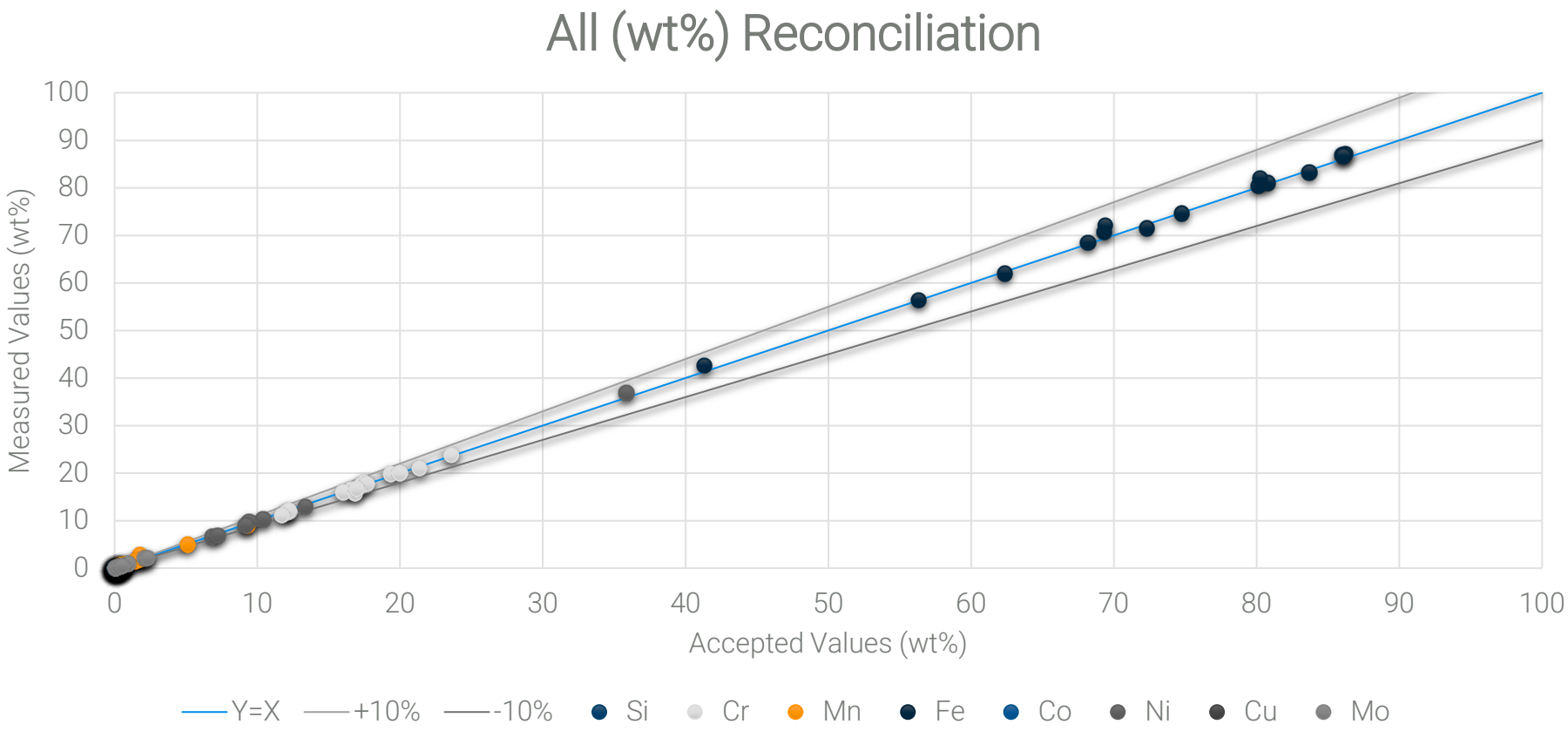
Trace elements possible with Micro-XRF

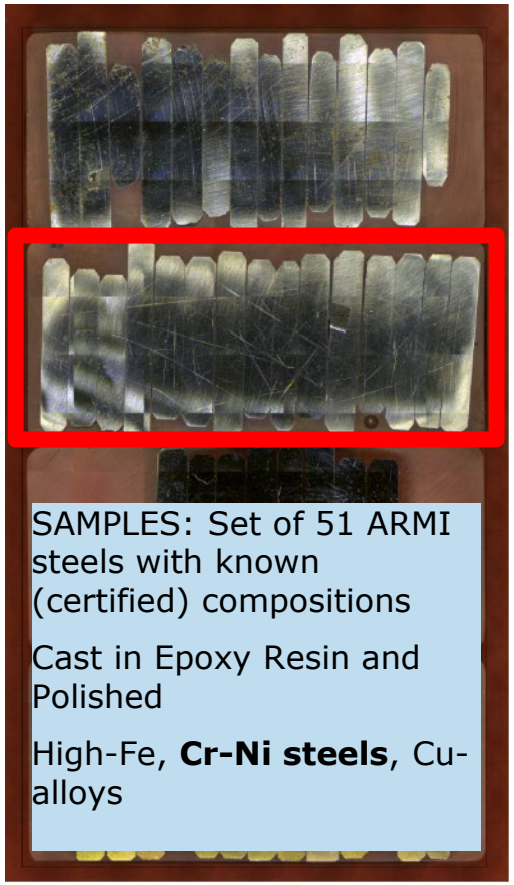
Element	Unit	90 sec	120 sec	180 sec
SiO2	(%)	39.04	39.17	39.20
TiO2	(%)	0.28	0.28	0.29
Al2O3	(%)	22.23	21.97	21.87
Cr2O3	(%)	0.11	0.11	0.11
FeO	(%)	21.16	21.05	21.02
MnO	(%)	0.49	0.48	0.48
MgO	(%)	12.29	12.57	12.63
CaO	(%)	4.35	4.31	4.33
Ni	(ppm)	26	18	28
Cu	(ppm)	3	5	4
Zn	(ppm)	173	143	150
Ga	(ppm)	7	0	28
Ge	(ppm)	17	22	17
As	(ppm)	28	28	28
Rb	(ppm)	41	69	59
Sr	(ppm)	28	0	28
Y	(ppm)	2	28	3
Zr	(ppm)	157	157	171
Nb	(ppm)	1	28	0



Analysis of Steels and Alloys
Excitation: Micro-XRF; Detector: EDS

Analytical Conditions Point Analysis: 50 kV, 600 uA, No Filter, 130 kcps, under vacuum, Working Distance 12 mm, 120 seconds





Analysis of Steels and Alloys
Combined Analysis

Sample 32: AISI 422-205B

Element	Certified	MicroXRF	SEM-EDS	Combined
C	**0.22**			
N	**0.05**			
Al	**0.01**			
Si	**0.37**		0.34	**0.33**
P	**0.01**			
S	**0.00**			
Ti	**0.00**	0.003		**0.00**
V	**0.26**	0.279		**0.26**
Cr	**11.72**	11.084	11.37	**11.32**
Mn	**0.68**	0.797	0.87	**0.75**
Fe	**83.70**	83.243	84.55	**83.20**
Co	**0.03**	0.024	0.49	**0.02**
Ni	**0.70**	0.692	0.54	**0.67**
Cu	**0.15**	0.177		**0.15**
Nb	**0.02**	0.012		**0.01**
Mo	**0.97**	0.970	0.95	**0.94**

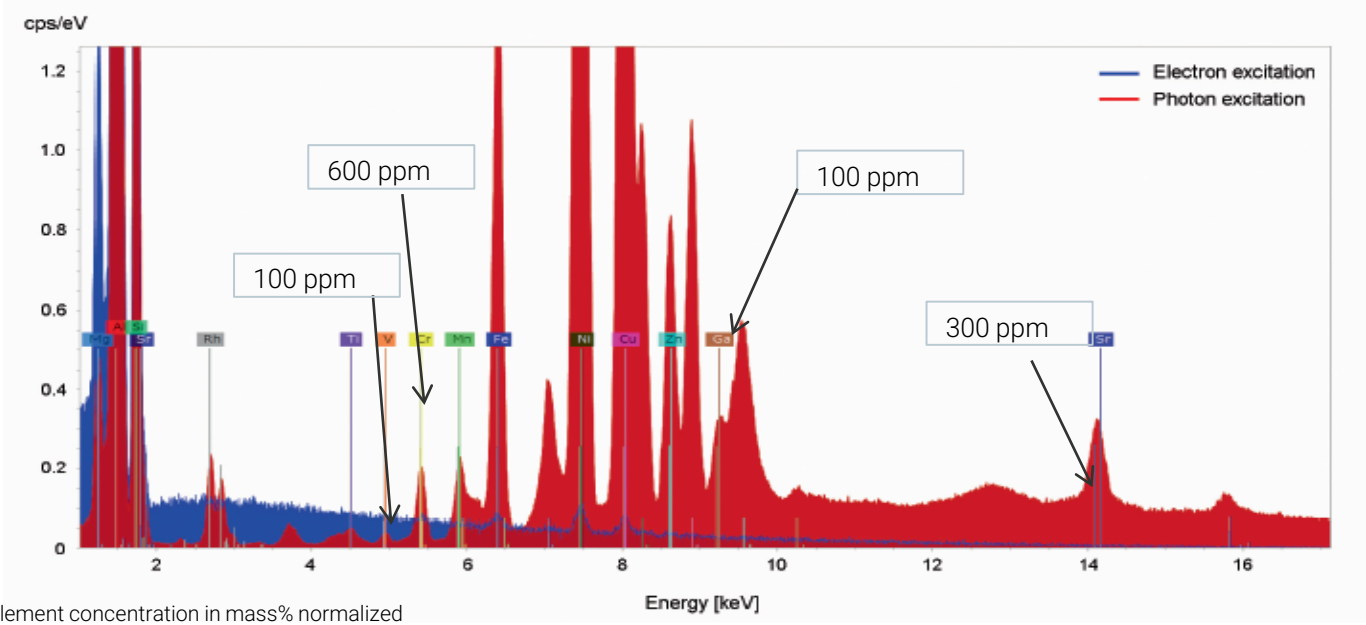
SEM-EDS/WDS

Low-Z elements

SEM-XRF

High-Z elements

Al Alloy
Improved LOD comparison EDS - XRF



Element concentration in mass% normalized

	Mg	Al	Si	Ti	V	Cr	Mn	Fe	Ni	Cu	Zn	Ga	Sr
EDS mean concentration value	1.20	85.09	11.43	n.d.	n.d.	b.d.	n.d.	0.36	0.91	0.91	0.10	n.d.	n.d.
Micro-XRF mean concentration value	0.85	83.87	12.83	0.03	0.01	0.06	0.03	0.36	0.93	0.93	0.10	0.01	0.03
Certificied values	1.1	84.52	12.00	0.011	0.0099	0.051	0.033	0.31	0.89	0.89	0.098	0.02	0.026

Fig. 1 Photograph of the analyzed specimen Alcoa Deltalloy® 4032

Accurate elemental analysis with high energy elemental lines

Rare Earth Elements have a range of X-ray energies that are detectable by EDS:

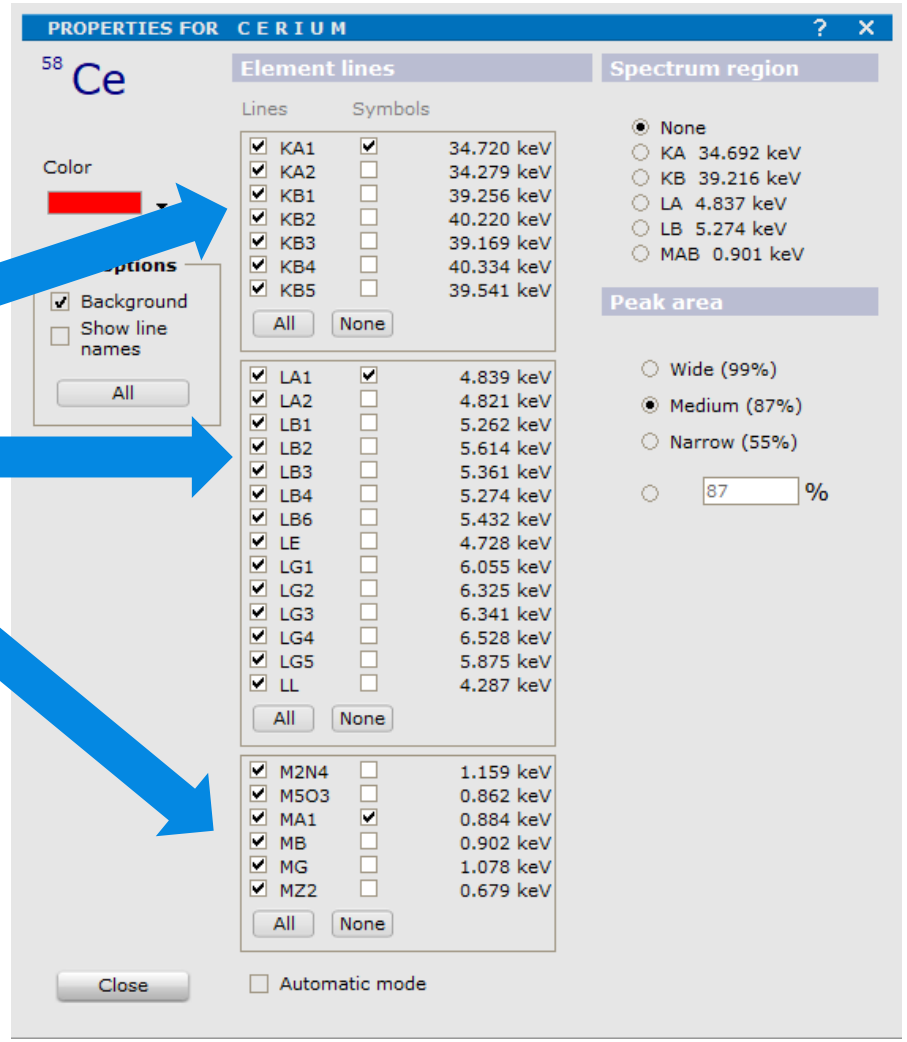
K-Series: 34 to 55 keV

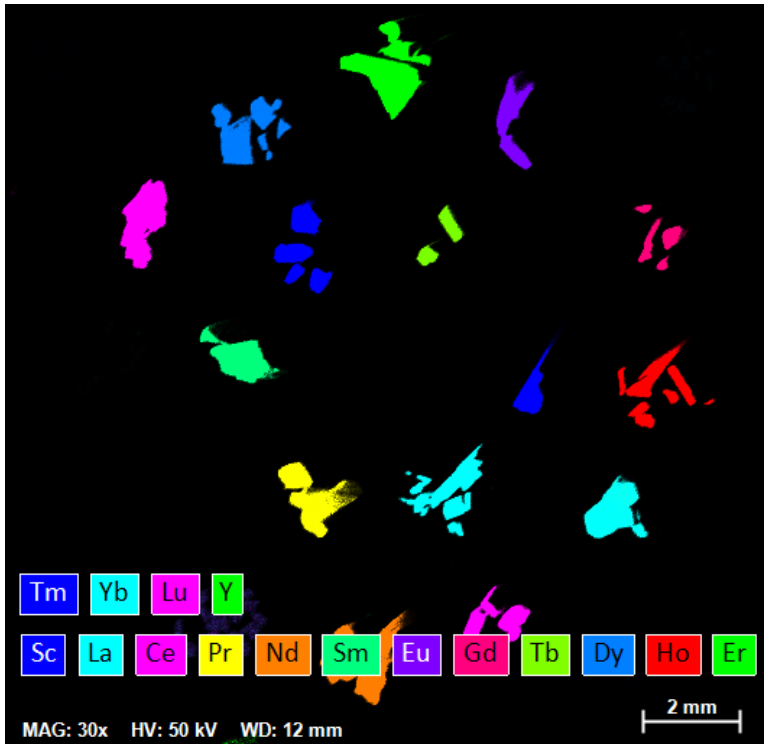
L-Series: 4 to 10 keV

M-Series: 0.5 to 1.5 keV

L-Series detectable with both electron and x-ray excitation source.

K-Series detectable with x-ray excitation source only.





Accurate elemental identification with high energy elemental lines

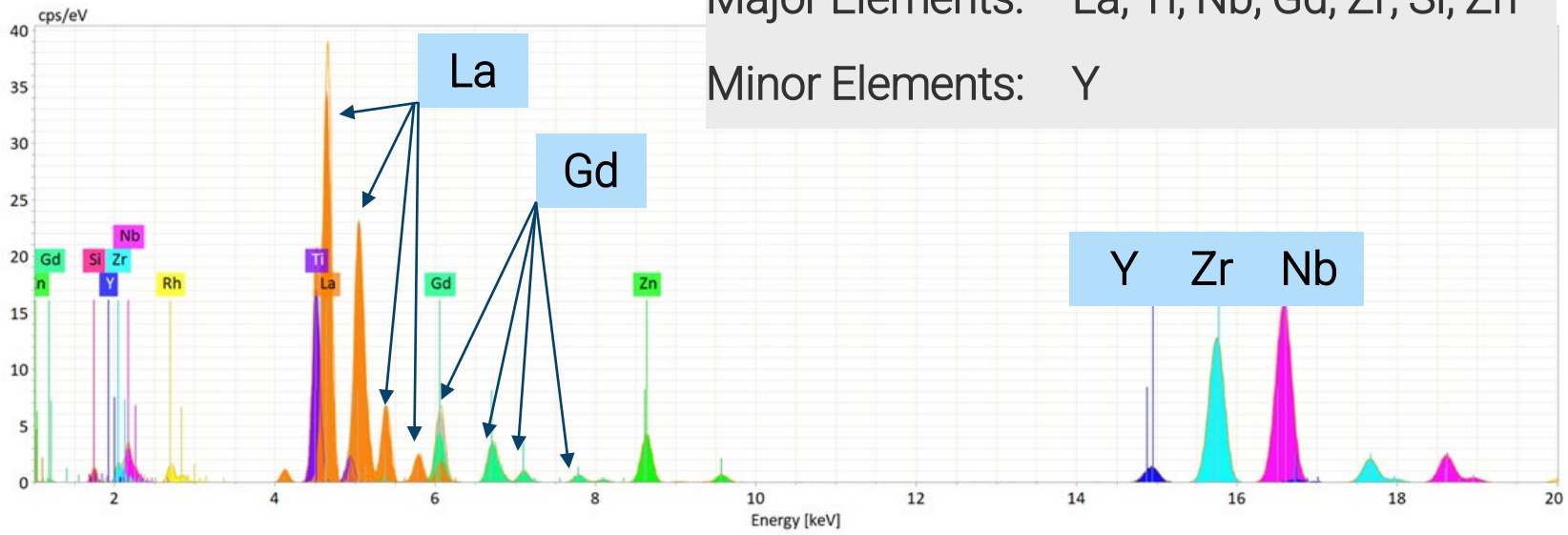
High Index Glasses

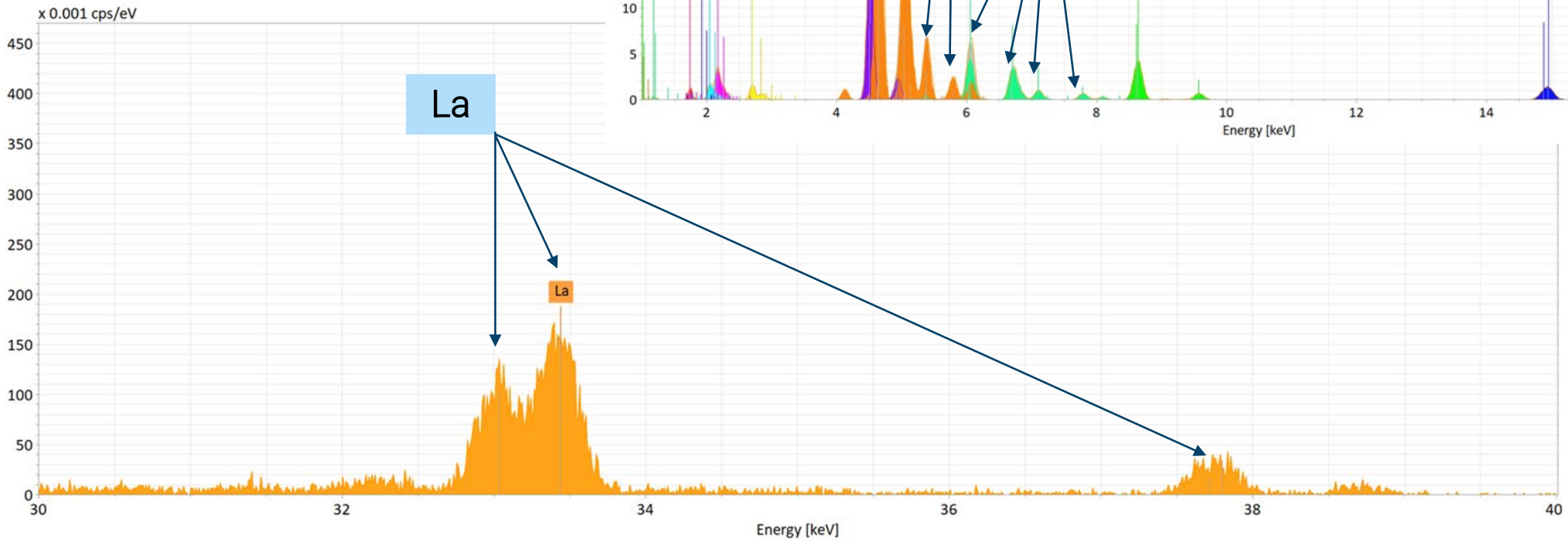
Spectrum X-axis Range:

Bottom: 30 to 40 keV

Right: 1 to 20 keV (Deconvolution)

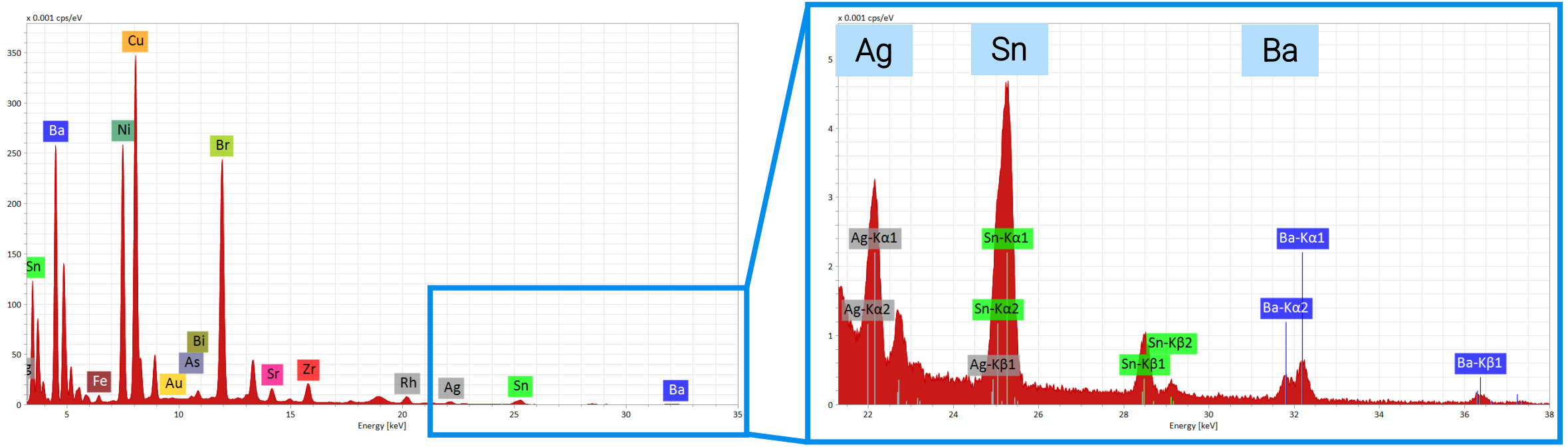
Major Elements:	La, Ti, Nb, Gd, Zr, Si, Zn
Minor Elements:	Y





Accurate elemental identification with high energy elemental lines
Ore Mineralisation

QUANTAX micro-XRF systems use a 50 kV X-ray source for the excitation to higher elemental energy lines, for example Ag Kα = 22.1 keV, Sn Kα = 25.3 keV and Ba Kα = 32.2 keV lines are all visible.

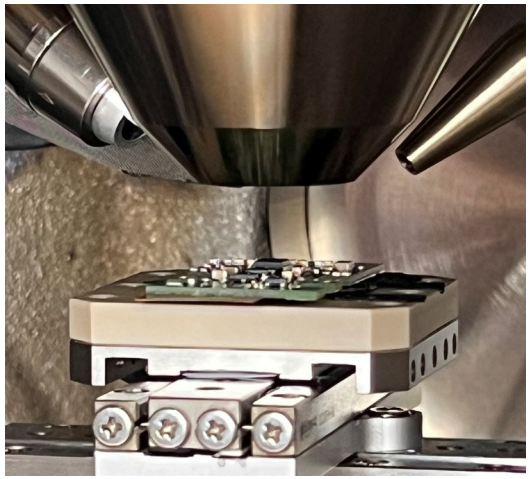


Full micro-XRF spectrum of a PCB showing how both major and trace elements can be detected using micro-XRF on SEM.

Zoomed spectrum energy range showing the detection of high energy elemental lines (higher than 20 keV)

Fast elemental mapping of the electronic components on a Printed Circuit Board (PCB)

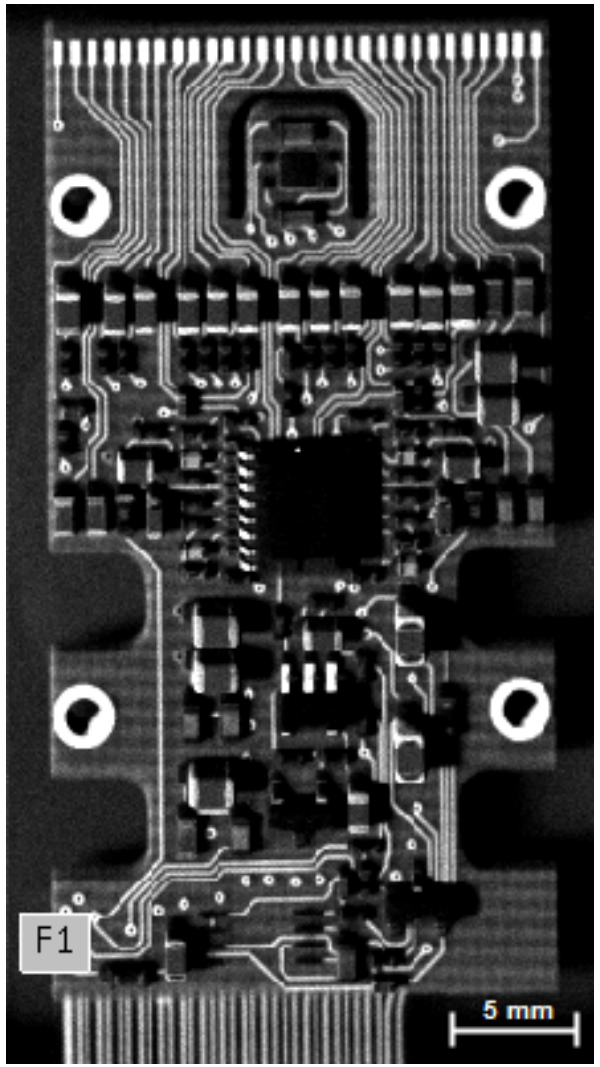
- Recycling of electrical components, such as Printed Circuit Boards (PCBs).
- High-speed micro-XRF on SEM can be used for the elemental analysis of electronic components at trace element sensitivity without any sample preparation required.

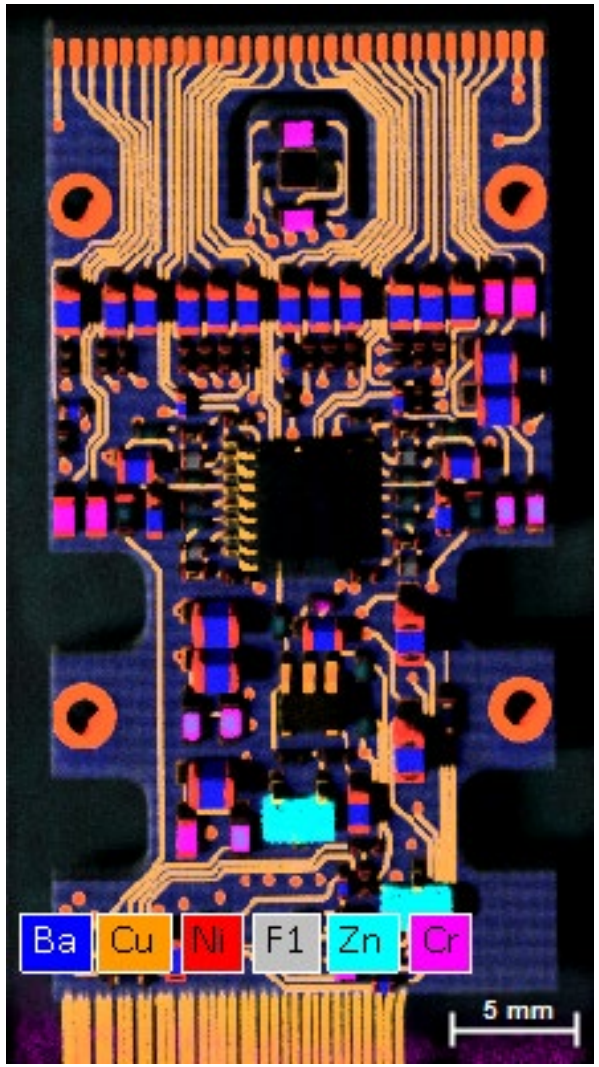


Left: Photo of PCB in SEM chamber

Right: Total X-ray intensity image

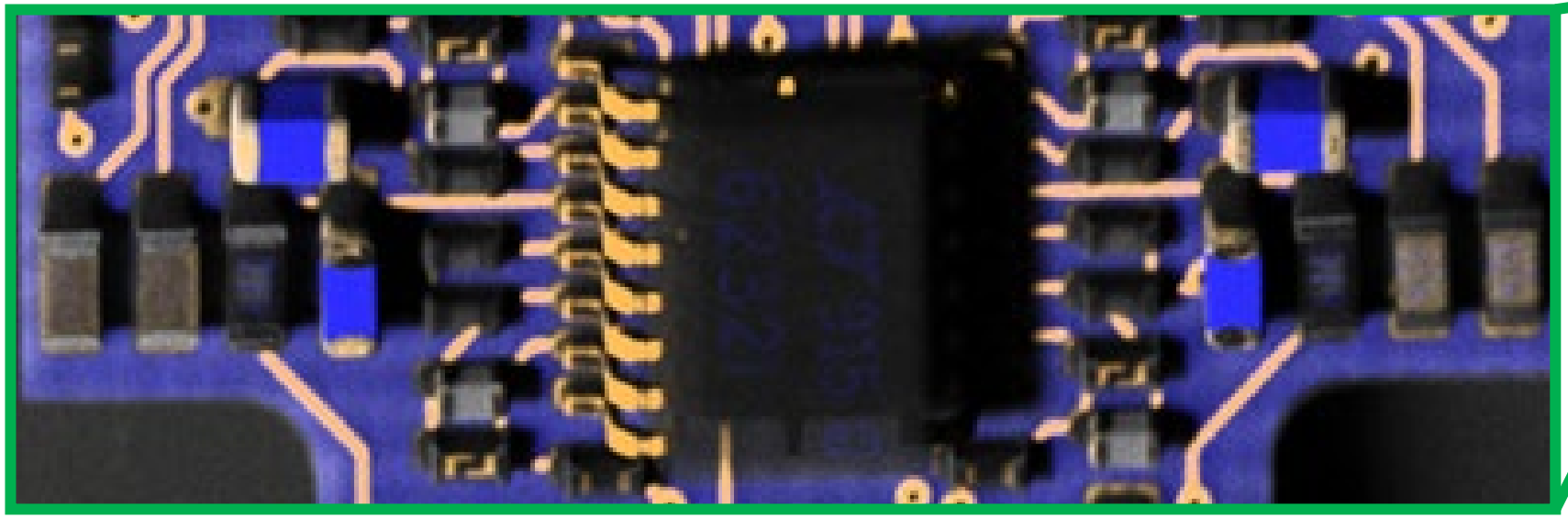
Far right: Mixed Elemental map of a PCB

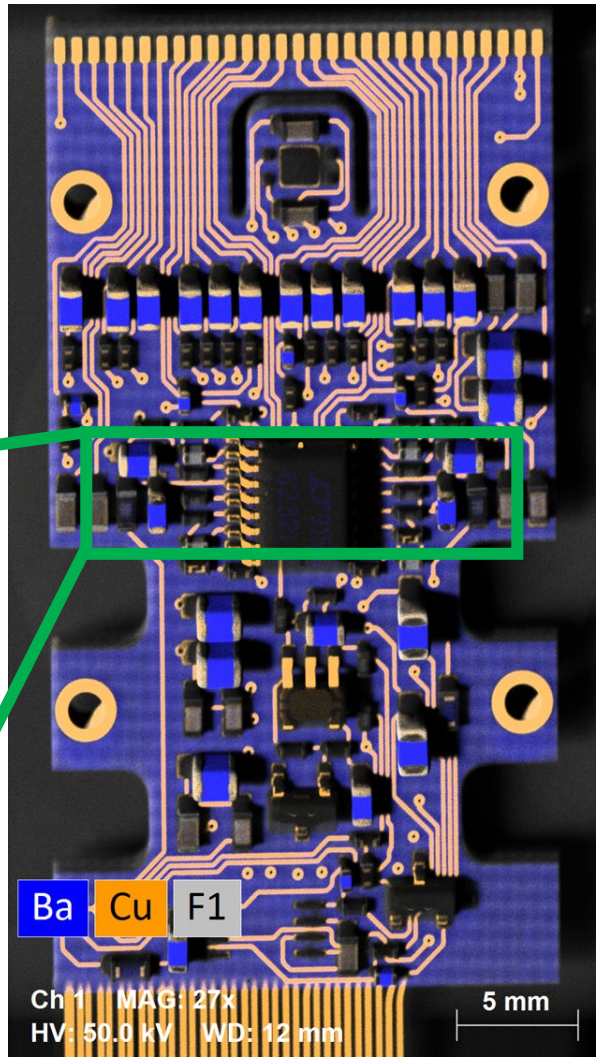




High-resolution elemental mapping of 3D features

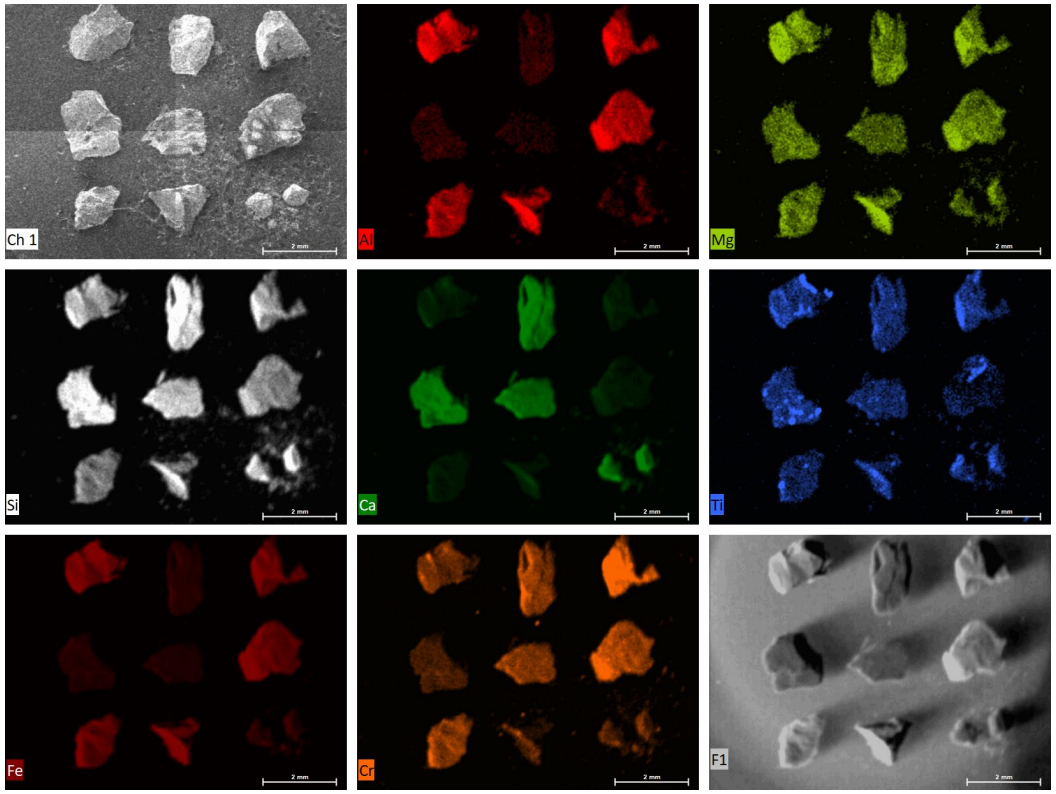
- The Aperture Management System (AMS) used in QUANTAX micro-XRF systems facilitates the high-resolution mapping of samples with 3D features, such as electronic components, at varying working distances.



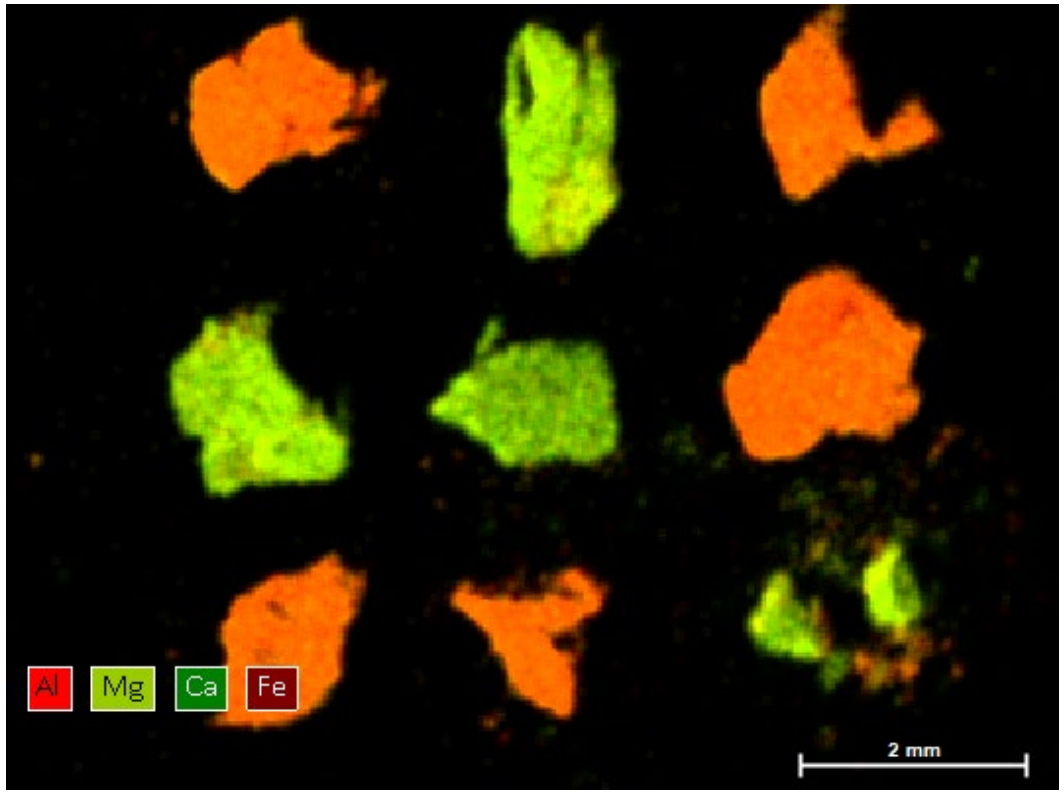


Analysis of Exploration Mineral Grains

Analysis of grains can help identify minerals of interest:
Two different grains – clinopyroxene and garnet derived from mantle eclogites associated with diamonds



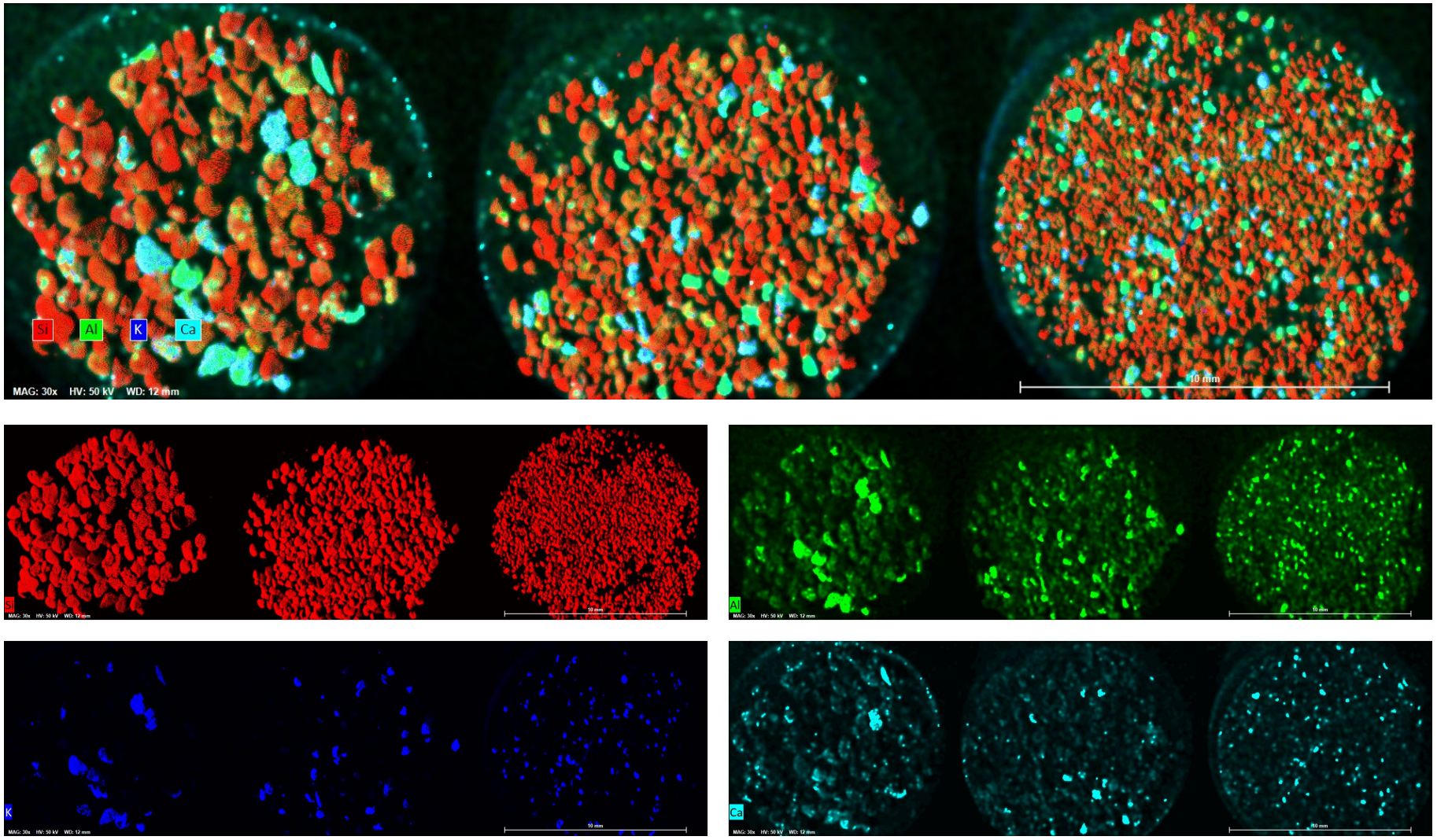
Clinopyroxene (green)
and
Garnet (orange)



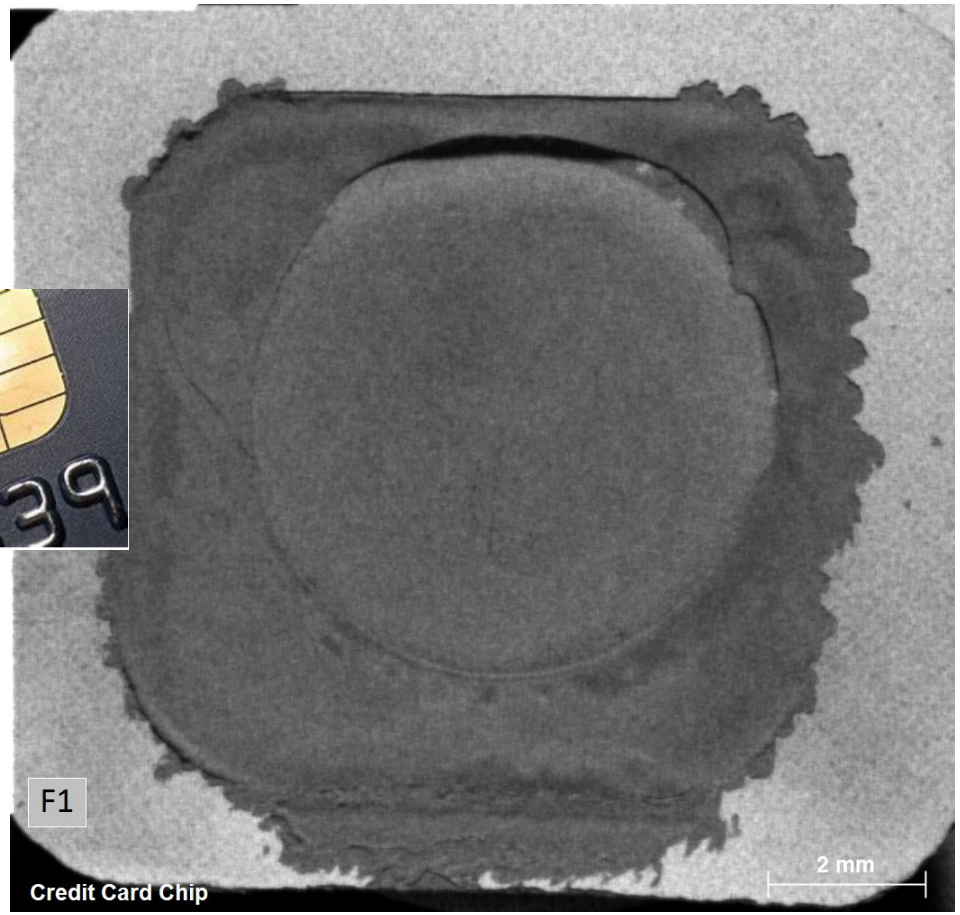
Analysis of Beach Sands

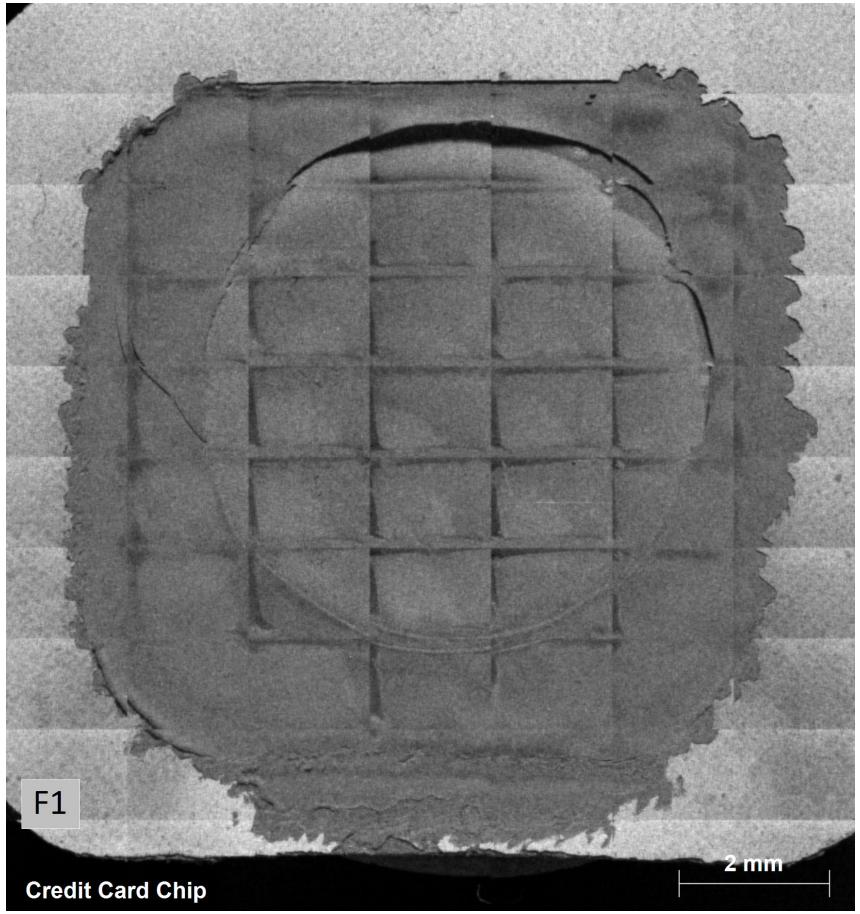
Analysis of Beach Sand

Loose Grains
Various Size Fractions
Uncoated
Large Area
High Speed Stage Movement



Analysis of Credit Card Chip
E-beam Excitation: Rapid Stage vs SEM Stage



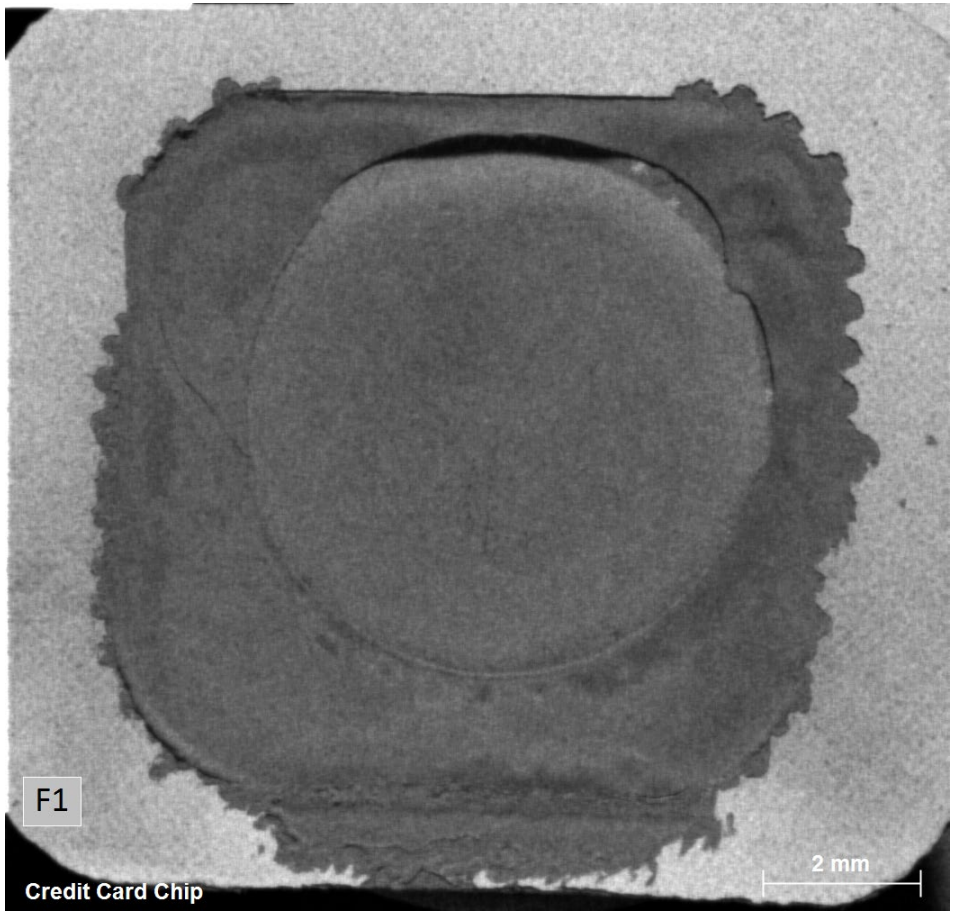


Sample courtesy of
Jeffrey Hannon

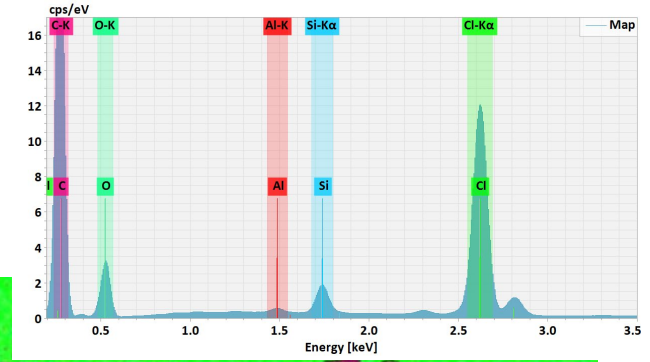
E-beam Map with
Rapid Stage

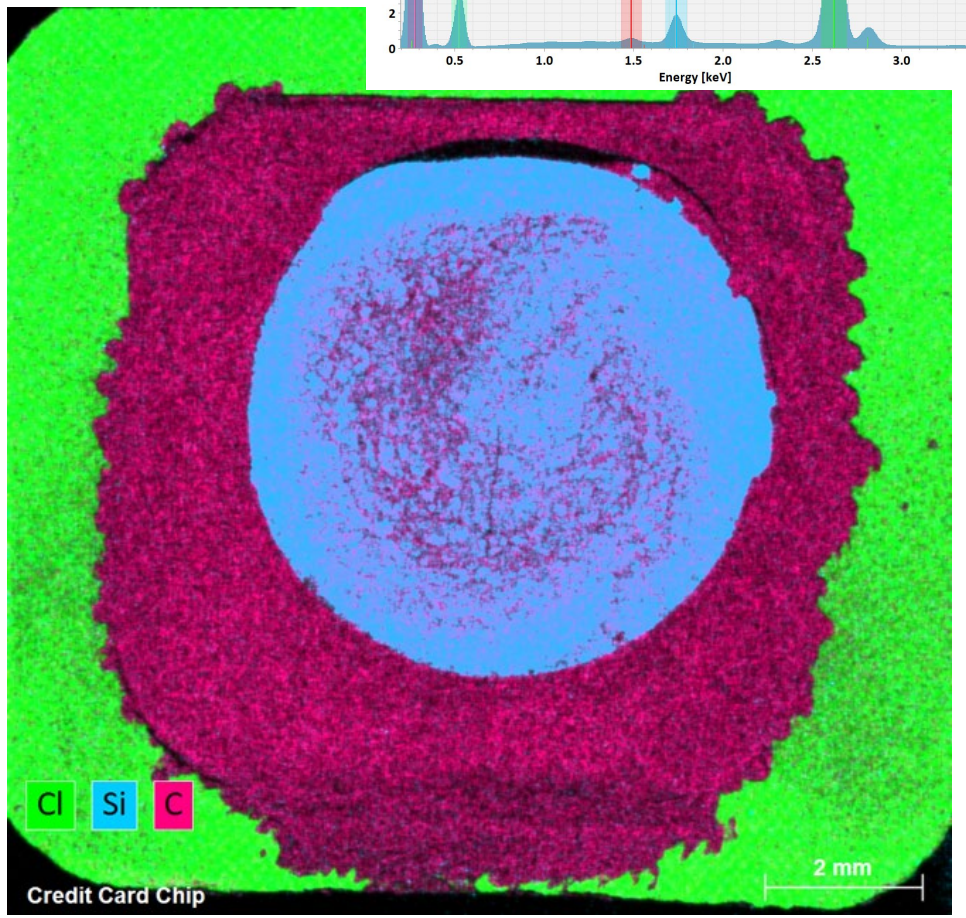
E-beam Map with
SEM Stage

Analysis of Credit Card Chip
E-beam Excitation



E-beam Map with Rapid Stage

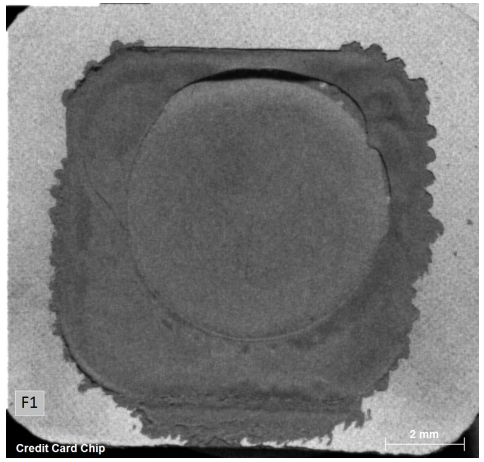


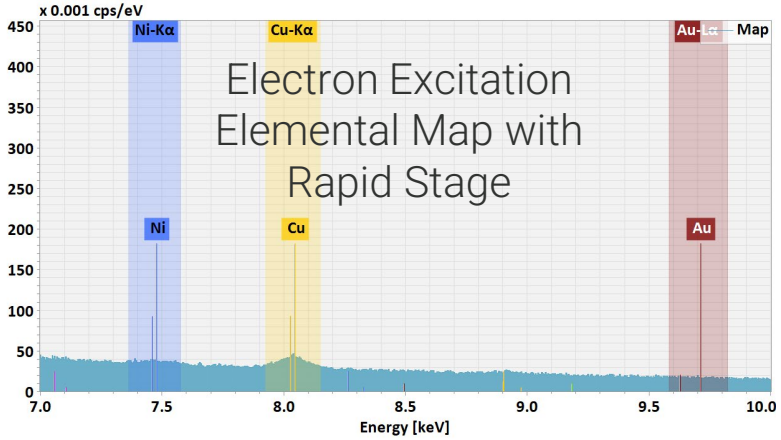


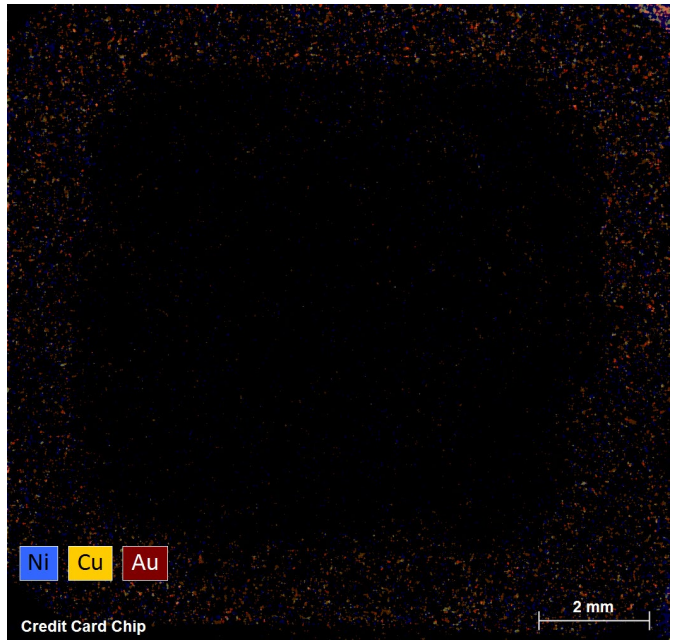
Electron Excitation Elemental Map with Rapid Stage

Sample courtesy of Jeffrey Hannon

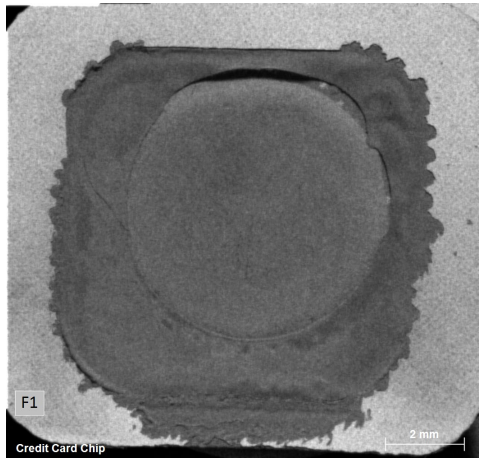
Analysis of Credit Card Chip
E-beam vs X-ray Excitation

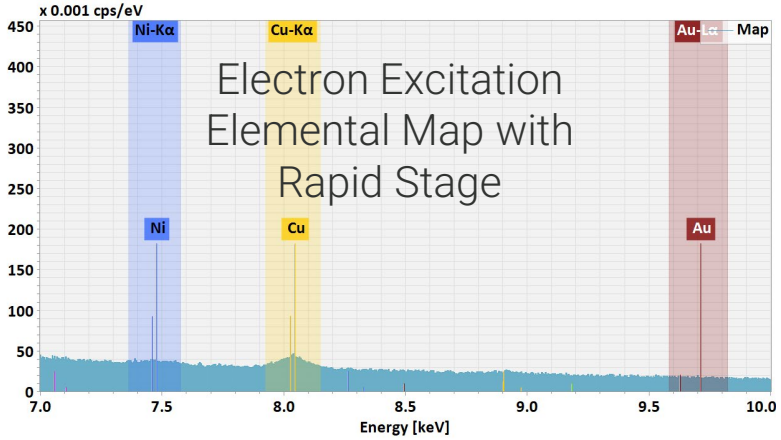


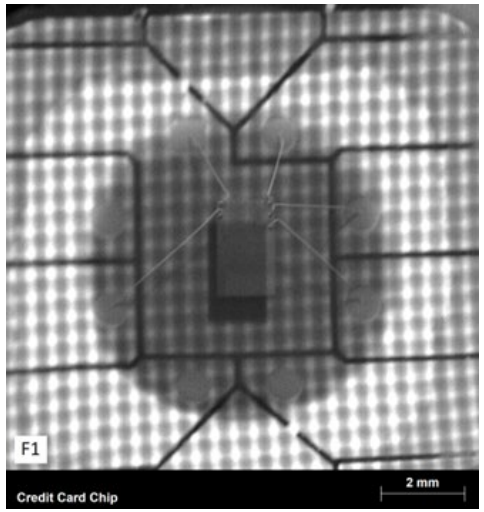


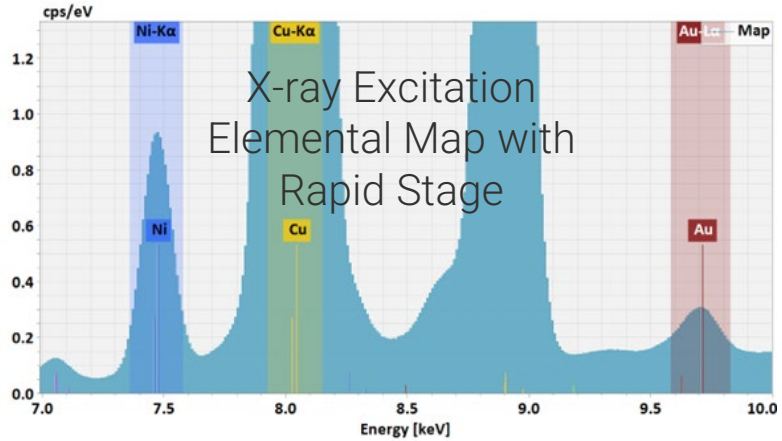


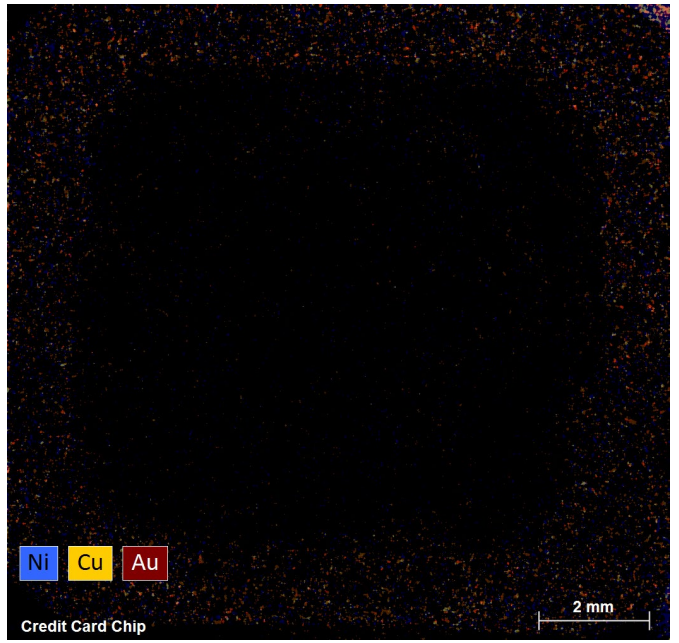
Analysis of Credit Card Chip
E-beam vs X-ray Excitation

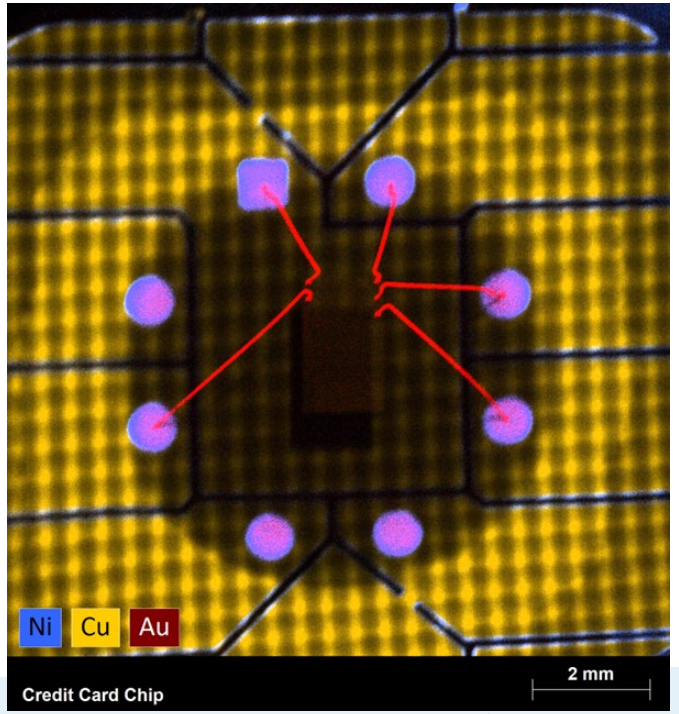




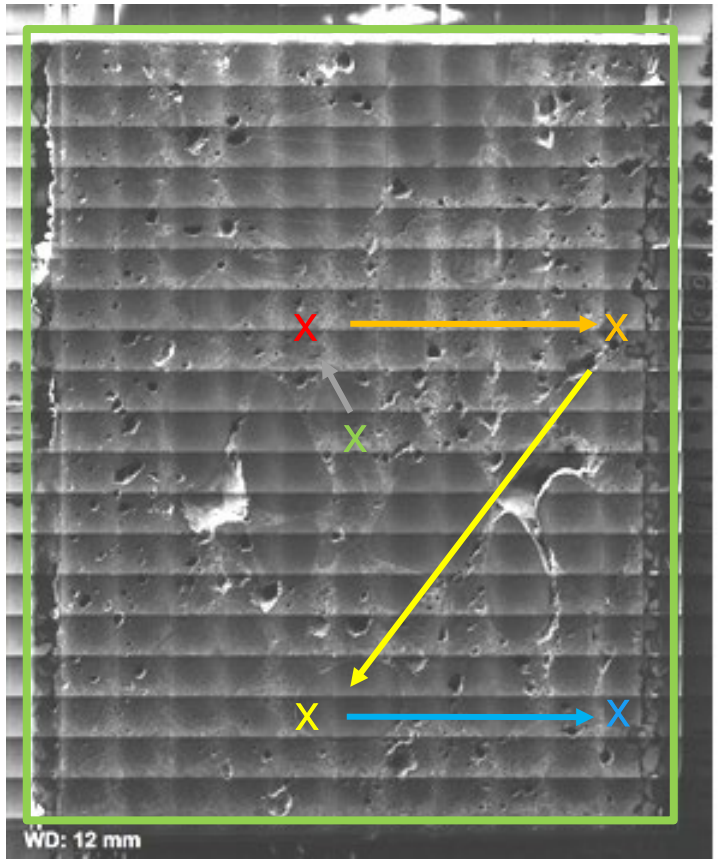


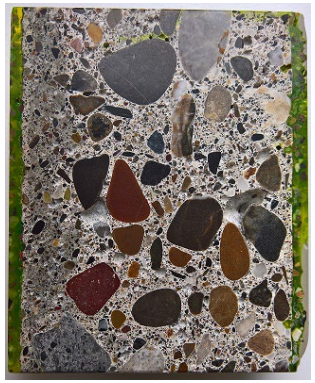




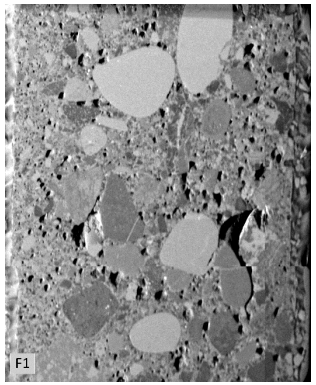


Large Area Maps
Rapid Stage + SEM Stage: SEM-XRF

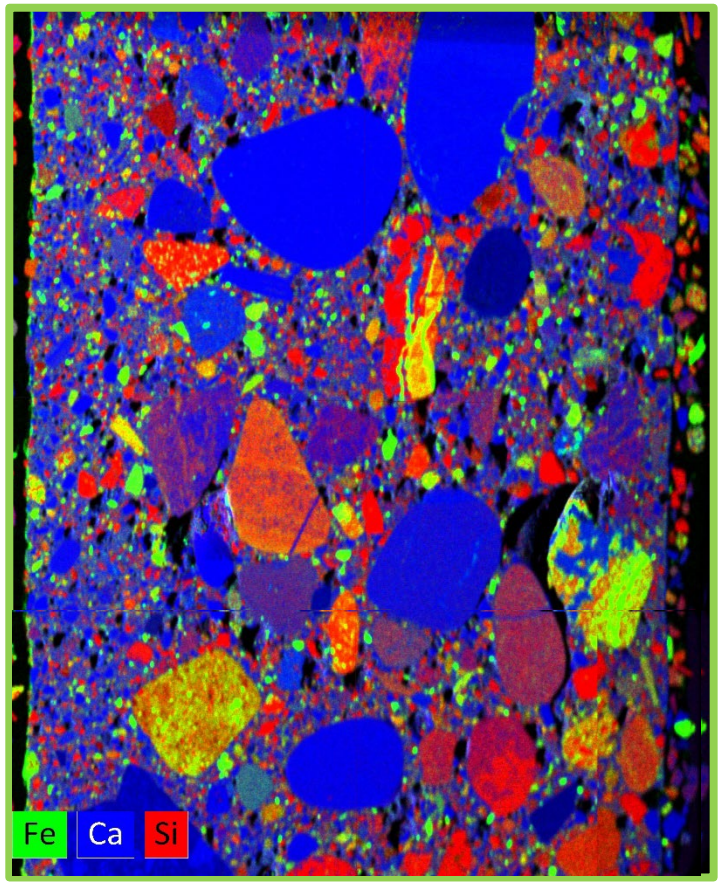




Photograph of the sample



X-Ray Intensity Map



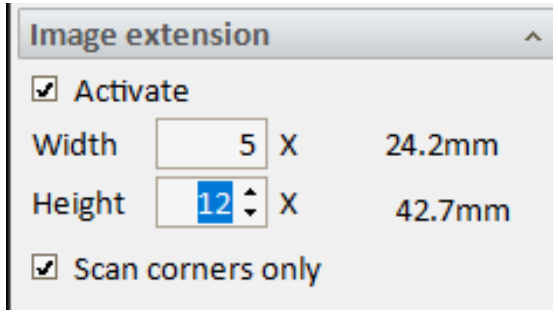
Large Samples:
Concrete Block: 61.8 mm x 74.4 mm

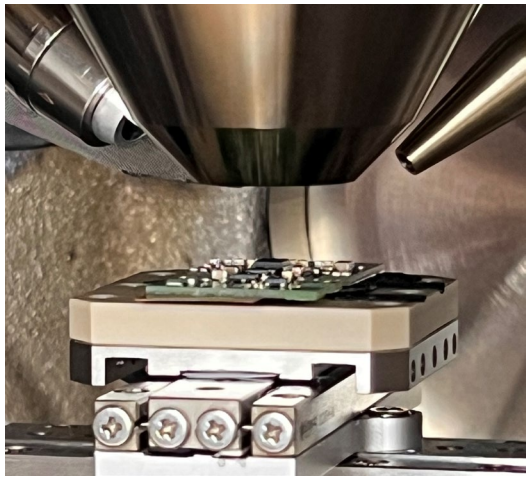
Such samples require a combination of the Specialised high speed stage + SEM Stage.

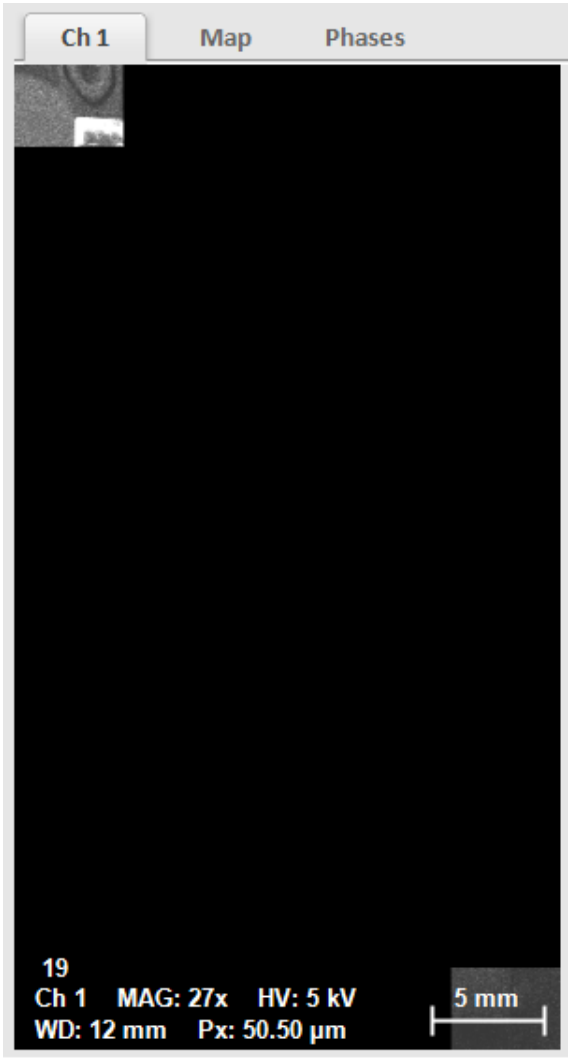
The sample is analysed in 4 maps which are mosaiced at the completion of the analysis.

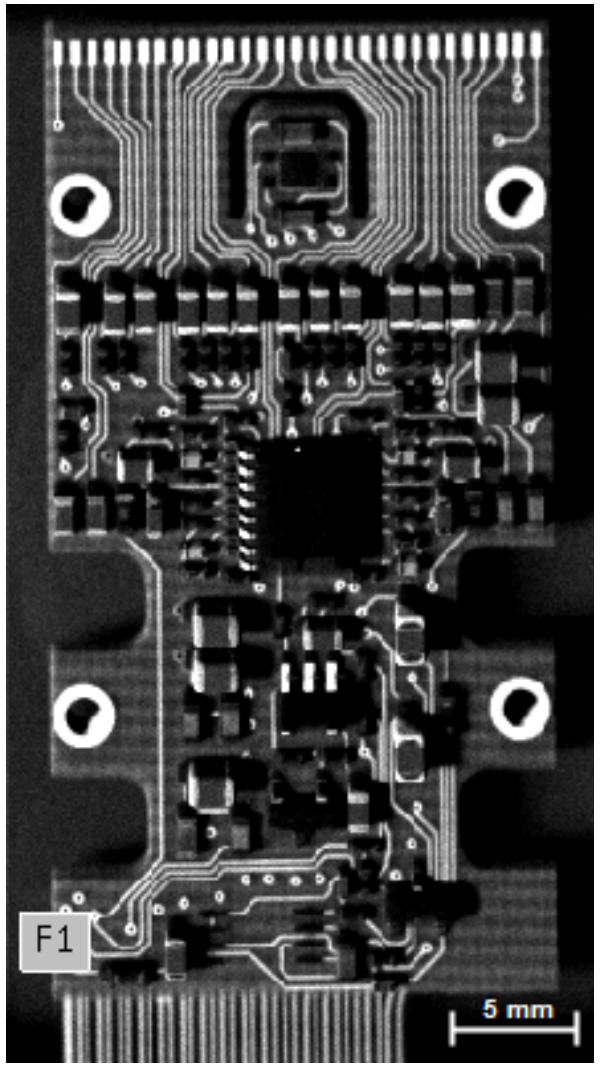
Image Extension: SEM is *14 x 22*

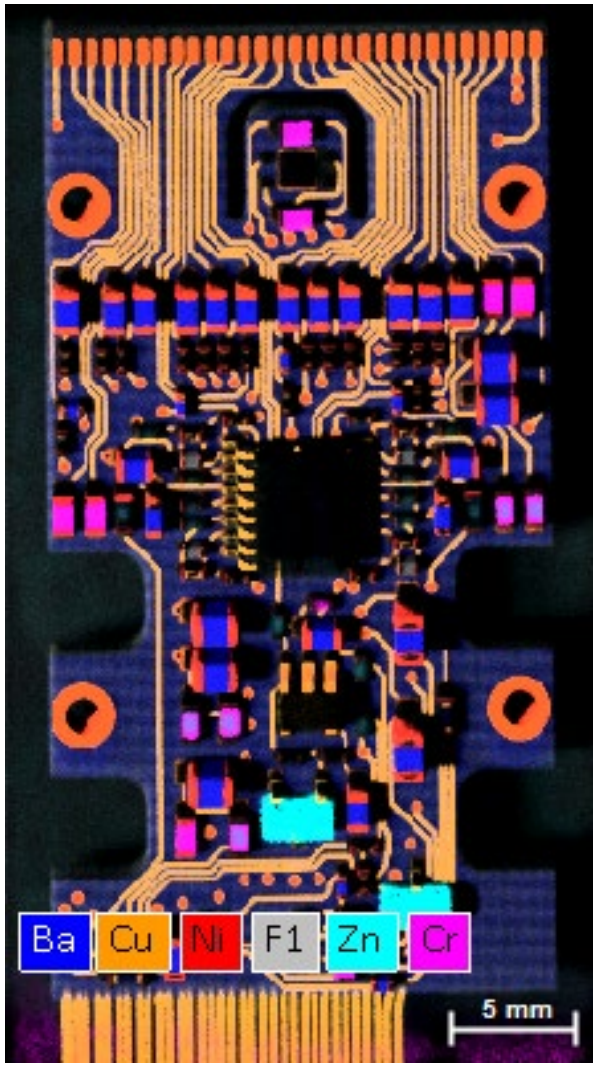
SEM-XRF and Rapid Stage
Integration in ESPRIT Software (version 2.6)









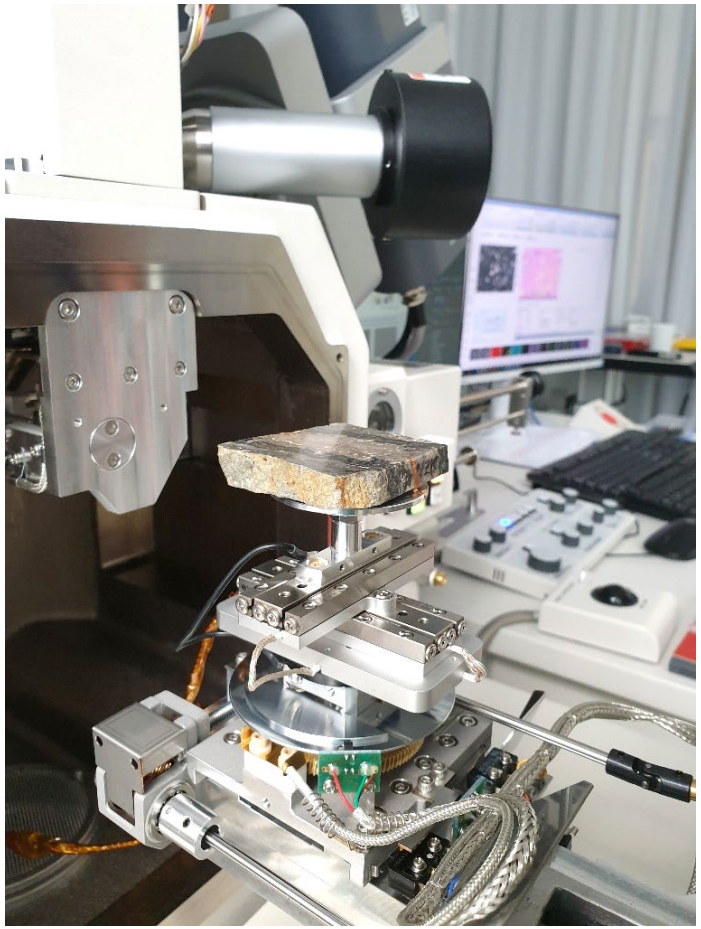


Beneficial Workflow: Epithermal Gold (Au)

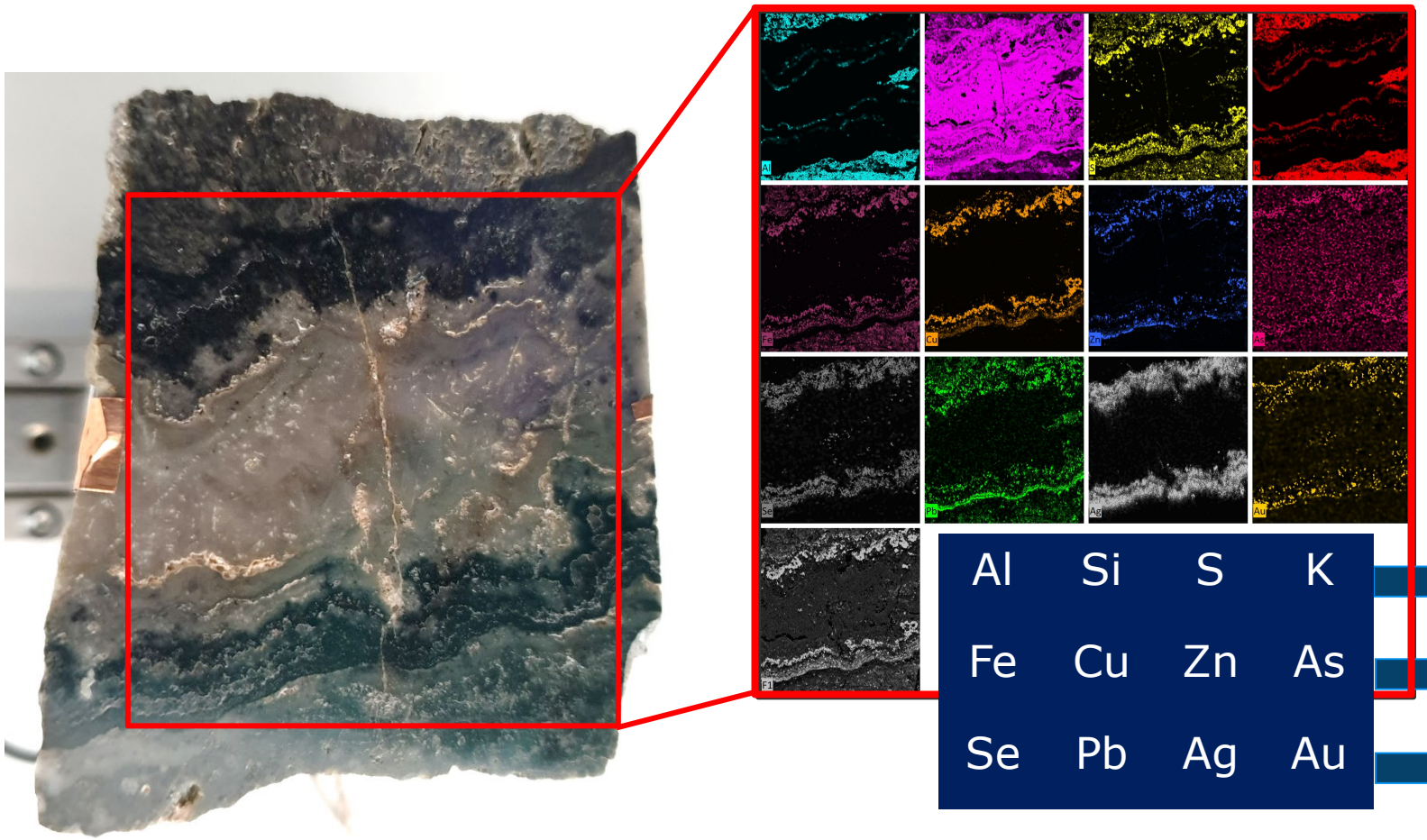
Gold-bearing sample from Karangahake Gold Mine, New Zealand.

Sample courtesy of Jeff Mauk

Mineral	Formula
Native Gold	Au
Native Silver	Ag
Sulphides	
Pyrite	FeS_2
Chalcopyrite	$CuFeS_2$
Galena	PbS
Sphalerite	ZnS
Gangue Mineralogy	
Quartz	SiO_2
Adularia	$KAlSi_3O_8$



SEM Micro-XRF Analysis: Epithermal Au
Large Area Mapping

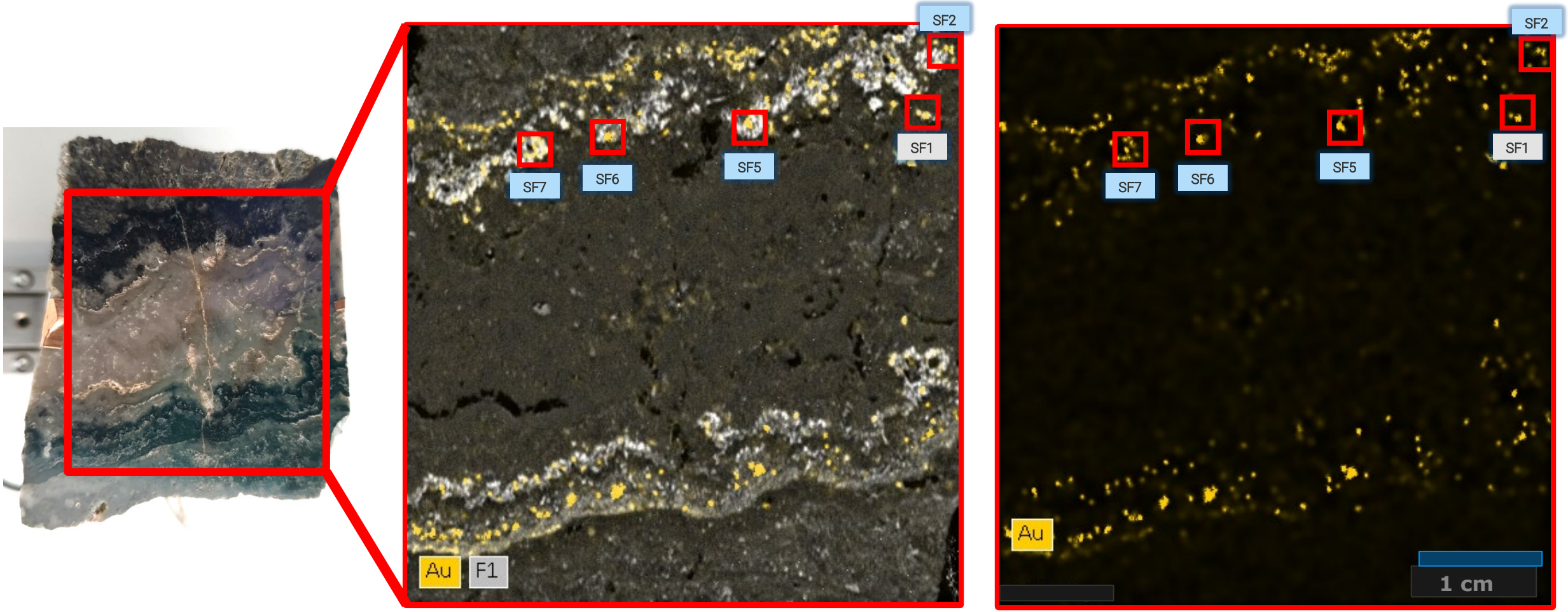


Beam:	X-ray
High Voltage:	50 kV
Anode Current:	600 µA
Analytical Spacing:	100 µm
Dwell Time:	64000 µs (64 ms)
Analytical Area:	4.5 x 4.5 cm
Total Analytical Time:	188 minutes
Spot Size:	25 µm
Interaction Depth:	10 – 100 µm

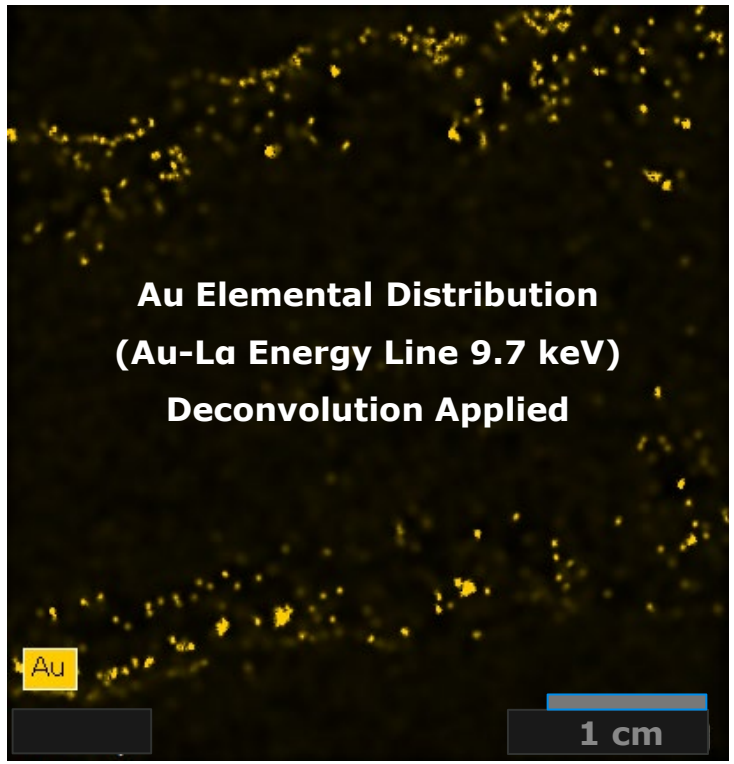
- Host Rock Elements: Al, Si, K
- Mineralisation: S, Fe, Cu, Zn, As
- Economic Mineralisation: Au, Ag, Se

Epithermal Gold-bearing rock sample from Karangahake, New Zealand

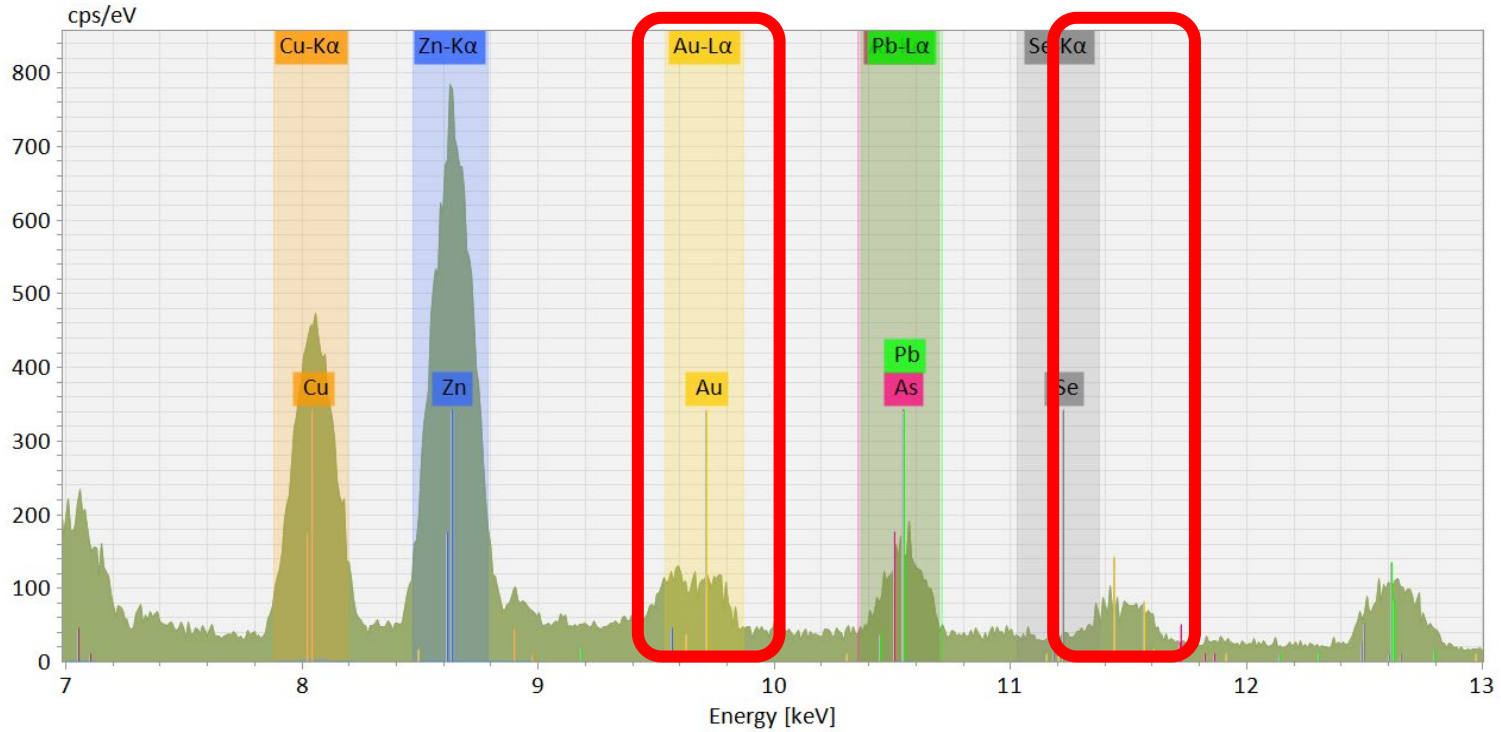
Micro-XRF on SEM (X-ray Excitation)
Identifying Gold (Au) in the Sample



Micro-XRF on SEM (X-ray Excitation)
Identifying Gold (Au) in the Sample



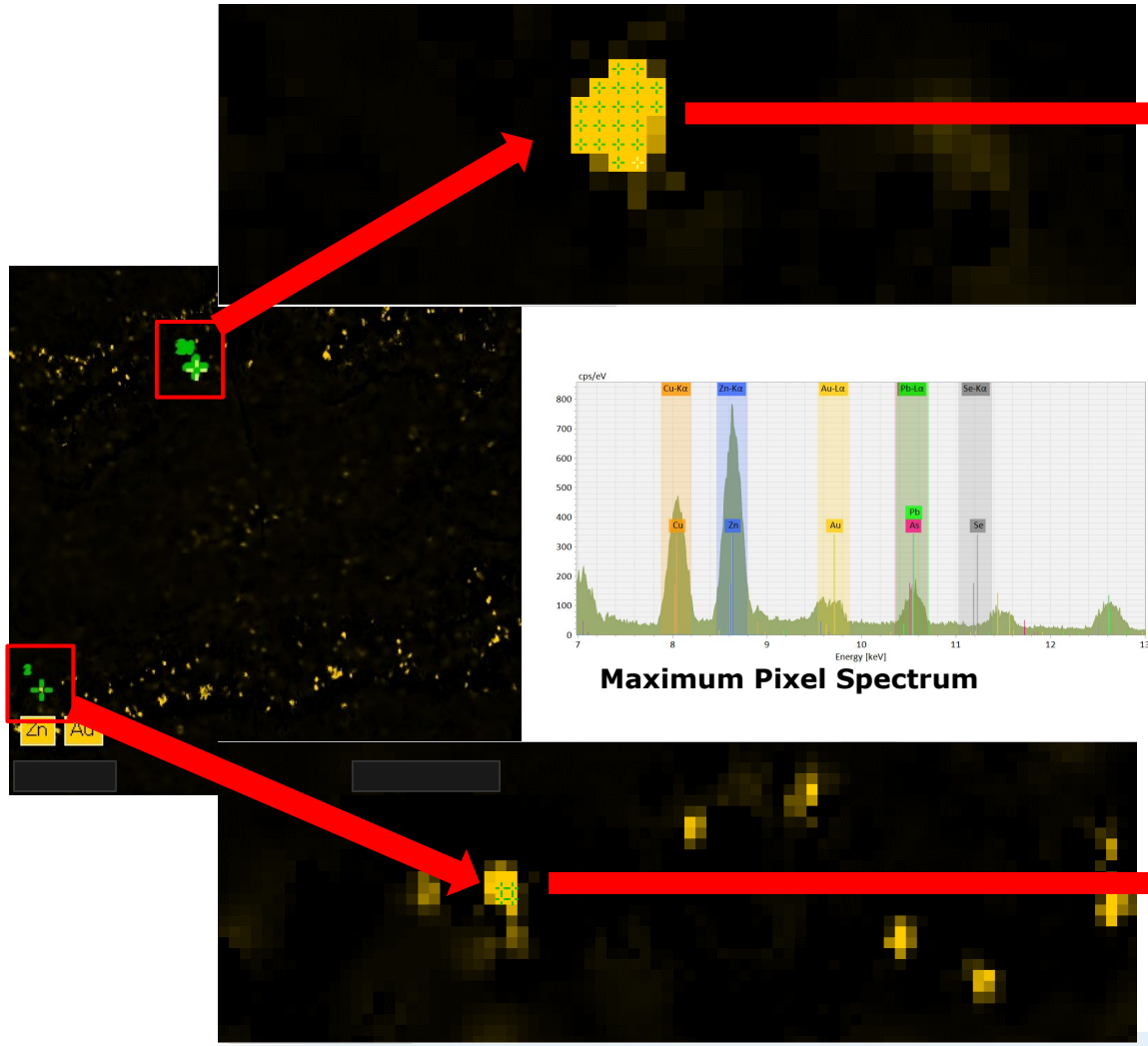
Maximum Pixel Spectrum



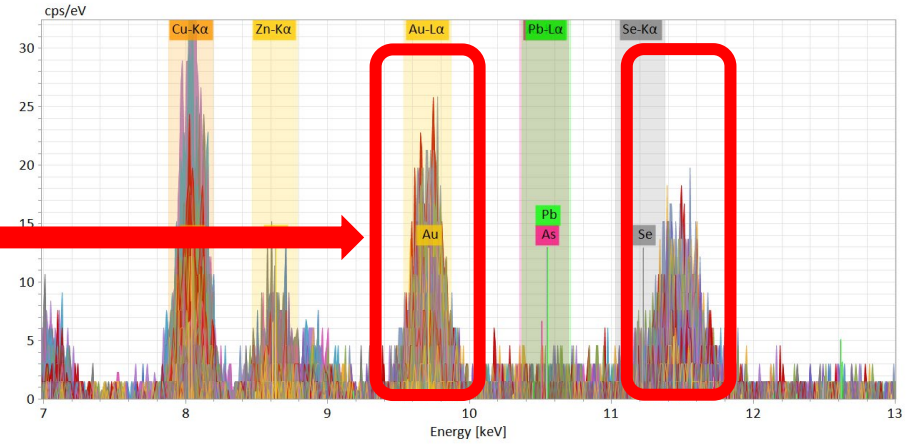
Distinct and clear Au: Au-Lβ X-ray energy Lines.

Presence of Au in the sample is confirmed. But is it identified correctly?

Micro-XRF on SEM (X-ray Excitation)
Identifying Gold (Au) in the Sample

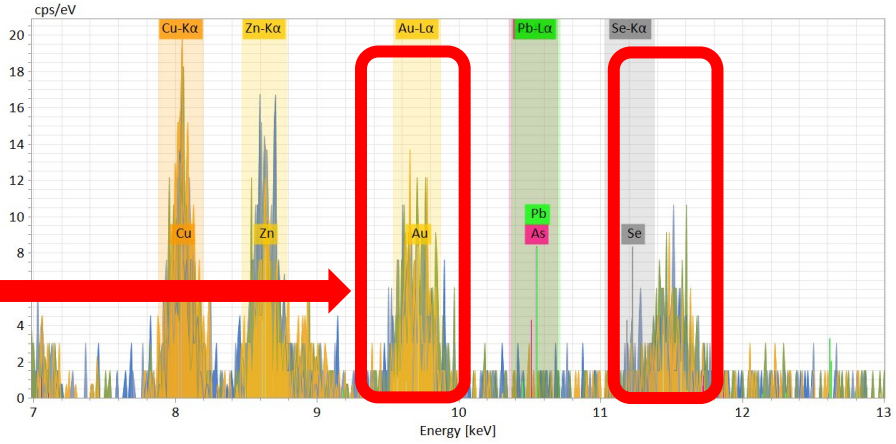


Maximum Pixel Spectrum

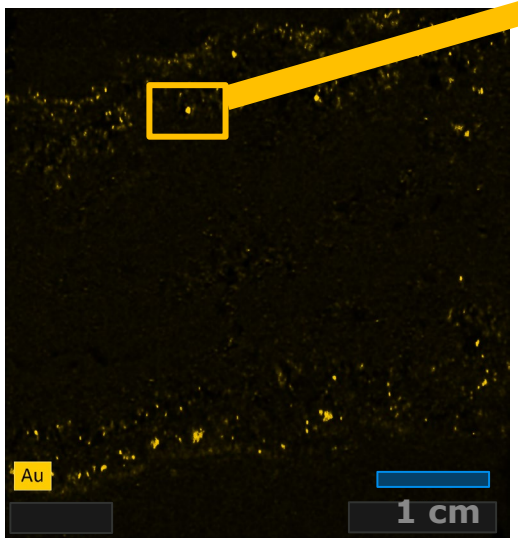


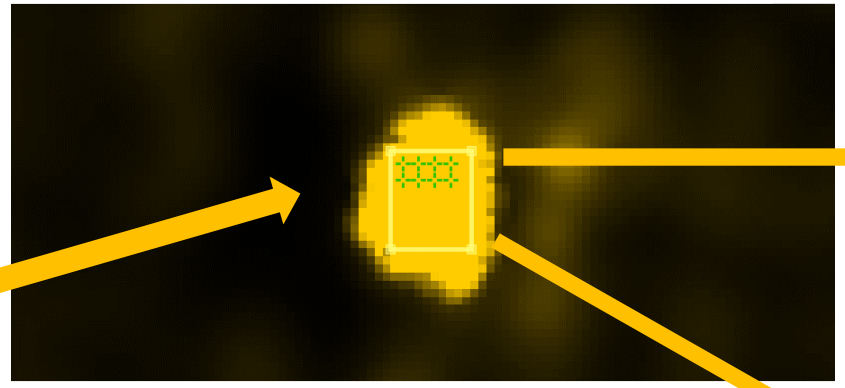
Distinct and clear Au-Lα and Au-Lβ X-ray energy Lines.

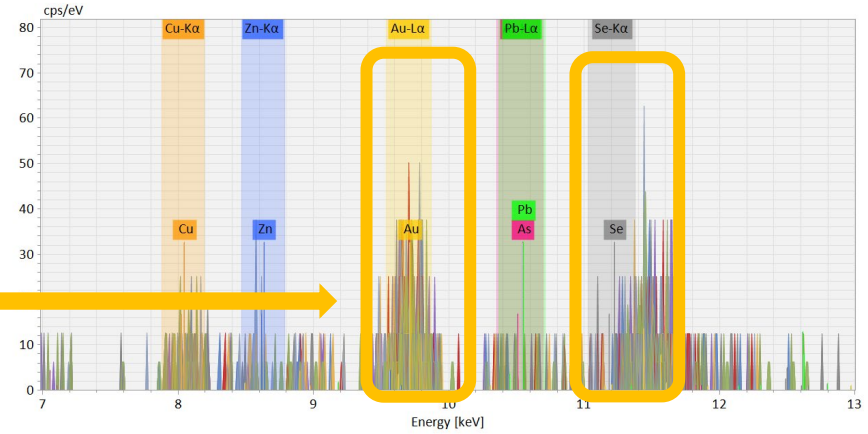
Presence of Au in the sample confirmed.



Micro-XRF on SEM (X-ray Excitation)
Identifying Gold (Au) in the Sample

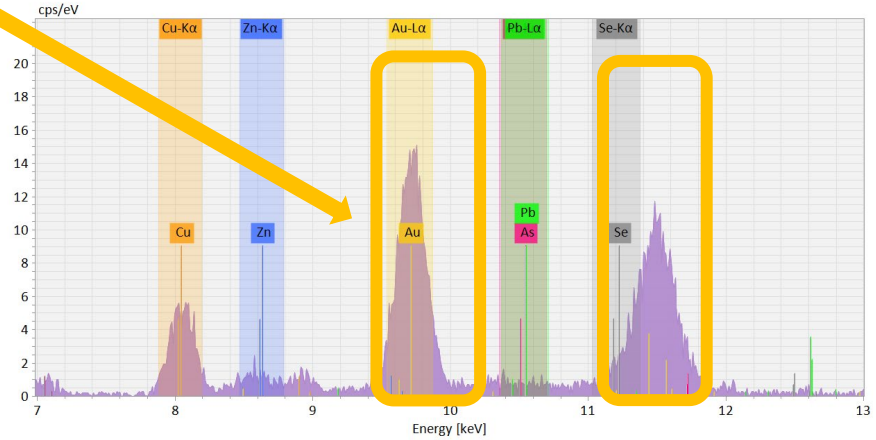






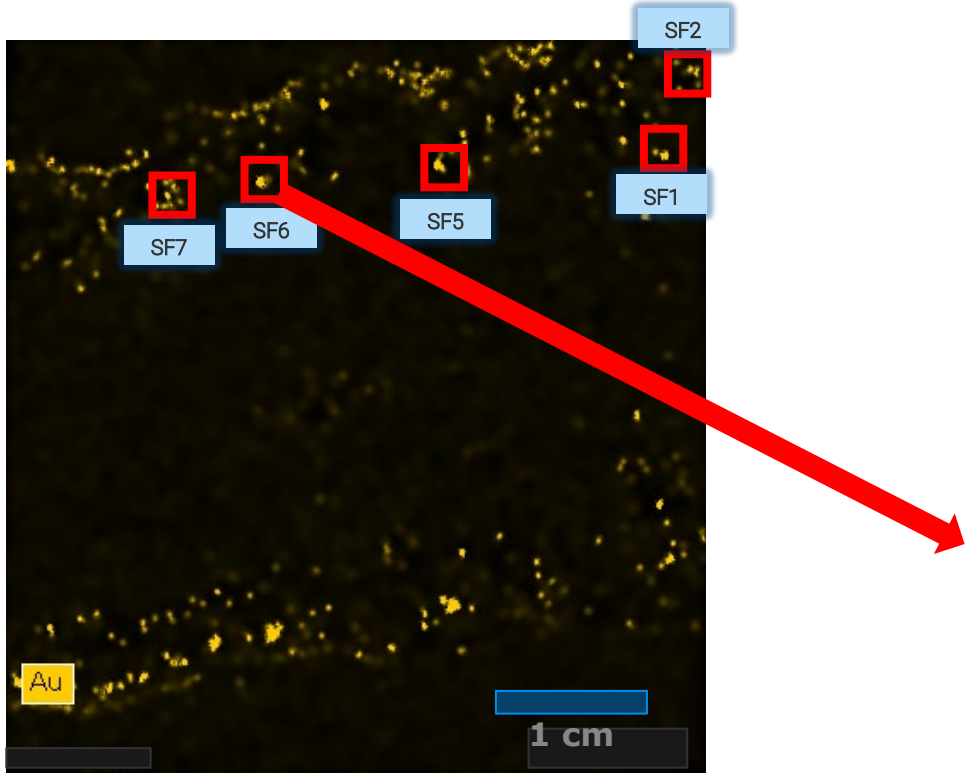
Individual Points Spectra – Low Counts

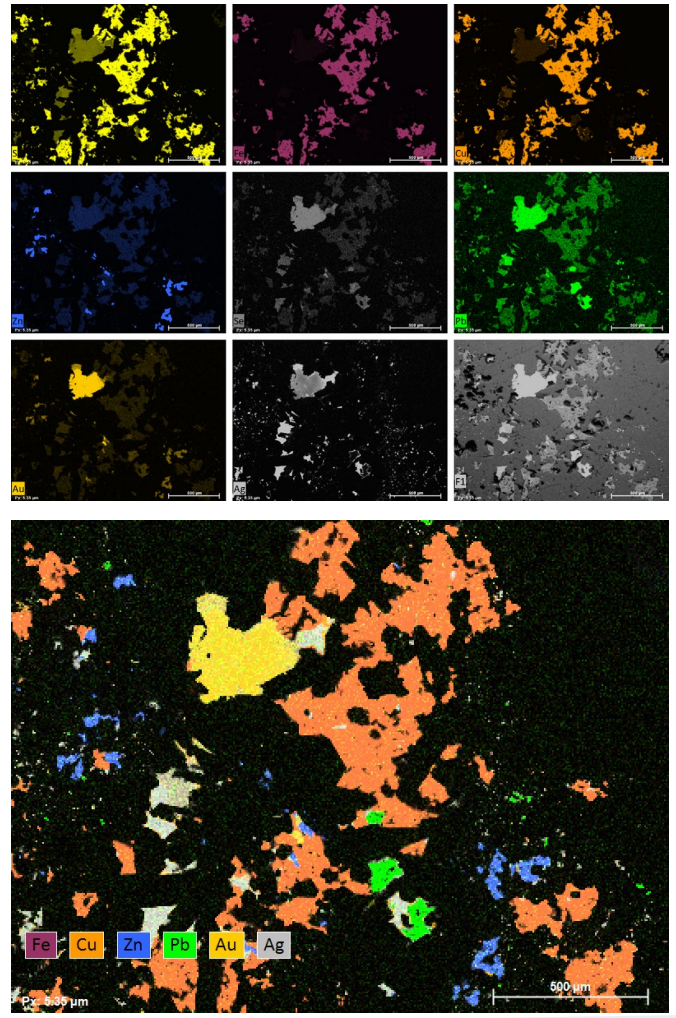
Distinct and clear Au-Lα and Au-Lβ X-ray energy Lines. Presence of Au in the sample confirmed



Summed points within Grain – High Counts, clear and confirmed elemental peaks

SEM-EDS (Electron Excitation)
Identifying Gold (Au) in the Sample





Single Field 6

Large Gold Grain;

Associated with Silver

Other mineralization:

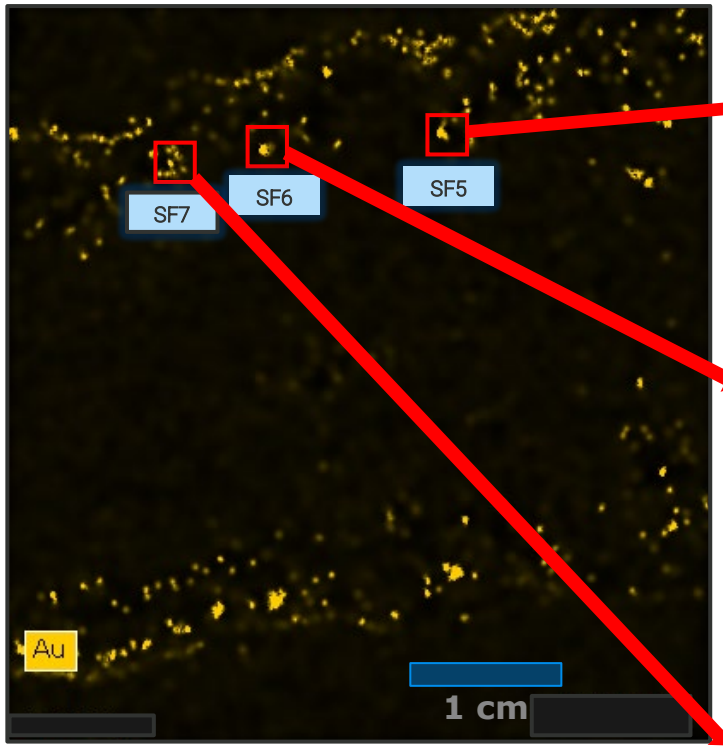
Pyrite (FeS2),

Chalcopyrite (CuFeS2),

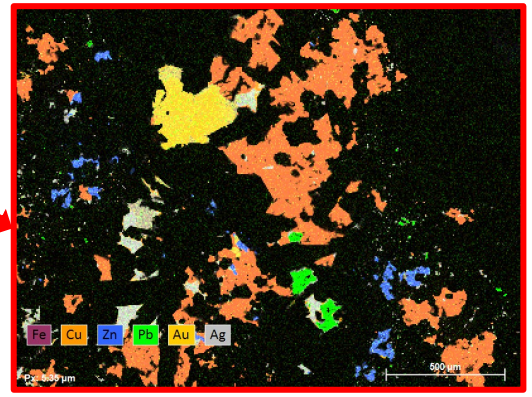
Galena (PbS),

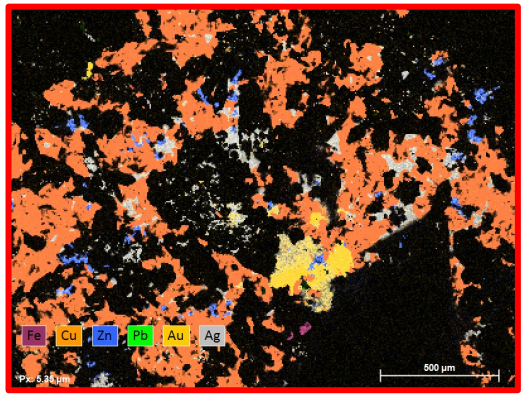
Sphalerite (ZnS)

SEM-EDS (Electron Excitation)
Identifying Gold (Au) in the Sample

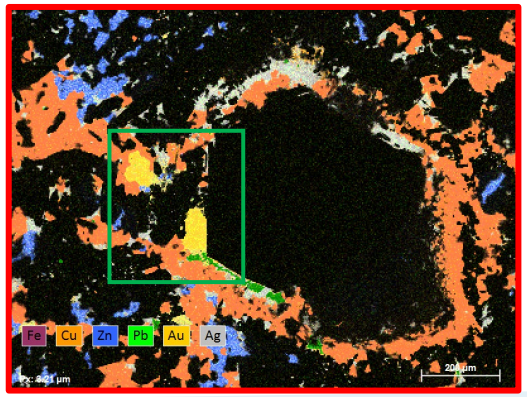


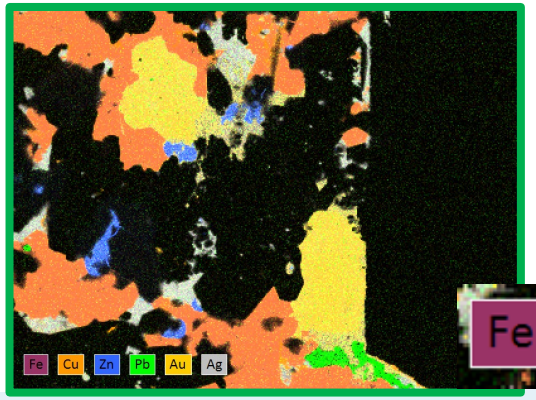
Micro-XRF: Large Area Map





SEM-EDS: Detailed Small Area Maps





Single Field 5, 6 and 7

Large Gold (Au) Grains;

Associated with Silver – Mineralogy Electrum

Other mineralization:

Pyrite (FeS2),

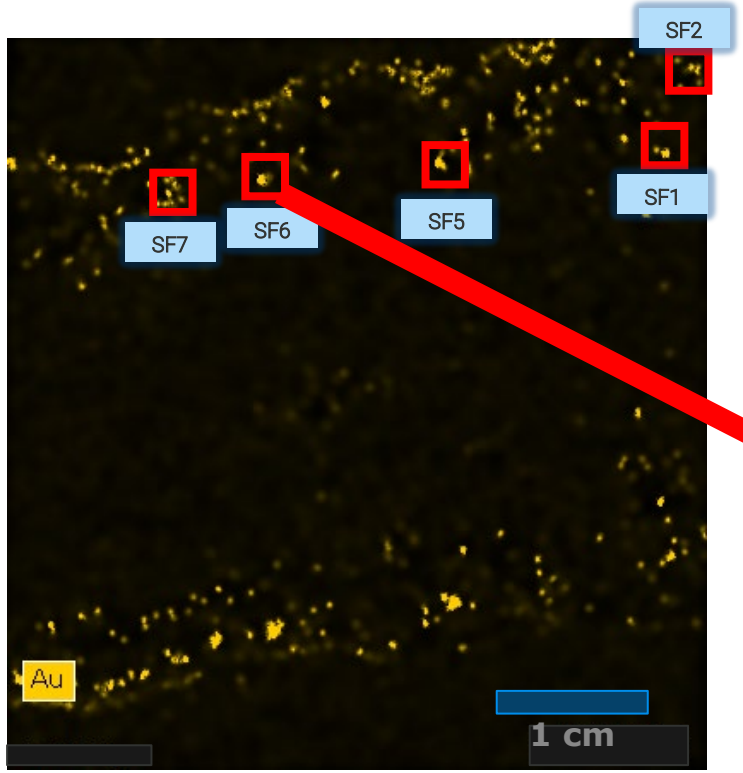
Chalcopyrite (CuFeS2),

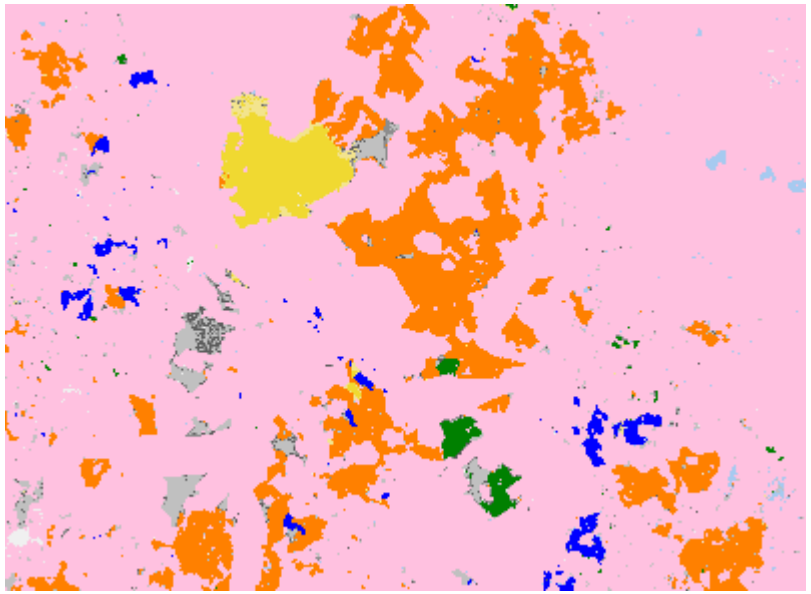
Galena (PbS),

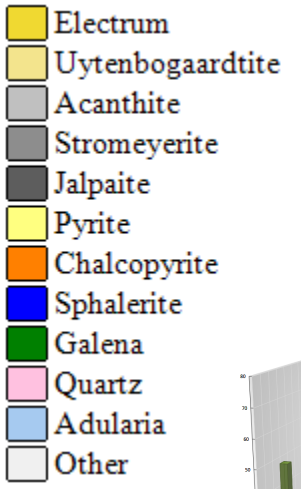
Sphalerite (ZnS)

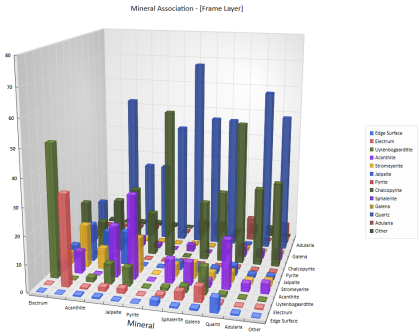
SEM-EDS (Electron Excitation) and AMICS

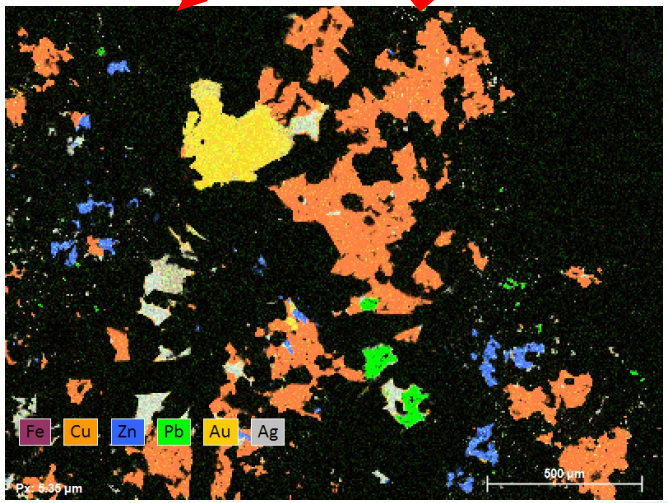
Area: SF6

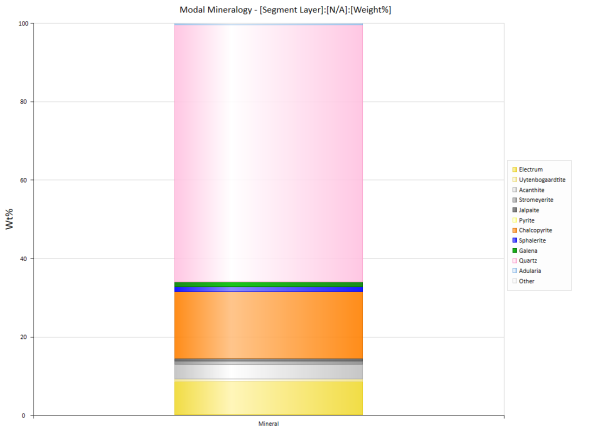


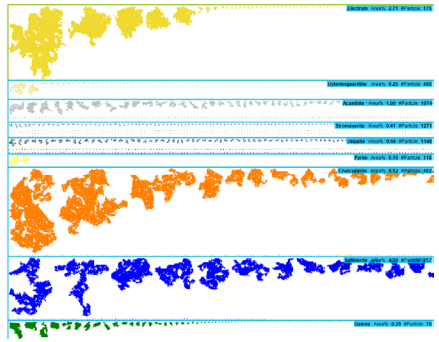






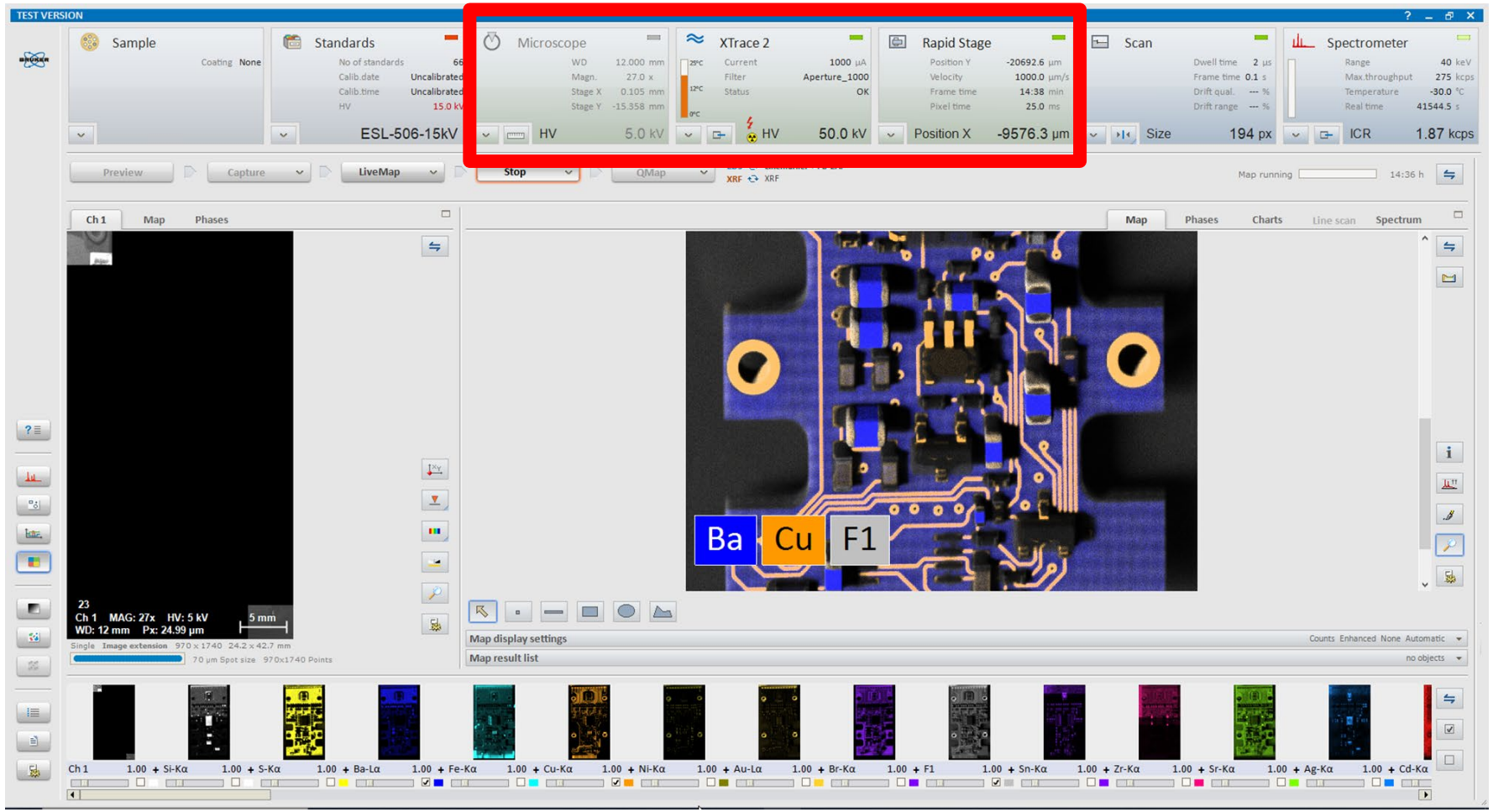






Full Range EDS Analysis: Software Updates – Esprit v2.6

Rapid Stage: Large Area Maps – SEM and X-Trace
Integration in ESPRIT Software (version 2.6)

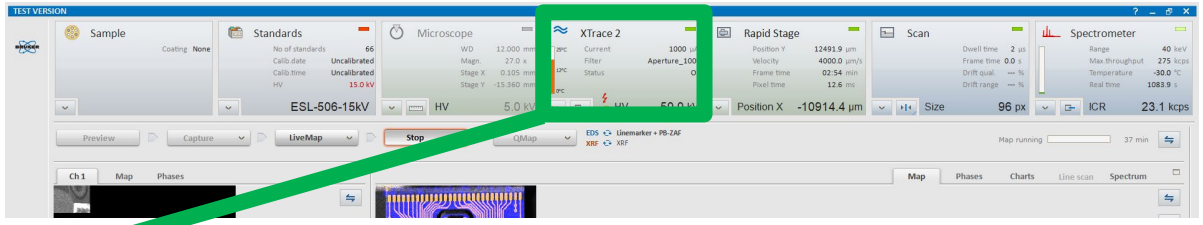


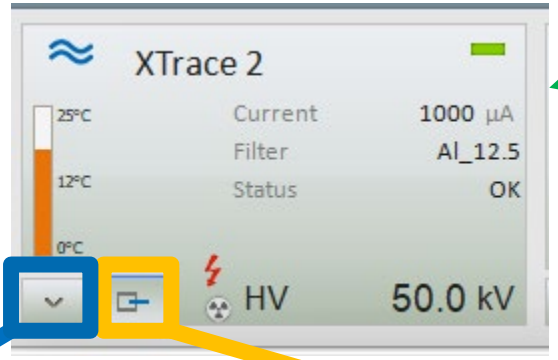
Large Area Mapping is possible with the Rapid Stage

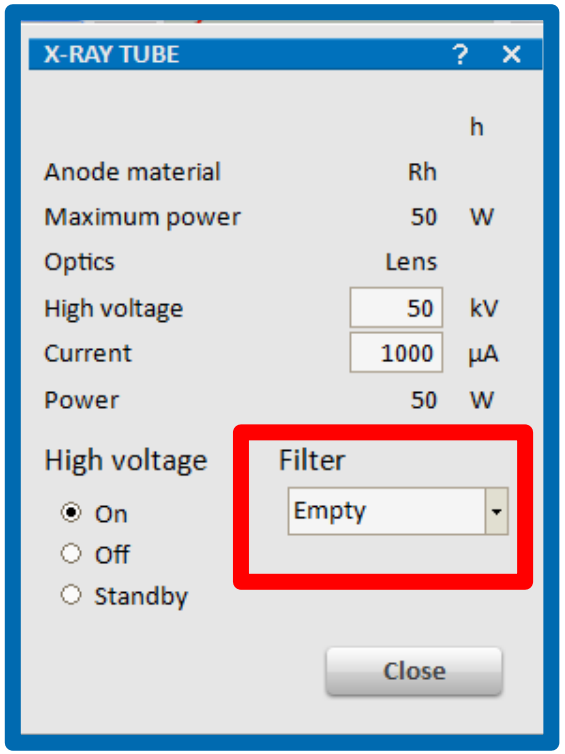
Integrated with both the SEM and the XTrace excitation sources

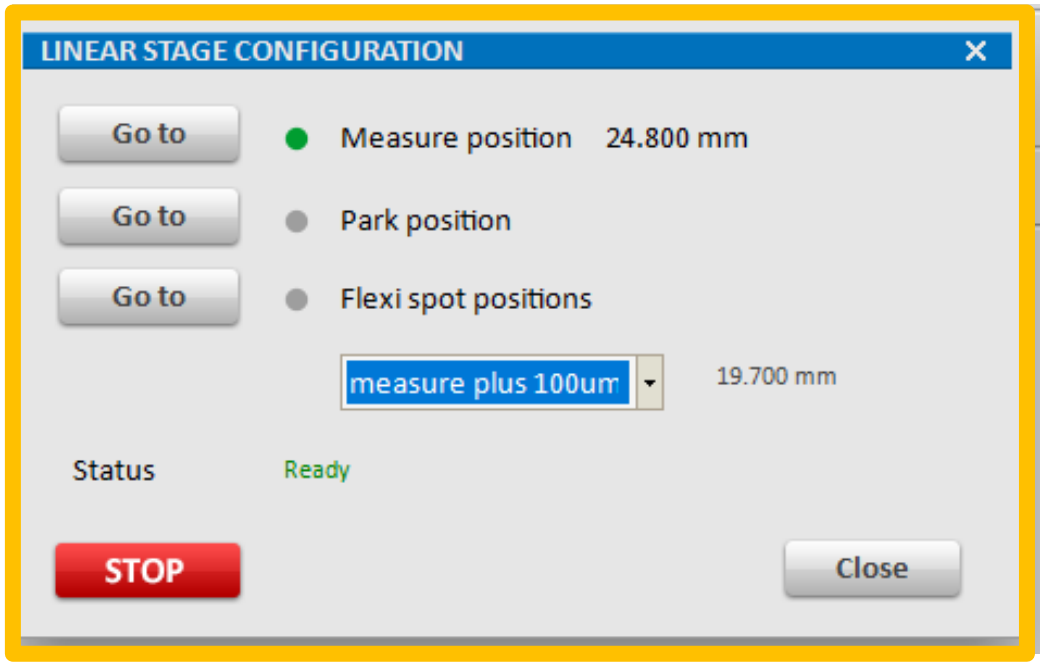
Benefits are that the a single are image is created (no mosaicing), thus removing potential aanalytical artifacts

SEM-XRF and Rapid Stage
Integration in ESPRIT Software (version 2.6)

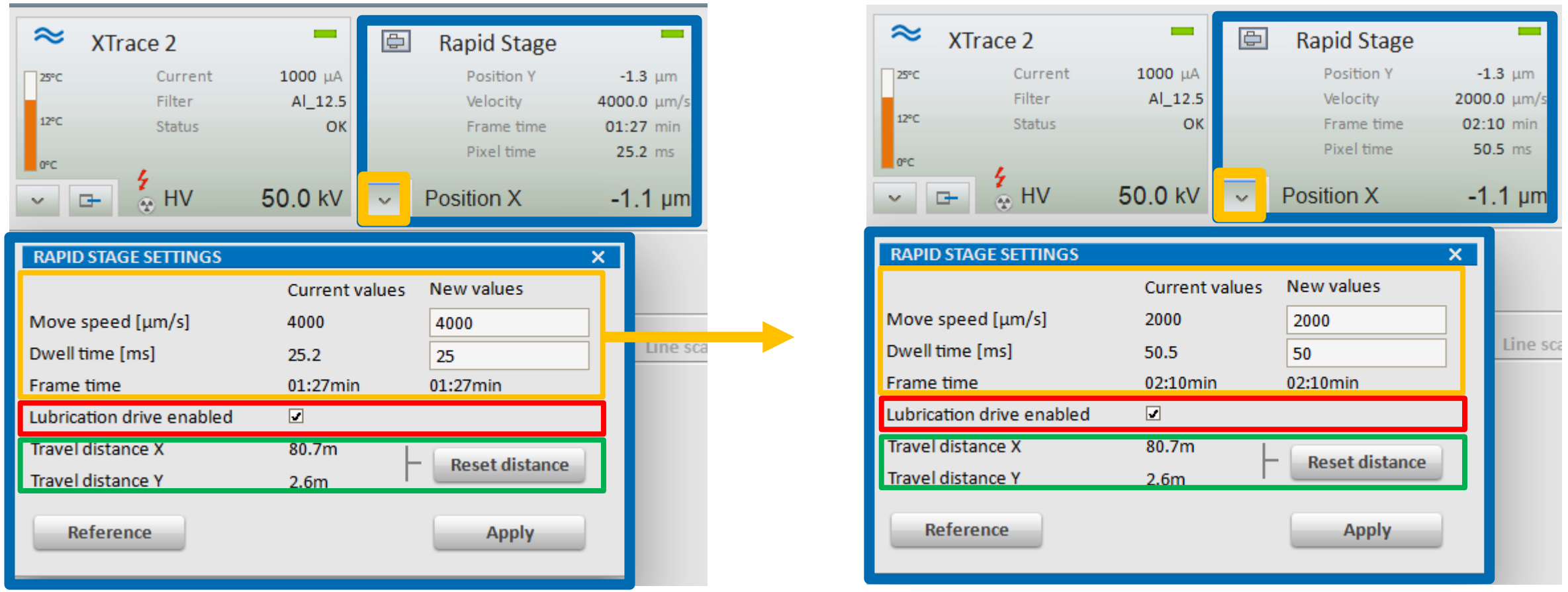


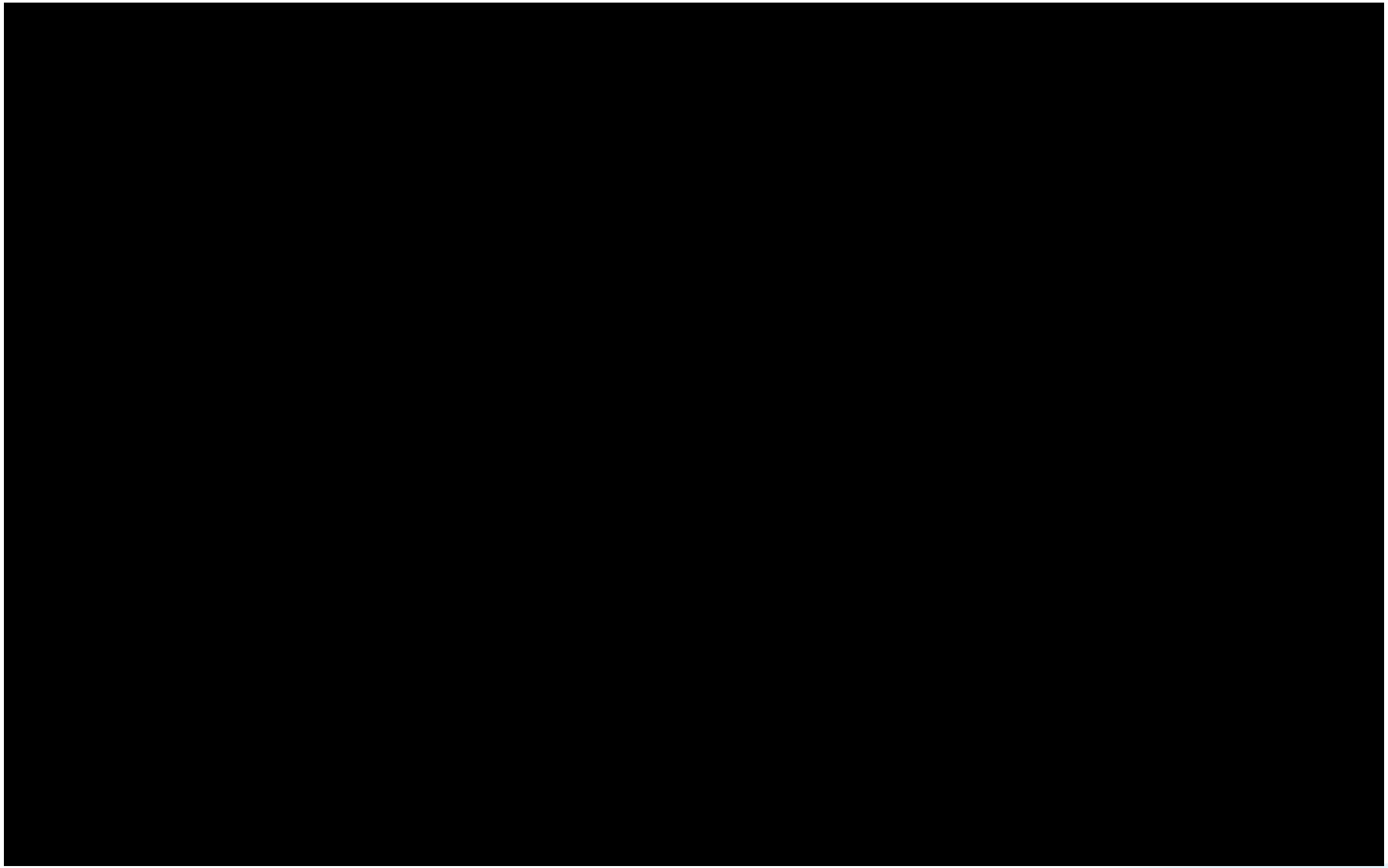






SEM-XRF and Rapid Stage
Integration in ESPRIT Software (version 2.6)





Full Range EDS Analysis with SEM-XRF
Summary and Conclusions

Summary and Conclusions: Full Range EDS

2 Excitation Sources:

Electron Beam (e-beam)

Micro-XRF (X-ray beam)

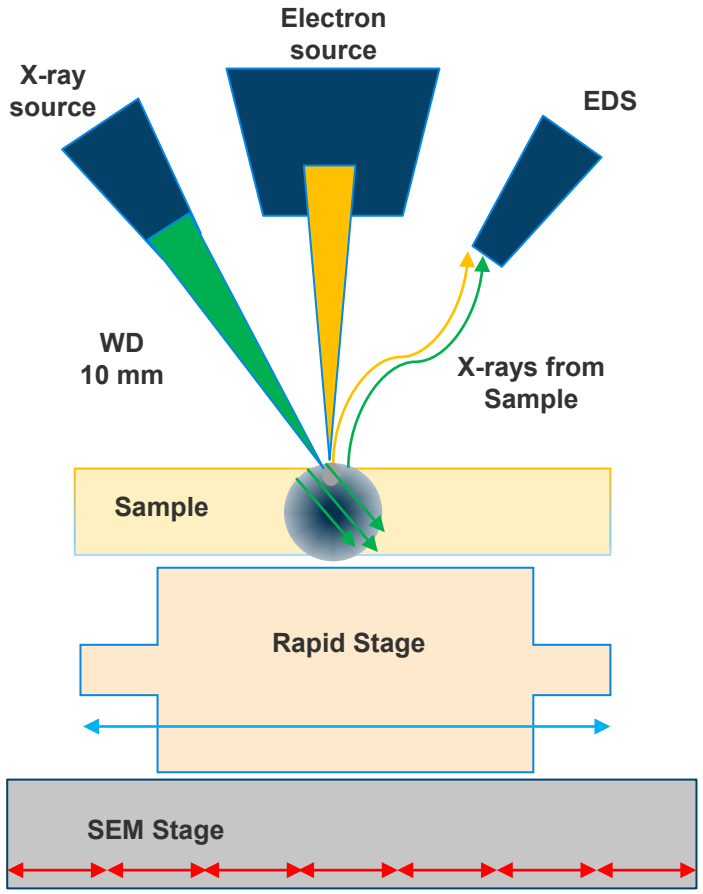
1 Detector:

Energy Dispersive Spectrometer (EDS)

2 Stages:

SEM Stage

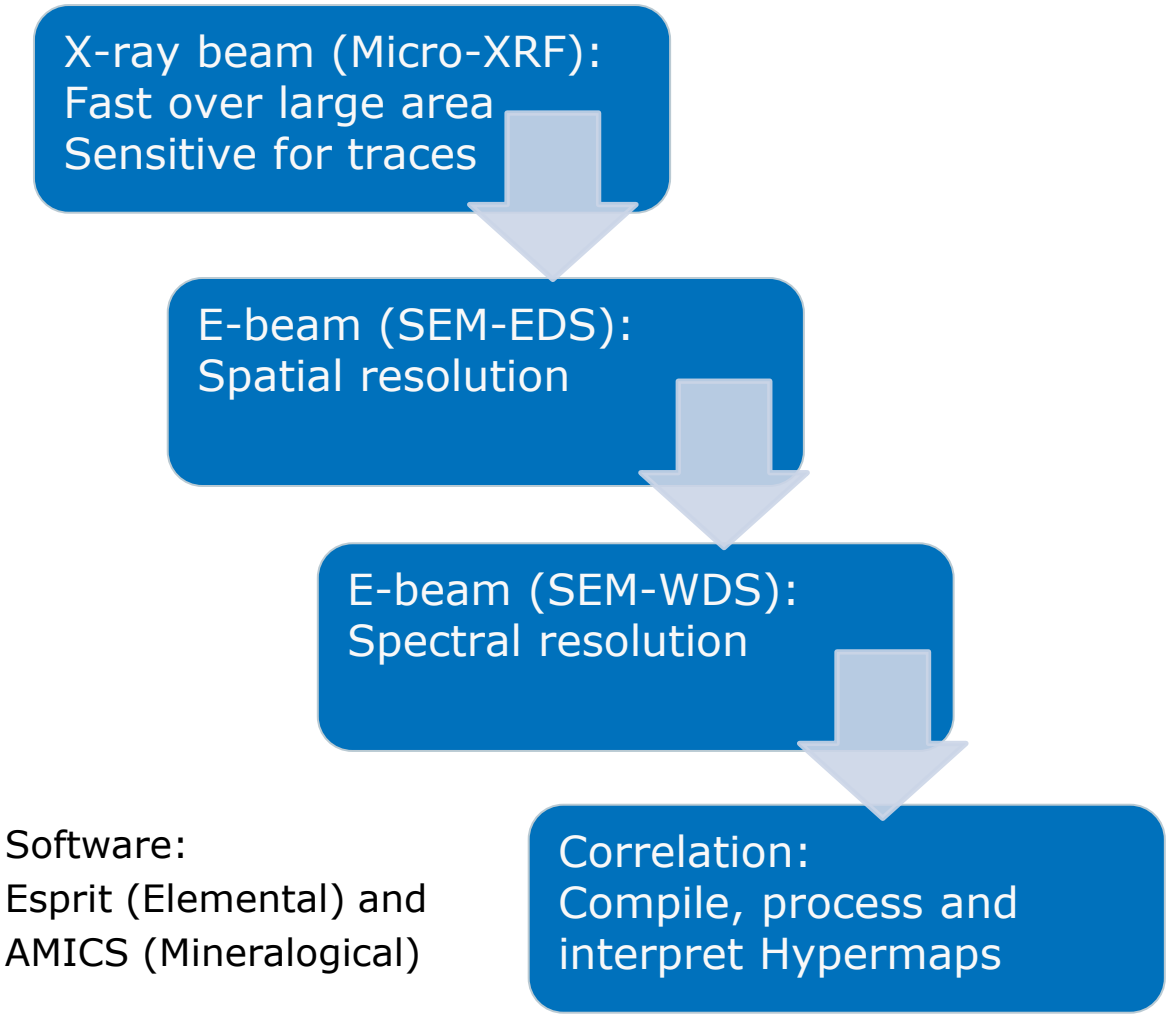
Rapid Stage



Micro-XRF Benefits for EDS Analysis:

- Non-destructive analytical technique
- No charging effects
- Minimal Sample Preparation Required
- Lower detection limits (down to 5 ppm)
- High Energy Lines Detection (Full Spectrum Range up to 40 keV)
- Ideal for Low kV or Beam sensitive samples
- Fast Large Area Mapping
- Micrometer scale measurement over cm

Workflow: Correlating Micro-XRF / e-beam / EDS / WDS analysis



Software:
Esprit (Elemental) and
AMICS (Mineralogical)

Micro-XRF (M6 JETSTREAM, M4 TORNADO, SEM-XRF (XTRACE))

- Fast analysis over large area
- Confirm presence of elements of interest
- Identify areas for further analysis
- Store stage positions of those areas

SEM-EDS

- High spatial resolution
- Fast analysis over small area
- Identify elemental and mineralogical relationships and associations on the micro- nano- scale.

SEM-WDS

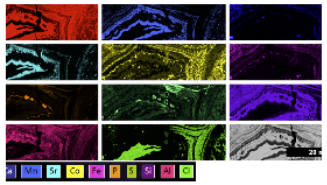
- High spatial resolution (similar to EDS)
- Resolution of peak overlaps
- Low detection limits
- High sensitivity for low X-ray energy range

Full Range EDS and Micro-XRF on SEM (XTrace): Further Information

LinkedIn Posts: #BrukerXTrace

https://www.bruker.com/

Search for:
QUANTAX Micro-XRF



Large Area Mapping of Mineralogical Samples

The new Rapid Stage is specifically designed for SEMs to enable large area mapping over millimeter (mm) to centimeter (cm) scales. This will eliminate potential SEM X-ray intensity variation artifacts associated with low magnification mapping and thus enhance elemental and mineralogical information in a timeous manor that was previously not possible.

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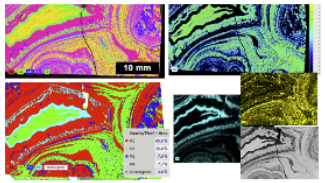

Elemental and Mineral distribution in Exotic-Cu Deposits

The ability to observe elemental changes within samples is important to understand geological processes and ore deposit genesis. The dual source system which incorporates a micro-XRF on a SEM enables elemental X-ray mapping over large areas, which shows major, minor and also trace elements on the ppm scale.

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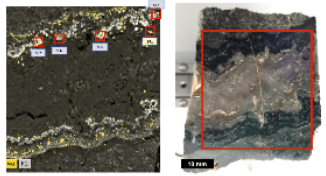

Dual Source Applications for Exploration and Mining: Au-bearing Epithermal Samples

The combination of micro-XRF with SEM enables the potential to analyze samples at multiple scales, from centimeters (cm) to millimeters (mm) to micrometers (µm) and below within a solitary system. Thus, by adding the micro-XRF to an SEM you convert your SEM to a dual source system, meaning that there are 2 excitations sources, the e-beam and photon beam. Either source can be used individually, or simultaneously, to generate sample X-rays that will be measured using the same EDS detector.

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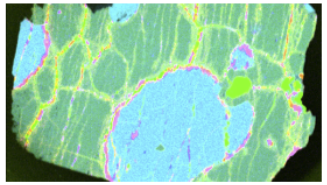

Mantle Petrology and the Source of Diamonds

We present a SEM-XRF element map of a mantle garnet-spinel peridotite from the diamond-bearing Newlands kimberlite (South Africa, Kaapvaal Craton). The intensity of the various elements indicates certain minerals that are present in the sample.

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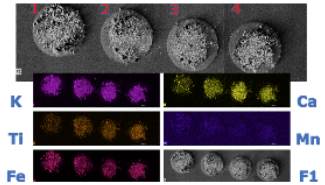
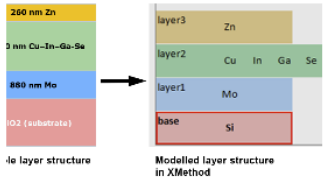


Identification of Contaminants and Toxins in Soils

Large Area Mapping (Hypermaps) using SEM-XRF can be performed on samples with topography. That is, minimal sample preparation is required and the sample can be analyzed directly without any degredation. This is particularly relevant in the analysis of soils, where any form of sample preparation, such as mounting and polishing or carbon coating, may alter the specimen.



Thin Film Analysis with SEM micro-XRF

As X-rays may pass through matter, X-ray Fluorescence (XRF) allows the determination of layer thickness. Using micro-XRF on SEM, the layer analysis (thickness and composition) is rendered feasible with spatial resolution at the micrometer scale. Layer analysis is strongly based on quantification using atomic fundamental parameter (FP).

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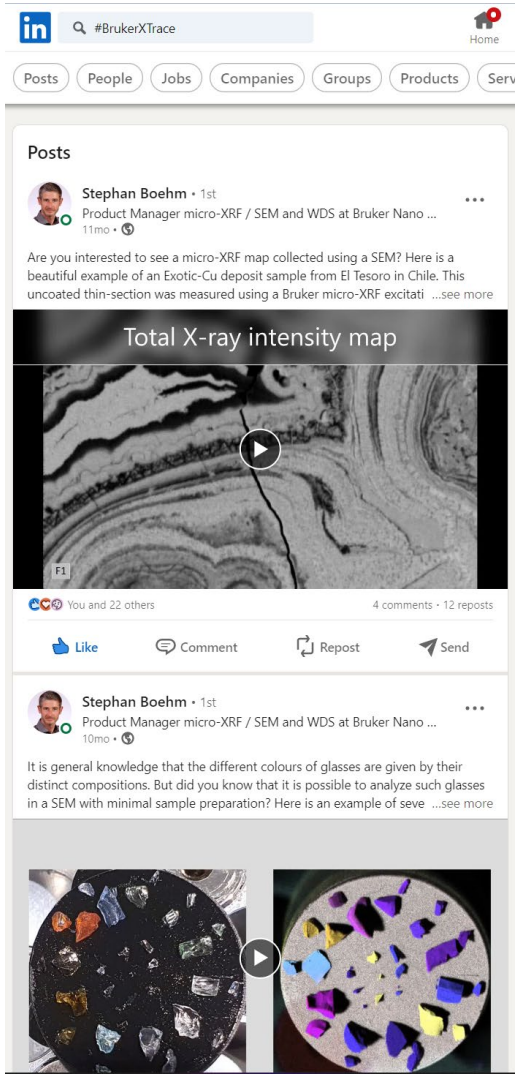

More Information

For more information, please contact us:

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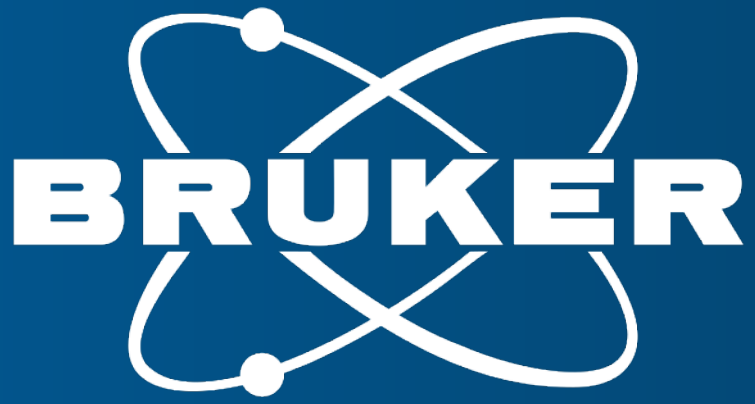
Thank you for your time.

Any Questions?



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