Introducing XTrace 2: The Next-Generation for Enhanced Micro-XRF on SEM

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SEM-XRF (XTrace): Introduction

01 Introduction : SEM-XRF and Rapid Stage

02 The New XTrace 2

03 XTrace 2 Features and Examples

04 Summary and Conclusion

05 Questions and Answers

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MicroXRF on SEM (SEM-XRF): Introduction

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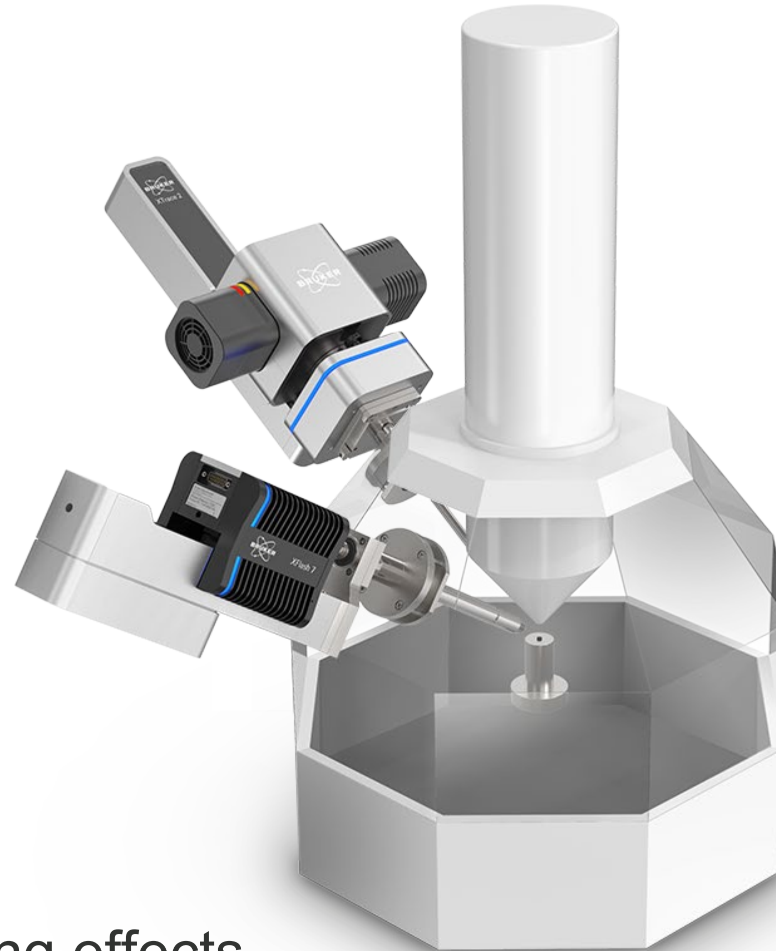
QUANTAX Micro-XRF System– Key facts:

10 ppm

detection limit

Trace element analysis dueto a low spectral background



1 nm – 40 µm

layer thickness range

Analysis of thin films and multi-layered structures

Minimal sample preparation

No coating required, no charging effects

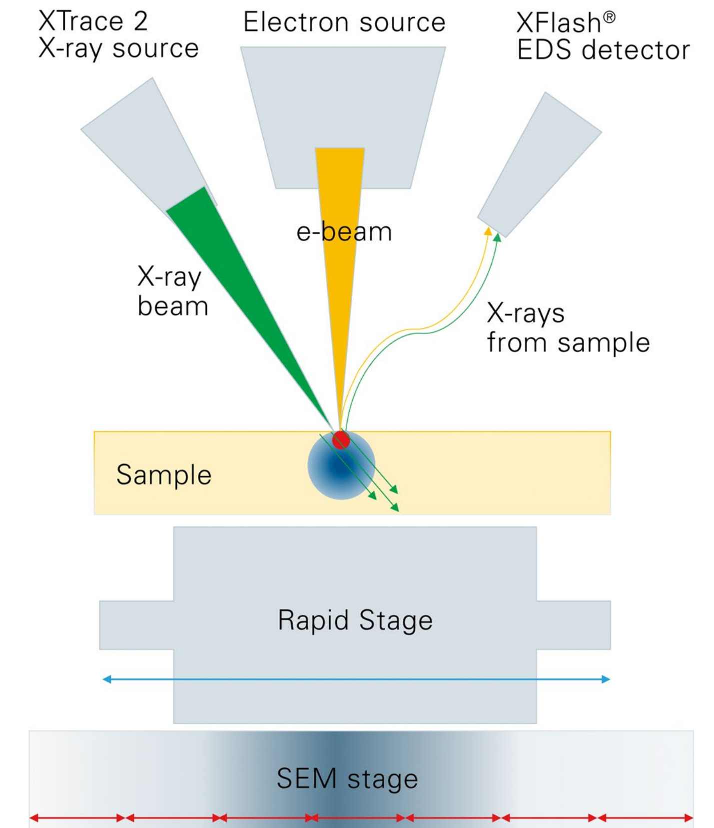
QUANTAX Micro-XRF System – Expand your SEM analytical capabilities

Micro-XRF on SEM as a complementary analytical technique to EDS analysis

Micro-X-ray Fluorescence (micro-XRF) spectroscopy is a non-destructive analytical technique that can be used alongside conventional Energy Dispersive Spectroscopy (EDS) on a Scanning Electron Microscope (SEM).

Micro-XRF on SEM, also known as SEM-XRF, empowers the SEM with a range of new analytical capabilities, using the same EDS detector already installed on the SEM.

Micro-XRF on SEM will convert the standard SEM to a dual beam source system (e-beam + photon beam), which can be activated either independently or simultaneously to obtain the benefits of each excitation method.



Complete EDS Analytical Solution

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QUANTAX Micro-XRF System – Expand your SEM analytical capabilities

Benefits of using a photon beam source on a SEM

- **Non-destructive analytical technique:** No heating or charging on e-beam sensitive samples
- **Minimal sample preparation:** No coating or polishing requried
- **Trace element detection limits:** Due to the low background (high signal-to-noise ratio), detection limits as low as 10 ppm are possible (element and matrix dependent)
- **Wide elemental identification range:** Majority of elements in the periodic table can be identified, from carbon (Z = 6) to uranium (Z = 92)
- **High energy line detection:** Extended X-ray spectral range (up to 40 keV)
- **Large area mapping:** Sample size for elemental mapping can be up to the SEM sample chamber size: micrometer scale measurement over centimenter scale sample size
- **Multi-layered sample analysis:** The larger depth of X-ray excitation allows for the characterization of multilayer systems

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Micro-XRF on SEM (XTrace): Further Information

LinkedIn Posts: #BrukerXTrace

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QUANTAX Micro-XRF

Trace Element Sensitivity with Minimal Sample Preparation

High-Speed Elemental X-ray Mapping even over Large Areas

Film Thickness Analysis

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 degradation. 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.

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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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Product Manager micro-XRF / SEM and WDS at Bruker Nano ...
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Are you interested to see a micro-XRF map collected using a SEM? Here is a beautiful example of an Exotic-Cu deposit sample from El Tesoro in Chile. This uncoated thin-section was measured using a Bruker micro-XRF excitati ...see more

Total X-ray intensity map

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It is general knowledge that the different colours of glasses are given by their distinct compositions. But did you know that it is possible to analyze such glasses in a SEM with minimal sample preparation? Here is an example of seve ...see more

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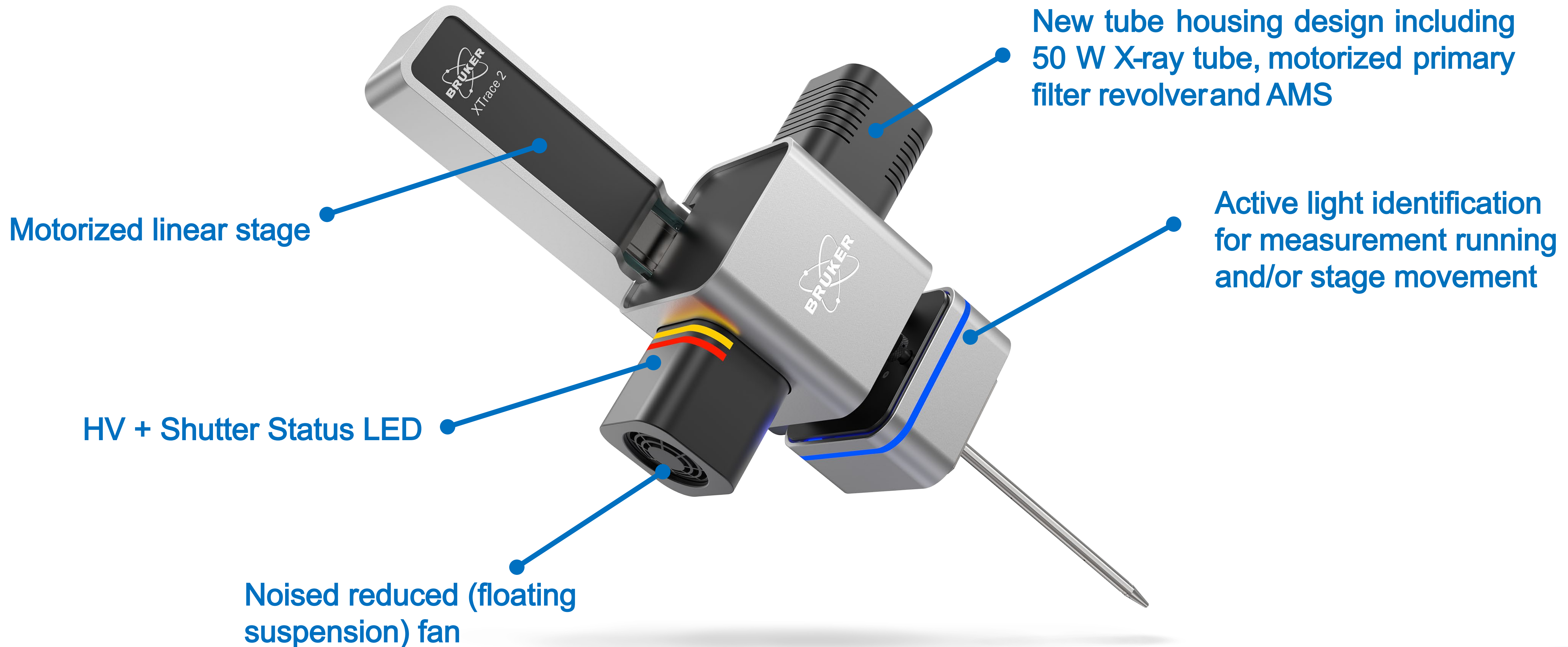
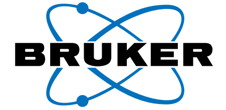
Introducing the new XTrace 2

XTrace 2 – Next Generation X-ray Source for Micro-XRF on SEM





XTrace 2 – Advanced X-ray Source for Micro-XRF on SEM

XTrace 2 is the next-generation X-ray source in the QUANTAX micro-XRF system. This new and innovative X-ray source enables fast micro-XRF spectral acquisition with high-resolution XRF data. Advanced features include:

- High tube power: Generate X-rays at **high energies of 50 kV** and **beam currents of 1000µA** for a high-count rate allowing accurate elemental data to be collected quickly and efficiently.
- Automatically switch between **6 primary filters** to reduce measurement background and precisely acquire low-count peaks.
- Scan topographic samples with high resolution XRF signal intensity using an **Aperture Management System (AMS)** that keeps the image in focus across variable measurement heights.
- Analyze inhomogeneous and/or irregular samples using **FlexiSpot mode**, allowing spectral measurements using a small or large spot size.
- Improved equipment lifetime with the **automatic tube warm-up procedure** and motorized linear stage with **automatic source retraction** (measuring and parking position).
- Software capability for **saving and correlating** micro-XRF and ebeam measurements.

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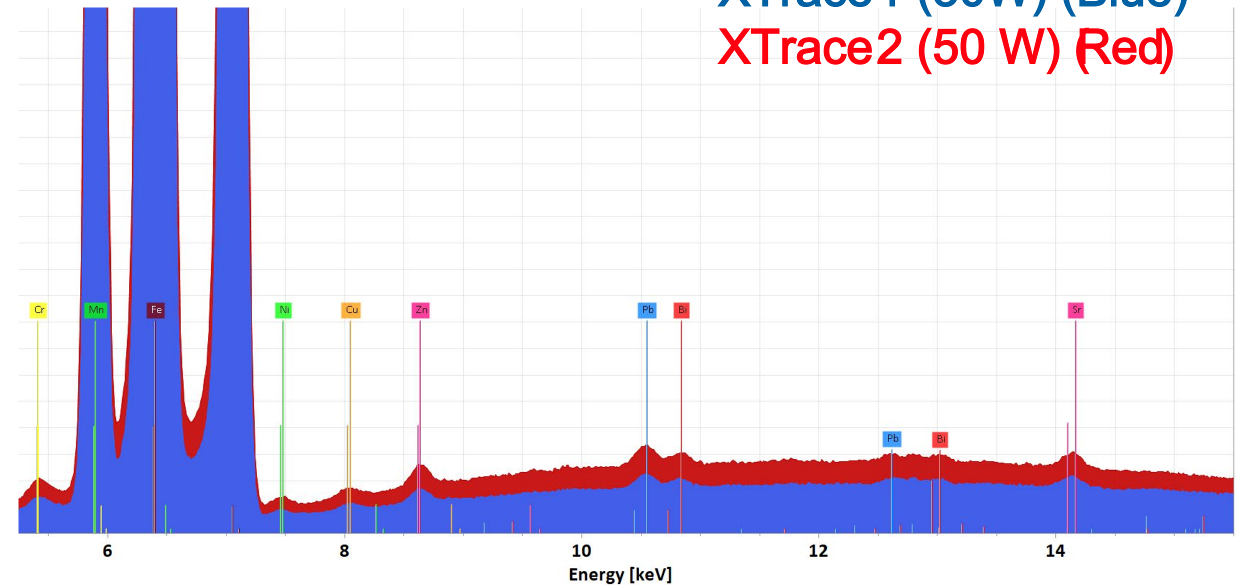
XTrace 2 – Significantly greater count rates

- XTrace 2 is equipped with a **50 W** X-ray tube / new tube housing
- Tube can now run at 50 kV / 1000 µA at maximum
- This enables up to **70 %** greater count rates and hence results in shorter acquisition times
- Can confirm the presence of trace elements much faster / allows to run X-ray maps at faster speed than before
- Automatic X-ray tube warmup regime for optimized X-ray tube lifetime

Spectrum:
XTrace 1 (30W) (Blue)
XTrace 2 (50 W) (Red)



Net Sum Counts

Ni 2010 (K)	Cu 5122 (K)	Zn 12715 (K)	Sr 12380 (K)	Zr 11381 (K)	Rh 131258 (K)	Pb 18716 (L)	Bi 11925 (L)
Ni 4014 (K)	Cu 8050 (K)	Zn 20336 (K)	Sr 16666 (K)	Zr 15289 (K)	Rh 178326 (K)	Pb 25533 (L)	Bi 17074 (L)

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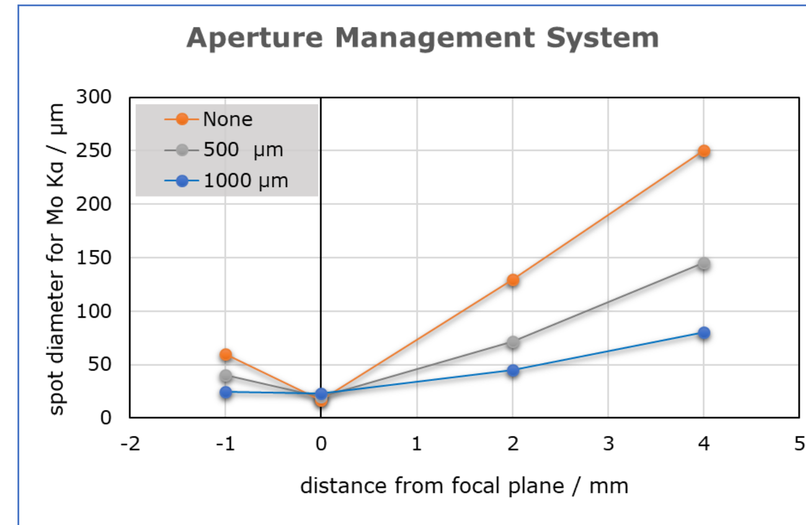
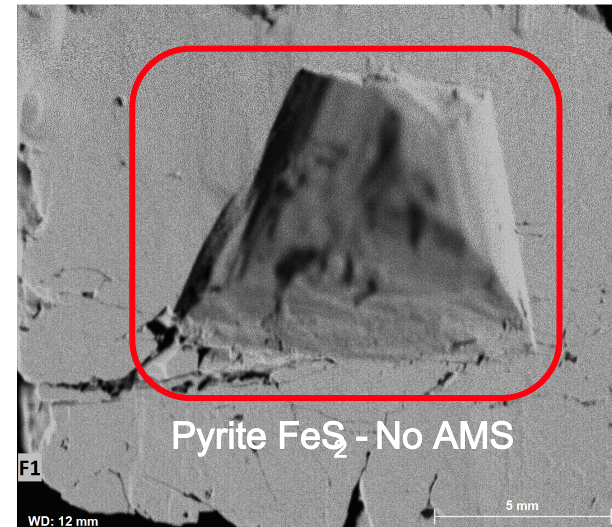
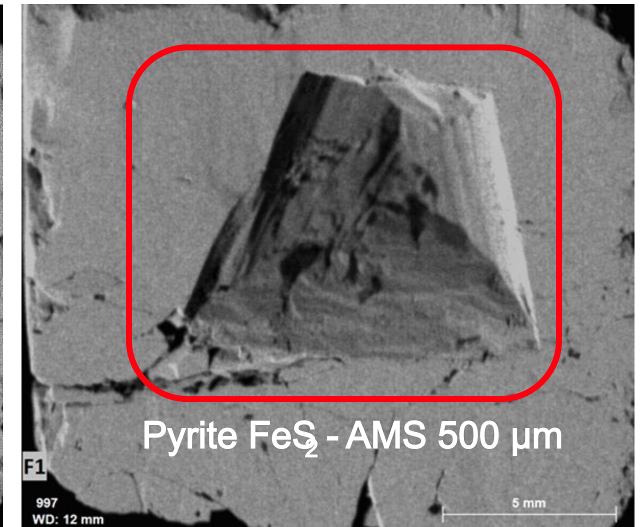
XTrace 2 – Scanning samples with topography

- X-ray polycapillary optics needs to be in correct WD to ensure smallest spot size, any changes in WD will automatically result in a much larger spot
- New (patent protected) aperture management system (AMS) keeps the optic in focus and retain measurement resolution over a certain range by increasing the depth of field
- This allows the examination & visualization of samples with topography even in a SEM



For standard micro-XRF analysis a 4 mm deviation from the working distance results in a ≈ 10 times increase in the spot size, i.e., a 10 times reduction in resolution. The new AMS will dramatically reduce this effect.





This example of a pyrite shows a micro-XRF map collected with no AMS (left) and with AMS (500µm) (right). Mapping the sample by using the AMS can even resolve the structures of the sample depth at **2.1 mm**.

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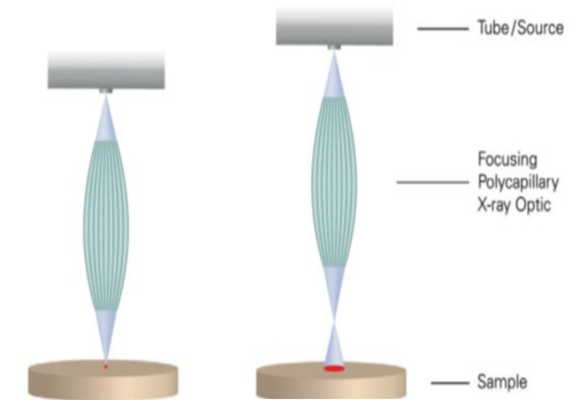
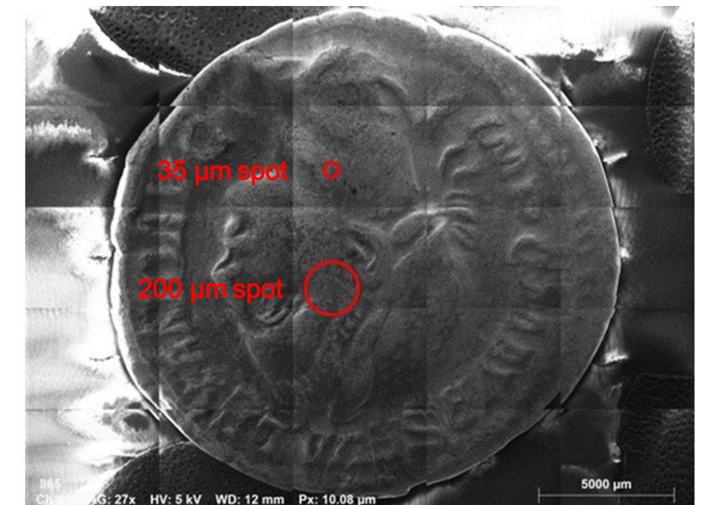
XTrace 2 – Working with variable spot sizes (FlexiSpot)

- Performing analysis with micrometer scale spatial resolution makes it challenging to find sample positions where the quantitative results are representative for the whole sample area (powders are typical examples)
- Larger X-ray spot´s allows more precise quantification of non-homogeneous and irregular shaped samples or even samples with uneven surfaces (powders), since sample information is integrated in the analysis over the enlarged detected area.
- FlexiSpot allows measurements at different spot sizes, ranging from 35 µm (standard optic spot size) to 500 µm and even beyond.
- FlexiSpot works by retracting the X-ray source, allowing the X-ray optic to be defocused out of the nominal optical working distance.
- The spot size will be selected in the ESPRIT software and allows the user to set multiple individual spot sizes

35 µm spot Defocused X-ray spot





Small (35 µm) and large (200 µm) X-ray spots on a sample.

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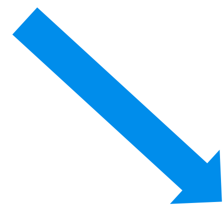
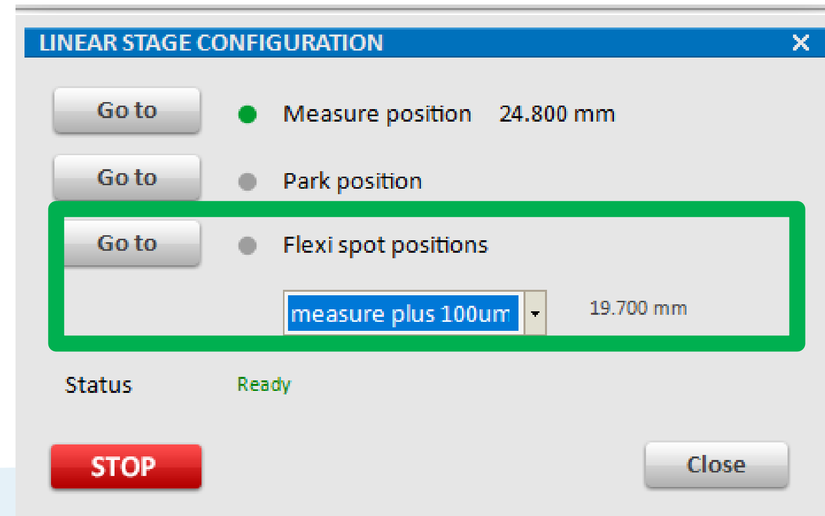
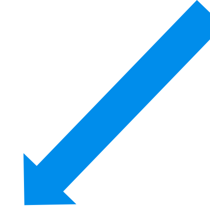
XTrace 2 – Working with variable spot sizes (FlexiSpot)



Software Menu

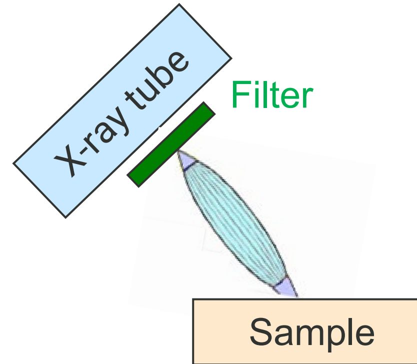


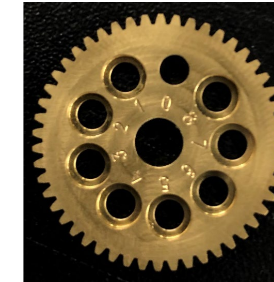


Dropdown Flexipoint Selection
Create your own analytical distance and thus spot size

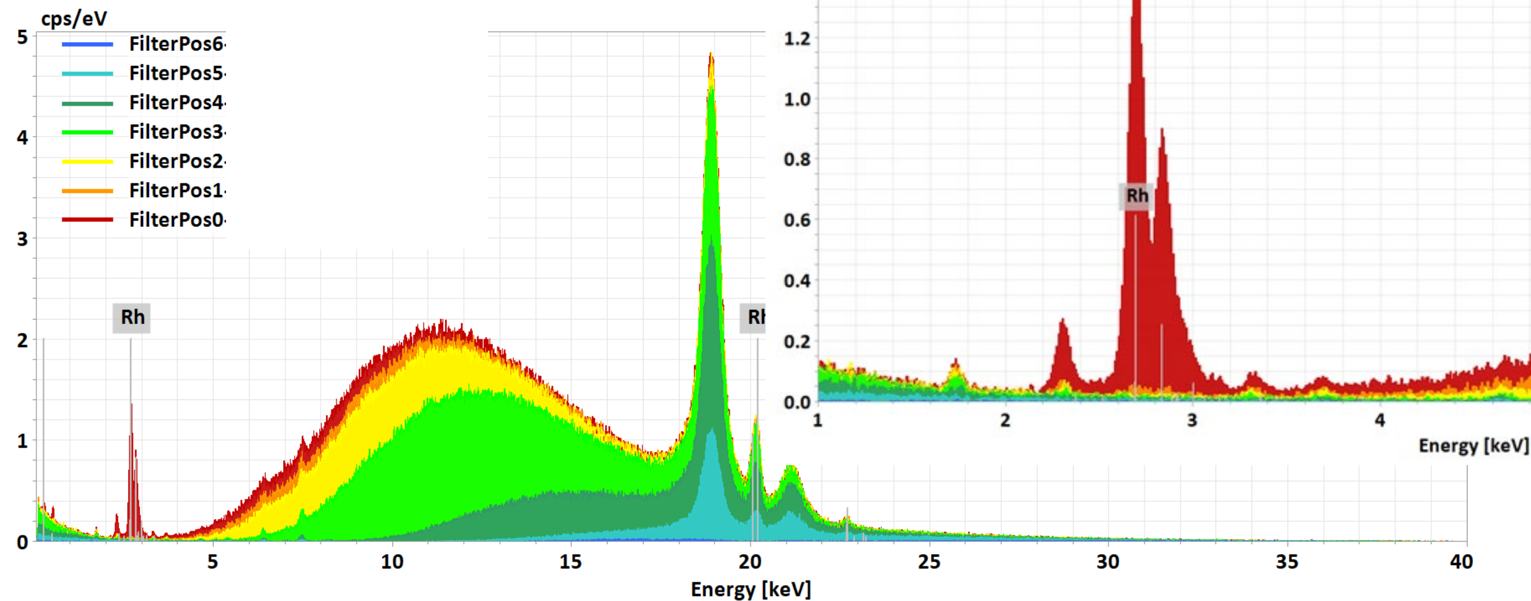
XTrace 2 – Enhanced background reduction / eliminating X -ray line overlaps

- Primary filters are a common practice in micro-XRF which:
  - Suppresses diffraction peaks
  - Eliminate X-ray line overlaps
  - smoothes the background for enhanced trace elemental sensitivity
- Primary filters are placed between the optic and the X-ray tube
- The new XTrace 2 has space for nine different options within the wheel
- Each filter allows further background reduction for various X-ray energy region´s. (up to 20 keV)





9 position filter wheel



Effect of different primary filters on an XRF excitation spectrum. Material and thickness of each individual filter impacts the spectrum region.

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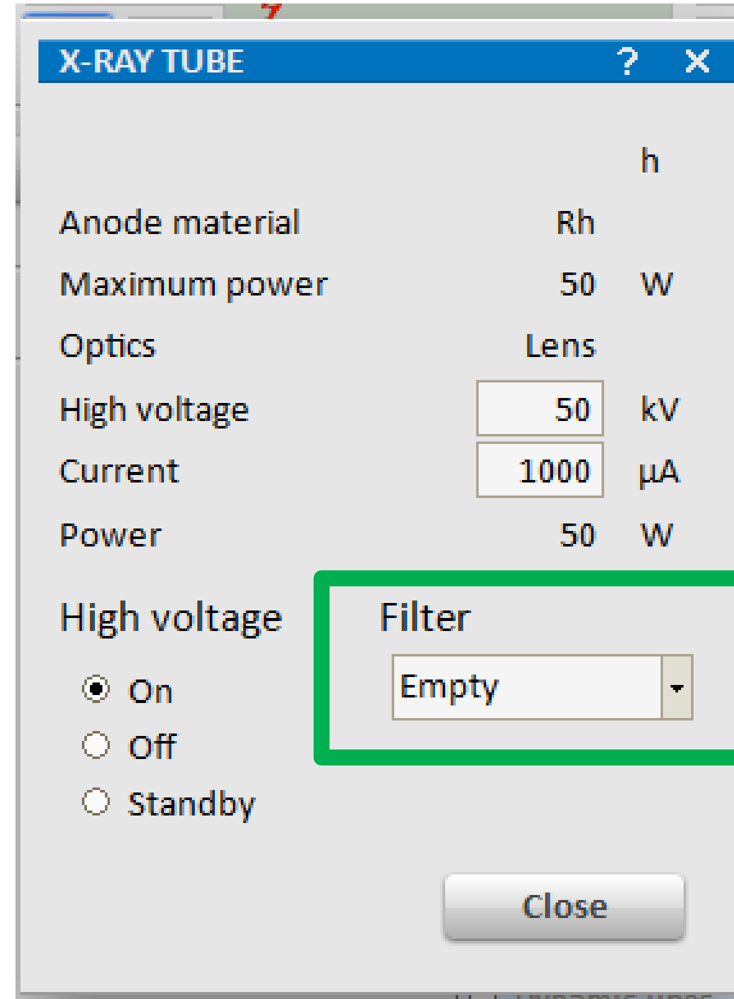
XTrace 2 – Enhanced background reduction / eliminating X -ray line overlaps

- Position "0" is left blank and is used for normal unfiltered analysis, whereas positions 1 to 6 are reserved for primary filters and positions 7 and 8 AMS apertures. Switching between positions is automatic via software control.



Software Menu

Dropdown Filter and AMS Options

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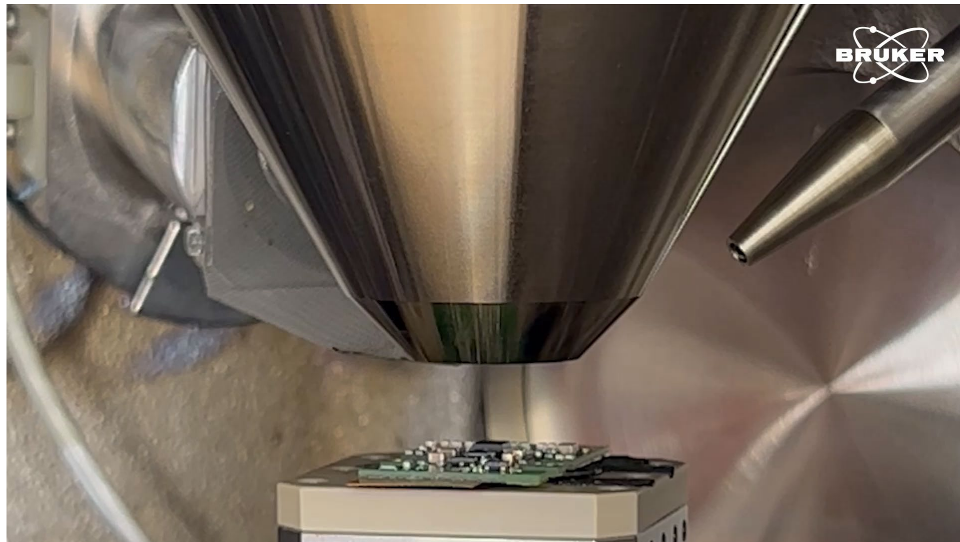
XTrace 2 – New safety features

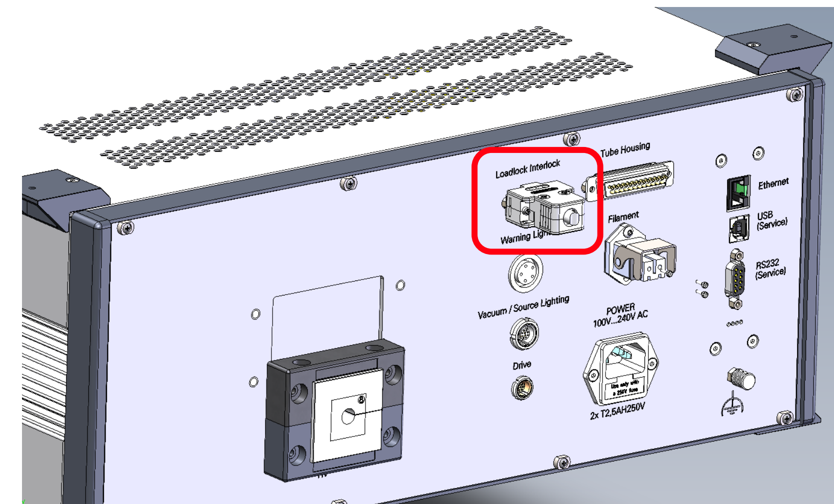
Auto source insertion and retraction mode

- The polycapillary optic of the X-ray source can be automatically inserted and retracted using XTrace 2 motorized linear stage.The source can also be retracted when the SEM sample chamber is vented for sample change.



Integration of SEM air - lock chamber status

- Status information of the SEM load lock chamber (open / closed) can be integrated in XTrace 2 security circuit for optimal X-ray safety purposes.
- Designed for the integration of various SEM load lock chambers (from different SEM vendors)



XTrace 2:
Feature and Application Examples

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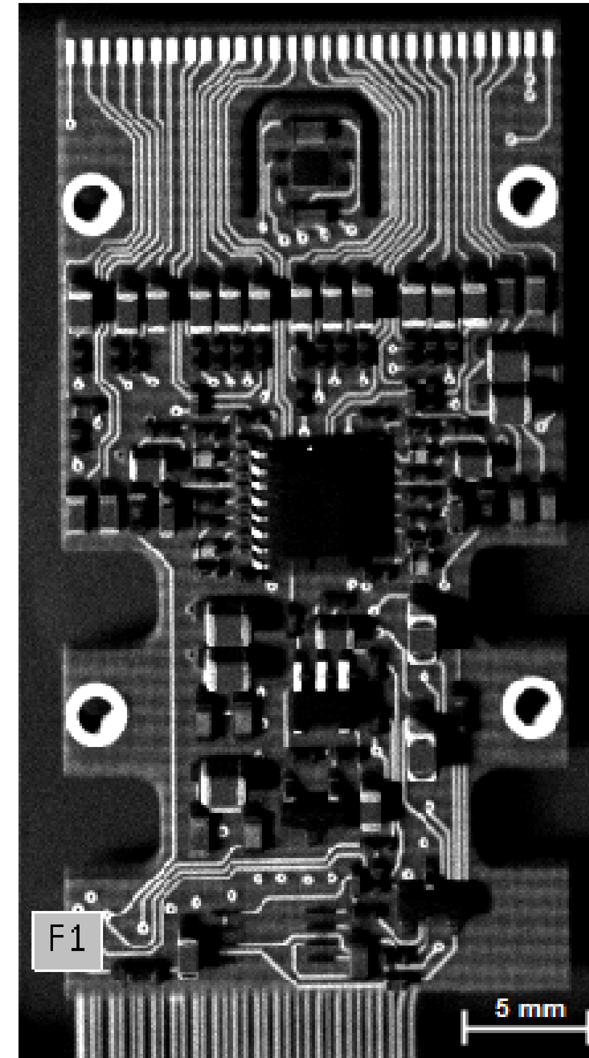
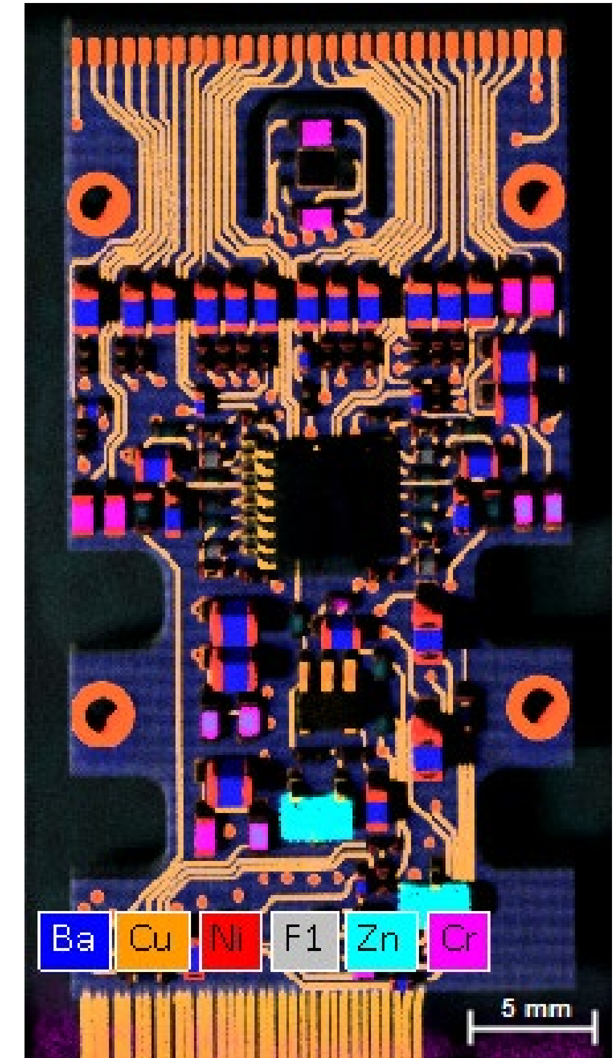
Fast elemental mapping of the electronic components on a Printed Circuit Board (PCB)

- 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

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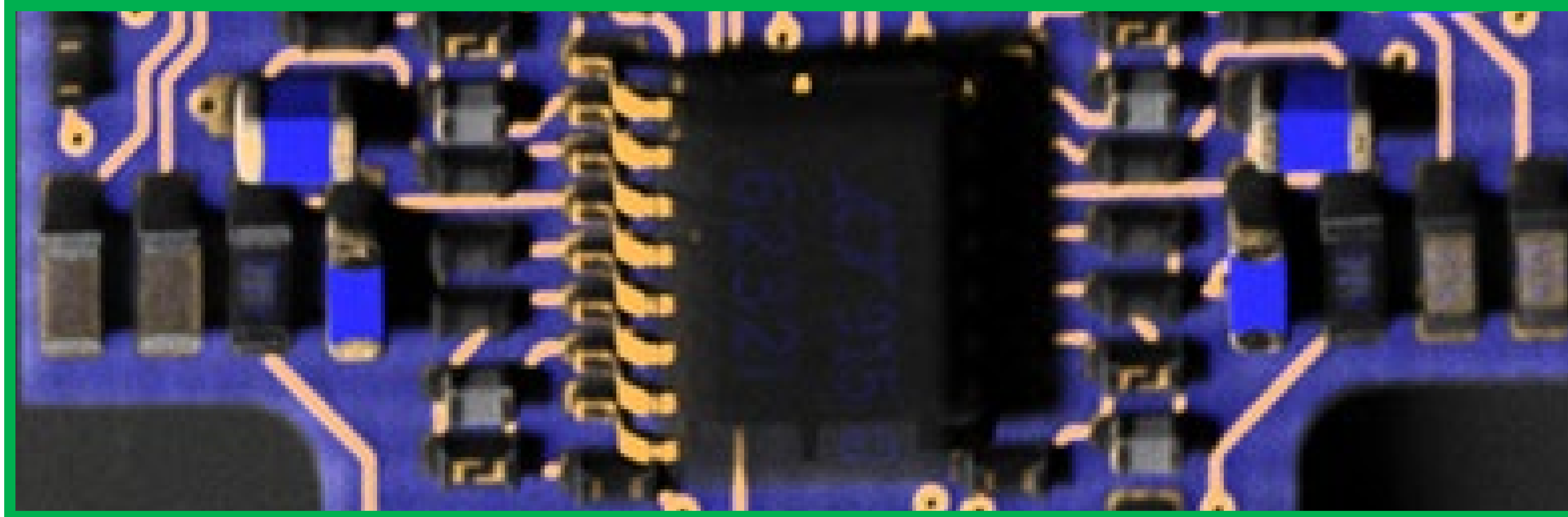
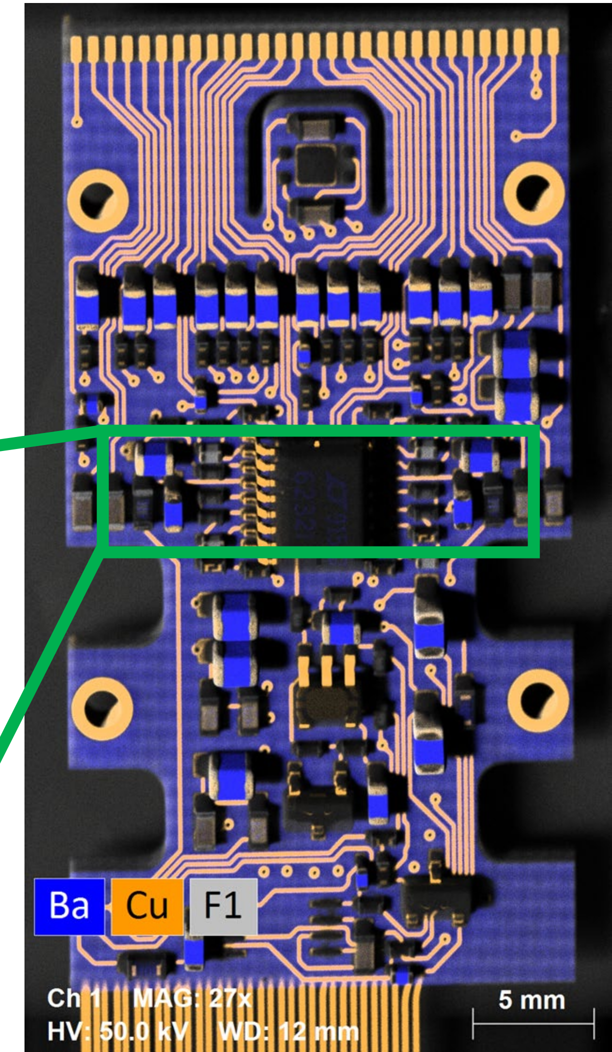
High-resolution elemental mapping of 3D features

- 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.





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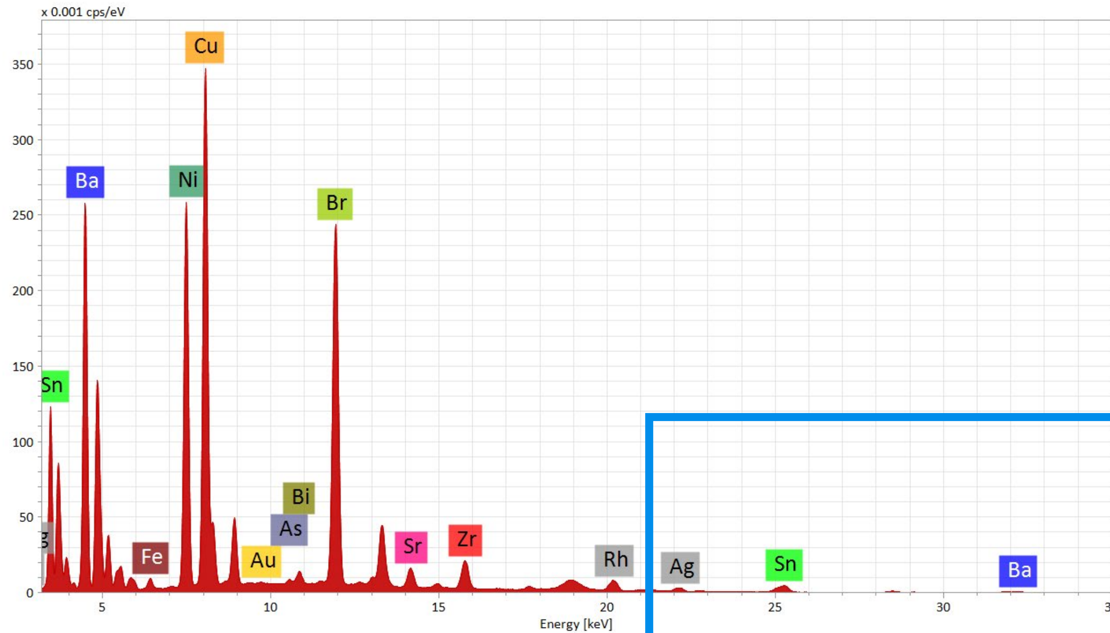
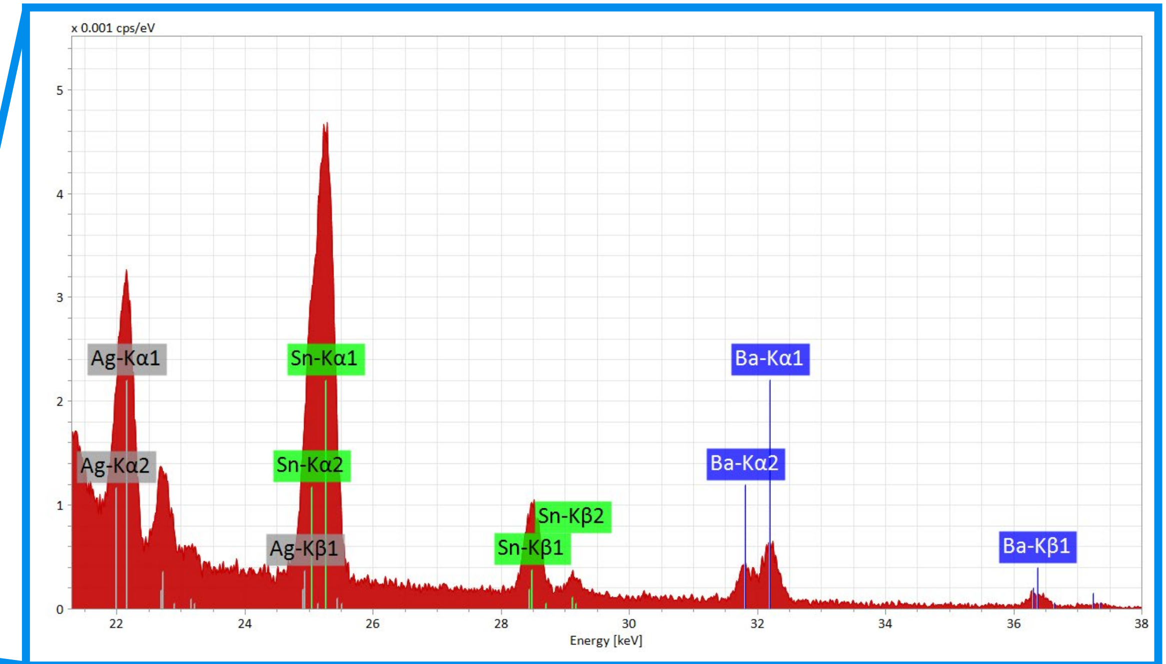
Accurate elemental analysis with high energy elemental lines

- 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)

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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.

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Color

Options
- [x] Background
- [ ] Show line names

All

Element lines

Lines	Symbols	
☑ KA1	☑	34.720 keV
☑ KA2	☐	34.279 keV
☑ KB1	☐	39.256 keV
☑ KB2	☐	40.220 keV
☑ KB3	☐	39.169 keV
☑ KB4	☐	40.334 keV
☑ KB5	☐	39.541 keV

All None

Lines	Symbols	
☑ LA1	☑	4.839 keV
☑ LA2	☐	4.821 keV
☑ LB1	☐	5.262 keV
☑ LB2	☐	5.614 keV
☑ LB3	☐	5.361 keV
☑ LB4	☐	5.274 keV
☑ LB6	☐	5.432 keV
☑ LE	☐	4.728 keV
☑ LG1	☐	6.055 keV
☑ LG2	☐	6.325 keV
☑ LG3	☐	6.341 keV
☑ LG4	☐	6.528 keV
☑ LG5	☐	5.875 keV
☑ LL	☐	4.287 keV

All None

Lines	Symbols	
☑ M2N4	☐	1.159 keV
☑ M5O3	☐	0.862 keV
☑ MA1	☑	0.884 keV
☑ MB	☐	0.902 keV
☑ MG	☐	1.078 keV
☑ MZ2	☐	0.679 keV

All None

- [ ] Automatic mode

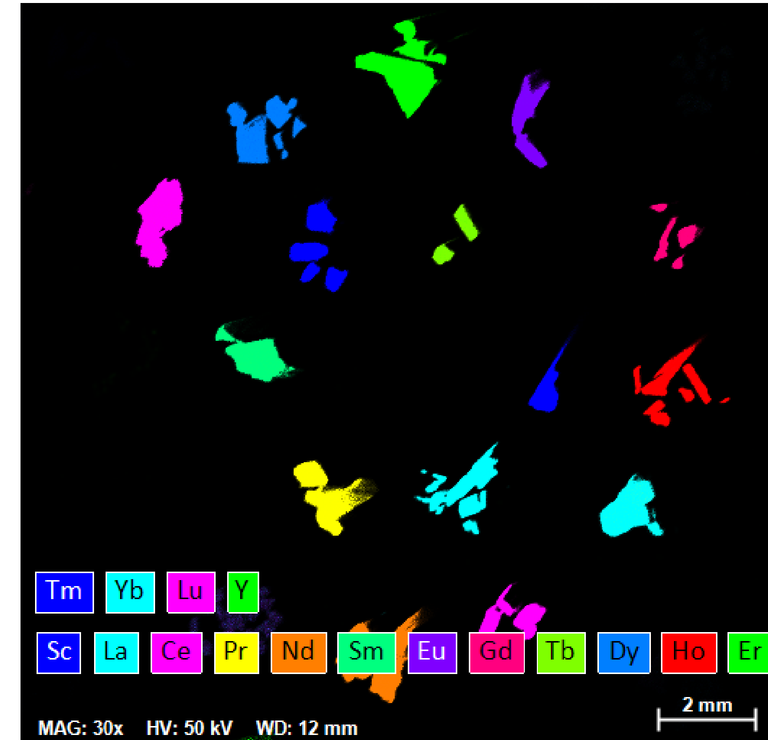
Close

Spectrum region

- (●) None
- ( ) KA 34.692 keV
- ( ) KB 39.216 keV
- ( ) LA 4.837 keV
- ( ) LB 5.274 keV
- ( ) MAB 0.901 keV

Peak area

- ( ) Wide (99%)
- (●) Medium (87%)
- ( ) Narrow (55%)
- ( ) 87 %



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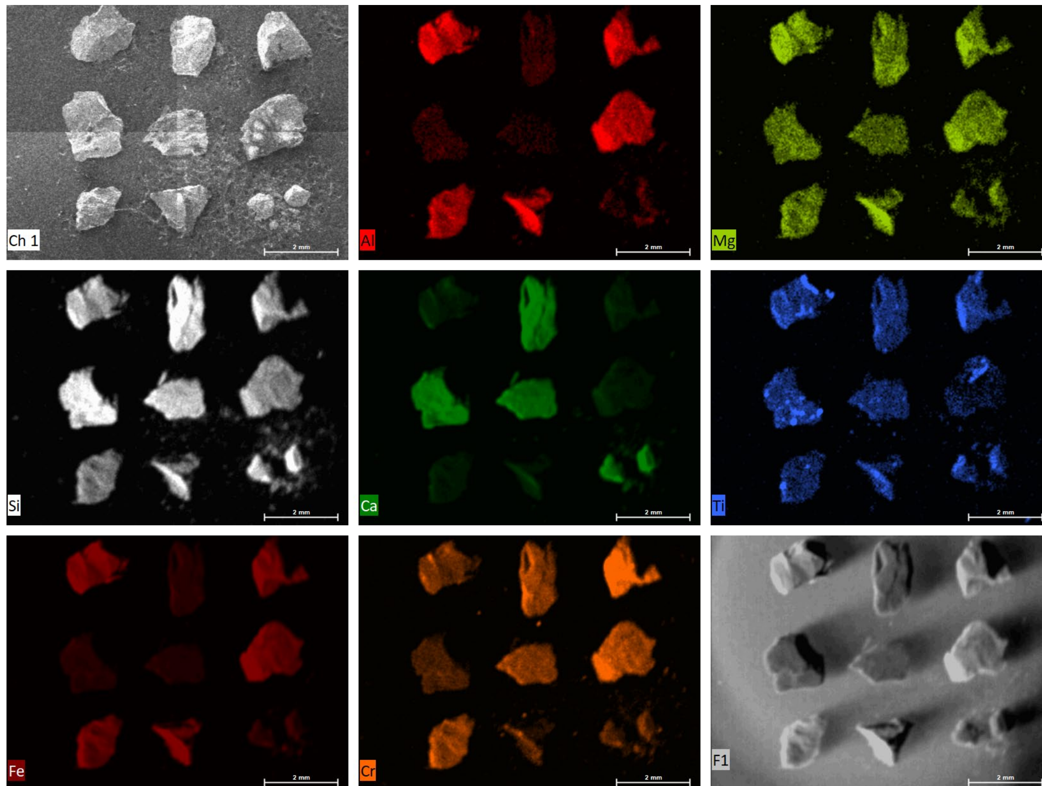
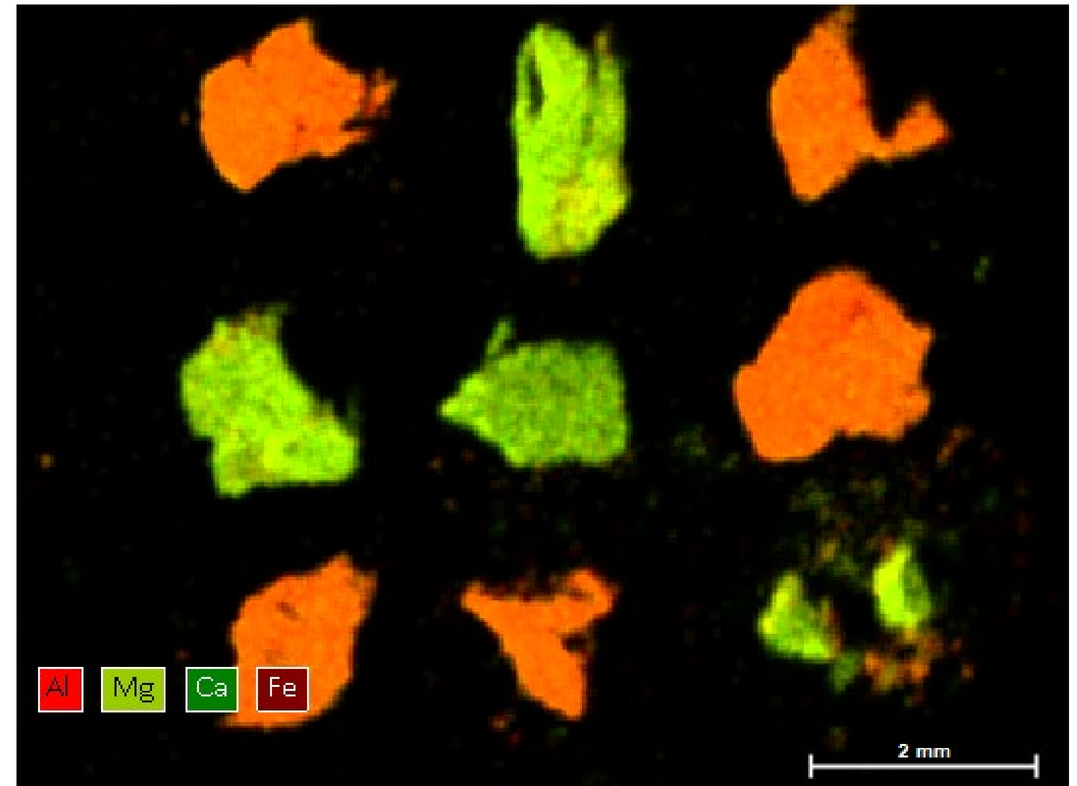
Analysis of Exploration Mineral Grains

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)





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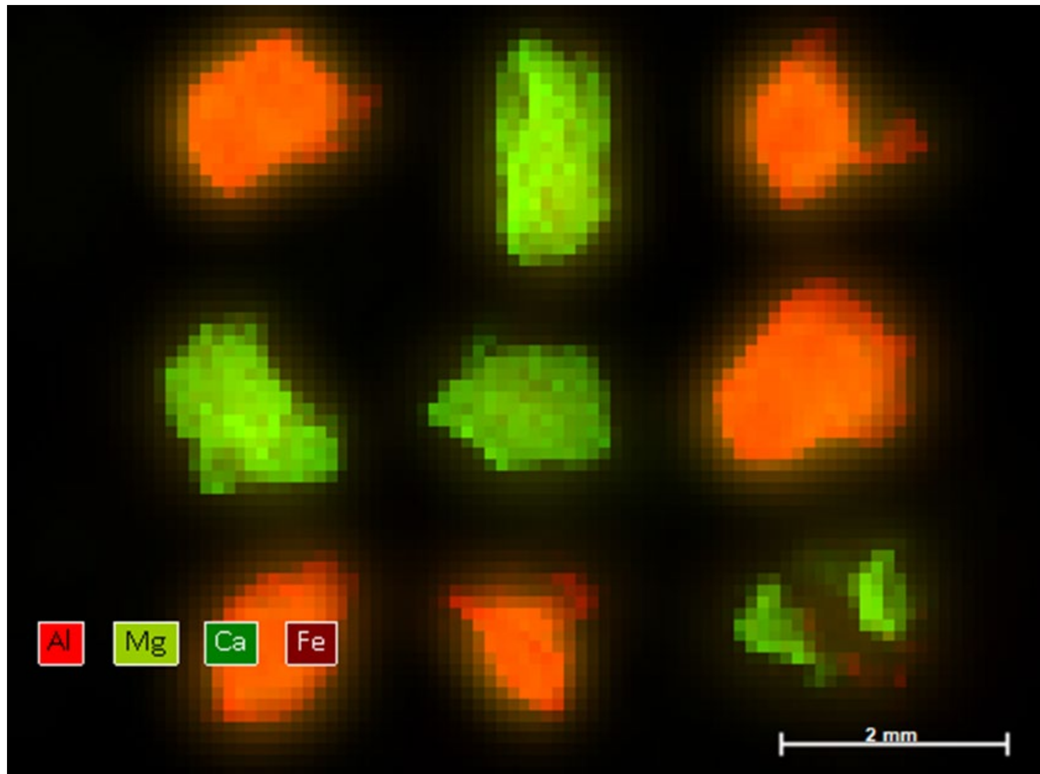
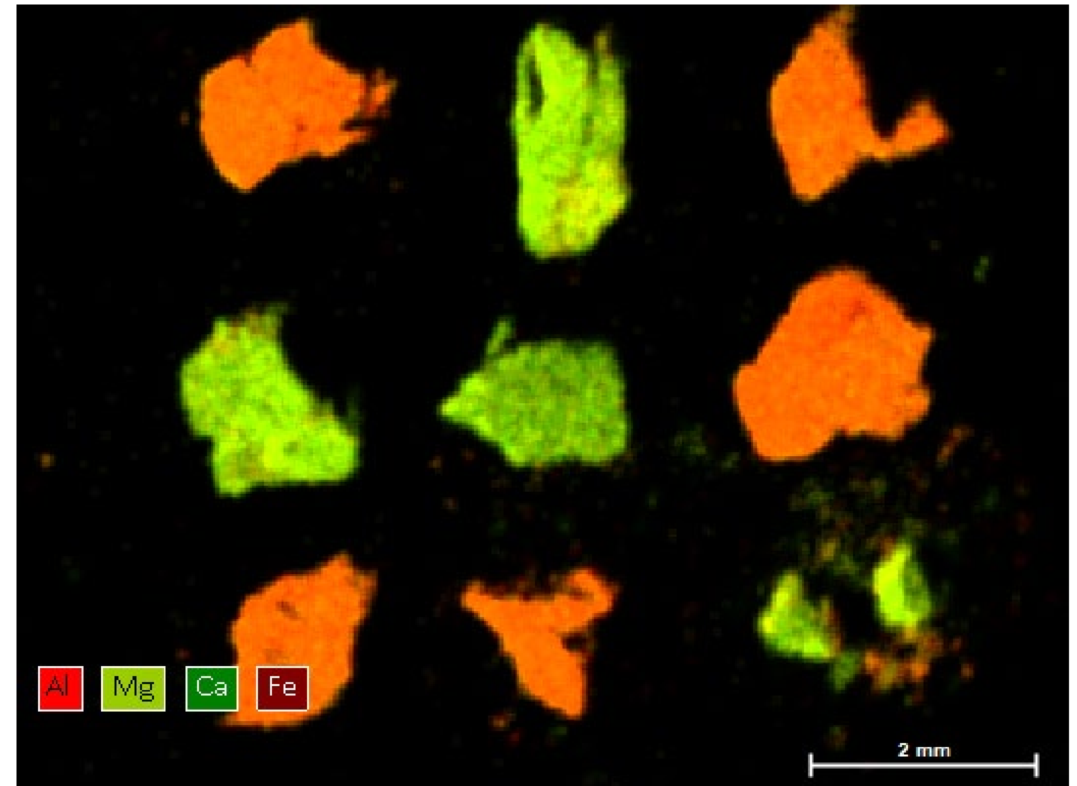
Analysis of Exploration Mineral Grains

High speed, coarse resolution analysis

Clinopyroxene (green)
and
Garnet (orange)





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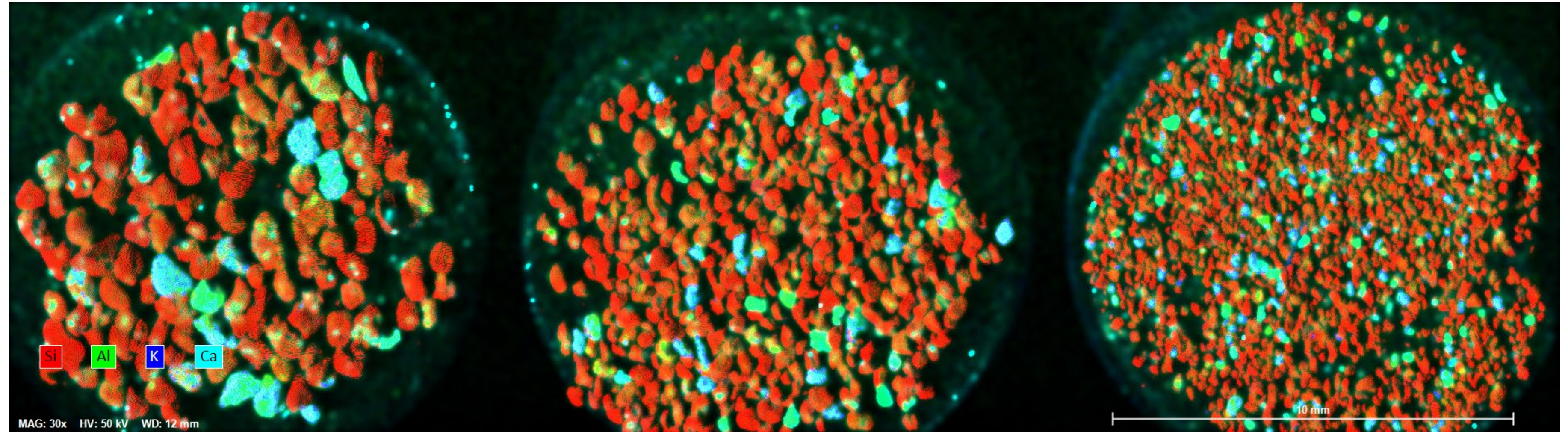
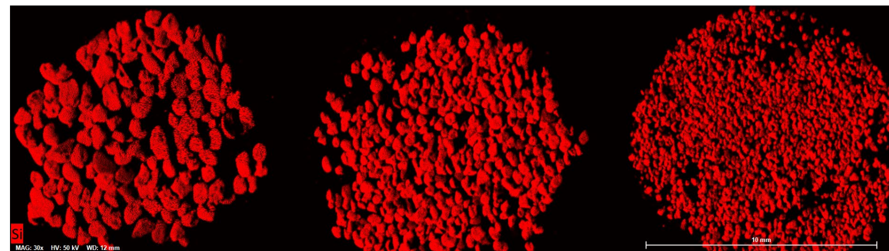
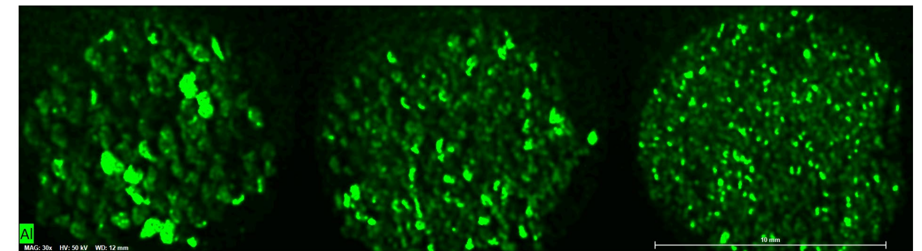
Analysis of Beach Sands

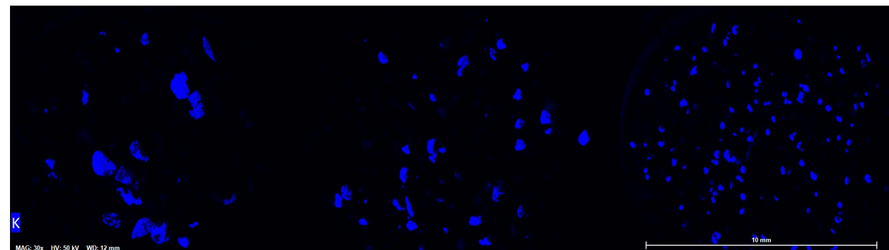
Analysis of Beach Sand

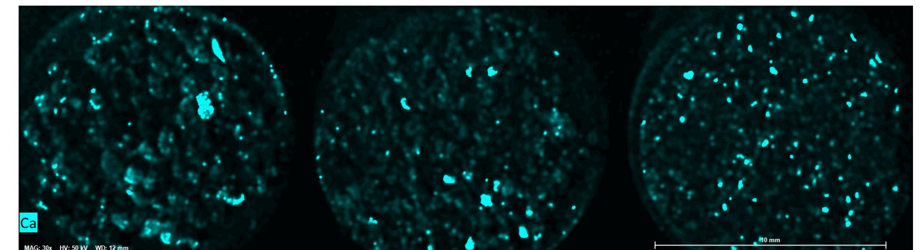
Loose Grains
Various Size Fractions
Uncoated
Large Area
High Speed Stage Movement











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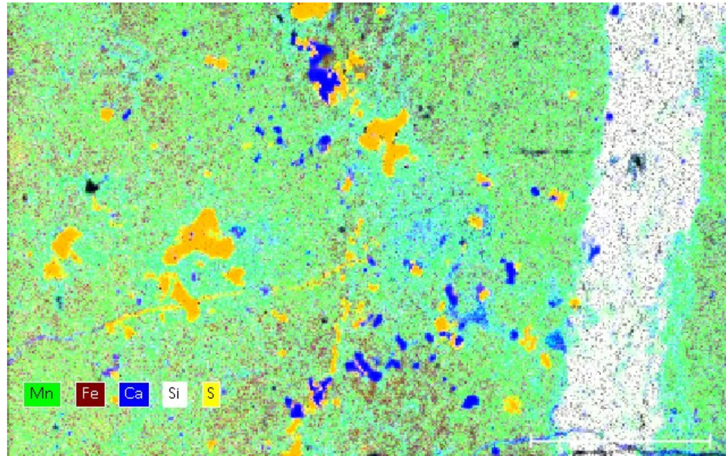
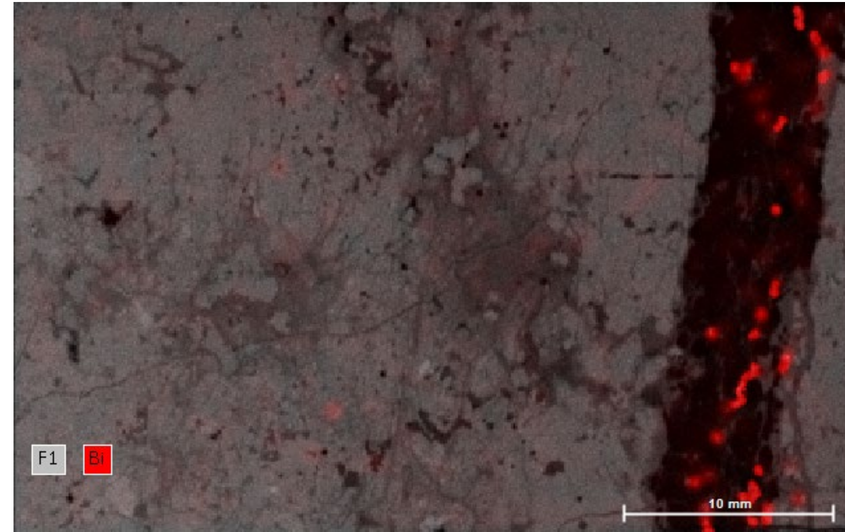
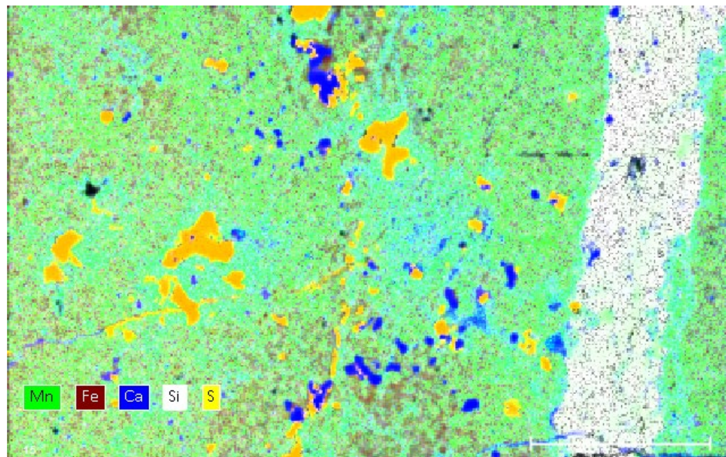
Analysis of Geological Sample

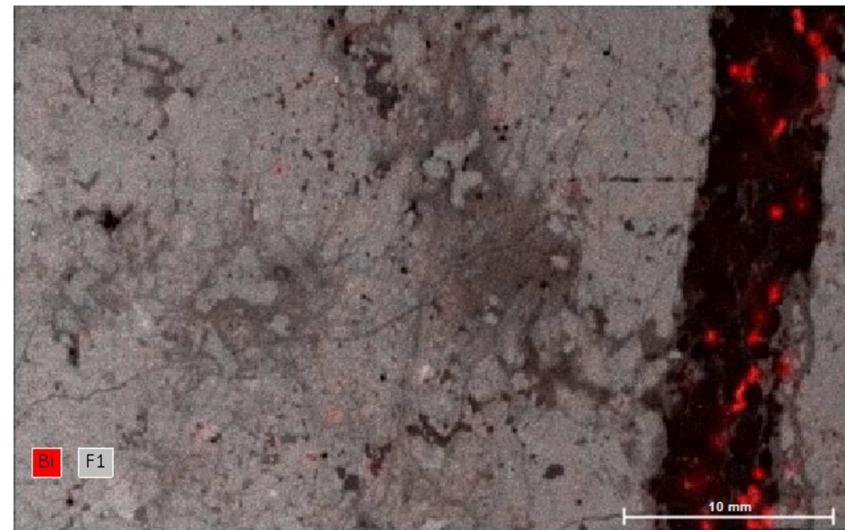
Sample:
Ilvaite skarn from Campiglia Marittima. Quartz vein with sulphides





1000 μA





600 μA

Sample courtesy of Kalin Kouzmanov, University of Geneva

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SEM-XRF:
Analysis of Vein Sample

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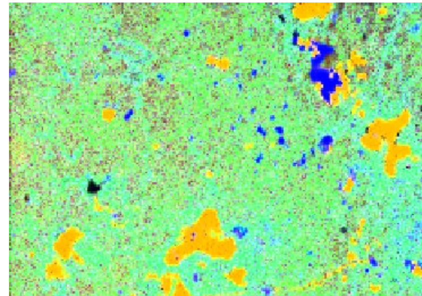
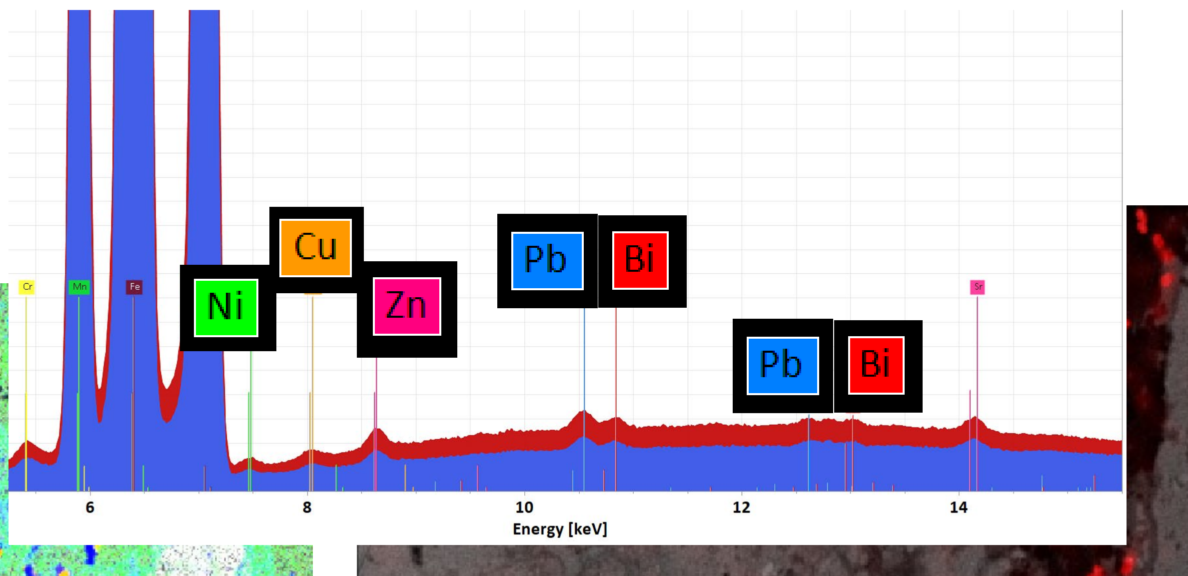
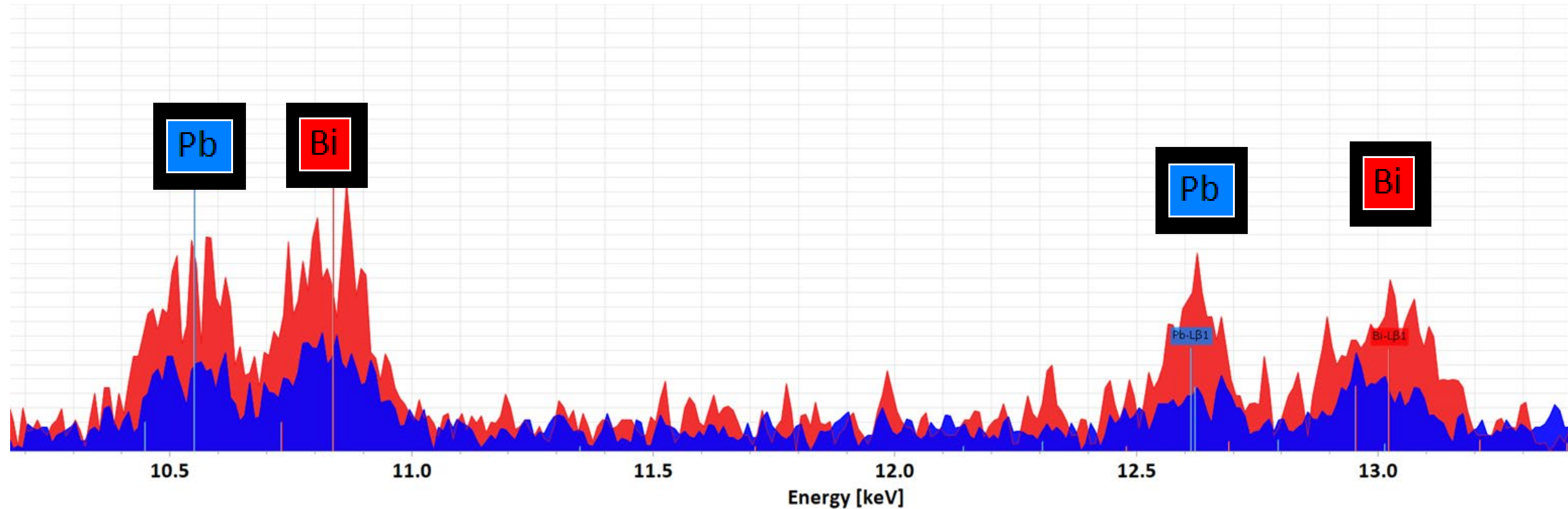
Sample:
Ilvaite skarn from Campiglia Marittima.
Quartz vein with sulphides





Ni 2010 (K)	Cu 5122 (K)	Zn 12715 (K)	Sr 12380 (K)	Zr 11381 (K)	Rh 131258 (K)	Pb 18716 (L)	Bi 11925 (L)
Ni 4014 (K)	Cu 8050 (K)	Zn 20336 (K)	Sr 16666 (K)	Zr 15289 (K)	Rh 178326 (K)	Pb 25533 (L)	Bi 17074 (L)



Sample courtesy of Kalin Kouzmanov, University of Geneva

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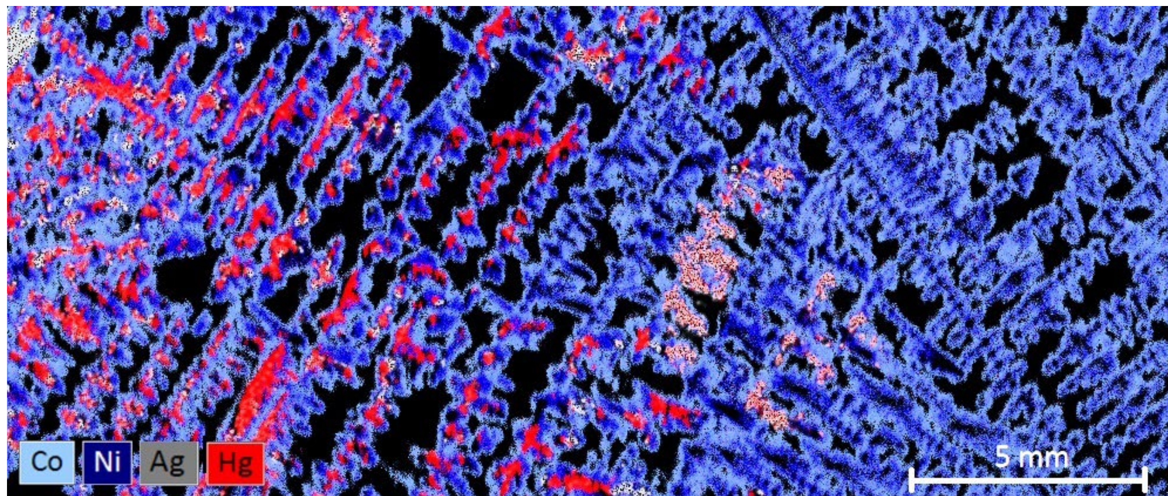
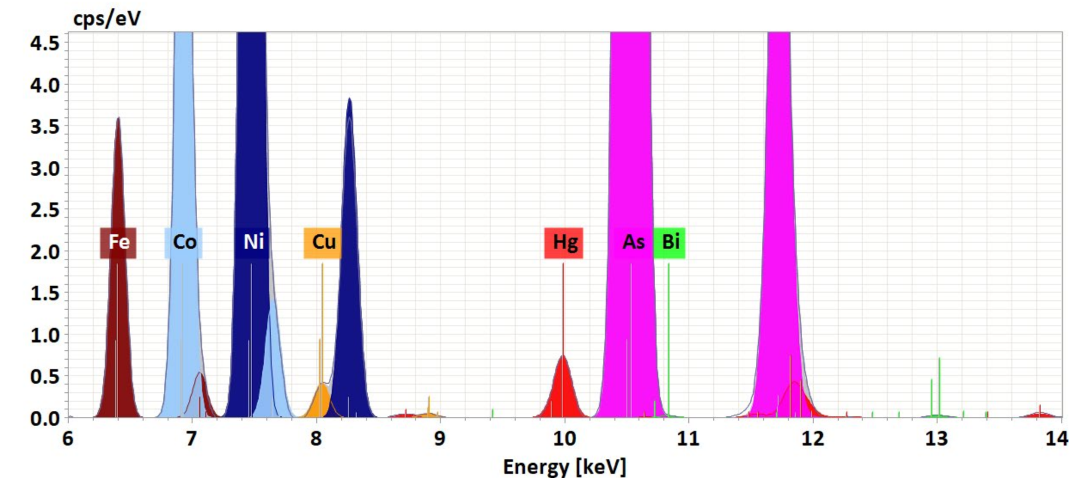
Identification and determination of the elements in ore at trace levels

- 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).





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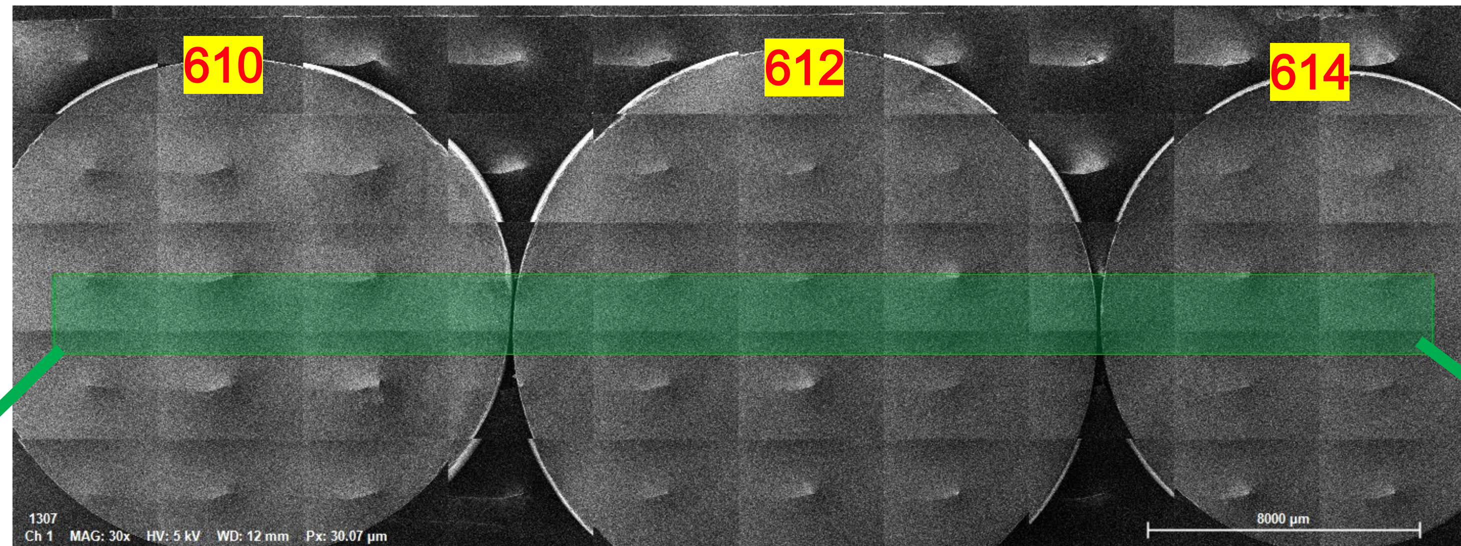
NIST Standard Glasses

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

NIST Glass Standards



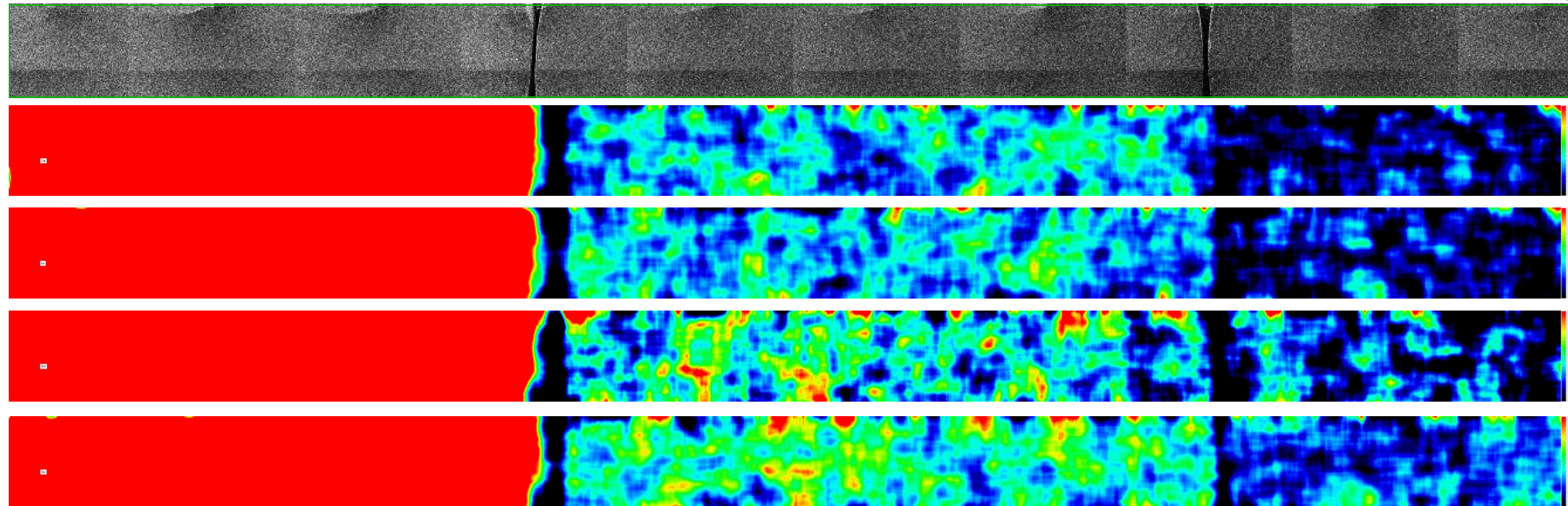
SE Image

Ce

La

Nd

Eu



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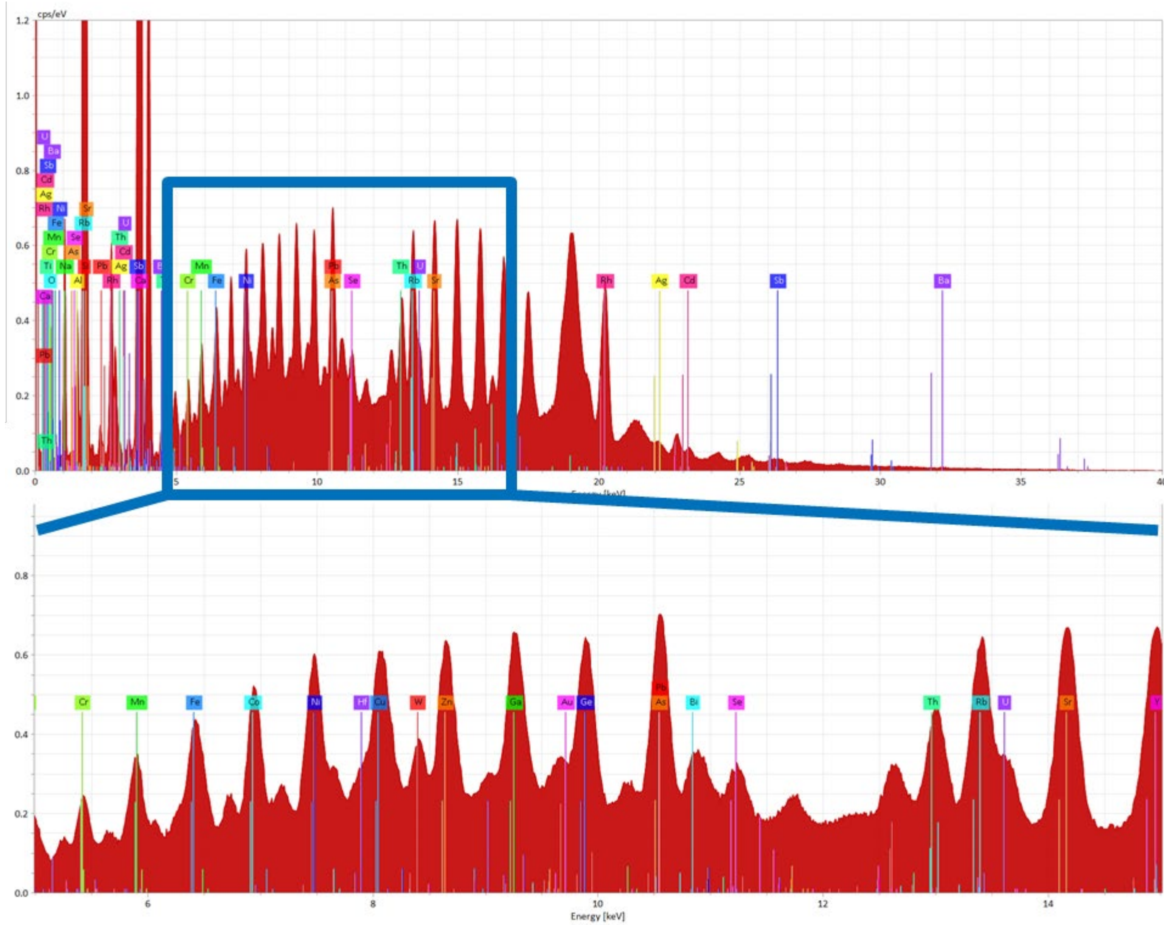
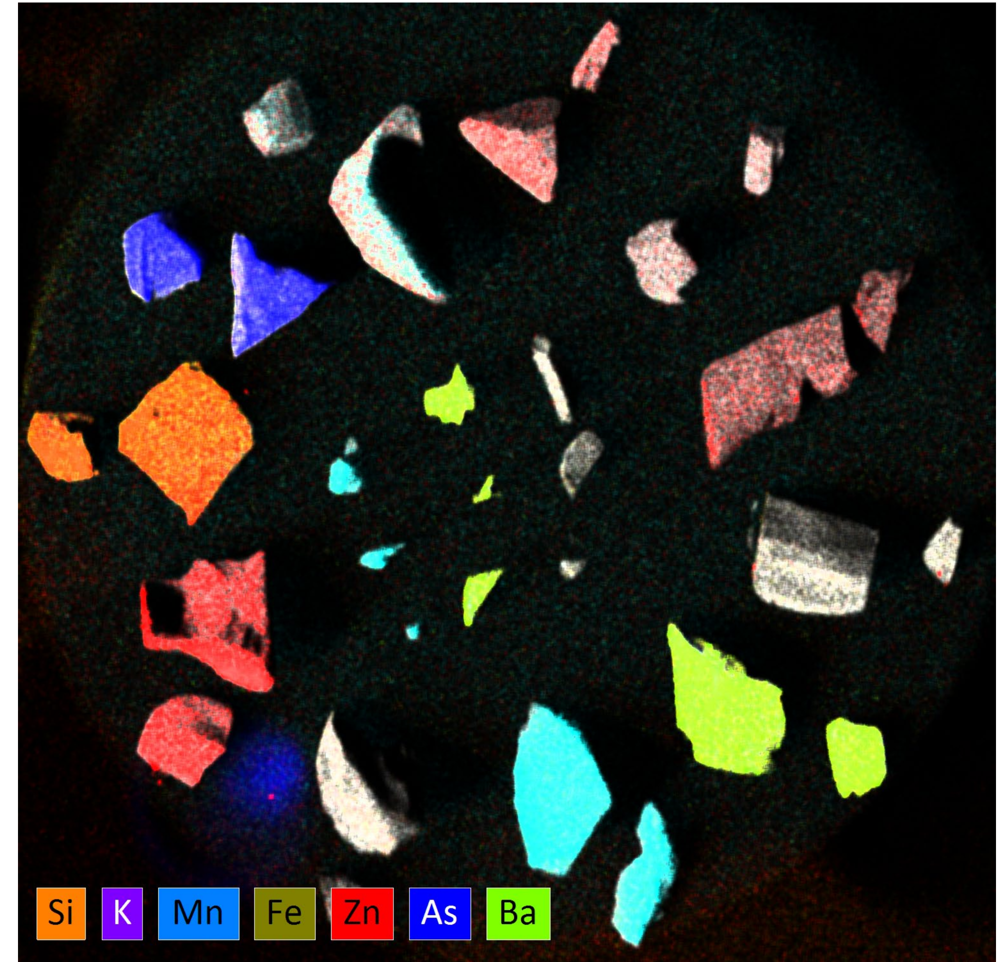
Flexispot Analysis



Glass analysis: NIST 610



Forensics: Glass fragment analysis

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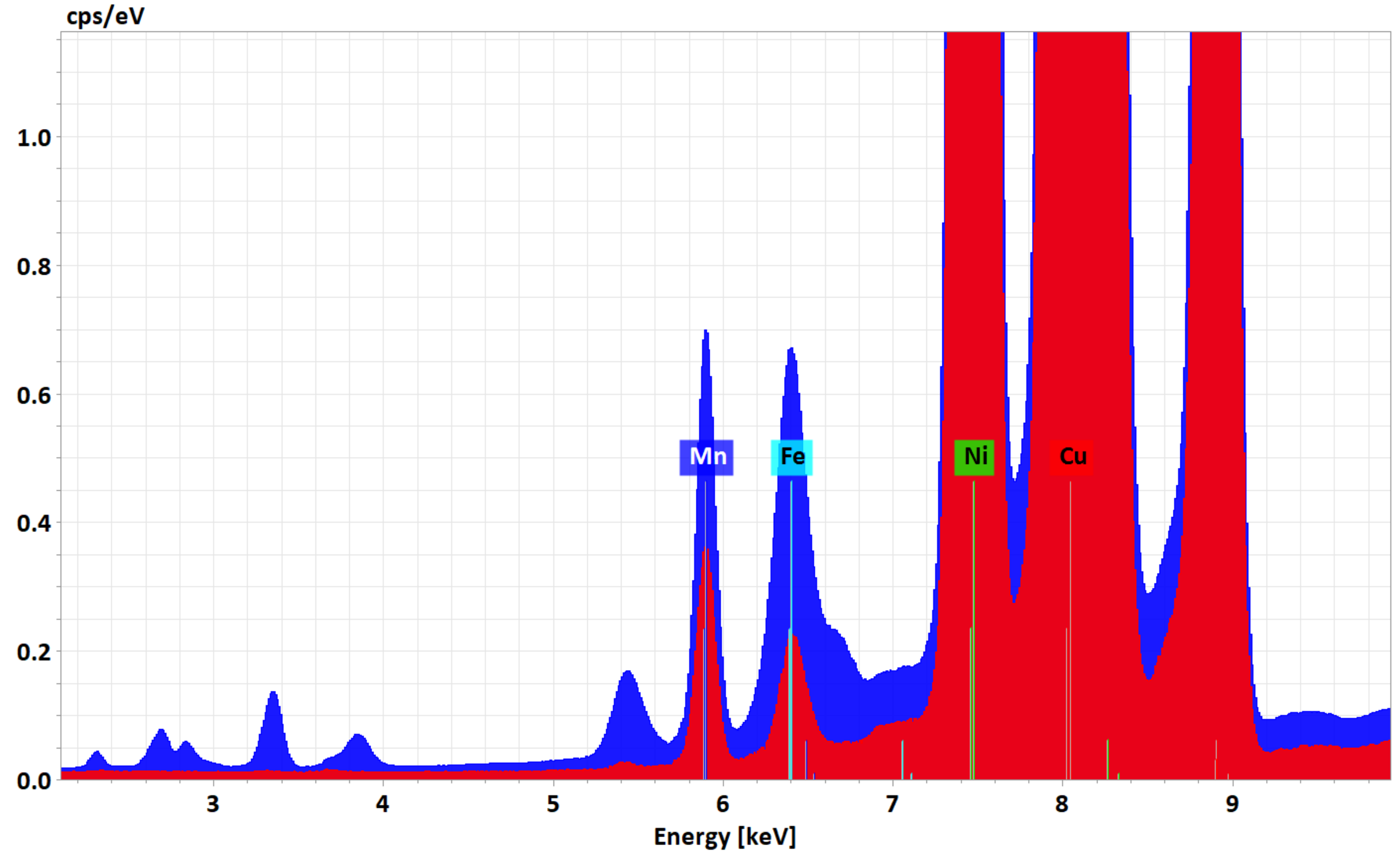
Benefits of Filters

Analysis of a coin

Blue Spectrum has no filter applied

Red Spectrum has appropriate filter for elements of interest

Presence of trace amounts of Mn and Fe confirmed and quantifiable



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XTrace 2:
Software Updates – Espirit v2.6

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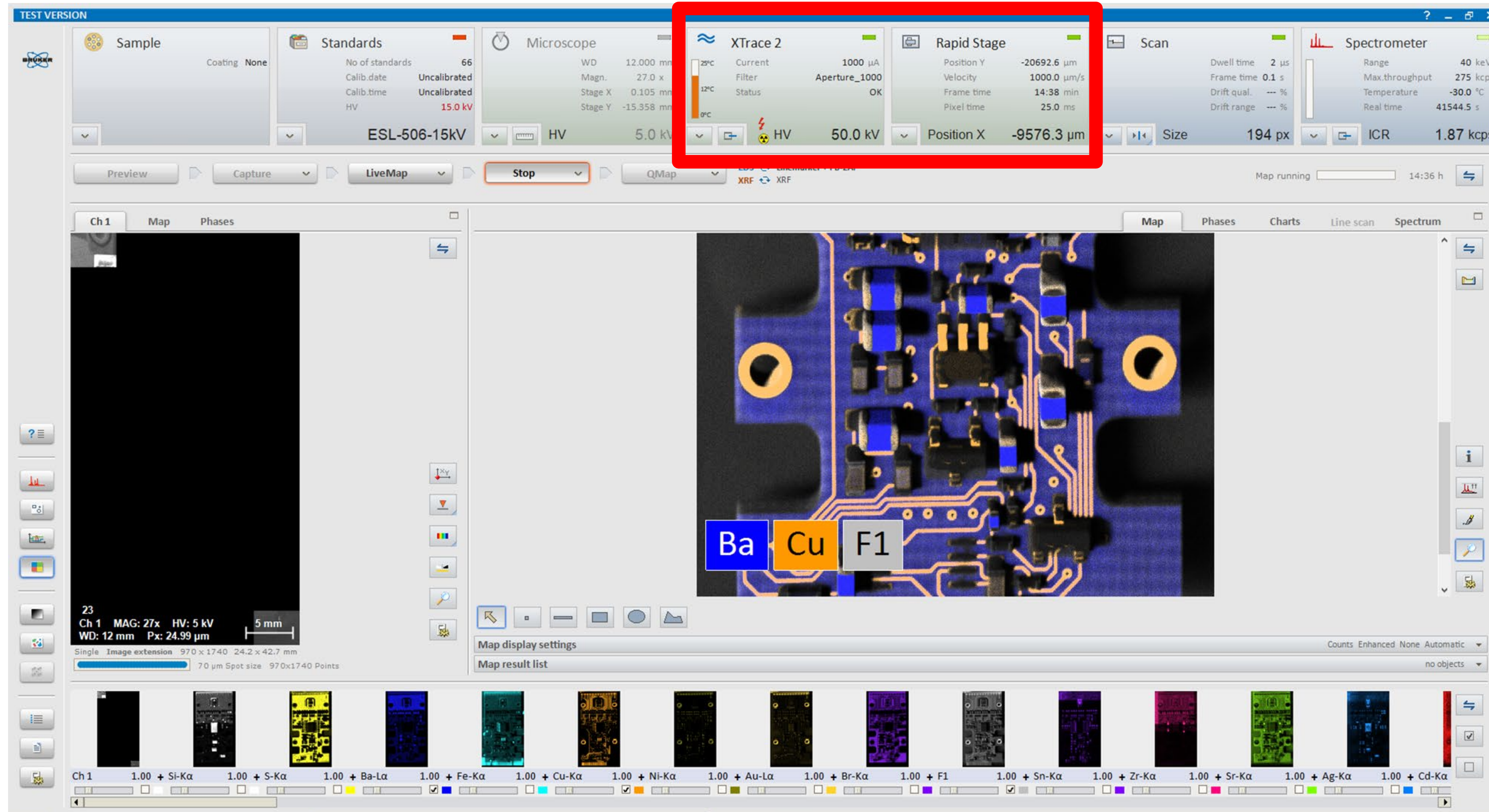
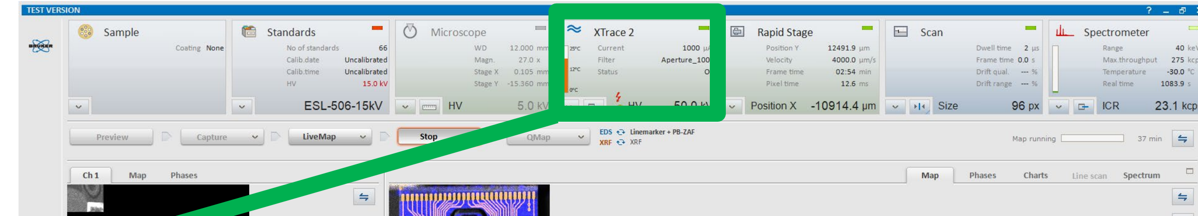
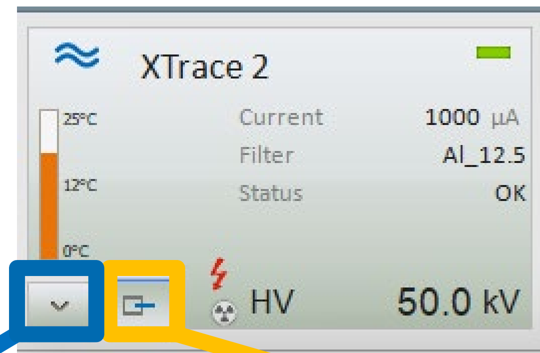
SEM-XRF and Rapid Stage
Integration in ESPRIT Software (version 2.6)

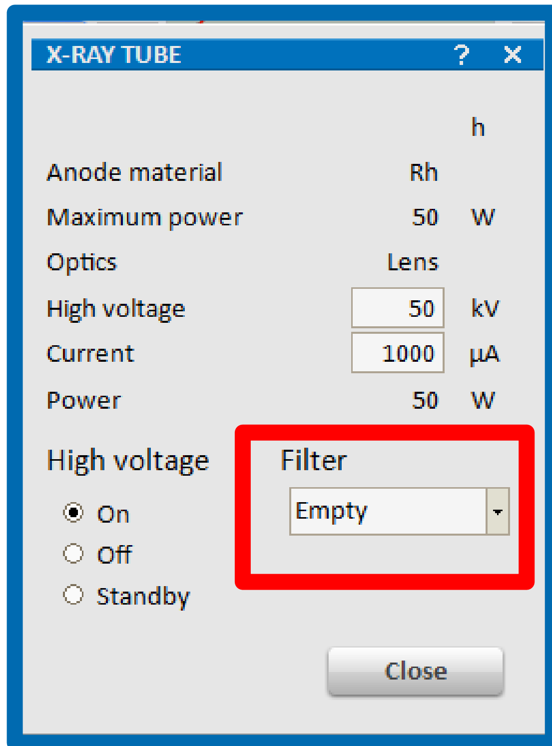


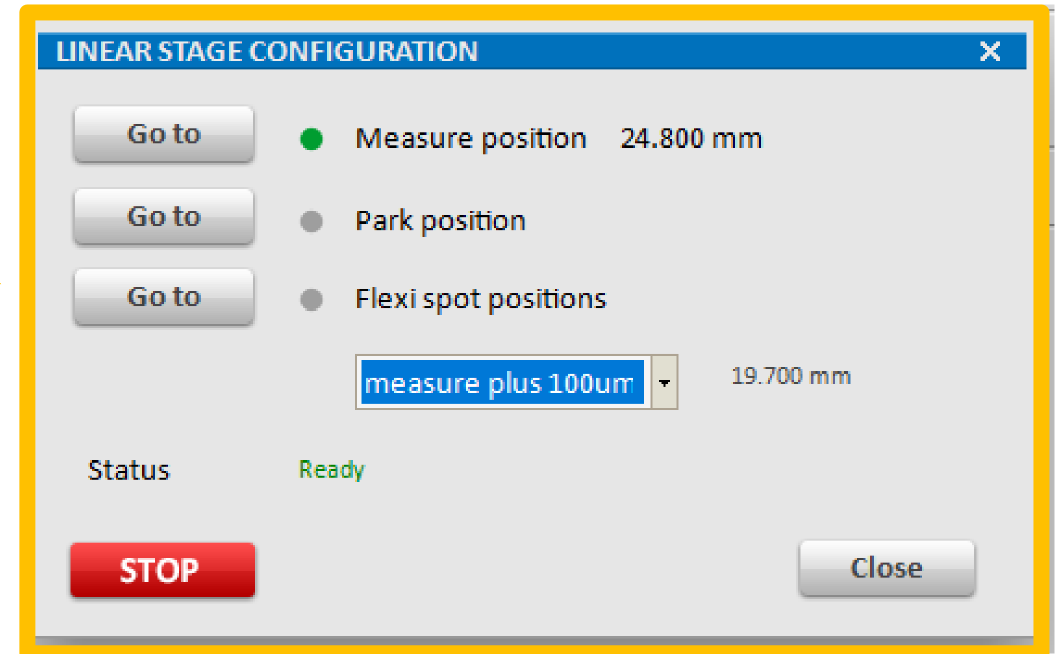
SEM-XRF and Rapid Stage

Integration in ESPRIT Software (version 2.6)









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

XTrace 2

Current	1000 µA
Filter	Al_12.5
Status	OK
HV	50.0 kV

Rapid Stage

Position Y	-1.3 µm
Velocity	4000.0 µm/s
Frame time	01:27 min
Pixel time	25.2 ms
Position X	-1.1 µm

RAPID STAGE SETTINGS

	Current values	New values
Move speed [µm/s]	4000	4000
Dwell time [ms]	25.2	25
Frame time	01:27min	01:27min
Lubrication drive enabled	☑	
Travel distance X	80.7m	Reset distance
Travel distance Y	2.6m	

Reference | Apply

→

XTrace 2

Current	1000 µA
Filter	Al_12.5
Status	OK
HV	50.0 kV

Rapid Stage

Position Y	-1.3 µm
Velocity	2000.0 µm/s
Frame time	02:10 min
Pixel time	50.5 ms
Position X	-1.1 µm

RAPID STAGE SETTINGS

	Current values	New values
Move speed [µm/s]	2000	2000
Dwell time [ms]	50.5	50
Frame time	02:10min	02:10min
Lubrication drive enabled	☑	
Travel distance X	80.7m	Reset distance
Travel distance Y	2.6m	

Reference | Apply

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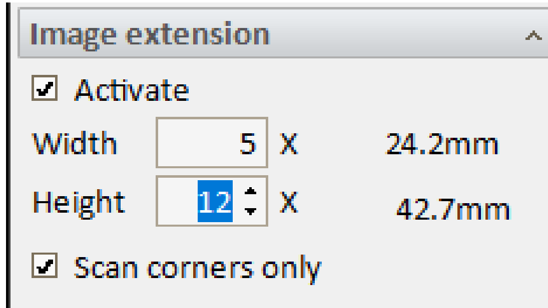
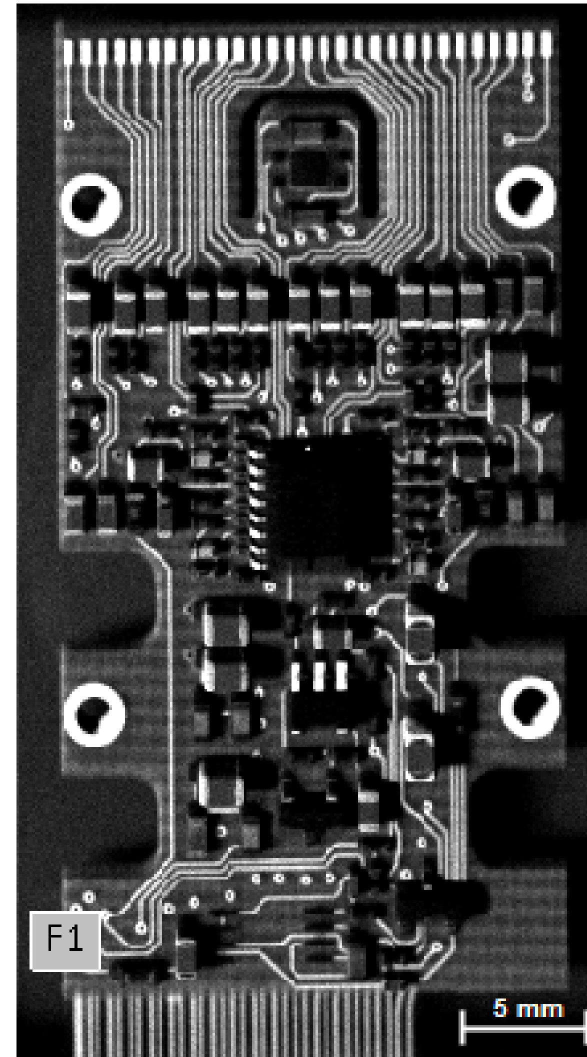
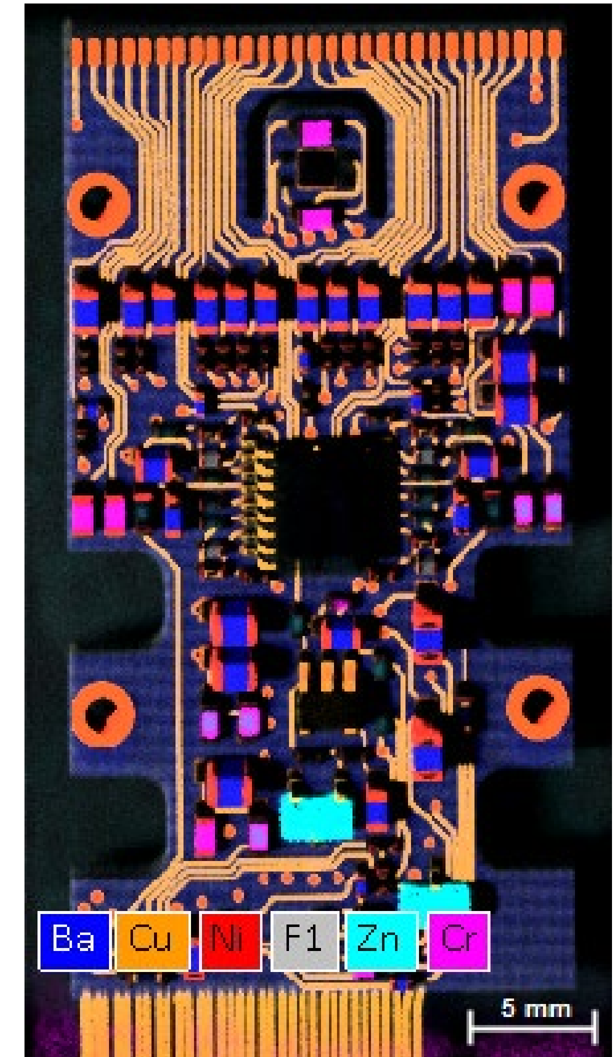
SEM-XRF and Rapid Stage

Integration in ESPRIT Software (version 2.6)







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SEM-XRF (XTrace): Summary and Conclusions

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

X-ray beam (Micro-XRF):
Fast over large area
Sensitive for traces

E-beam (SEM-EDS):
Spatial resolution

E-beam (SEM-WDS):
Spectral resolution

Correlation:
Compile, process and interpret Hypermaps

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

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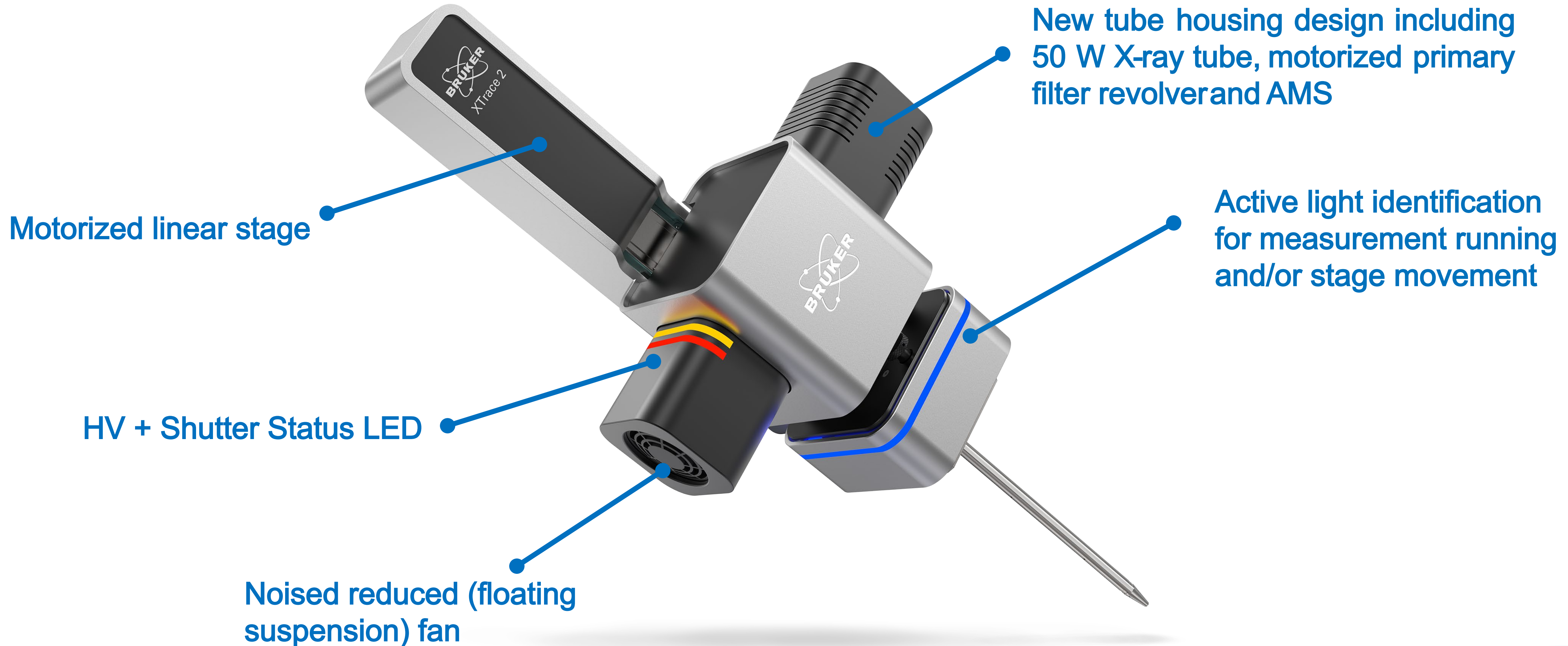
XTrace 2 – Next Generation X-ray Source for Micro-XRF on SEM



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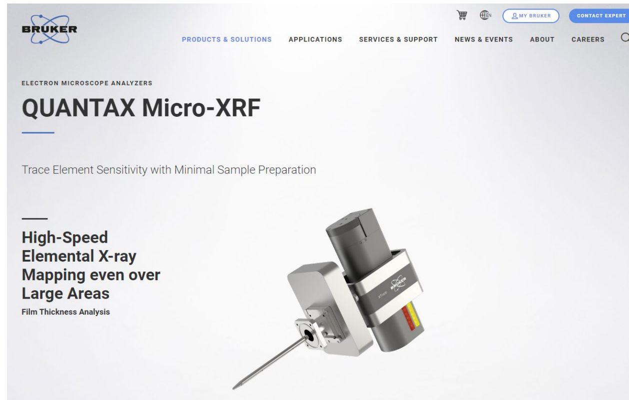
Micro-XRF General Information
Further Information

Web Site

https://www.bruker.com/

Search for:
QUANTAX Micro-XRF

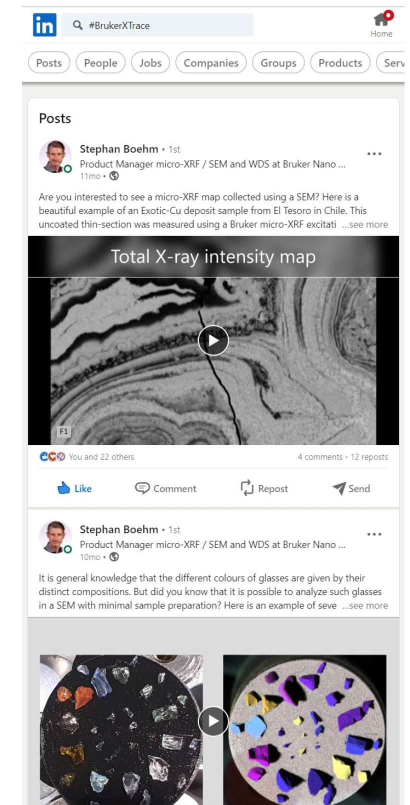


Upcoming Webinars:

Ultra-high spatial resolution EDS mapping of semiconducting materials with FEGSEM
Date: 29.06.2023 (10 am, 5 pm)

Geochemical Quantification of Geological Samples: Part I – Non-homogeneous samples
Date: 05.09.2023 (10 am, 5 pm)

LinkedIn Posts: #BrukerXTrace



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Thank you for your time.

Any Questions?

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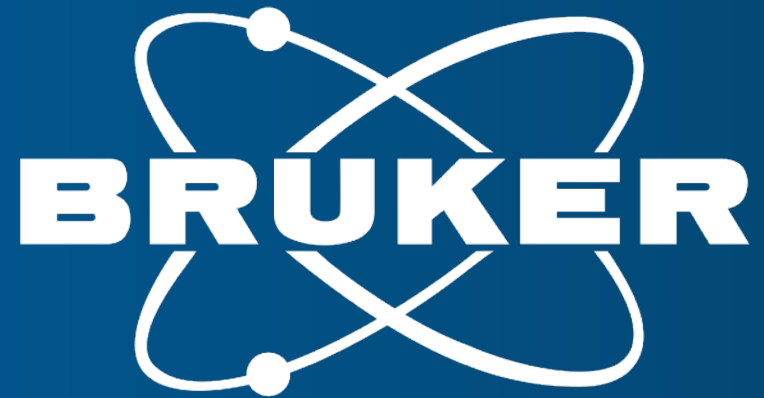

Innovation with Integrity