MICRO-XRF ON SEM

QUANTAX Micro-XRF

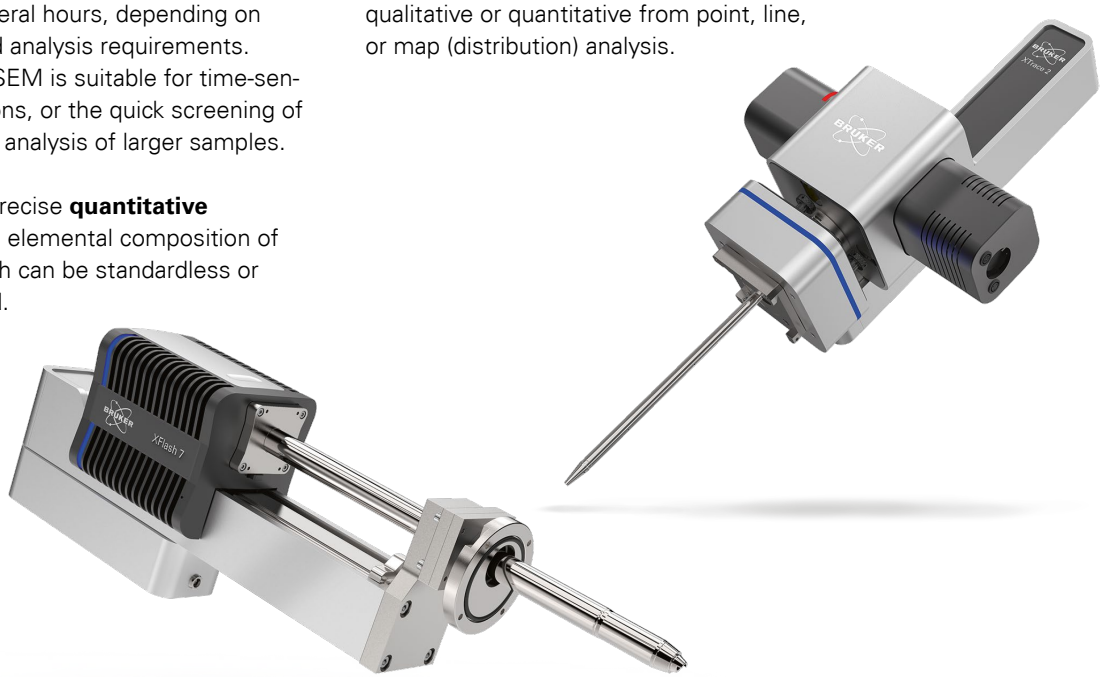
Trace element sensitivity with minimal sample preparation

Innovation with Integrity

Micro-X-ray Fluorescence Analysis

Micro-XRF on SEM, also known as SEM-XRF, empowers the scanning electron microscope with a range of new analytical capabilities, using the same EDS detector already installed on the SEM. Micro-XRF offers several novel analytical possibilities for elemental analysis in a SEM, including:

- **Non-destructive analysis** of valuable, delicate or irreplaceable samples. In addition, the photon X-ray source does not heat the sample, nor generate any electrical charging that may impact the analytical quality.
- **High spatial resolution analysis** at the micrometer scale, allowing for the detailed analysis of small or large areas or features within a sample, even with complex or heterogeneous elemental distributions, thanks to the precise spatial localization of elements of interest.
- Detection of a **wide range of elements** from carbon (Z = 6) to uranium (Z = 92), thus making it applicable to a wide variety of samples and applications.
- **Minimal sample preparation** required, i.e., no coating or polishing, thus saving time and effort. This allows for rapid analysis, making it efficient for high-throughput or routine sample analysis.
- **Fast analysis times**, typically from a few minutes to several hours, depending on the sample and analysis requirements. Micro-XRF on SEM is suitable for time-sensitive applications, or the quick screening of samples or the analysis of larger samples.
- Accurate and precise **quantitative analysis** of the elemental composition of a sample, which can be standardless or standard-based.
- **Trace element sensitivity** down to concentrations of 10 ppm or less (matrix and element dependent).
- **Large area elemental distribution maps** over the centimeter scale whilst measuring at the micrometer level is possible in combination with the optional Rapid Stage.
- Detection of **high energy X-ray lines up to 40 keV** with the 50 kV excitation source, allowing for the evaluation of elemental X-ray lines not normally considered when using a SEM.
- Determination of the **thickness of multi-layered structures** is possible due to the greater information depth of X-ray excitation.
- Micro-XRF on SEM is a **versatile analytical technique** that can be used in a wide variety of application fields, such as geology, archaeology, environmental science, materials science, forensics, pharmaceuticals, and more. The data collected can be qualitative or quantitative from point, line, or map (distribution) analysis.



Full Range EDS Elemental Analysis – Expand Your SEM Capabilities

Micro-XRF on SEM converts the standard SEM to a dual-beam source system (e-beam + photon beam) that can be operated either independently or simultaneously to benefit from the advantages of both excitation methods.

Using photon excitation on a specimen results in a similar interaction as that seen via electron excitation, causing the specimen to emit characteristic X-ray fluorescence radiation. This X-ray information is captured by a standard energy dispersive spectrometer (EDS) and can be utilized and processed as normal. Qualitative and quantitative data on a sample's elemental composition, including major, minor and trace elements, can be collected with high sensitivity and spatial resolution at the micrometer scale.

Micro-XRF analysis does not require any sample preparation, specifically that may alter the sample or even destroys it, making it suitable for the analysis of valuable or delicate samples in various applications.

Photons (X-rays) generated by a microfocus X-ray tube are focused via a polycapillary X-ray optic onto the sample surface. The optic spot size can range from 10 µm to 35 µm. Upon installation, this focused X-ray beam is aligned with the electron beam at a nominal SEM working distance (WD) maximized for EDS analysis. This allows the user to seamlessly switch between the two excitation sources to take advantage of both, or to use them simultaneously to qualitatively examine trace and light elements at the same time.

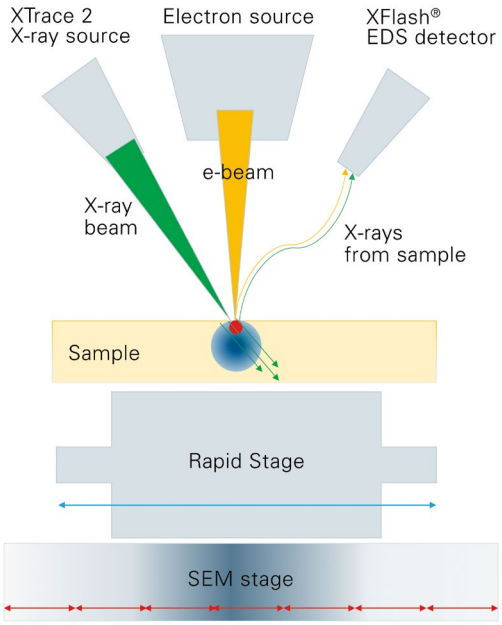


Figure 1
Schematic of a dual photon beam and electron beam source setup with a single EDS detector. The optional Rapid Stage sits on top of the normal SEM stage to enhance data acquisition whilst mapping.

The SEM, as the primary imaging instrument, provides high-resolution images of the sample surface. In addition to EDS analysis, the fully integrated XTrace 2 micro X-ray source enables X-ray fluorescence analysis of the same sample without sample transfer or additional sample preparation.

Utilizing a micro-XRF on a SEM will maximize the full range of an EDS detector's analytical possibilities, including spectral information up to 40 keV as well as major, minor and trace element concentrations and their distribution within the sample.

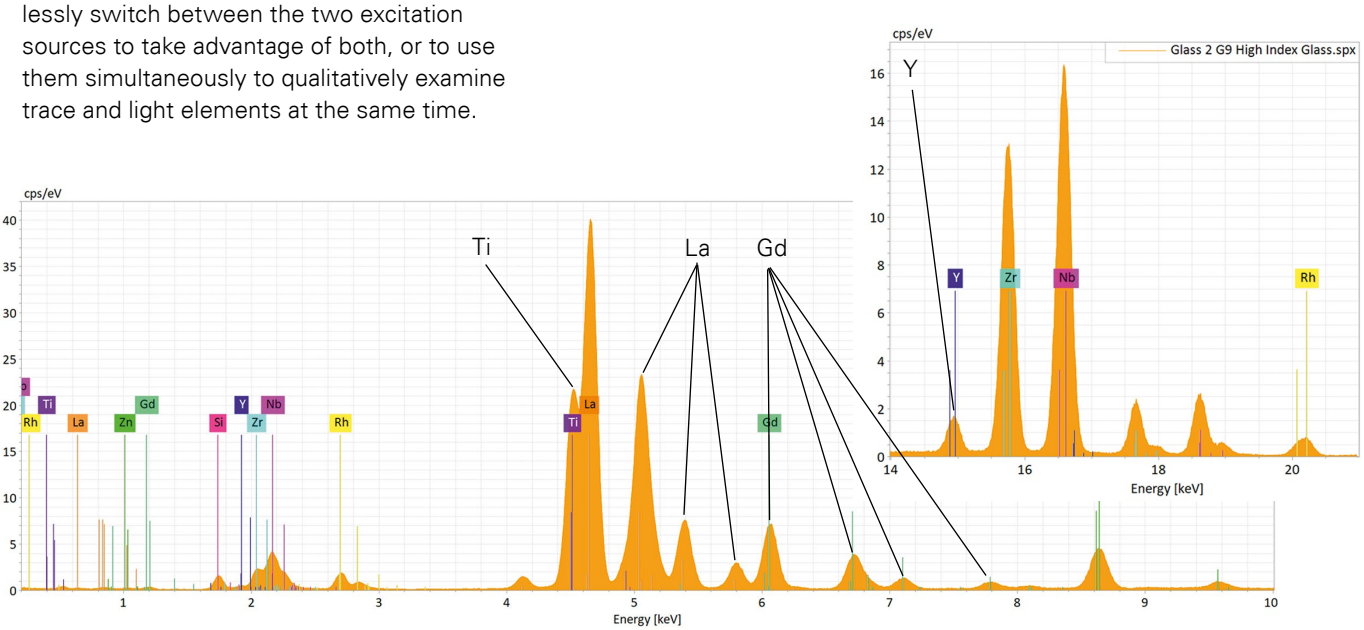


Figure 2
Analysis of Rare Earth Elements in a high index glass. Major elements such as La, Ti, Nb, Gd, Zr, Si, and Zn as well as the minor element Y were detected in the energy range from 0 to 20 keV.

XTrace 2 - Innovative X-ray source for micro-XRF analysis on SEM

XTrace 2 is the second-generation X-ray source for the QUANTAX micro-XRF system. It enables fast micro-XRF spectral acquisition with high-resolution data. Advanced features, such as the FlexiSpot mode, the Aperture Management System, and the motorized wheel for the selection of various filters facilitate the collection of robust data from even the most challenging samples.

XTrace 2 is designed for use with the Bruker XFlash® EDS detector series for optimum qualitative and quantitative data analysis and hence requires no additional X-ray detector. It can be adapted to a variety of inclined SEM ports.
The optional Rapid Stage, which can be temporarily mounted on the SEM stage, enables fast elemental X-ray mapping over large areas. This analytical setup allows the investigation of very large specimens up to 50 mm on a micrometer scale even in a SEM.

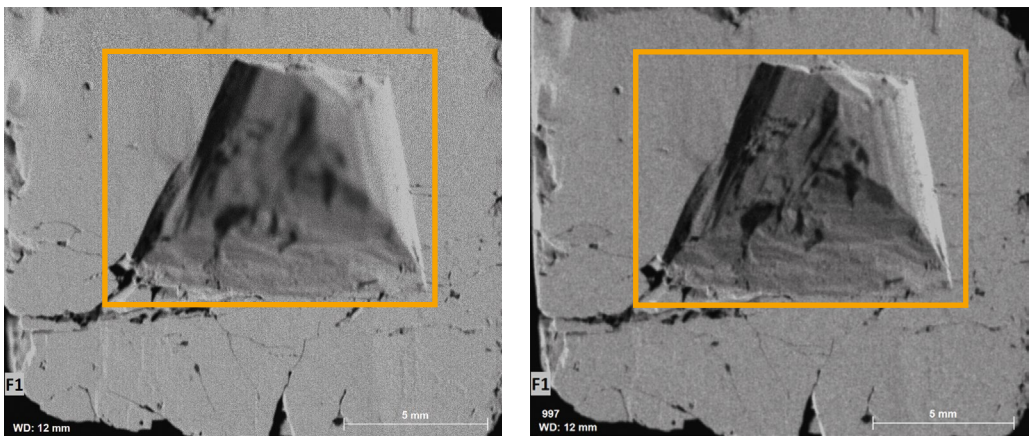
Full integration into the ESPRIT software makes QUANTAX micro-XRF an intuitive system for comprehensive SEM EDS and micro-XRF analysis.

Increased performance and usability

- High tube power generates X-rays at high energies of 50 kV and beam currents of 1000 µA for a high-count rate allowing accurate elemental data to be collected quickly and efficiently.
- Automatically switch between six primary filters for enhanced background reduction and precise acquisition of 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 focal depths.
- Analyze inhomogeneous and/or irregular shaped samples using the FlexiSpot mode, allowing spectral measurements using a small or a large spot size.
- Extended X-ray tube lifetime with the automatic tube warm-up procedure.
- Optimized safety and user functionality with the motorized linear stage incorporating automatic source retraction, including measure, park, and user specified positions
- Software capability for saving and correlating photon beam and e-beam measurements (control the analysis, select filters, and move the linear stage using the intuitive ESPRIT software).

Aperture Management System (AMS)

XTrace 2 is equipped with a patented Aperture Management System (AMS) to retain measurement resolution over a certain depth range, allowing the examination and visualization of topographical samples.



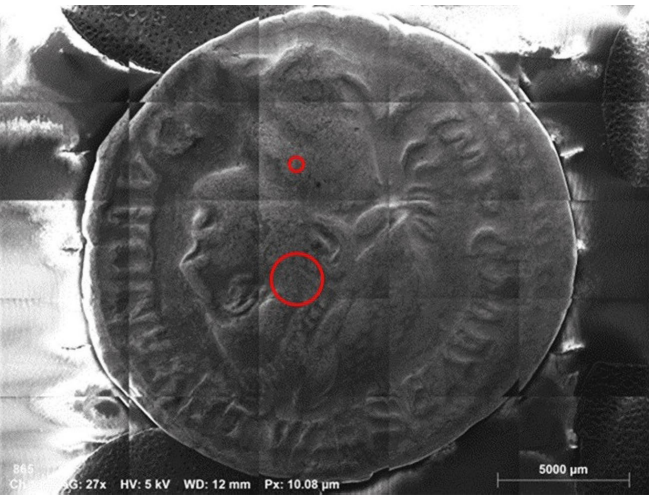
AMS
The AMS feature allows scanning of samples with topography in the SEM by increasing the focal depth. The surface of this pyrite crystal shows a total intensity X-ray map collected without AMS (left) and with AMS (500 µm, right). Mapping with AMS can even resolve the sample structures at a depth of 2.1 mm.

Any analysis that deviates from the micro-XRF focal plane (nominal working distance) will result in a larger spot size. The AMS keeps the optic in focus at varying working distances by increasing the depth of field. This means that any reduction in resolution due to deviations in the working distance is minimized, facilitating the high-resolution elemental mapping of topographical samples and their 3D features.

FlexiSpot mode

FlexiSpot allows measurements at different spot sizes, ranging from 35 µm (standard optic spot size) to 500 µm and even beyond. Large spot sizes are used for overall analysis, while small spot sizes are used for the analysis of specific features. Measuring at larger spot sizes allows more accurate quantification of inhomogeneous and irregular shaped samples as well as samples with uneven surfaces. A large spot area provides a more representative excitation of the whole sample composition in just one measurement.

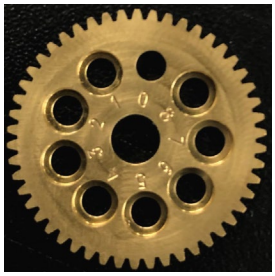
FlexiSpot works by retracting the X-ray source, allowing the X-ray optic to be defocused out of the nominal focal plane. Each individual spot size is selected in the ESPRIT software via an automated process. Multiple individual spot sizes can also be defined.



FlexiSpot
The ability to change the spot size from the X-ray optic spot (10 µm or 35 µm) and larger spot sizes is important for selecting the most appropriate analytical volume for sample representativity.

Motorized wheel with six primary filters and two AMS apertures

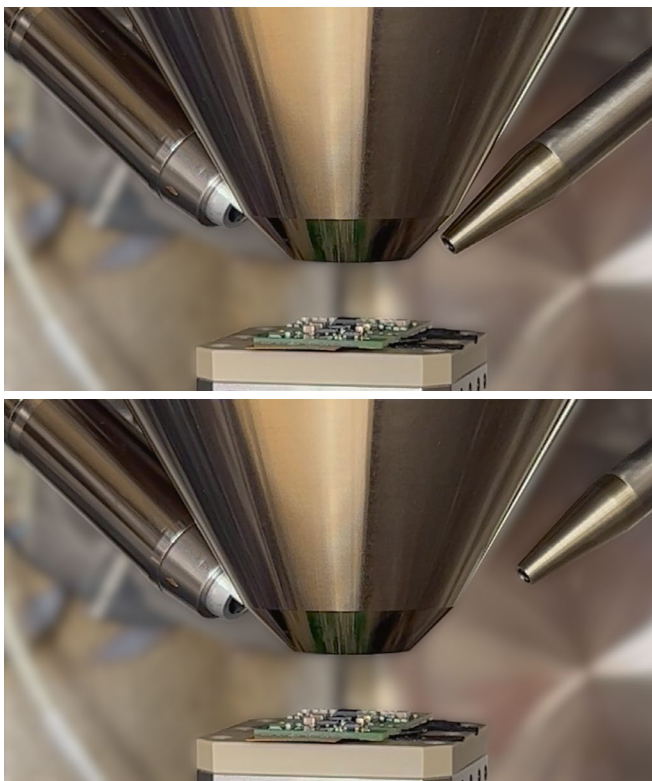
The new XTrace 2 has space for nine different options within the wheel, located between the X-ray tube and polycapillary optic, allowing further background reduction for each individual X-ray energy region.



Motorized wheel
Position 0 is left blank for normal unfiltered X-ray analysis. Positions 1 to 6 are reserved for several primary filters, whilst positions 7 and 8 are for AMS apertures. Switching between positions is automatic via software control.

SEM 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's motorized linear stage. The source can also be retracted when the SEM sample chamber is vented for sample change.
- **SEM air-lock integration.** The status information of the SEM air-lock chamber (open/closed) can be integrated into the XTrace 2's security circuit for optimal X-ray safety purposes. This feature is designed for the integration of various SEM air-lock chambers (from different SEM vendors).



Auto source insertion and retraction
Top: X-ray optic in measurement position, bottom: X-ray optic in parking position.

Applications

Micro-XRF on SEM has a wide range of applications across various fields.

Materials science

The elemental composition and distribution in a wide range of materials, including metals, alloys, polymers, ceramics, composites, coatings, and thin films can be determined using micro-XRF on SEM. It is used for quality control, failure analysis, characterization of surface treatments, and investigation of elemental segregation and diffusion in materials.

Analysis of Rare Earth Elements (REEs)

Rare earth elements have a range of X-ray energies, typically the L series are commonly used in standard EDS for identifying those REE´s which often overlap with the base metals (e.g., Cr, Mn, etc.). Micro-XRF is capable not only to detect the L series, but also the higher energy K lines (e.g., Ce at 34.7 keV) that have no peak overlaps, which makes the user confident of element identification and thus support the deconvolution process of the lower energy lines.

Smithsonian REE phosphate standards

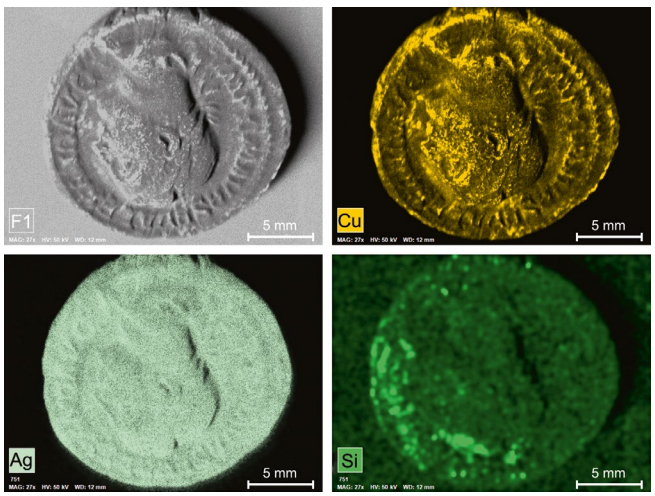
All rare earth elements can be clearly identified in a single map using SEM-XRF analysis with automated deconvolution.

Archaeology and art conservation

Cultural heritage objects, such as paintings, sculptures, ceramics and metal artifacts, can be studied using micro-XRF on SEM. It provides valuable information about the elemental composition of pigments, dyes, glazes, and corrosion products, which can help in identifying materials, studying artistic techniques, and assessing degradation processes.

Analysis of coins

Micro-XRF obtains surface information but also to detect sample information from underneath the sample surface which makes it possible to investigate also historic coins to determine their origin or to understand the aging process and avoid areas where surface ageing has occurred.



Elemental distribution analysis of coins

Analysis of a Roman coin highlighting the effects of burial and oxidation on the surface of the coin that will impact any quantification of the alloy composition.

Environmental science

The analysis of soil, sediment, water, and plant samples is possible using micro-XRF on SEM. It can determine the presence and distribution of trace elements, heavy metals, and contaminants in environmental samples, aiding in environmental monitoring, pollution assessment, and remediation studies.

Pharmaceuticals and cosmetics industries

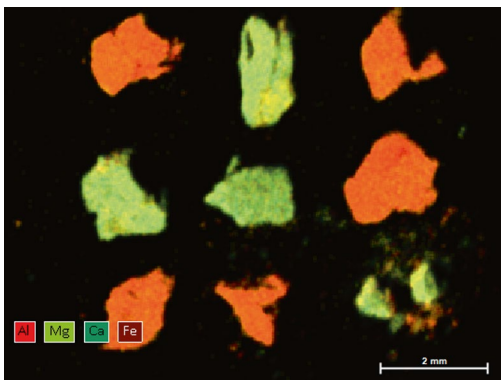
The application of micro-XRF in SEM in the pharmaceutical and cosmetics industries includes the analysis of raw materials, packaging materials and the quality assurance of finished products. It can determine the elemental composition of drugs, cosmetics, and packaging materials, aiding in quality control, compliance with regulations, and product development.

Geology and mineralogy

The elemental composition of rocks, minerals, ores, and soils can be determined and analyzed using micro-XRF on SEM. It can provide information about mineralogical composition, elemental zoning, and trace element distribution, which can be useful for mineral exploration, resource assessment, and geological research.

Analysis of exploration mineral grains

The composition of mineral grains can be easily determined with micro-XRF without any sample preparation. These grain fragments were simply mounted on a carbon stub without any carbon coating and quickly scanned to identify what these minerals are, in this case eclogitic garnet and clinopyroxenes from South Africa.



Element distribution map of various mineral grains
SEM-XRF analysis of unpolished garnet and clinopyroxene grains confirming that they are derived from a mantle eclogite, possibly associated with diamonds.

Forensics

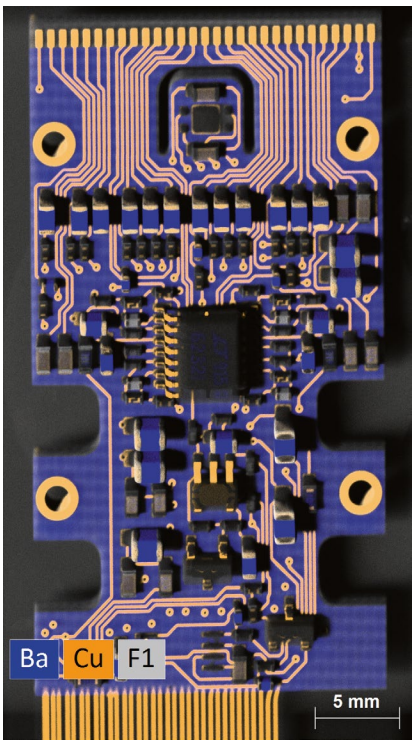
Forensic science benefits from micro-XRF on SEM over a wide range of applications including the analysis of trace evidence, gun shot residues, paint chips and fibers. It can provide information about the elemental composition of these samples, aiding in forensic investigations and criminalistics.

Electronics and semiconductor industries

Micro-XRF on SEM is a valuable tool in the electronics and semiconductor industries for the analysis of electronic components, circuit boards, and semiconductor materials. It can determine the elemental composition of solder joints, coatings, and contaminants, aiding in quality control, failure analysis, and process optimization.

Fast elemental mapping with AMS over large area of electronics components on a Printed Circuit Board (PCB)

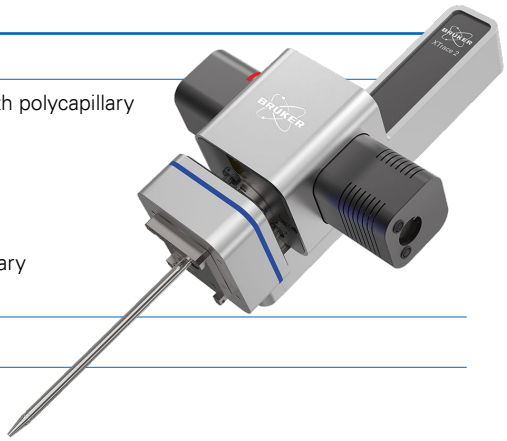
High-speed micro-XRF on SEM can be used for the elemental analysis of electronic components, such as this PCB, at trace element sensitivity without any sample preparation required. In addition, the new AMS keeps the various PCB components in focus, thus making it easy to interpret the results, even though the sample has a high topography.



Analysis of a PCB board
All the relevant components of a PCB board can be clearly identified via SEM-XRF analysis, despite the sample being three-dimensional. Image depth-of-field is maintained thanks to the unique Aperture Management System (AMS).

Technical Data

Sample types	Solids and particles
Excitation	High brilliance, air cooled micro spot X-ray tube with polycapillary X-ray optics, Be side window
- target material	Rh, optional Ag on request
- tube parameters	max. 50 kV, 1000 µA (50 W)
- standard spot size	< 35 µm for Mo Kα (17.5 keV)
- primary filters	software-controlled motorized wheel with six primary X-ray beam filters
Detection	XFlash® silicon drift detector*
Instrument control	No additional PC required, QUANTAX EDS PC can be used (recommended)*, instrument control unit connected via Ethernet
Instrument software	Bruker ESPRIT software, optional XMethod software package for thin film thickness analysis (layered samples)
Instrument control functions	Complete control of tube parameters and filters
Spectra evaluation	XRF peak identification, artifact and background correction, peak area calculation, standardless quantification, combined XRF and EDS quantification, thin film thickness analysis
Distribution analysis	HyperMap capability (hyperspectral database)
Result presentation	Quantification results, statistical evaluation, element distribution (line scan, mapping)
Power supply	100 - 240 VAC (1P), 50/60 Hz
Dimension and weight	XTrace 2 X-ray source: 428 mm x 297 mm x 179 mm, 14 kg XTrace 2 electronics: 489 mm x 500 mm x 196 mm, 15 kg
Quality and safety	XTrace 2 complies with the following safety regulation standards: - IEC 61010-1 (3.1) Europe, U.S., Canada - IEC 61010-1 (3.0) Japan - IEC 61010-1 (2.0) Australia, Korea



*XTrace 2 requires a pre-installed QUANTAX energy-dispersive X-ray spectrometer (EDS), consisting of XFlash® silicon drift detector, SVE signal processing unit and system PC.

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