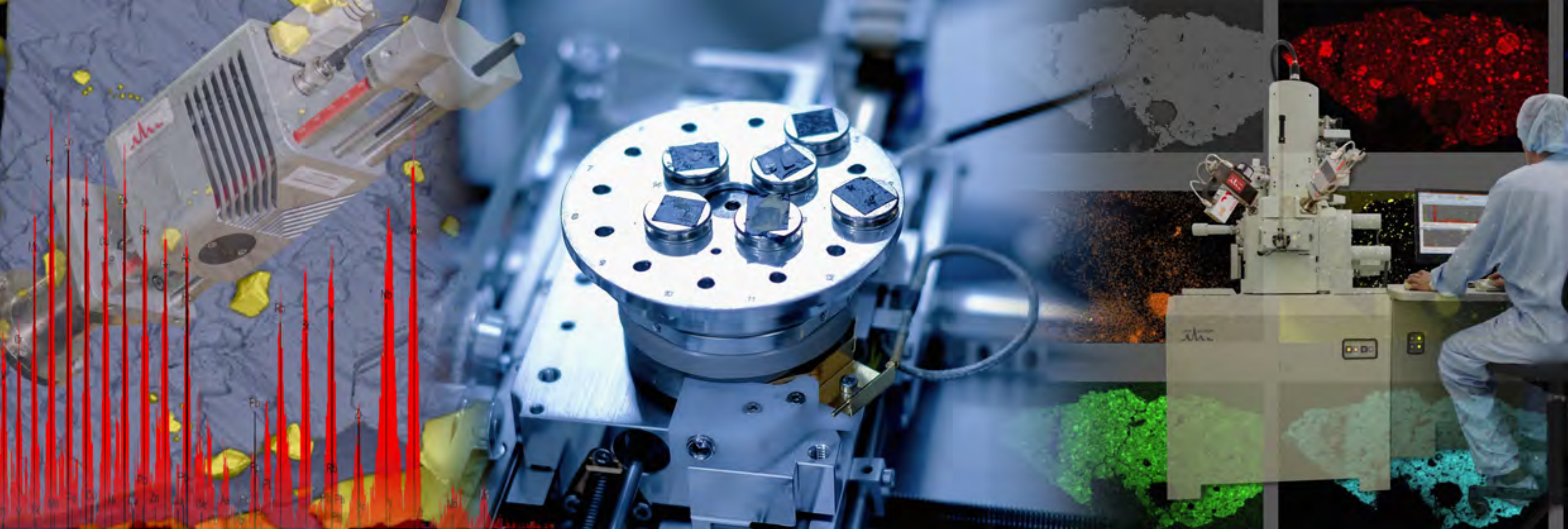
SEM-XRF

microXRF for SEM



https://www.ixrfsystems.com/sem-xrf/

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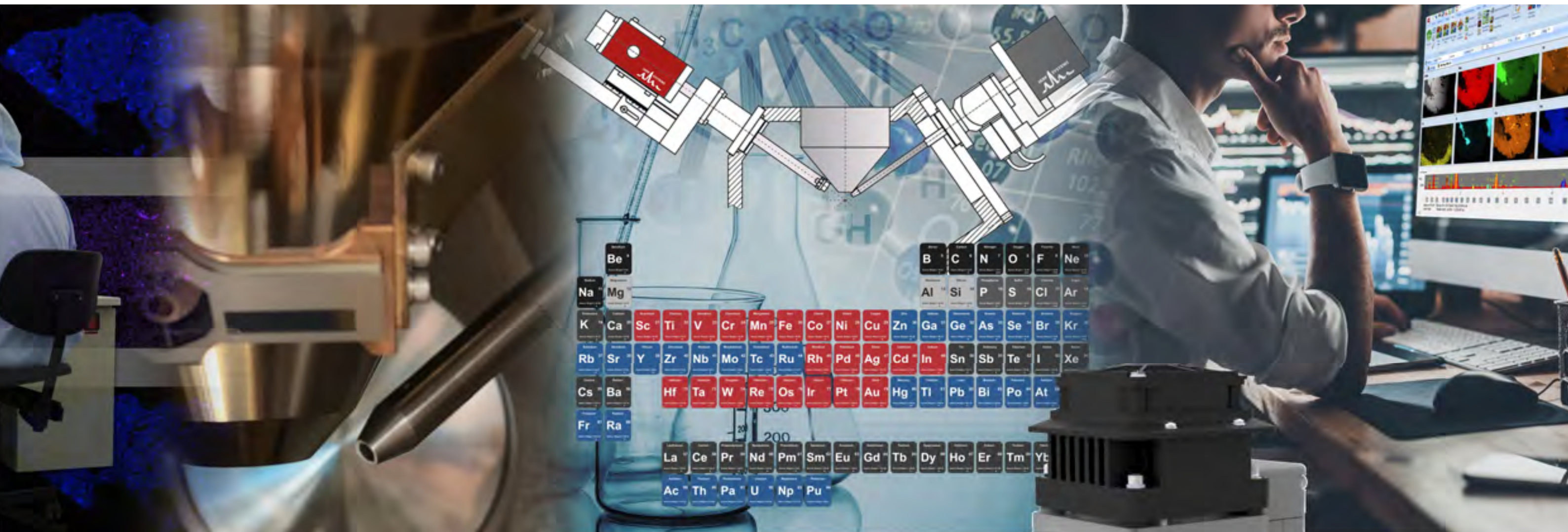
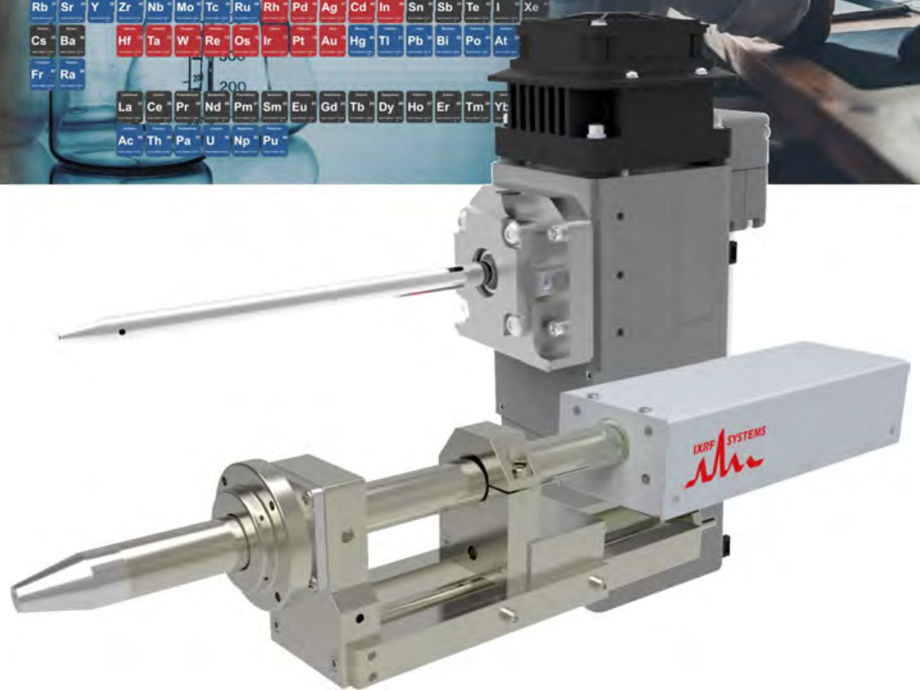




Microfocus X-ray sources for electron microscopy

Energy dispersive X-ray fluorescence spectrometry
for elemental analysis / hyperspectral XRF imaging

SEM-XRF: advanced hyperspectral imaging of heavy elements

Technological progress in the fields of small-spot low-power X-ray tubes and efficient polycapillary X-ray optics has enabled IXRF Systems to develop a line of compact X-ray sources that can be attached to scanning electron microscopes with energy dispersive X-ray spectrometer (SEM/EDS) systems. This innovation allows traditional X-ray fluorescence (microEDXRF) spectrometry to be performed within the SEM, where X-ray excitation allows efficient excitation and quantification of a far larger range of elements than are possible with e-beam excitation. The analytical power of SEM/EDS is significantly enhanced with addition of an X-ray exciation source.

Generally, SEM/EDS analysis takes place at an excitation energy of 20 keV. This method can detect, but not necessarily resolve, elements present in amounts above 0.1wt%. In terms of detectability, e-beam excitation is best suited for light elements with peaks below 2 keV, while SEM-XRF can detect heavier elements at lower concentrations than can be seen with SEM/EDS. Above ~2 keV, SEM-XRF can detect elements to ppm* levels. Additionally, peaks of heavier elements – at higher energies – can be fully resolved. An X-ray source is able to excite Mo K-lines at ~17.44 keV, whereas SEM/EDS is only able to excite the M-lines of molybdenum at 2.29 keV, which almost completely overlaps the K lines of sulfur and M-lines of lead.

*parts-per-million

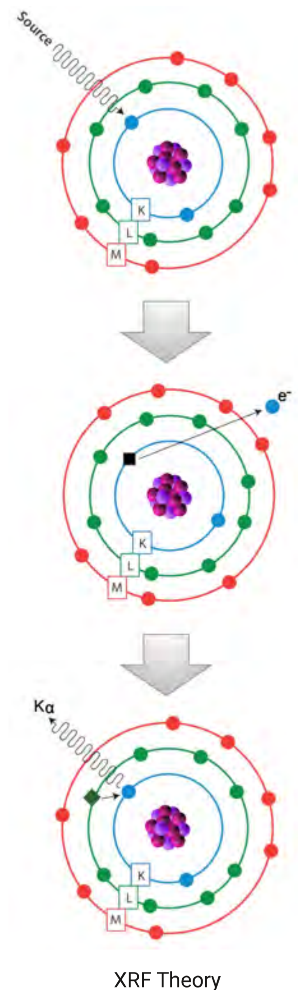
EDS w/ e-beam
< 500 ppm
< 200 ppm
< 50 ppm
< 20 ppm
< 10 ppm
< 5 ppm
Not measurable

Periodic table of the elements for XRF illustrating approximate lower limits of detection (LLD). Note that these values vary widely by matrix and acquisition conditions.

How does X-ray fluorescence work ?

In X-ray fluorescence (XRF), an electron can be ejected from its atomic orbital by the absorption of an electron or light wave (photon) of sufficient energy (see image at right). The energy of the exciting electron or photon (hv) must be greater than the energy with which the inner shell electron is bound to the nucleus of the atom. When an inner orbital electron is ejected from an atom (middle image), an electron from a higher energy level orbital will be transferred to the lower energy level orbital. During this transition a photon may be emitted from the atom (bottom image). This fluorescent light is called the characteristic X-ray of an element. The energy of the emitted photon will be equal to the difference in energies between the two orbitals occupied by the electron making the transition. Because the energy difference between two specific orbital shells, for a given element, is always the same, the photon emitted will always have the same energy.

For a particular energy (wavelength) of fluorescent light emitted by an element, the number of photons per unit time (generally referred to as peak intensity or count rate) is related to the amount of that element in the sample. The counting rates for all detectable elements within a sample are usually calculated by counting, for a set amount of time, the number of photons that are detected for the various elements' characteristic X-ray energy lines. Therefore, by determining the energy of the X-ray peaks in a sample's spectrum, and by calculating the count rate of the various elemental peaks, it is possible to qualitatively establish the elemental composition of the samples and to quantitatively measure the concentration of these elements.



XRF Theory

Interior of SEM showing (from left to right) the EDS detector nose, the SEM pole piece and IXRF's ƒX SEM small spot X-ray source.

What is SEM microXRF ?

Micro X-ray fluorescence (microXRF) spectroscopic analysis is a complementary, non-destructive analytical technique to traditional energy dispersive spectroscopic (EDS) analysis using a scanning electron microscope (SEM). Such analyses are important for the characterization of elemental composition within unknown samples ranging from millimeter sized inhomogeneous specimens (inclusions) down to small micron level particles. In addition, fitting an SEM with a high speed piezo XY stage allows hyperspectral XRF mapping of samples, providing images similar to SEM/EDS elemental maps.

MicroXRF with micron sized focal spots function as a type of XRF hyperspectral imaging microscope, where each pixel (in a map or image) contains information from 0.1–35 keV in the electromagnetic spectrum, affording greatly enhanced analytical capabilities.

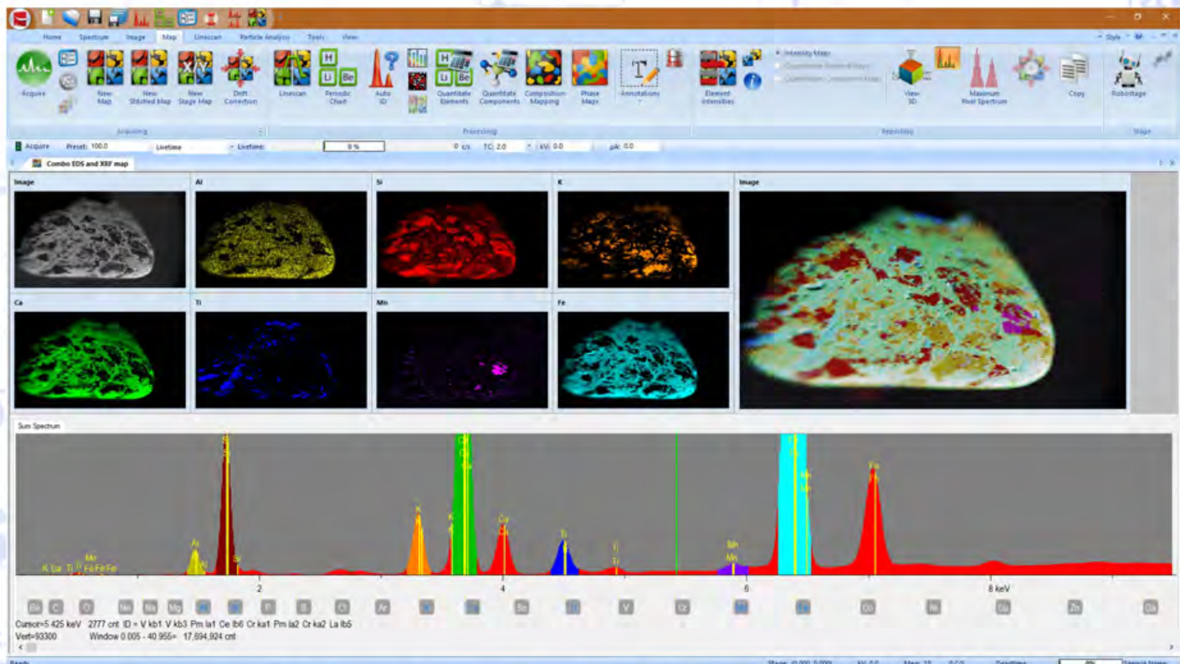
X-ray excitation yields much higher sensitivities for trace element detection (down to as low as 5 ppm for some elements), an extended X-ray spectral range up to 35 keV, as well as information from greater depths within a sample. Deeper "depth of interaction volume" allow characterization of multilayer thin-film (stack) systems, from 1 nm up to 40 µm thick, which is not possible with electron excitation.

Xb SEM™ fX SEM™

Polycapillary optics for10 µm spot X-ray excitation
A 50 kV, 50 W air-cooled X-ray tube with polycapillary focusing optic, Xb SEM is the definitive SEM-XRF solution. Available beam diameters are 10, 20 or 40 microns across a selection of anode materials: Ag, Cr, Cu, Mo, Rh and W. This advanced X-ray source does not interfere with normal SEM operation and may be used simultaneouly with e-beam excitation for combined XRF quantitative analysis.

Close coupled small spot minitaure X-ray source
Never compromise on sensitivity by adding a collimated transmission target X-ray source to your microscope. With a choice of anodes, the fX SEM is available in several spotsizes (down to 200 µm width) and maximum kV configurations. Like for Xb SEM, there is no interference with microscope operation. Simultaneous X-ray and e-beam excitation may be employed for complete analyses.

High precision modular XY linear piezo stage
While your existing microscope stage can be used with SEM-XRF, their different design criteria are less than ideal. Fitting a high speed piezo XY stage delivers the full potential for high-resolution imaging applications. Specially designed to mount on top of the existing SEM stage, open loop velocity is >20 mm/s with <1 micron resolution. This enables the acquisition of X-ray mapping data over a sample size of up to 35 x 35 mm (or higher) depending on microscope chamber dimensions.



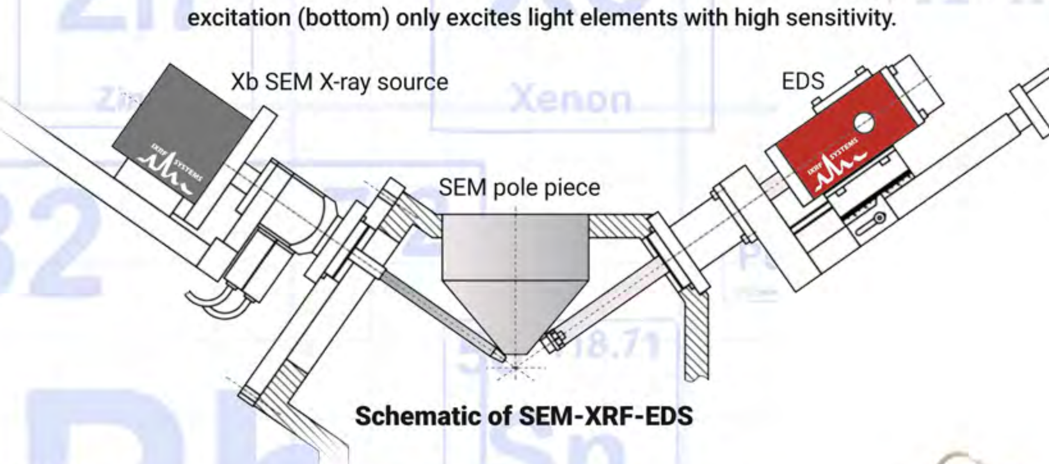
SEM-XRF mapping employing dual X-ray and e-beam excitation
Map tab displays IXRF Systems' microXRF-EDS imaging dashboard with a monochrome view (upper left) of the sample followed by elemental maps. A color keyed sum spectrum for the map is at screen bottom, where spectrum segment colors correspond to map colors. Upper right is a color keyed overlay image.

Quantify elements that are invisible with just SEM/EDS

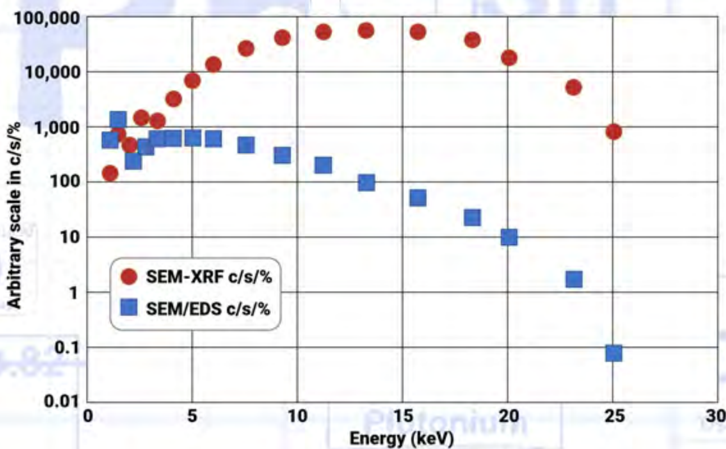
Enhanced microanalysis and hyperspectral XRF imaging

Excitation sensitivity comparison using NIST SRM 610 sample

SEM-XRF's X-ray excitation allows all elements to be observed (top) while SEM/EDS's e-beam excitation (bottom) only excites light elements with high sensitivity.



Schematic of SEM-XRF-EDS

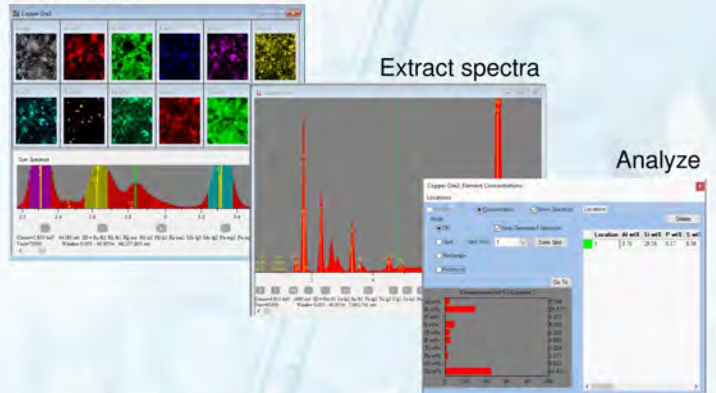


Relative sensitivities of K-lines at 30 kV excitation

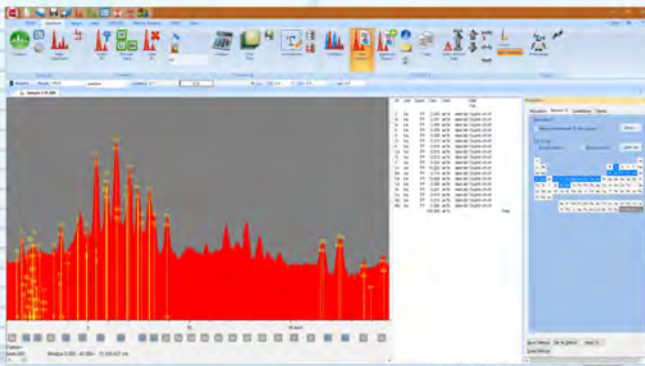
Plot of a theoretical silicon sample containing trace amounts of Na-Sn. At 0-2 keV, e-beam (SEM/EDS) is clearly more efficient. Above about 2 keV, X-ray photon excitation (SEM-XRF) becomes orders of magnitude more efficient. This allows for quantification of more elements at lower concentrations than is possible with SEM/EDS.

Iridium Ultra software

For our SEM-EDS, SEM-XRF and microEDXRF products, IXRF offers Iridium Ultra: an all-inclusive state-of-the-art software suite featuring a myriad of comprehensive qualitative and quantitative capabilities supporting both e-beam and X-ray excitation. Included are stage control and automation, data acquisition, spectral manupulation (including deconvolution and artifact removal), mapping, imaging, and statistical analysis tools. The platform is unsurpassed in it's ability to provide elemental and phase (PCA) mapping, line scans, critical dimensions as well as morphological analyses. As SEM-XRF does not require that samples be coated, it allows non-destructive measurement of a wider variety of matrices.

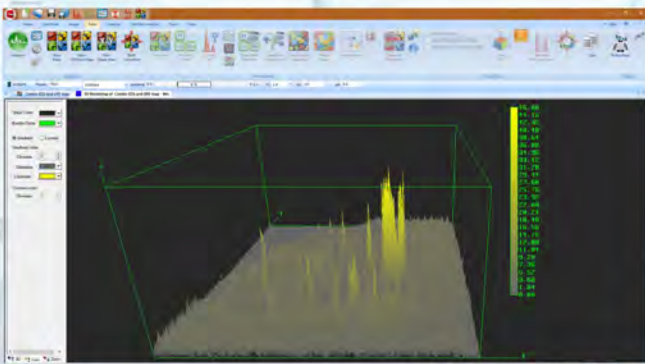


Workflow for SEM-XRF is straightforward. Typically a sample is mapped to reveal features and/or components. Spectra extracted from user defined areas are then analyzed. A formal report may be automatically created if desired.

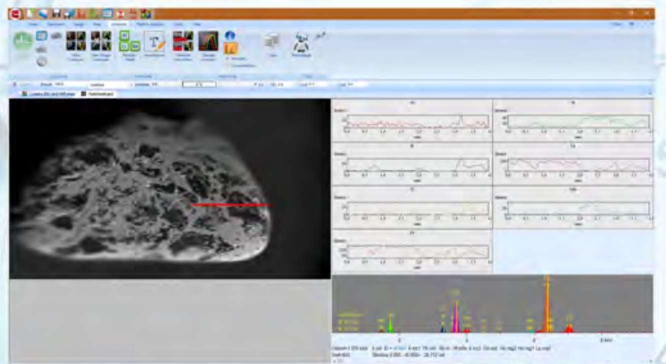


Log view for a stainless steel particle showing spectrum view for qualitative and quantitative data. Note KLM markers, FP quant results and prominate Mo K-lines (17.4 & 19.6 keV).

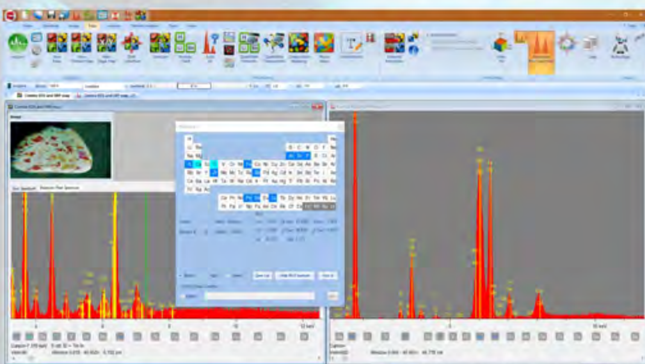
Comparison of SEM/EDS and SEM-XRF for a Al-Ta-Mn alloy specimen showing that X-ray excitation fully resolves all elements, allowing accurate quantification by standardless FP.



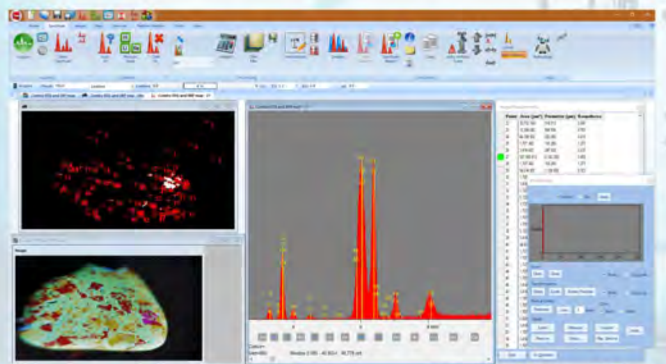
In 3D Viewer, the intensity of each pixel in a map is used as a height (Z axis), thus providing a topographical view. This example shows a 3D plot for Mn in a mineral sample.



In Linescan, an arbitrary line can be drawn to calculate the elemental composition across a map. It is an excellent tool for determining the location of phases, particles and features.



Maximum Pixel Spectrum is constructed by selecting the max pixel value within each energy plane, ignoring the remaining pixels. Highly localized trace constituents are easily found.



Using a polished mineral sample as an example, highly automated morphology tools provide sum spectra analysis of bright Gd-rich inclusions ... with just a few mouse clicks.

Iridium Ultra features*

* for a more comprehensive list with examples, please visit the product's page: ixrfsystems.com/iridium_ultra

- 4K x 4K images/maps
- ASTM E2926 (optional)
- Auto peak I.D.
- Automation
- Composition maps
- Custom reporting
- Elemental maps
- Ratio maps
- Linescans
- Three dimentsional viewer (3D)
- Scatter plot analysis
- Maximum pixel spectrum
- Morphology (feature analysis)
- Phase analysis & mapping
- Principal component analysis (PCA)
- Thermal maps
- Auto quantification
- ZAF
- Match
- Combined quantification
- MLF (multi-layer film) FP
- Standardless FP
- Type standard FP
- Least squares (LS)

About IXRF

For almost three decades IXRF has been designing and manufacturing high-end X-ray Microanalysis systems in the United States of America. We specialize in SEM/EDS, SEM-XRF and microXRF spectrometry.

Specifications

Configurations shown below are subject to change and do not include the necessary flange assembly. An open high angle port (35° degree elevation) is required for addition of a X-ray source. Typically, the open WDS port is employed.

	Xb SEM™	fX SEM™
Tube type	Side-window	End-window (transmission)
Anode materials	Ag, Cr, Cu, Mo, Rh or W	Ag, Cr, Cu, Ti, Mo, Rh or W
Voltage range	10 – 50 kV	4 – 35 kV
Emission current	1000 μA (max)	100 μA (max)
Beam diameter	10, 20 or 40 μm	200, 500 and 1000 μm (others available)
Optics	Polycapillary (focusing)	Pinhole collimation
Tube filters	Optional (up to 8, automated)	Optional (manual)
Cooling	Air (fan forced)	Air (convection)
Safety	Internal interlocked shutter. Interlocked to SEM, keyed power-on switch, HV-On lamp, warning beacon	Interlocked to SEM, keyed power-on switch, HV-On lamp, warning beacon
Control	Variable control kV/μA, X-ray on/off buttons, kV/μA display	Variable control kV/μA, X-ray on/off buttons, kV/μA display
Stage	High speed piezo type (optional)	High speed piezo type (optional)
Dimensions	244 (L) x 76 (W) x 249 (H) mm	520 (L) x 78 (W) x 54 (H) mm
Weight	5.9 kg (w/o flange)	1.0 kg (w/o flange)
Power	100-240 VAC, 1 phase, 50/60 Hz	100-240 VAC, 1 phase, 50/60 Hz