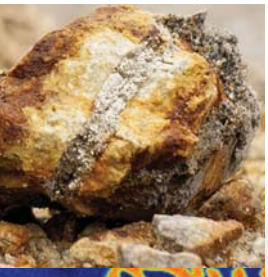
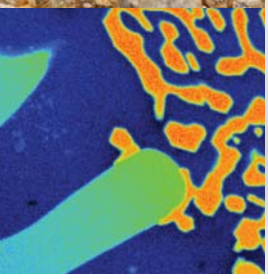
CAMECA
SCIENCE & METROLOGY SOLUTIONS

SX Five tactis

Electron Probe Microanalysis at your fingertips

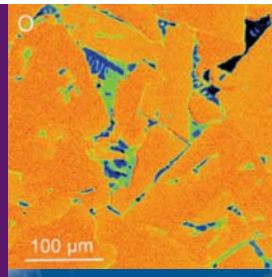


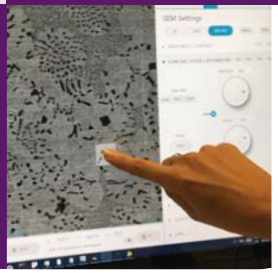


X-RAY SPECTROMETRY

CHEMICAL MICROANALYSIS

QUANTITATIVE MAPPING





AMETEK
MATERIALS ANALYSIS DIVISION

Sixty years of EPMA expertise and passion

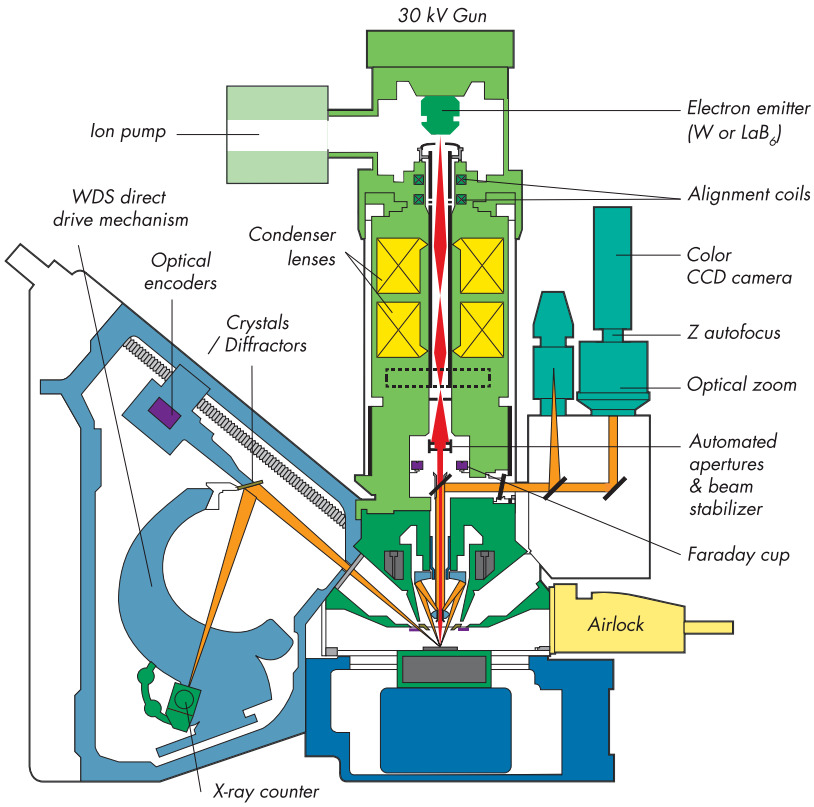
The SXFive-TACTIS Electron Probe MicroAnalyzer is the culmination of more than sixty years of our experience in EPMA technology.

In 1958, CAMECA's first electron microprobe was inspired by the originator of the technique, Prof. Castaing of the University of Paris. Since then, CAMECA has worked in close cooperation with leading scientists and academics to bring continuous improvements to Electron Probe MicroAnalyzer (EPMA) technology. We have succeeded in expanding EPMA from a laboratory-only technique to a workhorse analytical system useful in R&D labs as well as in industrial process control.

Offering higher and more stable beam currents as well as better X-ray peak-to-background ratios than other existing instruments such as SEM-EDS, the WDS-based CAMECA Electron Probe Micro-Analyzers allow precise and accurate quantitative analysis of major and trace elements. High intensity crystals ensure excellent sensitivity for all elements, while the choice of the electron source gives access to sub-micron lateral resolution microanalysis. Automation technology from our fab-line EPMA platform also ensures optimal long-term reliability.

The SXFive-TACTIS builds on this legacy to deliver superior imaging and quantitative analysis in a unique, user-friendly interface. Its dual operation mode (touchscreen or expert), makes it a perfect fit in multi-user facilities.

State-of-the-art EPMA technology



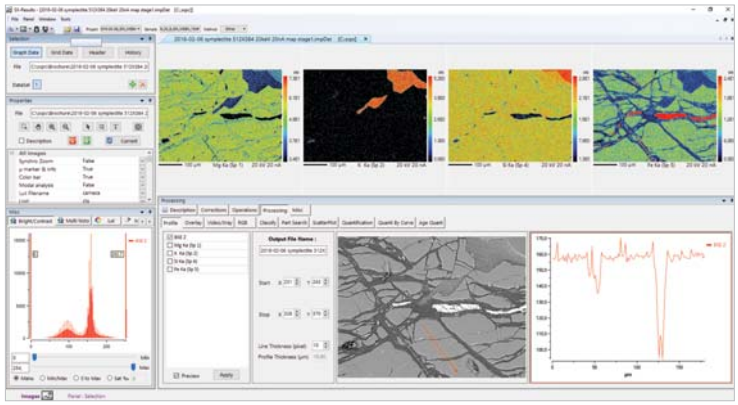
Excellence in every instrument detail:

- A choice of W/LaB_6 or Field Emission source with on-site upgrade possibility for more flexibility (shown here: synoptic of W/LaB_6 model).
- Optimized electron optics for higher lateral resolution.
- Superior vacuum system for better detection of light elements.
- Annular Faraday cup for enhanced reliability.
- Additional BSE detector for superior image quality, especially at low voltage.
- Choice of high intensity crystals (diffractors) for full spectrometer coverage or targeted analytical requirements.
- Enhanced automation for optimized throughput.

Five SXFive-TACTIS key wins

- Dual interface (beginner mode / expert mode) ⟶ Operational flexibility with a single tool at your multi-user facility.
- Additional BSE detector ⟶ Enhanced imaging especially at low beam voltages and/or low beam currents.
- Fully integrated EDS hyper-mapping module ⟶ Ultra fast quantitative analysis.
- Unique X-Live function ⟶ Real time acquisition of WDS and EDS X-ray images.
- Full remote control including SEM image ⟶ Run your experiment from your smartphone, tablet or any remote computer.

SXFive-TACTIS dual interface: Non-expert users can easily launch all mapping and quantification tasks in the touchscreen interface (left), and still access the complete set of powerful data processing features of the SX-Results module (right).



SX-Results module in PeakSight, the CAMECA EPMA automation & analysis software.

SXFive-TACTIS: EPMA at your fingertips

The world's best WDS technology

The Wavelength Dispersive Spectrometers (WDS) at the heart of CAMECA's SXFive-TACTIS offer the best resolution and sensitivity on the market, and deliver highly reproducible quantitative analyses.

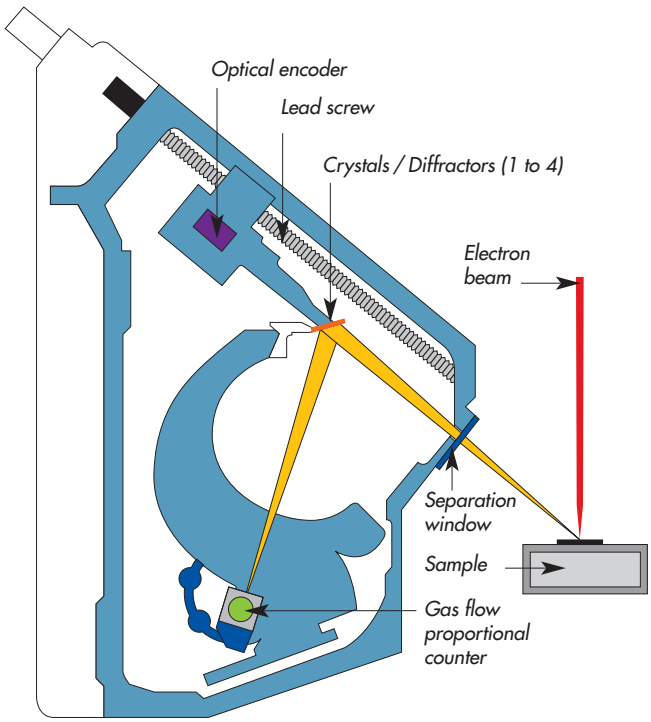
Wavelength Dispersive Spectrometry is acknowledged as the method of choice for high precision quantitative microanalysis. Exclusively designed by CAMECA, the SXFive-TACTIS WDS spectrometers have the highest resolution in the industry and, as a result, high peak-to-background ratios, thereby providing high sensitivities. The absolute spectral positioning is provided by optical encoders attached to the system. Only one peak measurement is needed to calibrate the entire spectrometer range!

Up to five WDS spectrometers (plus one Energy Dispersive Spectrometer - EDS) may be fitted to the instrument, depending on the application.

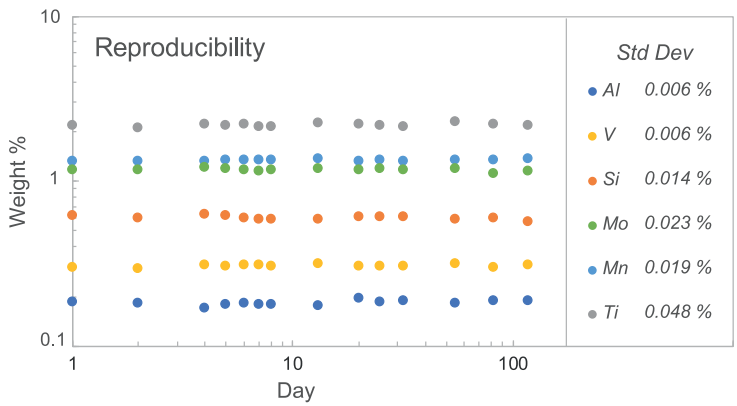
All spectrometers have the same 160 mm Rowland circle radius. Column separation windows and differential pumping guarantee that the spectrometers and diffracting crystals remain free of any contamination from the specimen chamber. Motors are under vacuum to eliminate feedthroughs, ensuring high reliability and reproducibility.

Thanks to the unique design of the CAMECA WDS, the entire spectrometer range can be scanned in only 15 seconds. Mounted at a take-off angle of 40°, the spectrometers are linear and fully-focussing. Their mechanics have been optimized to include a minimum number of moving parts (no belt-driven mechanism), thus ensuring excellent reproducibility and reliability.

Pre-aligned in the factory, the CAMECA WDS do not require either routine maintenance or periodic retuning.



Excellent reproducibility



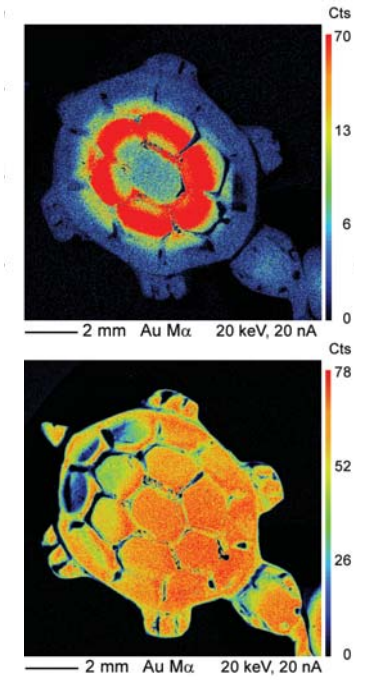
In this example, a stainless steel sample was analyzed repeatedly over a period of four months without re-calibration of the WDS.
The diagram clearly demonstrates the excellent reproducibility of the CAMECA WDS, ensuring reliable measurement of trace quantities and major components.

Optimized WDS orientation

The CAMECA spectrometers can be mounted vertically for flat and polished specimens, or in an inclined orientation for rough specimens.

A vertically mounted WDS is sensitive to small ΔZ variations in the specimen X-ray source away from the Rowland circle along the beam axis.

The inclined WDS (an exclusive design by CAMECA) is much less sensitive to the specimen X-ray source position along the electron optical axis. Variation in Z-focus across the sample has a minimal effect on the diffracted WDS X-ray intensity. This makes the inclined WDS particularly useful for the analysis or mapping of specimens with a rough surface (up to a few millimeters roughness).



Comparison of the signal given by a vertical and an inclined WDS:

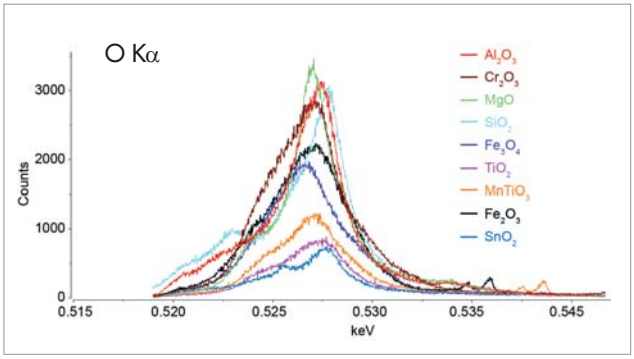
Mapping of the Au Ma X-ray signal on a turtle-shaped pendant.

The increased depth of field of the inclined spectrometer (bottom image) dramatically reduces the influence of sample topography.

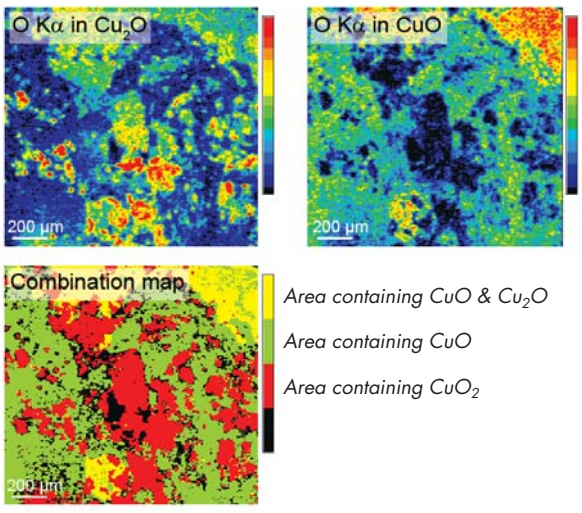
Extended spectrometer

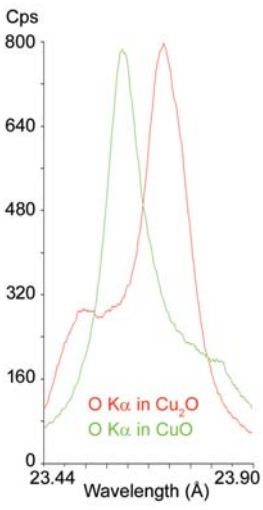
Mounted with a TAP or a LTAP crystal, the extended sine-theta spectrometer is the configuration of choice for challenging analytical applications, such as the detection of oxygen in different oxides. Excellent spectral resolution can be achieved, thus direct qualitative chemical mapping is also possible.

The WDS spectra of different oxides shown on the right clearly reveal the oxygen peak shift due to different oxidation levels.



Optimized phase mapping with Extended WDS & Image Overlay Software





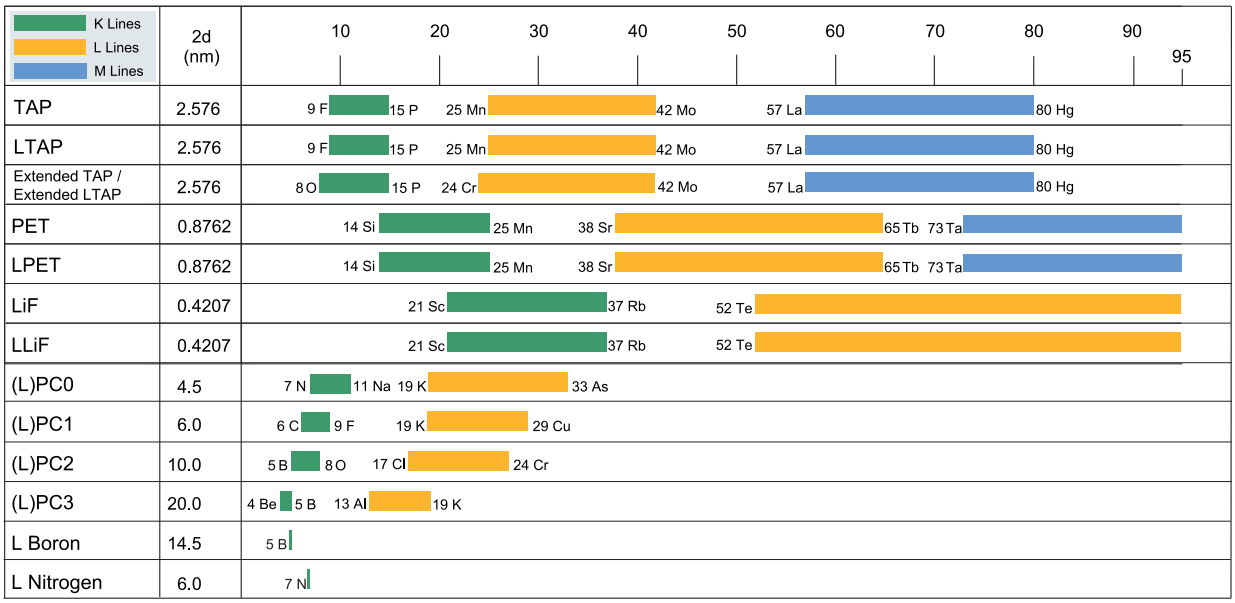
Analysis of a copper sample suspected to contain two types of oxides.

The Extended WDS - TAP crystal configuration enables the easy acquisition of maps from different oxygen peaks.

The *Image Overlay* software module can then be used to display different phases, distinguished by different colors, in a single combination map.

The largest choice of high intensity crystals

CAMECA offers several types of crystals to cover the entire periodic table and address all microanalytical challenges. Whatever the size of the crystals, the analytical range of our spectrometers remains unchanged.

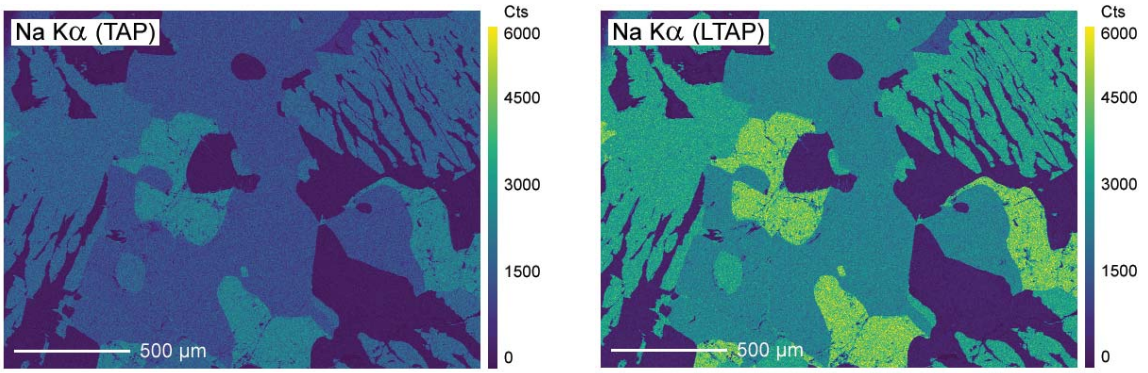


Large diffractors (natural or pseudo-crystals)

Large crystals with full spectrometer coverage and high peak-to-background ratio allow faster WDS analysis. Thanks to their high counting rates, they are extremely useful for measuring beam sensitive materials and trace elements.

Excellent signal / noise ratio with large crystals

X-ray images in nepheline syenite gneiss from Ontario, Canada, obtained simultaneously with regular and large crystals. The large crystal allows faster detection of Na X-rays from nepheline, albite, and sodic pyroxene, reducing beam dwell time needed to measure these beam sensitive minerals. Higher intensity and better signal/noise ratio can be obtained.



LTAP: maximum intensity & energy resolution

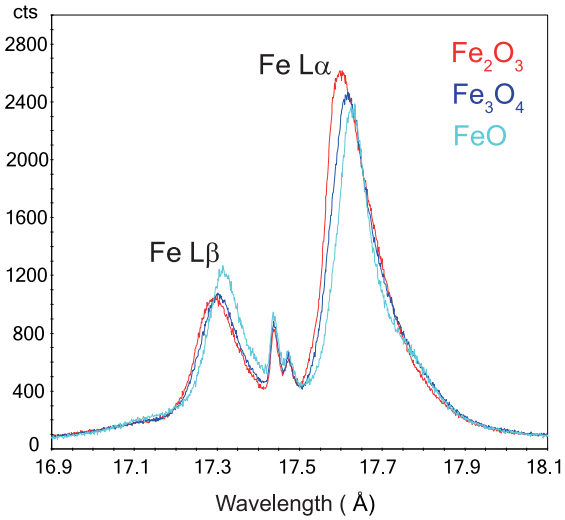
Uniquely available from CAMECA, the LTAP crystal enables a combination of maximum intensity and maximum energy resolution for a large range of elements, which is of great interest for mineral speciation, e.g., Fe.

Direct shift measurement on Iron with LTAP

Offering the highest count rate and energy resolution to measure Fe Lα and Fe Lβ peaks, the LTAP crystal is the ideal crystal for determining precise and accurate Fe speciation. These spectra can be used to differentiate Fe^{2+} and Fe^{3+} in hematite and magnetite as shown on the opposite graph.

Please refer to the 'Flank method' developed by Höfer et al.: Höfer, H.E. Hyperfine Interactions (2002) 144: 239. https://doi.org/10.1023/A:1025461907725.

The oxidation level or mineral can also be determined using the O Kα measurement by CAMECA extended spectrometer as shown on page 5.

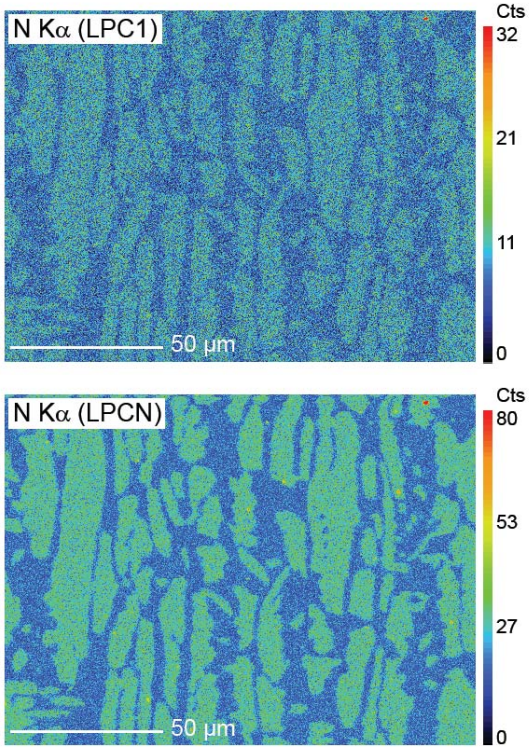


Special large pseudo-crystals for light element analysis

Special large pseudo-crystals for ultra high sensitivity B and N analysis and multilayered pseudo-crystals for light elements (Be to F) are also available.

Shown on the right are N X-ray maps of a duplex stainless steel, comparing counts obtained with LPC1 (standard large pseudo-crystal) and LPCN, our nitrogen-dedicated large pseudo-crystal at the same mapping conditions (beam voltage, beam current, dwell time).

The LPCN pseudo-crystal enables measurements at a much higher count rate at the N Kα position (an increase by a factor of 2.8) while the background is maintained at the same level, thus achieving excellent nitrogen detection sensitivity, even at short dwell times (40 ms/pixel).



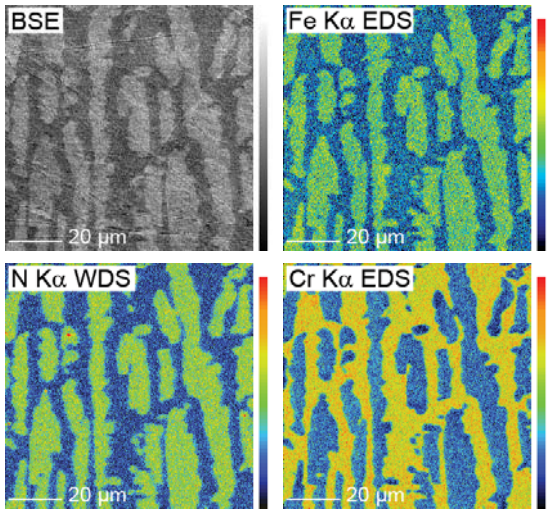
Fully integrated EDS analysis, now with hypermapping

Energy Dispersive Spectrometry combines with Wavelength Dispersive Spectrometry to offer the best of both worlds.

Simultaneous EDS/WDS acquisition

SXFive-TACTIS can be equipped with an Energy Dispersive Spectrometer (EDS) system suitable for rapid mineral or phase identification. The EDS detector can be used simultaneously in combination with all the WDS detectors in quantitative and imaging modes. Higher throughput is achieved with such WDS/EDS configuration by acquiring, for example, major elements on EDS and trace elements on WDS.

Shown here are simultaneously acquired EDS and WDS maps of a steel sample (15 keV, 200 nA).



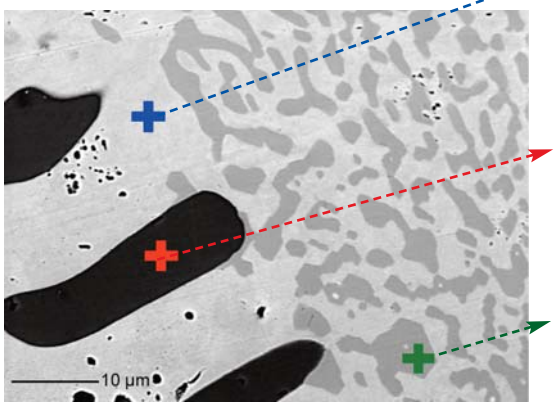
Qualitative images in counts

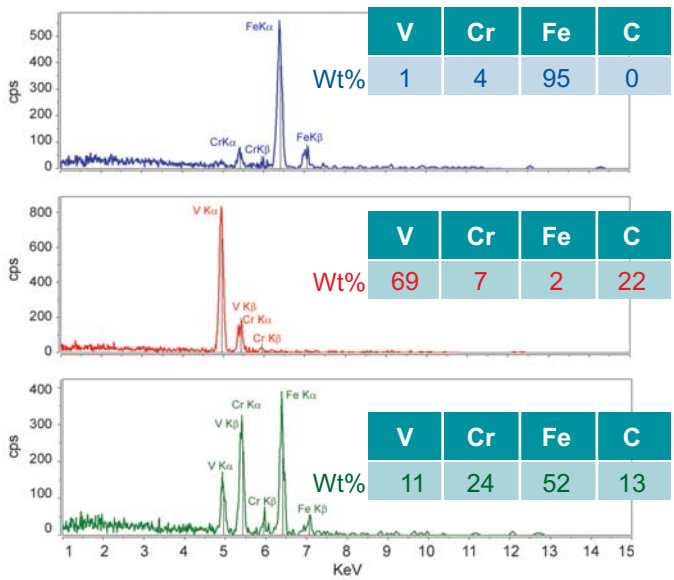
Ultra-fast quantitative analyses with EDS HyperMap

SXFive-TACTIS comes with a fully integrated EDS HyperMap module, making data processing and analysis faster and easier. Using EDS HyperMap, you can collect the full EDS spectrum at each pixel and extract your quantitative results simultaneously.

Quantitative data from each pixel in a snap

VCrFe sample analyzed at 15 kV / 20 nA.





	V	Cr	Fe	C
Wt%	1	4	95	0

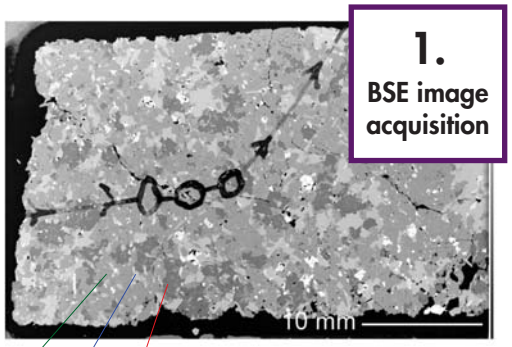
	V	Cr	Fe	C
Wt%	69	7	2	22

	V	Cr	Fe	C
Wt%	11	24	52	13

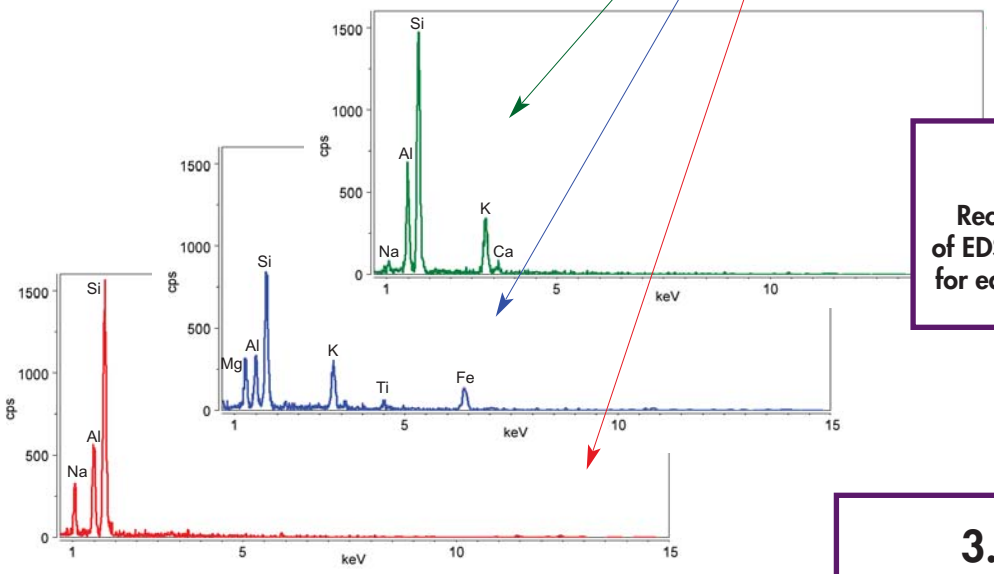
Easy EDS X-ray map processing

EDS HyperMap acquisition stores a full EDS spectrum at each map pixel. Therefore, individual x-ray maps for all EDS-detectable elements can be extracted from collected data, without taking additional time to acquire more maps. Also, the EDS spectrum at any pixel can be displayed by a simple point-and-click on an acquired image. Save time and mine your data for more information! Through Peaksight, WDS X-ray maps and EDS HyperMaps may be simultaneously collected.

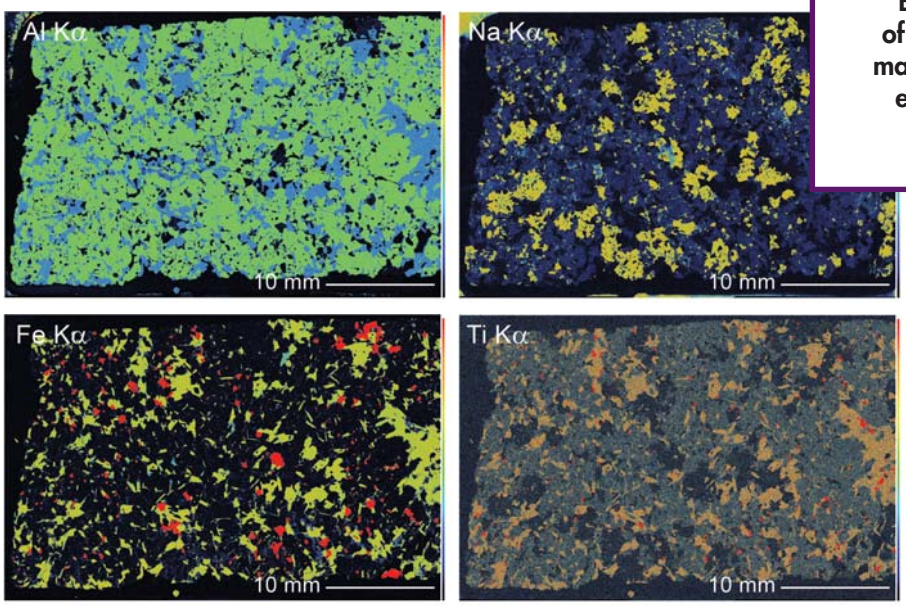
In this example, a complete geological thin-section (27 mm x 46 mm) of a nepheline syenite gneiss (Faraday, Ontario) was mapped at 15 keV, 35 nA using Peaksight's stage mosaic mode.



1.
BSE image acquisition



2.
Recording of EDS spectra for each pixel



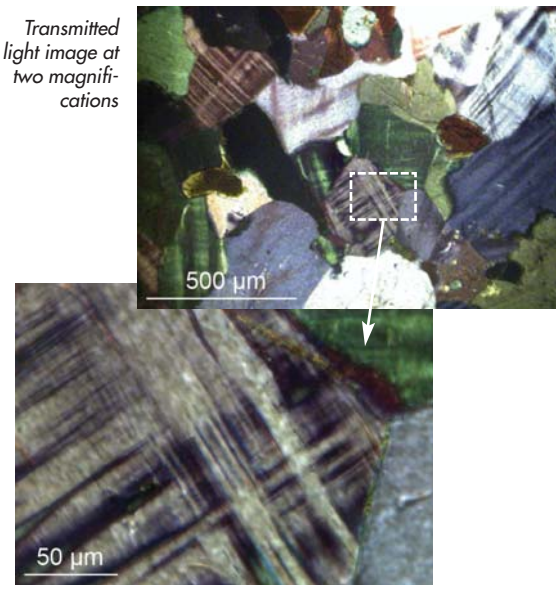
3.
Extraction of EDS X-ray maps for each element of interest

A robust environment:
easy sample survey & stable vacuum

The best optical image

SXFive-TACTIS offers the best optical image on the market thanks to a high quality optical microscope, coaxial with the electron beam. Imaged by a CCD camera, specimens are viewed in reflected light for sample location and precise height adjustment. An optional transmitted-polarized light option allows polarized reflected light for opaque specimens and polarized transmitted light for thin-sections (polarizers may be rotated and crossed). A motorized lens enables an optical zoom with a continuously variable field-of-view (from 250 to 1750 µm). A Z autofocus system guarantees that the specimen surface returns to the focused position as the specimen stage is moved between analysis points.

Transmitted light image at two magnifications



Excellent vacuum

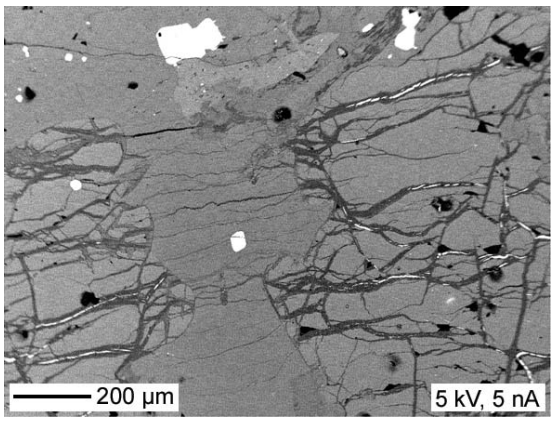
SXFive-TACTIS has been designed to ensure high vacuum quality. The base pressure is in the 10^{-4} to 10^{-5} Pa range inside the analysis chamber and 10^{-6} Pa inside the gun chamber (W/LaB_6 configuration). The high-vacuum turbomolecular pump and the use of an oil-free technology primary pump ensure a clean vacuum and excellent results on light element analysis.

Carbon and light element analyses are extremely important, especially in metallurgy. Carbon build-up must be reduced in order to achieve low detection limits. An optional anti-contamination device, consisting of a liquid nitrogen-cooled cold plate and/or a gas jet, allows the SXFive-TACTIS to measure traces of carbon in steel at very low concentrations.

The vacuum system is entirely automated and fully protected by all necessary safety and control devices. Pressures throughout the instrument are measured with gauges and are continuously monitored and displayed on a user-friendly synoptic panel.

BSE low kV detector

Accurate quantitative analysis of small phases is possible when working at low electron energy, with accurate identification of the region of interest. The additional BSE detector in SXFive-TACTIS delivers superior image quality, especially at low voltage and/or low beam currents. It will enable you to quickly and more accurately identify the areas of interest in your sample and achieve better detection of trace elements.



BSE images are helpful for obtaining compositional maps of a sample and for distinguishing different phases preceding spot probe analyses by either EDS or WDS.

Shown here: BSE image of an olivine-rich mineral specimen acquired at 5 kV/5 nA. Resolution: 2048x1536. Acquisition time: 18 sec.

Innovative dual interface

The SXFive-TACTIS dual interface has been specifically designed for multi-user facilities to take full advantage of a single tool, allowing both beginner and expert utilization.

Choose your interface

SXFive-TACTIS can be operated in two user modes:

- In beginner mode, the instrument configuration, tool operation, as well as basic imaging and data processing, are made easy thanks to a new, intuitive, touch screen interface providing immediate access to a wealth of simplified options.
- In expert mode, the interface is designed for skilled users who can benefit from the full set of tool parameters and software options.

XLive

Uniquely available on the CAMECA SXFive-TACTIS microprobes, the XLive feature allows the acquisition of real-time WDS and EDS X-ray images in one click, producing a rapid, yet meaningful overview of the specimen composition, either in composite or superposed mode.

Assisted quantification

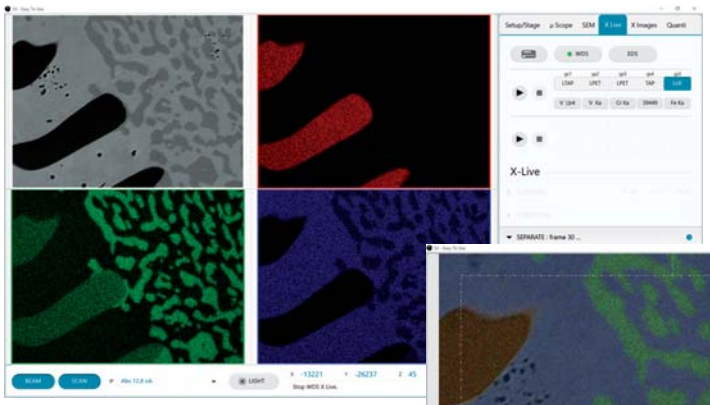
New in SXFive-TACTIS, the *Quanti* tool greatly facilitates the quantification process, allowing non-experts to easily obtain accurate elemental composition of their samples. Various scenarios are available depending on whether or not the user has prior knowledge of the species, and whether or not an appropriate analytical set-up is stored in the database.

As an example, in the case of a completely unknown sample, an EDS or WDS spectrum will automatically be acquired, allowing you to identify the elements and to determine the best analytical set-up.

Full remote control

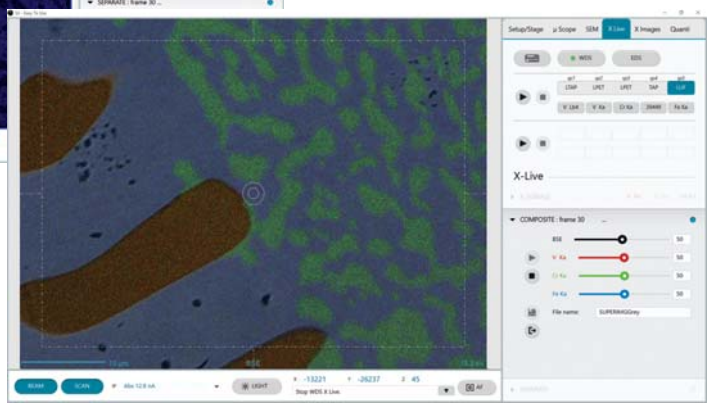
SXFive-TACTIS offers full remote control, including SEM imaging, allowing users to run experiments from their smart phone, tablet, or remote computer. The comprehensive online help has been entirely redesigned for easy access and continuous support during tool set-up and analyses.

Real time WDS & EDS X-ray imaging in one click with XLive



Use preset crystal configurations to acquire up to three X-ray signals plus one SE or BSE image.

VCrFe sample analyzed at 15 kV/20 nA.



Above: in "Accumulate" mode, the electron and the X-ray signals are displayed separately.

Right: in "Superpose" mode, the three X-ray signals are overlaid on the same image.

SXFive-TACTIS applications

CAMECA electron microprobes have been successfully applied to a diverse range of applications from geochronology, mineralogy and nuclear research to materials, metals, thin films and semiconductor research.

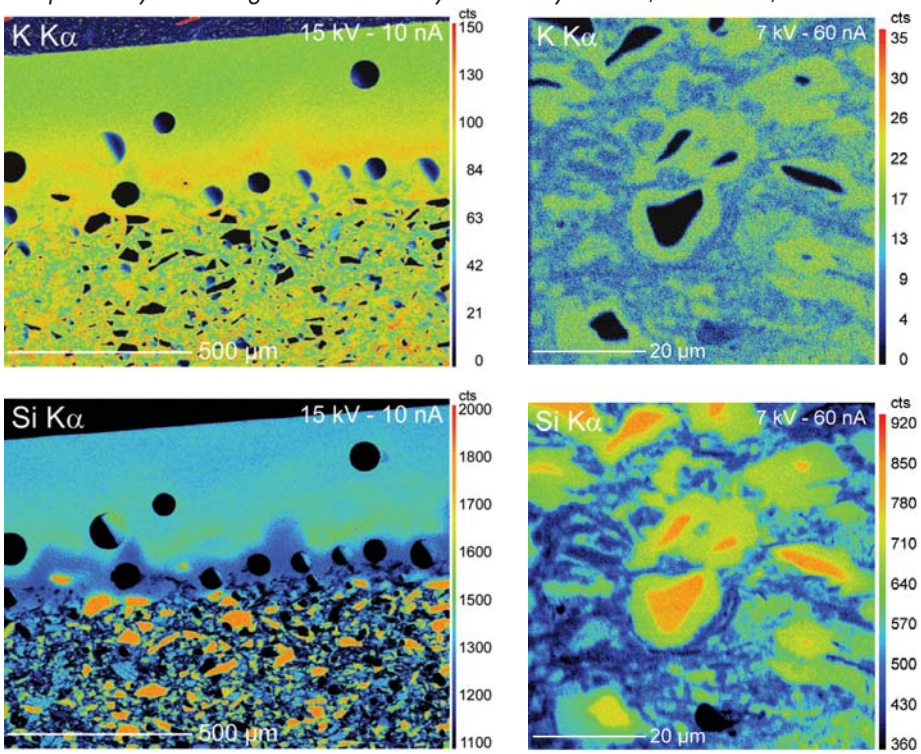
MATERIALS SCIENCE

Quantitative analysis of refractory materials

SXFive-TACTIS delivers true quantitative analysis thanks to the φρ(z) correction routine included in PeakSight. Quantitative data can be displayed such as wt%, oxide wt%, cations along with other measurements. The PeakSight quantitative analysis module also allows analyses with up to four experimental conditions for a single point, thus optimizing the detection parameters for each element of interest, a feature of particular interest for the analysis of challenging refractory materials such as glass or ceramics.

Below is an example of X-ray mapping and quantitative analysis of an ancient porcelain artifact specimen. Two regions are clearly visible on the X-ray maps; the enamel coating and the postherd matrix. During firing, the molten enamel was fused to the potsherd matrix. Note the fine features of this interdiffusion area on the pair of X-ray maps acquired at 7 keV (right). Not only major and minor elements but also oxides are quantified with excellent detection precision and accuracy.

Samples analyzed during round robin study initiated by J. Ruste, GN-MEBA, France.



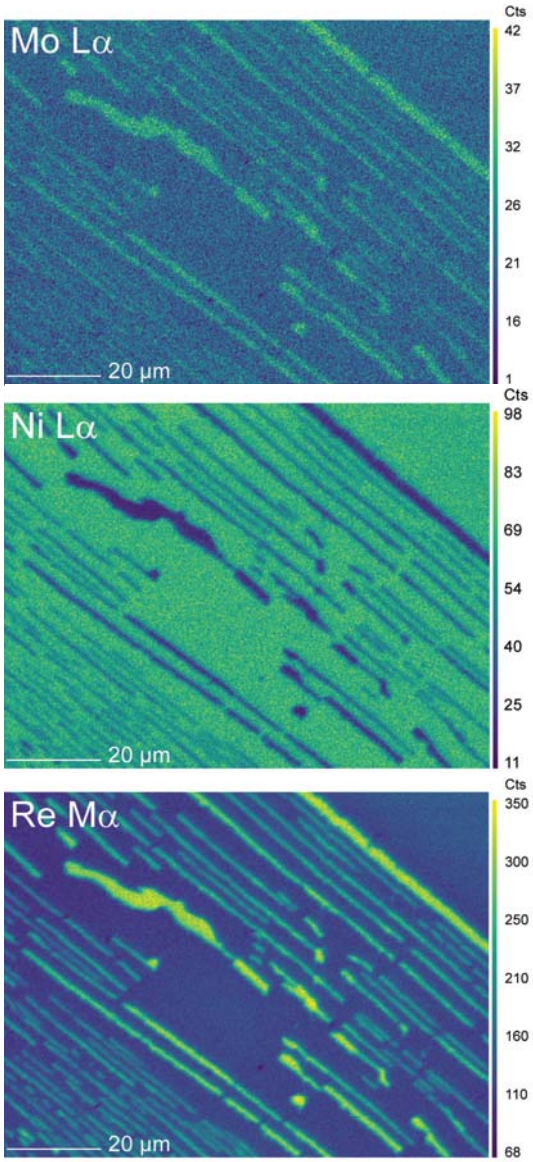
Quantitative analysis of the enamel coating

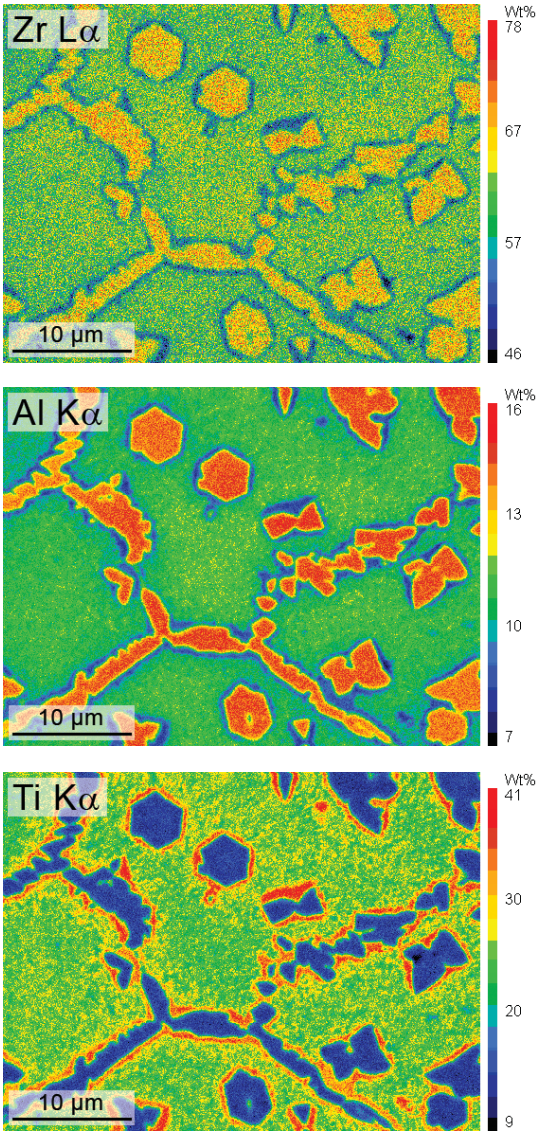
	Weight %
Major elements	
Si	34.53
Al	7.16
K	2.02
Ca	4.41
Mg	1.49
O	49.16
Minor elements	
Na	0.53
P	0.05
Fe	0.17
Ti	0.01
Ba	0.05
TOTAL	**99.58**

Oxides	SiO_2	MgO	Al_2O_3	K_2O	CaO	NaO	P_2O_5	FeO	TiO_2	BaO	**TOTAL**
Weight %	73.85	2.47	13.54	2.44	6.16	0.71	0.11	0.22	0.02	0.06	99.58

High spatial resolution mapping of precipitates in novel alloys

The CAMECA EPMA are widely utilized to support the developement of novel alloys. Applying low accelerating voltages (down to 5 keV) with intense beam currents in the SXFive-TACTIS, the electron-sample interaction volume is reduced, and high spatial resolution can be achieved with high X-ray count rates.





Above
X-ray maps of novel Zr-Al-Ti alloys were acquired at 7 keV / 100 nA, which makes it possible to observe the variation of interface composition around the precipitates, enabling understanding of their formation processes.
Sample courtesy of J. LaCombe, University of Nevada, Reno, USA.

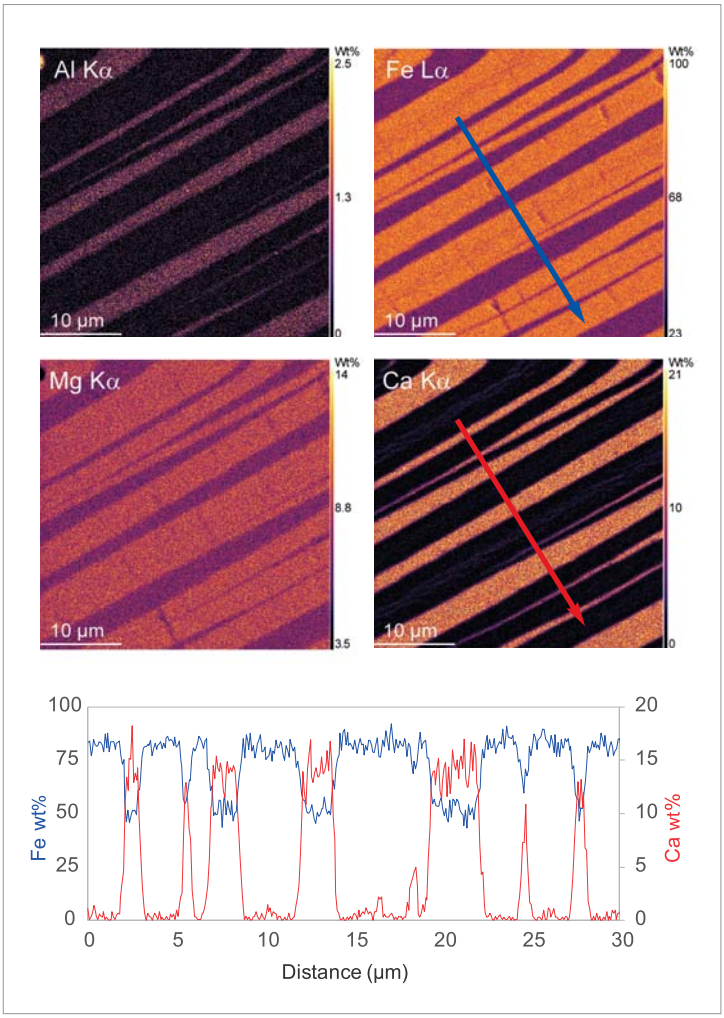
Left
An isothermal section (1200°C) of a Mo-Ni-Re system was imaged at 5 keV / 100 nA with a beam spot smaller than 100 nm. The Re Ma map shows very clear details in the range of sub-micron lateral resolution.
Sample courtesy of K. Yaqoob, E. Leroy and J.M. Joubert, ICMPE, Thiais, France.

SXFive-TACTIS applications

EARTH & PLANETARY SCIENCES

The frontier of geological applications for EPMA is in precise and accurate trace element measurements. Thanks to their high-precision WDS and stable, reproducible current, the CAMECA EPMAs have been the platform of choice for the development of emergent applications in monazite geochronology and many other trace element applications.

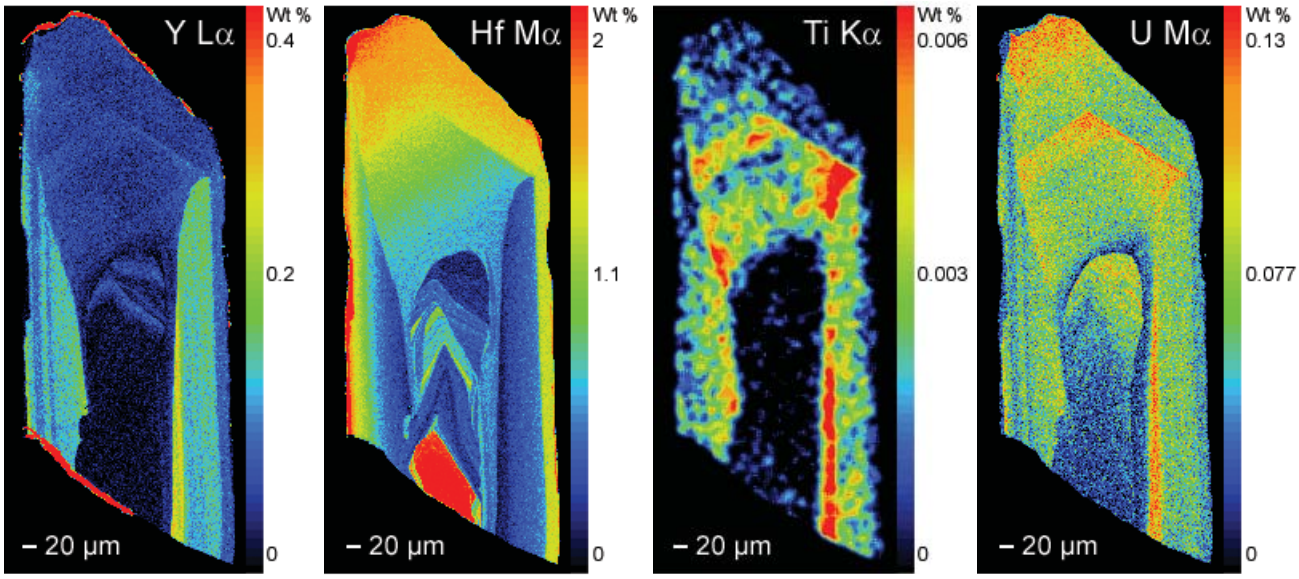
Quantitative analysis of complex meteorite breccias



Quantitative X-ray images of a Howardite meteorite (Tindouf area, Sahara) were acquired at high spatial resolution using a focused electron beam of 7 keV and 30 nA.
Precise and accurate compositional analysis of this kind of meteoritic sample is quite challenging because the rock was intensely brecciated by meteoritic impact and mixes different lithologies (eucrite and diogenites in the broad sense and chondritic or metallic impactors).

The profile plot of Fe Lα and Ca Kα concentrations (in wt%) taken along the red overlay line demonstrates a lateral resolution better than 400 nm.

Sample courtesy of B. Moine, J-L. Devidal and T. Hammouda, University of Clermont-Ferrand, France.



Trace element zoning in zircon

Quantified X-Ray maps at 15 keV and 200 nA of Y, Hf, Ti and U in a zircon from a Papua New Guinea eclogite showing trace element zoning reflecting formation conditions. Hafnium X-ray map reveals zoning within a ca. 4.6 Ma old zircon. Quantified background subtraction at each pixel allows accurate visualization of very low concentration elemental distributions.

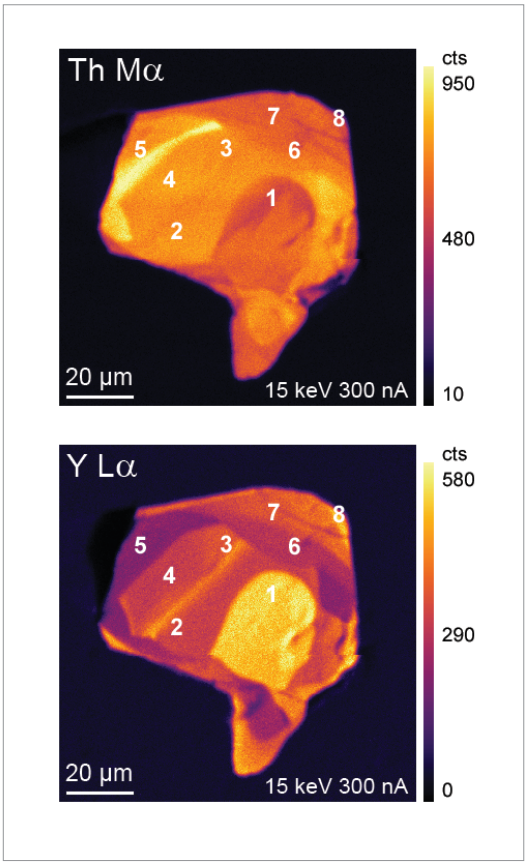
Sample courtesy of J. Desormeau and S. Gordon, University of Nevada, Reno, USA.

GEOCHRONOLOGY

Non-destructive dating of monazite grains

Monazite is a remarkably robust mineral for geochronology, and is commonly measured by conventional isotopic methods. However, multiple ages are often recorded within a single monazite grain, and on a spatial scale smaller than can be resolved by even small-spot isotopic instruments. Non-destructive EPMA analysis can be used for accurate in situ measurement of Th, Y, U and Pb in order to determine the crystallization age of subdomains, even on a submicron scale. The high spectral resolution and reproducibility of the CAMECA WDS brings unmatched precision and accuracy to the trace element measurements that support this technique.

Y Kα and Th Mα WDS map of monazite grain from the eastern Adirondack Mountains, New York, USA. The center of this grain has been dated by EPMA at 1175 +/- 5 m.y. whereas the edge of the grain has been dated at 975 +/- 15 m.y. Eight distinct compositional domains can be distinguished. Domains 1 through 5 grew in a left-right orientation whereas domains 6, 7, 8 grew in a perpendicular orientation, suggesting a change in the active geological forces.



Sample courtesy of M. Jercinovic and M. Williams, University of Massachusetts, Amherst, USA.

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