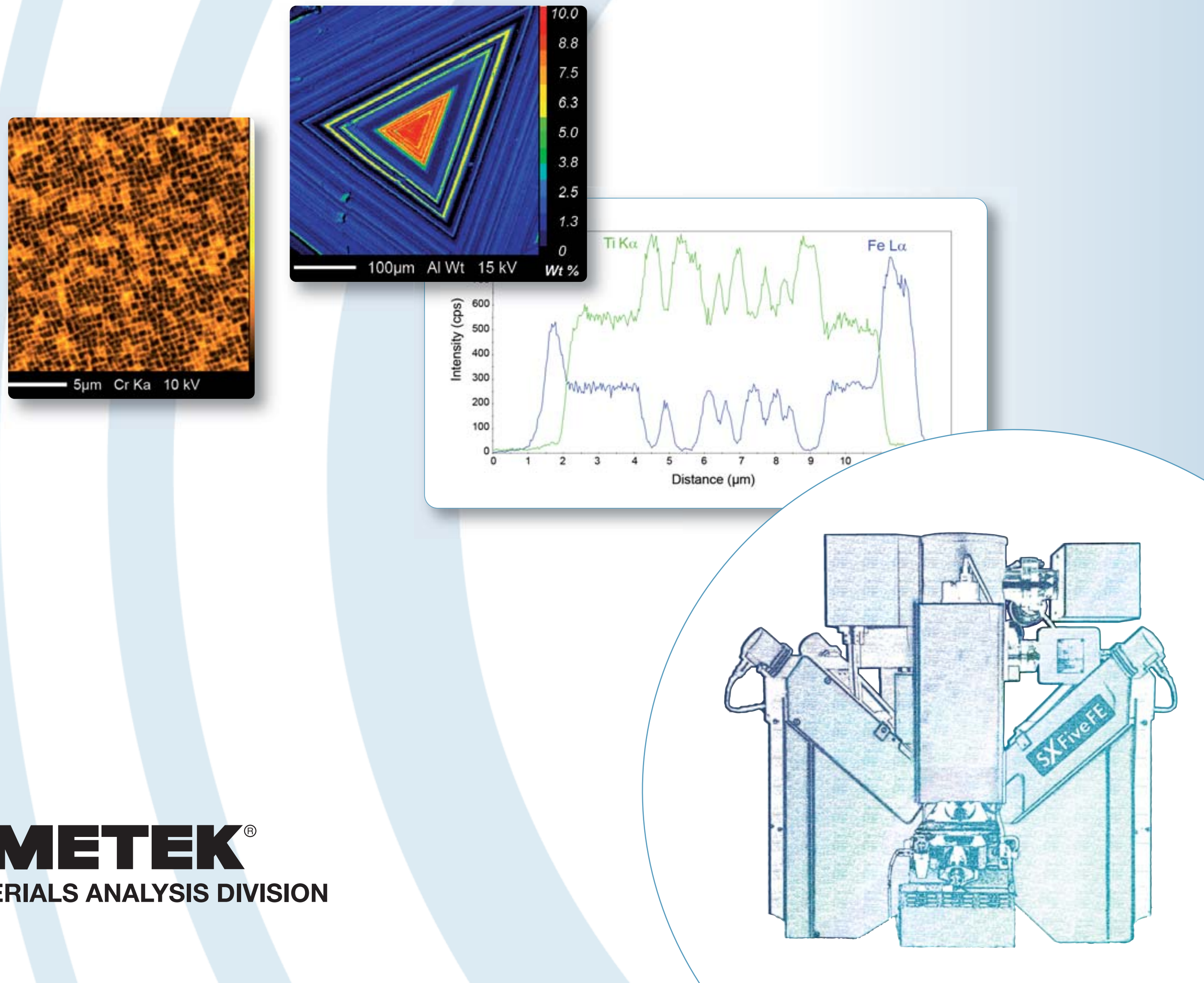
SCIENCE & METROLOGY SOLUTIONS

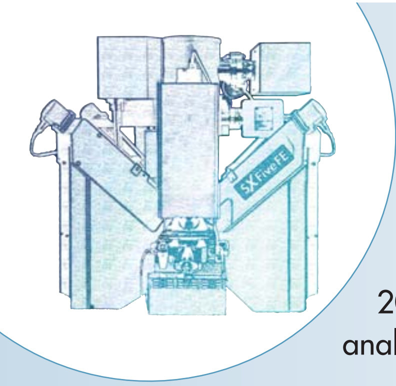
SXFiveFE

Application note

Quantitative analysis & X-ray mapping at high spatial resolution with Field Emission EPMA



MATERIALS ANALYSIS DIVISION



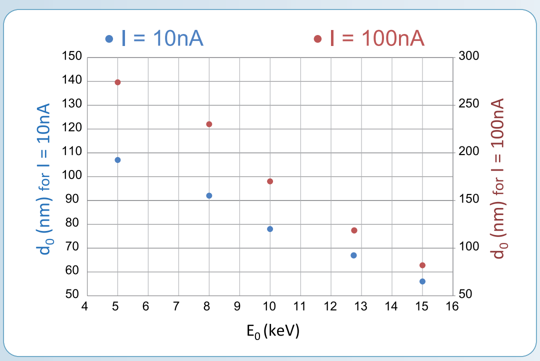
Thanks to its Field Emission source, the CAMECA **SXFiveFE** Electron Microprobe delivers a focused electron beam at **low voltage** and with **very stable current**. At low beam voltage, the penetration depth of the primary electrons, **and thus the interaction volume decrease to sub-µm scale**, compared to micron scale at 15 or 20 keV with conventional, non-FE EPMA. Spatial resolution of quantitative EPMA analyses is now greatly improved with the Field Emission **SXFiveFE** !

Improved Analytical Resolution

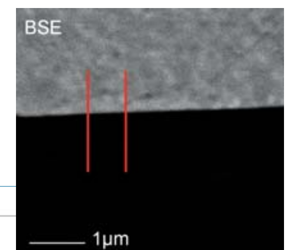
Analytical resolution (AR) is defined as the quadratic sum of the electron beam diameter and the diameter of the X-ray emission volume.

Influence of the electron beam diameter

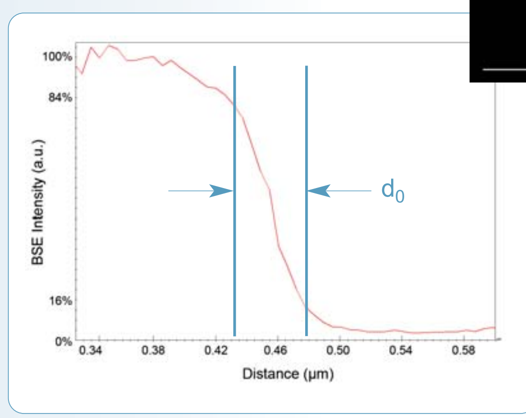
The diameter of the electron beam (d_0) is measured as the width at 16%-84% of the total signal amplitude from a reconstructed band scan across a sharp interface of a BSE picture. The electron density achieved with a FE source is greatly improved compared to that achieved with conventional W/LaB_6 sources. The graph below shows that at given electron impact energy, the lower the beam current, the smaller the electron beam diameter, **hence an improved analytical resolution.**



SXFiveFE: *beam diameter vs beam energy at 2 beam currents. As a comparison, d_0 = 600 nm at 15 keV and 100 nA with W filament.*



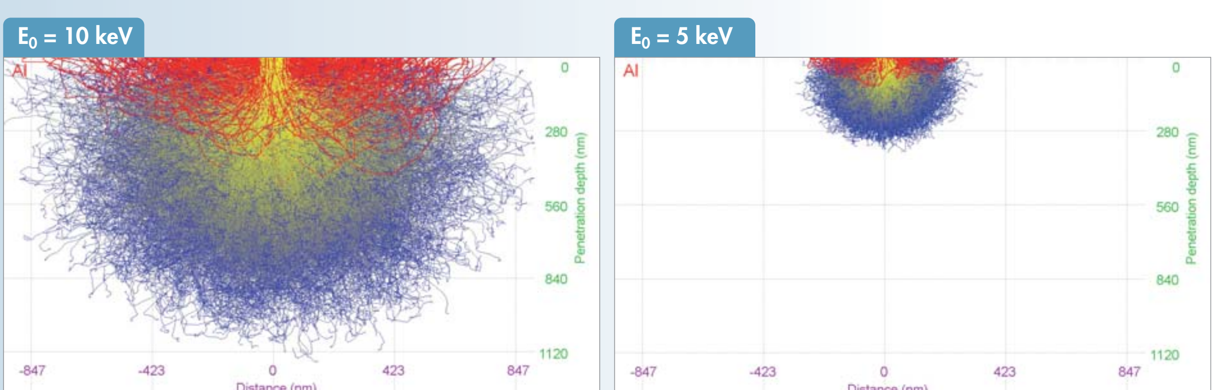
BSE picture of V/Fe interface acquired on a V, Cr, Fe superalloy



Example of a reconstructed band scan from above BSE picture.

Influence of the electron beam energy

At constant beam diameter, the lower the impact energy, the smaller the excitation volume, and the better the analytical resolution as shown on the below Monte Carlo simulations.

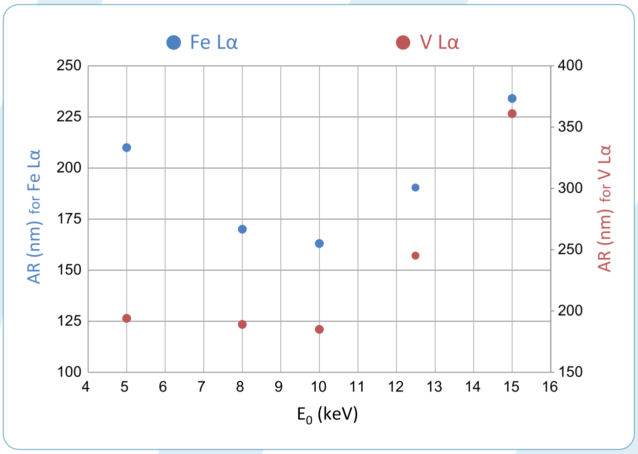


Simulations performed with CASINO program.

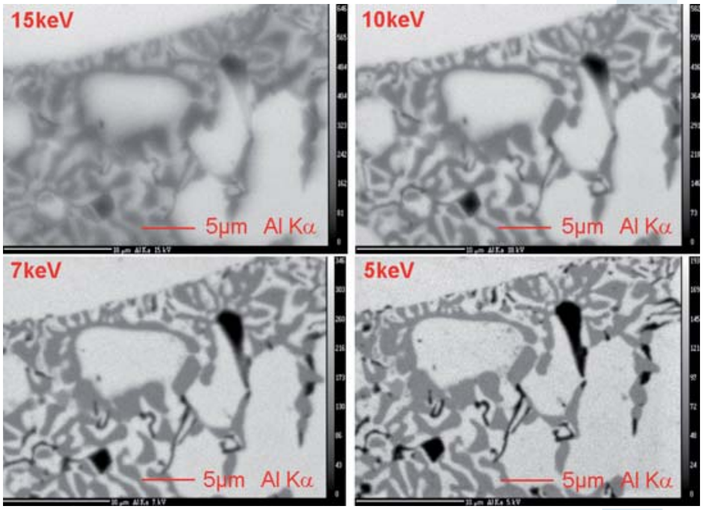
Trajectory of electrons in a Al matrix at 10 keV and 5 keV visualized by energy of electrons.

Optimum analytical resolution

For a given material, the improvement of analytical resolution (AR) requires the optimum balance between beam diameter and energy. The CAMECA **SXFiveFE** is setting new standard, as demonstrated on the right side graph for a V, Cr, Fe superalloy. The X-ray analytical resolution of Fe Lα and V Lα lines is measured at 16%-84% of the total signal amplitude from the band scan acquired across the interface at 10 nA for 5 impact energies: 15 keV, 12.5 keV, 10 keV, 8 keV and 5 keV. In this superalloy, the **best X-ray analytical resolution** is achieved at 10 keV primary electron energy.



Application



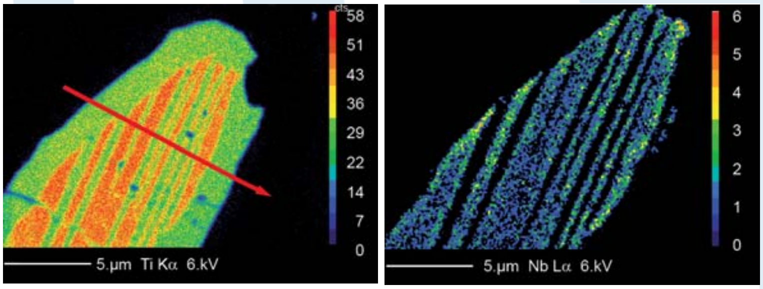
Metallurgy

The improvement of the analytical resolution as a function of impact energy is illustrated in the Al Kα X-ray maps of a brass specimen at progressively lower electron beam energies: 15 keV, 10 keV, 7 keV and 5 keV.

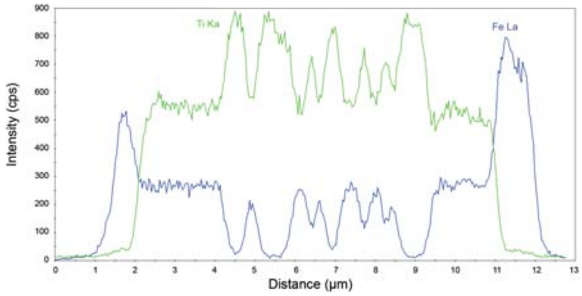
The highest analytical resolution is achieved for this speciment at 5 keV and a beam diameter of 80 nm. The Si metal phase (in black) is well resolved under these conditions, while it remains difficult to distinguish under conventional microanalysis column conditions of 10-15 keV.

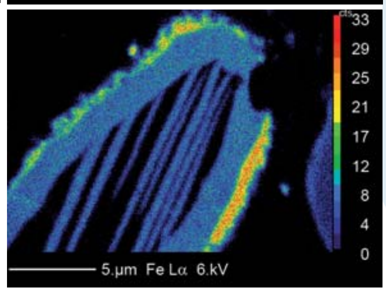
Geosciences

The **SXFiveFE** offers unequaled imaging capabilities at sub-micron spatial resolution as illustrated by the X-ray maps on the right. Shown here are Fe, Ti and Nb maps acquired at 6 keV beam voltage and 50 nA beam current in a composite Fe-Ti oxide grain from a metasomatized peridotite xenolith.



An **optimum X-ray analytical resolution of 310 nm** is achieved for the Ti Kα line measured at 16%-84% of the total signal amplitude from the line scan acquired across the sample (red line on Ti Kα image).





Sample courtesy of F. Kalfoun, D. Ionov, C. Merlet.

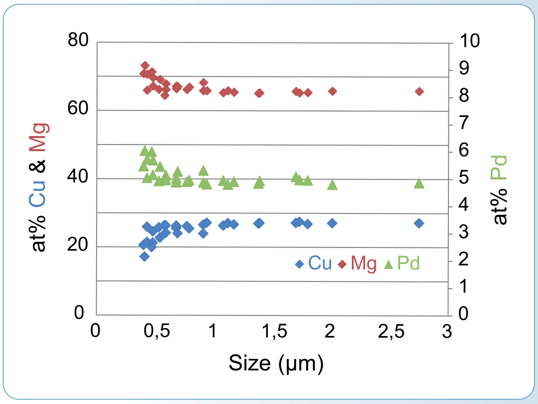
CAMECA's SXFiveFE is the EPMA of reference for quantitative analysis and X-ray imaging with sub-micron spatial resolution.

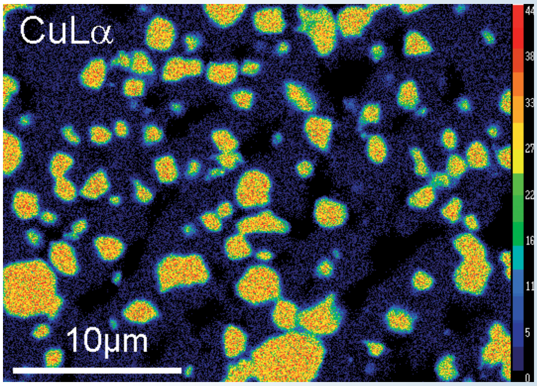
Thanks to its Wavelength Dispersive Spectrometers with a long acclaimed reputation for high reproducibility, high sensitivity and high spectral energy resolution, our **SX FiveFE** electron microprobe delivers **quantitative analysis with benchmark accuracy**.

Accurate quantitative analysis of **small phases** is possible when working **at low electron beam energy** as illlustrated on the right side graph, showing measured composition of precipitates ranging between 0.3 µm and 2.5 µm.

The specimen of interest exhibits various sizes of Cu, Mg, and Pd precipitates in a matrix of Mg-Pd. For sizes above 0.6 µm in diameter, concentrations of Mg, Cu and Pd are stable, indicating that the X-rays are coming only from the phase. Below 0.6 µm in diameter, signals of Pd and Mg increase while the Cu signal decreases indicating that the analyzed volume becomes larger than the precipitate itself.

The **X-ray map** of Cu acquired on this specimen **at 6 keV** beam energy shows a clear **sub-micron spatial resolution** together with **reliable precipitate composition down to 0.6 µm in diameter**.





Sample courtesy of Dr Eric Leroy, CNRS ICMPE Thiais, France

CAMECA is the reference in micro- and nano-analysis, providing cutting-edge science and metrology solutions with unequaled customer support through a worldwide network of subsidiaries and agents.

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