



XFlash® 7 – The New EDS Detector Series

BRUKER NANO ANALYTICS

XFlash® 7 - The EDS Detector
for SEM STEM and FIB-SEM

XFlash® 7
Unique 4-segment annular EDS detector XFlash®
FlatQUAD

Présentation de la société



SEMPA 2024 ENSICAEN/CIMAP

SYNERGIE⁴, c'est le service avant tout...

Produits

1994-2024 30 ans !

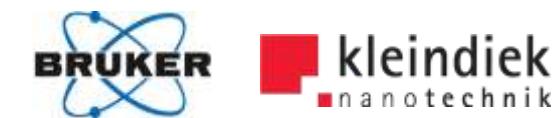
Microscopie Electronique à Balayage de table



Préparation d'échantillons



Microanalyse et analyse *In-Situ*



Spectrométrie par fluorescence X



Nano et Micro-tomographie à rayons X



Synergie⁴, c'est le Service avant tout

Présentation de la société

Produits

MEB de table EDS intégré



1 à 30kV résolution 5nm

SE /BSE/EBSD

HV / LV

Platine motorisée en 5 axes

EDS intégré



Préparation d'échantillons



Métalliseurs et
évaporateurs carbone



Préparation mécanique,
polissage ionique



Présentation de la société

Produits

Microanalyse et analyse *In-Situ*



EDS (Energy-Dispersive x-ray Spectroscopy)

WDS (Wavelength Dispersive Spectroscopy)

EBSDD (Electron Backscatter Diffraction)

μ -XRF (Micro X-Ray Fluorescence)

Analyse In-Situ par pico-indentation

Sondes et micromanipulateurs



EDS Quantax



WDS XSENSE



μ -XRF XTrace



Pico-Indenteur
Hysitron pour MEB et
MET



Sondes
Micromanipulateurs

Présentation de la société

Produits

Spectrométrie par fluorescence X



Fluorescence X (XRF)



M6 JetStream

Micro-fluorescence X (μ XRF)



M4 Tornado

Fluorescence X en totale réflexion (TXRF)



S4 T-STAR



Elio



Système de table et transportable par TXRF Analyse élémentaire des Ultra Traces

Produits



Lab Report XRF 458

S2 PICOFOX Rapid and cost-effective monitoring of sludge and wastewater by TXRF spectroscopy

International standards define analytical methods for the determination of dissolved and particulate bound elements in water (e.g. ground, surface, fresh, drinking and wastewater) [1]. Although, total reflection X-ray fluorescence analysis (TXRF) is not approved yet for wastewater monitoring.

Procedures for the quantitative analysis of Hg or As, Cd, Cr, Ni, Pb, and other elements in wastewater were described recently and their results were compared with ICP-OES/MS data [2, 3]. This lab report demonstrates the use of TXRF spectroscopy for trace element analysis of synthetic wastewater samples and sludges from industrial processes. A special focus is set on the analysis of thallium (Tl), which typically fails when using ICP-OES.



Fig. 1: Outflow pipe for wastewater containing sludge - a nightmare for analytical chemists

TXRF

S2 PICOFOX

Clean Water Package (CWP)

The S2 PICOFOX Clean Water Package (CWP) is an attractive offer for on-site analysis campaigns of fresh water, wastewater and effluents. It is supplied with the wastewater reference standard SPS-WW2 already prepared on quartz discs.

The S2 PICOFOX is a compact, portable benchtop TXRF spectrometer for simultaneous detection of ultra-trace element in various samples. The spectrometer provides cost-effective on-site screening of a wide range of samples and a subsequent selection of samples for laboratory analyses.

The S2 PICOFOX requires no media and can be easily transported to remote sites to screen any environmental sample for trace elements. This helps to reduce the number of samples to be measured in the laboratory and thus minimizes the amount of hazardous chemicals and CO₂ released during power generation.

CWP specifications

- S2 PICOFOX spectrometer equipped with automatic sample changer
- Capacity of 25 sample discs
- Line focus X-ray tube with Mo target
- 60 mm² XFlash® silicon drift detector
- Wastewater standard SPS-WW2 prepared on three quartz discs
- Wastewater standard operation procedure (SOP) including calibration
- Notebook PC
- TXRF accessories

Typical power consumption for the spectrometer and PC is about 220 W. For on-site campaigns without power supply the use of a portable power station with a minimal capacity of 750 Wh and at least two power sockets is recommended.



Innovation with Integrity

Présentation de la société

Produits

Nano et
Micro-tomographie
à rayons X



Sciences des Matériaux
et de la terre
(AXS)

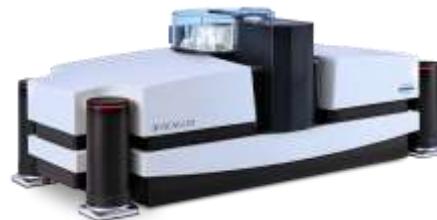
Sciences de la vie (PCI)



SkyScan 2214



SkyScan 1273



SkyScan 1272



SkyScan 1275

Ex-Vivo



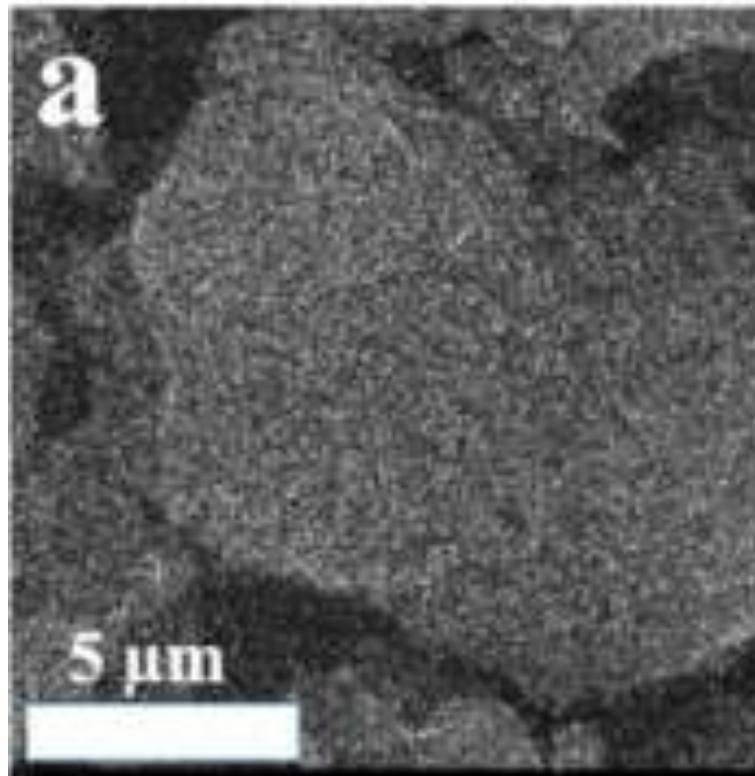
SkyScan 1276



SkyScan 1278

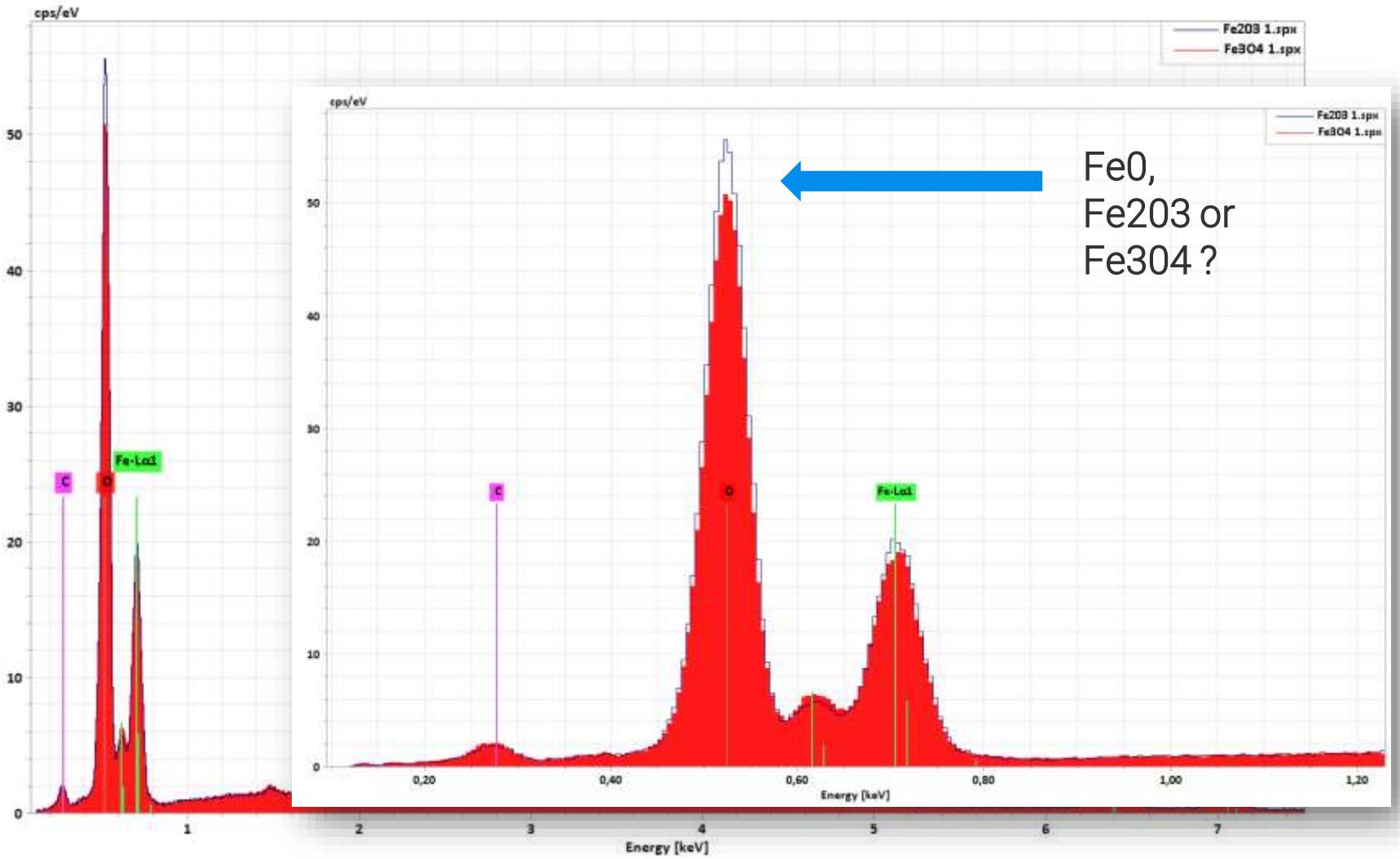
In-Vivo

XFlash® 7 – The best solid angle with lower surfaces



1980- Analog X-ray mapping with pulses

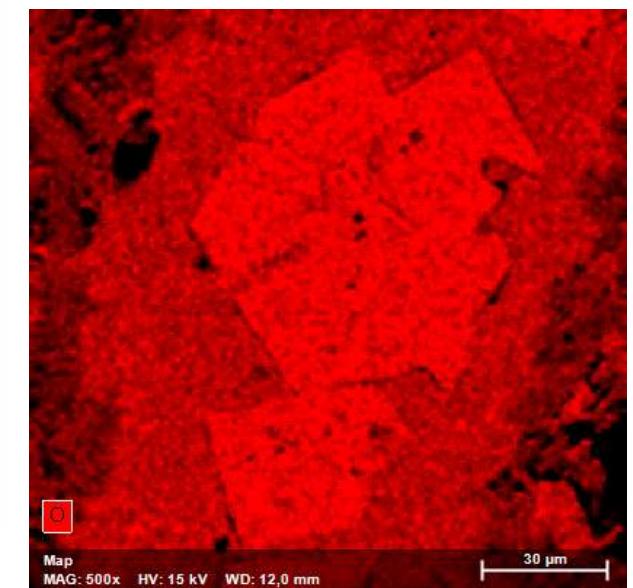
XFlash® 7 – The best solid angle with lower surfaces



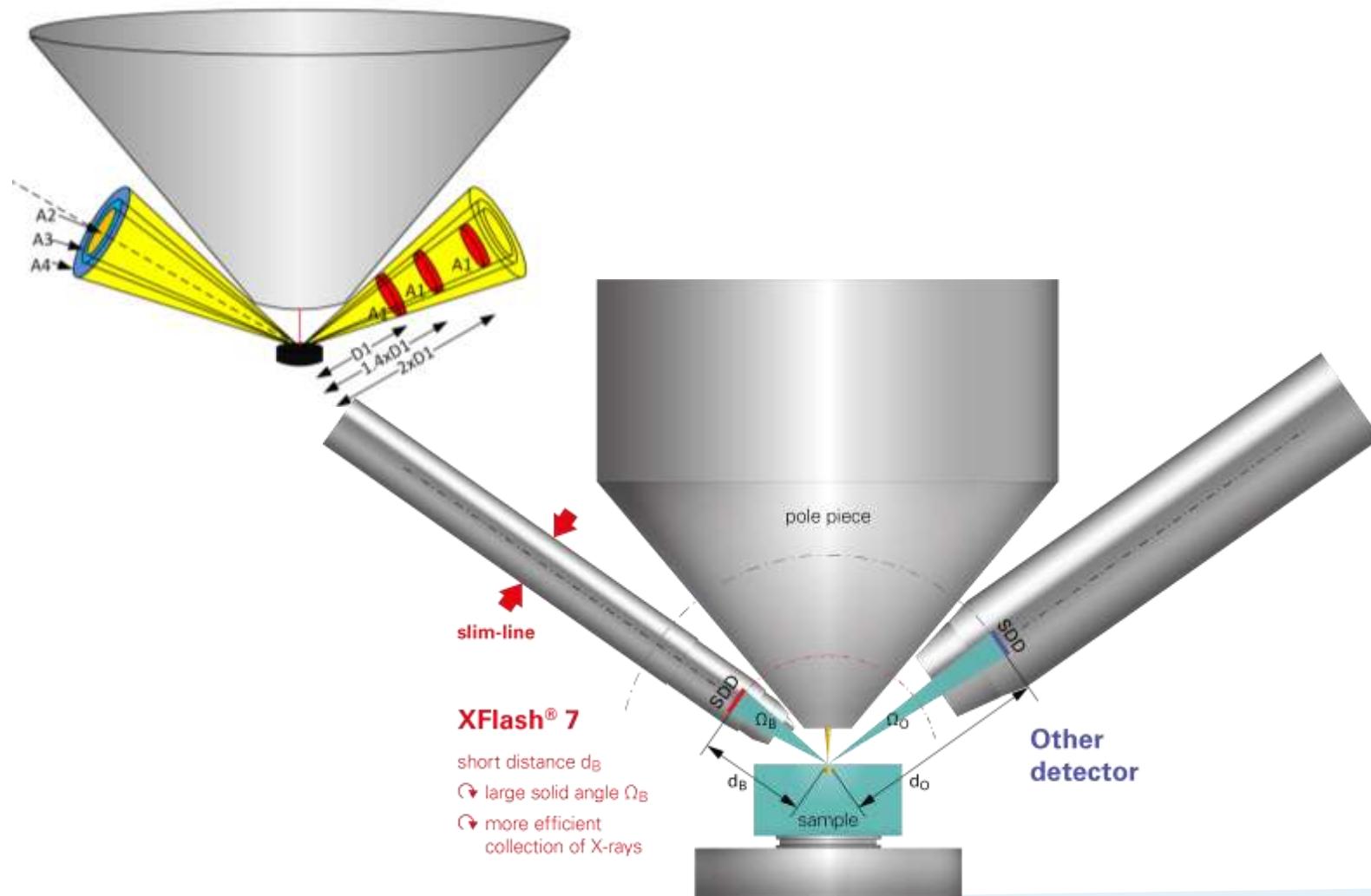
We need counts for accurate identification, quantification and fast mapping.

5 sec / 100 000 cps
= 500 000 counts

100 by 100 pixels
= 10000 pixels
1sec/pixel=3 heures



XFlash® 7 – The best solid angle with lower surfaces



Solid angle (also called collection angle)

- X-rays radiate in all directions.
- Simply put: the solid angle is the portion of a hemisphere above or below a sample that the detector sees.
- The larger the solid angle of a detector, the more sample radiation is collected.
- There are basically two methods to improve solid angle:
 - Increase the detector chip size – this leads to a linear increase of solid angle (left side).
 - Decrease the detector sample distance – this squares the solid angle when the distance is halved (right side).
- Bruker uses both methods to provide best solid angle in relation to detector chip area.

XFlash® 7 – The right solid angle for better analysis

Fast. Precise. Reliable.

XFlash® 7 - detectors 30 - 60 and 100mm²
for SEM and FIB-SEM (fully motorized)



XFlash® 7 for SEM – Key facts

New Electronic Up to 1,000,000 cps

Real analytical throughput

Achieve unmatched analysis speed

> 2,200

Element lines

Quantify complex data using the most comprehensive atomic database incl. K, L, M and N lines



> 1.1 sr (FQ)

Largest solid angle for X-ray collection

Maximize sample throughput with optimum geometry for most efficient collection of the generated X-rays

XFlash® 7 – Benefits

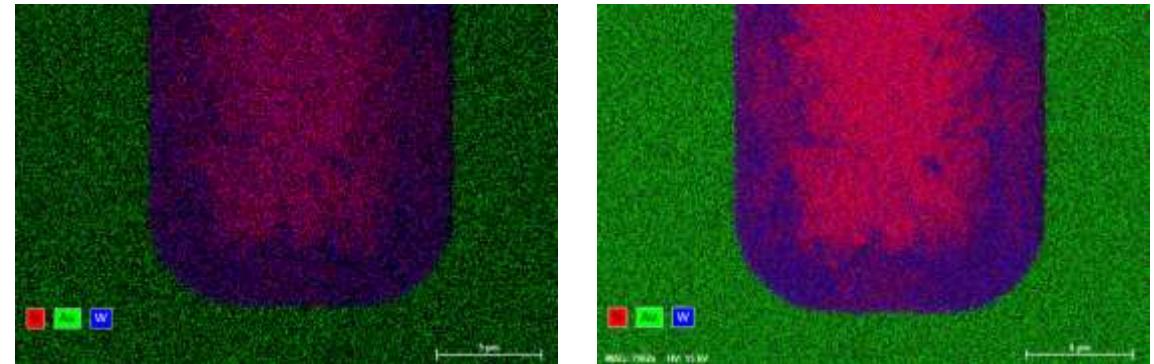
Make your element analysis more efficient

- Individually optimized EDS systems to provide unmatched speed and precision.
- Shorten measurement time with maximized throughput.
- The most efficient geometry for collection of generated X-rays to analyze challenging samples.
- Benefit from accurate and reliable quantification results with optimized geometry.
- Detect smaller quantities of matter thanks to better detection limits, lower background and less absorption.



XFlash® 7 – Be faster!

- Get best results in the shortest time
- No risk of signal loss caused by slow read-out electronics
- Maximize your sample throughput without compromising quality
- Avoid tweaking microscope parameters
- Acquire quantitative EDS data at any speed



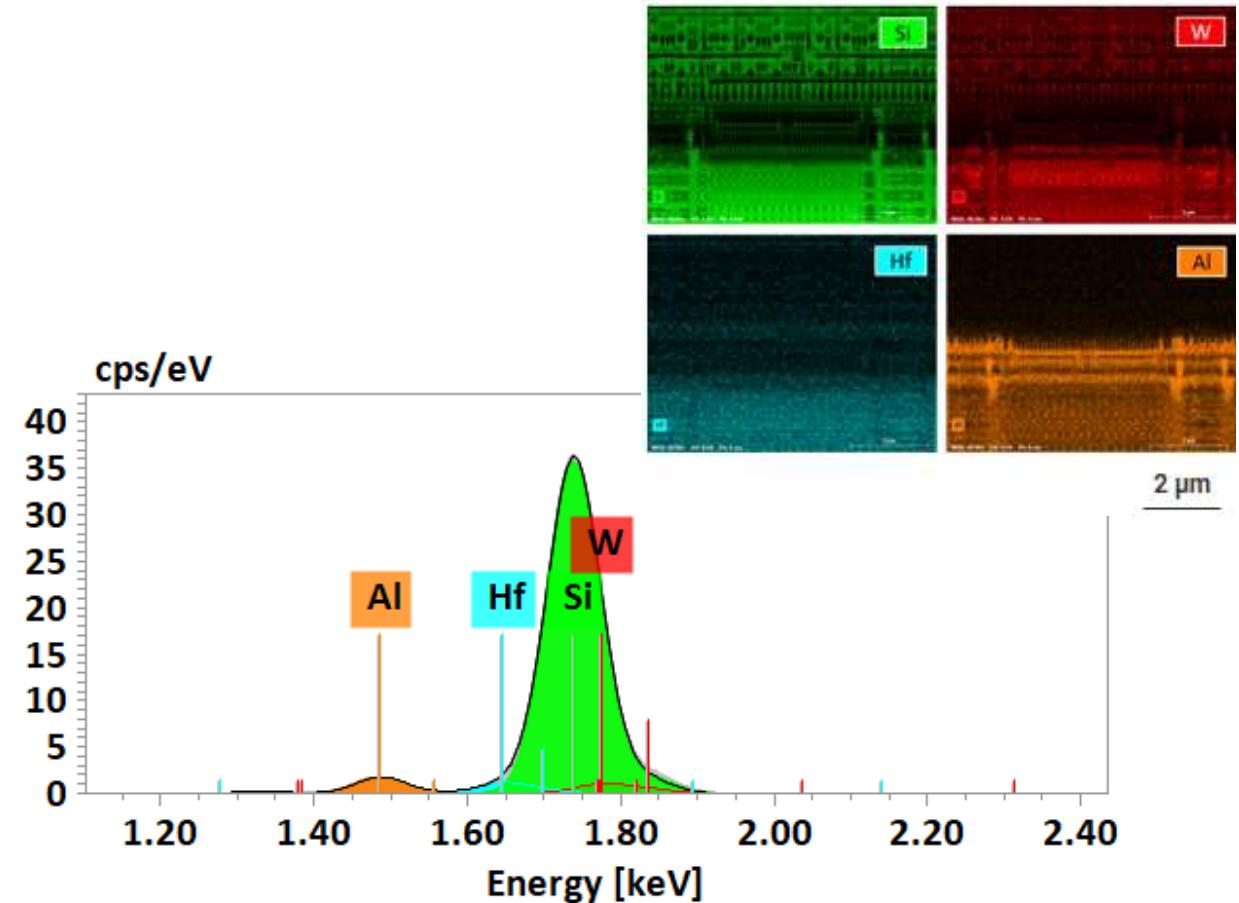
Element distribution maps based on deconvolved net intensities of Si-K and W-M lines at 15 kV.

| Measurement conditions | Left | Right |
|------------------------|-----------------------|---------------|
| Detector | Conventional detector | XFlash® 760 |
| Measurement time | 7 s (1 frame) | 7 s (1 frame) |
| Input count rate | 63 kcps | 700 kcps |
| Dead time | 18 % | 38 % |

XFlash® 7 – Get precise results!

The most **comprehensive atomic database** including more than **2,200 element lines** leads to an unrivaled quality in ESPRIT peak ID and separation, meaning:

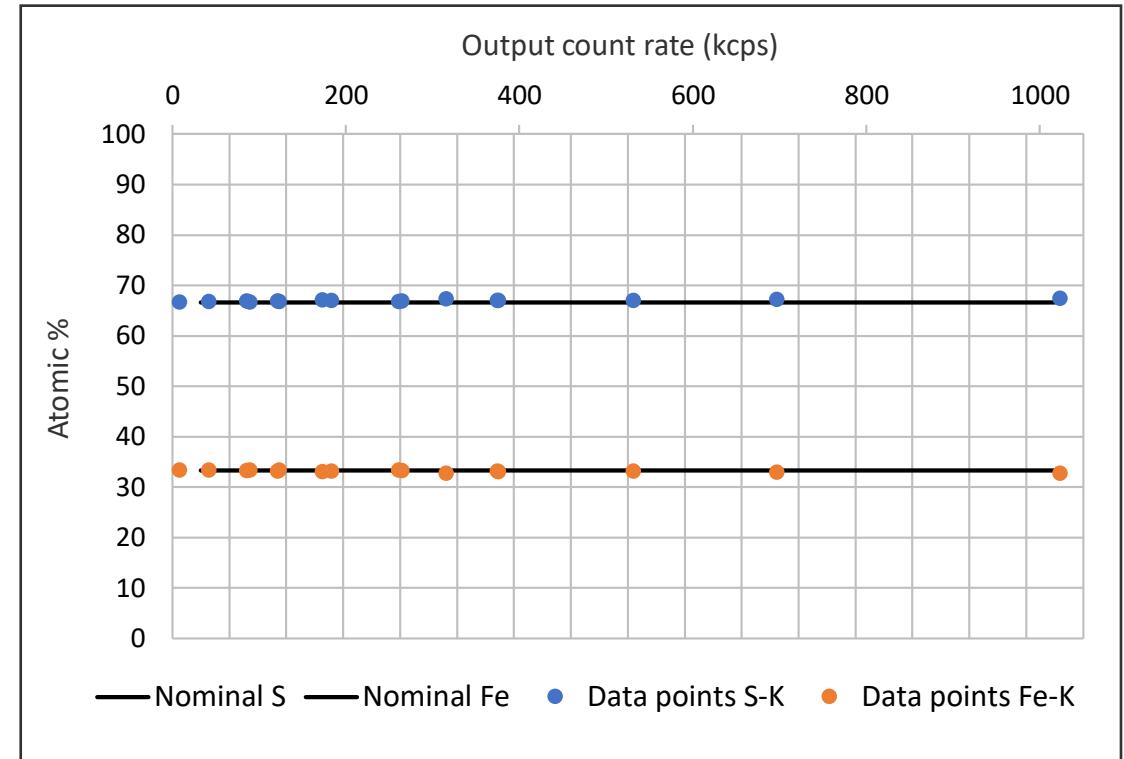
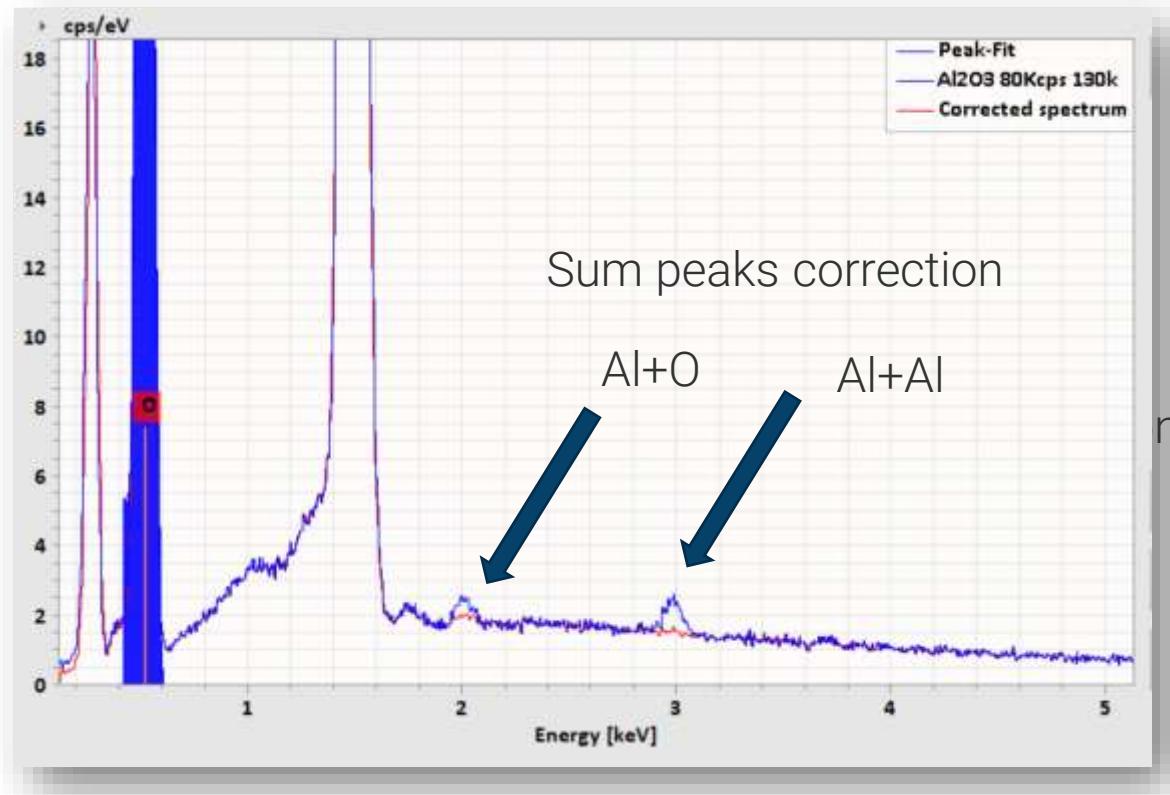
- Advanced peak deconvolution algorithms enables online (live, during acquisition), and offline visualization of overlapping elements
- The combination of good resolution with best deconvolution means even close overlaps can be resolved with confidence
- Best quantification results also for challenging samples using low accelerating voltages



Automatically deconvolved heavily overlapped lines.

XFlash® 7 – Ensure reliability!

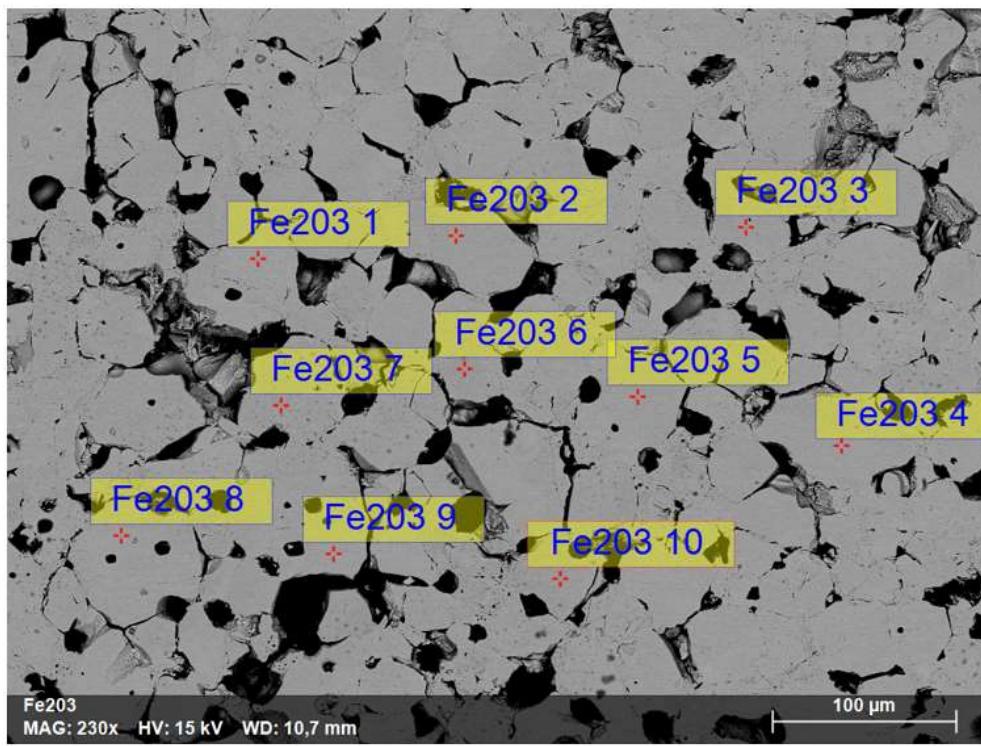
Detector stability is the basis for data acquisition and reliable quantification results regardless of microscope settings and measurement conditions, meaning:



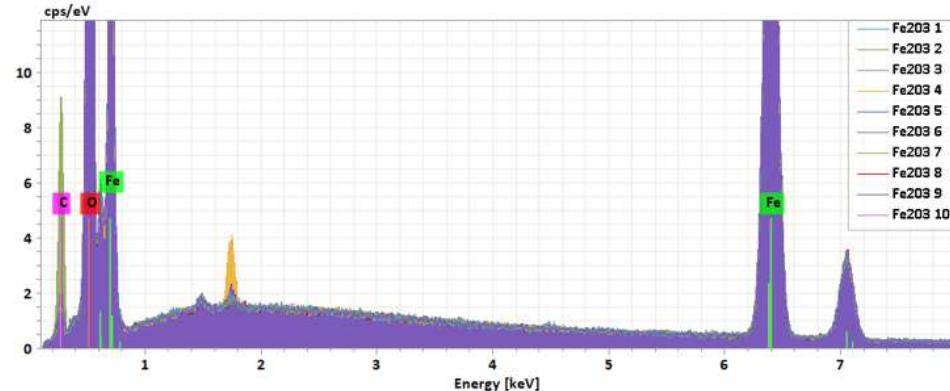
Fe and S quantification results of stoichiometric FeS₂ at different output count rates (OCR)

Echantillon 1 : Analyse quantitative
Hématite Fe₂O₃

Image BSE et pointés d'analyse:
500 000 coups/spectre, 2 nAmp, 100 000 cps (5s)

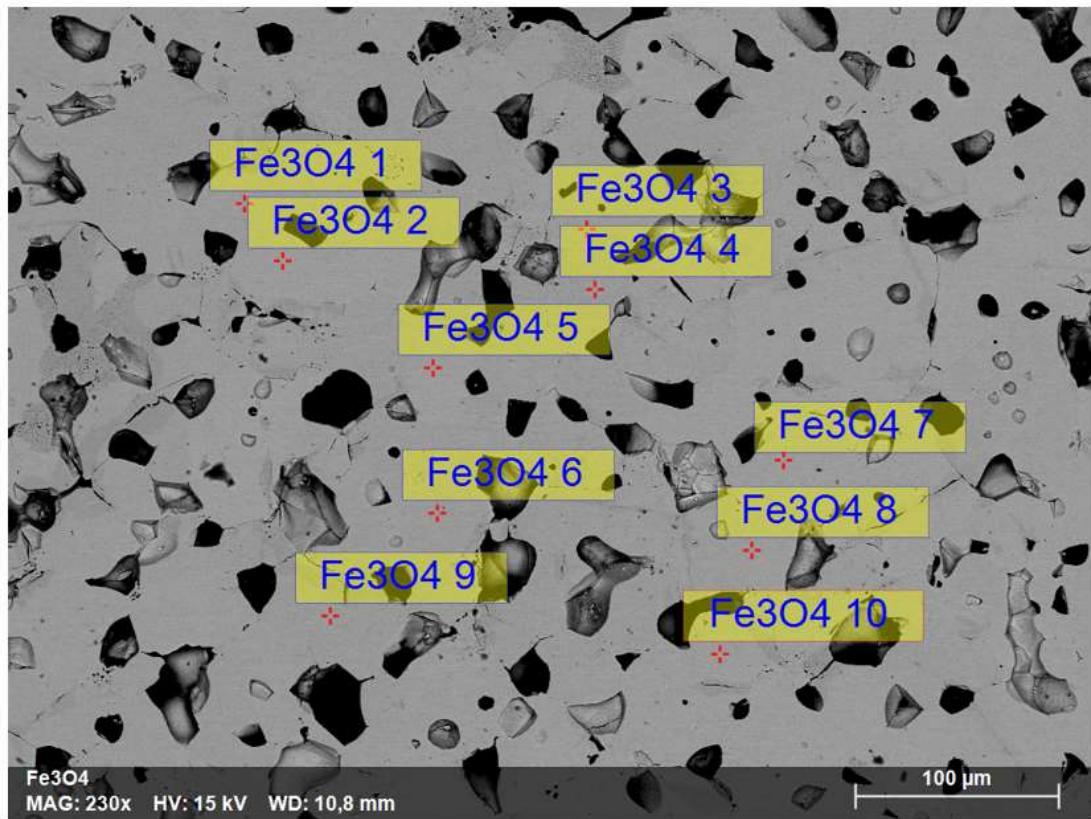


Analyses quantitatives PhiRoZ avec témoins

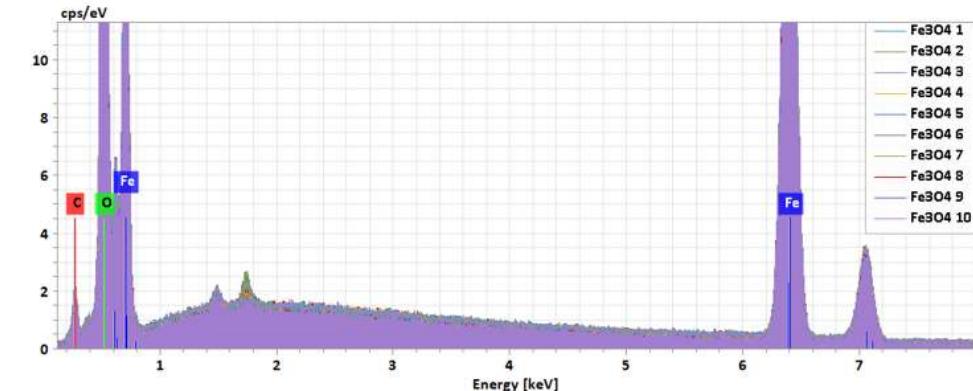


Echantillon 1: Analyse quantitative
Magnétite Fe₃O₄

Image BSE et pointés d'analyse:
500 000 coups/spectre, 2 nAmp, 100 000 cps (5s)



Analyses quantitatives PhiRoZ avec témoins



WT% non-normalisés

| Spectrum | Carbon | Oxygen | Iron | Sum |
|-----------|--------|--------|-------|--------|
| Fe3O4 1 | 0,00 | 27,69 | 71,94 | 99,63 |
| Fe3O4 2 | 0,00 | 27,60 | 71,04 | 98,64 |
| Fe3O4 3 | 0,00 | 27,95 | 72,35 | 100,30 |
| Fe3O4 4 | 0,00 | 27,84 | 72,43 | 100,28 |
| Fe3O4 5 | 0,00 | 27,55 | 71,25 | 98,80 |
| Fe3O4 6 | 0,00 | 27,65 | 71,93 | 99,58 |
| Fe3O4 7 | 0,00 | 28,24 | 72,31 | 100,55 |
| Fe3O4 8 | 0,00 | 27,75 | 72,28 | 100,02 |
| Fe3O4 9 | 0,00 | 27,48 | 71,71 | 99,19 |
| Fe3O4 10 | 0,00 | 27,72 | 72,44 | 100,16 |
| Mean | 0,00 | 27,75 | 71,97 | 99,71 |
| Sigma | 0,00 | 0,22 | 0,50 | 0,66 |
| SigmaMean | 0,00 | 0,07 | 0,16 | 0,21 |

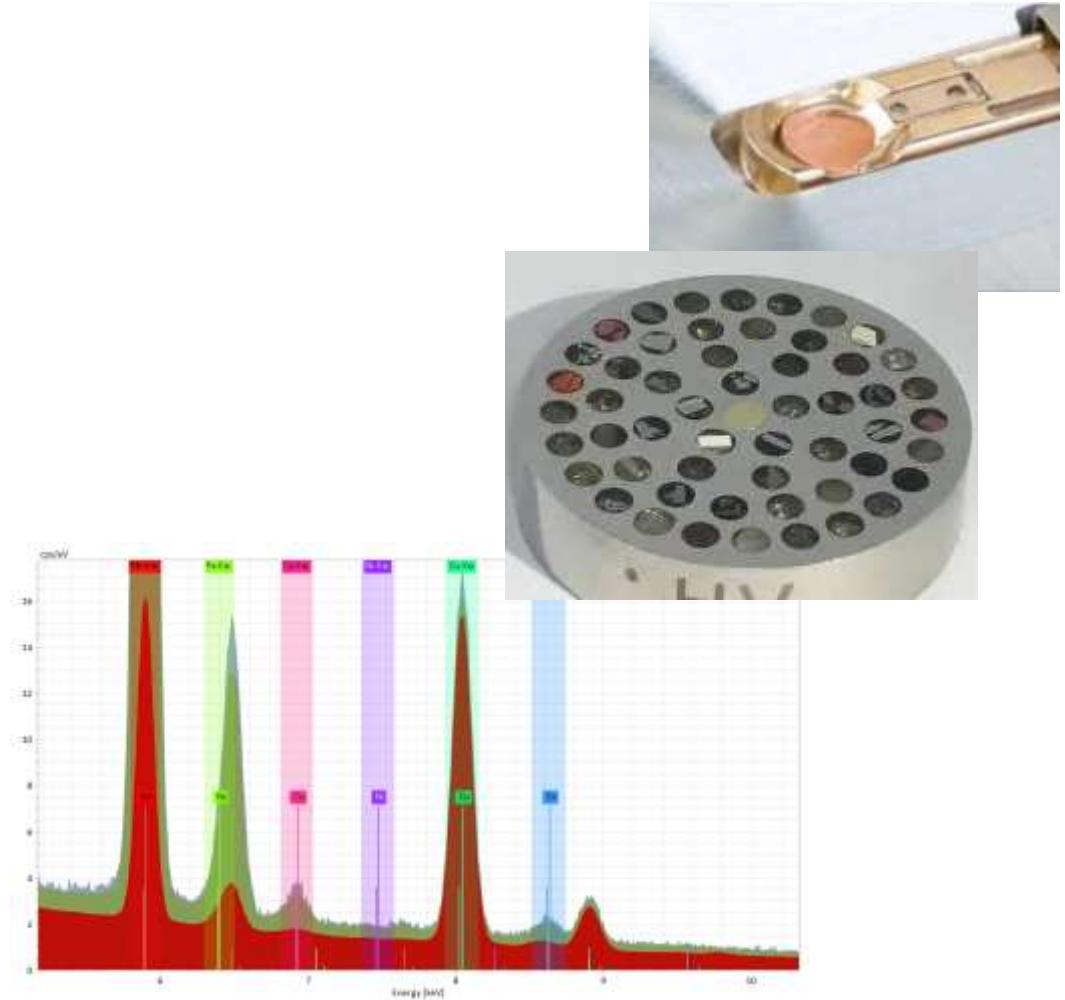
Atom% normalisés

| Spectrum | Carbon | Oxygen | Iron |
|-----------|--------|--------|-------|
| Fe3O4 1 | 0,00 | 57,33 | 42,67 |
| Fe3O4 2 | 0,00 | 57,56 | 42,44 |
| Fe3O4 3 | 0,00 | 57,41 | 42,59 |
| Fe3O4 4 | 0,00 | 57,30 | 42,70 |
| Fe3O4 5 | 0,00 | 57,44 | 42,56 |
| Fe3O4 6 | 0,00 | 57,29 | 42,71 |
| Fe3O4 7 | 0,00 | 57,68 | 42,32 |
| Fe3O4 8 | 0,00 | 57,26 | 42,74 |
| Fe3O4 9 | 0,00 | 57,22 | 42,78 |
| Fe3O4 10 | 0,00 | 57,18 | 42,82 |
| Mean | 0,00 | 57,37 | 42,63 |
| Sigma | 0,00 | 0,16 | 0,16 |
| SigmaMean | 0,00 | 0,05 | 0,05 |

ESPRIT Quant Plus

Full user control when needed

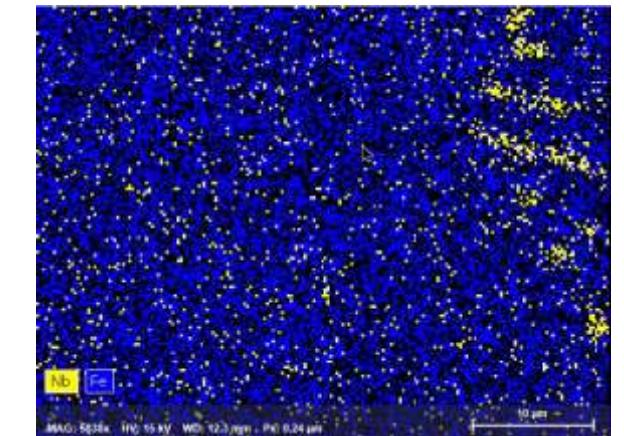
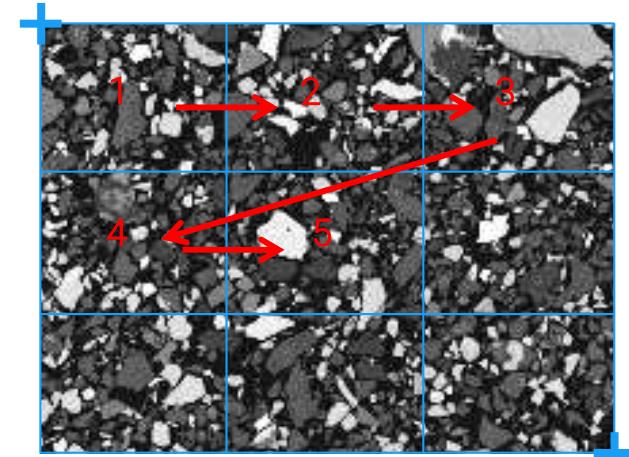
- Upgrade your ESPRIT software suite with advanced quantification tools to solve demanding tasks
- Features:
 - Quantitative line scans and element mappings
 - Standard-based quantification using given or own standards
 - Quantification of electron transparent samples using Cliff-Lorimer or Zeta factor methods
 - Maximum Pixel Spectrum to identify traces of elements



ESPRIT Image Plus

Optimize your information output

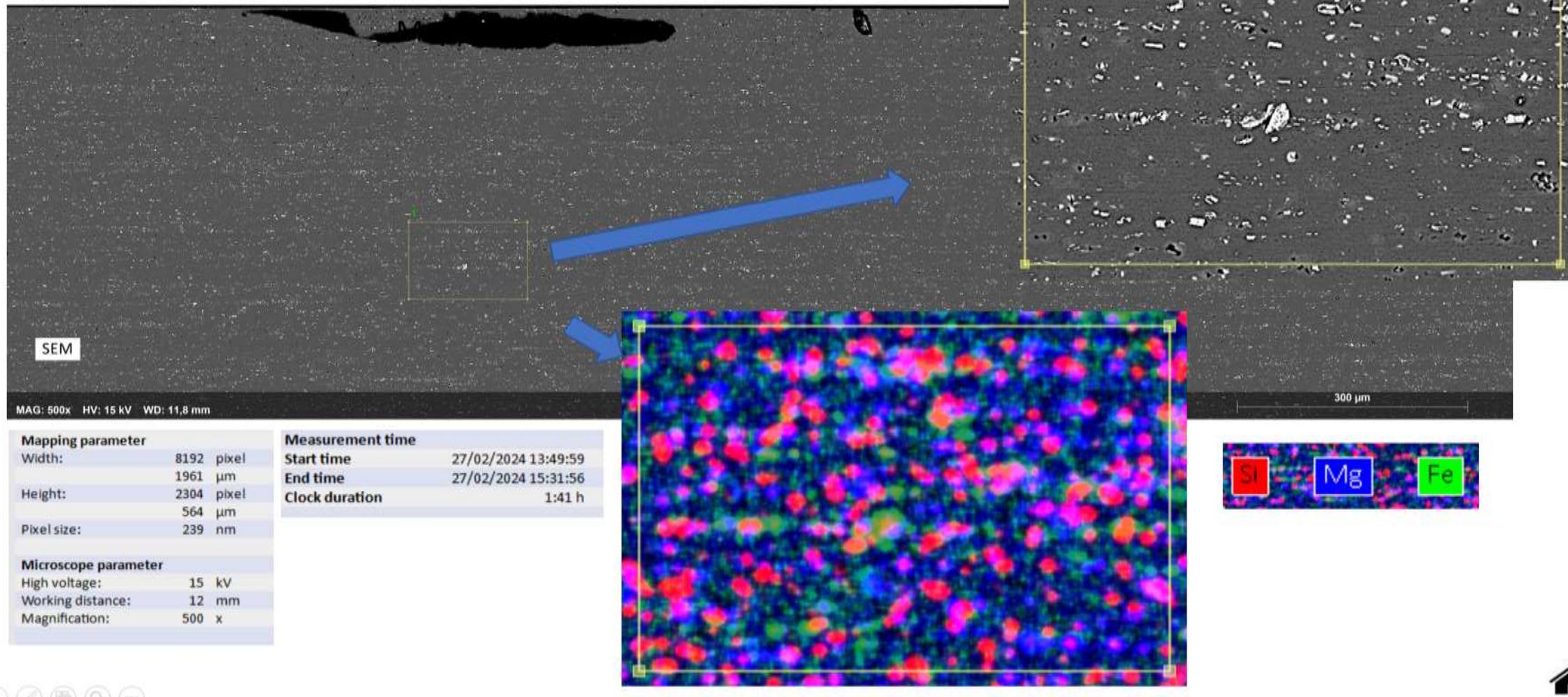
- Upgrade your ESPRIT Software Suite with the most advanced Imaging tools to visualize hidden features of your sample and extend the field of view independent of the SEM scan range
- Features
 - Image processing tools such as ESPRIT Vision and ColorSEM
 - Automatic drift correction
 - Automatic chemical Phase Analysis
 - Extended field of interest using Stage scan also in combination with scanning the electron beam to analyze large sample areas in cm range
 - ESPRIT LiveMap ([see YouTube example](#)): visualize the element distribution in real time while driving the microscope stage to search live for sample areas of interest



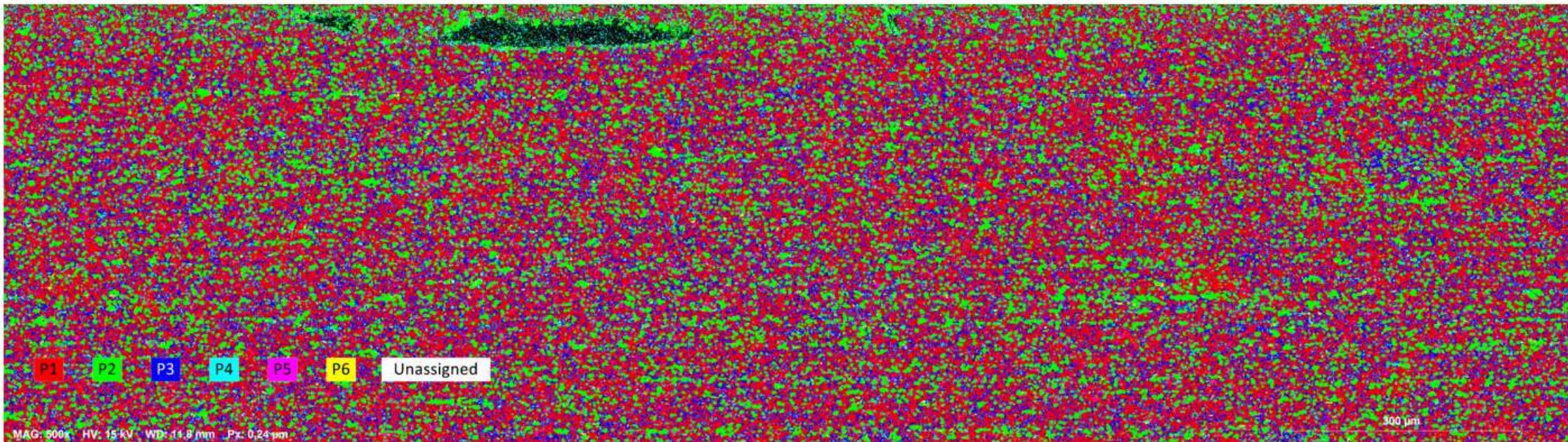
ESPRIT Image Plus Large Area Mapping

Alliage Aluminium alloy

Mosaïc Mapping (24 images 1024x760 pixels)



ESPRIT Image Plus Automatic chemical Phase Analysis

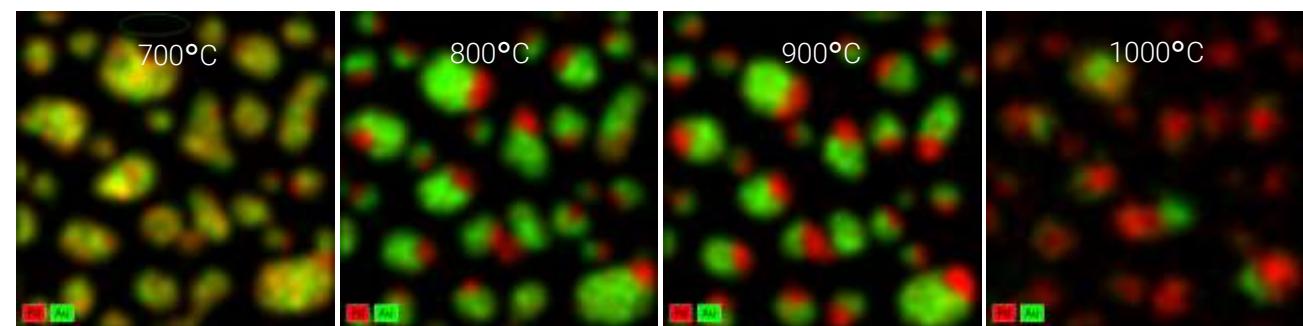
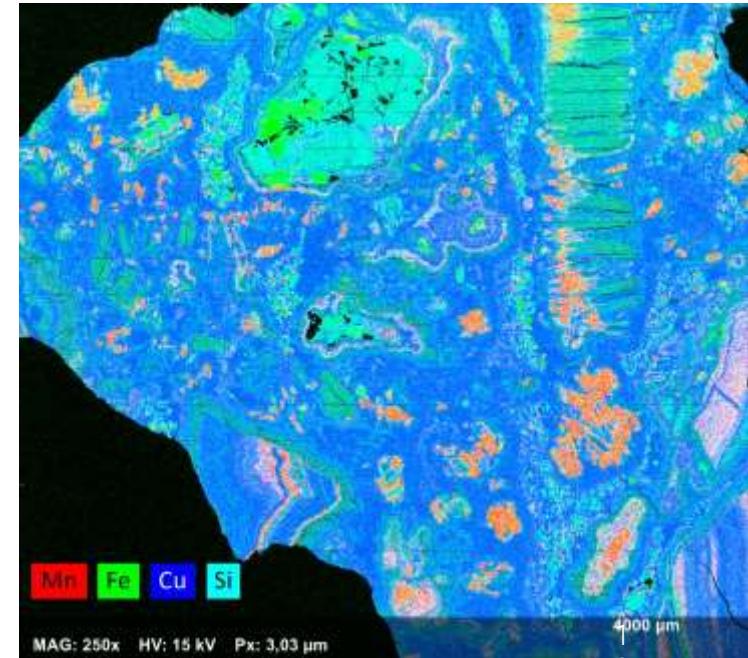


| <u>Counts/Pixel</u> | <u>Area</u> | <u>SEM</u> | <u>Si-Kα</u> | <u>Mg-Kα</u> | <u>Fe-Kα</u> | <u>Al-K</u> |
|---------------------|-------------|------------|--------------------------------|--------------------------------|--------------------------------|-------------|
| ■ P1 | 46,0 % | -- | 14,99 | 39,67 | 1,13 | -- |
| ■ P2 | 21,0 % | -- | 239,02 | 53,91 | 5,83 | -- |
| ■ P3 | 20,3 % | -- | 15,13 | 28,37 | 10,38 | -- |
| ■ P4 | 5,8 % | -- | 15,70 | 9,97 | 16,10 | -- |
| ■ P5 | 4,6 % | -- | 51,39 | 60,60 | 3,83 | -- |
| ■ P6 | 1,3 % | -- | 46,10 | 20,43 | 18,72 | -- |
| □ Unassigned | 0,1 % | -- | 44,82 | 6,72 | 13,24 | -- |

ESPRIT Automation Plus

Enhance your productivity with automated measurements

- Adding the ESPRIT Automatic Job Control allows unattended measurements and sample analysis independent of time and sample area
- ESPRIT Automation includes ESPRIT Feature – the automatic particle analysis combining morphological and chemical information to classify any type of particles
- The unique TRM software module allowing Time Resolve Mappings for in-situ analysis such as heating experiments tensile tests in SEM and STEM also in combination with EBSD



Au/Pd sputtered on a silicon-nitride membrane, 700 °C → 1000 °C

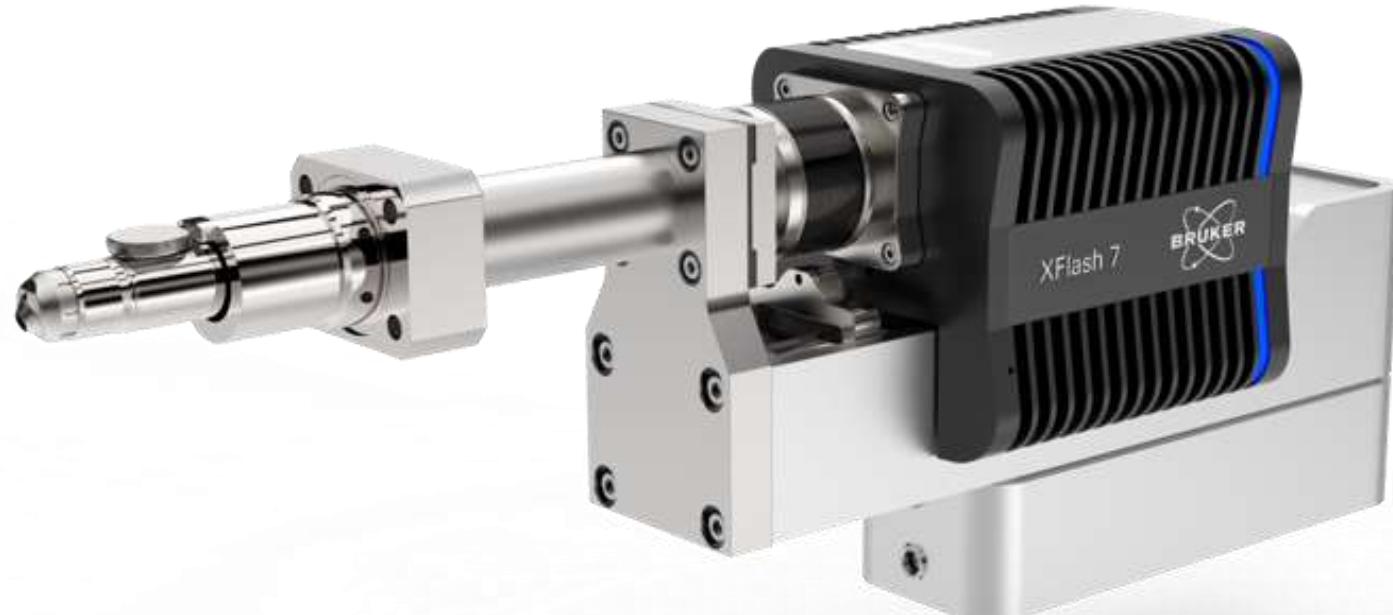


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XFlash® 7T - The EDS Detector for TEM and STEM

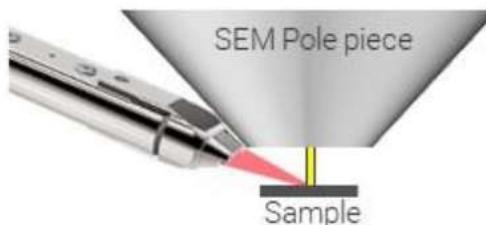
XFlash® 7 – The right angle for better analysis

Fast. Precise. Reliable.



XFlash® 7T - the detector
for TEM and STEM

XFlash® 7 – Racetrack Windowless for STEM and SEM-STEM



XFlash ® Oval 100mm²



- Oval shaped SDD chip geometry
- High solid angle of 0.4 sr
- High collection efficiency
- High take-off angle of 35°
- Ultra-high spatial resolution – high sensitivity
- Excellent spectral quality at low and high kV
- Light element, low kV - low probe current analysis
- TEM-like EDS measurements in SEM
- Fast data processing (up to 600,000 cps output count rate)

XFlash® 7T for TEM – Key facts



1 Å

Stable resolution

Map periodic structures (atoms, layers) with high stability using enhanced drift correction features

80 keV

Unprecedented upper energy limit

Unequivocally identify and quantify all present elements

3 TEM-Quantification models

Succeed in TEM, STEM and T-SEM with easy-to-use powerful quantification based on theoretical and experimental Cliff-Lorimer factors as well as Zeta-factor interpolation

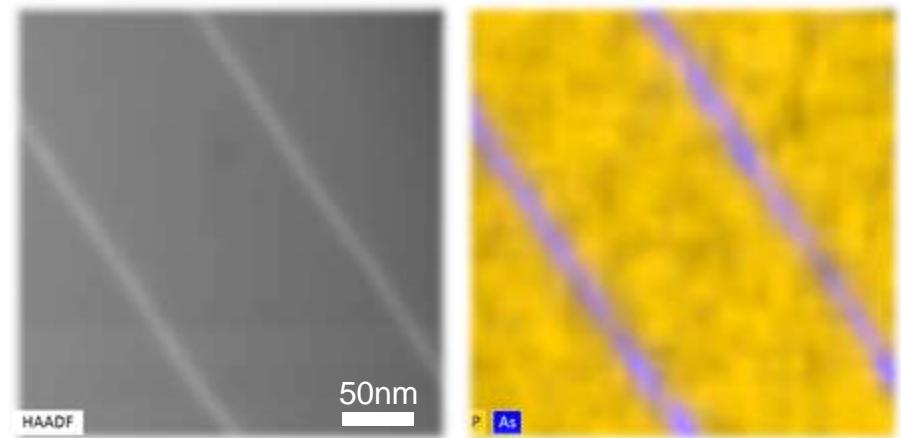
XFlash® 7T – Benefits

- Fast acquisition of precise and reliable data thanks to
 - High count rate at minimized absorption and shadowing effects, rarely need to tilt
 - Easy to understand user interface: you see what you do!
 - Comprehensive data mining during and after data acquisition
 - Unequivocal element ID relying on element lines up to 80 kV
 - High spatial resolution with drift correction routines adapted for periodic including atomic structures
 - Fast-moving stable detector stage
 - Minimized mechanical and electromagnetic interference
- In-situ monitoring of processes in transmission with highest spatial resolution using
 - Time resolved data acquisition during in-situ experiments
- Automation of data acquisition and analysis processes using scripting and API options for
 - Generation of specific analysis jobs
 - Batch processing

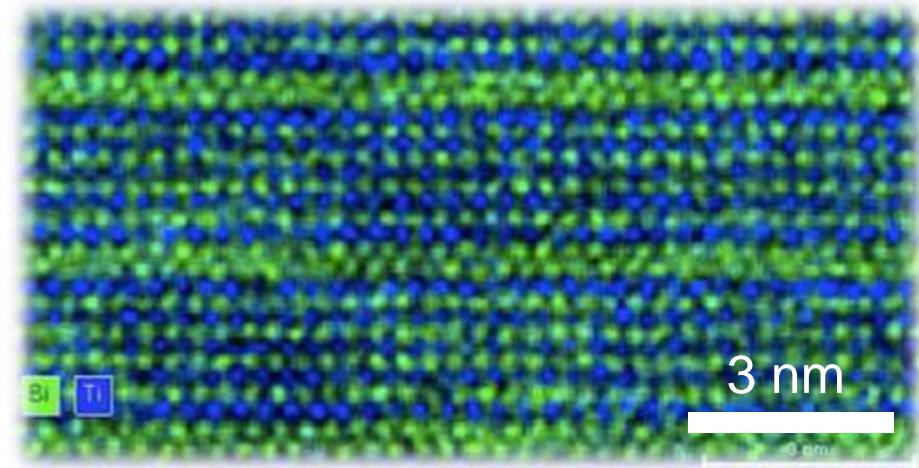


XFlash® 7T – Stable resolution from nm to 1Å

- Slim-line design and geometry optimization for each microscope pole piece type ensure **maximum collection and take-off angle** for fast data acquisition
- Avoiding specimen tilt, absorption, shadowing and system peaks.
- **No mechanical or electromagnetic interference** with high-end TEM performance, even at atomic resolution.
- EDS element mapping in TEM, STEM and SEM (T-SEM) on the **nanoscale with drift correction for periodic features**, such as quantum wells and atom columns.



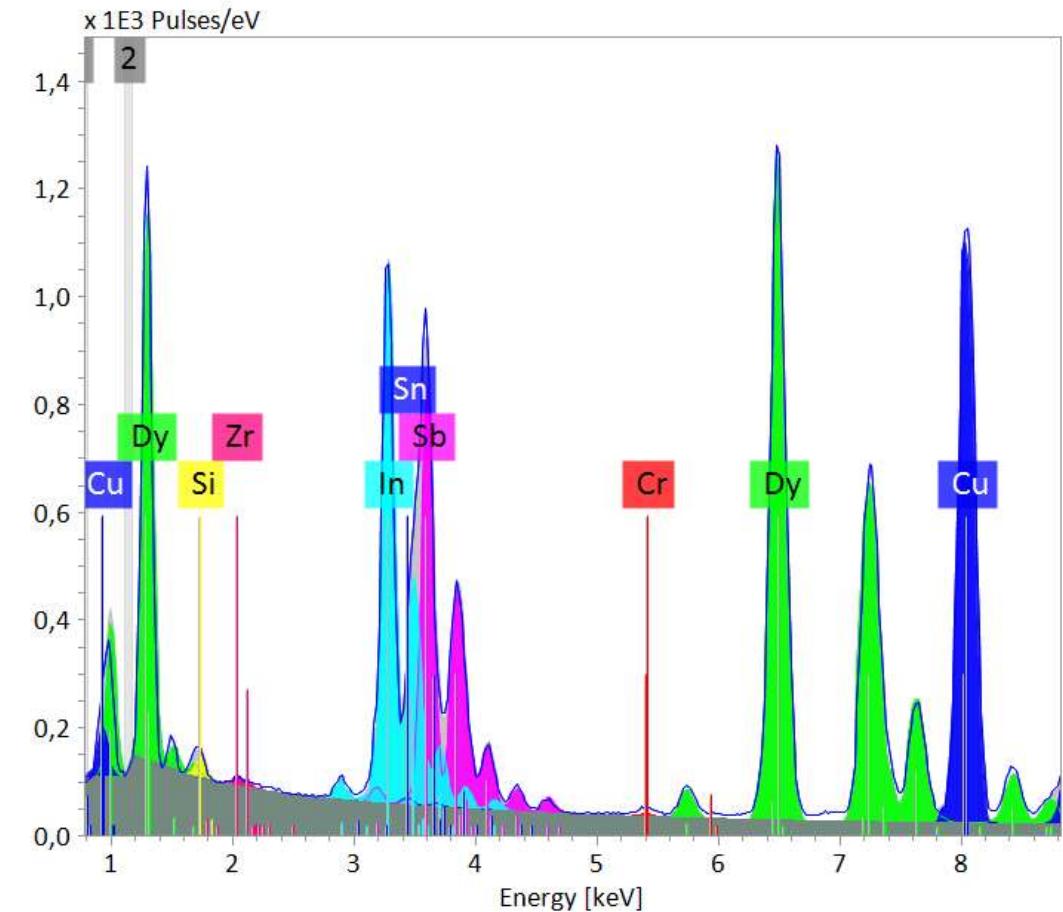
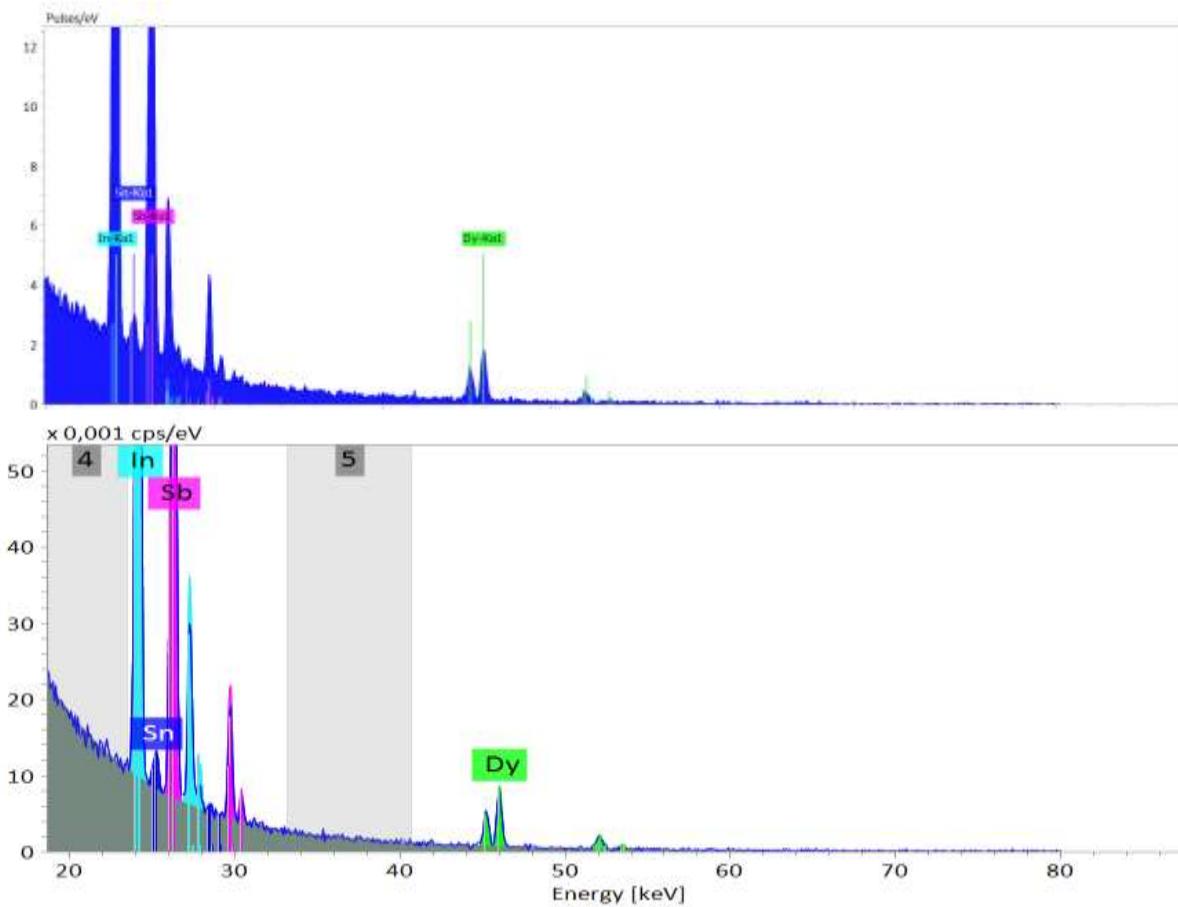
Data courtesy: Nion Co.: III-V quantum wells



Data courtesy: L. Keeney et.al, TCD, Dublin; Multiferroic material

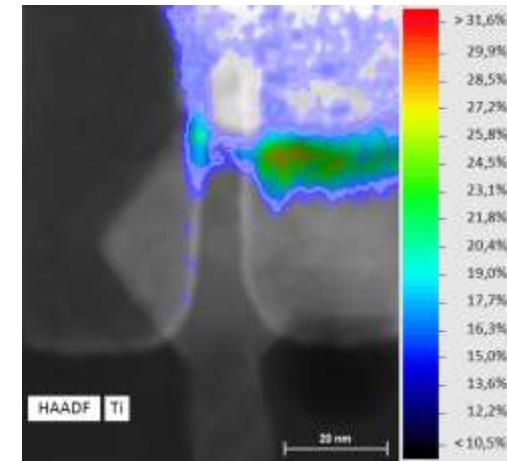
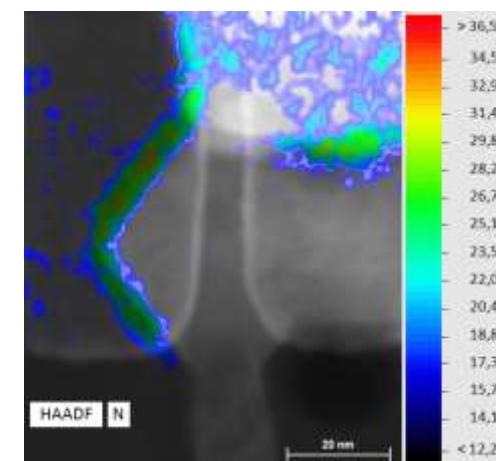
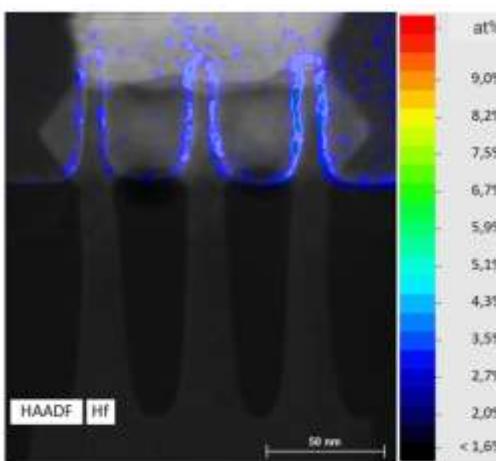
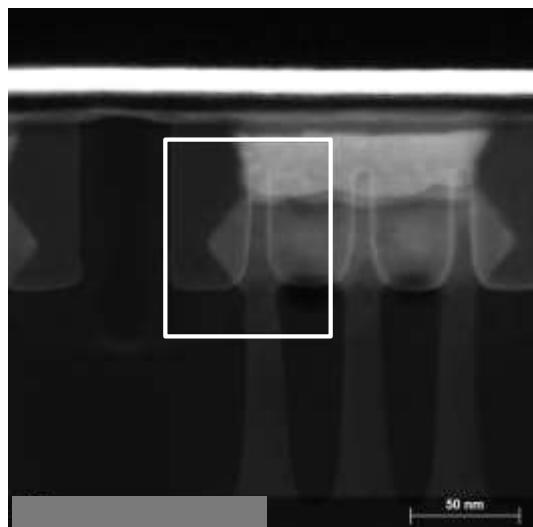
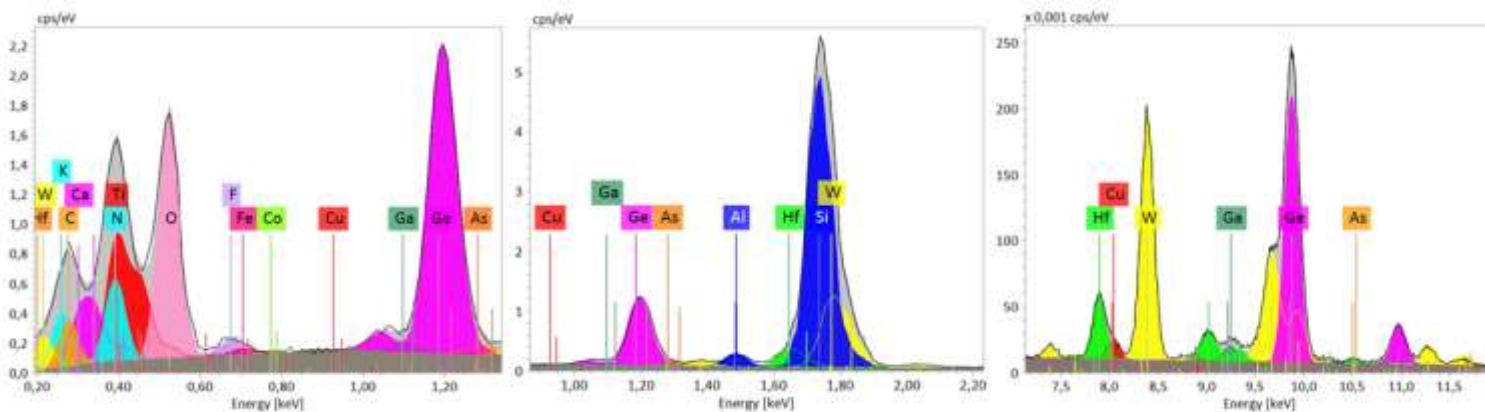
XFlash® 7T – 80 kV element lines for quantification

Example: Dy₂InSbO₇ powder, high and low energy line deconvolution for quantification



XFlash® 7T – 3 quantification models for TEM, STEM and STEM in SEM (T-SEM): Si-based semiconductors

- Quantitative EDS in STEM using the Cliff-Lorimer method allows to distinguish nitrogen and titanium nitride in semiconductor nanostructures.



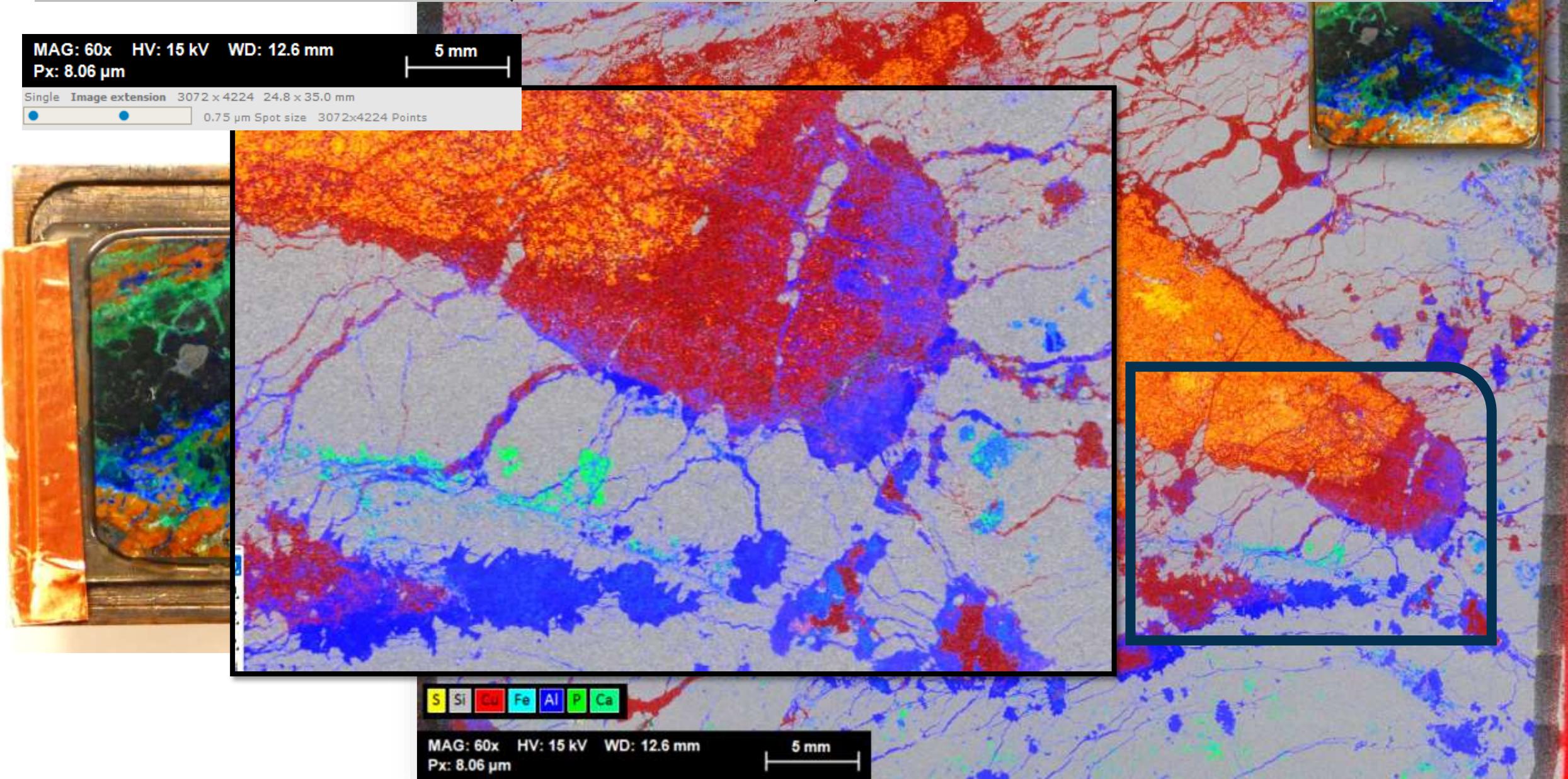
Data courtesy: ACE



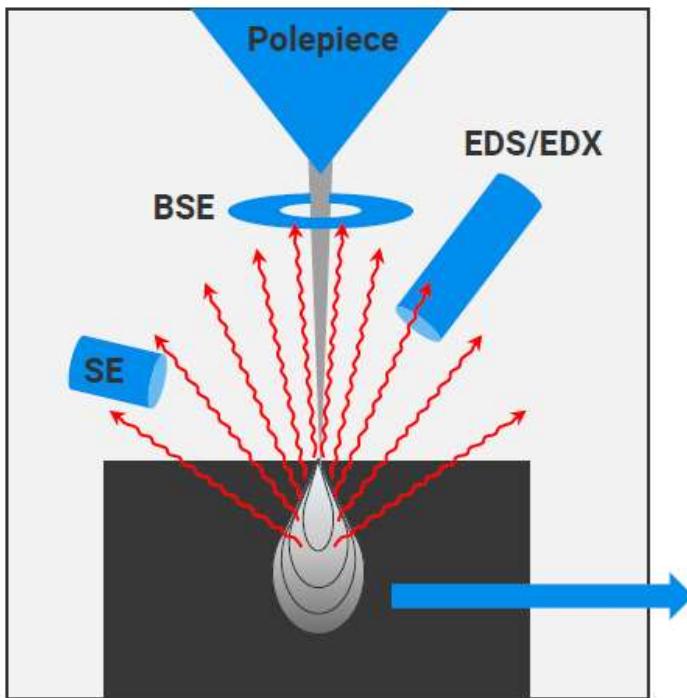
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SEM and FEG-STEM EDS Application Examples

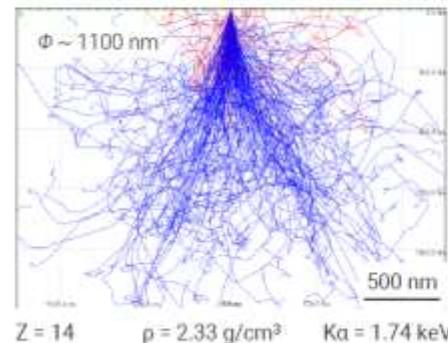
THIN SECTION COPPER ORE (ATLAS MOROCCO) PREPARED BY THIN SECTION LAB TOUL



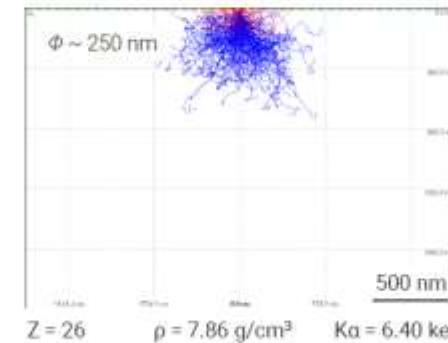
: SEM EDS analysis of bulk sample (Monte Carlo simulations)



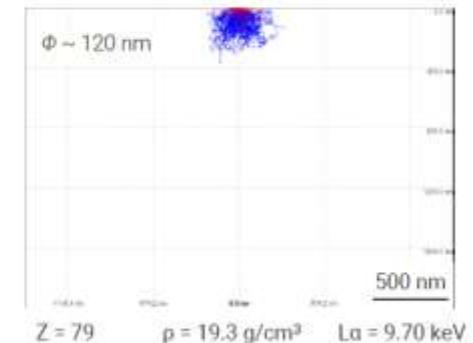
Si - 15 kV



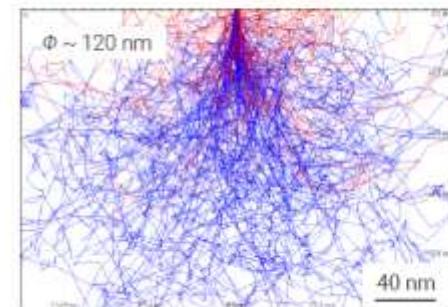
Fe - 15 kV



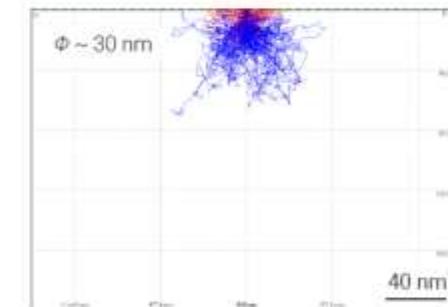
W - 15 kV



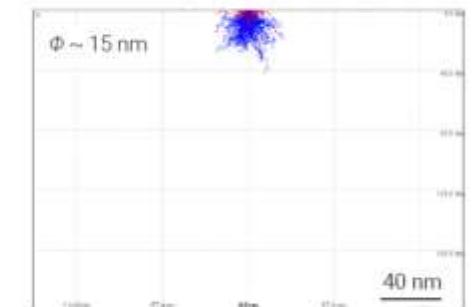
W - 15 kV



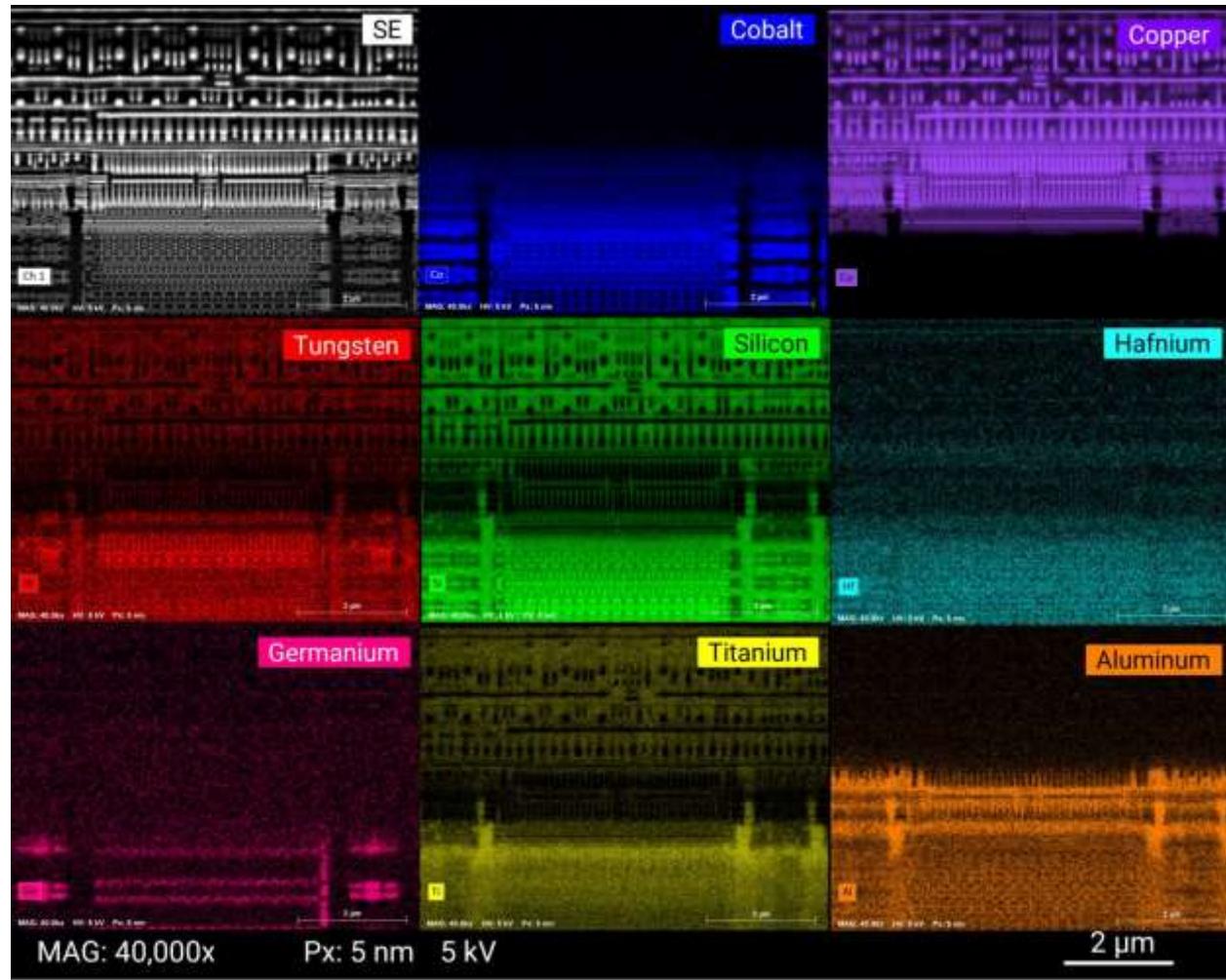
W - 5 kV



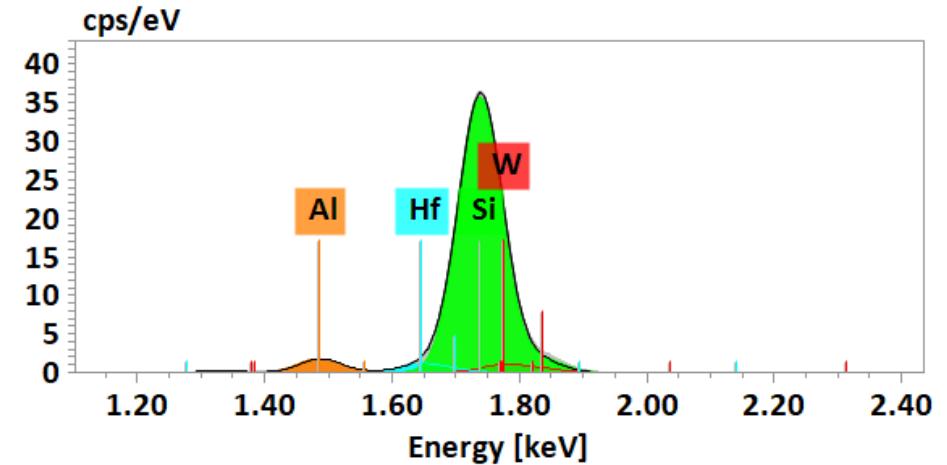
W - 3 kV



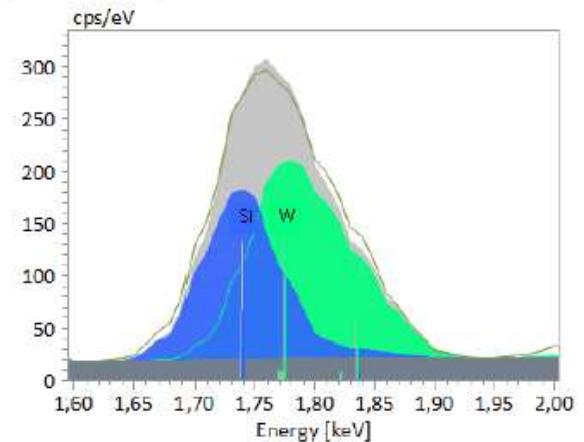
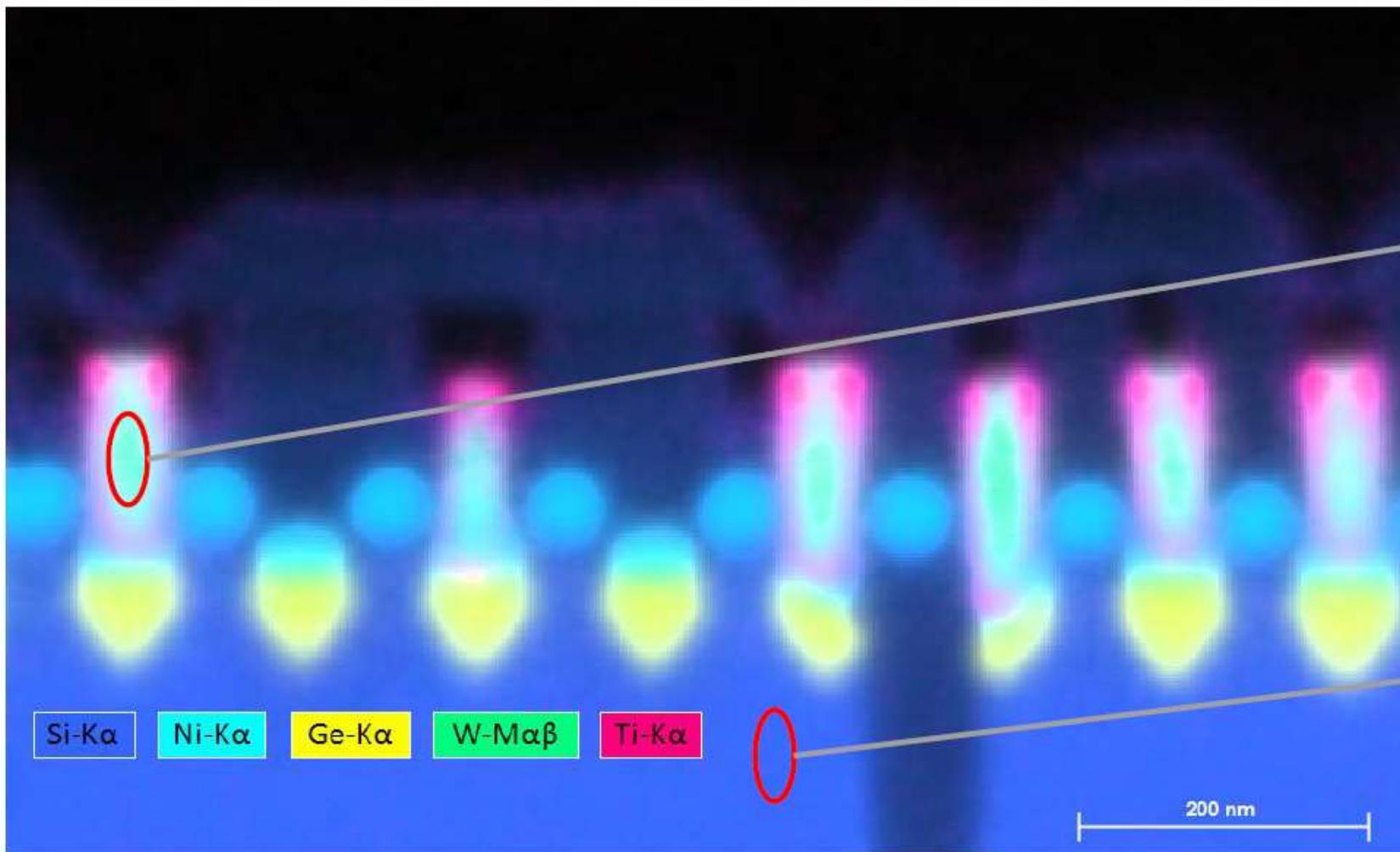
SEM EDS analysis of bulk 7 nm process FinFET @ 5kV



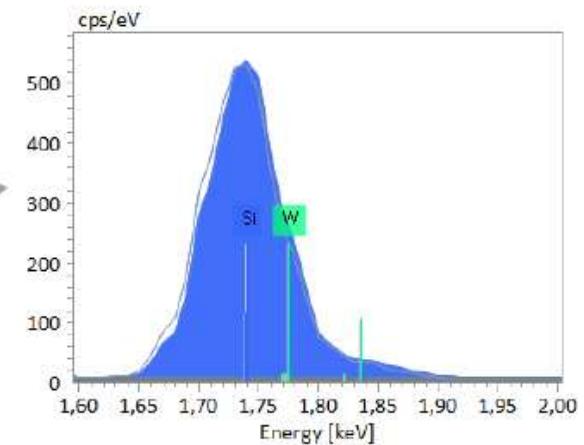
- Semiconducting IC chip (FinFET) delayered with FIB
- Low kV, EDS spatial resolution ~10 nm (theoretical bulk lateral resolution), Co-Cu and Si-W-Hf peak overlaps deconvoluted
- XFlash® 7 features: High collection angle ideal for low X-ray yield samples - SNR



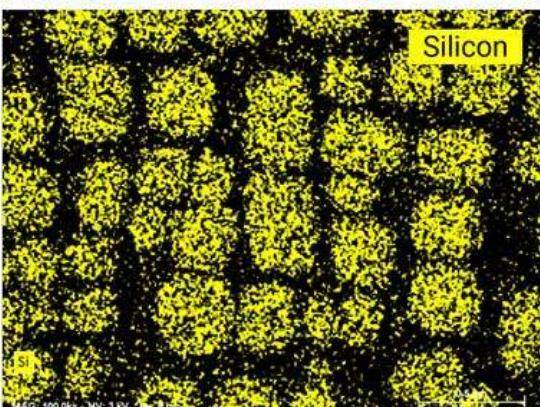
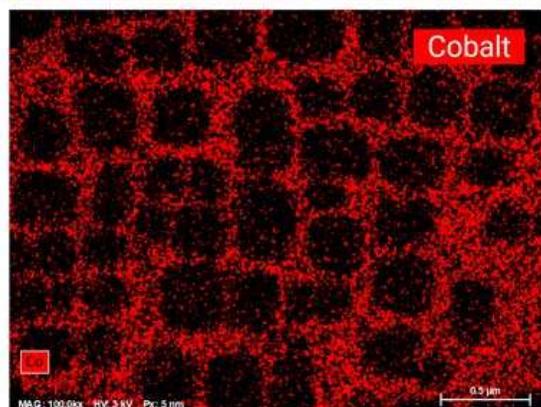
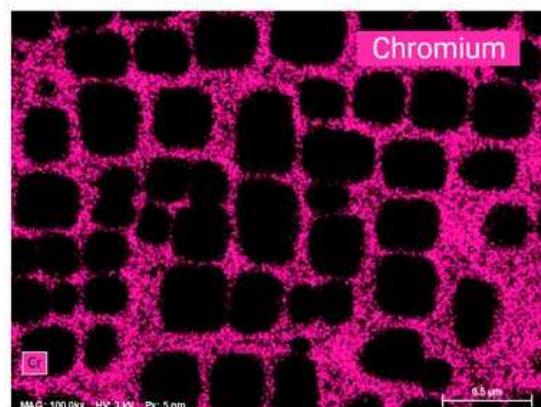
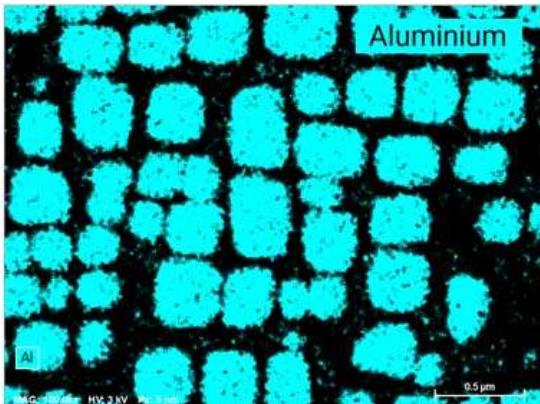
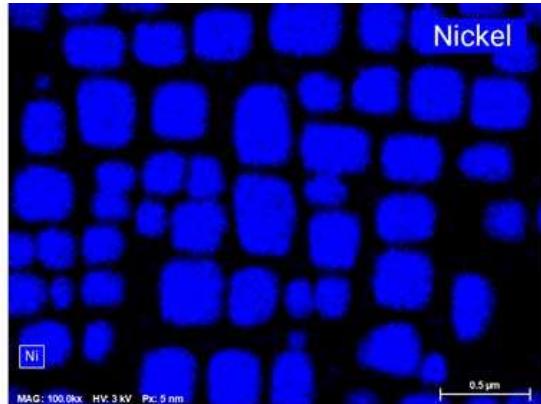
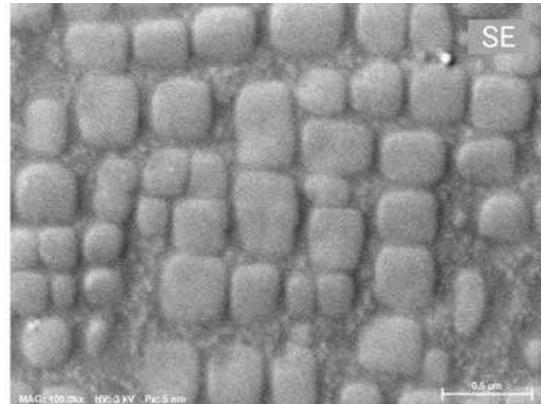
High spatial resolution mapping - Semiconductor structure (RAM)



Distinguishing overlapping element peaks with deconvolution



Resolving γ and γ' phases of Ni-based single crystal superalloy



MAG: 100,000x

Px: 5 nm

3 kV

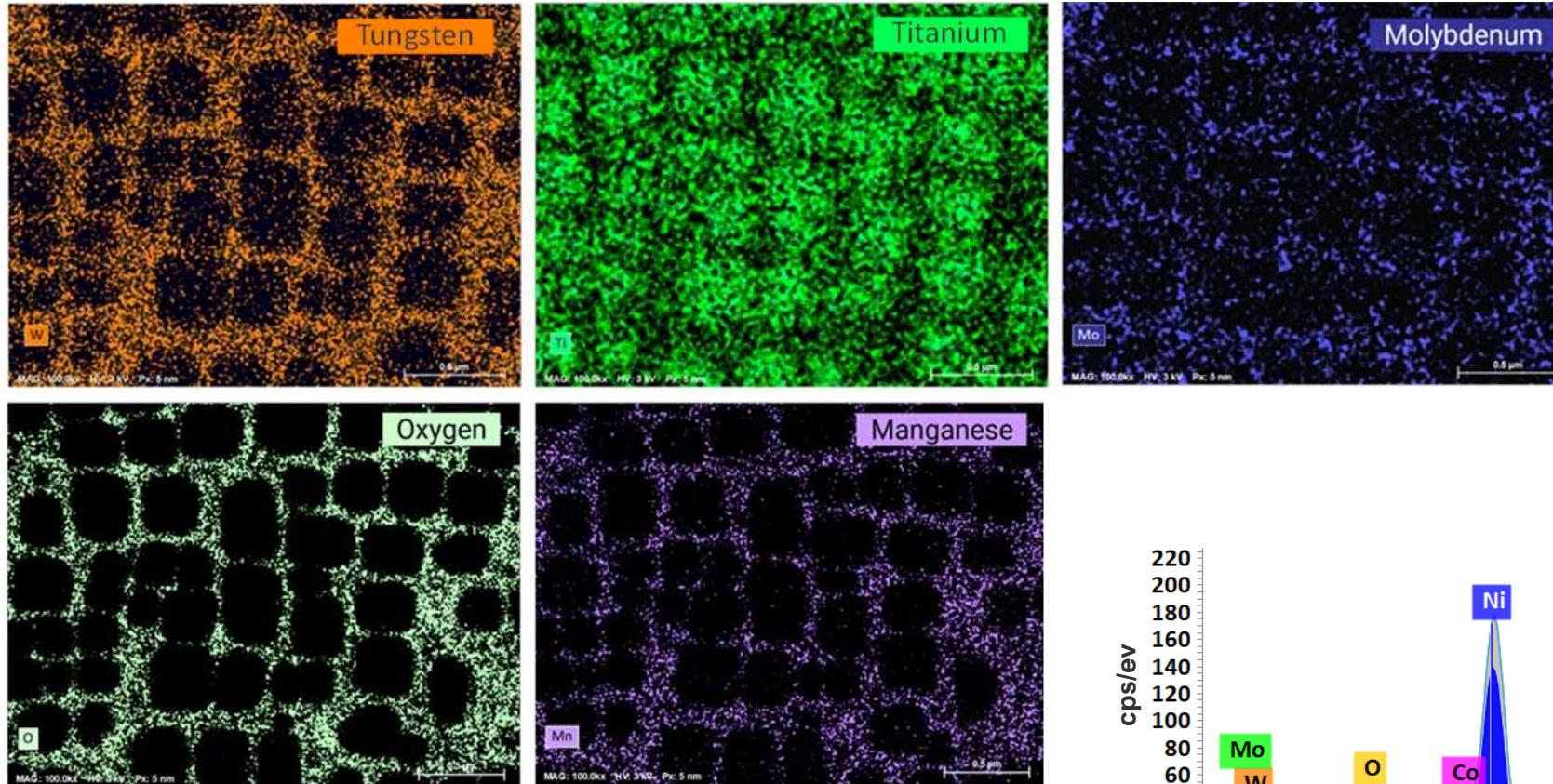
Major elements (>1.0 mass%)

500 nm

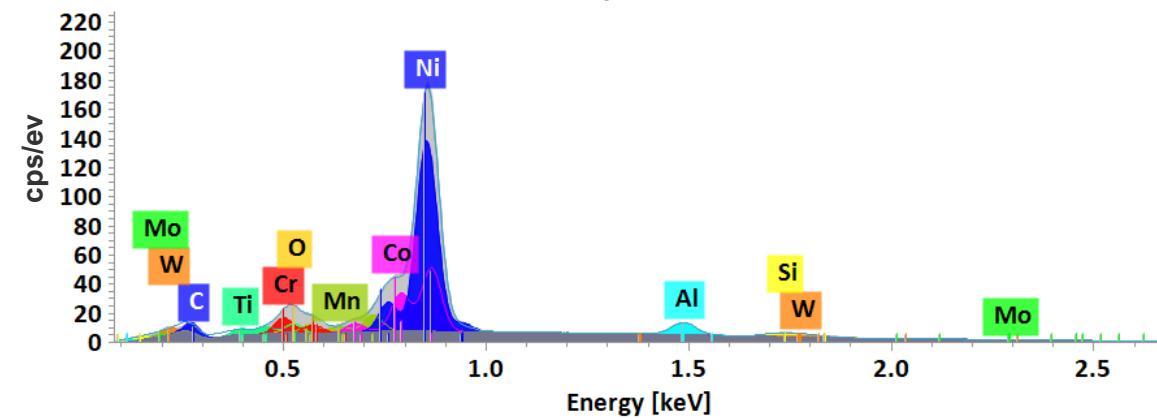
- High EDS spatial resolution of ~15 nm acquired at 3 kV
- Distribution of trace elements at high resolution from the matrix
- Large solid angle: High sensitivity for low X-ray yield samples



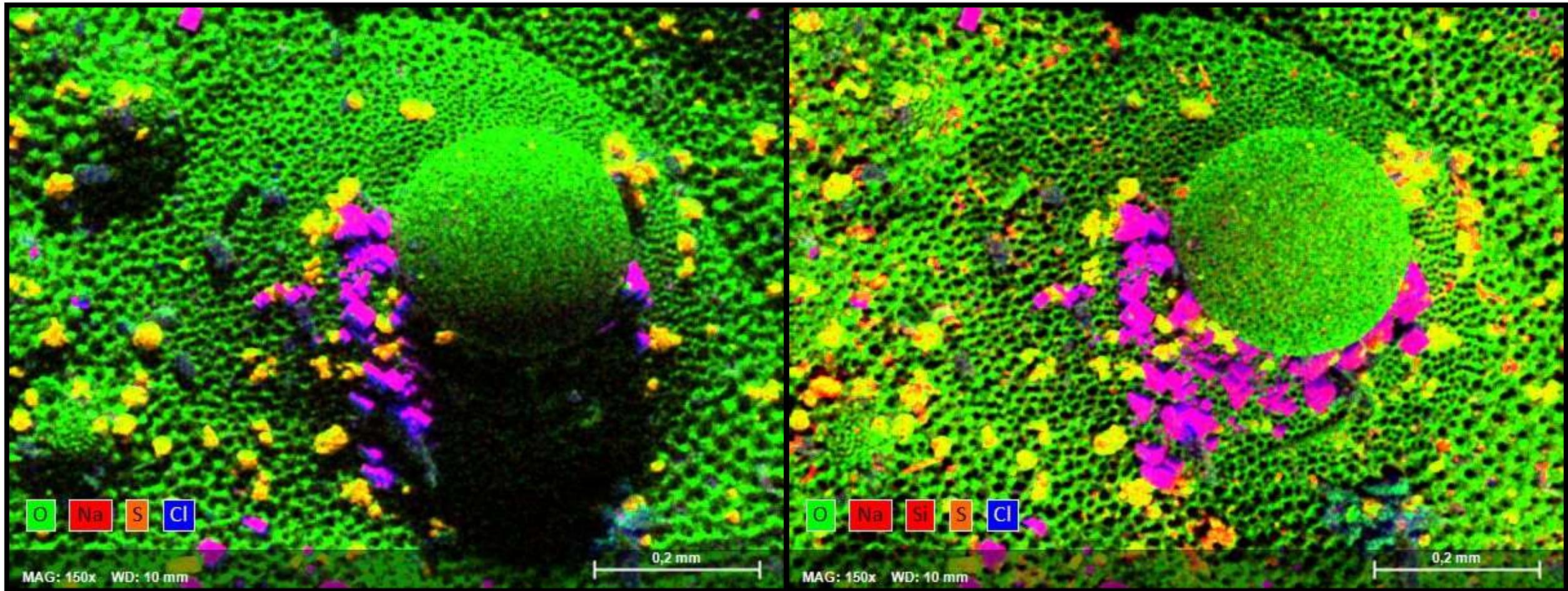
Example 5: Resolving γ and γ' phases of Ni-based single crystal superalloy



Trace elements (<1.0 mass%)

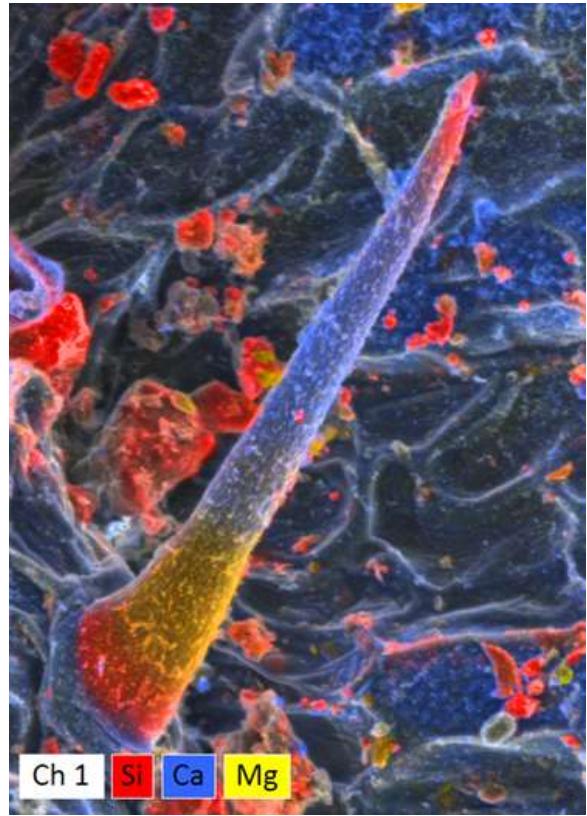


Single detector Dual versus dual detector (face to face)

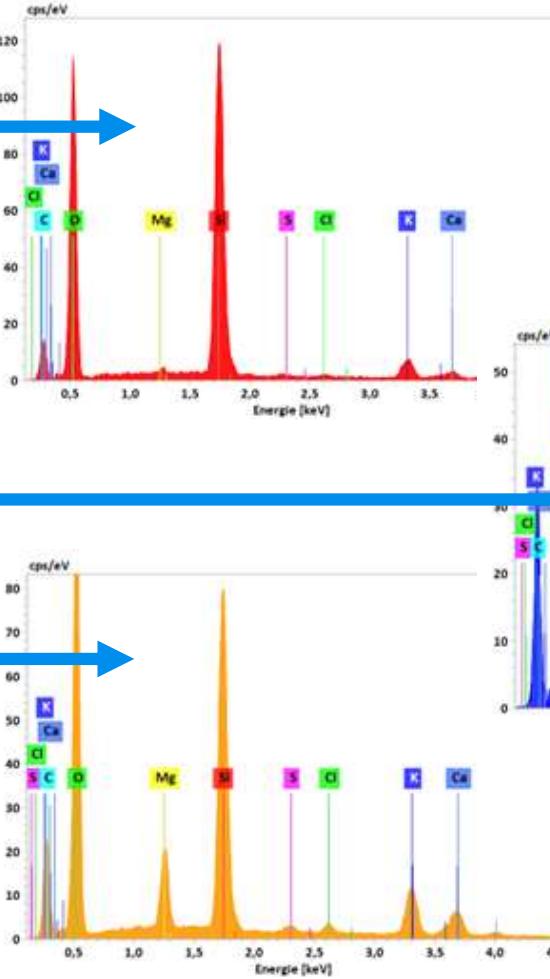
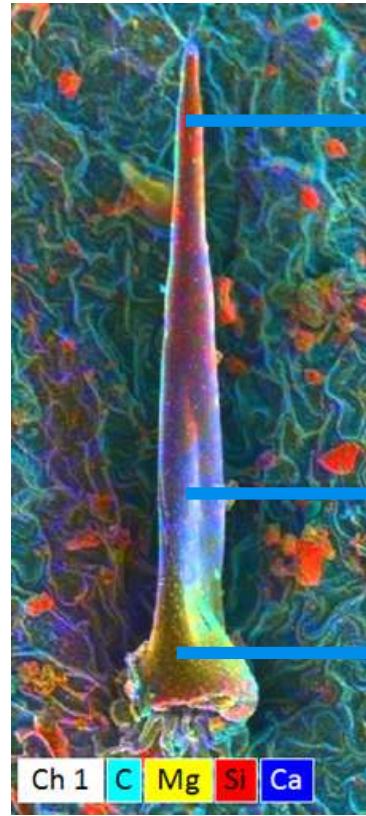


SEA URCHIN SHELL

Analysis of a stinging nettle plant by a dual EDS detector system



ESPRIT Hypermap of a trichome from a stinging nettle (*Urtica sp.*). The tip and base are enriched in Si (in red), whereas Ca (in blue) and Mg (in green) contents are higher in the middle part of the trichome.



EDS spectra extracted from the ESPRIT HyperMap confirm the presence of 3 different biominerals:

The tip and base of the trichome have Si-rich minerals, silicon dioxide acts as a hardening agent in the material but may also break easily when necessary.

The rest is made of Ca- and Mg-rich minerals, giving the structure the necessary flexibility.

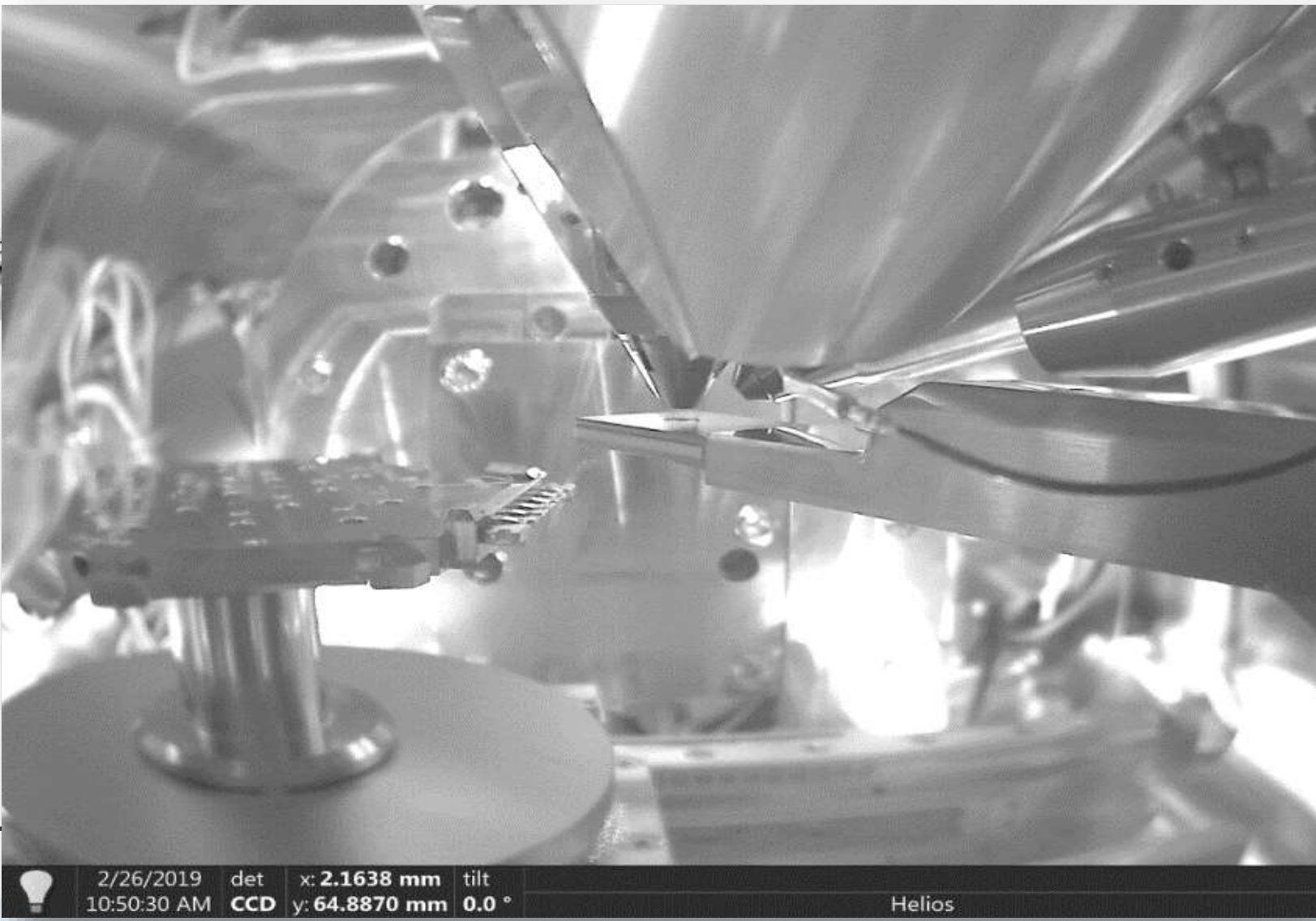
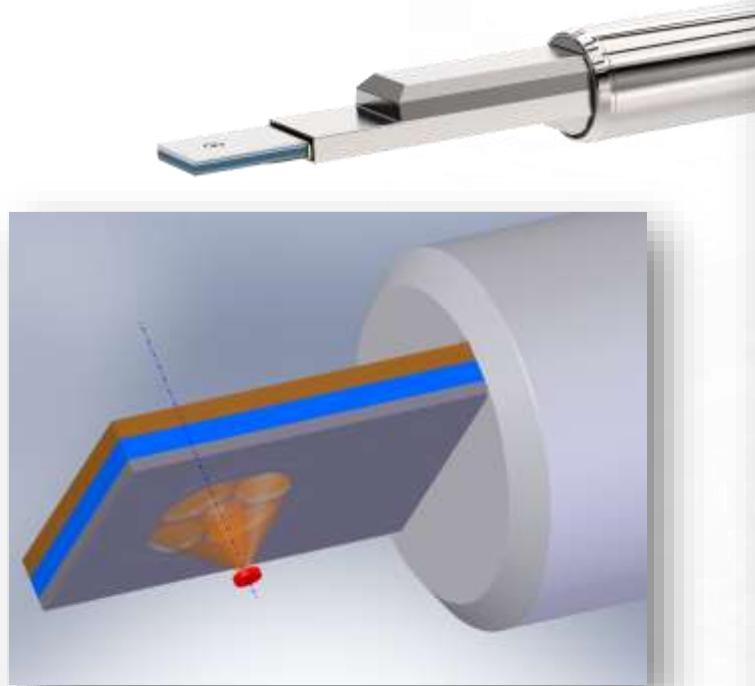


XFlash® 7 – The New EDS Detector Series

BRUKER NANO ANALYTICS

XFlash® 7
4-segment annular EDS detector -XFlash®FlatQUAD

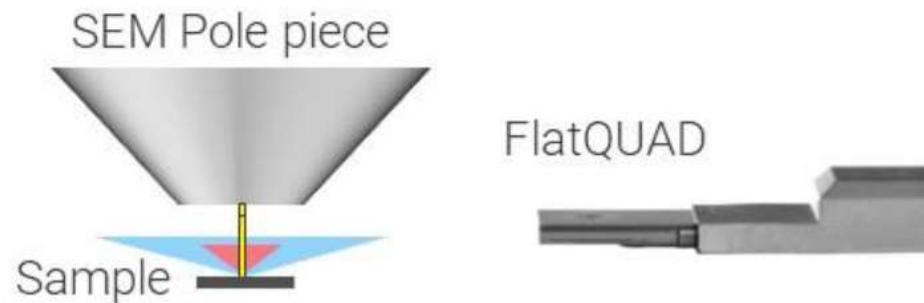
XFlash® 7 – The right solid angle for better analysis



XFlash® 7 – Flat Quad Annular

XFlash® 7 – FlatQuad

XFlash® FlatQUAD

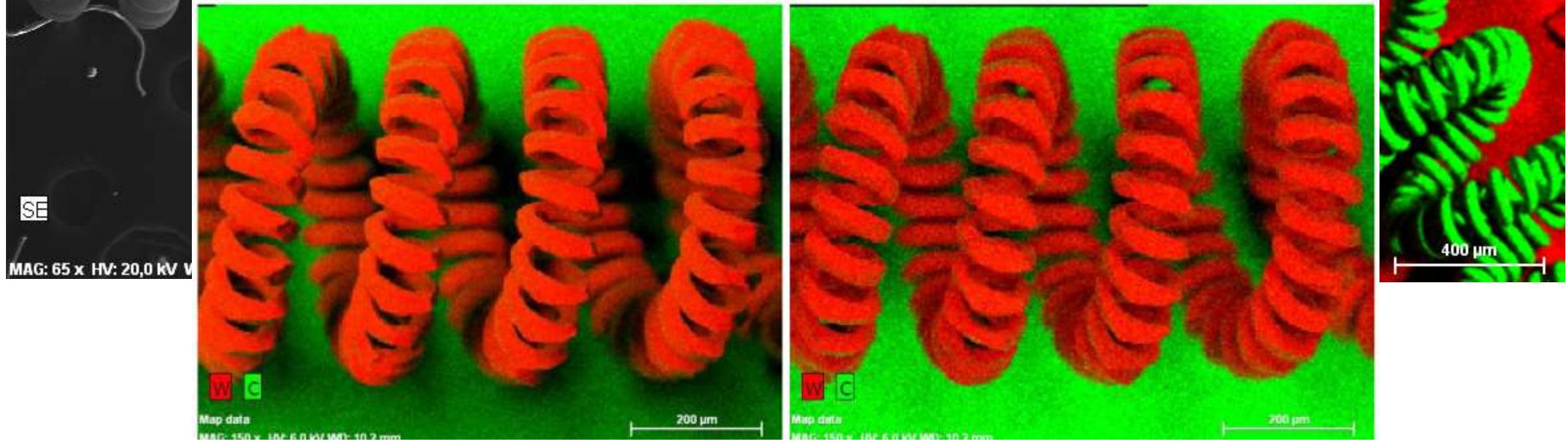
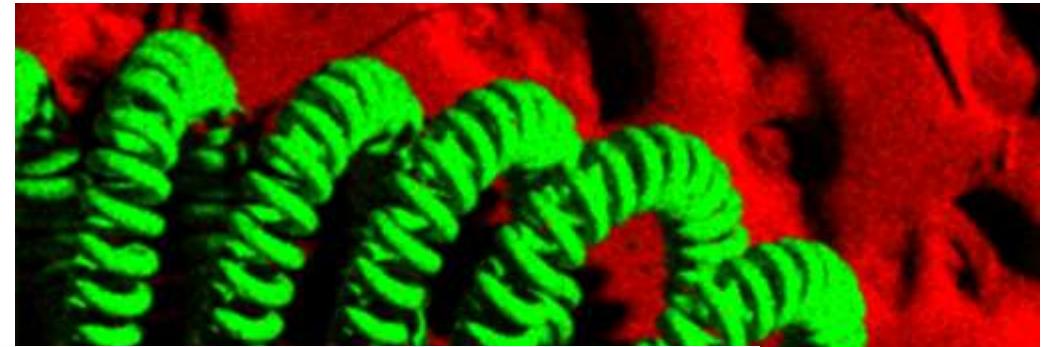
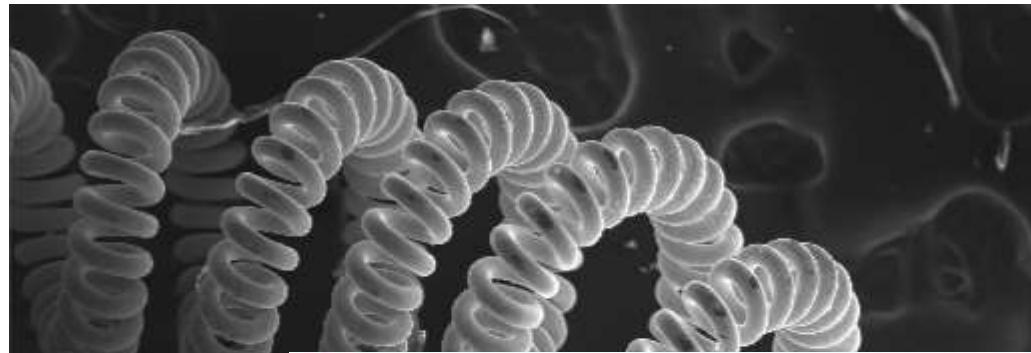


- Annular 4-segment (4x) SDD geometry, central ap.
- Side entry EDS (STEM/BSE like)
- Large solid angle of 1.1 sr
- High take-off angle (~60°)
- Optimal signal collection geometry



- High sensitivity at very low probe currents ~few pA
- Minimize sample charging/damage/C-deposition at low PC
- High vacuum conditions EDS – high resolution
- Low vacuum capability
- Moderate probe currents for high-speed EDS mapping
- Low x-ray yield samples: Low PC – High resolution
- Nanoparticles, Thin lamellae, beam sensitive materials

XFlash® 7 – The right solid angle for better analysis

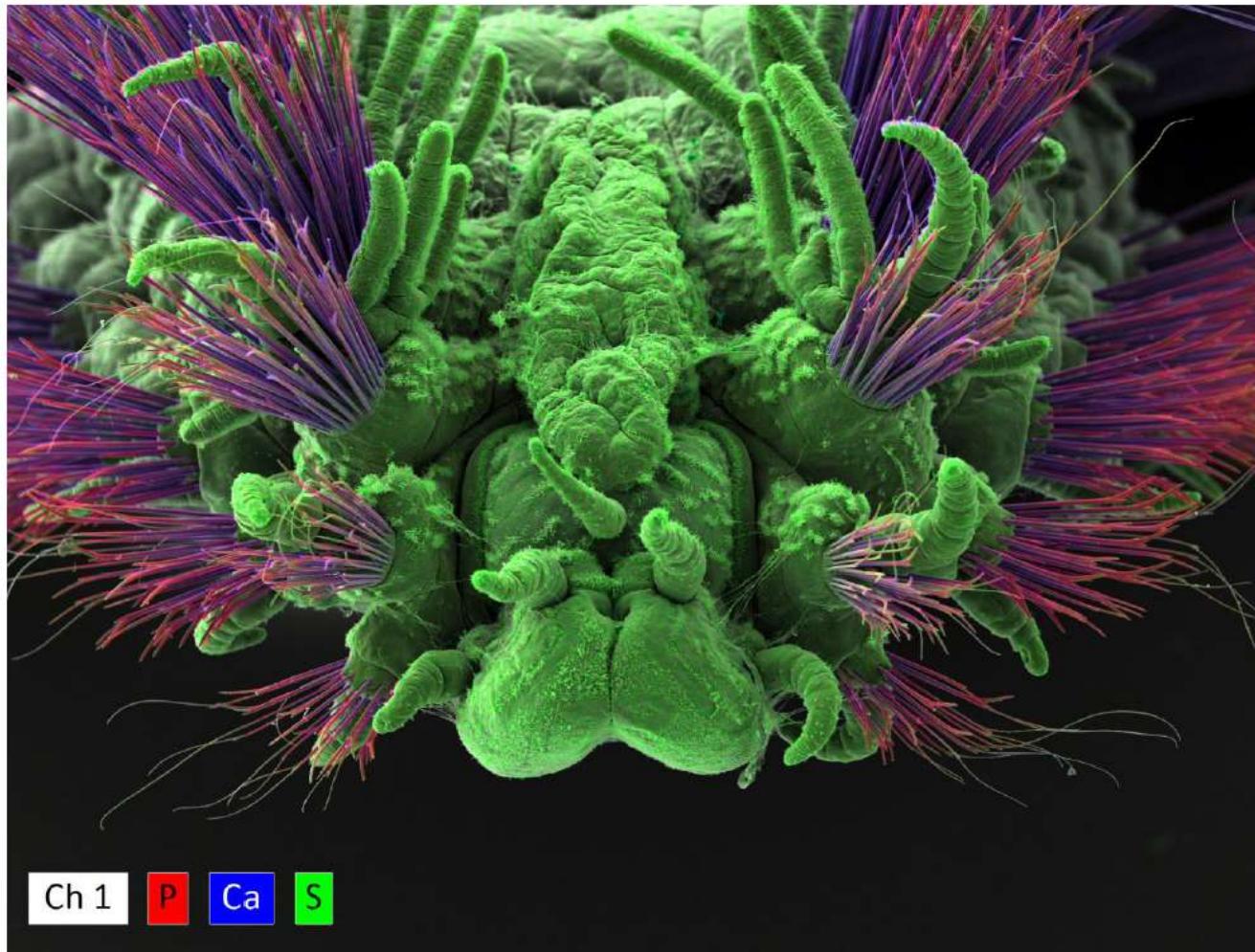


XFlash® 7 – Flat Quad Annular detector (with one diode at left and four diodes at right)

Unlock the secrets of marine life with cutting-edge technology

Head of the polychaeta

polychètes



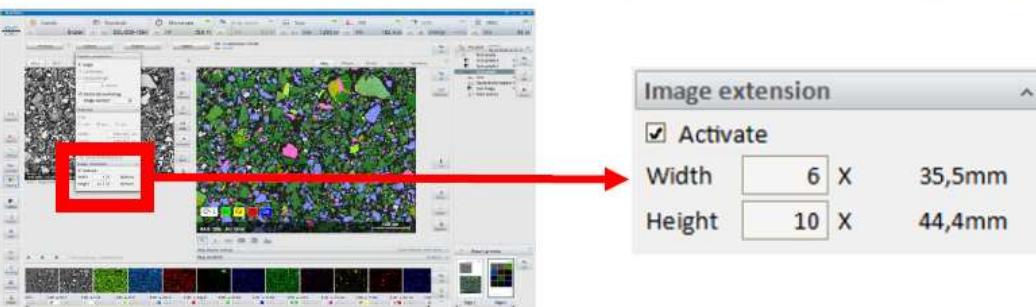
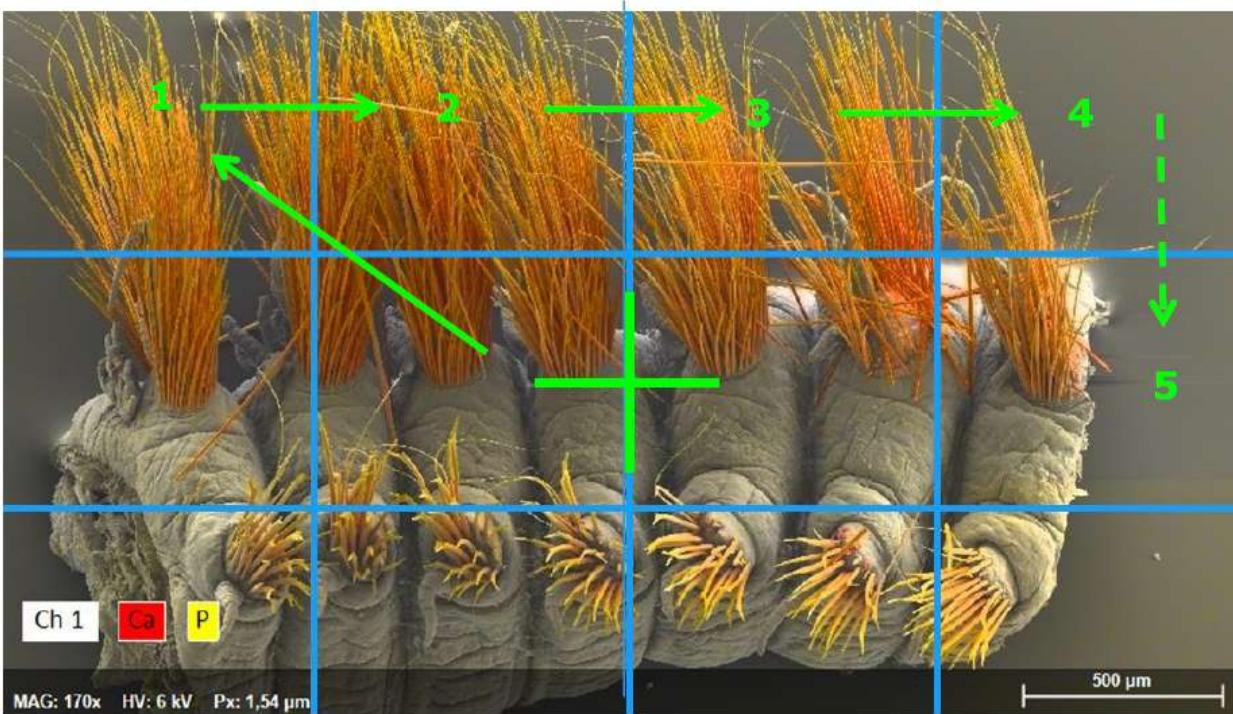
- The head of Polychaeta, adorned with its mineralized bristles composed of calcium phosphate,
- These bristles, crafted from calcium phosphate, not only provide structural support to the organism but also serve a vital function in its defense mechanism
- Mapping parameters:
 - 10 kV
 - Image 2048x1536 pixel
 - Magnification: 160
 - Pixel spacing: 0,8 µm
 - Pd coated
 - Time: 176 sec.
 - WD 13 mm



Sample courtesy of University Bonn

Increase sample measurement area with one click

Image Extension



▪ Image Extension

- Use actual sample position as central Mapping position and define number of x/y frames around
- Result: **one** Hypermap file
- Image extension can be enlarged for a full sample map

Sample courtesy of University Bonn



UNIVERSITÄT BONN

XFlash® 7 – Flat Quad Annular detector

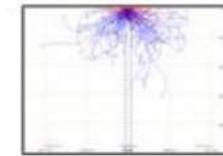
How to achieve ultra high resolution?



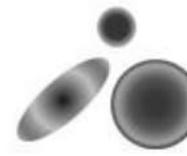
Drift
correction



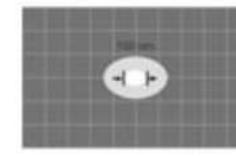
Interaction
volume



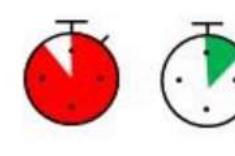
Material
density/matrix



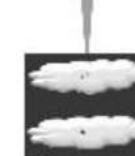
Beam
footprint



Pixel/map
Size/statistics

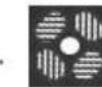


Dwell time
compromise



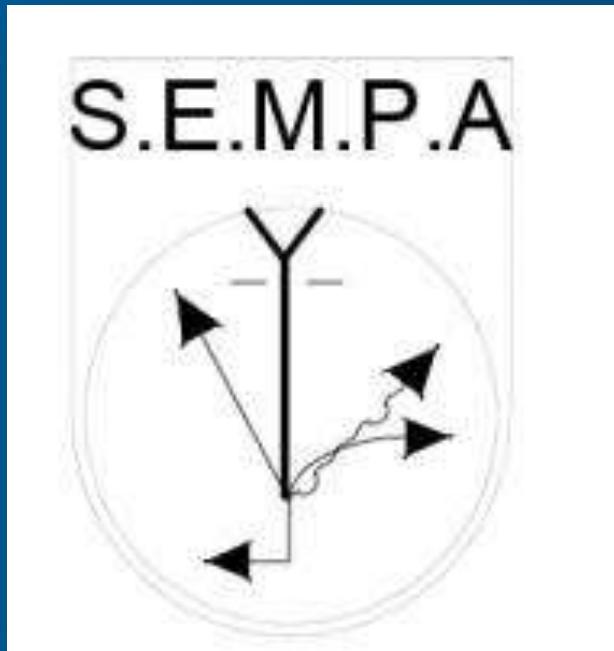
Beam
conditions

- **HW requirements:** Beam alignment, probe current, environment control – mechanical/magnetic interactions.
- **Analytical detectors:** High collection efficiency EDS detectors; stable, efficiency and high throughput HW or electronics, backed by a powerful SW.
- **Specimen requirements:** Specimen thickness, matrix, mounting, contamination control.
- **XFlash® FlatQUAD:** Highest sensitivity (few pA) with 1.1 sr solid angle, suitable for low and high kV analysis.
 - Optimized for speed and sensitivity for challenging applications.
- **XFlash® Oval 100mm² windowless:** Ultra high EDS spatial resolution capable detector.
 - Light element, low kV low probe current analysis with high sensitivity, TEM-like EDS measurements in SEM.



XFlash® 7 – Feature highlights

- Bruker's latest generation of QUANTAX EDS features the XFlash® 7 detector family providing the largest solid angle for X-ray collection and highest throughput
- The XFlash® 7 continues to set standards in performance and functionality for Scanning Electron Microscope (SEM), Focused Ion Beam (FIB) and Electron Probe Micro Analyzer (EPMA)
- The XFlash® 7 detectors offer optimized solutions for EDS analysis of electron transparent specimens in TEM and SEM
- The unique annular XFlash® FlatQUAD answers your questions on challenging samples, e.g., low kV, low beam current, and surface topography
- Slim-line technology, large collection angle design, latest generation pulse processing
- Highest spectral performance obtained with best energy resolution
- Increased results accuracy by sophisticated quantification algorithms and a unique combination of standardless and standard-based methods
- Time resolved data acquisition for in-situ experiments
- Maximized system uptime through predictive maintenance



Thank you!