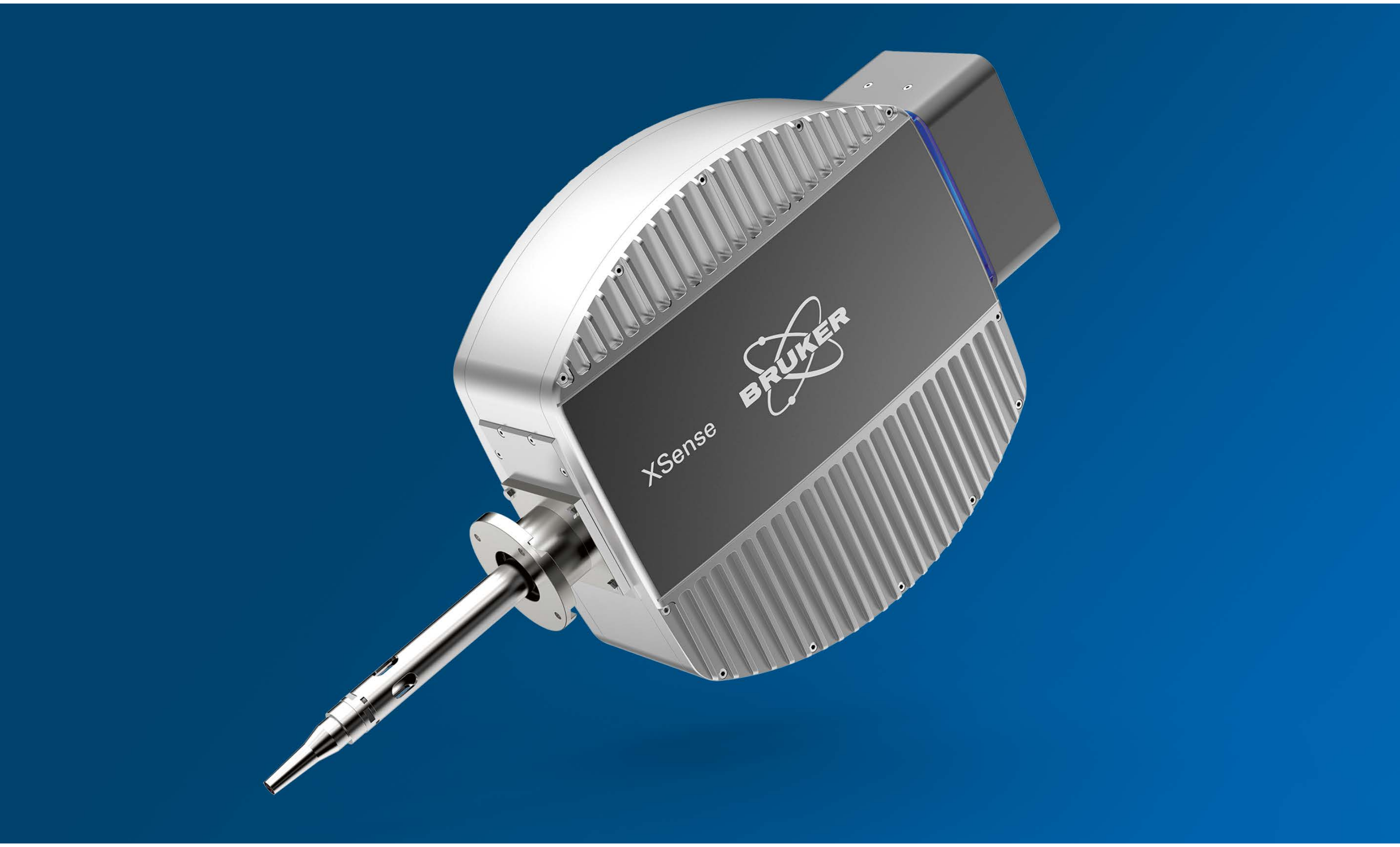


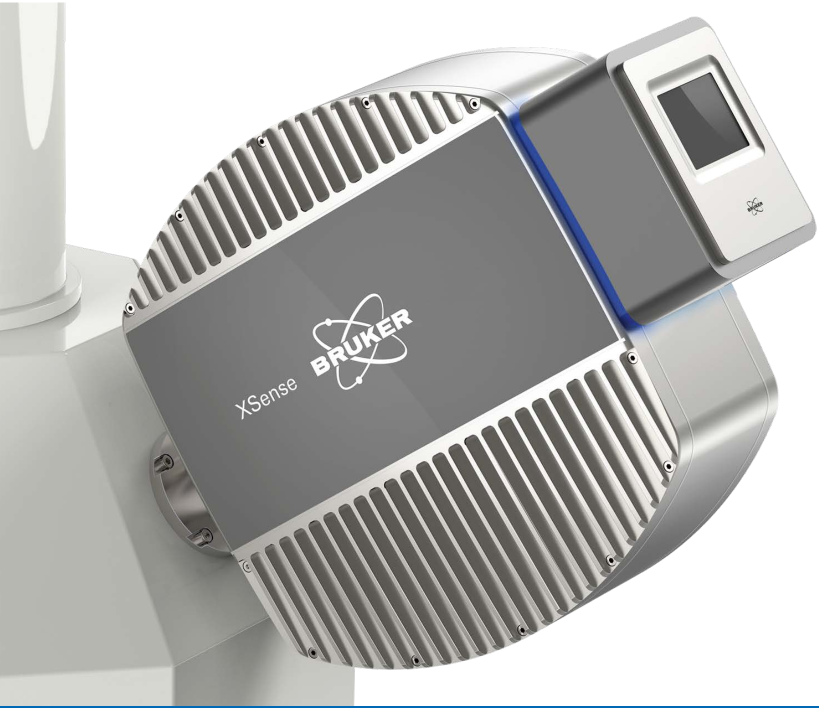

# QUANTAX WDS

Wavelength Dispersive Spectrometry for SEM

WDS

Innovation with Integrity

# High Resolution, High Sensitivity, Low Detection Limits




The QUANTAX WDS for SEM system uses the XSense wavelength dispersive spectrometer. This parallel beam spectrometer has the finest grazing incidence optic on the market and up to six analyzer crystals. The automatic optic alignment and pressure controlled proportional counter systems allow the operator to acquire accurate results easily and quickly. Seamless integration into the ESPRIT software enables fully combined EDS and WDS analyses.

SEM-WDS is an ideal technique to complement SEM-EDS in demanding applications which require:

- substantially higher spectral resolution
- enhanced P/B-ratios, i.e., lower detection limits
- outstanding sensitivity for light elements.

Wavelength dispersive spectrometry is based on Bragg diffraction using natural crystals and synthetic multilayers for constructive interference of defined X-ray energies. This well-established technique yields X-ray peaks that are up to 20 times narrower than those of any high-resolution EDS.

The unbeatable performance of a WDS allows resolution of close-by X-ray lines for the analysis of challenging samples. With its grazing incidence optic, the XSense spectrometer delivers spectral resolution better than any WDS using a polycapillary optic.

The wavelength selective nature of the WDS technique and the detection system using a proportional counter also lead to superb signal to noise ratios, being ca. 10 times higher than those of an EDS. High signal to noise ratios translate directly into lower detection limits: a WDS can determine trace element concentrations down to a hundred parts per million, and even lower.

The grazing incidence optic used with the XSense spectrometer captures X-rays close to their source of origin with a large solid angle and thus results in higher signal intensities for low X-ray energies compared to classical Rowland-circle spectrometers. This characteristic is advantageous for the analysis of light elements or the exploration of higher line series (e.g., L and M lines), surpassing previous limits for low-kV and low-current applications.

# Designed for Highest Performance

## High resolution where it really counts

Equipped with up to six analyzer (diffracting) crystals, the XSense wavelength dispersive spectrometer covers the 70 eV to 3.6 keV energy range most relevant for high resolution X-ray microanalysis. The large number of crystals with partially overlapping energy ranges provides the optimum choice for every application.

## Ultimate sensitivity through advanced kinematics and high-end optics

XSense's advanced kinematics maintains perfect positioning of the diffracting crystal with respect to the incoming beam over the full Bragg angle range, resulting in unparalleled performance. The sophisticated non-magnetic optics avoid beam shift and image distortion and pushes resolution, peak-to-background ratios and sensitivity to their limits.

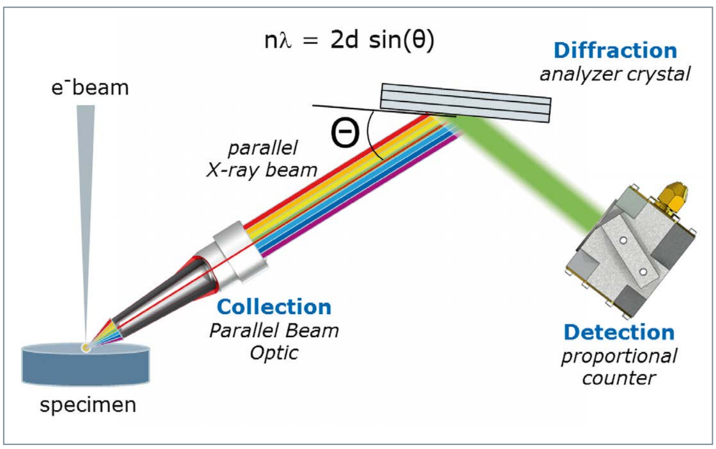



Fig. 1 Schematic working principle of a parallel beam WDS. While Bragg diffraction is the common basis for all WDS types, a collection optic with a high solid angle provides the advantages of the present system.

## Auto-alignment system for optimum measurement conditions

The precise alignment of the spectrometer's optical axis with respect to the electron beam is of paramount importance in parallel-beam spectrometry. XSense's internal fully motorized 3-axis stage enables fast and stable positioning of the parallel-beam optics with sub-micrometer resolution. A smart algorithm provides correct alignment at the touch of a key.

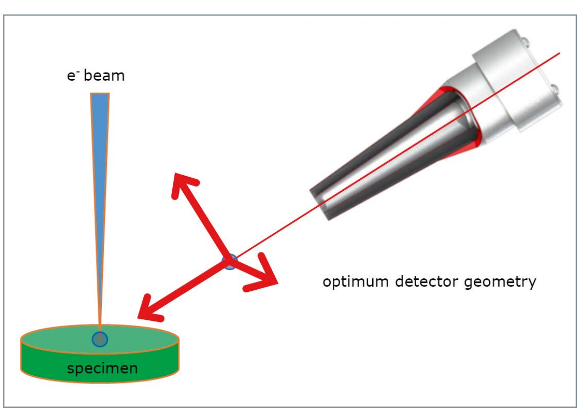



Fig. 2 The XSense is equipped with a unique automatic optical alignment system for fast and easy operation.

## Finest optic system available for a WDS

The 3-cone parallel-beam mirror optic ensures extremely low residual beam divergence. A unique secondary optic between crystal and detector suppresses background artefacts. In combination with the auto-optic alignment system this results in the highest possible resolution achievable by a parallel beam (PB) WDS.

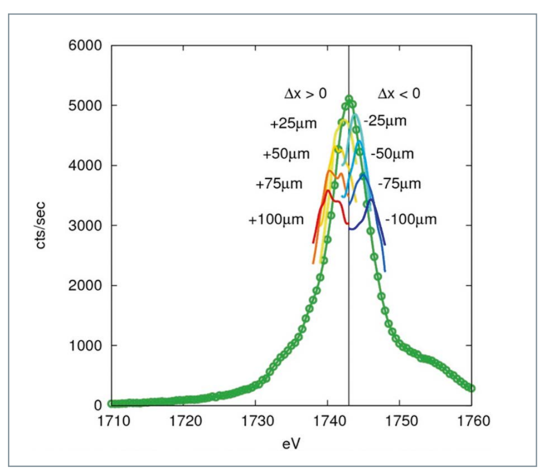



Fig. 3 Highest resolution, maximum count rates and best reproducibility achieved with the XSense's automatic optical alignment system.

# Designed for Ease-of-Use

## Easy setup – short time to measurement

Numerous automation features make the QUANTAX WDS easy to operate and relieve the user from tedious and time-consuming adjustments. This includes perfect optical alignment with minimum user intervention, automatic choice of appropriate analyzer crystal, automated gas flow proportional counter and many more.

## Reliable acquisition with the flow-controlled proportional counter system

Bruker's unique gas and counter management system actively controls the counter's internal gas pressure and automatically performs all high voltage and discriminator settings. While greatly simplifying device operation and minimizing gas consumption, this maintains constant counter characteristics under all environmental conditions, enhancing reproducibility and system reliability.

## Touch control panel for easy spectrometer setup and monitoring

The XSense spectrometer is equipped with a touch panel display for direct control of the spectrometer status. Check interlock functions and perform basic operations like gate valve closing or retraction of the optics using the display.

## Full integration with EDS through the ESPRIT analytical software

Using the intuitive ESPRIT software interface allows EDS and WDS measurements on the same spot. Measurement modes can be changed with a mouse click. Qualitative or quantitative point and object analyses, line scans and mapping are available. The combined EDS-WDS quantification can be done entirely using standards or with coupled standardless EDS and standard-based WDS.

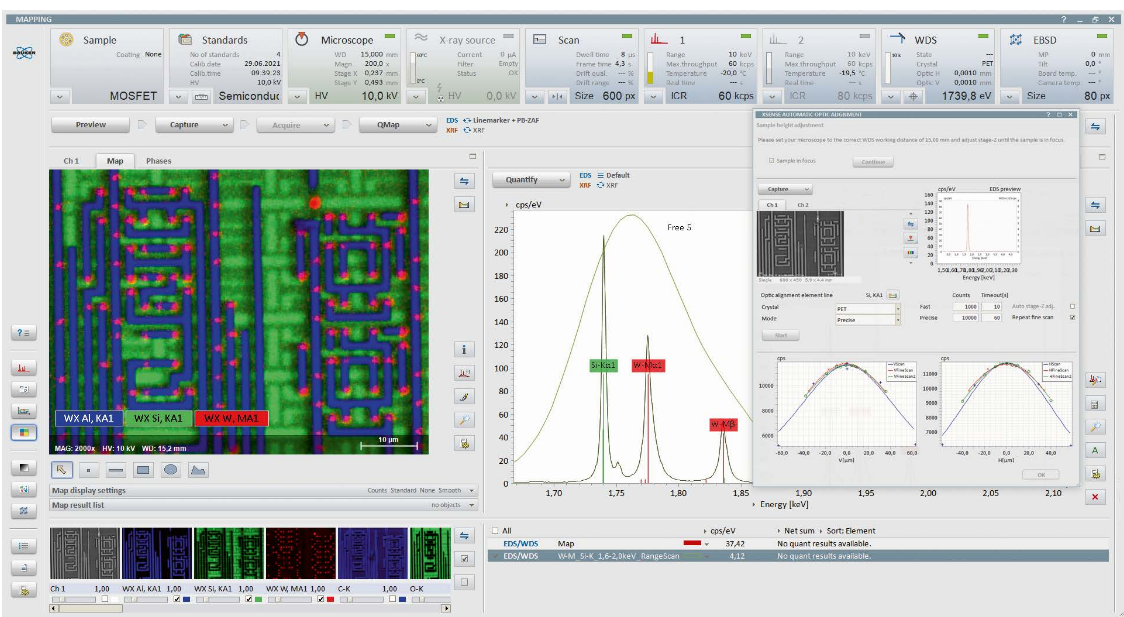



Fig. 4 ESPRIT provides seamless integration of QUANTAX WDS and EDS. All device control and fully combined analyses are performed with one software.

# Application Examples

***Focus A: Resolving X-ray peak overlaps***

## Elemental identification in semiconductors

To achieve high spatial resolution during quality control of semiconductor microchips, analyses must be carried out at low accelerating voltages. However, these conditions complicate the distinction between W and Si: Only the W M series energy lines, which severely overlap with the substrate Si Kα line are excited at low accelerating voltages.

In contrast to EDS, elements such as W are identified with confidence using QUANTAX WDS due to its superior spectral resolution (Fig. 5).

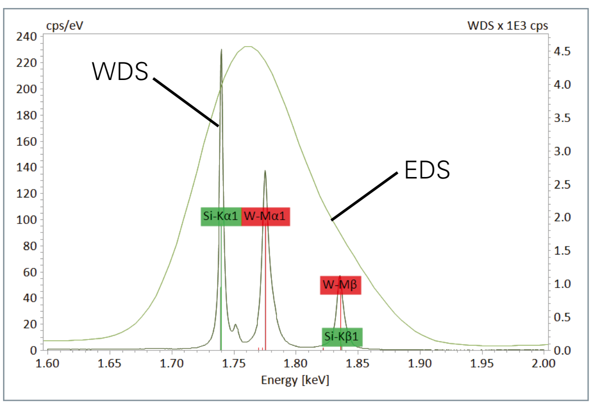



Fig. 5 X-ray spectrum section for tungsten silicide ($WSi_2$) in the energy region of 1.6 - 2.0 keV showing the high spectral resolution of WDS.

## Resolving peak overlaps in sulfide minerals

EDS spectra of galena are characterized by severe peak overlaps of Pb (M series) and S (K series), the major constituent elements (Fig. 6). In contrast to EDS, QUANTAX WDS clearly resolves the individual peaks of sulfur and lead in the corresponding spectrum.

High spectral resolution at low X-ray energies is especially important when low kV has to be applied to the analysis in order to achieve high spatial resolution and low depth penetration on small mineral grains.

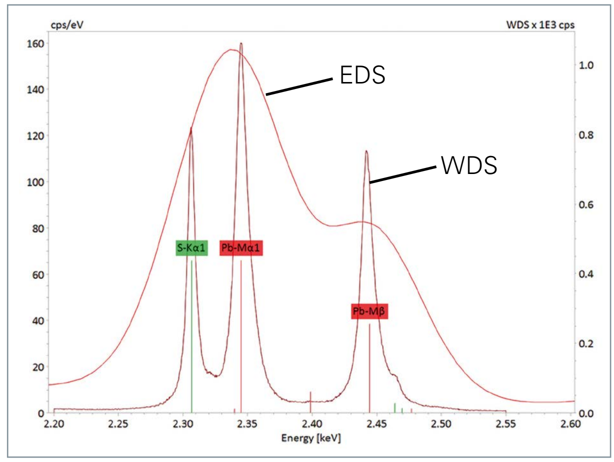



Fig. 6 X-ray spectrum section for galena (PbS) in the energy region of 2.2 - 2.6 keV showing the high spectral resolution of QUANTAX WDS compared to EDS.

## Sulfate alteration of lead acid batteries

Sulfation is an alteration process in lead-acid batteries characterized by the formation of permanent crystalline lead sulfate ($PbSO_4$) deposits. The nature, kinetics and spatial distribution of such deposits is of major interest during battery research.

QUANTAX WDS easily resolves X-ray overlaps between Pb and S to accurately determine the nature and spatial distribution of chemical phases generated during battery alteration (Fig. 7).

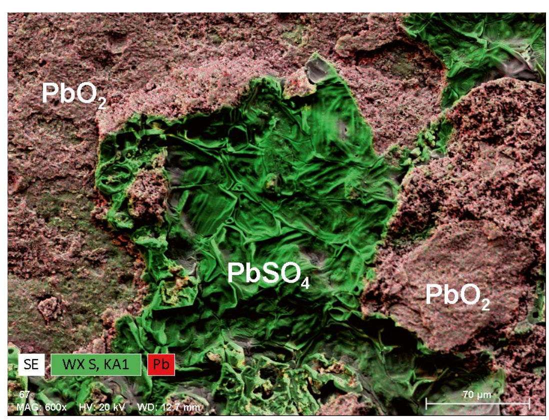



Fig. 7 X-ray element distribution map for S and Pb acquired on an electrode of a lead acid battery.

# Application Examples

### *Focus B: Analysis of Trace Elements*

## Trace element determination in stainless steel

Trace elements may have a critical influence on the properties, affecting workability and durability of steel. Since WDS achieves a 10 times higher peak to background ratio for many X-ray lines compared to EDS, trace element contents as low as 100 µg/g or below can be determined and quantified by WDS. For the present study we investigated stainless steel SRM 160b (AISI 316) with Si (0.509 wt.%) and P (0.020 wt.%) as known trace elements (Fig. 8).

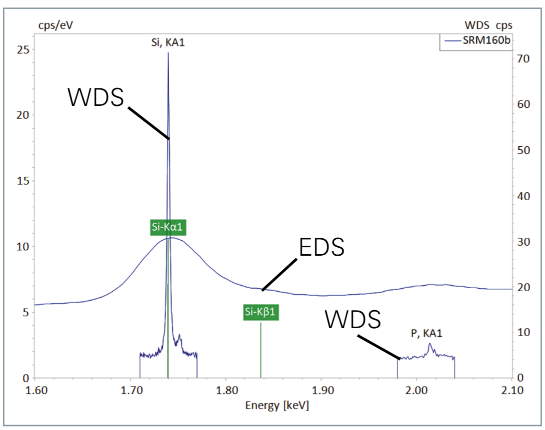



Fig. 8 Trace elements Si and P in a stainless steel standard material using energy range scans by QUANTAX WDS, Si = 0.509 wt.%, P = 0.02 wt.%.

## Trace elements in peridotite Cr-spinel

Peridotite rocks are the dominant rock type of the Earth's mantle and their composition can teach us about the mantle and crust formation during the early Earth's history.

Alpine peridotites were analyzed for major and trace elements using QUANTAX WDS. Cr-spinels occurring with orthopyroxene, altered olivine and minor clinopyroxene contain traces of 230 ppm Si and 3450 ppm Zn (Fig. 9).

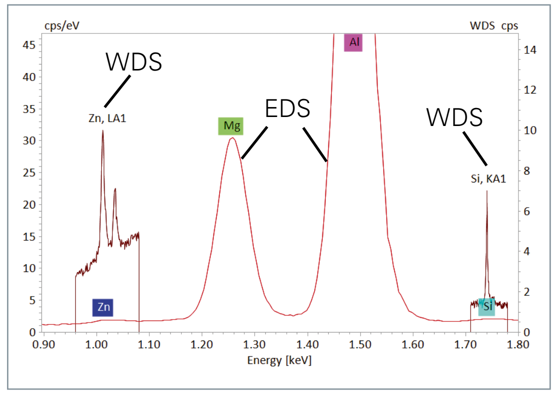



Fig. 9 Energy range scans acquired by QUANTAX WDS for qualitative identification of Zn and Si in Cr-spinel from peridotite (sample courtesy of Prof. Giancarlo Capitani, University of Milano, Italy).

## Trace element distribution in plagioclase

Mapping the distribution of the trace element Sr in a zoned plagioclase mineral helps to unravel the evolution of a volcanic complex (Fig. 10). The analytical challenge in this case is not only related to the low concentration of the element of interest (Sr < 1000 ppm), but also to the distinct EDS peak overlap of Si Kα and Sr Lα.

QUANTAX WDS is perfectly suited for mapping and in-situ analyses of trace elements in minerals. The WDS technique is indispensable especially under such analytical circumstances where overlapping major and trace element peaks have to be resolved.

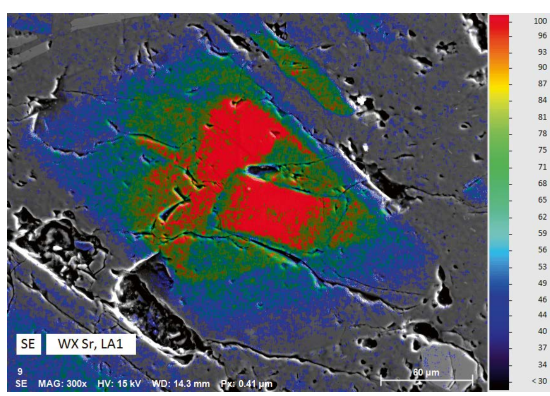



Fig. 10 X-ray element distribution map for Sr, a trace element in plagioclase. (Volcanic rock sample courtesy of Dr. Daniel Smith, University of Leicester, UK).

***Focus C: Analysis of Light Elements***

## Investigation of borides

The QUANTAX WDS system has an outstanding sensitivity for light elements, such as Be, B, C, N, O, and F. High count rates and high signal to noise ratios enable the acquisition of a map within a few minutes.

The elemental boron and carbon map of $ZrB_2$ grains in an epoxy resin matrix (Fig. 11) was acquired within 5 minutes mapping time per element using a 5 kV acceleration voltage. $ZrB_2$ is an ultra-high temperature ceramic material with a boron content of 19.3 wt.%.

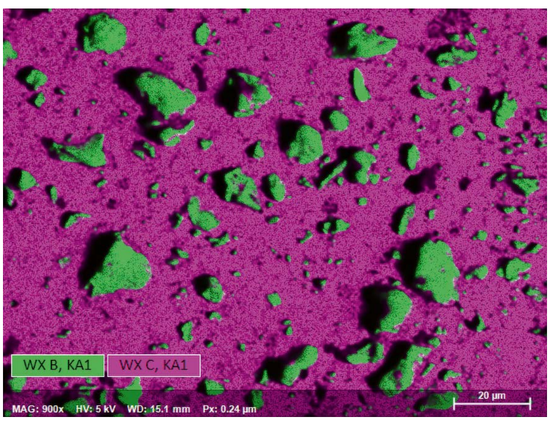



Fig. 11 Element distribution map for B (green) and C (magenta) on $ZrB_2$ grains in epoxy resin. Analytical conditions: 5 kV, 19 nA, 5 min mapping time per element.

## Quantification of nitrogen in steels

Accurate quantification of nitrogen in steel is an important topic of research since the element affects both mechanical and corrosion resistance properties. For the present study, 15 Acer inox steels with certified compositions were analyzed for N.

The results in Fig. 12 show that nitrogen even in trace element levels of a few hundred ppm in a dense matrix can accurately and precisely be determined by QUANTAX WDS.

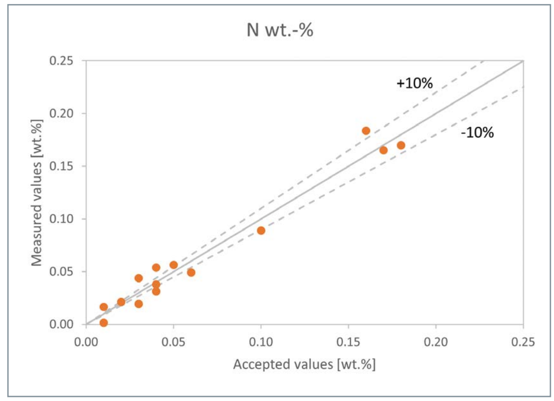



Fig. 12 The diagram shows a good correlation between the measured and accepted values for N contents in steel standards down to as low as 100 ppm.

## Carbon distribution in steels

Precisely studying the relationship between carbon concentration and the material properties of steel is of major interest to the steel producing and processing industries.

Dual phase steel may show variations in the carbon content which are directly related to the two phases martensite and ferrite. QUANTAX WDS is a perfect analytical tool to determine trace amounts of carbon in the shortest analytical times.

The map shown in Fig. 13 was acquired in five minutes. It shows martensite in green (relatively high carbon content of 0.3 wt.%) and ferrite in red (relatively low carbon content of 0.01 wt.%).

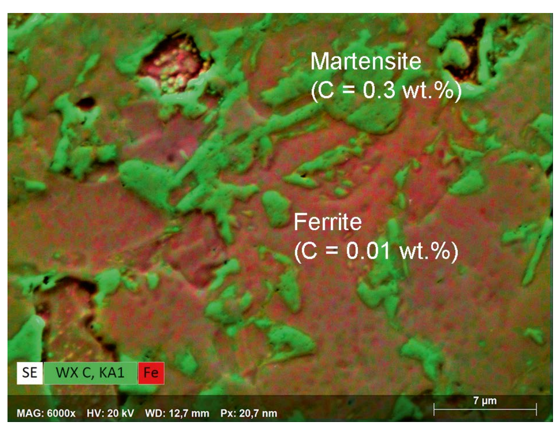



Fig. 13 Carbon distribution in a dual phase steel mapped with QUANTAX WDS (sample courtesy of TU Eindhoven).

# Technical Specifications

- XSense parallel beam WD spectrometer with the finest grazing incidence collimating optics
- Distortion-free non-magnetic optics, fully retractable
- Up to six diffracting crystals: 200 Å, 80 Å, 60 Å, (optional 30 Å) multilayers, TAP and PET
- Energy range covering 70 eV to 3,600 eV dedicated to low energy applications
- Element range covering Be (4) to U (92)
- Energy resolution of ≤ 4.6 eV for Si Kα and ≤ 15 eV for C Kα
- Count rates of ≥ 310 cps/nA for Si Kα on PET and ≥ 900 cps/nA for C Kα on BRML80
- Full functionality using low vacuum conditions
- Unique secondary optic for superior background resolution at X-ray energies ≥ 2 keV
- Ingenious automatic optic alignment system with fully motorized advanced kinematics
- P10 proportional counter with unique gas flow and pressure control
- Fully integrated in the ESPRIT software for spectrometer control, qualitative and quantitative analysis, line scan and mapping
- Seamless integration with Bruker EDS, EBSD and micro-XRF on the same SEM
- Low weight of 18 kg
- Compact design with slim optics mount
- Flexible adaptation to either a WDS or EDS SEM port


**Bruker Nano Analytics**

Headquarters Berlin · Germany
Phone +49 (30) 670990-0
Fax +49 (30) 670990-30
info.bna@bruker.com

**www.bruker.com/quantax-wds**