BRUKER NANO WEBINAR

Fundamentals and characteristic applications of a Parallel Beam Optics-WDS on SEM

Bruker Nano Analytics, Berlin, Germany

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Presenters

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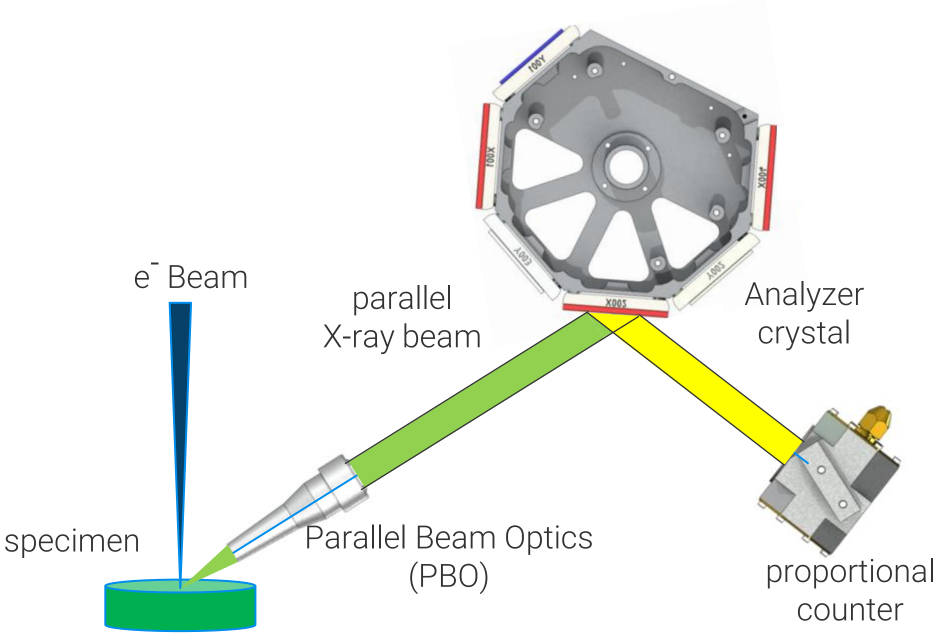
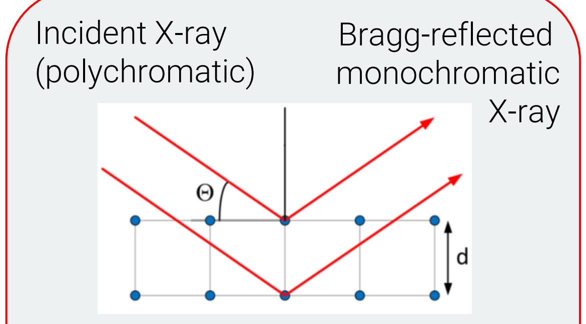
Outline of the presentation

WD Spectrometer
A brief introduction

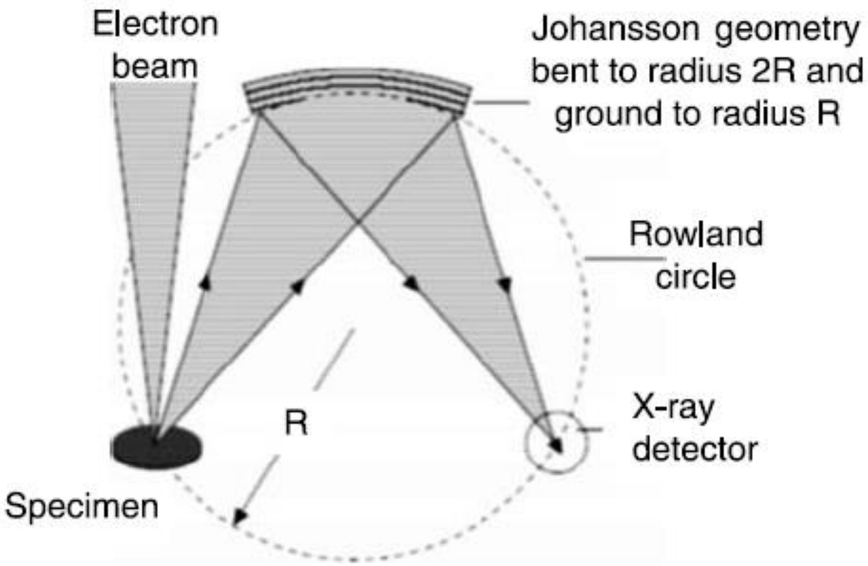
Parallel Beam WD Spectrometer (XSense)





Bragg equation: $n\lambda = 2d\,\sin(\theta)$

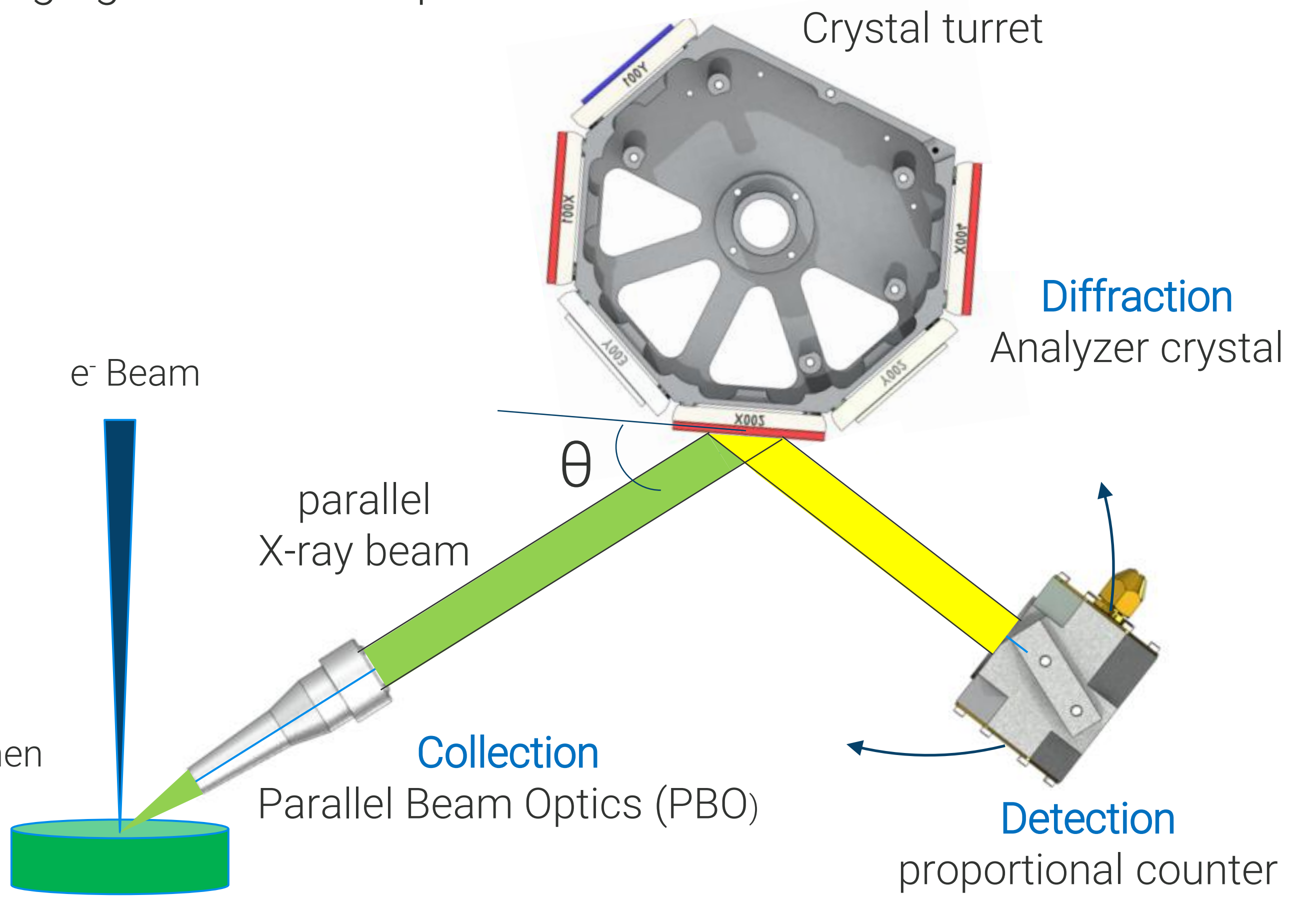
Rowland Circle based WD spectrometer



- WD spectrometer are based on Bragg's Law of diffraction
- Sample X-rays are diffracted by a crystal and those diffracted X-rays are counted by a proportional counter
- Geometry of the WD spectrometer will be adjusted by changing the angle of the diffractor relative to the sample
- This angle determines the X-ray energy that satisfies Bragg's Law

XSense (PBO) WD Spectrometer Working Principle

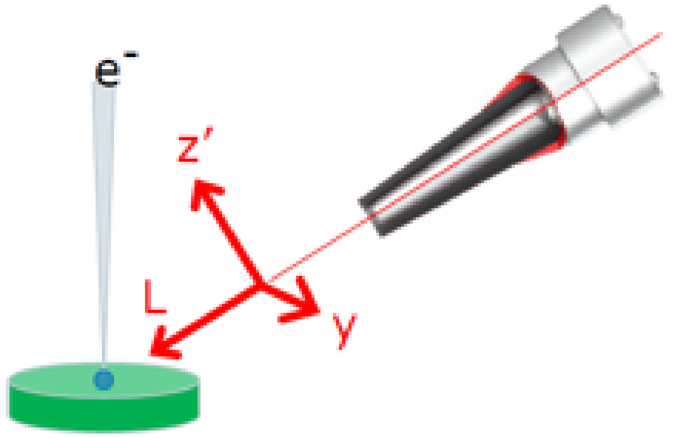
- In Parallel Beam Optics (PBO) WDS systems, an X-ray optic (mirror optic) is placed near the sample and transforms X-rays diverging from the sample into a parallel beam
- This results in:
  - A high solid angle for all X-ray energies
  - more compact spectrometer
- Bragg diffraction takes place at flat analyzer crystal (crystal turret)
- Angle Θ between beam and crystal surface and crystal lattice constant 2d determine the energy that passes through to the detector
- Detector rotates in order to set the right reflection angle to satisfy Bragg's Law
- X-ray detection with flow proportional counter



XSense Technical Design Advantages
Auto-Aligning Optics

- In PBO WD spectrometer, the optic must be aligned properly with the sample at the proper WD so that the parallel X-ray beam is incident on the diffractor
- Deviations from the proper WD can result in significantly reduced count rates, peak shape deformations or degraded resolution
- Auto- optic alignment system is fully integrated within Bruker ESPRIT SW and allows to compensate deviation in sample positioning from the proper WD by automatically adjusting the angle of the optic vertically and horizontally

Auto- alignment capability and the more compact design of a PBO WD Spectrometer allows the installation either on an EDS or WDS port.



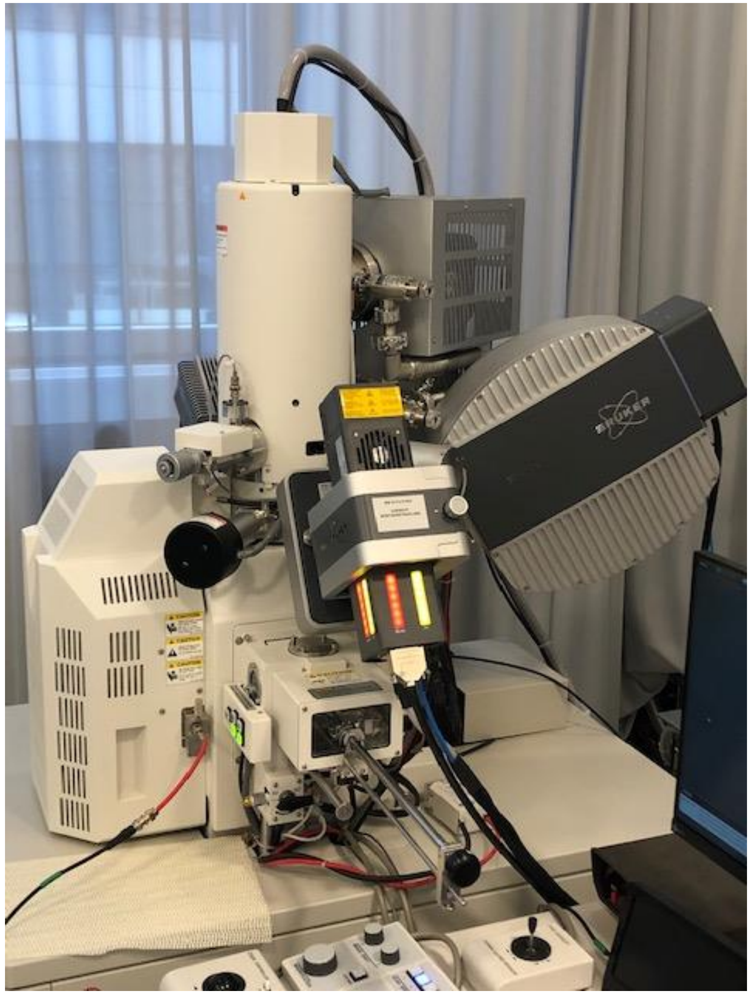
3-axis optic positioning unit:

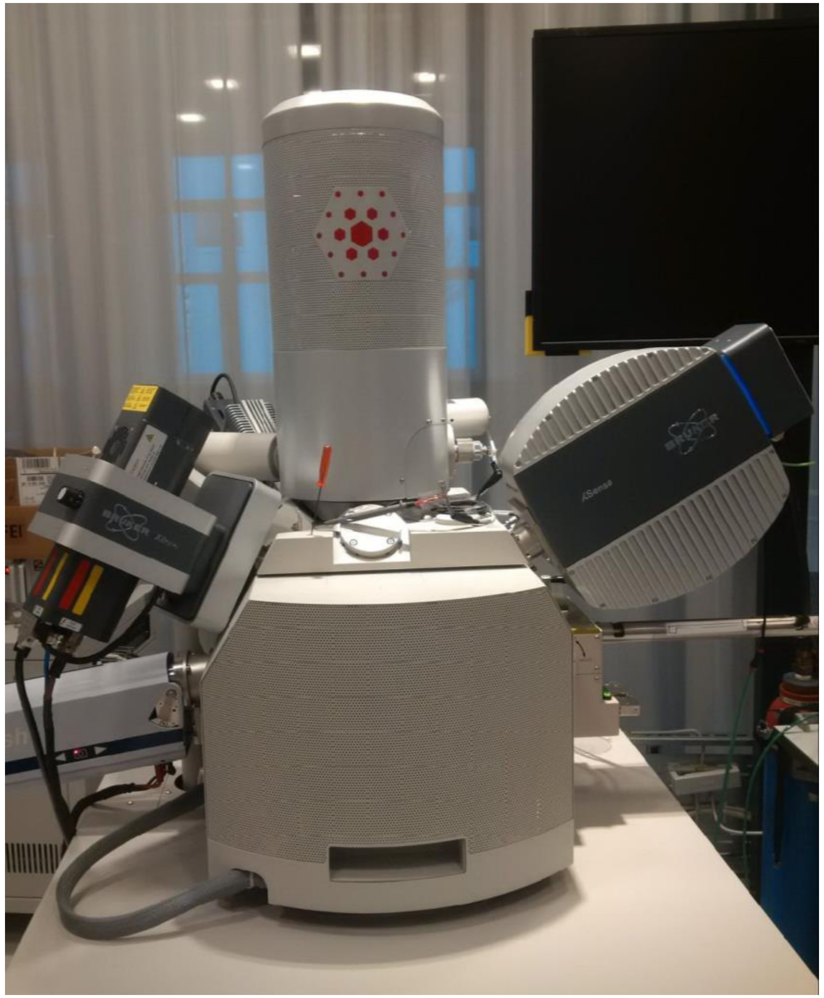
- ±1mm adjusting range for the y- and z'-axes, resolution <1µm
- 6mm adjusting range for the L-axis, resolution 1.25µm
- Full optic retraction

Auto-alignment algorithm:

- Fast and accurate optimum position estimation

QUANTAX WDS can be mounted on a WDS- or EDS-port





EDS – WDS comparison XSense specs

EDS – WDS comparison

- From the physics there is no substantial difference between EDS and WDS, just the different detector abilities
- Energy dispersive and wavelength dispersive X-ray spectroscopy are complementary techniques, where:
  - EDS is measuring all elements simultaneously (fast)
  - WDS is measuring elements sequentially (slow)
  - Standard-based quant is a must for WDS
  - WDS has much higher peak/ background ratios, important for detection of low concentration in the ppm range
  - WDS provides much better energy resolution which is important for resolving peak overlaps

XSense (PBO) WD spectrometer specs:

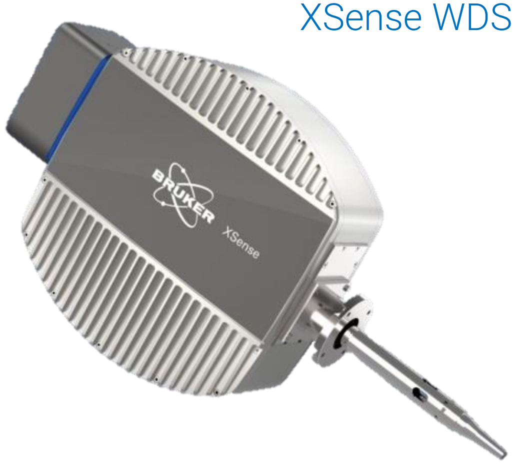
- Energy resolution: 4 eV for SiKa
- Energy range: 70 eV – 3.6 keV (K-lines: Be – Ca; L- lines: Al – Sb; M –lines: Zr – U; N –lines: Tb – U)
- Concentration range: down to 100 ppm (enhanced P/B-ratio)
- Large solid angle
- Outstanding sensitivity for (very) light Z- elements (Be, B etc.)

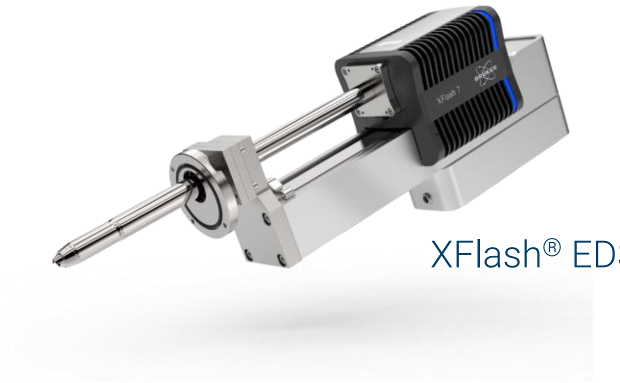
QUANTAX WDS - APPLICATIONS

Characteristic applications for PBO-WDS

Advantages of the PBO-WDS for applications

XSense WDS





XFlash® EDS

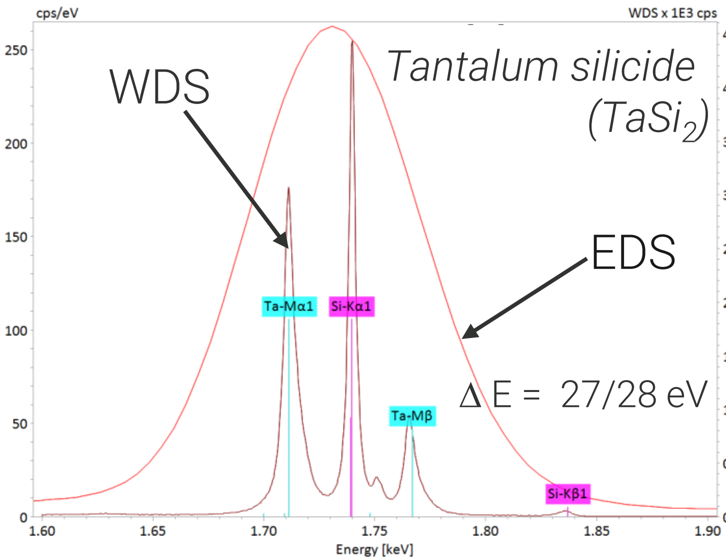
Compared to EDS the WDS shows:

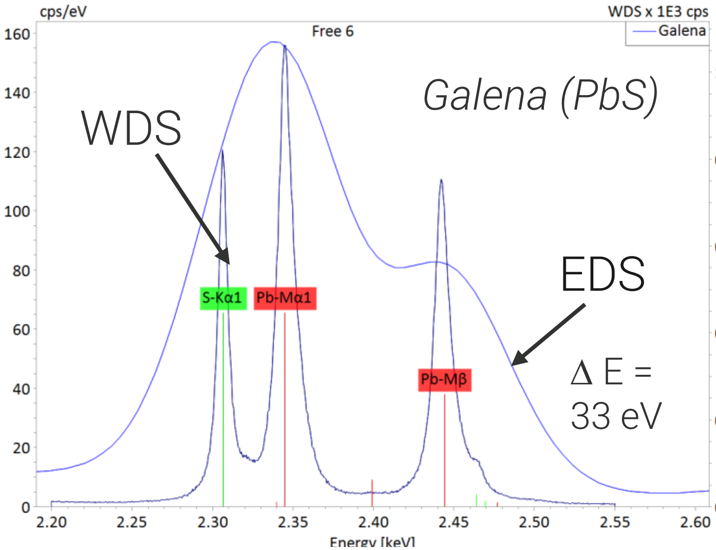
- substantially higher spectral resolution
- enhanced P/B-ratios, therefore lower detection limits
- outstanding sensitivity for light elements

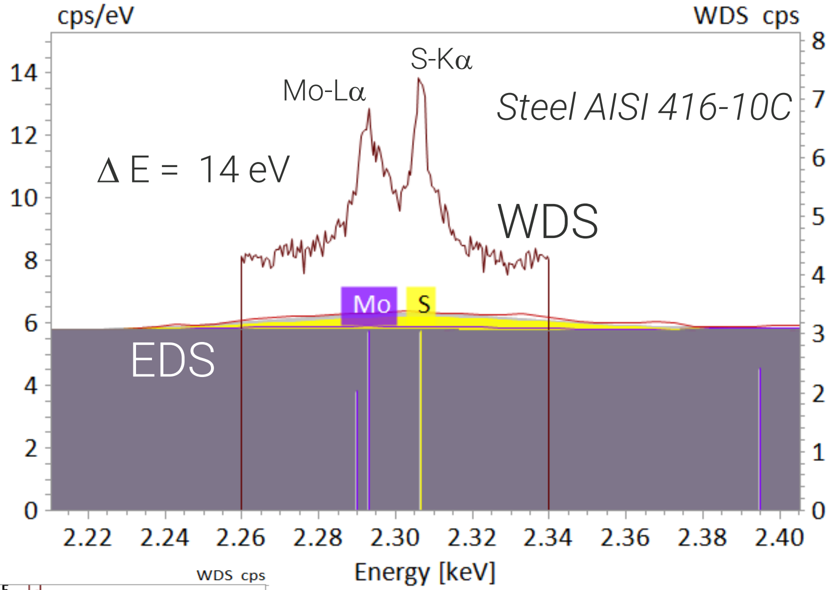
WDS is an ideal technique to complement EDS in demanding applications

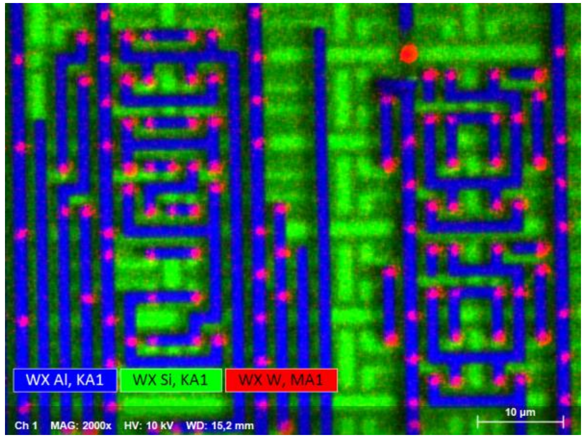
Spectral resolution

Resolution of EDS-peak overlaps in various materials



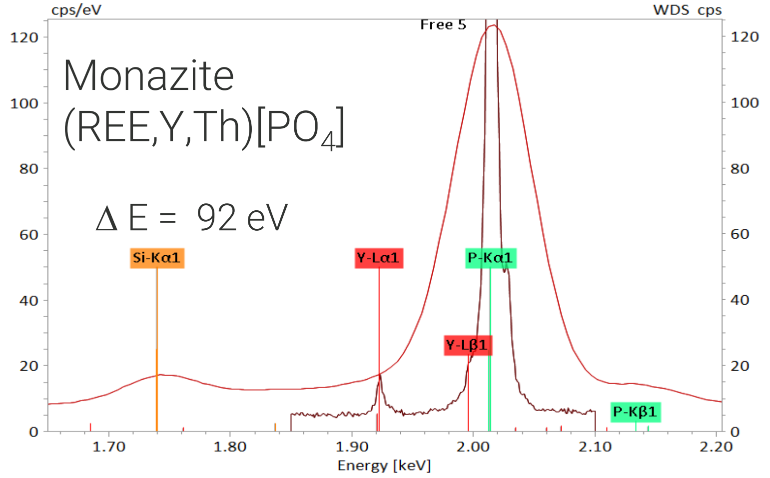






MOSFET

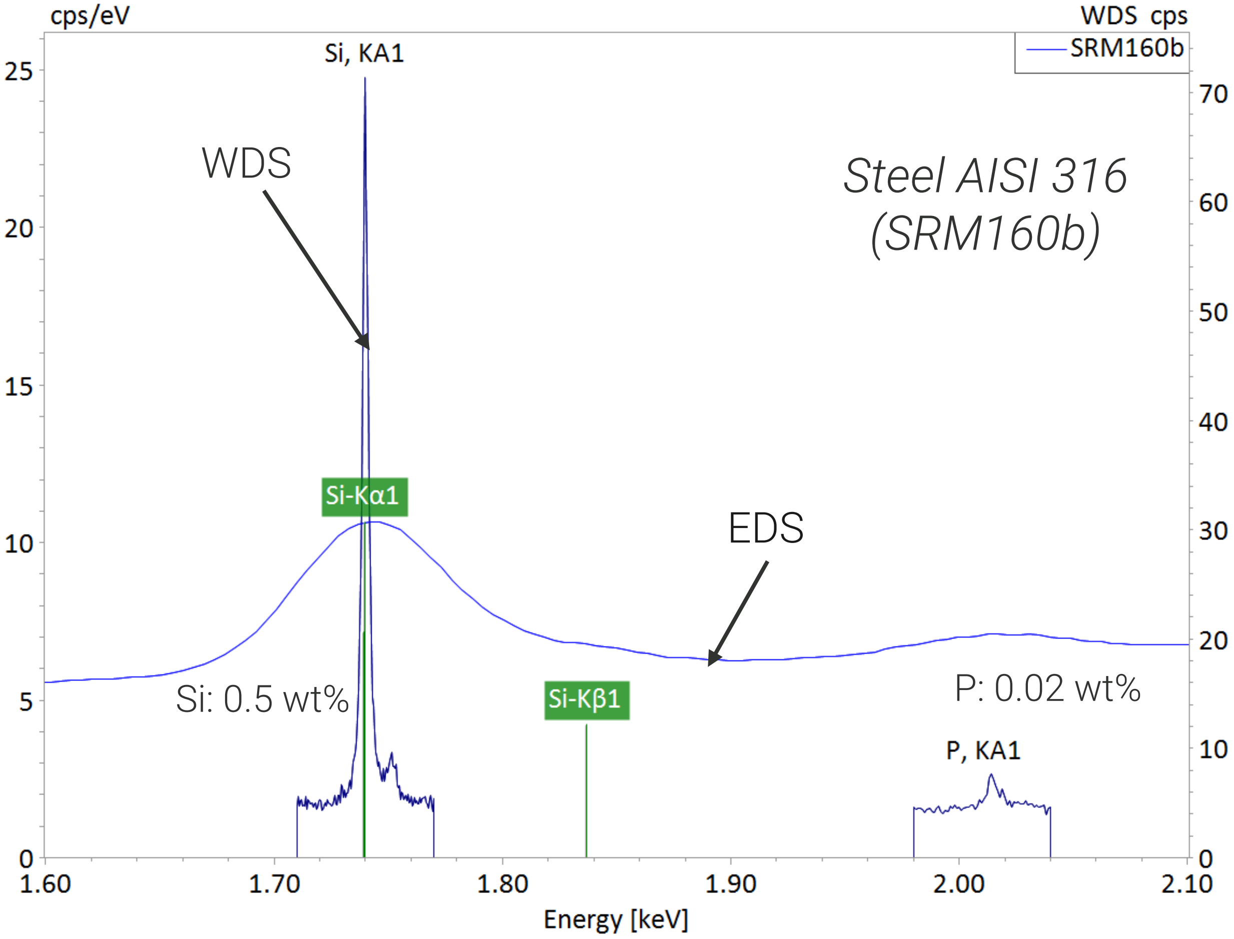
Red: W
Green: Si
Δ E = 35 eV

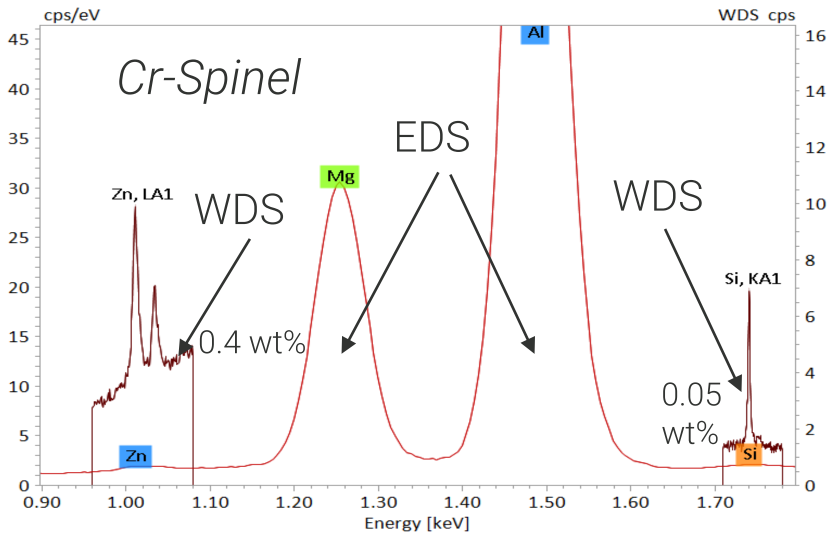


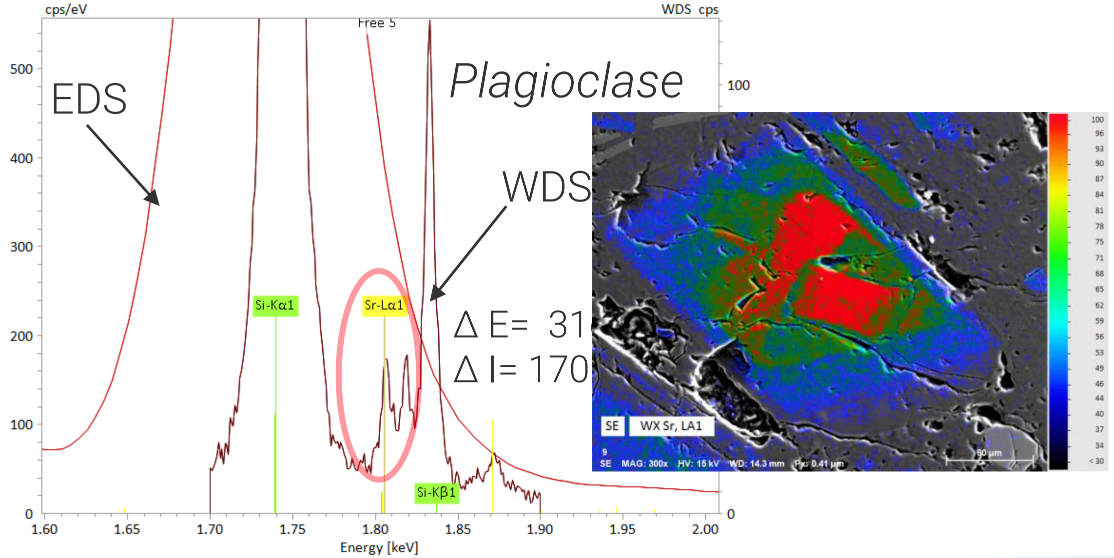
QUANTAX WDS - APPLICATIONS

Trace elements

Trace elements in various materials



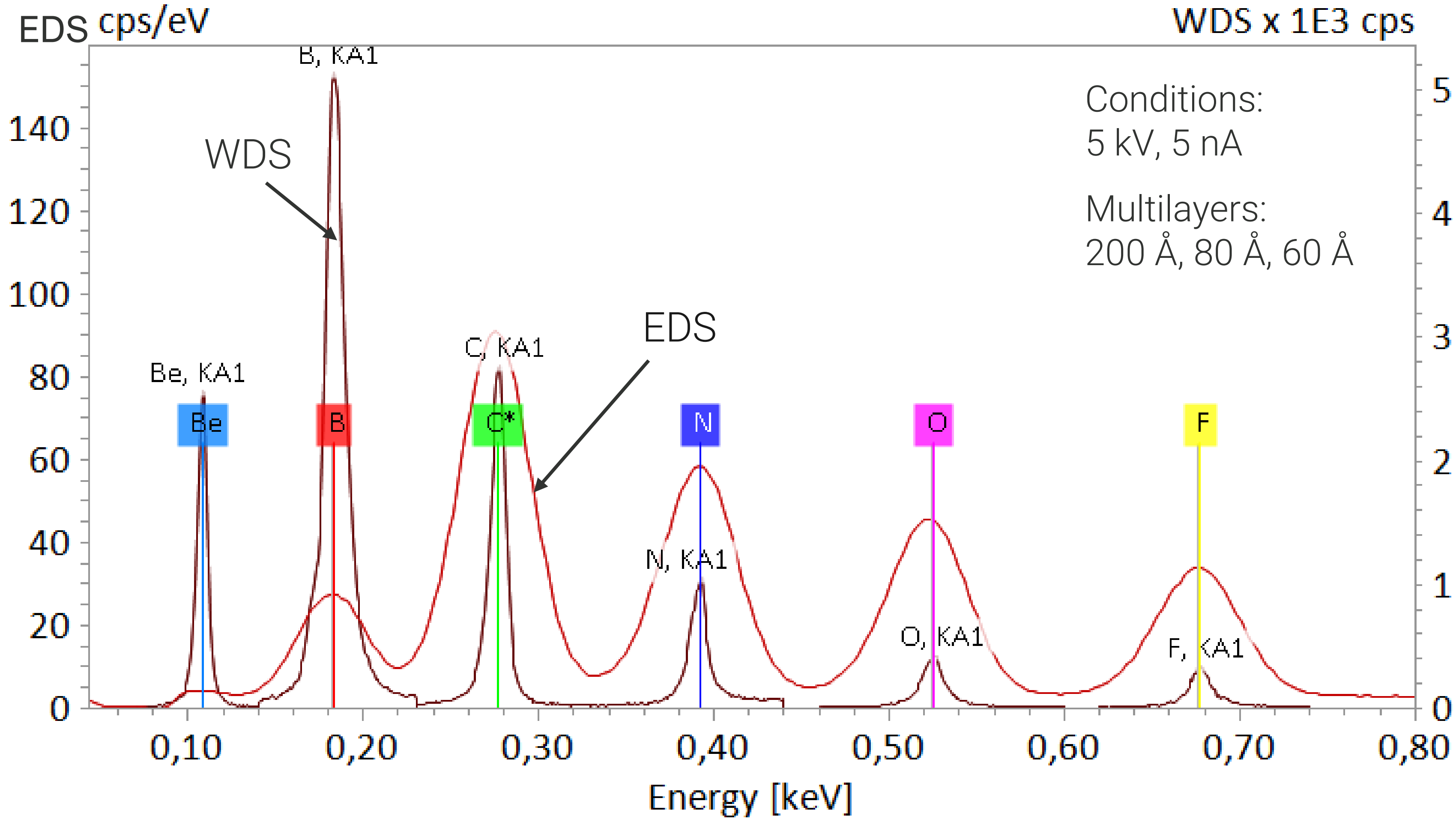


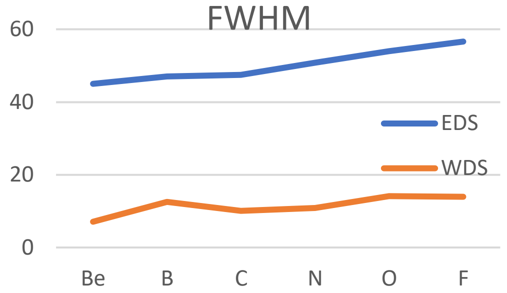


QUANTAX WDS - APPLICATIONS

Light elements

Light element spectra acquired by EDS and WDS





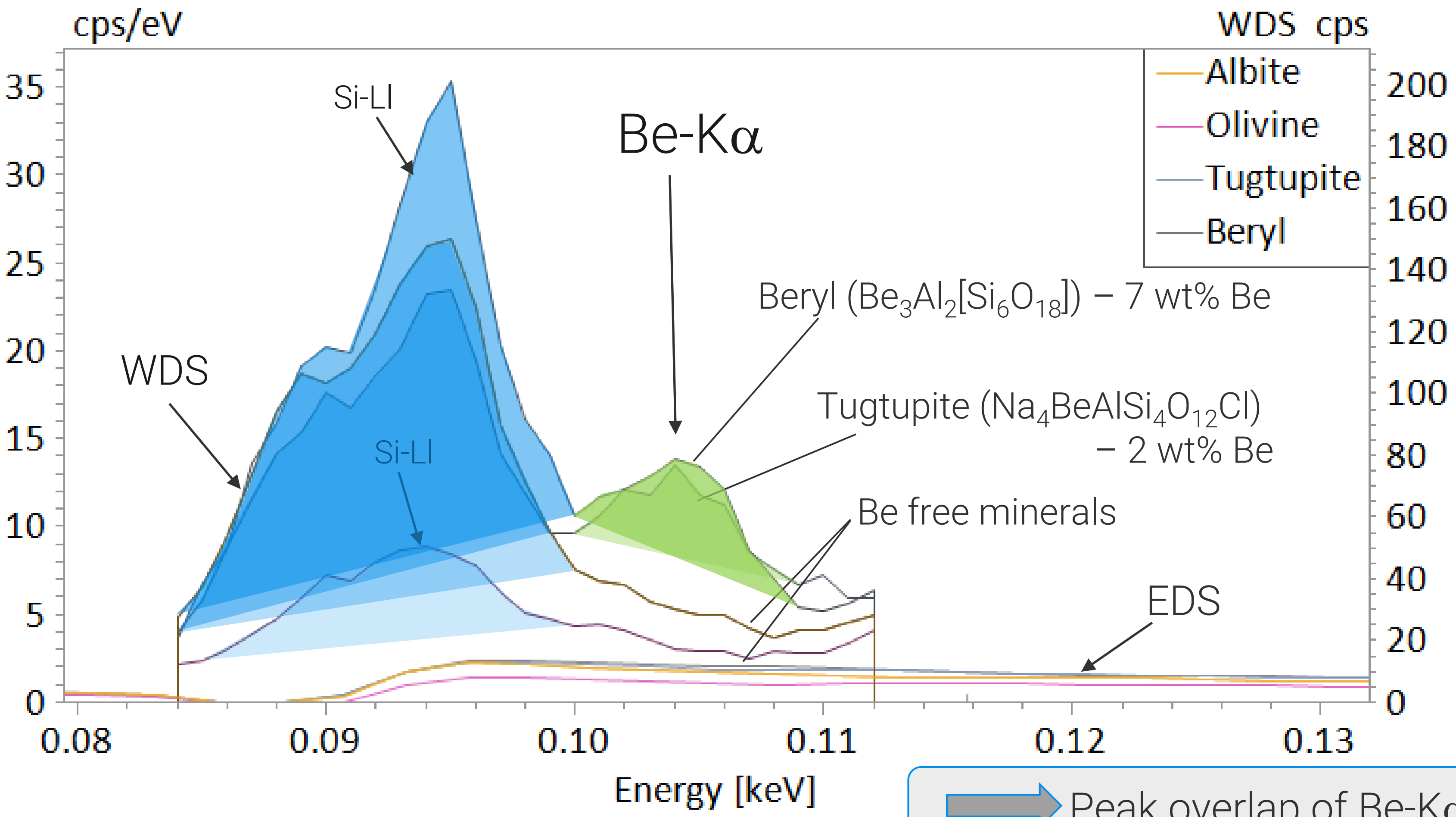
Samples:

Pure elements: Be, C and compounds: BN, SiO_2 CaF_2

WDS shows higher sensitivity, higher peak/background ratios and better spectral resolution

Beryllium-minerals vs. Be-free minerals

- WDS energy range scan for Be-Kα

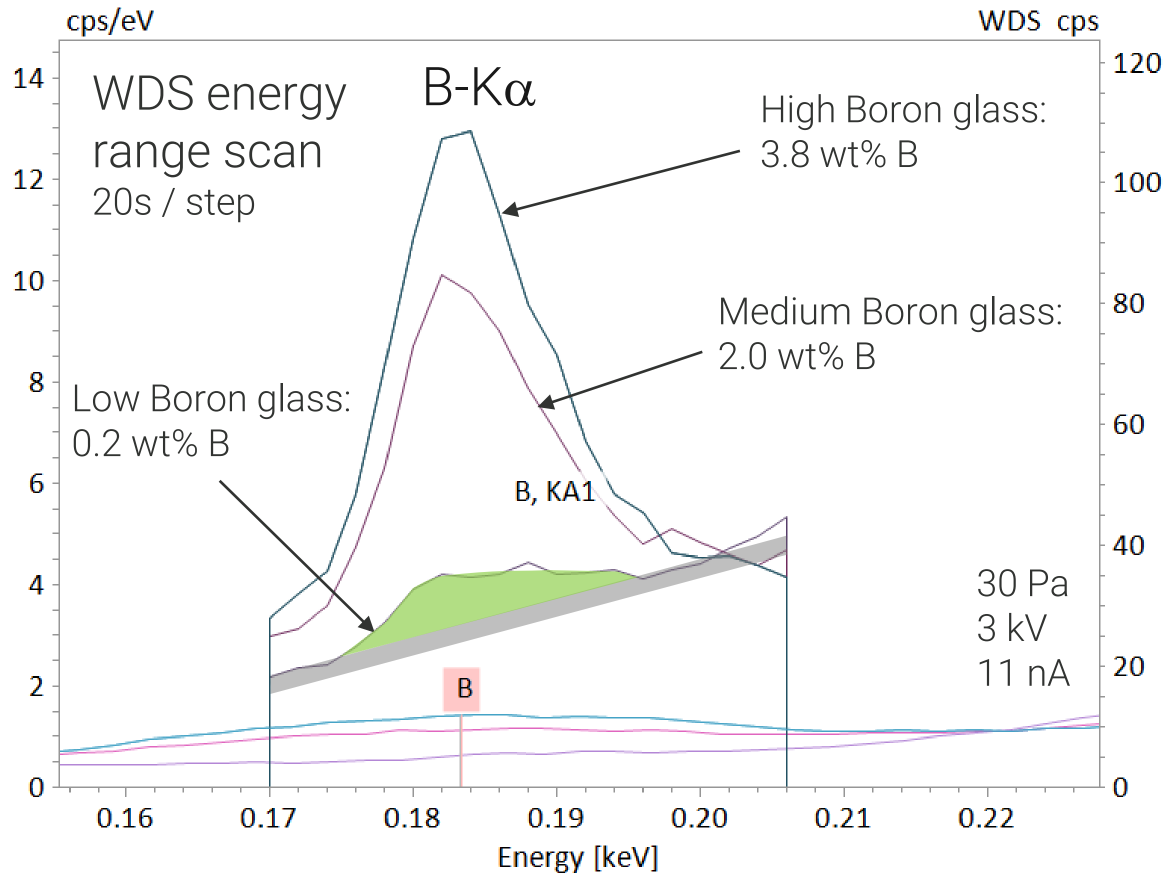


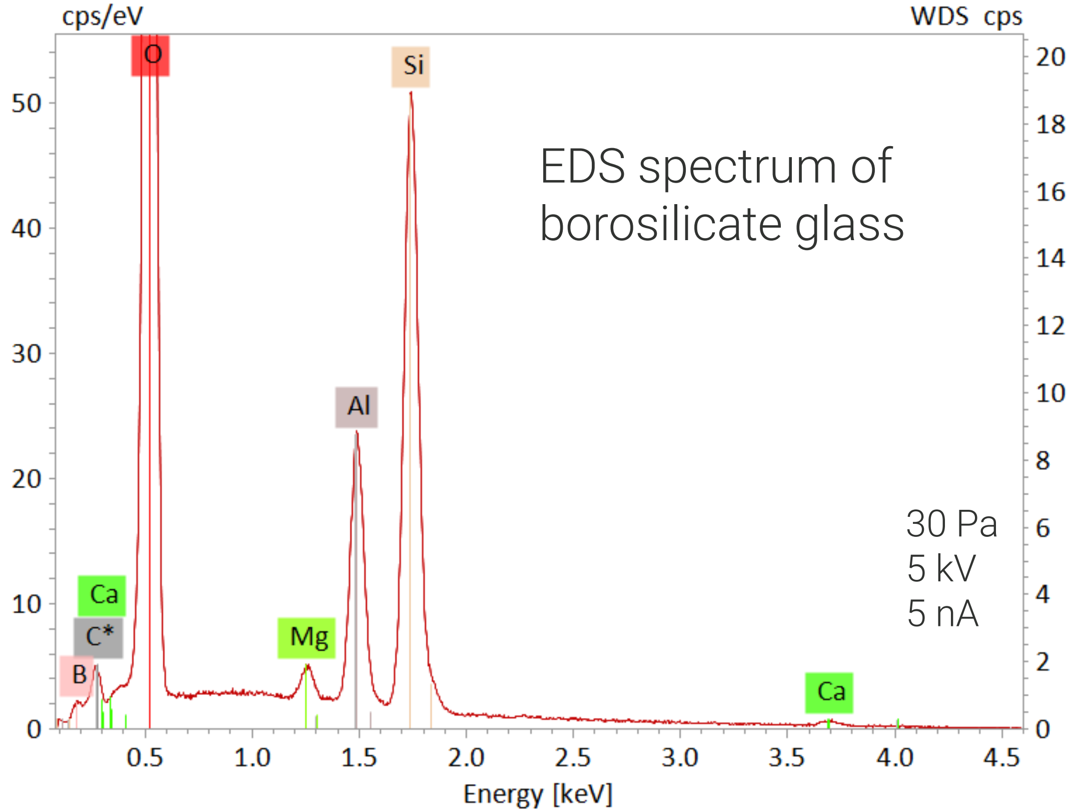
5 kV, 17 nA
Dwell time:
10 s / step

Peak overlap of Be-Kα and Si-L is resolved

Boron in glasses

- Low-vacuum analyses of uncoated glasses

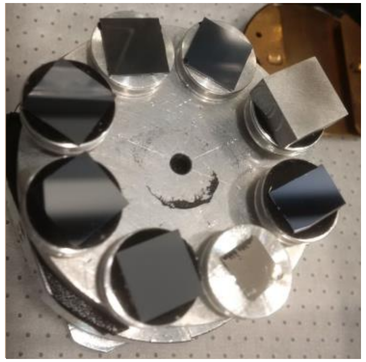


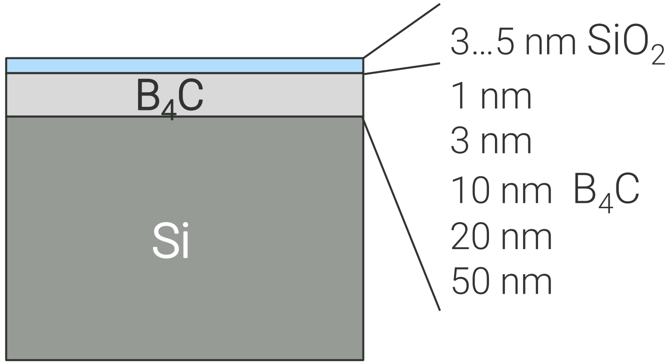


WDS can successfully quantify low B contents in glass

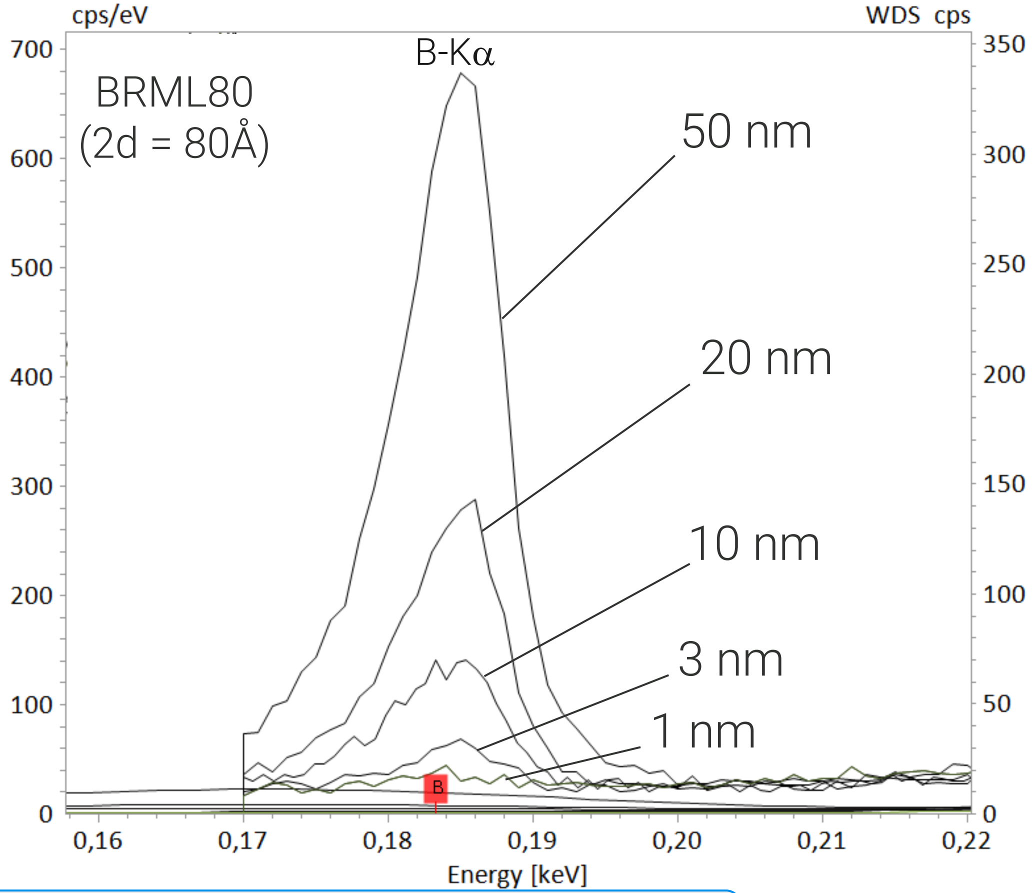
Boron in layered samples

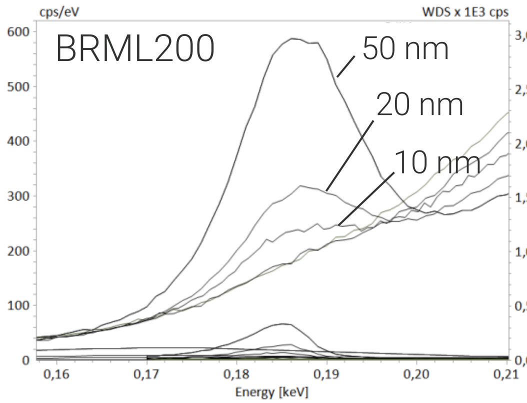
- Detection of Boron in thin B_4C layers

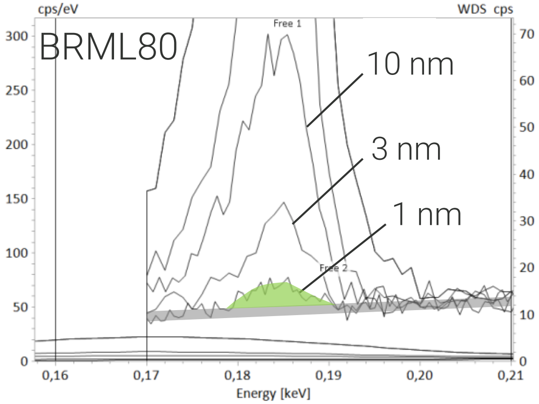




5 kV, 3.5 nA, 5 s/eV



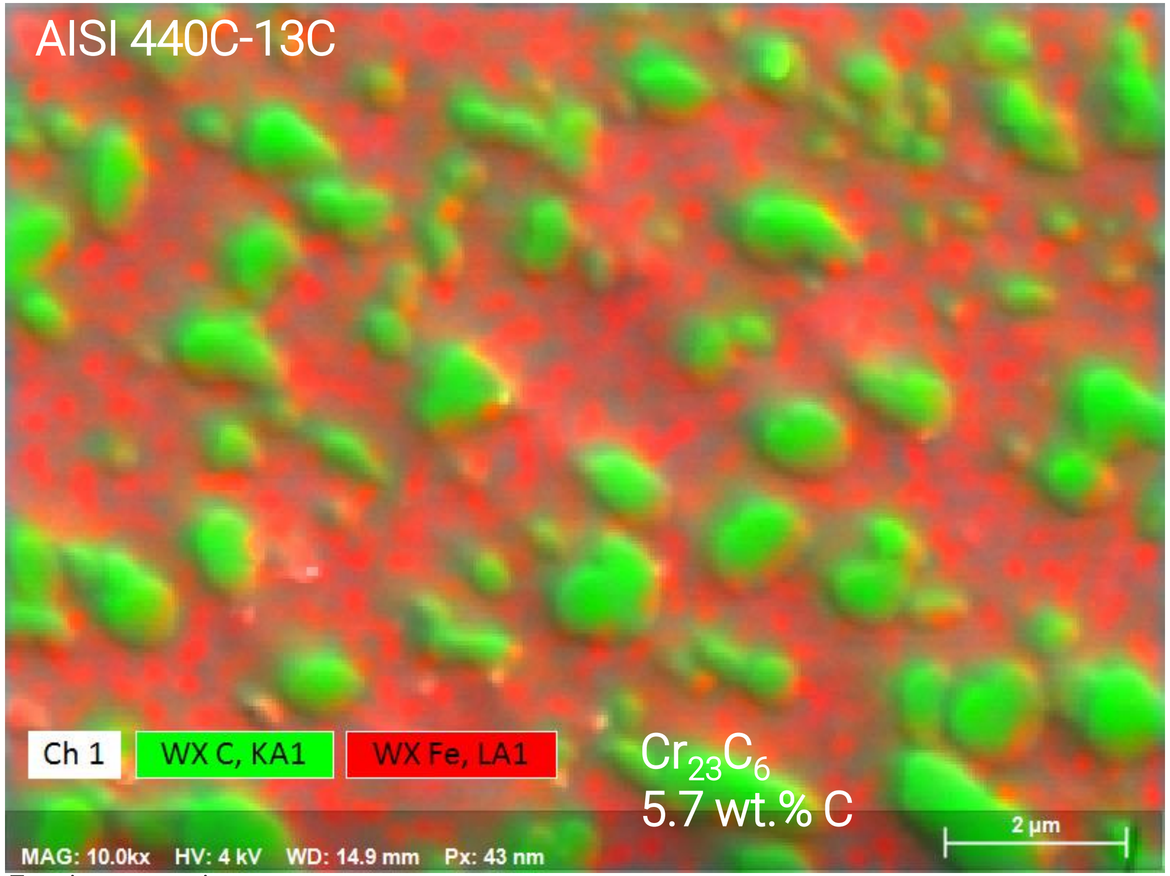




Evidence for Boron down to the thinnest layer

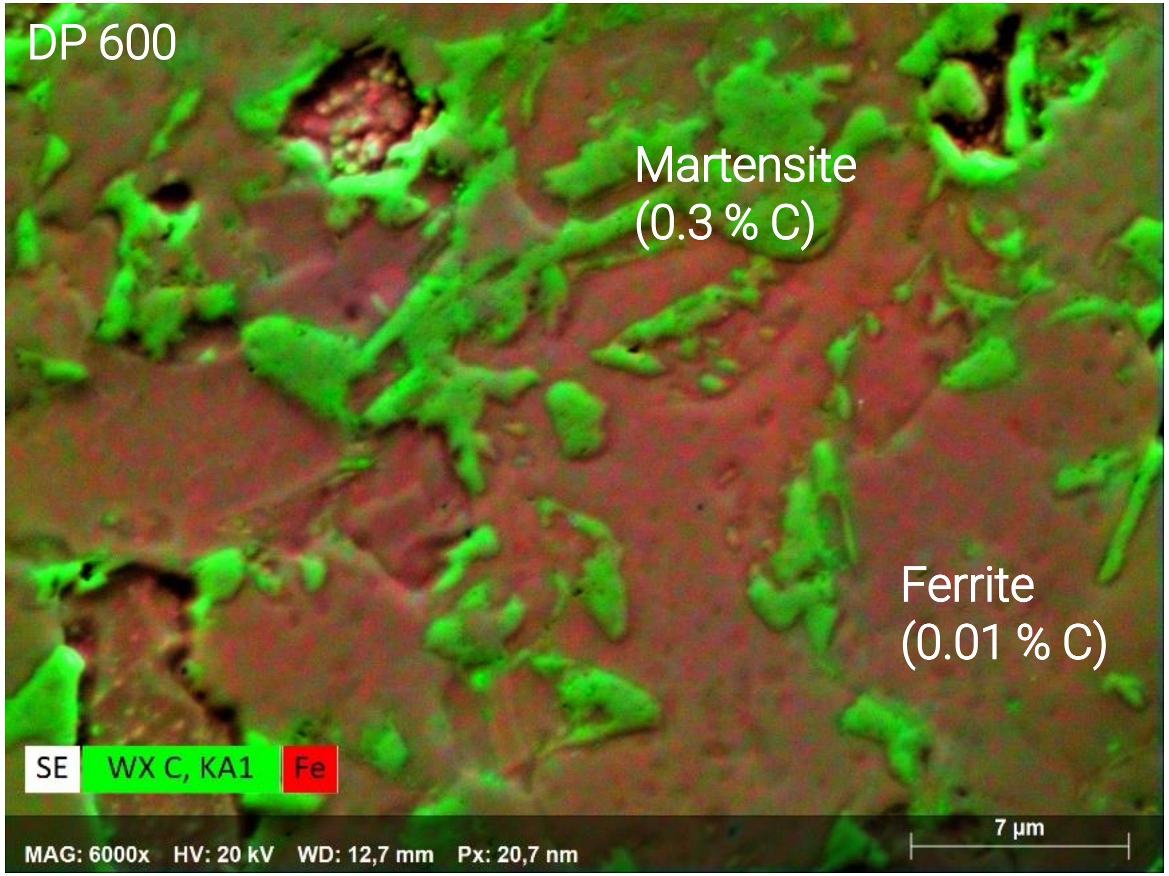
Mapping carbon distribution in steel

- Carbide bearing steel



5 min mapping

- Dual phase steel

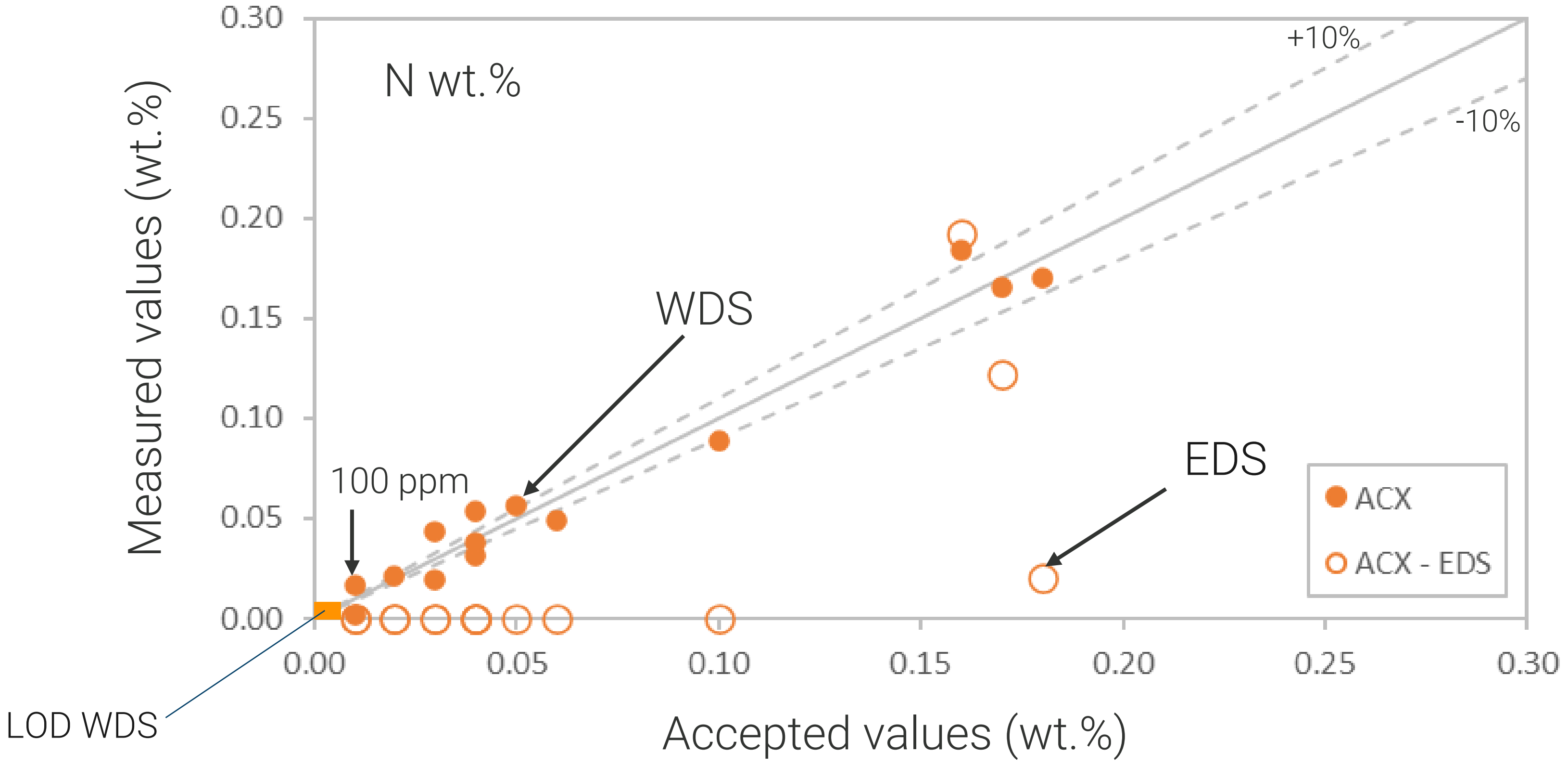


Sample: TU Eindhoven – 5 min

→ Fast mapping is possible and preferred to minimize contamination

Nitrogen in steel

- Trace element quantification on certified reference steel samples



Samples:

15 certified steels (Acerinox) with nitrogen 100 – 1800 ppm

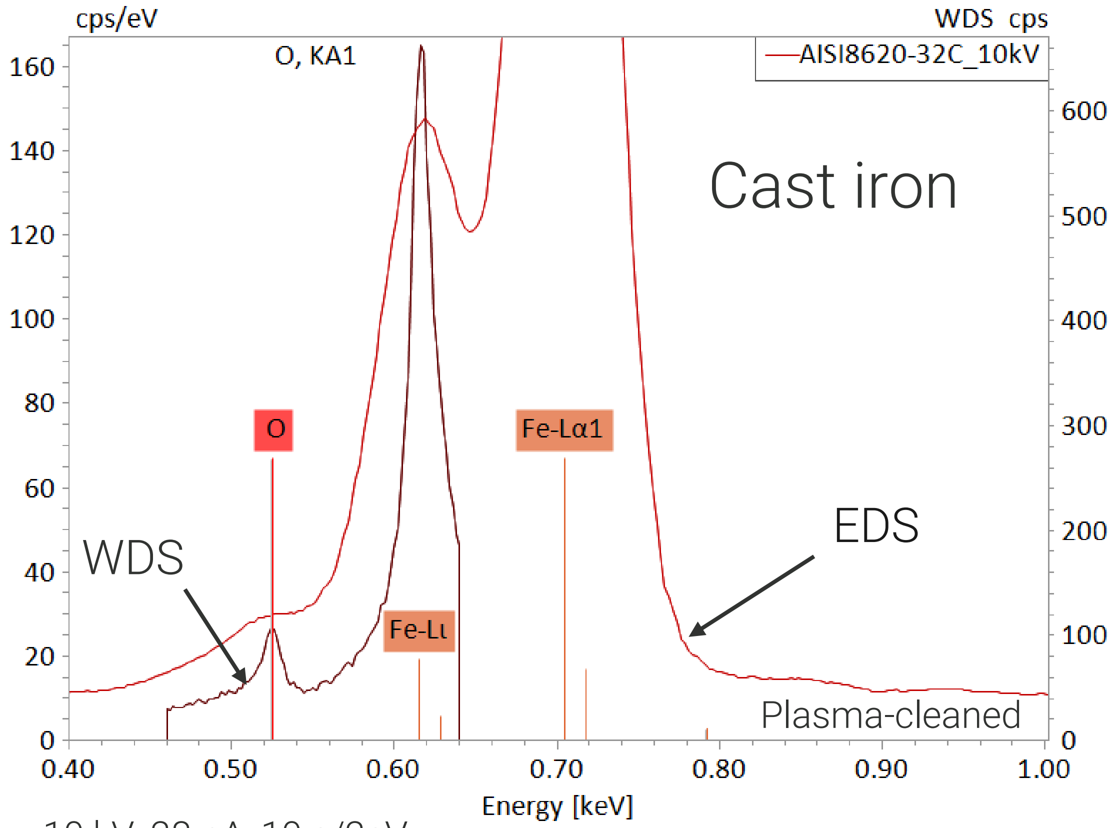
Parameter:

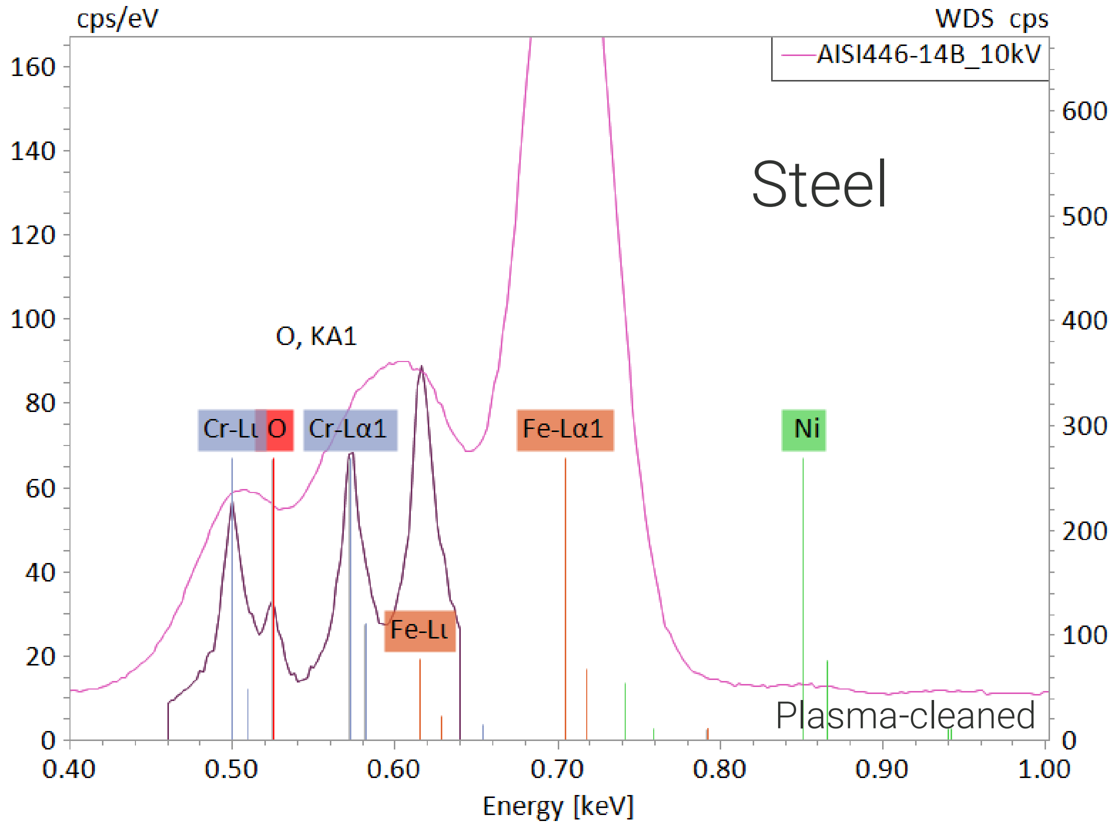
5kV, 10nA, 60s on peak

WDS has lower limit of detection (LOD) than EDS

Oxygen in cast iron and stainless steel

- Scan for O-Kα in samples with and without Cr





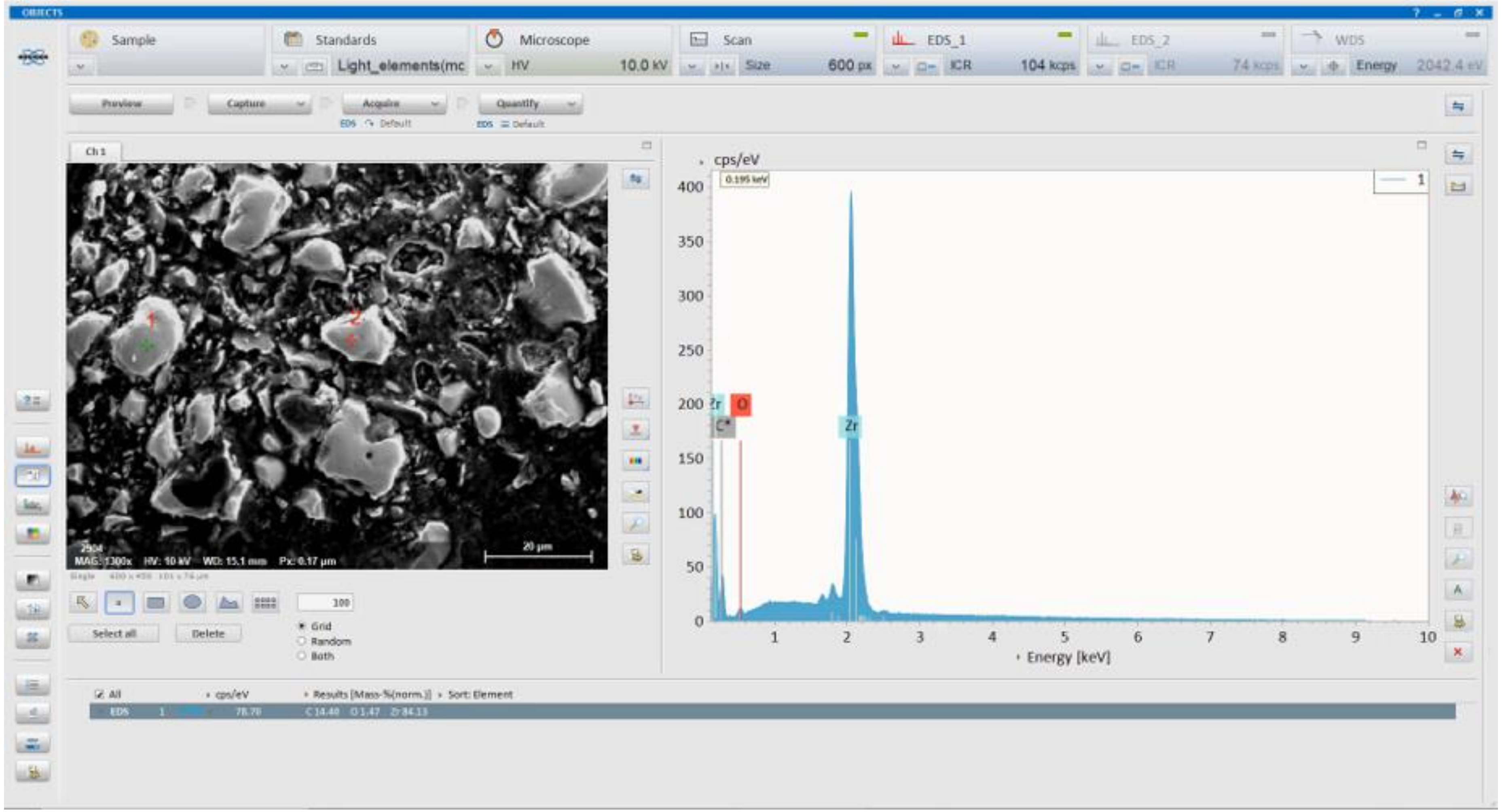
10 kV, 28 nA, 10 s/2eV

➡ WDS resolves peak overlap of O-Kα and Cr-L lines

Element identification and mapping by PBO-WDS

Sample:

ZrB_2 in epoxy resin



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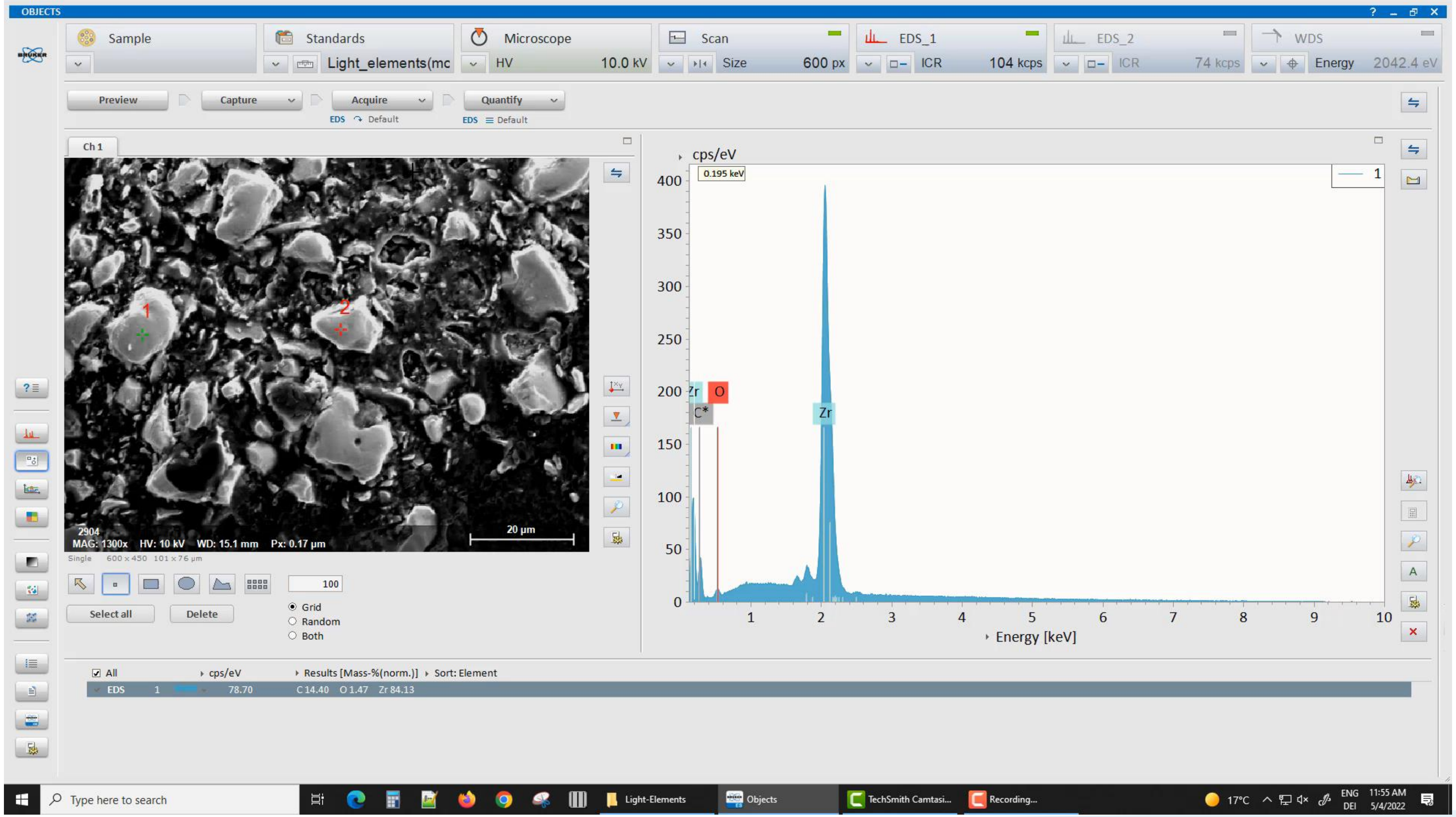

Summary and Conclusions

QUANTAX WDS

- High spectral resolution
  - Resolving peak overlaps
  - Resolution instead of deconvolution
- Trace element determination
  - Low detection limits
  - Traces as low as a few 100 ppm and below
- Light element analyses
  - High sensitivity for light elements
  - Including Be, B

Thank you!

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Questions and Answers

Are There Any Questions?

Please type in the questions you might have

in the Q&A box and press *Send*.

Innovation with Integrity