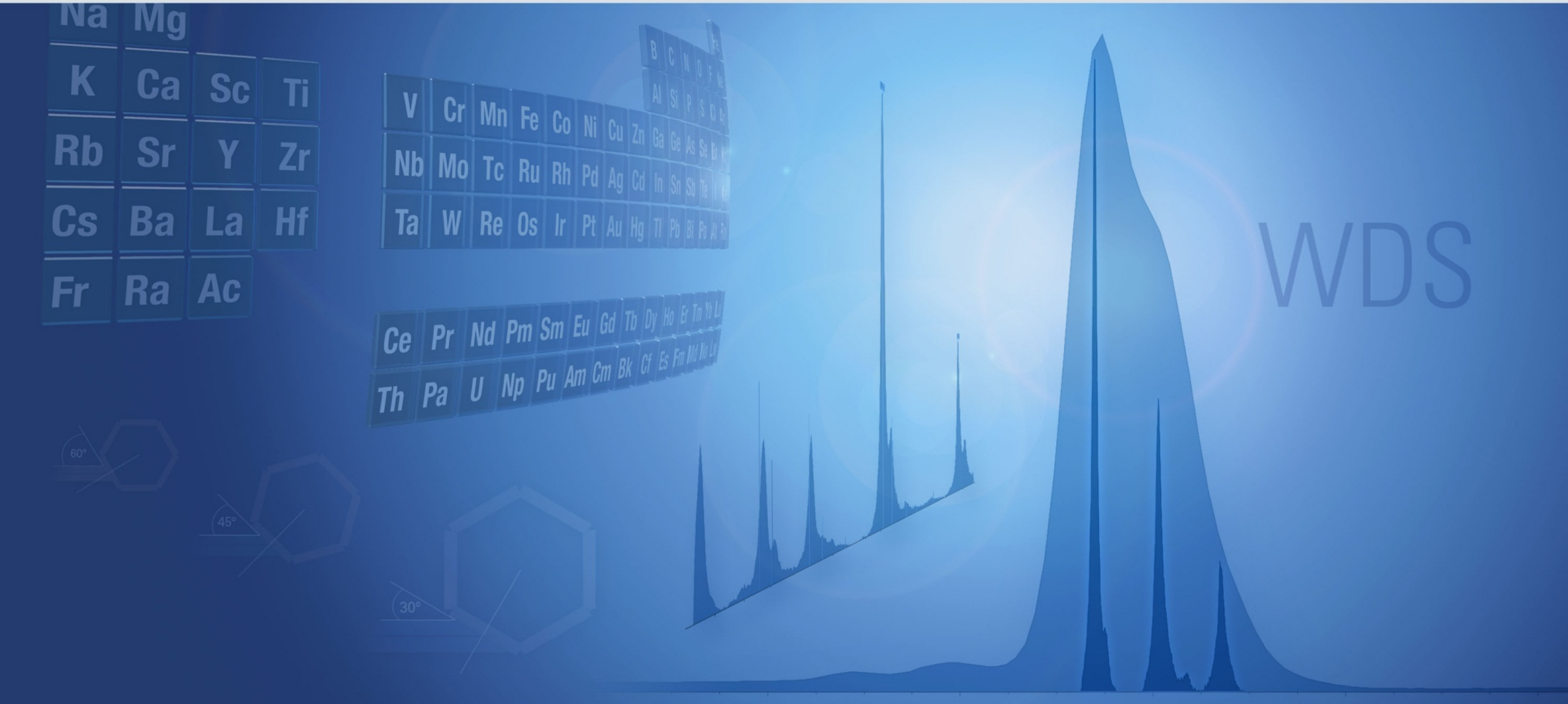
Microanalysis with high spectral resolution: the power of QUANTAX WDS for SEM

Bruker Nano Analytics, Berlin, Germany
Webinar, September 14, 2017



Presenters

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Bruker Nano Analytics, Berlin, Germany

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Bruker Nano Analytics, Berlin, Germany

Microanalysis with high spectral resolution: the power of QUANTAX WDS for SEM

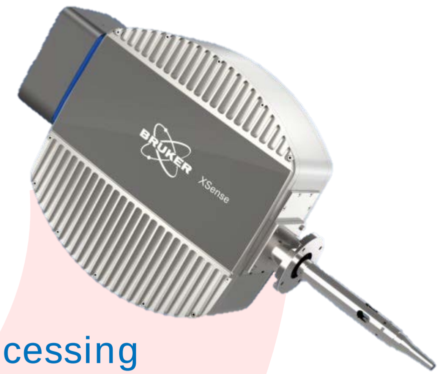
OUTLINE:

- QUANTAX WDS – an overview
- The XSense™ spectrometer: working principle and spectral resolution
- EDS vs. WDS: the need for high spectral resolution
- Sample measurement data and application examples focusing on high spectral resolution

QUANTAX WDS
System Components

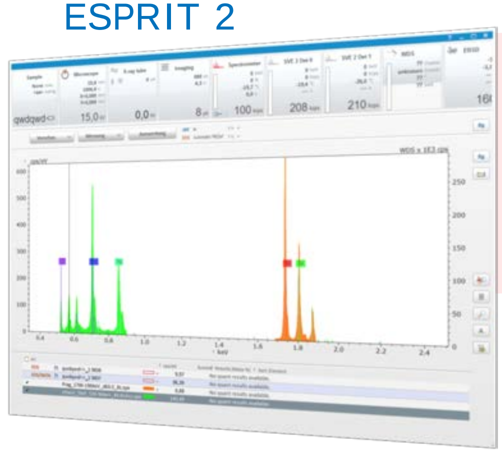
QUANTAX WDS: integral part of the QUANTAX family

XSense™ WD spectrometer



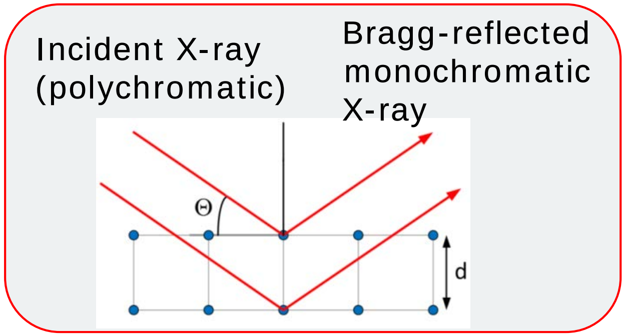
Signal processing unit SVE 6

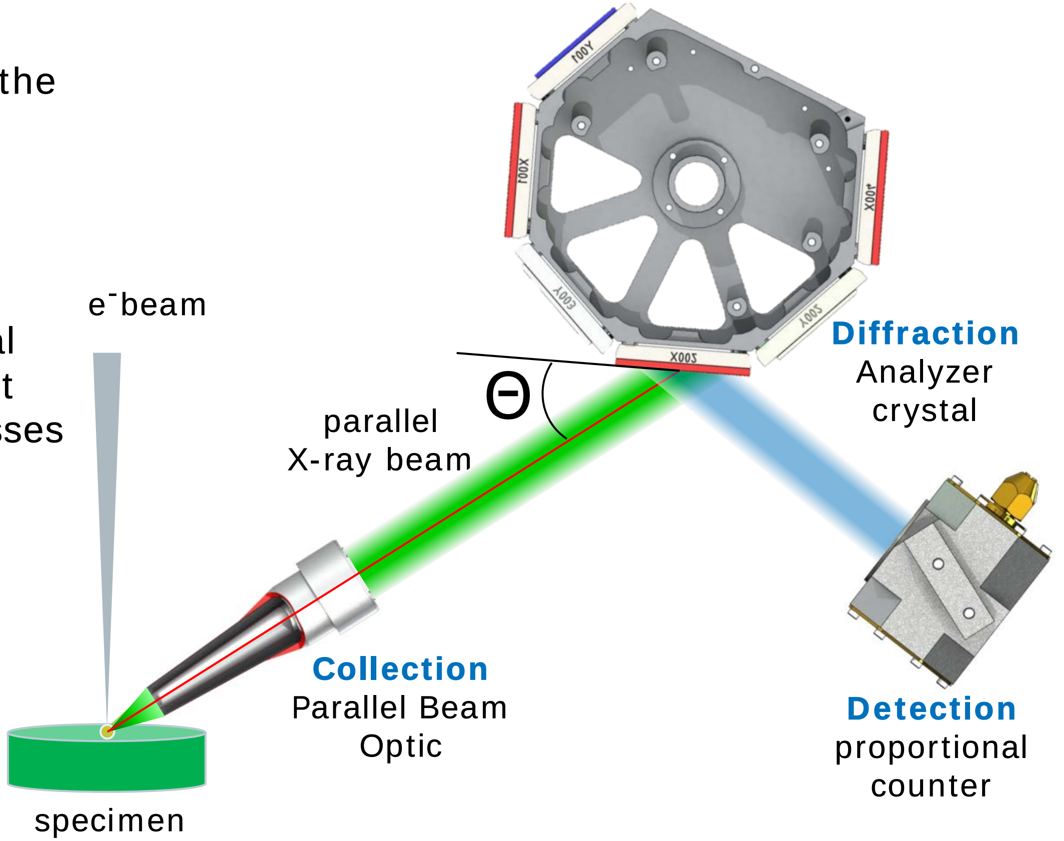
ESPRIT 2



XSense WD Spectrometer
Setup and Working Principle

- Parallel Beam Optic (PBO) transforms X-rays diverging from the sample into a parallel beam
- Polychromatic beam undergoes Bragg diffraction at flat analyzer crystal
- Angle Θ between beam and crystal surface and crystal lattice constant $2d$ determines the energy that passes through to the detector
- X-ray detection by a flow proportional counter





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

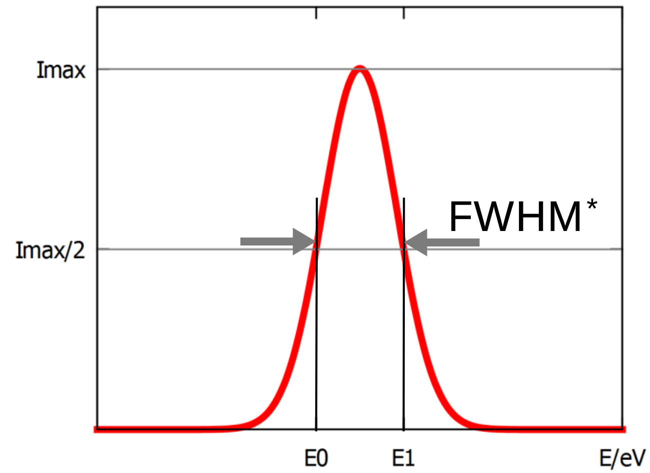
XSense WD Spectrometer
Spectral resolution

- Spectral resolution usually defined via the *full width at half maximum* (FWHM) of an elemental line peak
- Natural line widths are in the 0.2-3 eV range
- Peak broadening due to spectrometer effects:

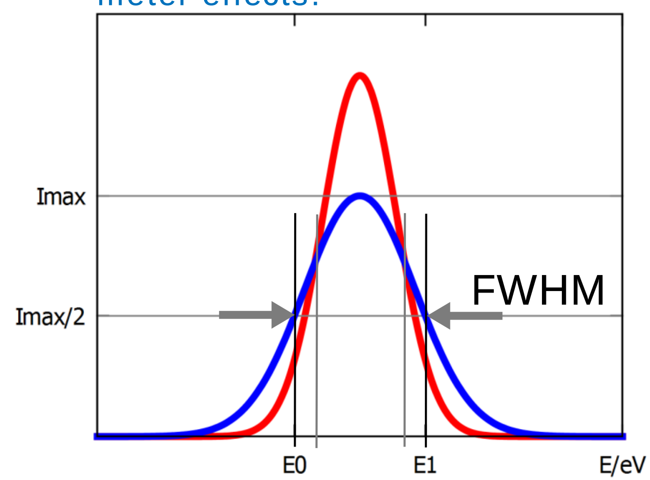
  In a PB-WDS:

  - Imperfections of analyzing crystals/multi-layers (crystal defects, inter-layer diffusion, waviness of bilayer boundaries)
  - Imperfect parallelization of beam due to (**A**) aberrations of the optic, (**B**) optical misalignment.

Natural line width:



Peak broadening due to spectro-meter effects:



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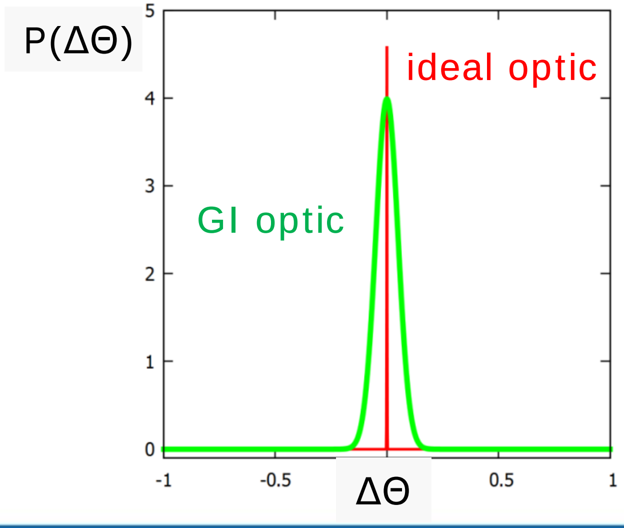
XSense: Minimization of **A**:

- Use of a grazing incidence mirror optic which (in comparison with a polycapillary-based optic) produces a highly parallel beam of low divergence

Grazing incidence mirror optic:

low beam divergence



XSense WD Spectrometer
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  In a PB-WDS:

  - Imperfections of analyzing crystals/multi-layers (crystal defects, inter-layer diffusion, waviness of bilayer boundaries)
  - Imperfect parallelization of beam due to (**A**) optic figure/slope errors, (**B**) optical misalignment.

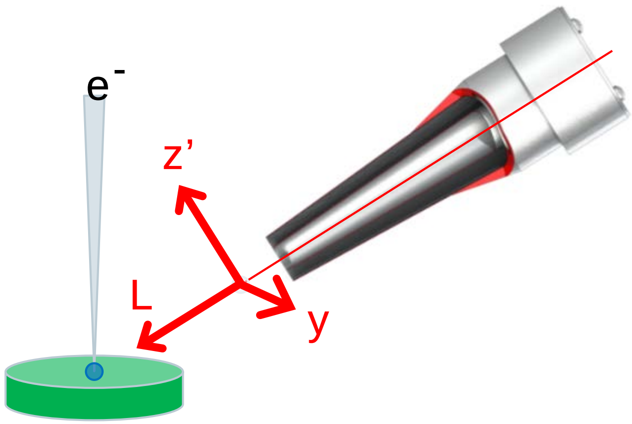
XSense: Minimization of **B**:

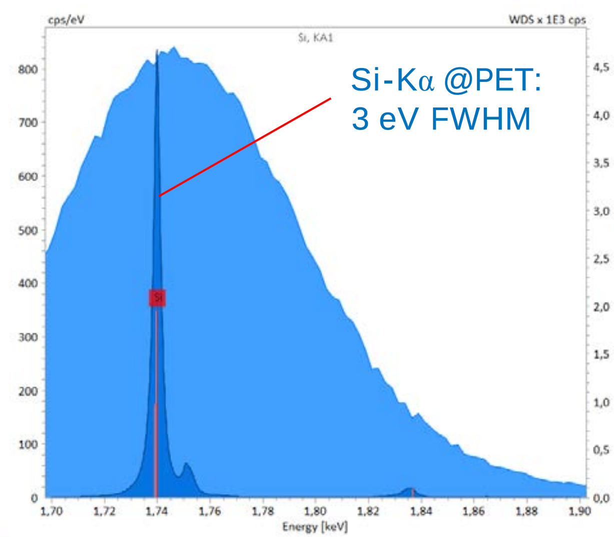
- Spectrometer equipped with 3-axis optic positioning unit + powerful optical alignment software algorithm

grazing incidence optic + auto-optic alignment:

highest possible resolution from a PB-spectrometer

3-axis optic positioning unit

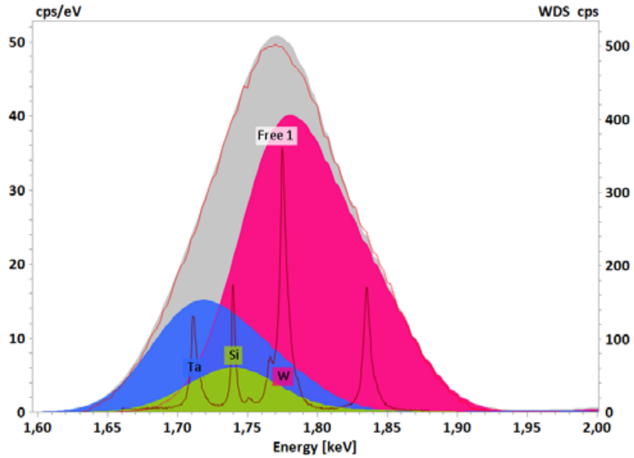




QUANTAX WDS
EDS vs. WDS

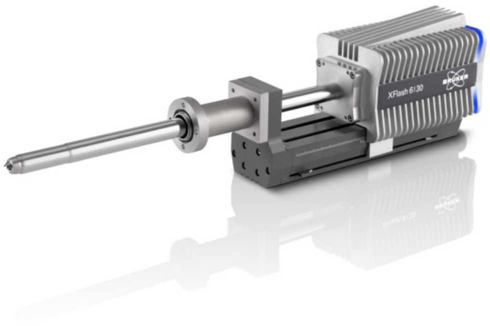
- In SDD-based EDS peak broadening is much more pronounced, resolutions are in the 40-120 eV range
- Limited resolution of EDS leads to frequent peak overlaps, mainly in the low energy range

Typical EDS peak overlap:



- XSense features substantially higher spectral resolution (typically 4-20 eV FWHM)

XFlash™ EDS detector



XSense™ WD spectrometer

WDS ideally complements EDS in demanding applications, where resolution is critical

QUANTAX WDS

Resolving common overlaps in EDS microanalysis

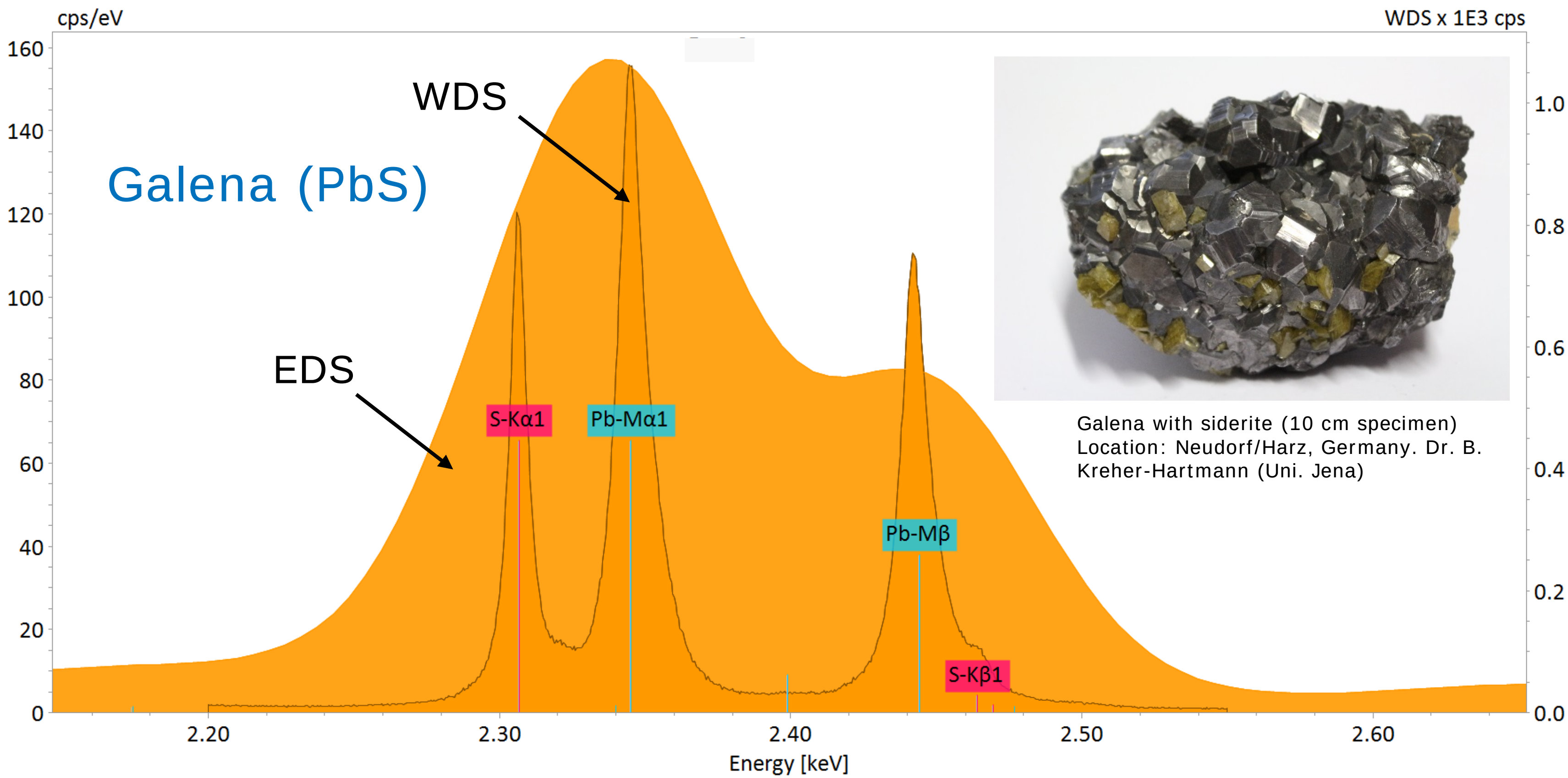
Element and line	Interferences with	Δ eV	Samples or applications where the overlaps are found
Cu-L	Na-Kα	18	Biological samples (grid)
As-L	Na-Kα	79	Biological samples (stain or fixative)
Ag-L	Cl-Kα	10	Biological samples (stain or fixative)
Ru-L	S-Kα	54	Biological samples (stain or fixative)
Os-M	Al-Kα	5	Biological samples (stain or fixative)
U-M	K-Kα	22	Biological samples (stain or fixative)
Sr-Lα	Si-Kα	31	Silicates (feldspars in particular)
Y-Lβ	P-Kα	18	Phosphates
Y-Lβ	Zr-Lα	46	Silicates (zircon), oxides (zirconia)
S-Kα,β	Mo-Lα; Pb-Mα	14; 38	Minerals, lubricants, sulfides, sulfates
Ti-Kβ	V-Kα	20	Steels, Fe-Ti oxides
V-Kβ	Cr-Kα	13	Steels
Cr-Kβ	Mn-Kα	47	Steels
Mn-Kβ	Fe-Kα	87	Steels
Fe-Kβ	Co-Kα	128	Steels, magnetic alloys
Co-Kβ	Ni-Kα	169	Steels, hard surfacing alloys
W-Mα,β	Si-Kα,β	35	Semiconductor processing
Ta-Mα,β	Si-Kα,β	27	Semiconductor processing
Ti-Kα	Ba-Lα	45	Optoelectronics, silicates
As-Kα	Pb-Lα	8	Pigments

Overlaps known from biological, geological and material sciences and industries

Modified after Goldstein et al. (2007). Scanning Electron Microscopy and X-Ray Microanalysis. Springer

QUANTAX WDS and EDS

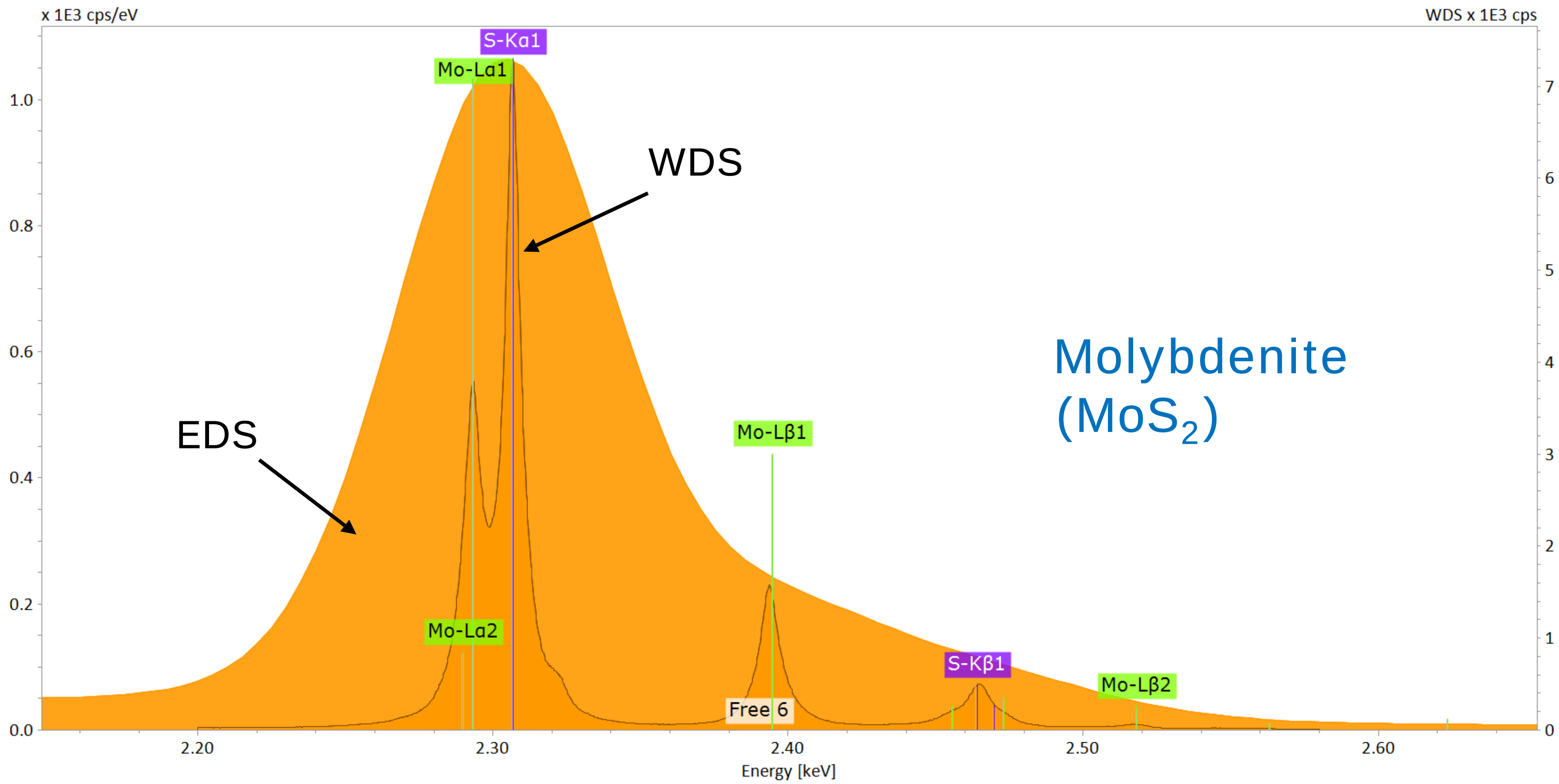
Geological samples I: Pb sulfide



Galena with siderite (10 cm specimen)
Location: Neudorf/Harz, Germany. Dr. B. Kreher-Hartmann (Uni. Jena)

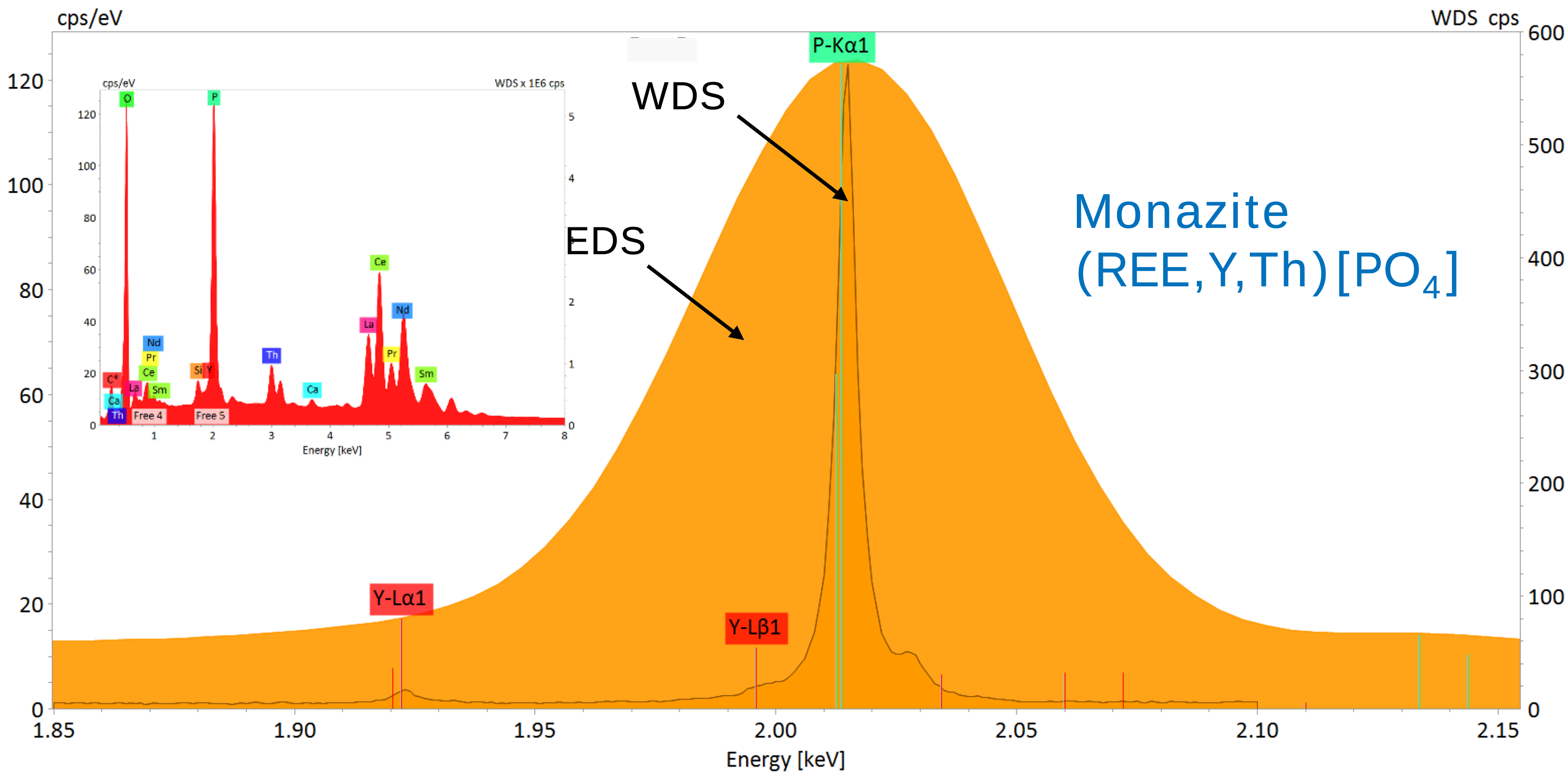
Δ S-Kα – Pb-Mα: 38 eV

QUANTAX WDS and EDS
Geological samples II: Mo sulfide



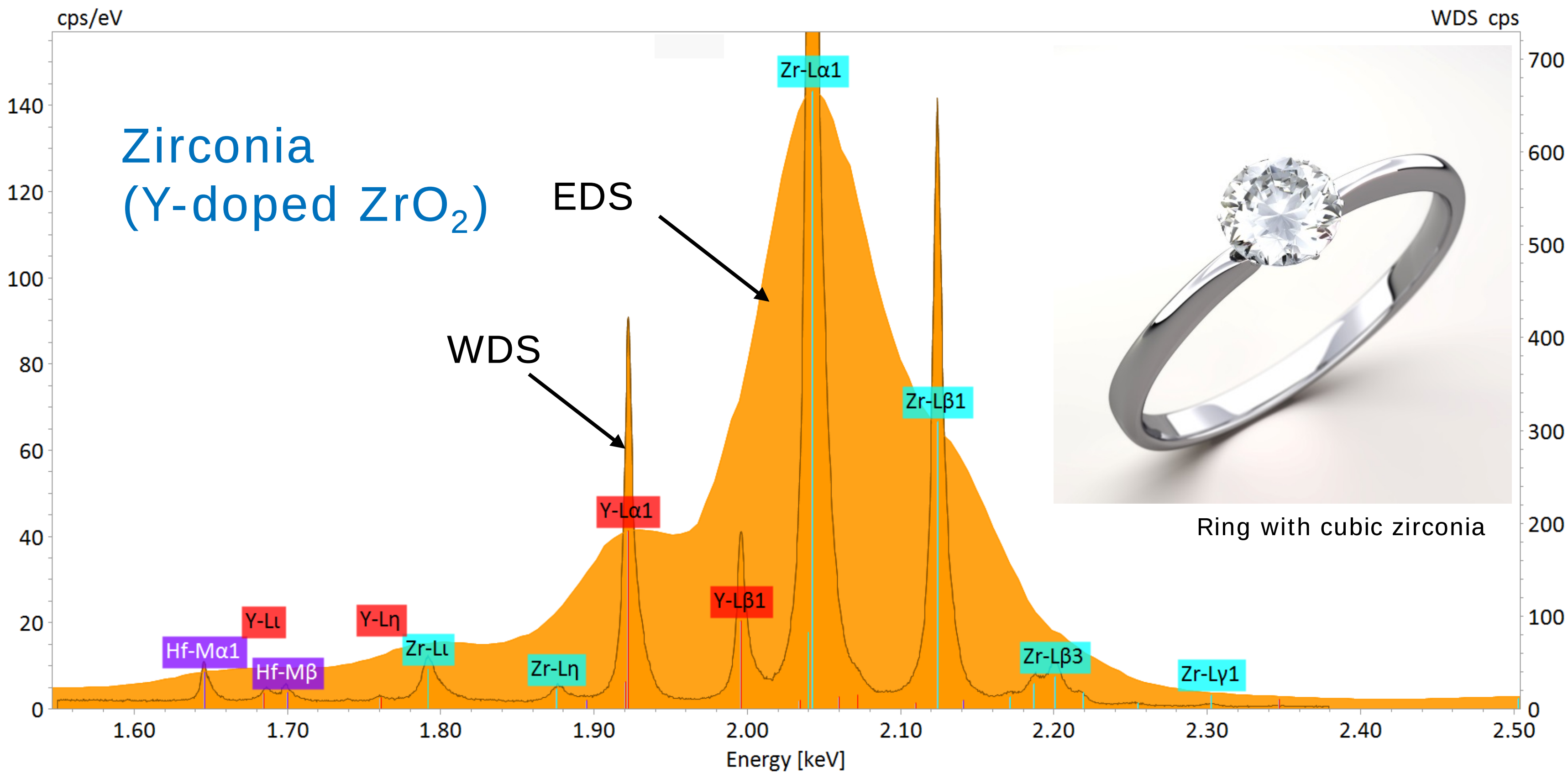
Δ S-Kα – Mo-Lα: 14 eV

QUANTAX WDS and EDS
Geological samples III: REE phosphates



Δ P-Kα – Y-Lβ: 18 eV; Δ P-Kα – Y-Lα: 92 eV

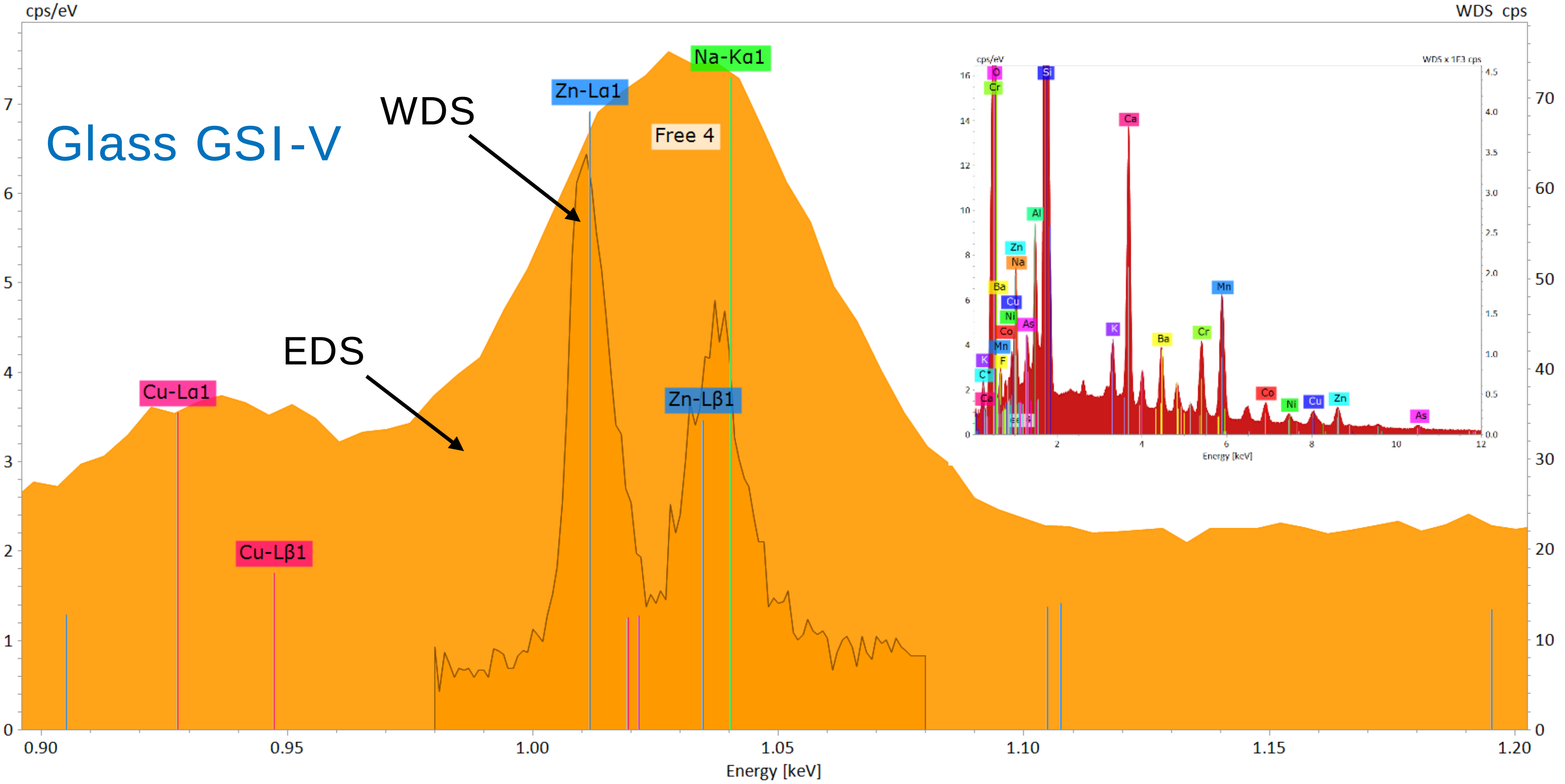
QUANTAX WDS and EDS
Geological & material sciences: Zr oxide



Δ Zr-Lα – Y-Lα: 120 eV; Δ Zr-Lα – Y-Lβ: 46 eV

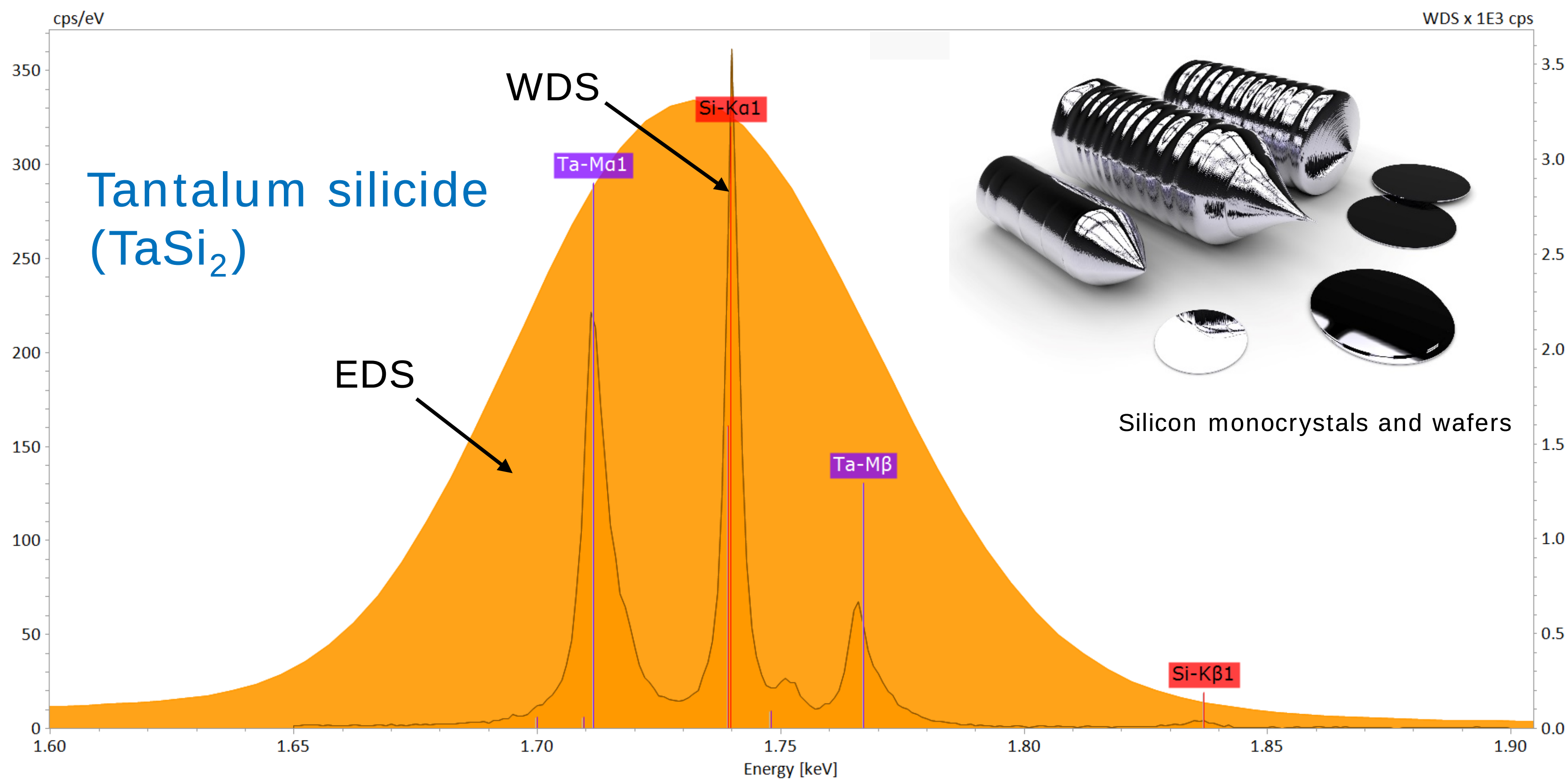
QUANTAX WDS and EDS

Application in material science II: glass



Δ Na-Kα – Zn-Lα: 28 eV; Δ Na-Kα – Zn-Lβ: 5 eV

QUANTAX WDS and EDS
Material science III: semiconductors



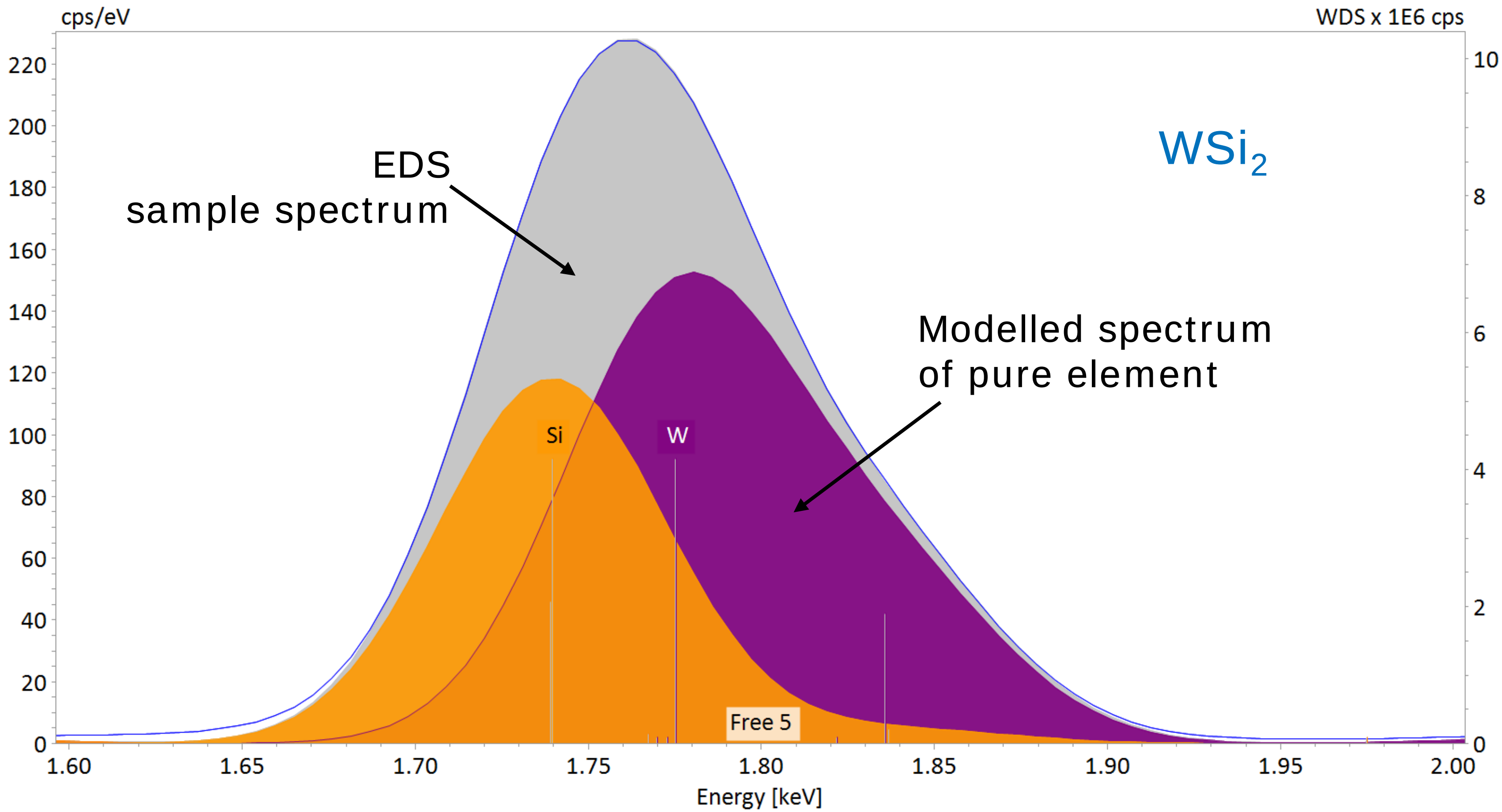
Δ Si-Kα – Ta-Mα: 28 eV; Δ Si-Kα – Ta-Mβ: 27 eV

QUANTAX WDS and EDS

Comparison of WDS and EDS resolution

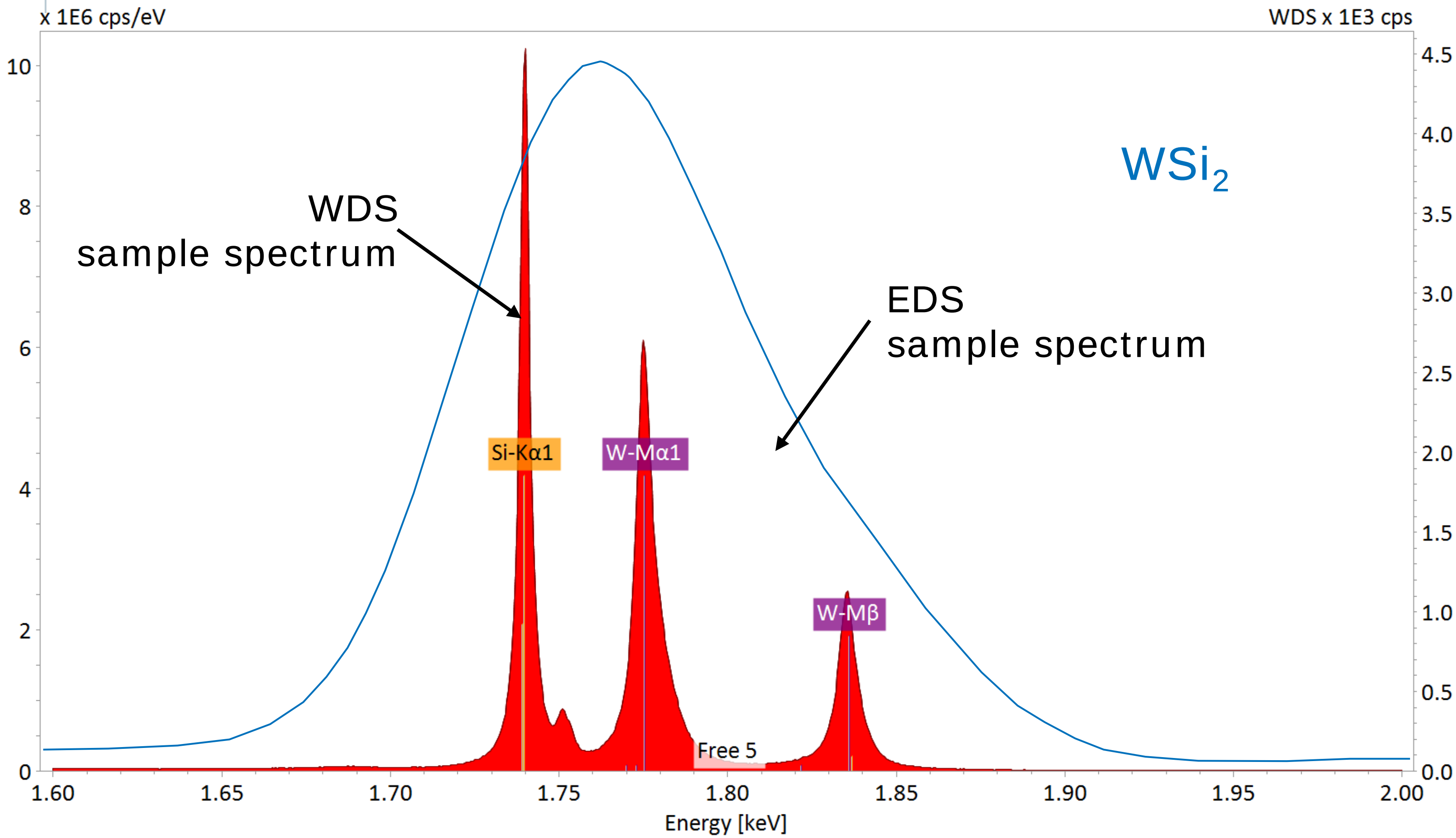
Element	Atomic No.	X-ray line	Energy [keV]	FWHM EDS [eV]	WDS diffractor	FWHM WDS [eV]	Resolution improvement
Si	14	Kα	1.740	75	PET	3.5	21x
P	15	Kα	2.014	77	PET	5	15x
S	16	Kα	2.307	85	PET	7	12x
Y	39	Lα	1.922	82	PET	6.3	13x
Zr	40	Lα	2.042	83	PET	7.2	12x
Mo	42	Lα	2.293	87	PET	9.5	9x
Ta	73	Mα	1.712	71	PET	6	12x
W	74	Mα	1.775	74	PET	6.4	12x
Hg	80	Mα	2.195	80	PET	9	9x
Pb	82	Mα	2.345	91	PET	11.9	8x

QUANTAX WDS and EDS
Resolution vs. deconvolution I



Deconvolved EDS spectrum of tungsten silicide (WSi_2)

QUANTAX WDS and EDS
Resolution vs. deconvolution I



Highly resolving WDS spectrum of tungsten silicide (WSi_2)

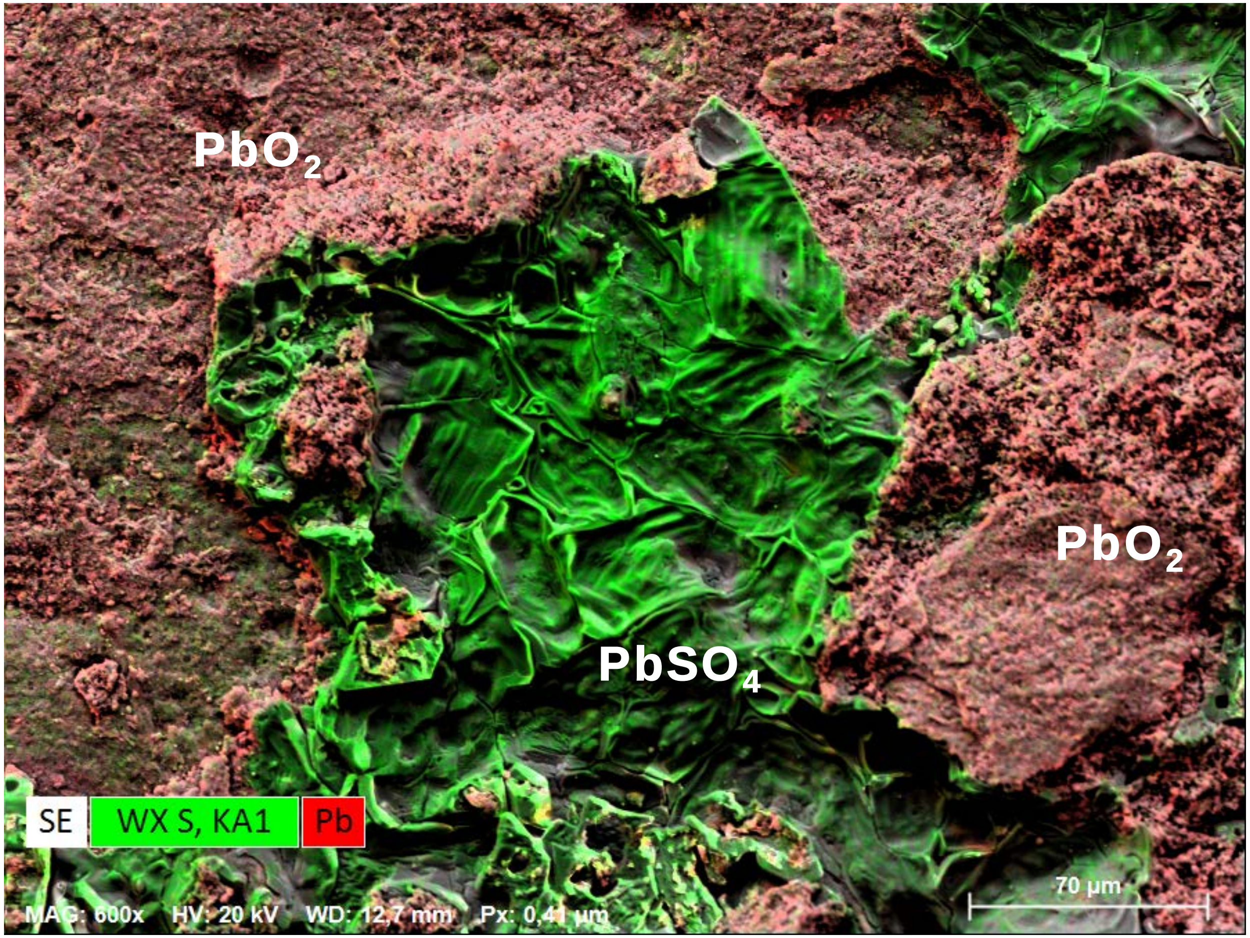
QUANTAX WDS and EDS
Resolution vs. deconvolution II

Quantitative results in atomic percentages (5kV)

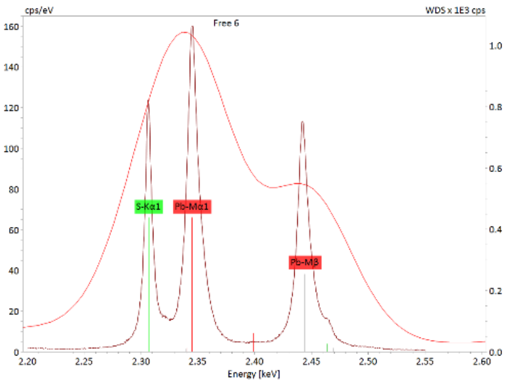
Sample	Element	Stoichio-metry	EDS[1]	EDS[2]	WDS
MoS_2	Mo	33.3	39.0	34.5	33.9
	S	66.7	61.0	65.5	66.1
WSi_2	W	33.3	20.2	32.6	33.4
	Si	66.7	79.8	67.4	66.6

[1]standardless, [2]standard-based

QUANTAX WDS and EDS
High spectral resolution for mapping



Combined WDS and EDS mapping



Combined WDS and EDS spectra

Lead accumulator showing surface deposits of $PbSO_4$

Summary of todays WDS Webinar

- Bruker QUANTAX PB WDS on SEM
- XSense WDS facilitates high spectral resolution analyses
- Applications include biological, geological and material sciences and industries
- Deconvolution methods cannot replace true spectral resolution
- High spectral resolution is important for qualitative and quantitative analyses as well as mapping
- QUANTAX WDS is a powerful tool for scientific and industrial applications

Are There Any Questions?

Please type in the questions you might have in the Q&A box and press *Send*.

Innovation with Integrity