BRUKER NANO WEBINAR

Advanced materials microanalysis using QUANTAX WDS with a grazing incidence X-ray optic

Bruker Nano Analytics, Berlin, Germany

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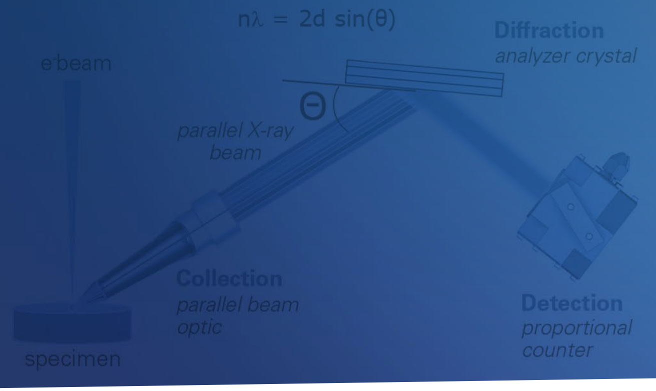
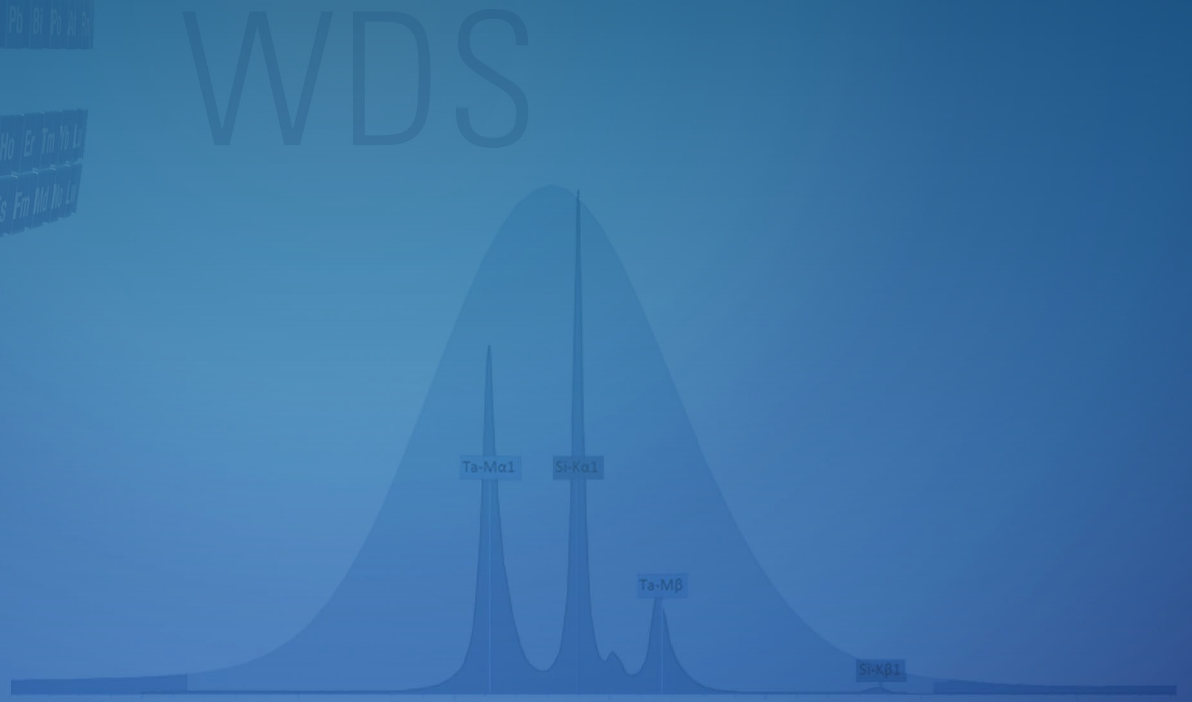
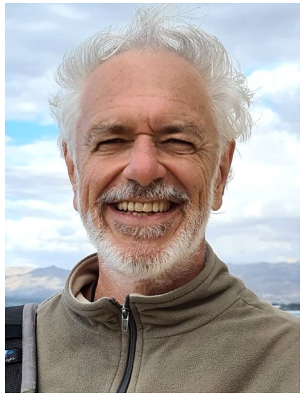
CEA-IRIG, Université Grenoble Alpes, France





Presenters



Dr. Eric Robin
Senior Researcher, CEA-IRIG, Université Grenoble Alpes, France

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Sr. Applications Scientist WDS, Bruker Nano Analytics, Berlin, Germany

Outline of the presentation

01 Introduction to Wavelength Dispersive Spectrometry with Parallel Beam Optic (PBO-WDS)
- principles and advantages

02 Complementing microanalysis techniques:
- What WDS can add to EDS analysis

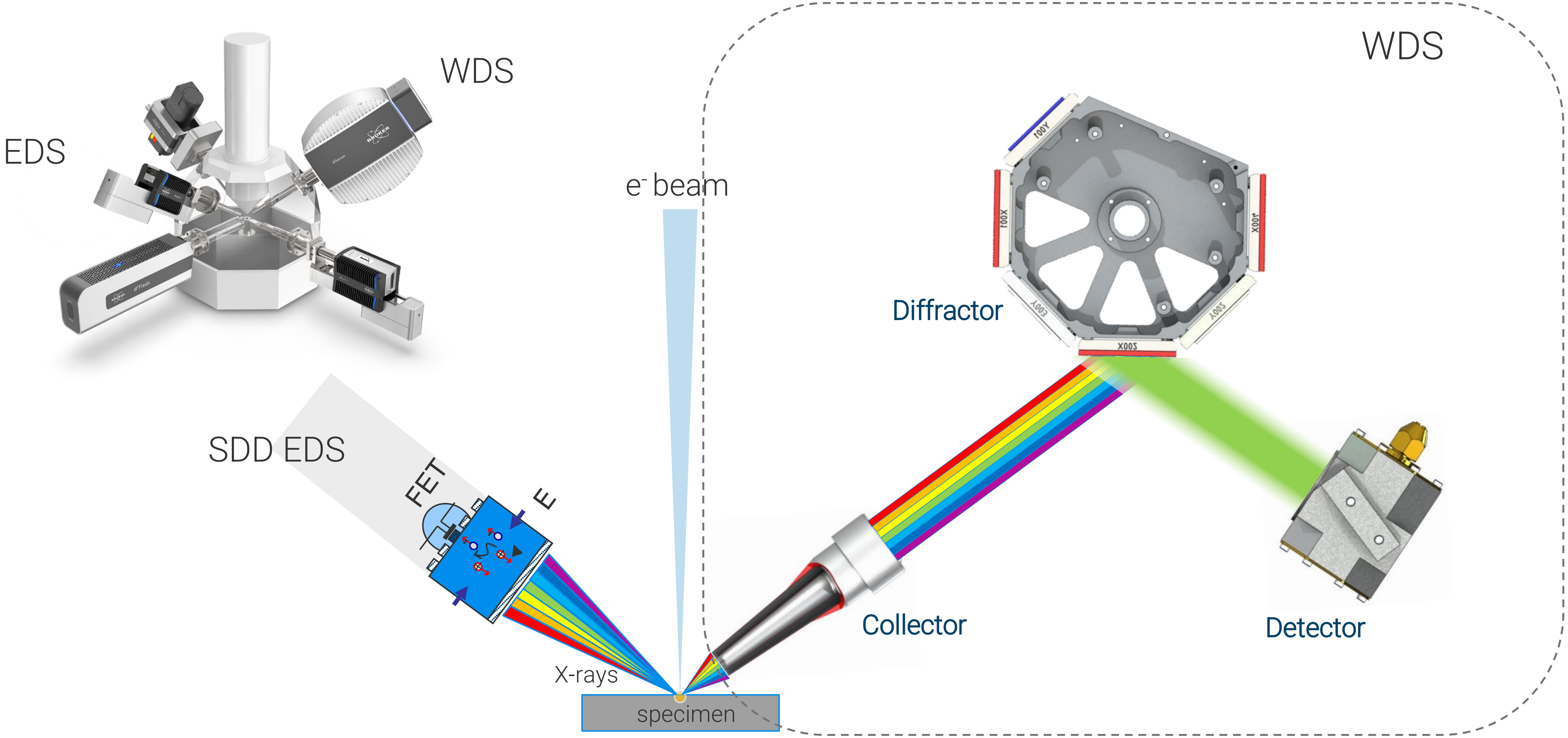
03 Application examples from CEA- IRIG:
- characterization of B-rich phases in permanent REE magnets
- quantification of Mg dopant in GaN nanowires

04 Conclusions and Q&A

QUANTAX WDS WEBINAR

Wavelength Dispersive Spectrometry

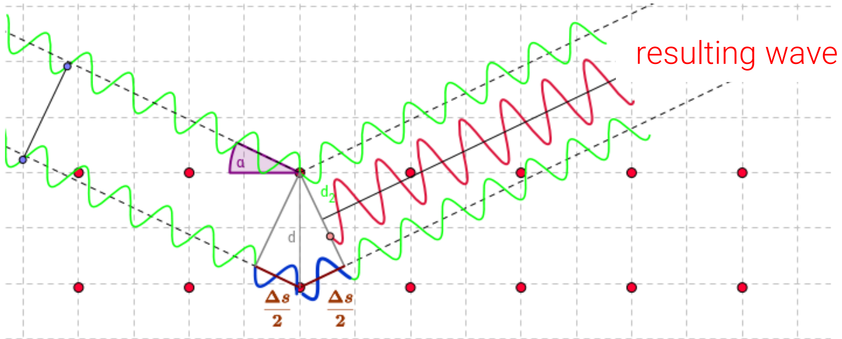
Technical principles of WDS & EDS



Technical principles of WDS

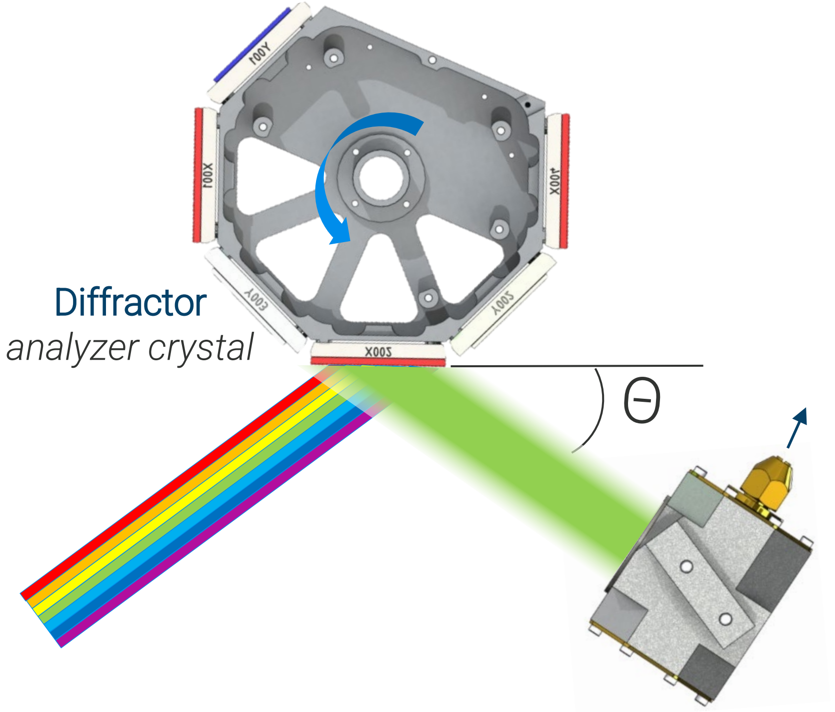
Bragg equation

$$n\lambda = 2d \sin(\theta)$$



X-rays are diffracted on the crystal lattice

- Bragg diffraction at analyzer crystal
- Measurement energy determined by Bragg angle Θ and crystal lattice constant
- X-ray detection with flow proportional counter

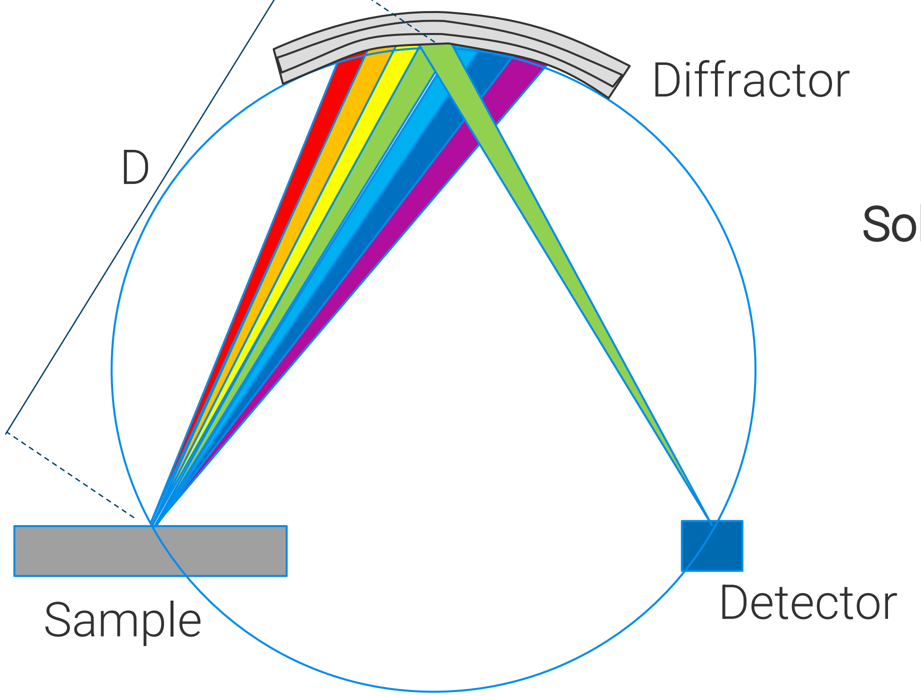


QUANTAX WDS WEBINAR

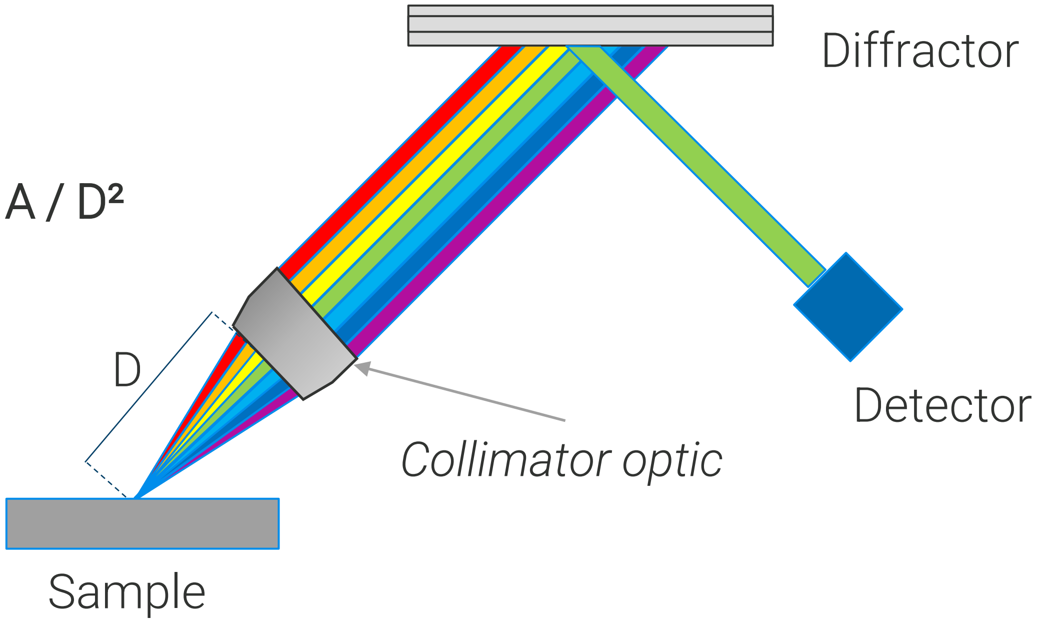
WDS with Parallel Beam Optic (PBO-WDS)

Rowland Circle vs. PBO type WDS

Classical Rowland circle design



Modern Parallel Beam Optic design



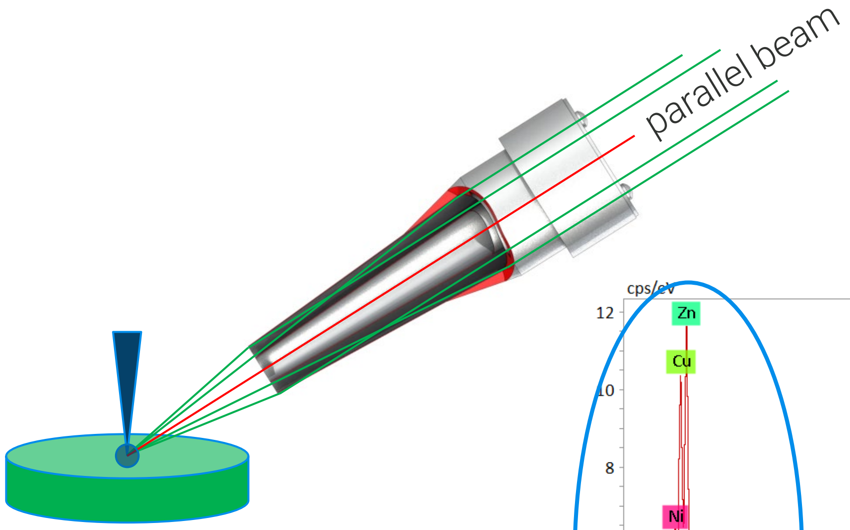
Solid Angle, $\Omega = A / D^2$

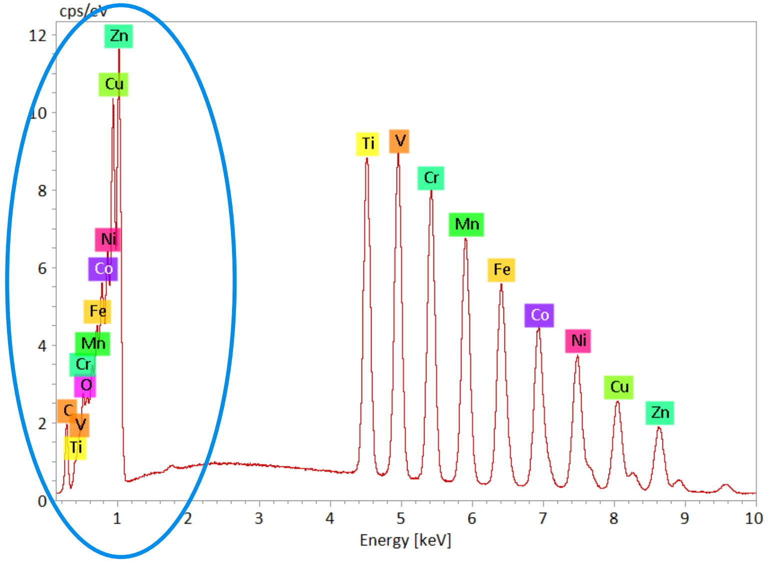
- Diverging beam creates small solid angle
- Requires 20-100 nA probe current
- Very long acquisition time at lower currents
- Causes damage to beam-sensitive samples

- Collects X-rays near the sample
- Requires only 2-20 nA probe current
- Faster acquisition, less damage

Different types of parallel-beam optics (PBO)

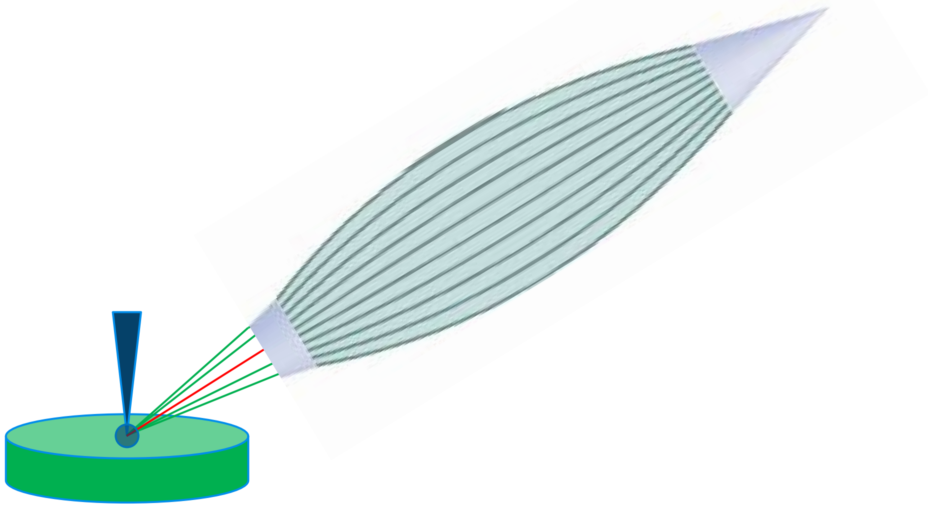
Grazing incidence mirror optic





- X-ray reflection in conic mirrors
- Higher spectral resolution
- Limited upper energy range
- Optimized for low X-ray energies

Polycapillary optic



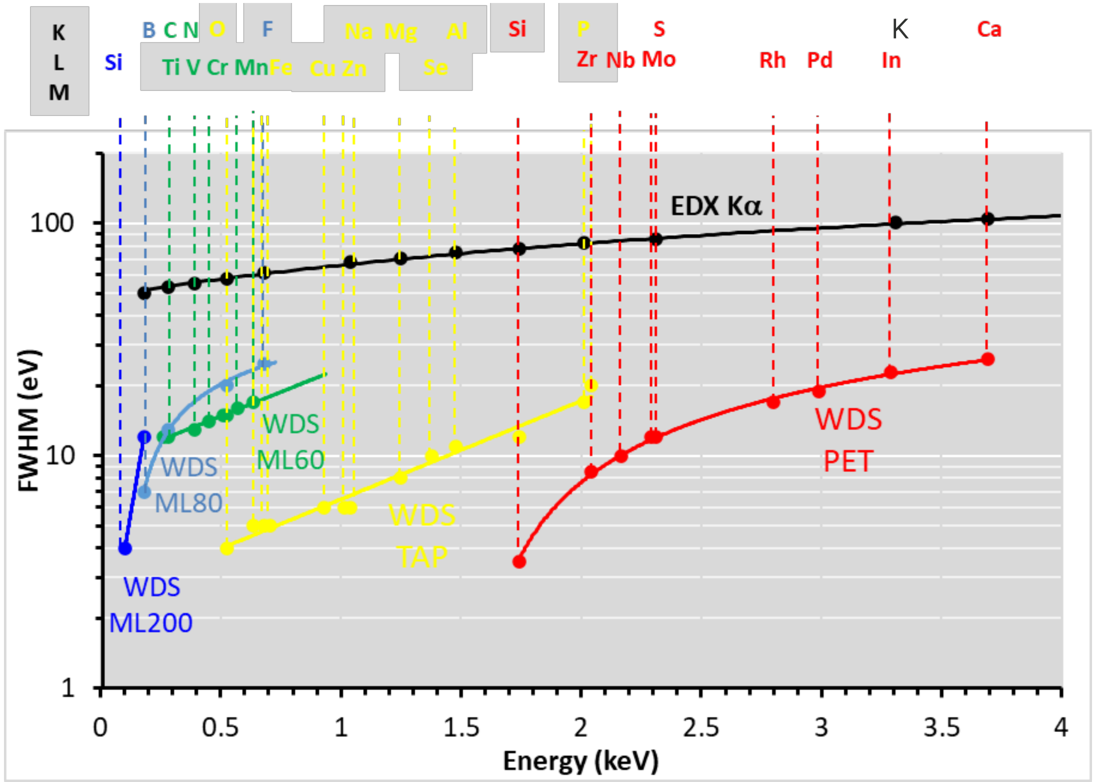
- Based on total reflection with optic fibers
- Optimized for higher X-ray energies
- Degraded spectral resolution

QUANTAX WDS WEBINAR

What WDS can add to microanalysis (EDS)?

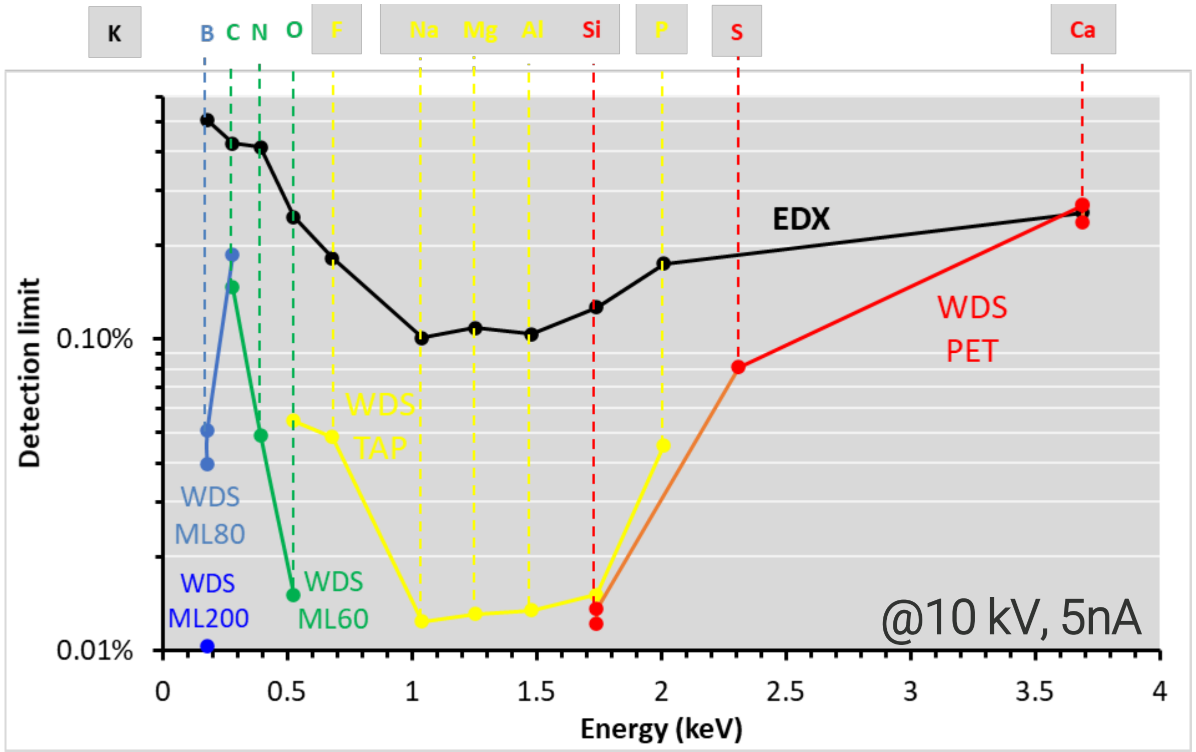
QUANTAX WDS and EDS parameters

Spectral resolution



Spectral resolution of WDS is generally better than EDS (up to 20x). Parameters vary with the different diffractors and X-ray energy. FWHM = full width at half peak maximum.

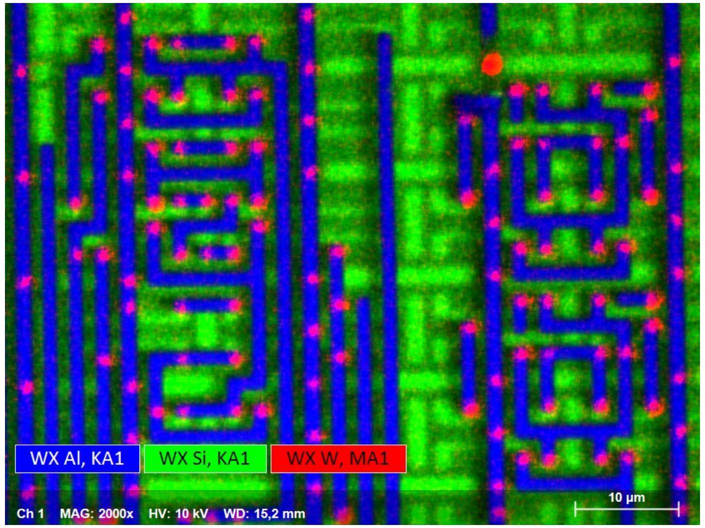
Limits of detection



Up to 10x higher signal/noise ratios result in 10x lower limits of detection for WDS, thus better trace element detection. Note the low HV and probe current for the present measurements.

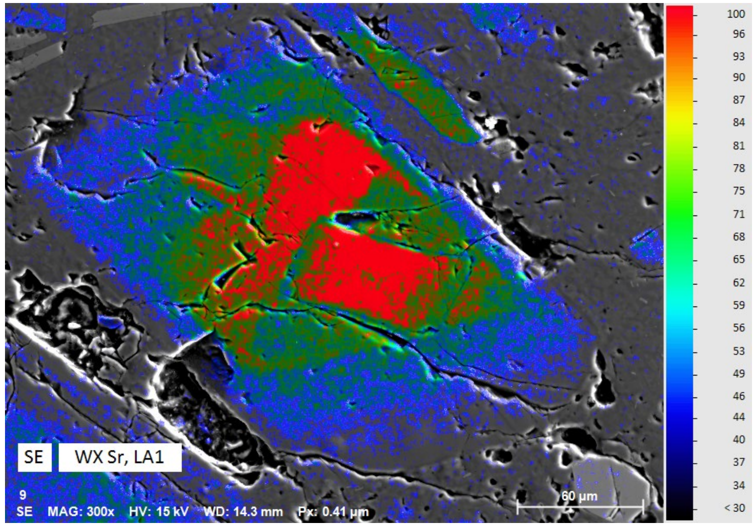
Application fields for PBO-WDS on SEM

Resolution of EDS peak overlaps



Example: MOSFET with gates made of tungsten on silicon base.

Determination of trace elements



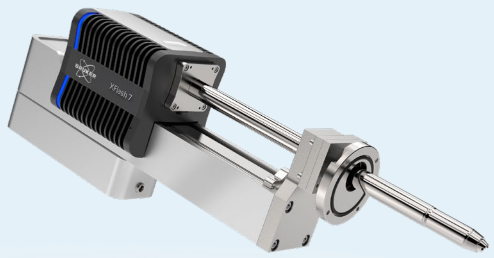
Example: Traces of Sr enriched in the core of a plagioclase of a volcanic rock.

Determination of light elements

Example: Distribution of carbon in two-phase steel DP600.

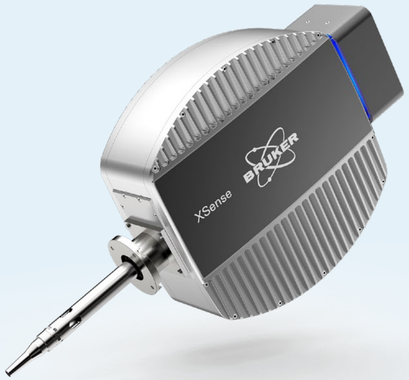
QUANTAX WDS and EDS characteristics

XFlash® ED spectrometer



- limited spectral resolution (40–130 eV FWHM)
- lower Peak/Bg-ratios
- covers full energy range
- fast (entire spectrum all at once)

XSense WD spectrometer



- high resolution (typically 3–15 eV FWHM)
- high signal/noise-ratios → low limits of detection
- outstanding sensitivity for soft X-rays
- limited energy range
- slower (sequential measurement)

WDS is an ideal technique to complement EDS in demanding applications

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Application on advanced materials

Alternative Energies and Atomic Energy Commission

DE LA RECHERCHE À L'INDUSTRIE

Wavelength Dispersive X-ray Spectrometry

Advanced Chemical Analysis of Nanostructures using a WDS spectrometer for SEM

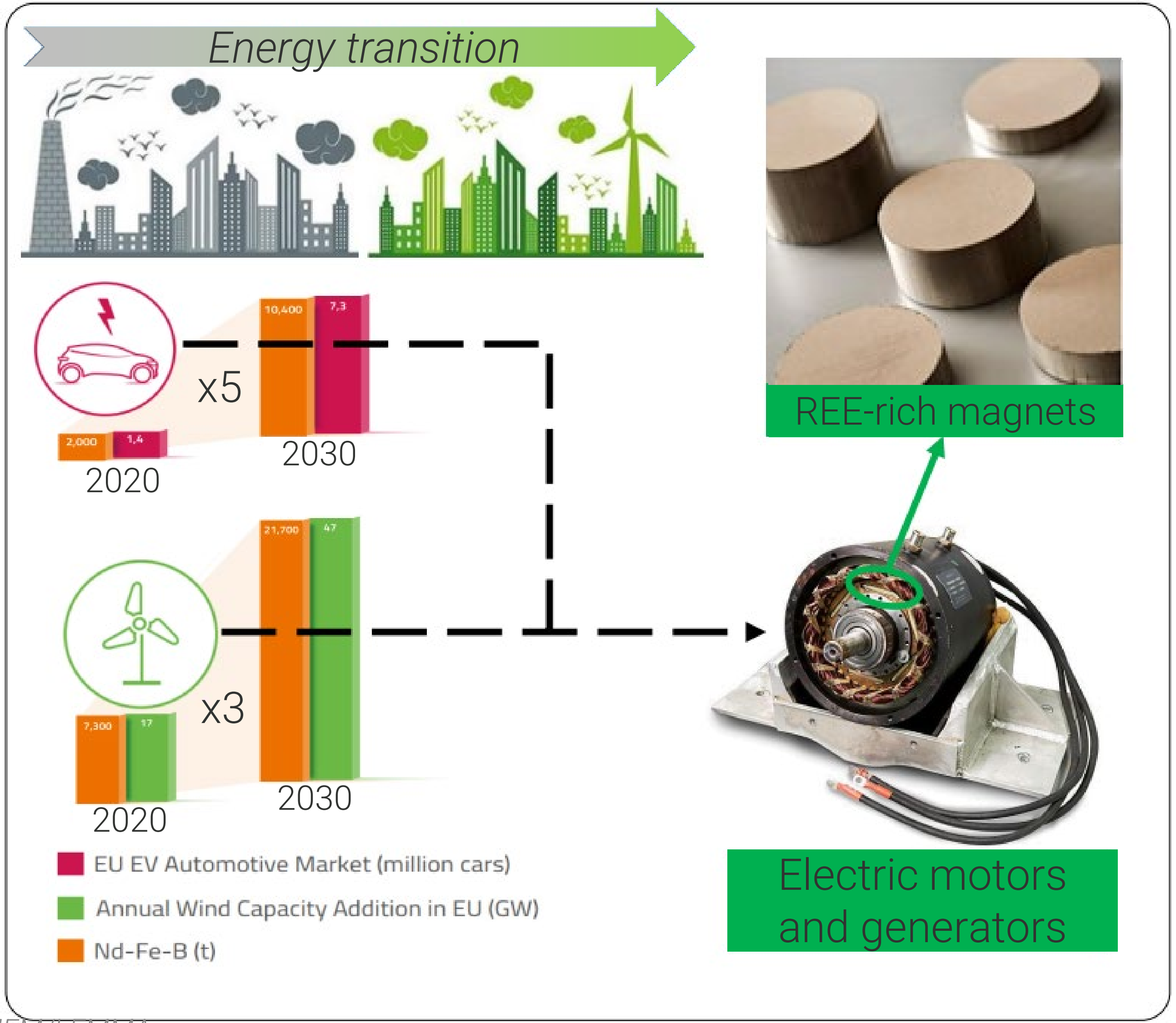
XSense

BRUKER

Eric Robin
IRIG/MEM/LEMMA
PFNC CEA-Grenoble

www.cea.fr

IDENTIFICATION AND CHARACTERIZATION OF REE-RICH PHASES IN RECYCLED MAGNETS



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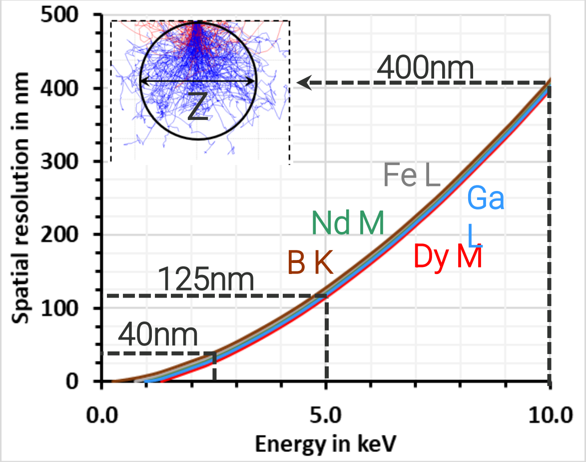
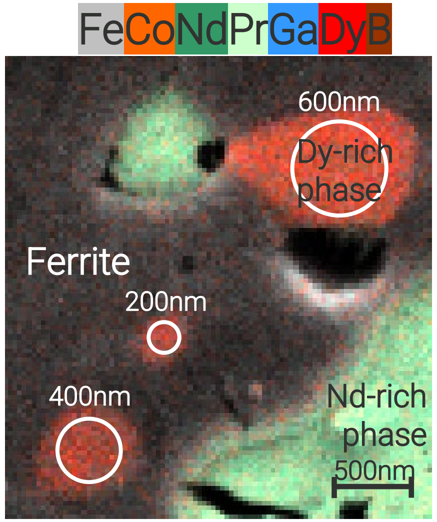
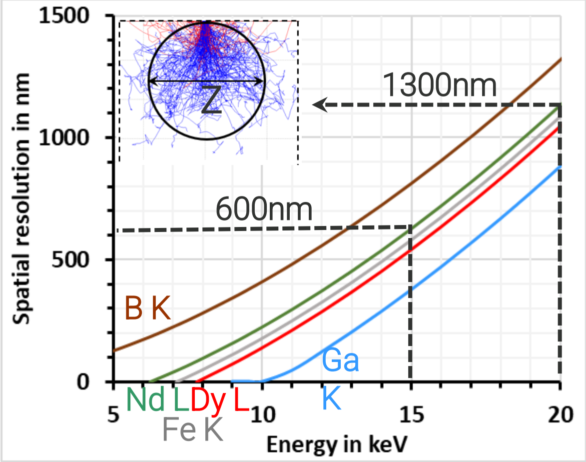
IDENTIFICATION AND CHARACTERIZATION OF REE-RICH PHASES IN RECYCLED MAGNETS

$$Z\,(nm) = 64(E^{1.68} - Ec^{1.68})/\rho$$

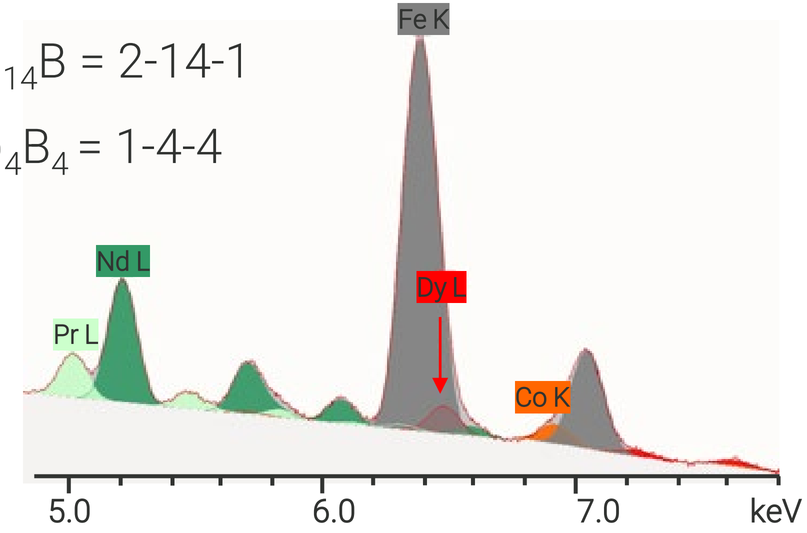






$REE_2(Fe,Co)_{14}B$ = 2-14-1

$REE(Fe,Co)_4B_4$ = 1-4-4



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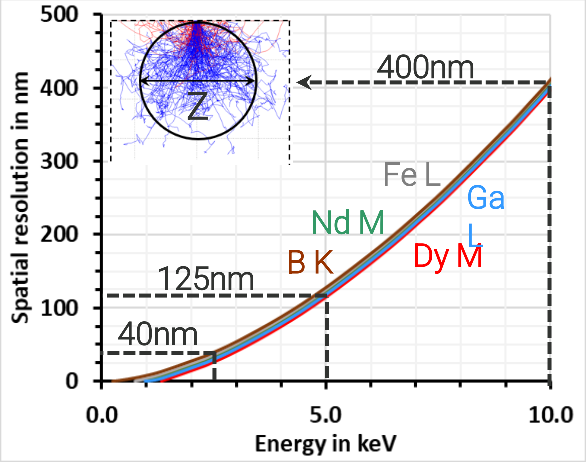
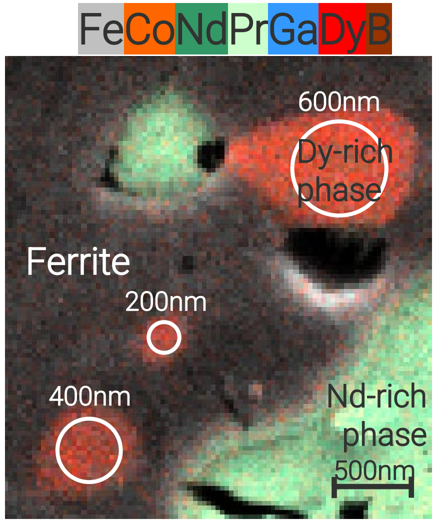
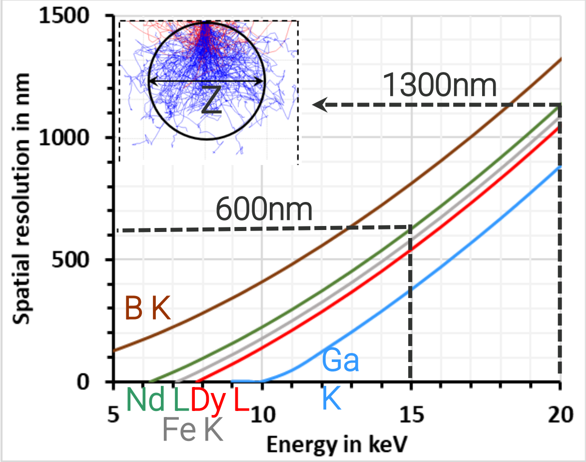
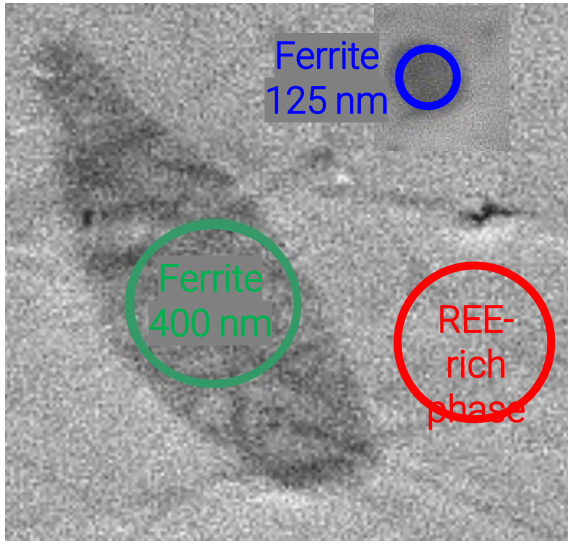
IDENTIFICATION AND CHARACTERIZATION OF REE-RICH PHASES IN RECYCLED MAGNETS

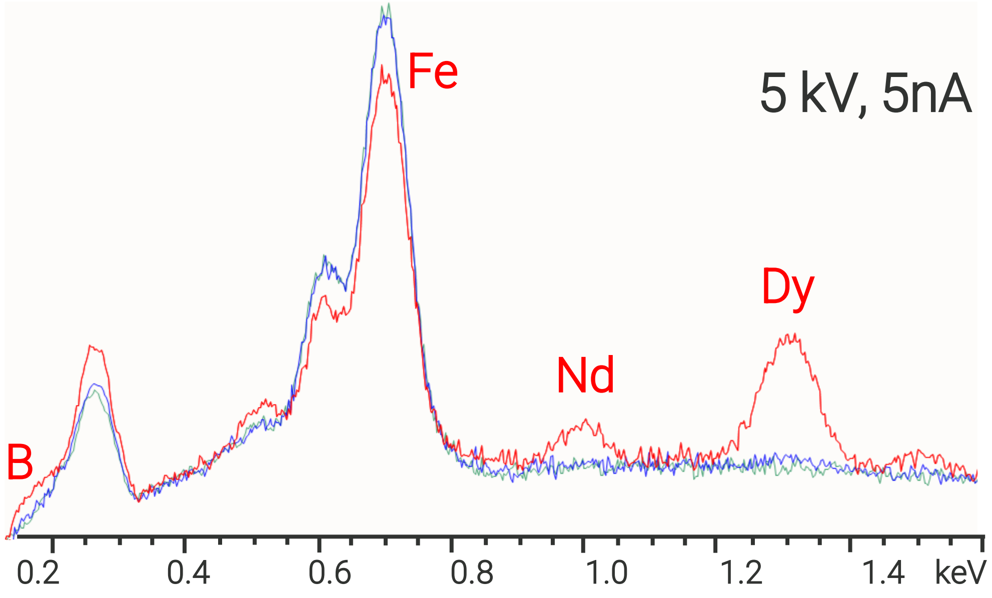
$$Z\,(\text{nm}) = 64(E^{1.68} - Ec^{1.68})/\rho$$











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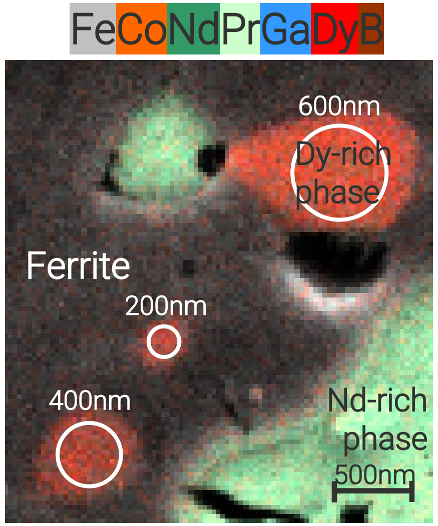
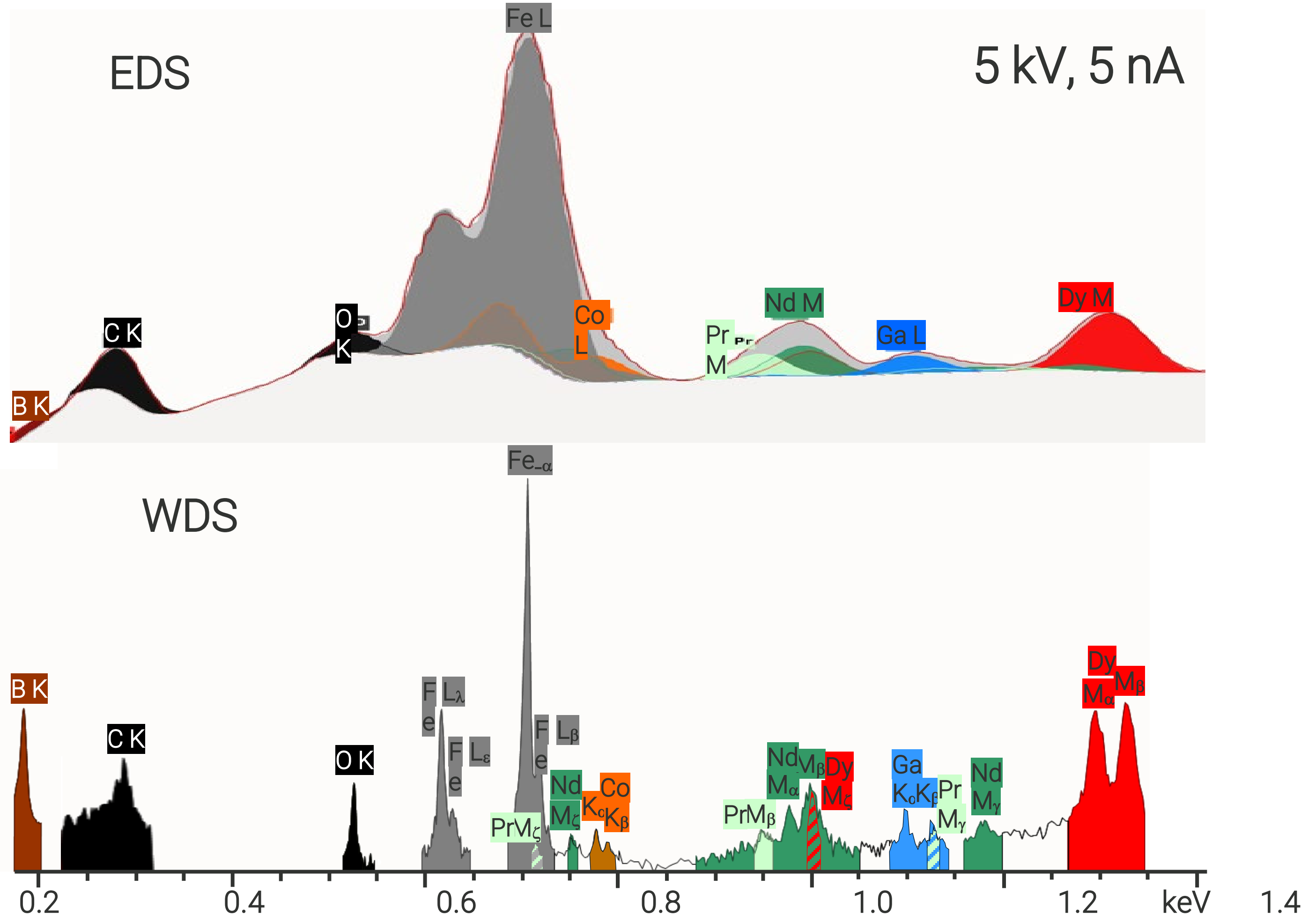
IDENTIFICATION AND CHARACTERIZATION OF $REE_2Fe_{14}B$ IN RECYCLED MAGNETS



Elt	EDS Line	EDS wt%	WDS Line	WDS wt%
Fe	L	67.4	LA1	71.3
Co	L	2.6	LA1	1.6
Ga	L	1.1	LA1	0.8
Pr	M	3.2	MG	4.1
Nd	M	20.3	MZ1	10.8
Dy	M	11.1	MB	10.9
B	K	?	KA1	1.0
		105.6		100.5
	REE	2.8		2.0
	Fe+Co	14.0		14.0
	B	?		1.0



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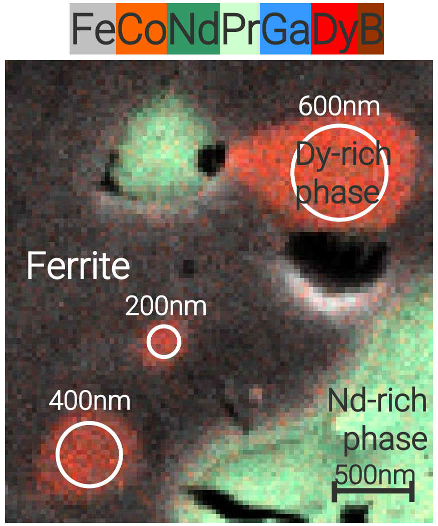
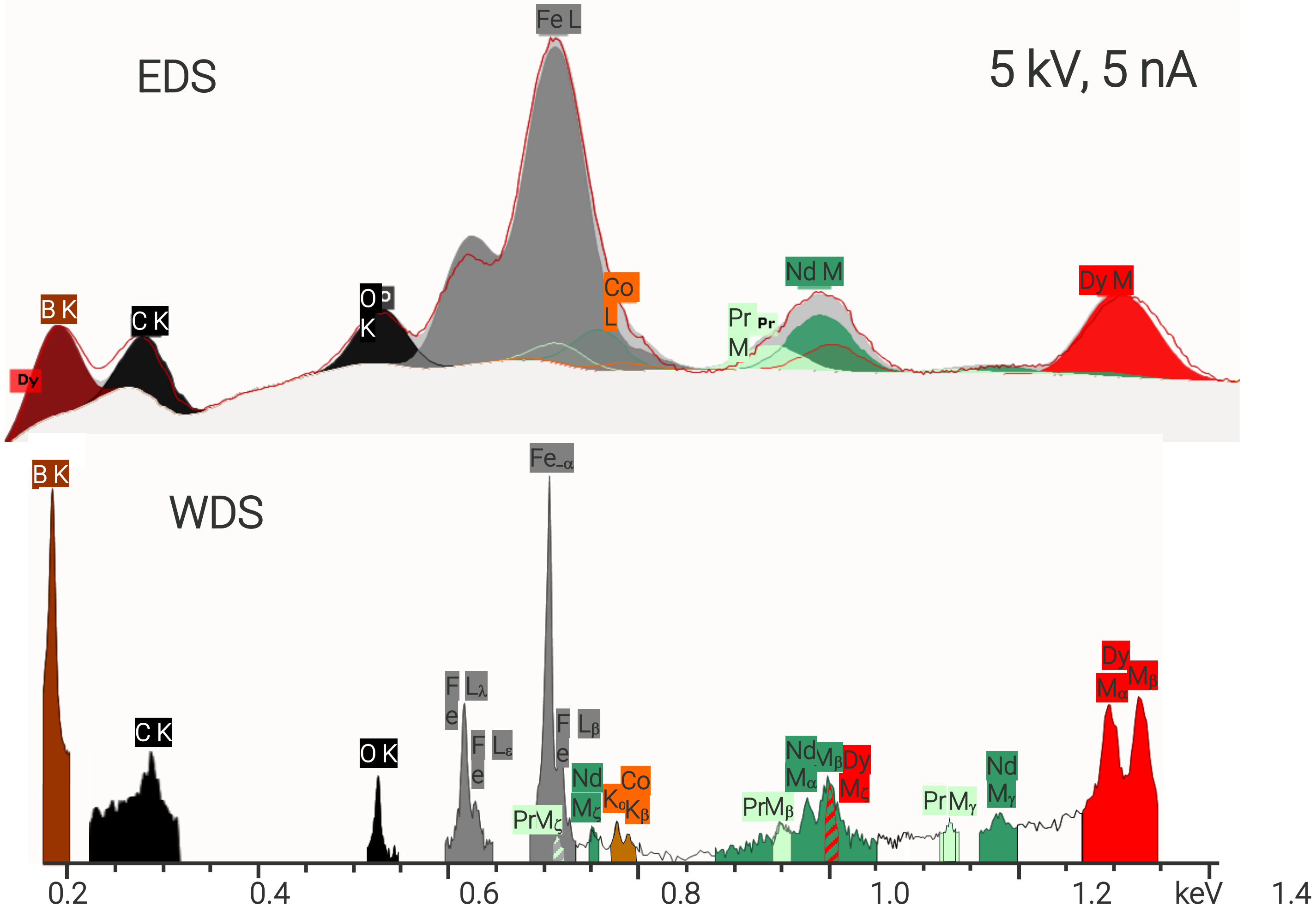
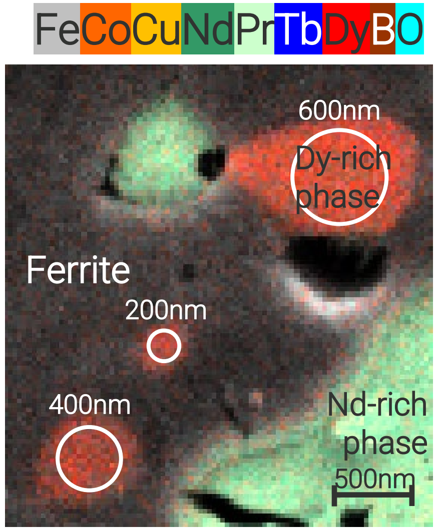
IDENTIFICATION AND CHARACTERIZATION OF REEFe_4B_4 IN RECYCLED MAGNETS



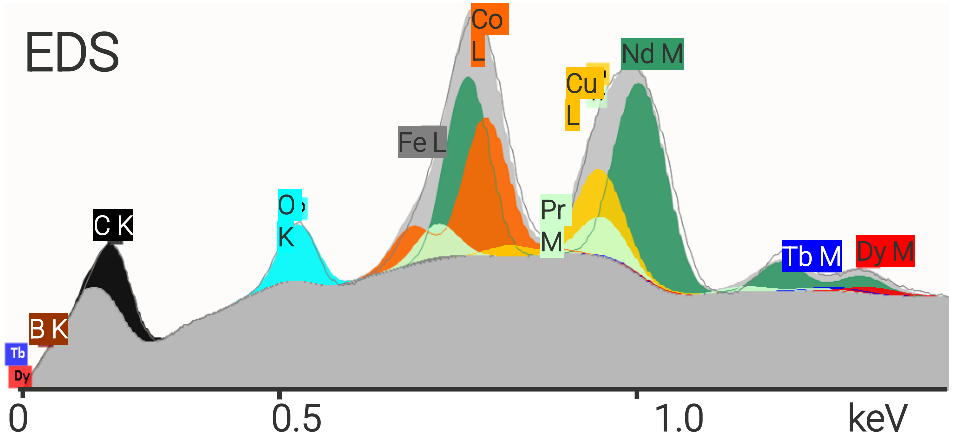
Elt	EDS Line	EDS wt%	WDS Line	WDS wt%
Fe	L	49.4	LA1	50.7
Co	L	0.5	LA1	1.1
Ga	L	< 0.1	LA1	< 0.1
Pr	M	10.9	MG	3.5
Nd	M	17.3	MZ1	18.3
Dy	M	13.8	MB	14.7
B	K	8.2	KA1	10.0
		103.8		98.3
	REE	1.3		1.0
	Fe+Co	4.0		4.0
	B	3.4		4.0



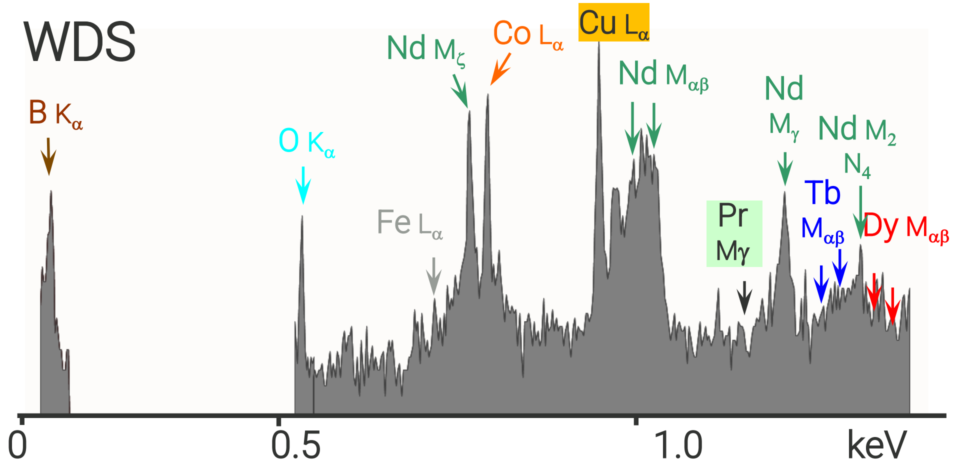
IDENTIFICATION AND CHARACTERIZATION OF ND-RICH PHASE IN RECYCLED MAGNETS



10 kV, 5nA



Elt	wt%	at%
Nd	98.35 ± 0.29	64.3
Pr	11.21 ± 1.12	7.5
Tb	0.82 ± 0.05	0.5
Dy	0.78 ± 0.04	0.5
Co	9.11 ± 0.04	14.6
Cu	3.11 ± 0.16	4.6
Fe	0.19 ± 0.04	0.3
B	< 1.00	
O	1.30 ± 0.01	7.7
Sum	124.88	100.0



Elt	wt%	at%
Nd	82.68 ± 0.38	60.8
Pr	< 0.30	
Tb	< 0.10	
Dy	< 0.10	
Co	7.85 ± 0.01	14.1
Cu	4.65 ± 0.01	7.8
Fe	2.32 ± 0.16	4.4
B	0.51 ± 0.01	5.0
O	1.18 ± 0.01	7.8
Sum	99.19	100.0

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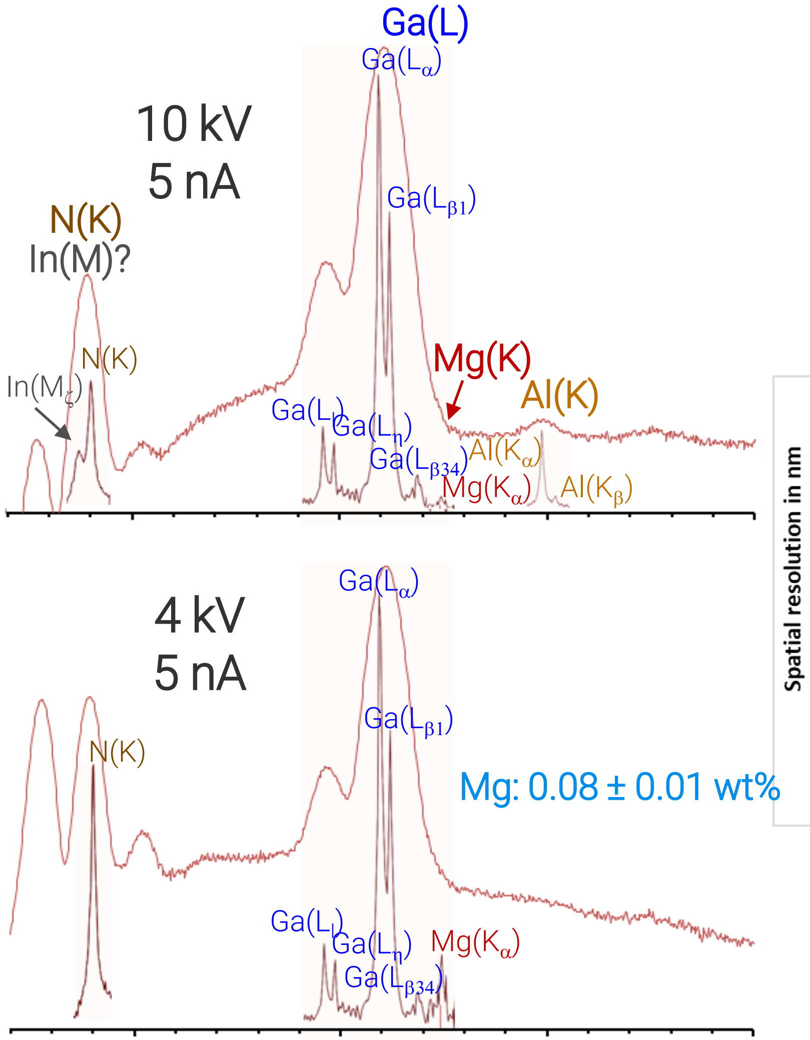
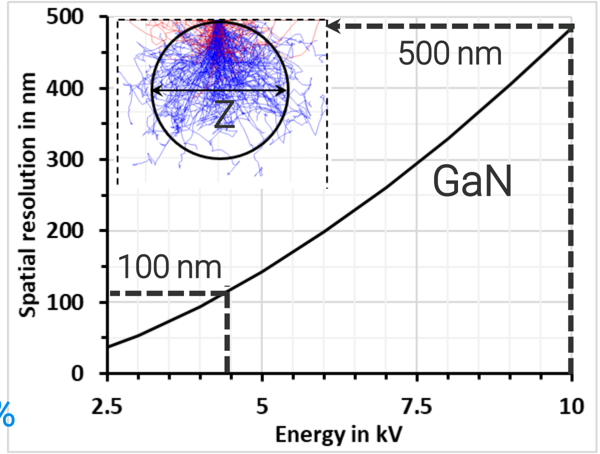
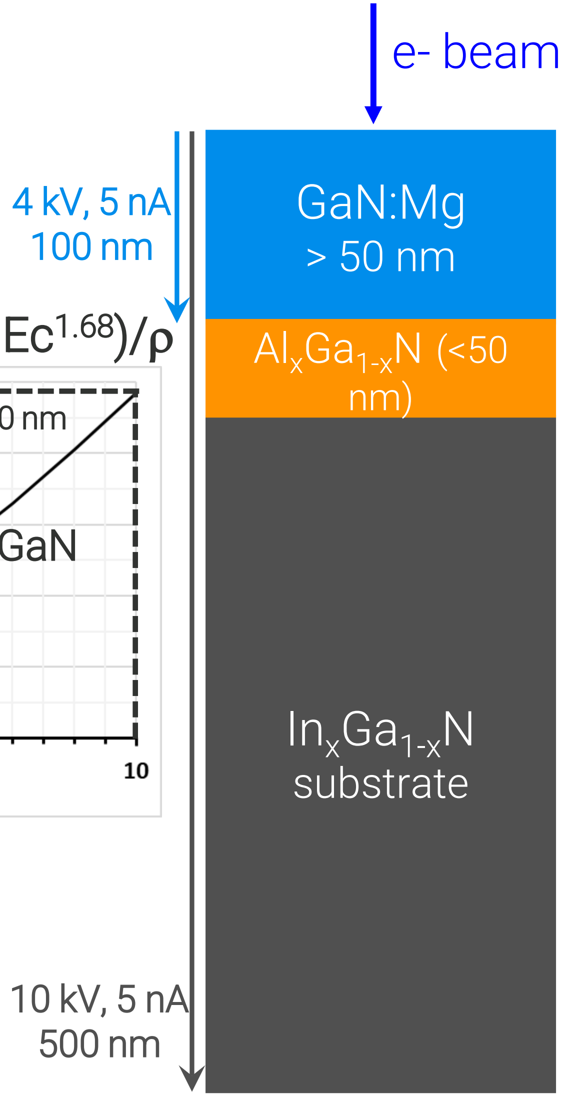
QUANTIFICATION OF Mg DOPANT IN GAN NANOWIRES



$$Z\,(\text{nm}) = 64(E^{1.68} - Ec^{1.68})/\rho$$



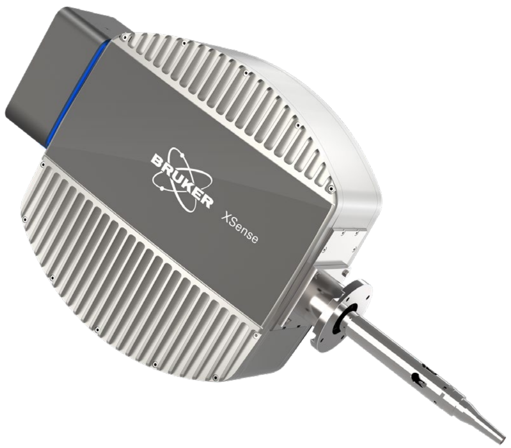


Motivation for a WDS on SEM

How does a WDS complement an EDS?

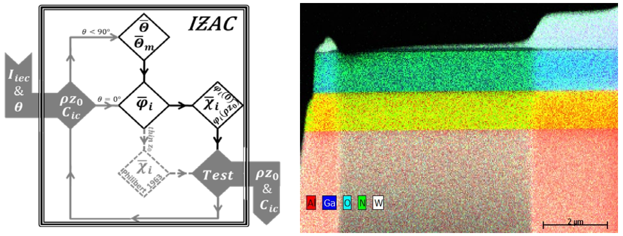
- Higher spectral resolution
  - ✓ Resolving peak overlaps
  - ✓ Resolution instead of deconvolution
- Light and trace element analyses
  - ✓ Low detection limit (including Be, B)
  - ✓ A few 100 ppm and below
- High spatial resolution
  - ✓ Ability to work at low voltage

XSense

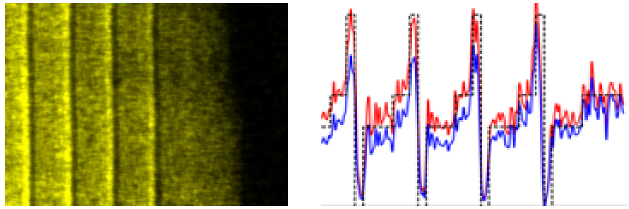


↳ Opens the way to the analysis of nanostructures in SEM!

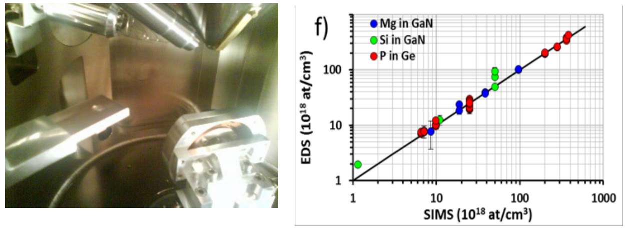
IZAC code

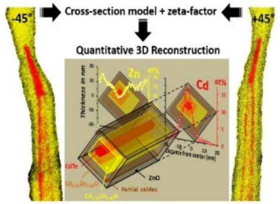


k- & ξ-factors



Doping





D3D tomo

Thank you for your attention

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Innovation with Integrity

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