

Energy dispersive X-ray spectroscopy: How far can we take line deconvolution?

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The main characteristic of silicon drift detectors (SDDs) is their extremely high pulse load capacity of up to 750,000 counts per second at good or reasonable energy resolution (123 eV Mn-K α , 46 eV C-K α at 100,000 counts per seconds). Compared to crystal wavelength dispersive X-ray spectroscopy (WDS), EDS provides better geometric collection efficiency and overall quantum efficiency. This and the high pulse throughput capability of the SDD result in a significantly fast signal collection compared to WDS. The inferior energy resolution of SDDs that causes peak overlaps can be compensated by deconvolution. Even for strong overlaps it is possible to deconvolve element lines in order to determine peak intensities using an extended atomic database. The latter has been updated by Bruker with a focus to line families in the low energy range ($E < 1$ keV) and M lines leading to over 250 additional lines in the energy range of 0–4 keV [1].

An example with strong peak overlaps is seen in samples which contain rare earth elements (REE), conventionally requiring a rather complex analytical protocol by WDS. Diagenetic concretions of the mineral monazite (REE)PO₄, from Upper Ordovician mudrocks of Central Wales, display REE zonation as revealed by intensity element maps and area spectra which were extracted out of a hyperspectral imaging EDS database (Figs. 1 A/B, Figs. 2 A/B). Standardless quantification with deconvolution of all spectra in the EDS database allows the display of quantitative element distributions through maps or linescans (Fig. 2C). This reveals concentration of gadolinium within the earliest deposited core of the concretion, followed by progressive sequential incorporation of the lighter REE, lanthanum dominating the outermost rim.

The second example focuses on a heterogeneously composed diffusion layer ~ 1 μ m in size at a Si₃N₄-Ti joint (Fig. 3) which was produced using a capacitor discharge joining technique and later heat treated at 900 °C [2]. A low accelerating voltage of 4 kV must be used in order to decrease the excitation volume for generated X-rays in the bulk sample. The strong peak overlap of nitrogen-K (392 eV) and titanium-L₁ (395 eV, Fig. 4A) and restriction of measurement time due to sample drift at high magnification exclude practical analysis with high current WDS methods. A hyperspectral linescan, acquired by EDS and quantified with reference standards shows a different spatial distribution for nitrogen and titanium (Fig. 4B) confirming the accuracy of the peak deconvolution model. A concurrent presence of titanium with silicon in the diffusion layer indicates the formation of a Ti_xSi_y compound. Point analysis of this phase corresponds to a stoichiometry of Ti₅Si₃ (62.6 \pm 2.5 at.% Ti and 37.4 \pm 2.5 at.% Si, n=5). These results are supported by TEM investigations [2] and the fact that Ti₅Si₃ is the thermodynamically most favorable stoichiometry.

This study has successfully demonstrated that the element distribution of overlapping lines at the intermediate and lowermost energy range can be analyzed on the micro- and nanoscale by EDS in a short time by peak deconvolution using complete element line families.

References

- [1] A. Abmann and M. Wendt, *Spectrochim. Acta*, 58 (2003) 711–716.
- [2] O. Tunçkan, *Joining ceramics using capacitor discharge technique and determination of metal ceramic interface reactions*, PhD thesis (2010), Anadolu University, Eskişehir, Turkey.

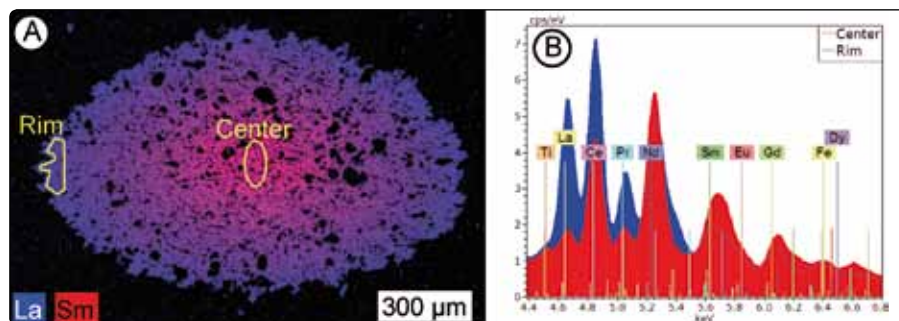


Fig. 1: (A) Intensity map of a zoned monazite and (B) area spectra extracted out of the EDS database displaying zonations of REE with enrichment of LREE (La, Ce) and depletion of intermediate REE (Nd, Sm, Gd) at the rim when compared to the center.

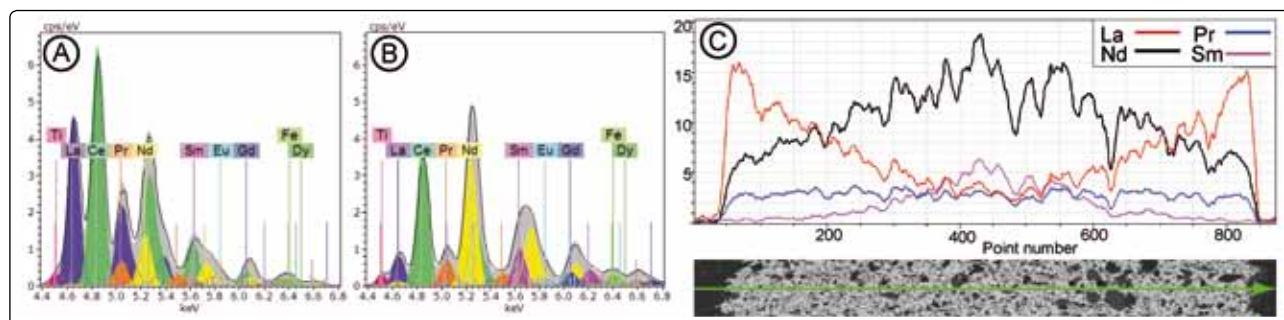


Fig. 2: Deconvolution result of the background corrected spectrum of (A) center and (B) rim shown in Fig. 1A. Colored peaks are deconvolved peaks for each element. The grey area is the sum of all colored peaks and perfectly matches the experimental result (black line). (C) Quantitative line scan (wt.%) showing the distribution of selected REE.



Fig. 3: BSE image of a Ti-Si₃N₄ joint showing a heterogeneously composed diffusion layer.

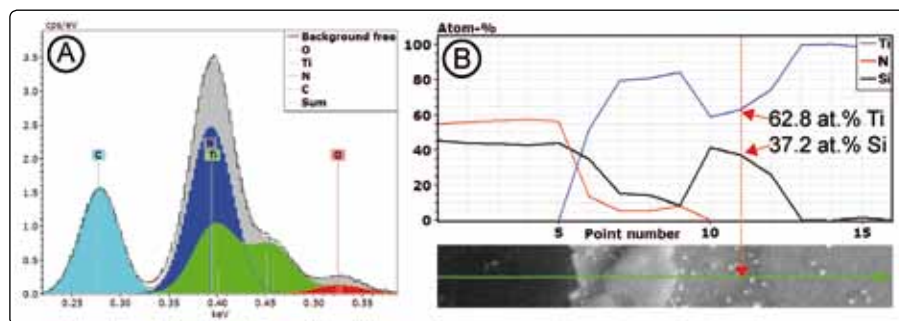


Fig. 4: (A) Deconvolution result of Ti and N. See Fig. 2A for additional information. (B) Quantitative linescan (at.%) of 16 points with 250 nm distance (4 μm line). Measurement conditions: ZEISS Supra 55LV, Bruker XFlash SDD 5010; 4 kV, 1.1 nA, 1.2 keps, 315 s (~20 s per point, limited by sample drift).