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From the micro- to nanoscale: How far can peak deconvolution be taken using Energy Dispersive X-ray Spectroscopy (EDS) with SEM?

T. Salge (1), O. Tunckan (2), S. Turan (3), A. Kearsley (1), and R Terborg (1)

(1) Bruker Nano GmbH, Schwarzschildstr. 12, 12489 Berlin, Germany (tobias.salge@bruker-nano.de), (2) Civil Aviation School, İki Eylul Campus, Anadolu University, 26555 Eskişehir, Turkey, (3) Materials Science and Engineering Department, Anadolu Univ., 26555 Eskişehir, Turkey, (4) Mineralogy Department, Natural History Museum, SW7 5BD London, UK

The main characteristic of silicon drift detectors (SDDs) is their extremely high pulse load capacity of up to 1,500,000 counts per second and good or reasonable energy resolution (121 eV Mn-Kα, 38 eV C-K). 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 algorithms. Even for strong overlaps it is possible to deconvolve peaks in order to determine their 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)PO_4, from Upper Ordovician mudrocks of Central Wales, display REE zonation as revealed by integrated peak intensity element maps and area spectra which were extracted out of a spectrum imaging EDS database. Standardless quantification with deconvolution of all spectra in the database allows display of quantitative element distributions by maps or linescans. 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 $\sim$1 μm in size at a Si_3N_4-Ti joint which was heat treated at a temperature of 900 ˚C using a capacitor discharge joining technique [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-Ll (395 eV) 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 confirming the accuracy of the peak deconvolution model. A concurrent presence of titanium with silicon in the diffusion layer is indicating the formation of a Ti_xSi_y compound. Point analysis of this phase corresponds to a stoichiometry of Ti_5Si_3 (62.6±2.5 at.% Ti and 37.4±2.5 at.% Si, n=5). These results are supported by TEM investigations [2] and the fact that Ti_5Si_3 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

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