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Correlated EDXS and EELS compositional analyses in Dy-doped Nd-Fe-B-based magnets

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The coercivity of Nd–Fe–B-based permanent magnets depends strongly on the microstructural features, such as the structure/chemistry properties of the matrix grains, the triple pockets (TPs) and the various internal interfaces. The partial replacement of the Nd by a heavy-rare-earth (HRE) element (Dy or Tb) as part of the grain-boundary diffusion process is known to have a large, positive influence on the coercivity, due to the formation of core-shell grains with a HRE-containing shell and a Nd–Fe–B-based core. It is believed that the shell, with its higher anisotropy field resulting from the HRE, hinders magnetisation reversal at the grain edges and leads to an increase in the coercivity of the magnet. To gain a deeper insight into the critical demagnetization processes in relation to the presence of HREs, it is vital to properly detect and quantify their location in the microstructure on the nanoscale.

In this study Dy-doped Nd–Fe–B-based magnets were used, allowing us to establish analytical procedures that facilitate the reliable quantification of HREs using transmission electron microscopy (TEM). The compositional analysis of the sample was obtained by using a scanning transmission electron microscope (STEM/TEM UHR Jeol 2010F) equipped with an energy-dispersive x-ray spectrometer (EDXS) (LINK ISIS EDS 300) and electron energy-loss spectroscopy (EELS) (Gatan PEELS 766).

The EDXS analysis of the matrix grain, which was performed with a large, 100x100nm analytical window, confirmed the expected concentrations of Nd and Fe. In addition, minor amounts of Pr, Co and Cu were identified. However, the presence of Dy could not be detected by EDXS. The problem of a reliable detection and quantification of small concentrations of Dy dopant in the Nd–Fe–B system by EDXS is related to the strong overlap of the Fe-K and Dy-L spectral lines. In addition, the reliability of EDXS analysis is further compromised by the overlapping spectral lines of elements that are required for the fine tuning of the Nd–Fe–B's magnetic properties and are typically present in small concentrations, for example, Co.

In contrast, the EELS Fe-$L_{2,3}$, Co-$L_{2,3}$ and Dy-$M_{4,5}$ ionization edges do not suffer from signal overlap and can therefore be reliably quantified. In addition, EELS offers the possibility for the detection and quantification of light elements, such as B and O. A serious overlap in EELS though can be found between the Pr-$M_{4,5}$ and Nd-$M_{4,5}$ edges, which are superimposed on the Cu-$L_{2,3}$ edge. To perform a complete compositional analysis of the Dy-doped Nd–Fe–B-based system we propose a correlated EDXS and EELS analytical procedure. Such a combined spectra acquisition facilitates not only a complete quantification of the most important elements (Nd, Fe and Dy), but also a full quantification of the minor (Co, Cu) and light elements (B, O) from the same specimen area.

Figure 1 shows a HAADF-STEM image of a Nd–Fe–B grain and the adjacent TP. Several EDXS and EELS line-scans were acquired across the interface. The TP is characterized by large amounts of Nd and Pr, about 42 at% and 13 at%, respectively. In addition, the concentration of Fe and Cu in the TP was found to be approximately 4 at%. While the concentration of these elements in the TP stays constant, the Co shows a gradual decrease in concentration from the interior of the TP towards the interface. The concentration of Co in the grain was found to be constant at around 2 at.%. The averaged background-subtracted EELS spectrum obtained from the 19 individual spectra acquired from the matrix grain is shown in Figure 2a. The presence of the Fe, Pr and Nd signals is clearly seen.In addition, the enlarged high-energy loss spectrum region clearly contains a Nd signal (Figure 2b). The average composition obtained by quantification of the EELS spectrum, applying theoretical cross-sections for selected elements, yields 80 at.% of Fe, 3 at.% of Pr and 15 at.% of Nd, which is roughly consistent with the EDXS results. In addition, the EELS analysis reveals that there is around 3 at.% of Dy present in the Nd–Fe–B grain close to the interface. The presented study confirms that correlated EDXS and EELS analysis can be successfully applied to fully quantify the Nd–Fe–B system, which includes a reliable analysis of small amounts of Dy.

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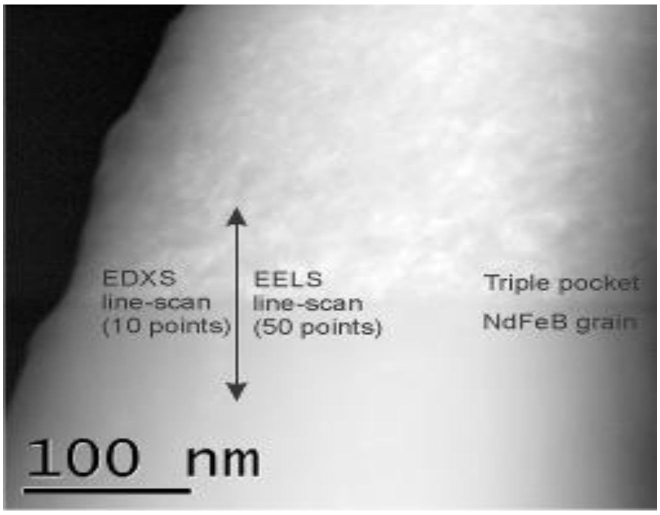

Figure 1. HAADF-STEM image of a Nd–Fe–B grain adjacent to a triple pocket. The EDXS and EELS line-scans are marked on the image.

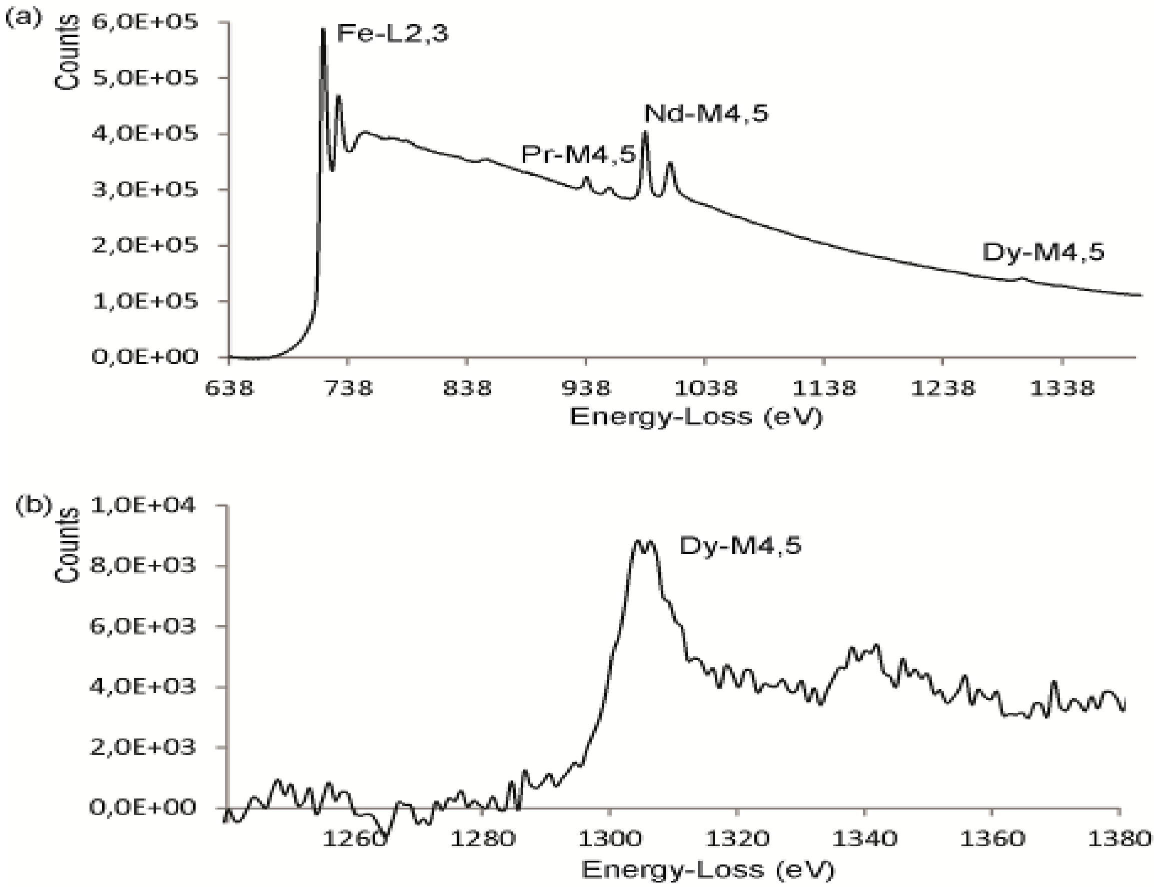

Figure 2. Background-subtracted (a) averaged EELS spectrum acquired from the Nd–Fe–B grain adjacent to the TP and (b) enlarged Dy-$M_{4,5}$ energy-loss region.