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ROLE OF Dy DIFFUSION IN SYNTERED Nd-Fe-B-TYPE HARD MAGNETS

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Abstract

High coercivity Nd-Fe-B permanent magnets play an important role in rapidly-growing renewable energy sector (electric vehicles or wind turbines). To retain the coercivity at high operating temperature, heavy-rare-earth elements (HRE), such as Dy and Tb, are added using grain-boundary diffusion (GBD) process. The addition of HRE results in a significant improvement of the coercivity due to the increase of the intrinsic resistance to demagnetization.

In the present study, we report on the correlation between magnetic properties and the distribution of the DyF_3 in the melt-spun $Nd_2Fe_{14}B$ ribbons that were coated with 2 wt.% Dy, spark-plasma sintered (SPS) and annealed at 600 °C for 30 h. During SPS and annealing process Dy diffused along grain boundaries (GB) into the outer parts of Nd-Fe-B grains, thus forming core-shell grains with Dy-rich shell and Nd-Fe-B core. The magnetometry measurements showed that the addition of Dy increased the coercivity in heat-treated samples by 25 %. For the structural and compositional studies, we used a Cs-corrected scanning transmission electron microscope (FEI Titan 80-200) equipped with SuperX electron dispersive X-ray (EDX) spectrometer and electron energy-loss (EEL) spectrometer (Gatan Enfinium ER model 977).

The analyses were carried out in the so-called wheel side region of the annealed sample where the size of the Dy-treated Nd-Fe-B grains varies between 50-100 nm. The EDX maps confirmed the core-shell-like structure. However, the reliability of the EDX analysis is compromised by overlapping of the Fe-K, Dy-L and Co-K lines. Therefore, to separate the contribution of these elements and provide more reliable compositional maps, EEL spectra of the same grains were recorded. Direct integration of the power law background-subtracted spectra does not allow to fully separate the different contributions as the Fe-L edge overlaps with the F-K edge tail overestimating the Fe contribution. An overlap of the Nd-M_3 and Dy- $M_{4,5}$ edges is also present at higher energies and prevent an accurate determination of the Dy concentration. To unwrap the spectra the vertex component algorithm (VCA) was applied and thus we were able to determine the local repartition of the present elements and extract Fe-$L_{2,3}$/Co $L_{2,3}$, Nd-$M_{4,5}$, Dy-$M_{4,5}$, F-K and O-K maps. The results confirmed the Dy,Nd-Fe phase formation at the shell around the pure Nd-Fe-B core grains. Further studies will focus on quantitative analysis of the as SPS sample which will at the end pave the way to observe the diffusion of DyF_3 as a function of heat-treatment process and correlate it with the magnetic properties of the magnet.