TEM analysis of high-coercive Nd-Fe-B sintered magnets treated by Tb-metal vapor sorption

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Coercivity of Nd-Fe-B sintered magnets is believed to be influenced by so-called "Nd-rich phase". However the crystal structure of the Nd-rich phase is not clarified yet. Recently, Tb-metal vapor sorption treatment has attracted much attention as a technique for great improvement of the coercivity [1]. We found that the Tb-treatment forms a thin and continuous wetting layer around $Nd_2Fe_{14}B$ grains [2]. In this study, behaviors of small amount elements in the Tb-treatment have been investigated by using TEMs equipped with an energy dispersive X-ray spectroscopy (EDS) and an electron energy loss spectroscopy (EELS), in order to reveal the structure and formation mechanism of the wetting layer phase.

TEM specimens were fabricated using a focused ion beam (FIB: HITACHI FB-2000K) mill with a micro-sampling unit from a central area of the Nd-Fe-Co-B magnets. Elemental analysis was carried out with an EDS (FEI TECNAI-20F) and with an EELS (JOEL JEM-3200FSK).

After the Tb-vapor sorption treatment, $Nd_2Fe_{14}B$ grains are corvered with thin layer of amorphous. A triple junction of grain boundaries consists of the amorphous and small Nd-O grains about 200 nm in diameter. The detailed EDS analysis showed that the amorphous phase scarcely contained Fe but Co and Nd. The distribution of Tb is not clear, because of the week intensities of the Tb-peaks for its small amount and the overlaps of other peaks; the Tb-$L_{\alpha 1}$ (6.27 keV), Fe-$K_{\alpha 1,2}$ (6.40 keV), Tb-$L_{\beta 1}$ (6.98 keV) and the Co-$K_{\alpha 1}$ (6.93 keV). The Tb-treatment allows phase transformation of the Nd-rich phase to the amorphouse phase and Nd-O phase, and migration of Co from the main $Nd_2Fe_{14}B$ to form the Co-Nd amorphous thin layer which wraps each $Nd_2Fe_{14}B$ grain. The wrapped structure prevents the nucleation of magnetic reversed domains and then improves the coercivity.

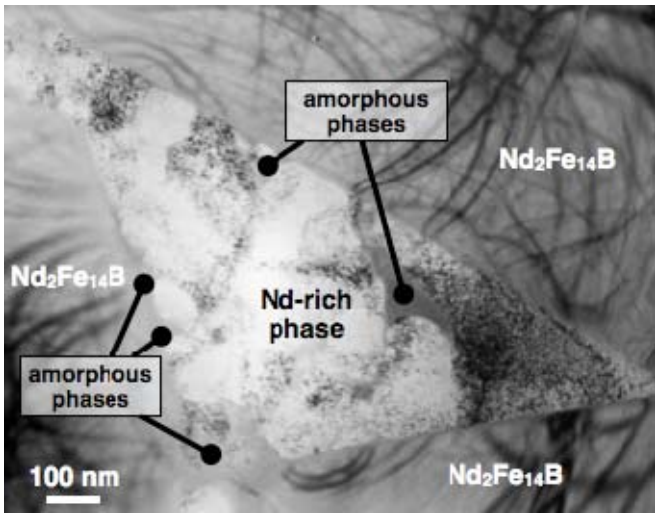

Fig.1 Typical microstructure of a triple junction of Tb-treated Nd-Fe-Co-B magnet.

Reference

1. S. Suzuki, K. Machida: Material Integration. **16** (2003) 17
2. N. Watanabe, M. Itakura, N. Kuwano, D. Li, S. Suzuki and K. Machida: *Materials Transactions* 48 (5), 915-918 (2007).