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[P2] Raw Materials & Recycling

Session Chair: Mr. Johann Fischbacher (University for Continuing Education Krems, Austria), Dr. Yusuke Hirayama (AIST, Japan)

[P2-32] Boosting the Coercivity of the Nd-Fe-B Alloy Recovered from Oxidized Scrap Magnets

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Securing a supply chain of rare earths has led to efforts in recycling Nd-Fe-B magnets (1). One common strategy is the magnet-to-magnet approach, where scrap magnets are reintroduced into the production chain and processed using traditional powder metallurgy (2). This approach, although straightforward, heavily depends on the condition of the scrap. If the magnet has been exposed to humidity, high temperatures, or corrosive environments, it is likely that the scrap will be oxidized to some degree. High oxygen levels result in poor magnetic properties in recycled magnets due to depletion in the Nd-rich phase, which is crucial for obtaining an adequate microstructure during processing (3). In extreme cases, oxidation can destroy the Nd₂Fe₁₄B phase partially. This scenario renders the magnet-to-magnet approach ineffective unless the Nd-Fe-B alloy is somehow recovered. The literature has addressed the recovery of Nd-Fe-B alloy from magnet machining waste (4). The reduction-diffusion (RD) process has been explored to recover the Nd-Fe-B alloy. However, this recovery process has not been thoroughly addressed for oxidized end-of-life magnets (5,6), which is the focus of the present work. The starting point involves oxidized magnet scrap recovered from a magnetic levitation unit. During operation, corrosion occurred, leading to an oxygen content of around 15000 ppm in the end-of-life condition. XRD analyses revealed that the scrap surface was primarily composed of iron oxides/hydroxides and neodymium oxides/hydroxides, with some Nd₂Fe₁₄B phase present, accounting for 15% wt. according to Rietveld refinement. Oxidation was limited to the surface and subsurface regions, leaving a preserved magnetized core in the end-of-life condition. The work focused on the oxidized portion, separating the preserved core from the corroded products. The scrap underwent demagnetization heat treatment under vacuum. The demagnetized product was sieved with 500 µm openings, yielding two portions: a <500 µm O-rich portion and a >500 µm O-poor portion. The O-rich portion had an oxygen content of 60000 ppm, and XRF analyses showed it contained 31% wt. of rare earths, balanced mainly by iron and trace elements. The RD process was then conducted to recover the Nd-Fe-B alloy from the O-rich portion. Metallic calcium was used as a reducing agent, followed by a series of heat treatments with varying time and temperature parameters. XRD analyses showed successful recovery of Nd₂Fe₁₄B after RD processing, alongside an iron-calcium perovskite by-product. Mössbauer spectra revealed the formation of a Nd-rich phase,

corresponding to 8% wt. of all Fe species present in the RD product. In addition to recovering Nd₂Fe₁₄B and Nd-rich phases, both the demagnetization and RD processes were tailored to obtain a final microstructure that increased the coercivity of the RD powder. Optimal heat treatment conditions resulted in RD powder with coercivity values greater than 1000 kA/m, representing 86% of the original magnet's coercivity before use. SEM analysis observed Nd₂Fe₁₄B grains in the RD product with an average diameter of $1.8 \pm 0.3 \mu\text{m}$. The key to achieving such a fine-grained microstructure was the demagnetization step. XRD analyses of the demagnetized O-rich portion revealed it was mainly composed of alpha iron and neodymium oxides/hydroxides. A closer look at the XRD pattern showed broad peaks for alpha iron, indicating fine crystallite sizes of 21.5 nm, according to the Scherrer equation. Alpha iron was absent before demagnetization and was likely formed during treatment due to preserved metallic phases like Nd₂Fe₁₄B acting as reducing agents for iron oxides/hydroxides. Mössbauer spectra analysis after demagnetization revealed that 60% of all Fe species corresponded to alpha iron, whereas no alpha iron was detected before demagnetization. This nanostructure favored the formation of a fine-grained Nd-Fe-B microstructure during RD processing, resulting in a coercive powder. (1) J. Ormerod, et. al. *Sustainability*, 2023. vol. 15. 14901. (2) S. Rivoirard, et. al. *Proc. 16th Int. Workshop on Rare-Earth Magnets and Their Applications* (Sendai Japan), 2000. p. 347. (3) M. Zakotnik, I.R. Harris, A.J. Williams. *Journal of Alloys and Compounds*, 2009. vol. 469, p. 314 321. (4) M. Yue, et. al. *ACS Sustainable Chemistry & Engineering*, 2018. vol. 6. issue 5. (5) A. Chetouani, F. Richomme, J.M. Le Breton. *Materialia*, 2018. vol. 36, 102189. (6) S.X. Lima, et. al. *Transforming Highly Corroded Scrap Magnets Into Coercive Powders*. *IEEE International Magnetism Conference (INTERMAG)*, 2024. vol. 60, no. 9, 2100704.