

Poster | Material, processing, and characterization

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## **[P1] RE-Fe-B Magnets**

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

[P1-1]

High performance HREE-free hot-deformed Nd-Fe-B magnets by Nd-Cu grain boundary diffusion

\*Kazumasa Fujimura<sup>1</sup>, Takanori Kajiwara<sup>1</sup>, Takashi Oikawa<sup>1</sup>, Hiroshi Miyawaki<sup>2</sup> (1. Corporate R&D center, Daido Steel Co., Ltd. (Japan), 2. Material solution dept., Daido Steel Co., Ltd. (Japan))

[P1-2]

Development of Heavy Rare-Earth, Co-free Nd-Fe-B Injection Molded Anisotropic Bonded Magnet with High Corrosion Resistance

\*Kazuaki Shimba<sup>1</sup>, Takumi Otaki<sup>1</sup>, Masaya Shintaku<sup>1</sup>, Satoshi Sugimoto<sup>2</sup>, Hironari Mitarai<sup>1</sup> (1. Aichi Steel Corporation (Japan), 2. Department of Management Science and Technology, Graduate School of Engineering, Tohoku University (Japan))

[P1-3]

Impact of the RFe<sub>2</sub> phase in Ce-containing magnets on annealing optimization and magnetic performance

Fengqiao Liu<sup>1</sup>, \*Yunqiao Wang<sup>1</sup>, Wei Zhu<sup>1</sup>, E Niu<sup>1</sup>, Zhan Wang<sup>1</sup>, Xiaolei Rao<sup>1</sup>, Boping Hu<sup>1</sup> (1. Beijing Zhong Ke San Huan Research (China))

[P1-4]

Coercivity Enhancement and Synergistic Suppression of CeFe<sub>2</sub> Phase in Ce Magnets with High Ce Content

\*Minggang Zhu<sup>1,2</sup>, Xiaolong Song<sup>2</sup>, Qisong Sun<sup>2</sup>, Yikun Fang<sup>2</sup>, Wei Li<sup>2</sup> (1. AT&M North Technology Co.,Ltd (China), 2. Central Iron and Steel Research Institute (China))

[P1-5]

Enhanced Magnetic Properties and Microstructural Characterization of Hot-Deformed (Ce,L)-Fe-B Magnets with Eutectic Alloy Incorporation

\*Kyungmi Lee<sup>1</sup>, Ye Ryeong Jang<sup>1</sup>, Wooyoung Lee<sup>1</sup> (1. Yonsei Univ. (Korea))

[P1-6]

Simultaneous improvement in coercivity and remanence of (Nd, Pr)-ultra-saving Ce-substituted RE-Fe-B sintered magnets by grain boundary diffusion process using low-melting Nd-Cu-Al-Ga alloy

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[P1-7]

Microstructural Optimization and Coercivity Enhancement in Nd-Ce-Fe-B Magnets through Grain boundary diffusion of Pr-La Mixed Alloy

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[P1-8]

### Hysteretic properties of (Nd,Ce,Tb)-(Fe,Co)-(Al,Cu,Ti)-B permanent magnets prepared by in-situ grain boundary diffusion

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[P1-9]

### Utilization of high-abundance rare earth elements in Tb-Cu-Al alloy for high efficient grain boundary diffusion of Nd-Fe-B magnets

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[P1-10]

### A macroscopic perspective on the sintered Nd-Fe-B magnets prepared by Tb grain boundary diffusion

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[P1-11]

### Regulation of Grain Boundary Structure in NdFeCoB Magnets through Grain Boundary Diffusion of DyAlCu Alloy

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[P1-12]

### Coercivity enhancement of Nd-Fe-B sintered magnet through grain boundary restructuring using Dy<sub>80</sub>Ga<sub>20</sub> eutectic alloy

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[P1-13]

### Nb assisted grain boundary pinning in Nd-Cu diffused Nd-Fe-B magnets for enhancing the Coercivity

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[P1-14]

### Effects of trace elements on the grain boundary diffusion of sintered NdFeB magnets

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[P1-15]

### Effect of Coating Methods on the Magnetic Properties of Grain Boundary Diffusion Processed Nd-Fe-B Sintered Magnets

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[P1-16]

Magnetization reversal of core-shell structured grain of GBDP Nd-Fe-B sintered magnet

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[P1-17]

Novel Aspects in Nd-Fe-B Grain Boundary Engineering: Integrating (Electro)chemistry and Materials Science

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[P1-19]

Features of the magnetization reversal processes in sintered permanent magnets Nd-Fe-B and Sm-Co type

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[P1-20]

Potential of Cryogenic Treatment Applications on Rare-Earth-Based Functional Magnetic Materials

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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-1] High performance HREE-free hot-deformed Nd-Fe-B magnets by Nd-Cu grain boundary diffusion

\*Kazumasa Fujimura<sup>1</sup>, Takanori Kajiwar<sup>1</sup>, Takashi Oikawa<sup>1</sup>, Hiroshi Miyawaki<sup>2</sup> (1. Corporate R&D center, Daido Steel Co., Ltd. (Japan), 2. Material solution dept., Daido Steel Co., Ltd. (Japan))

Keywords : xEV、Nd-Fe-B magnet、HREE-free、Hot-deformed magnet、Grain boundary diffusion

Nd-Fe-B magnets used in EVs require high coercivity to withstand high temperatures and high antimagnetic fields. One of the methods to improve the coercivity of hot-deformed Nd-Fe-B magnets without using heavy rare earth elements such as Dy and Tb is the grain boundary diffusion process using light rare earth elements [1], [2]. In this process, a eutectic alloy such as Nd-Cu is applied to the magnet surface followed by heat treatment at a low temperature of 500-700°C. This allows the eutectic alloy to diffuse from the surface to the interior of the magnet while inhibiting the growth of fine crystal grains, widening the grain boundary phase, weakening the magnetic interaction between the Nd<sub>2</sub>Fe<sub>14</sub>B particles resulting in an improvement in coercivity.

Fig. 1(a) shows changes in remanence ( $B_r$ ) after Nd-Cu alloy diffusion, and Fig. 1(b) shows increases in coercivity ( $H_{cj}$ ) from the base magnet. The decrease in  $B_r$  due to grain boundary diffusion is constant regardless of  $H_{cj}$  of the base magnet. However, the larger the amount of increase in coercivity, the smaller the coercivity of the base magnet. This suggests that higher hard magnetic properties can be obtained by using a magnet with higher  $B_r$  as the base magnet. Therefore, a base magnet with low RE composition is desirable. In this report, hot-deformed magnets with  $B_r$  of 1.42 T at room temperature and  $H_{cj}$  of 553 kA/m at 150°C and  $H_k/H_{cj}$  of 98% at 150°C was used as the base magnet and Nd-Cu alloy was diffused into the grain boundary. The  $H_{cj}$  of the magnet was improved to 704 kA/m at 150°C with  $B_r$  of 1.39 T at room temperature without HREE. The  $H_k/H_{cj}$  is still maintained at 96% at 150°C, which is adequate for usage of EVs.

#### References

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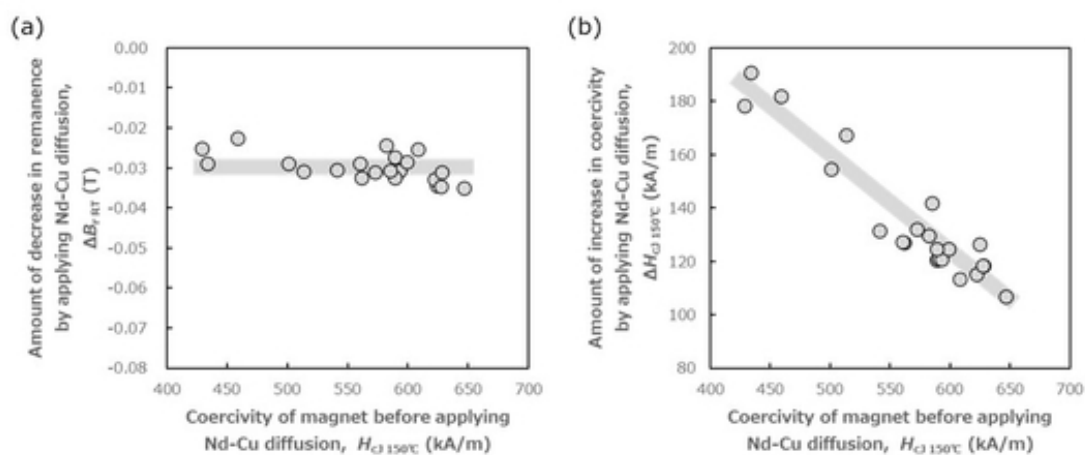


Fig. 1. (a, b) Change in remanence at room temperature and coercivity at 150°C by Nd-Cu diffusion

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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-2] Development of Heavy Rare-Earth, Co-free Nd-Fe-B Injection Molded Anisotropic Bonded Magnet with High Corrosion Resistance

\*Kazuaki Shimba<sup>1</sup>, Takumi Otaki<sup>1</sup>, Masaya Shintaku<sup>1</sup>, Satoshi Sugimoto<sup>2</sup>, Hironari Mitarai<sup>1</sup> (1. Aichi Steel Corporation (Japan), 2. Department of Management Science and Technology, Graduate School of Engineering, Tohoku University (Japan))

Keywords : Nd-Fe-B Anisotropic Bonded Magnet、Nd-Fe-B HDDR powder、Corrosion Resistance、Surface treatment of powders、Injection Molding

#### [Introduction]

An electric water pump (EWP) is an essential component of electric vehicles and is used to cool main functional components. Owing to size reduction requirements, Nd-Fe-B bonded magnets are used in EWP applications [1]. In EWPs, bonded magnet requires long-term durability, which implies low flux loss in hot water. Bonded magnets with high corrosion resistance have been fabricated using phosphoric acid-treated rare-earth powders [2-3]. Additionally, we investigated the phosphoric acid treatment of heavy rare-earth and Co-free anisotropic Nd-Fe-B powders prepared by dynamic hydrogenation disproportionation desorption recombination *d*-HDDR [4-5] treatment. Phosphoric acid-treated powders baked above 250 °C exhibits high corrosion resistance [6]. In this study, we develop injection-molded magnets with high corrosion resistance by applying oxidation treatment and non-kneading process.

#### [Experiment]

A base alloy with a composition of Nd<sub>12.5</sub>Fe<sub>ba1</sub>Ga<sub>0.3</sub>Nb<sub>0.2</sub>B<sub>6.2</sub> (at. %) was prepared. After *d*-HDDR treatment and sieving (< 212 μm), raw powders were obtained. Before the phosphoric acid treatment, the raw powders were oxidised in air at 160–180 °C for 3 h. The oxidised powders were treated with phosphoric acid using the method described in the literature [6]. The baking temperature was 300 °C. After sieving (< 212 μm), oxidised and phosphoric acid-treated (referred to as OPAT in short) powders were obtained. The non-kneading compounds were fabricated as follows: The OPAT powders were coated with 0.5 wt. % amino-silane coupling agents. The treated powders were mixed with 11–14 wt. % polyphenylene-sulfide resin. The mixture was heated at 300 °C and blended using the extruder that was excluded parts adding strong shear stress to powders. Magnets measuring 11×11×11 mm<sup>3</sup> were fabricated by heating the compounds at 300 °C and injection molding its, in oriented magnetic field of 1.5 MA/m. The magnet fabricated using the non-kneading process with OPAT powders are called as MA, and raw powders are called as MB. Magnets were also fabricated using compounds prepared by kneading raw powders and resin by a typical process, in which powders were subjected to strong shear stress. This sample is called as MC.

The magnetic properties of the powders were measured at RT by VSM. STEM was employed for microstructural observations. In this study, the corrosion resistance of the powders was evaluated based on the change ratio of the coercivity ( $\Delta H_{cj}$ ) before and after the anticorrosion test. This test was performed by immersing the powders in an aq. solution of 50 % long life coolant (LLC) at 150 °C for 100 h.

The magnetic properties of the magnets were measured using a BH tracer at RT. The irreversible flux loss ( $\Delta F_{ir}$ ) of the magnets was calculated based on the surface magnetic flux density measured using a flux meter after immersion in an aq. of 50 % LLC at 80 °C and cooling to RT.

#### [Results and discussion]

The  $\Delta H_{cj}$  value of the OPAT powder was -1.6 %, whereas that of a sample without oxidation treatment was -3.0 %. Based on microstructural observations, an oxidised layer with a thickness of 300–400 nm was fabricated below the phosphate layer. The thickness of the phosphate layer was 100–200 nm and remained unchanged with or without the oxidation treatment. These results indicate that the corrosion resistance of the OPAT powder was improved by the thick multilayers.

Based on the microstructural observations, the powders in MA maintained a multilayer structure. The magnetic properties of MA included a remanence ( $B_r$ ) of 0.73 T and a coercive force ( $H_{cj}$ ) of 1060 kA/m. Fig. 1 shows the  $\Delta F_{ir}$  values of the samples after immersion in 80 °C aqueous solution. The  $\Delta F_{ir}$  of the magnet MA was -2.4 % at 8000 h, whereas those of MB and MC were -8 % and -15 % at 1000 h, respectively.

#### [Conclusion]

The corrosion resistance of the phosphoric acid-treated powder was improved by oxidation treatment. The surface layer of the OPAT powder was maintained after injection molding. In conclusion, Nd-Fe-B anisotropic bonded magnets with high corrosion resistance were obtained, which can be used in applications operating in harsh environments, e.g. EWP.

#### [Acknowledgements]

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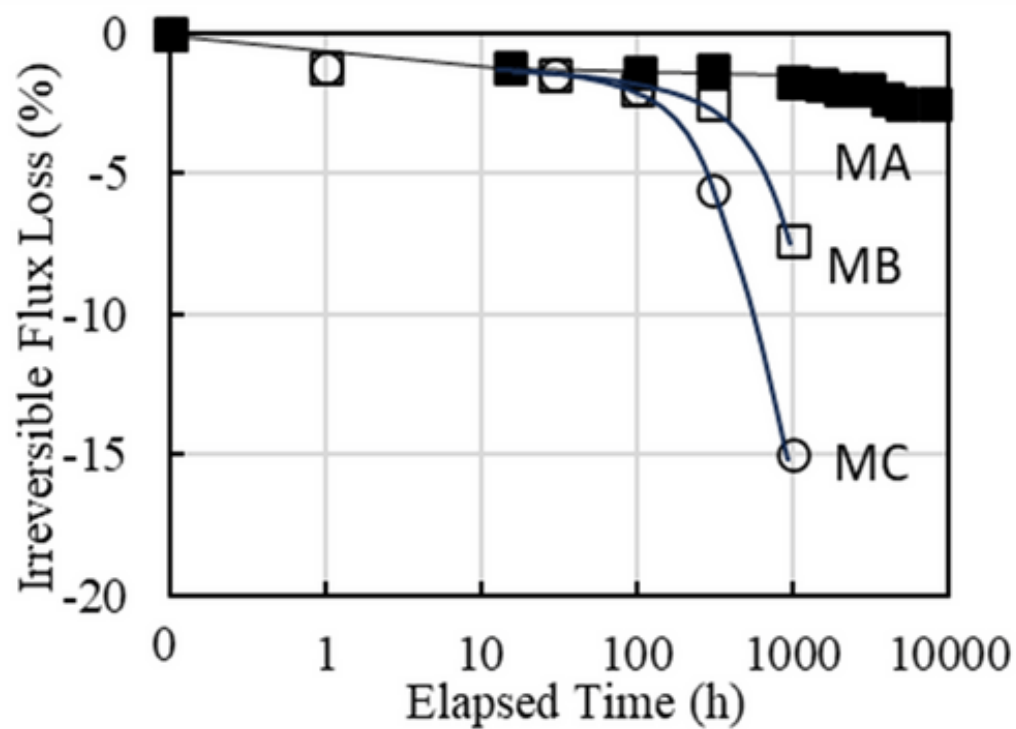


Fig. 1. Irreversible flux loss of samples MA, MB, and MC after exposure to 80 °C in an aqueous solution of 50 % LLC.



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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-3] Impact of the RFe<sub>2</sub> phase in Ce-containing magnets on annealing optimization and magnetic performance

Fengqiao Liu<sup>1</sup>, \*Yunqiao Wang<sup>1</sup>, Wei Zhu<sup>1</sup>, E Niu<sup>1</sup>, Zhan Wang<sup>1</sup>, Xiaolei Rao<sup>1</sup>, Boping Hu<sup>1</sup> (1. Beijing Zhong Ke San Huan Research (China))

Keywords : Ce - containing magnet、RFe<sub>2</sub> phase、annealing、Ce/R ratio、transition temperature

Ce - containing magnets have been widely applied in many fields due to their high cost - performance. Compared with Nd magnets, it is well - established that Ce - containing magnets exhibit a greater tendency to form the RFe<sub>2</sub> (R = Ce, Nd) phase. This phase exerts a relatively substantial influence on their magnetic properties. Moreover, it impacts the grain - boundary diffusion process. Additionally, owing to the complexity of phase formation, the annealing process for Ce - containing magnets is generally more intricate than that of Nd magnets. This complexity has implications for both the production and application of Ce - containing magnets. Therefore, in this study, we conducted systematic research to reveal the relationships among the evolution of the RFe<sub>2</sub> phase, the annealing process, and the magnetic properties.

Ce - containing magnets with different Ce contents (5 - 13%) were annealed at various temperatures. Their magnetic properties were tested, and microstructural observations were carried out using a scanning electron microscope (SEM). Meanwhile, RFe<sub>2</sub> (R = Ce, Nd) alloys featuring different Ce/R ratios were fabricated and heat-treated. Subsequently, differential scanning calorimeter (DSC) was employed to determine their phase - transition temperatures. Through the research, we found that within this content range, as the Ce/R ratio increases, the transition point of RFe<sub>2</sub> gradually rises. Correspondingly, the optimal heat treatment temperature for Ce - containing magnets also increases. SEM analysis also revealed that as the amount of Ce substitution increases, the area of RFe<sub>2</sub> in the magnets after being treated at the optimal heat treatment temperature increases. Moreover, the Ce/R ratio in the RFe<sub>2</sub> phase also rises. It is concluded that the Ce/R ratio of RFe<sub>2</sub> phase in magnets increases with increasing Ce content, resulting in the variation of transition temperature of RFe<sub>2</sub> phase being enhanced simultaneously, which seriously impacts on annealing temperature.

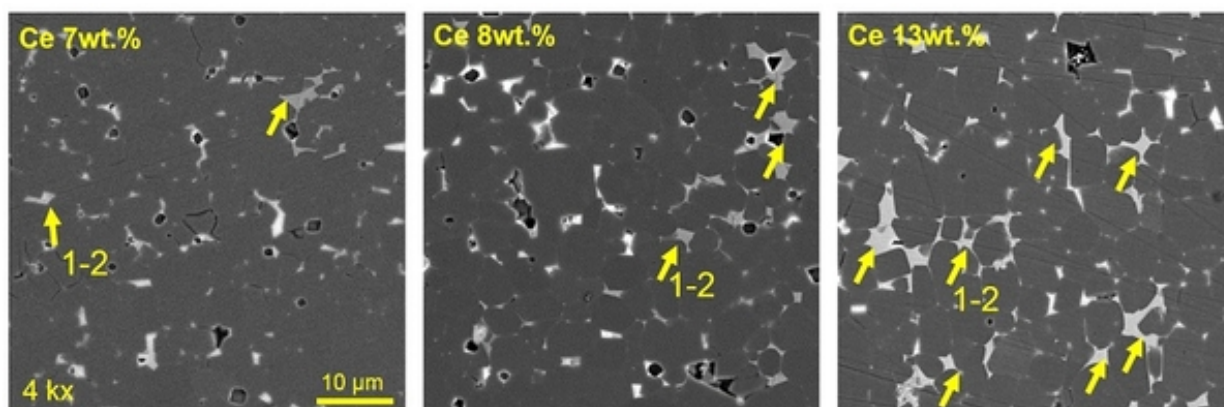


Fig.1. The area ratio of  $RFe_2$  increases with the increase of Ce content in Ce - containing magnets annealed at the optimal temperature

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## [P1] RE-Fe-B Magnets

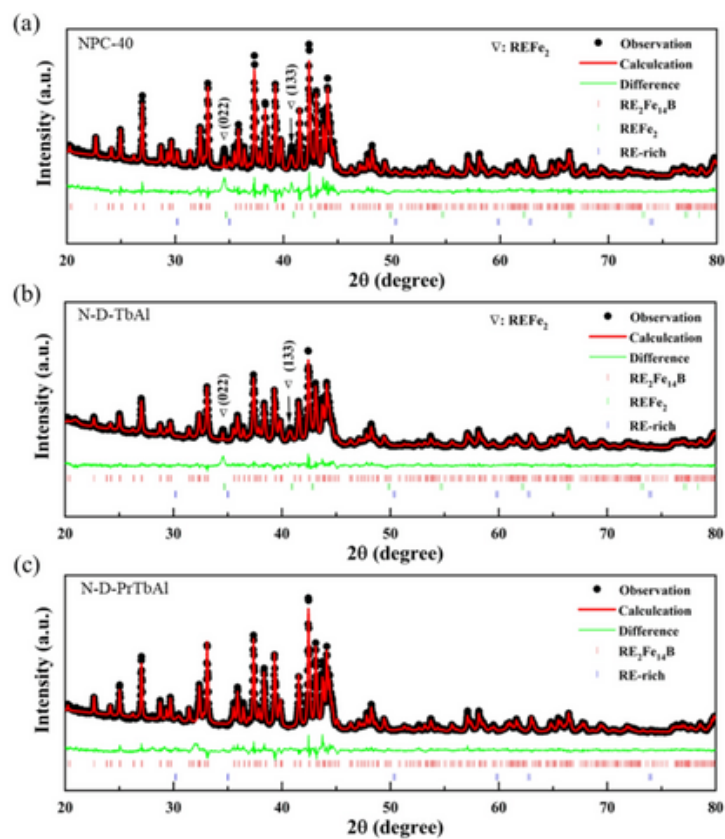
Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-4] Coercivity Enhancement and Synergistic Suppression of CeFe<sub>2</sub> Phase in Ce Magnets with High Ce Content

\*Minggang Zhu<sup>1,2</sup>, Xiaolong Song<sup>2</sup>, Qisong Sun<sup>2</sup>, Yikun Fang<sup>2</sup>, Wei Li<sup>2</sup> (1. AT&M North Technology Co.,Ltd (China), 2. Central Iron and Steel Research Institute (China))

Keywords : Ce magnet、Coercivity、High Ce Content、CeFe<sub>2</sub>

Earlier, we developed dual-main-phase sintered Ce magnet successfully, which solved the problem of the application of Ce element in magnets to a large extent. Nowadays, Ce magnets have already formed an industry in China, standing firmly within the permanent magnet sector, and have consistently been a hot pursuit among researchers. Our recent studies have successfully developed quasi-trivalent Ce magnets free from CeFe<sub>2</sub> through valence state regulation of Ce. However, the traditional cerium magnets produced in factories still suffer from the presence of the CeFe<sub>2</sub> phase, which deteriorates the magnetic properties. To expand the application areas of Ce magnets with high Ce content, it is crucial to improve their performance and to mitigate of CeFe<sub>2</sub> phase. In this work, we propose a strategy for the efficient utilization of heavy rare earth Tb to enhance the coercivity while synergistic suppressing of CeFe<sub>2</sub> phase in Ce magnets with high Ce-content. The design of diffusion-source alloy compositions is based on first-principles calculations, where diffusion behavior of Tb elements in Ce magnets with high Ce content is improved by the mechanism of synergistic action of Pr elements. The synergistic diffusion of Pr and Tb elements in the experiment contributes to the improvement of coercivity and suppress the CeFe<sub>2</sub> phase in the Ce magnet. The microstructure, phase composition, magnetization reversal, and magnetic domain evolution processes reveal the mechanism of performance enhancement. The strategy of efficient coercivity enhancement and suppression of the CeFe<sub>2</sub> phase will provide new support for expanding the application areas of Ce magnets with high Ce content.



**Fig.1** The Rietveld refinement results of XRD data: (a) Original magnet; (b) Tb-Al diffused magnet; (c) Pr-Tb-Al diffused magnet.

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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-5] Enhanced Magnetic Properties and Microstructural Characterization of Hot-Deformed (Ce,Lu)-Fe-B Magnets with Eutectic Alloy Incorporation

\*Kyungmi Lee<sup>1</sup>, Ye Ryeong Jang<sup>1</sup>, Wooyoung Lee<sup>1</sup> (1. Yonsei Univ. (Korea))

Keywords : Permanent Magnet, Hot-Deformed

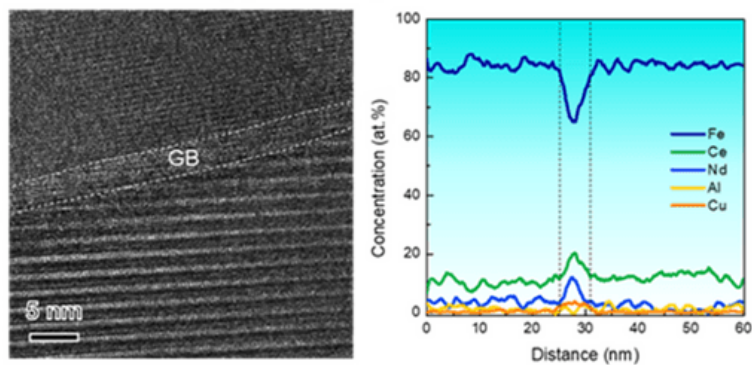
Due to increasing issues regarding the depletion and rising costs of heavy rare earth (HRE) elements, research on reducing HRE usage while maintaining high-performance permanent magnets has gained significant attention. As a result, light rare earth (LRE) elements, which are more abundant and cost-effective, have been widely explored as potential substitutes, with Ce being widely considered as a candidate. However, Ce-Fe-B alloys exhibit inferior magnetic properties compared to Nd-Fe-B due to the lower intrinsic performance of the 2:14:1 phase and the formation of the paramagnetic CeFe<sub>2</sub> phase, which adversely affects the overall magnetic properties.

In this study, we aimed to enhance the magnetic properties of Ce-Fe-B-based magnets by producing fine-grained powders through the melt-spinning process, followed by hot deformation to develop anisotropic magnets. However, in Ce-Fe-B magnets, the formation of CeFe<sub>2</sub> secondary phases inhibited grain alignment, resulting in low remanence and discontinuous, weakly defined grain boundaries, leading to a decrease in coercivity. To address this issue, we fabricated (Ce,Lu)-Fe-B magnets by partially substituting Ce with Lu and further improved the magnetic properties by incorporating a eutectic alloy.

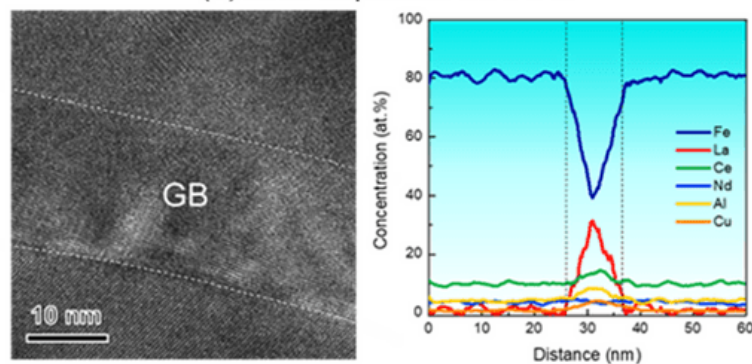
The experimental results indicated that the Ce-Fe-B magnet exhibited minimal grain boundary formation after hot deformation due to the absence of a RE-rich phase. Additionally, the high concentration of CeFe<sub>2</sub> phases inhibited diffusion, limiting the improvement in magnetic properties even after eutectic alloy incorporation. In contrast, in (Ce,Lu)-Fe-B magnets, the decrease in Ce content inhibited the formation of the CeFe<sub>2</sub> phase, which enabled the formation of a RE-rich phase essential for grain boundary formation during hot deformation. Furthermore, eutectic alloy incorporation further inhibited CeFe<sub>2</sub> formation, promoted grain boundary formation, and enhanced texture alignment.

As a result, the remanence and coercivity of the hot-deformed magnets were enhanced, achieving a maximum (BH)<sub>max</sub> of 5.7 MGOe in (Ce,Lu)-Fe-B magnets with a small amount of eutectic alloy incorporation.

(a) NAC doped Ce-Fe-B



(b) NAC doped La-Ce-Fe-B



**Figure 1.** High-resolution TEM images and corresponding EDS line profiles of grain boundaries (GB) in eutectic alloy-incorporated Ce-Fe-B and (Ce,La)-Fe-B magnets

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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-6] Simultaneous improvement in coercivity and remanence of (Nd, Pr)-ultra-saving Ce-substituted RE-Fe-B sintered magnets by grain boundary diffusion process using low-melting Nd-Cu-Al-Ga alloy

\*Sujin Lee<sup>1</sup>, Sumin Kim<sup>1</sup>, Tae-Hoon Kim<sup>1</sup>, Kyoung-Hoon Bae<sup>2</sup>, Dong-Hwan Kim<sup>2</sup>, Jung-Goo Lee<sup>1</sup> (1. Korea Institute of Materials Science (Korea), 2. Star Group Ind. CO., Ltd. (Korea))

Keywords : Ce-based magnets, Grain boundary diffusion process, Coercivity, High Nd/Pr-saving, Remanence, High-performance

The Nd-Fe-B-based magnets are widely employed in the various industries, including wind power generation, intelligent robotics, and electric/hybrid vehicles, due to their superior hard magnetic properties derived from high coercivity and remanence of Nd<sub>2</sub>Fe<sub>14</sub>B (2-14-1) grains<sup>1</sup>). However, the increasing demand for the Nd-Fe-B magnets has led to a substantial depletion of critical rare earth elements (RE), particularly Nd and Pr, resulting in concerns on their cost and supply stability<sup>2</sup>). Therefore, significant research efforts have been directed toward developing high-performance permanent magnets with reduced Nd and Pr<sup>3</sup>). Among potential substitutes, Ce have emerged as a promising candidate for partially replacing Nd/Pr in the magnets due to its abundant availability and cost-effectiveness<sup>4</sup>). However, the inferior intrinsic properties of Ce<sub>2</sub>Fe<sub>14</sub>B compared to Nd<sub>2</sub>Fe<sub>14</sub>B present a significant challenge in achieving high-performance (Nd,Pr)-saving magnets. In particular, excessive Ce substitution (>40 % of the total RE) severely degrades the hard magnetic properties<sup>5</sup>), which creates a significant limit for increasing Nd/Pr-saving rate of the magnets. In this work, we demonstrate a simultaneous enhancement of coercivity and remanence in ~57 % (Nd, Pr)-saving magnets *via* the grain boundary diffusion process (GBDP) using a low-melting Nd-Cu-Al-Ga (NCAG) alloy as the diffusion source.

The Nd/Pr-saving base magnets with the composition of (Nd, Pr)<sub>13.0</sub>Ce<sub>14.3</sub>Ho<sub>2.7</sub>B<sub>0.9</sub>Co<sub>1.0</sub>Cu<sub>0.2</sub>Al<sub>1.7</sub>Ga<sub>1.4</sub>Fe<sub>bal.</sub> (wt.%) were prepared for GBDP. A small amount of Ho, which can increase the anisotropy field of 2-14-1, was co-doped with Ce. Therefore, the Nd/Pr-saving level [(Ce+Ho)/(Nd+Pr+Ce+Ho)] of the magnets prepared for this work was ~57 %. To resolve the deterioration of magnetic properties induced by Ce in 2-14-1 grains, the Nd-based alloy (NCAG) was selected as the diffusion source satisfying two criteria: (1) RE in the GBD source should have a negative substitution energy for Ce in the 2-14-1 lattice to extract the Ce from 2-14-1 to Nd-rich GBP, and (2) the RE in the GBD source should be able to increase both the anisotropy field and saturation magnetization of 2-14-1 in Ce-substituted magnets. The coating amount of NCAG source for the base magnet was 8 wt.%. The coated magnets were heat-treated at 970°C for 15 h for GBD,

followed by post-diffusion annealing at 550°C for 2h. Subsequently, we examined the magnetic and microstructural analyses for the GBD-treated and post-diffusion annealed magnets.

As shown in the second-quadrant demagnetization curves in Fig.1(a), the coercivity of the as-sintered (Nd, Pr)-saving magnets substantially increased from 0.90 T to 1.53 T by the NCAG-GBDP, and it further increased to 1.65 T after the post diffusion annealing. Additionally, the remanence of the as-sintered magnets also improved to 1.07 T after NCAG-GBDP, exhibiting the gain of 0.03 T. The NCAG-GBDP simultaneously improved the coercivity and the remanence of (Nd, Pr)-saving magnets. Figs. 1(b) and (c) show the back scattered electron (BSE) images and electron probe micro-analyzer (EPMA) maps for Ce and Nd acquired from center region (~1650  $\mu\text{m}$  depth) of the GBD-treated and post-diffusion annealed magnets, respectively. For the GBD-treated magnet, the substitution of Nd with Ce in the outer region of 2-14-1 grains was observed even at the magnet center, indicating that the Nd-rich shell, which can suppress the reverse domain nucleation<sup>6)</sup>, is formed in the entire region of (Nd, Pr)-saving magnets. Note that post-diffusion annealing facilitated further extraction of Ce from the shell, allowing more Nd to diffuse from the Nd-rich GBP into the shell. These results provide a clear evidence for the enhancement of both coercivity and remanence of high Ce-substituted magnets *via* NCAG-GBDP. In this presentation, we will discuss the detailed microstructural changes induced by the NCAG-GBDP, specifically focused on the compositional changes of shell before and after the GBDP. Based on these results, we will suggest the key factors in the GBDP for improving the hard magnetic properties of high-Ce-content magnets to a level of commercial (Nd, Pr)-Fe-B sintered magnets.

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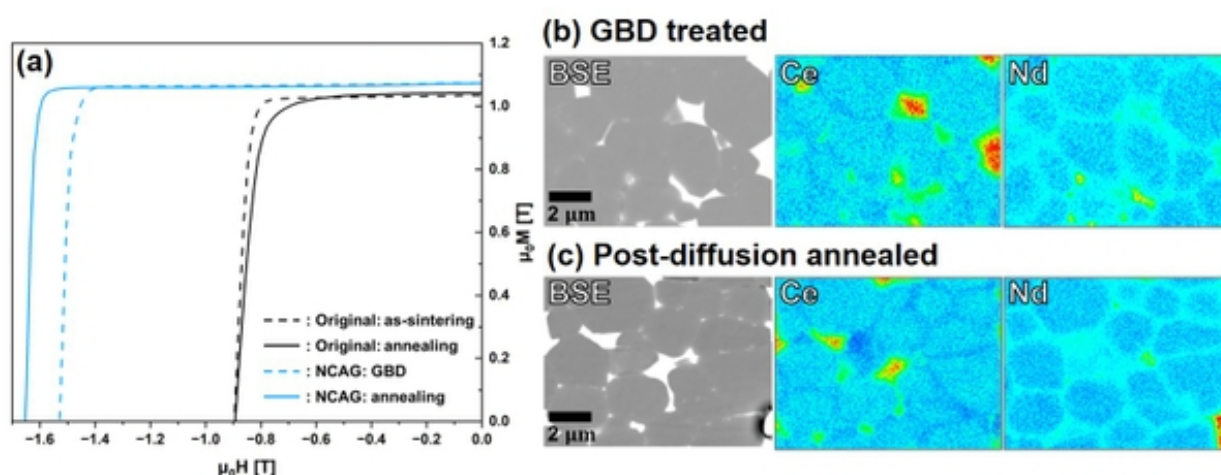


Fig.1 (a) Demagnetization curves for as-sintered / annealed base magnets, NCAG-GBD treated and post-diffusion annealed magnets. (b) and (c) BSE images and EPMA elemental maps for Ce and Nd taken from the center region (~1650  $\mu\text{m}$  depth) of the NCAG-GBD treated and post-diffusion annealed magnets, respectively.



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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-7] Microstructural Optimization and Coercivity Enhancement in Nd-Ce-Fe-B Magnets through Grain boundary diffusion of Pr-La Mixed Alloy

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Keywords : Rare earth magnet、Nd-Ce-Fe-B、Grain boundary diffusion process、Light rare earth、Chemically induced liquid film migration

We report on the magnetic and microstructural characteristics observed in Nd-Ce-Fe-B sintered magnets following grain boundary diffusion process (GBDP) with Pr-La-Cu-Ga alloys. The microstructural feature of the shell and grain boundary phase (GBP) in GBD treated with Pr-La-Cu-Ga and Pr-Cu-Ga were characterized via the electron probe microanalysis and high-angle annular dark-field scanning transmission electron microscopy analysis. The GBDP with Pr-containing alloys forms a high-anisotropy Pr-rich shell, enhancing coercivity; however, this shell formation causes chemically induced liquid film migration (CILFM), resulting in undesirable grain growth in the magnets, which restricts the improvement of coercivity. The introduction of La in the boundary diffusion source increased coercivity from 13.7 kOe (11.3kOe before heat treatment) to 20 kOe without the use of heavy rare earth (HRE) elements. The formation of Pr-enriched thin RE-rich shell is more advantageous for enhancing coercivity. The RE concentration in the high-anisotropy RE-rich shell can be more effectively controlled by suppressing CILFM. It was confirmed that CILFM effectively suppressed using La due to its low solubility in the 2:14:1 phase. Our study shows that utilization of La can be a cost-effective solution to prevent undesirable grain growth by CILFM and increase the coercivity.

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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-8] Hysteretic properties of (Nd,Ce,Tb)-(Fe,Co)-(Al,Cu,Ti)-B permanent magnets prepared by in-situ grain boundary diffusion

\*Pavel Alexandrovich Prokofev<sup>1</sup>, Natalia Borisovna Kolchugina<sup>1</sup>, Nickolay Andreevich Dormidontov<sup>1</sup>, Nickolay Vladimirovich Kudrevatykh<sup>2</sup>, Anna Sregeevna Bakulina<sup>1</sup>, Mark Vladimirovich Zheleznyi<sup>1</sup> (1. Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences (Russia), 2. Ural Federal University named after the First President of the Russian Federation B. N. Yeltsin (Russia))

Keywords : Permanent magnet, rare earth alloy, hysteretic properties

Preparing the Ce-containing Nd-Fe-B hard magnetic materials exhibiting both a high coercive field and a high magnetic energy product is a great challenge. As is known, there is an overstocking of the Ce element, since the  $\text{Ce}_2\text{Fe}_{14}\text{B}$  compound is inferior in magnetization, anisotropy field and Curie temperature to the  $\text{R}_2\text{Fe}_{14}\text{B}$  compounds with  $\text{R} = \text{Nd, Pr, Tb, Dy}$ , which are currently characterized by the greatest use. In order to balance the utilization of rare-earth resources, researchers have partially replaced Pr and Nd with Ce to produce sintered magnets. Such a substitution requires the addition of a heavy rare-earth metal to improve the coercivity of sintered magnet. A hydrogenated Tb-Co-Cu- $\text{H}_x$  intermetallic compound was used as the diffusion source to fulfill the in-situ grain-boundary diffusion and grain-boundary structuring of sintered magnet containing 8 wt% Ce. In this case, the  $(\text{Nd,Ce})_2\text{Fe}_{14}\text{B}$  main phase grains with the Tb-diffusion shell (determined by scanning electron microscopy in using a wave detector) are formed in the sintered RE-Fe-B magnets (see Fig. 1).

The introduction of terbium and cobalt into the main-phase grain via the grain-boundary diffusion and copper accumulation at a triple junction were found. The precise in-situ engineering of the microstructure of permanent magnets is the method which allowed us to fabricate Nd-Ce-Fe-B magnets, in the case of resource-saving substitution of Ce for Nd, with the high hysteretic properties via the formation of core-shell grain structure giving the local increase in the magnetic anisotropy of the Ce-containing 2-14-1 phase. The degree of texture of the Ce-containing sintered magnet was estimated based on measurements of the remanence along and across the magnet texture; it is 0.96. The measurement of the field dependences of magnetization showed that the anisotropy field of the hard-magnetic  $\text{Nd}(\text{Ce})_2\text{Fe}_{14}\text{B}$  phase at 50, 75, 125, and 150 °C is 52.7, 49.9, 37.5, and 33.5 kOe, respectively; the values are close to those of the Ce-free main magnetic phase. The hysteretic properties of the magnet measured at (1) 25, (2) 50, (3) 75, (4) 100, (5) 125, (6) 158, (7) 175, and (8) 200 °C are given in Table 1.

The temperature coefficients of remanence  $\alpha$  and coercive force in magnetization  $\beta$  for the Ce-containing sintered magnet prepared with 4 wt % Tb-Co-Cu- $\text{H}_x$  are given in Table

2.

This study provides an experiment basis for manufacturing high-coercivity Nd-Ce-Fe-B magnets by in-situ diffusing Tb-Co-Cu material in industry. The experimental results obtained indicate the possibility of the use of Ce-containing permanent magnets low-alloyed with Tb for creating various electronic devices. The study was performed in terms of state assignments nos. № 075-00320-2400 and FEUZ 2023-0020 and in part supported by State Corporation ROSATOM, state contracts nos. N.4shch.241.09.21.1101 and N.4shch.241.09.22.1141.

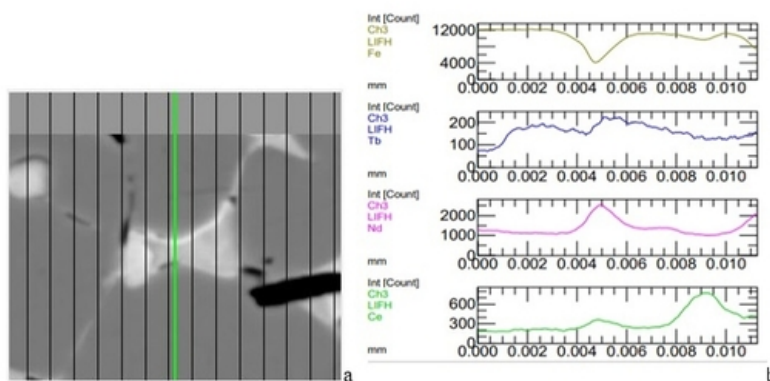


Fig. 1. Scanning electron microscopy data of sintered permanent magnet (Nd,Ce,Tb)-(Fe,Co)-(Al,Cu,Ti)-B prepared with 4 wt% of Tb-Co-Cu-Hx: (a) secondary-electron image and (b) element distribution along a scanning line.

Table 1

	1	2	3	4	5	6	7	8	
$B_r$	= 1,11	1,08	1,05	1,01	0,970	0,922	0,866	0,791	T
$H_{cB}$	= 10,5	10,2	9,87	9,41	8,36	6,30	4,24	2,26	kOe
$H_{cJ}$	= 20,0	17,6	14,3	11,4	8,76	6,41	4,29	2,35	kOe
$(BH)_{max}$	= 29	27	26	24	22	20	17	10	MGOe
$B_a$	= 0,549	0,533	0,521	0,501	0,489	0,466	0,462	0,508	T
$H_a$	= 5,23	5,12	4,94	4,73	4,46	4,19	3,60	2,04	kOe
$J_k$	= 0,997	0,973	0,945	0,909	0,873	0,829	0,779	0,712	T
$H_k$	= 17,5	15,7	13,3	10,7	8,20	5,98	3,95	2,04	kOe
$H_{lmax}$	= 20,0	20,0	19,9	19,9	19,8	19,7	19,5	19,4	kOe
T	= 25	50	75	99	125	150	174	199	°C

Table 2

Temperature range	25-50°	50-75°	75-100°	100-125°	125-150°	150-175°	175-200°
$\alpha$ , %/°C	-0.094	-0.115	-0.152	-0.159	-0.199	-0.239	-0.345
$\beta$ , %/°C	-0.488	-0.733	-0.828	-0.920	-1.071	-1.324	-1.811

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## [P1] RE-Fe-B Magnets

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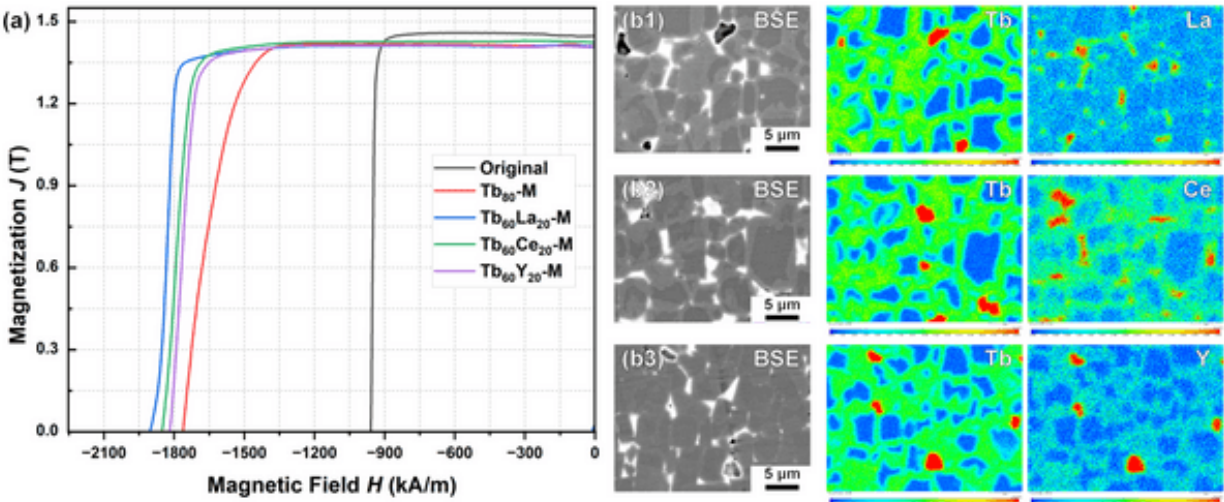
### [P1-9] Utilization of high-abundance rare earth elements in Tb-Cu-Al alloy for high efficient grain boundary diffusion of Nd-Fe-B magnets

\*Mingpeng Kou<sup>1</sup>, Hongya Yu<sup>1</sup>, Xichun Zhong<sup>1</sup>, Zhongwu Liu<sup>1</sup> (1. School of Materials Science and Engineering, South China University of Technology (China))

Keywords : Nd-Fe-B、Grain boundary diffusion、High-abundance rare earth elements、Diffusion behavior

Grain boundary diffusion (GBD) is an effective approach to enhance coercivity for Nd-Fe-B magnets with reduced consumption of heavy rare earths (HRE). Nevertheless, it is also confronted with the challenge of shallow diffusion depth, causing a low utilization efficiency of HRE. To address this issue, a significant portion of the current research efforts have been centered on promoting the diffusion effects of HREs along the GBs by replacing the HRE by light REs of Pr and Nd. In our previous work, we proposed the introduction of La and Ce into binary alloys of Tb, which achieved a remarkable improvement in the utilization efficiency of HRE due to synergistic effect of those three REs (Tb, Ce, La), thereby inspiring the exploration of high-abundance RE as components of diffusion source. However, the diffusion behavior of high-abundant REs and the characteristics of elemental distribution are still unclear.

In this study, we systematically investigated the diffusion behavior of the La, Ce, Y in Tb-Cu-Al alloy, as well as the synergistic mechanism between La, Ce, Y and Tb, ultimately achieving the modulation of diffusion behavior of Tb by high-abundant REs. After the GBD process at 900 °C for 10 h, even though 25 at% of Tb is substituted by La, Ce, or Y, the coercivity enhancement ( $\Delta H_{cj}$ ) of the Tb<sub>60</sub>La<sub>20</sub>Cu<sub>10</sub>Al<sub>10</sub>, Tb<sub>60</sub>Ce<sub>20</sub>Cu<sub>10</sub>Al<sub>10</sub>, Tb<sub>60</sub>Y<sub>20</sub>Cu<sub>10</sub>Al<sub>10</sub> diffused magnet were 1.19 T, 1.12 T, 1.08 T, respectively, even higher than that of Tb<sub>80</sub>Cu<sub>10</sub>Al<sub>10</sub> with  $\Delta H_{cj}$  of 1.01 T. The BSE images and EDS mapping in Fig. 1 indicate that La tends to aggregate at grain boundaries while Ce and Y prefer to enter into the grains. Further analyses demonstrated that the notable performance enhancement of Tb<sub>60</sub>(La,Ce,Y)<sub>20</sub>Cu<sub>10</sub>Al<sub>10</sub> diffused magnet is mainly attributed to the synergistic effect of La, Ce, Y on the diffusion of Tb. The competitive diffusion of Ce or Y and Tb effectively inhibited the lattice diffusion of Tb in the grains. La tends to combine with oxygen to form La<sub>2</sub>O<sub>3</sub>, which reduces the consumption of Tb in the grain boundary oxide, improving the effective function depth for magnetic hardening shell structure. This work provides insight for the design of low-cost diffusion alloy containing high-abundance rare-earths.



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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-10] A macroscopic perspective on the sintered Nd-Fe-B magnets prepared by Tb grain boundary diffusion

\*Jinghui Di<sup>1</sup>, Hui Meng<sup>2</sup>, Chaoyue Zhang<sup>3</sup>, Min Zou<sup>4</sup> (1. Hangzhou Magmax Technology Co., Ltd. (China), 2. Hangzhou Foresee Group Holdings Co., Ltd. (China), 3. Hangzhou Kede Magnetic Components Co., Ltd. (China), 4. Lab Magnetics Inc. (United States of America))

Keywords : Nd-Fe-B, grain boundary diffusion, macroscopic demagnetization, thick GBD magnets

Heavy rare earths grain boundary diffusion in sintered Nd-Fe-B has attracted tremendous amount of attention due to the achieved broader demagnetization curves and competitive cost efficiency of Dy/Tb [1, 2]. The enhanced magnetic performance of the GBD magnets can be attributed to their unique composition and distribution of materials. Within the microstructure, a GBD magnet is characterized by the presence of (Tb/Dy,Nd)<sub>2</sub>Fe<sub>14</sub>B shells enveloping the Nd<sub>2</sub>Fe<sub>14</sub>B cores. This arrangement serves to enhance the local anisotropy within the material. While at the macroscale, it is augmented by the gradation of Tb/Dy concentration from the surface inward, resulting in a differentiated coercivity within the material. Despite these advancements, lingering challenges remain in pinpointing the exact locations where magnetic nucleation begins during demagnetization, a pivotal aspect in fine-tuning magnetic performance. Prior studies using magnetic force microscopy and magneto-optical Kerr effect microscopy have investigated potential nucleation sites, tending toward the edges of the core or the (Tb/Dy)<sub>2</sub>Fe<sub>14</sub>B shells that exhibit a higher anisotropy field [3]. In this context, the interaction between the high coercivity layers, which ostensibly act as shields, and the lower coercivity layers, where magnetization reversal is likely to initiate, has been a topic of extensive debate [4]. Nevertheless, investigations into macroscopic demagnetization processes have been conspicuously absent and a comprehensive examination of the underlying coercivity mechanisms in GBD magnets is still needed. In this study, we report the macroscopic demagnetization [5] of the Tb GBD magnets and a method to make thick GBD blocks. The broader application area can be achieved together with a verification method for the high-performance and GBD magnets.

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## [P1] RE-Fe-B Magnets

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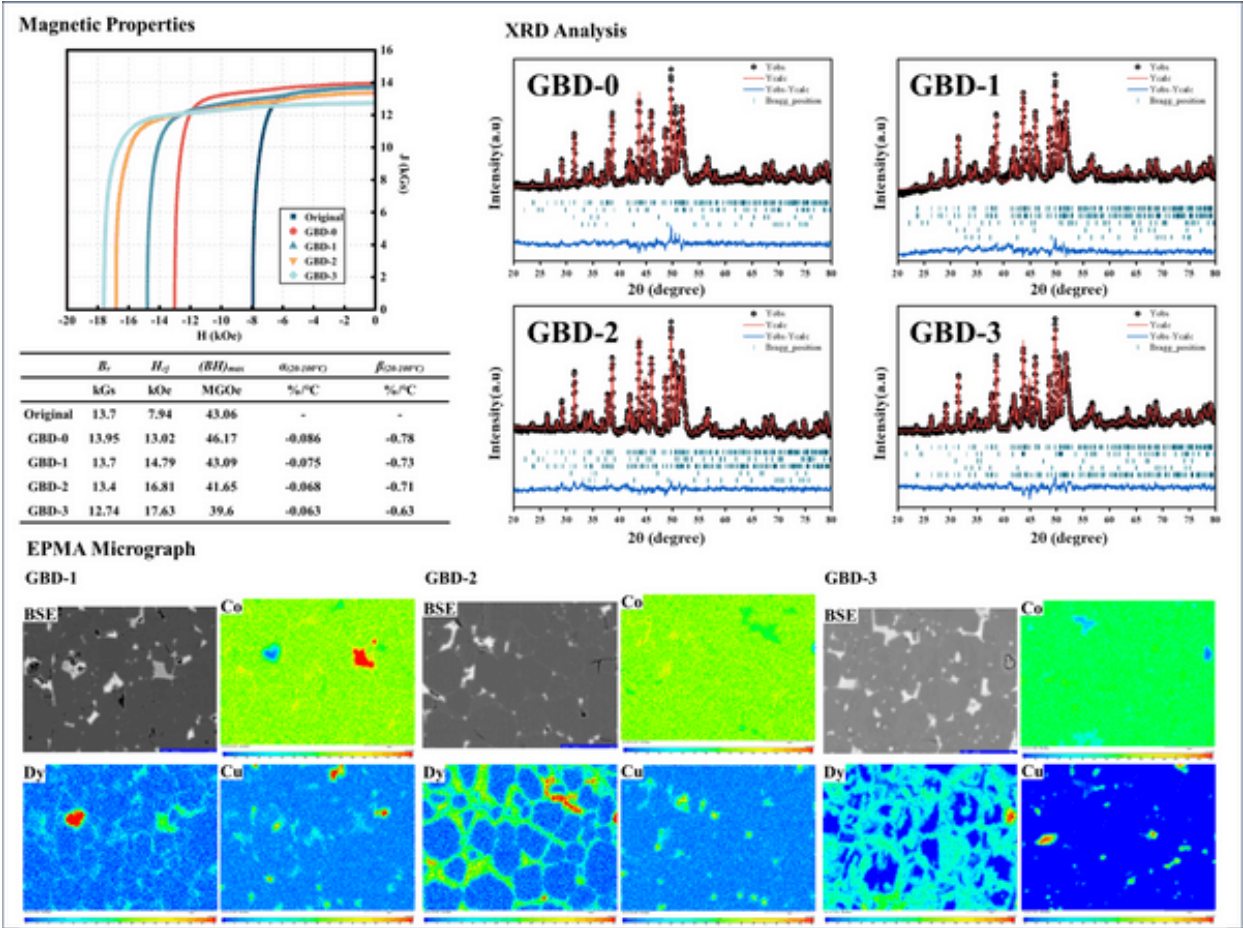
### [P1-11] Regulation of Grain Boundary Structure in NdFeCoB Magnets through Grain Boundary Diffusion of DyAlCu Alloy

\*Shengzhi Dong<sup>1</sup>, Haibo Feng<sup>1</sup>, Jing Liu<sup>1</sup>, Jiateng Zhang<sup>1</sup>, Wei Li<sup>1</sup> (1. Central Iron & Steel Research Institute (China))

Keywords : Grain Boundary Diffusion、NdFeCoB Magnet、Nd<sub>2</sub>Co<sub>17</sub>、Coercivity、Low-melting-point alloy

The optimization of grain boundary structure in sintered NdFeCoB magnets was studied using the grain boundary diffusion process (GBDP) with Dy<sub>70</sub>Al<sub>20</sub>Cu<sub>10</sub> alloy flakes as the diffusion source. These flakes were attached to magnets with a nominal composition of Pr<sub>7.8</sub>Nd<sub>23.4</sub>Co<sub>12</sub>Fe<sub>bal</sub>Al<sub>0.3</sub>Cu<sub>0.2</sub>Zr<sub>0.1</sub>B<sub>1</sub>. Magnets with various coating thicknesses were labeled as follows: GBD-0 (0 mm), GBD-1 (0.03 mm), GBD-2 (0.09 mm), and GBD-3 (0.15 mm). Among them, GBD-3 exhibited optimal performance, increasing the coercivity from 7.94 to 17.63 kOe, which represented a significant enhancement of 122%. And the diffused magnet exhibited a more optimized temperature coefficient. Prior to GBDP, Co aggregated at grain boundaries with Nd<sub>2</sub>Co<sub>17</sub> phase. After GBDP, microstructural analysis revealed the formation of Dy-rich shells with high anisotropy fields around the 2-14-1 phase within the magnet. Additionally, Al and Cu were uniformly distributed, which reduced Nd<sub>2</sub>Co<sub>17</sub> content and enhanced coercivity. This study shows GBDP with "Heavy Rare Earth-Low Melting Point" alloys improves NdFeCoB magnets' magnetic properties, offering a theoretical basis for high-performance, temperature-stable sintered NdFeCoB magnets.





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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-12] Coercivity enhancement of Nd-Fe-B sintered magnet through grain boundary restructuring using Dy<sub>80</sub>Ga<sub>20</sub> eutectic alloy

\*PAULRAJ S<sup>1</sup>, VIVEKANANDHAN P<sup>1</sup>, VIJAYARAGAVAN G<sup>1</sup>, SADHASHIVAM M<sup>2</sup>, PRADEEP K. G.<sup>2</sup>, PRABHU D<sup>1</sup>, GOPALAN R<sup>1</sup> (1. Centre for Automotive Energy Materials, International Advanced Research Centre for Powder Metallurgy and New Materials, Chennai – 600 113, Tamil Nadu, India (India), 2. Department of Metallurgical and Materials Engineering, Indian Institute of Technology Madras (IITM), Chennai – 600 036, Tamil Nadu, India (India))

Keywords : Sintered Nd-Fe-B magnet, Grain boundary diffusion, Dy<sub>80</sub>Ga<sub>20</sub> alloy, Magnetic properties

#### Abstract

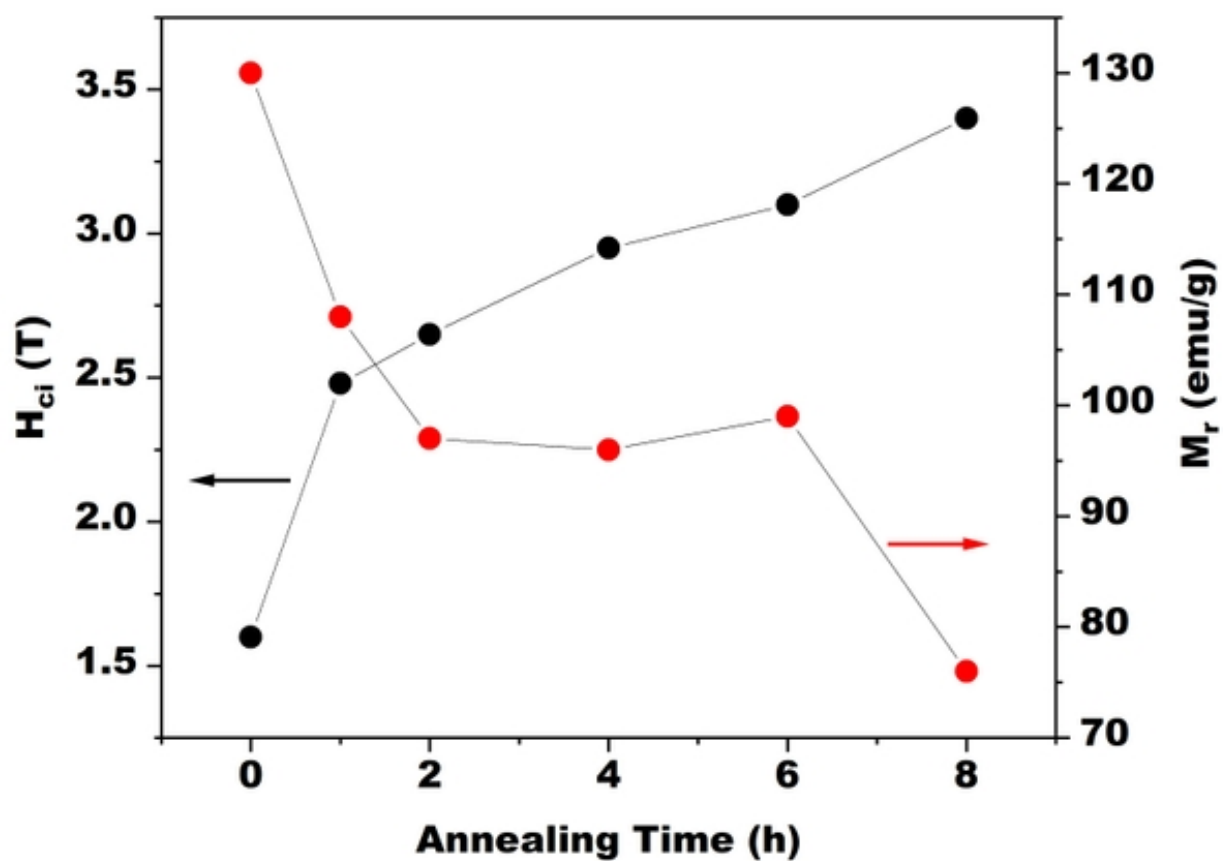
The sintered Nd-Fe-B magnets, invented by Sagawa in 1983, is unrivalled in terms of its high energy product [1]. The typical operating temperature of traction motors used in electric vehicles is about 150°C and addition of heavy rare earth (HRE) is necessary to retain the coercivity of these magnets to be used for these applications. Partial addition of Dy/Tb for Nd at the alloying stage is one of the approaches to enhance the coercivity of sintered magnet. However, high cost and the scarcity of Dy/Tb elements has always been a limitation. Grain boundary diffusion process (GBDP) is the one of the best routes to improve the coercivity with relatively less consumption of HRE [2-3]. In this work, we report relatively high coercivity obtained using a novel GBDP of Dy<sub>80</sub>Ga<sub>20</sub> eutectic source. A coercivity enhancement of 113% yielding a coercivity value of 3.4 T was achieved under optimized conditions. GBDP carried out on the same magnet using one of the well reported Dy<sub>70</sub>Cu<sub>30</sub> source only yielded ~78% enhancement in coercivity demonstrating the efficacy of the new alloy in enhancing the coercivity. Preliminary results suggest that the diffusion depth of Dy-Ga source is much higher as Ga is known to promote efficient diffusion of RE elements from the triple junctions [4]. In this presentation we shall detail the optimization parameters and report the evolution kinetics of the core shell microstructure and analyze the role of grain boundary chemistry using 3-Dimensional atom tomography and transmission electron microscopy studies.

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#### Figure Caption

Figure. 1 Variation of intrinsic coercivity and remanent magnetization in  $\text{Dy}_{80}\text{Ga}_{20}$  diffused Nd-Fe-B magnet at different annealing duration



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## [P1] RE-Fe-B Magnets

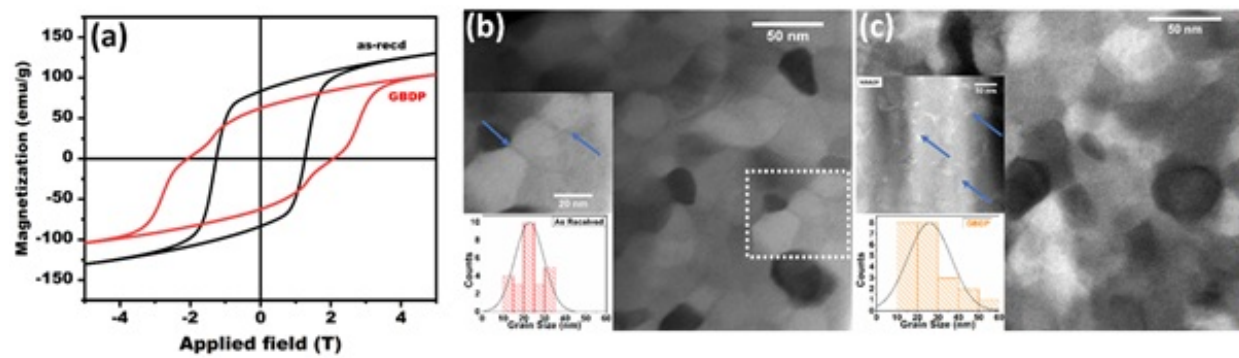
Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-13] Nb assisted grain boundary pinning in Nd-Cu diffused Nd-Fe-B magnets for enhancing the Coercivity

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Keywords : Nd-Fe-B, coercivity, microstructure, melt-spinning, magnetic properties

Grain boundary diffusion process (GBDP) using low-melting eutectic alloys is an effective method to enhance the coercivity of Nd-Fe-B permanent magnets by enriching grain boundaries (GBs) with Nd and other low-melting elements. However, the accompanying grain growth during annealing often has an adverse impact on coercivity. To overcome this, alloying with Nb has been introduced to impede grain growth while maintaining a fine microstructure during GBDP. Melt-spun ribbons of Nd-Fe-B alloys with Nb doping provide a favourable microstructure, as the rapid solidification process produces fine grains and a homogeneously distributed Nb-rich phase. Therefore, GBDP in Nd-Fe-Nb-B can be advantageous. Here we report the GBDP of Nd-Cu eutectic phase and the influence of Nb on the microstructure and magnetic properties of Nd-Fe-B melt-spun ribbons. The coercivity was enhanced from 1.2 T to 2.1 T with Nd-Cu GBDP. Coercivity enhancement mechanisms were evaluated in detail through magnetic measurements, microstructural characterization, and advanced techniques such as transmission electron microscopy (TEM), exchange coupling analysis, and atom probe tomography (APT). The results provide insights into the synergistic role of Nb-rich clusters and Nd-Cu-enriched GBs in suppressing grain growth and promoting domain wall pinning. The detailed microstructure and magnetic properties of the Nd-Cu GBDP Nd-Fe-Nb-B would be discussed[1] H. Sepehri-Amin, D. Prabhu, M. Hayashi, T. Ohkubo, K. Hioki, A. Hattori, K. Hono, , Scripta Materialia, 68, 167 (2013)[2] M.B Sivakumar, D.Prabhu, M. Sadhasivam, B. Manjusha, N. Chandrasekaran, K.G. Pradeep, G. Sundararajan and R. Gopalan, Materials Research Letters, 10(12), 780 (2022)



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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-14] Effects of trace elements on the grain boundary diffusion of sintered NdFeB magnets

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Keywords : Sintered Nd-Fe-B magnet、Trace elements、Grain boundary diffusion、Coercivity

In response to the substantial increase in demand for high-performance rare earth permanent magnets, cost-effective grain boundary diffusion technology has become an important technology for preparing Nd-Fe-B based magnets with high coercivity. Generally, there are various unintentionally added trace elements in NdFeB magnets such as O, H, N, and C. To improve the performance of the magnets, a small amount of Ga, Zr, or Nb may also be doped in the magnets. Until now, the effects of these trace elements on the grain boundary diffusion of NdFeB magnets have not been well understood. In this work, grain boundary diffusion was carried out on the sintered Nd-Fe-B magnets containing different contents of O, C, and Nb. Tb-Cu alloy was employed as the diffusion source. The influences of these three elements on the microstructure and magnetic properties of the diffused magnets were investigated. Fig.1 shows the coercivity enhancement ( $\Delta H_{cj}$ ) and Tb concentrations against diffusion depth from the surface of the diffused magnets with different levels of O, C, and Nb contents. Fig.1a shows that the  $\Delta H_{cj}$  decreases from 683 kA/m to 513 kA/m with the increase of O content from 320 ppm to 460 ppm in the magnets. Fig.1b shows that the increase in C content also decreases the diffusion efficiency. As C content increased from 550 ppm to 1200 ppm, the  $\Delta H_{cj}$  decreases from 664 kA/m to 297 kA/m. Similarly, the increase of niobium from 0.5 wt.% to 1 wt.% also decreases the  $\Delta H_{cj}$  of diffused magnets from 1174 kA/m to 920 kA/m, as shown in Fig.1c. The microstructure analysis was carried out to understand the effect mechanism of the trace elements. The results indicated that the increased O and C content leads to more high melting point oxides in the magnet, which impedes the Tb diffusion and reduces the diffusion efficiency of Tb. For the effect of Nb, more high melting point Nb-rich phases presented in the magnets with high Nb content also impedes the Tb diffusion. As shown in Fig1, the weight percentages of Tb content inside the diffused magnets with higher O (Fig.1d), C (Fig.1e) and Nb (Fig.1f) contents are lower than those in the magnets with lower O, C, Nb content, respectively. The results indicated that the high melt-point intermetallic compound presented in the grain boundary is not beneficial to the grain boundary diffusion. More detailed investigations on the microstructure of the studied magnets have also been carried out to verified above results. This work is important for the optimization of sintered Nd-Fe-B magnets for efficient grain boundary diffusion.

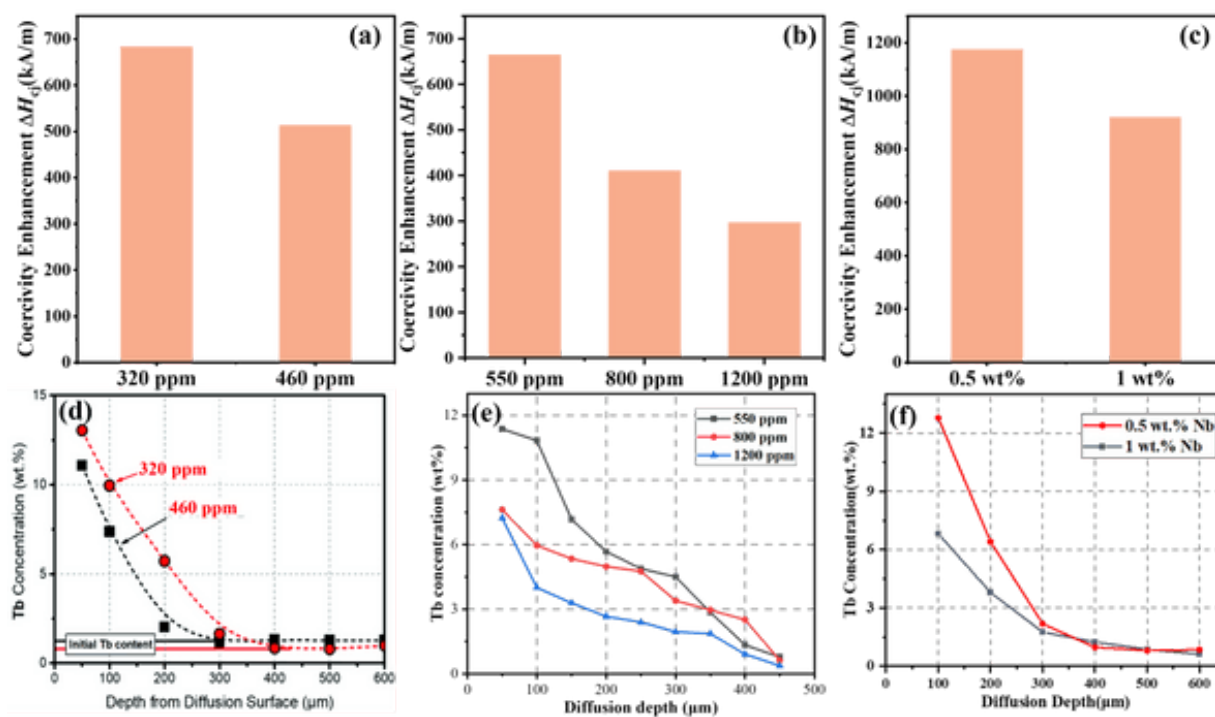


Fig.1  $\Delta H_{cj}$  of the diffused magnets with different O (a), C (b), and Nd (c); Tb concentrations against diffusion depth from the surface of the diffused magnets with different O (d), C (e), and Nd (f).

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## [P1] RE-Fe-B Magnets

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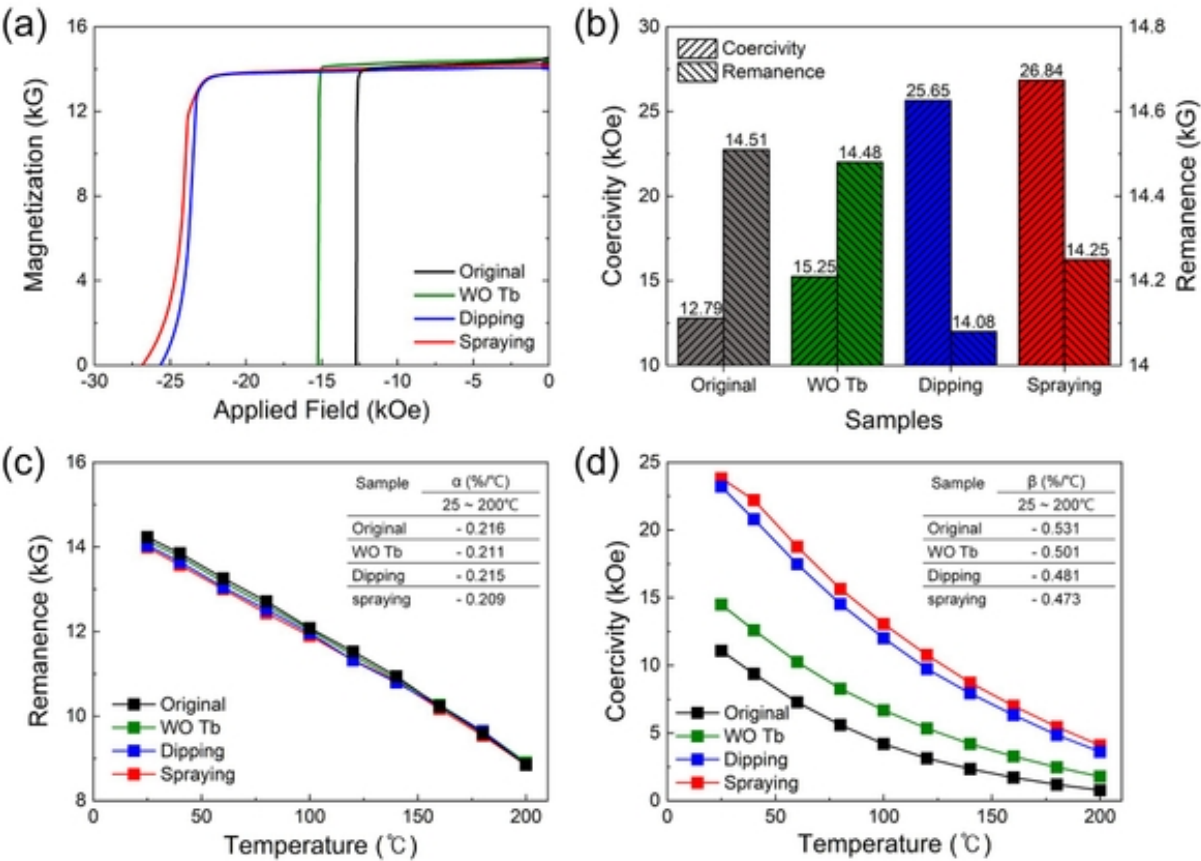
### [P1-15] Effect of Coating Methods on the Magnetic Properties of Grain Boundary Diffusion Processed Nd-Fe-B Sintered Magnets

\*Jaehyuk Kim<sup>1,2</sup>, Ye Ryeong Jang<sup>1</sup>, Dong Hyun Lee<sup>4</sup>, Seong Chan Kim<sup>2</sup>, Ju Young Beak<sup>2</sup>, Donghwan Kim<sup>5</sup>, Sang Hyub Lee<sup>5</sup>, Sumin Kim<sup>6</sup>, Jong Wook Roh<sup>4</sup>, Dalhyun Do<sup>3</sup>, Dong Hwan Kim<sup>2</sup>, Jeongmin Kim<sup>2</sup>, Wooyoung Lee<sup>1</sup> (1. Yonsei Univ. (Korea), 2. DGIST (Korea), 3. Keimyung Univ. (Korea), 4. Kyungpook National Univ. (Korea), 5. Star Group (Korea), 6. KIMS (Korea))

Keywords : Nd-Fe-B, Grain Boundary Diffusion Process, Coating method, Magnetic properties

The grain boundary diffusion (GBD) process has emerged as an effective method for enhancing the coercivity of sintered Nd-Fe-B magnets while minimizing the consumption of heavy rare-earth elements (HREs) such as Tb and Dy. Conventional bulk doping leads to a homogeneous distribution of HREs throughout the magnet, resulting in unnecessary material waste and a reduction in remanence (Br). In contrast, the GBD process selectively diffuses HREs along grain boundaries, thereby improving coercivity more efficiently. However, the effectiveness of this process is significantly influenced by the coating method and diffusion pathway. In this study, the effect of different TbH<sub>x</sub> coating methods on the magnetic properties of GBD processed Nd-Fe-B magnets was analyzed. Commercial sintered Nd-Fe-B magnets were coated using dip coating and spray coating, followed by the diffusion process. The coercivity (Hc) increased from 12.8 kOe to 24.4 kOe with dip coating and further to 26.8 kOe with spray coating. Meanwhile, a slight decrease in remanence (Br) was observed, from 14.5 kG to 14.0 kG for dip-coated magnets and 14.3 kG for spray-coated magnets. The results indicate that spray coating enables a more uniform diffusion process, leading to superior coercivity enhancement compared to dip coating. In dip-coated samples, diffusion from side wall regions was inefficient, and the diffusion direction was perpendicular to the magnet's alignment. This caused HREs to penetrate the grain interior rather than diffusing along the grain boundaries. In contrast, spray coating facilitated a more uniform and controlled diffusion pathway along the grain boundaries, thereby maximizing coercivity improvement. This study highlights the critical role of optimizing the HRE coating method in the GBD process to achieve high coercivity while minimizing rare earth consumption. The findings demonstrate that spray coating provides higher diffusion efficiency than dip coating, offering a cost effective and sustainable approach for producing high performance Nd-Fe-B magnets.





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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-16] Magnetization reversal of core-shell structured grain of GBDP Nd-Fe-B sintered magnet

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Keywords : GBDP Nd-Fe-B sintered magnet、 Magnetization reversal、 Magnetic domain、 In-situ observation、 Lorentz TEM

The GBDP sintered Nd-Fe-B magnet exhibits improved magnetic properties without excessive use of expensive heavy rare earths such as Dy and Tb. The Dy- or Tb-rich shell, which possesses a high anisotropy field, along with an improved grain boundary, has been attributed to the increased coercivity and minimal degradation of remanent magnetization [1]. However, the mechanism behind coercivity enhancement remains not fully understood. Various experimental techniques, including Kerr microscopy, MFM, and Lorentz TEM, have been employed to observe the magnetization reversal process [2,3]. Most observations conducted under varying magnetic fields and temperatures focus on domain evolution on the surface perpendicular to the easy axis. However, the demagnetization field significantly alters the domain structure, making the observed results not directly correlated with the magnet's magnetic properties. In this study, utilizing a self-developed strong in-plane magnetic field holder in Lorentz TEM [4], we observed core-shell structured grains along the plane of the easy axis, obtaining direct experimental insights into the magnetization reversal process of the magnet. GBDP Nd-Fe-B was prepared by diffusing DyH<sub>3</sub> into an N55 commercial Nd-Fe-B magnet. This process increased the coercivity from 14.2 kOe to 21.8 kOe. A TEM sample was cut using FIB at a depth of 40 μm from the diffusion surface. Fig. 1(a) presents the SEM image of the TEM sample, where grain ⑥, located at the center, exhibits a full core-shell structure. It is surrounded by some RE-rich phases and six uncomplete grains, the remaining portions after FIB etching. Fig. 1(b) shows the thermally demagnetized state, revealing a multi-domain structure with domain walls spanning across all grains except for grain ③. The domain walls deflect within the grains along their easy axes. As the applied field increased slightly, the domain walls shifted and merged rapidly, bringing the sample to a saturated state—an essential characteristic of nucleation-type magnetic domains. When an opposing field was applied, as indicated by the long red arrows in Fig. 1, magnetization reversal occurred grain by grain. Fig. 1(c)-(f) are four frames from a continuous video, before which grains ①–③ had already reversed their magnetization. The magnetization reversal is evident as an instantaneous flip in diffraction contrast, clearly observable in the video. Another notable feature is the contrast change at the grain boundaries, as exemplified in Fig. 1(c) and 1(d) when magnetization reversal occurred in

grain ④. The grain boundary, marked by white arrows in Fig. 1(c), became “black” in Fig. 1(d), which is attributed to changes in Lorentz forces between magnetization of grains ④ and ⑥, as well as ④ and ⑦. The directions of the magnetization are indicated by yellow and red arrows. As the field increased to 1.21 T and 1.29 T, magnetization reversal was observed in grains ⑤ and ⑥, as shown in Fig. 1(e) and 1(f), respectively. It is worth noting that the contrast of grain boundaries indicated by white arrows in Fig. 1(f), becomes almost identical in Fig. 1(c), which is the result of magnetization reversals in all three grains ④-⑥.

In this experiment, we observed that magnetization reversals occurred first in the surrounding grains, followed by the central grain. This is in anticipation as the defected regions of the surrounding grains have lower anisotropy fields. The exception of grain ⑦ is likely due to its thick Dy-rich shell layer. More importantly, we found that diffraction contrast jumped only seven times, and no domain walls like those in Fig. 1(b) were observed during the process. This indicates that the magnetization reversal of each grain happens simultaneously, with no distinct sequence between the core and shell regions. Although micromagnetic simulations suggested that demagnetization could nucleate in either the core or shell first, this phenomenon was not observed within the frame rate of 30 fps (33.3 ms per image) in the video, which suggests strong exchange coupling between the core and shell within individual grains. The one-by-one reversal of grains demonstrates good magnetic decoupling by the grain boundaries. Magnetization reversal at high temperatures will also be reported at the conference.

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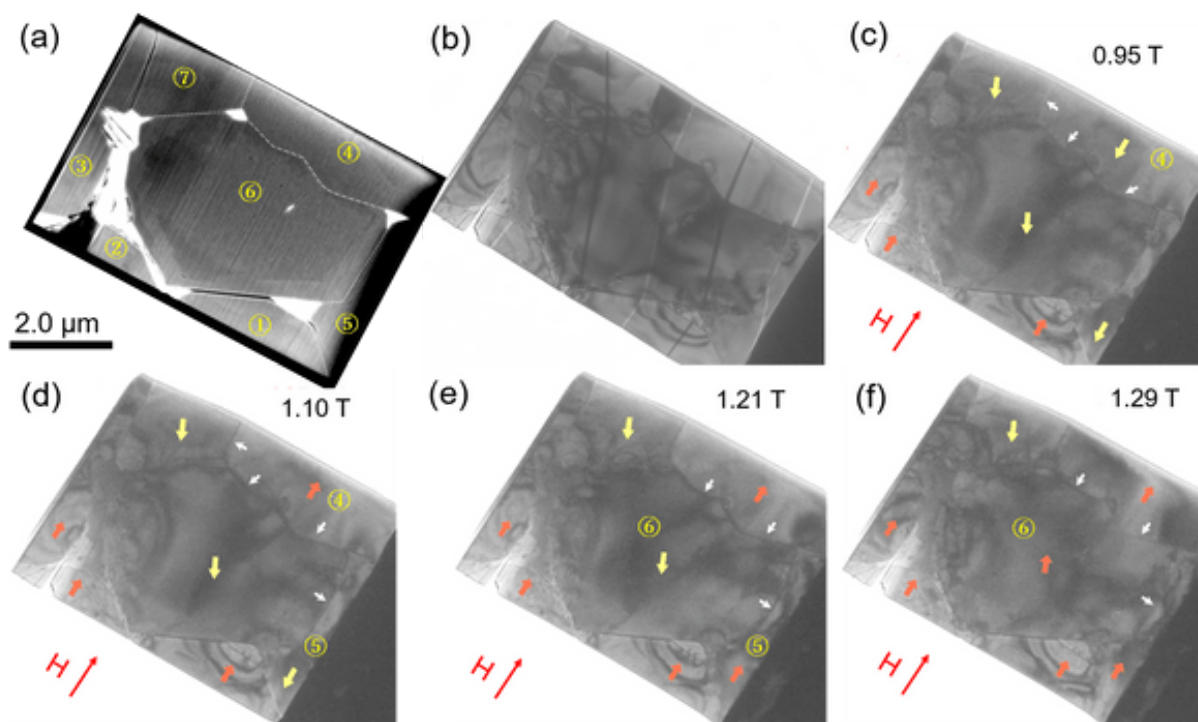


Fig.1 (a) SEM image of the TEM sample. (b) Thermally demagnetized state. (c) Magnetization reversals have occurred for grains ① - ③. (d)-(f) Magnetization reversal happens for grains ④, ⑤ and ⑥, respectively.

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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-17] Novel Aspects in Nd-Fe-B Grain Boundary Engineering: Integrating (Electro)chemistry and Materials Science

\*Kristina Zuzek<sup>1</sup>, Tomaz Tomse<sup>1</sup>, Mihaela Rebernik<sup>1</sup>, Fabian Burkhardt<sup>1</sup>, Amit Mishra<sup>1</sup>, Crt Saksida<sup>1</sup>, Laurence Schieren<sup>2</sup>, Carlo Burkhardt<sup>2</sup>, Johann Fischbacher<sup>3</sup>, Thomas Schrefl<sup>3</sup>, Sourur Semsari Parapari<sup>1</sup>, Saso Sturm (1. Jozef Stefan Institute (Slovenia), 2. Pforzheim University (Germany), 3. Danube University Krems (Austria))

Keywords : Nd-Fe-B、 recycling、 processing、 grain boundary engineering

Controlling the interfaces between the primary Nd<sub>2</sub>Fe<sub>14</sub>B phase and its surrounding subphases is reported to be crucial for enhancing coercivity<sup>1</sup>. Our study focuses on these interfaces—especially between the Nd<sub>2</sub>Fe<sub>14</sub>B phase and the grain-boundary phase—as they play a key role in magnetization reversal. To address this, we introduce a novel approach: single-grain in-situ grain boundary engineering, where magnets are directly created from Nd<sub>2</sub>Fe<sub>14</sub>B matrix grains. The feedstock was thus produced from end-of-life Nd-Fe-B magnets via selective electrochemical and organic acid treatments that have been upgraded to effectively recover Nd<sub>2</sub>Fe<sub>14</sub>B matrix phase grains without any Nd-rich phase and the Nd-oxide phases<sup>2,3</sup>. As it is known that trace amounts of copper positively influence the coercivity, we employed an electrochemical approach as a proof-of-concept to deposit Cu on the Nd<sub>2</sub>Fe<sub>14</sub>B matrix grains. We have used Na<sub>2</sub>SO<sub>4</sub> for the supporting electrolyte instead of the commonly used H<sub>2</sub>SO<sub>4</sub> which enabled us to electrodeposit copper on easily oxidizable substrates like Nd-Fe-B. It was found that potentials, such as −0.5 V, resulted in a negative current throughout the whole deposition experiment, indicating prevailing Cu reduction over the Nd-Fe-B oxidation, representing a critical first step in advancing grain-boundary engineering in the Nd-Fe-B system, introducing new phases that enhance corrosion resistance and promote cost and resource efficiency. Further we succeed in processing of Nd-Fe-B permanent magnets using the matrix Nd<sub>2</sub>Fe<sub>14</sub>B phase grains, with recourse efficient grain boundaries based on Nd-Cu consolidated using spark plasma sintering. The impact of Nd<sub>70</sub>Cu<sub>30</sub> addition ranging from 0 to 30 wt.%, on the microstructure and magnetic properties was investigated. The bulk density and the remanence saturated at 10 wt.% of added Nd<sub>70</sub>Cu<sub>30</sub>, reaching 7.63 g/cm<sup>3</sup> and 1.03 T, respectively. Given the strong affinity of Nd to oxygen, part of the Nd from the two-phase Nd<sub>70</sub>Cu<sub>30</sub> alloy interacts with oxygen present at the Nd<sub>2</sub>Fe<sub>14</sub>B grain surfaces, forming Nd-rich oxides, which segregate in triple pockets, as indicated by SEM and TEM. We demonstrate that the removal of oxygen from the grain boundaries significantly enhances the coercivity, which increased from 50 to 825 kA/m for 2.5 and 30 wt.% addition. The positive influence of the texture improvements and the oxygen

redistribution on the final coercivity was also proven by micromagnetic calculations. Novel electro and chemical recycling routes opened up new possibilities for reengineering Nd-Fe-B magnets, breaking away from conventional approaches and potentially improving magnet performance. Acknowledgment: ARRS P2-0084, HEU REESILIENCE (101058598), HEU GREENE (101129888).

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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-19] Features of the magnetization reversal processes in sintered permanent magnets Nd-Fe-B and Sm-Co type

\*Andrey Urzhumtsev<sup>1,2</sup>, Viktoria Maltseva<sup>1</sup>, Alexey Volegov<sup>1</sup> (1. Ural Federal University (Russia), 2. POZ-Progress LLC (Russia))

Keywords : permanent magnets、coercivity、magnetisation reversal processes、pinning、nucleation

In the modern technology industry, there are a large number of areas for the application of hard magnetic materials, the main volumes of consumption of which fall on the energy sector for electric motors and generators. The issue of magnetization reversal mechanisms in sintered permanent magnets (PM) of the Nd-Fe-B and Sm-Co type remains controversial. The main reason for this is the complex heterogeneous micro- and nano-structure formed during sintering, the properties of which are difficult to describe using structural analysis and numerical modeling.

The paper presents a number of approaches to measuring and assessing magnetic properties that allow us to determine the role of each of the magnetization reversal mechanisms in the formation of the coercivity of the most common commercial magnets of the N35, N48, N48SH and Sm(Co, Fe, Zr, Cu)<sub>2</sub> grades.

Magnetic measurements of the initial magnetization and susceptibility curves were carried out on MPMS XL 7 in fields up to 7 T. Magnetic susceptibility was measured in alternating AC magnetic field with an amplitude  $h \sim 3.7$  Oe and a frequency  $f = 7$  Hz. Partial hysteresis loops were measured using a PPMS DynaCool with a VSM setup attachment in a field of up to 9 T with a step of 1 kOe.

The study analyzes initial magnetization curves  $\sigma(H)$  and  $\sigma_r(H)$  from a thermally demagnetized state, magnetic susceptibility curves  $\chi(H)$  and  $\chi_r(H)$ , angular dependences of coercivity  $H_c(\Theta)$  within the framework of the Kondorsky and Stoner-Wohlfarth models, assesses the reversible and irreversible contribution to magnetization, and evaluates the role of intergranular magnetostatic interaction. The results of the presented magnetometric methods are combined into a general concept for assessing the role of pinning, nucleation and coherent rotation in magnetization reversal processes for sintered PM.

The considered methods of analysis show that the mechanism of magnetization reversal in permanent magnets based on the Nd<sub>2</sub>Fe<sub>14</sub>B phase is more complex than predicted by purely nucleation or pinning models [1]. A similar picture is observed in magnets of Sm(Co, Fe, Zr, Cu)<sub>2</sub> type [2], but the effects of changes in magnetic susceptibility, reversible contribution to magnetization and the role of magnetostatic interaction are manifested approximately an order of magnitude weaker than in Nd-Fe-B magnets.

The study showed that the approach under consideration for assessing the mechanism of formation of a high-coercivity state is an effective tool that complements such methods as microstructural analysis and numerical modeling and allows predicting the prevailing role of the pinning or nucleation. For example, it has been shown that in the Nd-Fe-B PM N35 grade magnetization reversal almost entirely through domain wall movements, while N48SH behaves close-up to nucleation process. It was found that the magnetization reversal mechanisms in sintered rare-earth permanent magnets can indicate themselves in a very diverse manner and are largely determined by the history of the creation of a given magnet and differ from PM the same type, but different grades.

This work was financially supported by FEUZ-2024-0060.

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## [P1] RE-Fe-B Magnets

Session Chair: Dr. Imants Dirba (Technical University of Darmstadt, Germany), Dr. Tae-Hoon Kim (Korea Institute of Materials Science, Korea)

### [P1-20] Potential of Cryogenic Treatment Applications on Rare-Earth-Based Functional Magnetic Materials

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Keywords : Cryogenic treatment, Functional materials, Rare earth permanent magnets, Sustainability

Rare-earth (REs)-based permanent magnets are one of the classes of functional materials that are essential for low carbon energy technologies, such as green energy applications (i.e. wind energy) and fossil-fuel-free mobility (i.e. electromobility). Among all the hard magnetic materials used in industry, Nd-Fe-B permanent magnets stand out as the magnet of choice for many applications due to its unique combination of properties, such as elevated extrinsic magnetic properties, thermal resistance, suitable mechanical properties, and high corrosion resistance. Typically, the improvement of such properties can be achieved through chemical composition manipulation and the use of alloying elements, tailoring of microstructure via thermo-mechanical processes and the use of coatings, in that order. Recent studies have focused on the post-processing that can be cost-effective, relatively easy to apply, sustainable in terms of resource(s) consumption, and that can simultaneously improve/manipulate various properties of such functional materials. In recent years, cryogenic treatment (CT) has been widely used as a multi-property tailoring process for different ferrous and non-ferrous alloys, including microstructural, structural and surface properties modification. During CT material is subjected to sub-zero temperatures for a certain period of time. There are known 3 different types of CT, first is so-called conventional CT (CCT in the range 273-193 K), second is shallow CT (SCT is in the range of 193-113 K) and third is deep CT (DCT is below 113 K). Ferrous and non-ferrous alloys subjected to such cryogenic treatments exhibited improvements on mechano-chemical-physical behavior, corrosion and wear resistance through microstructural changes, such as increased precipitation of carbides, grain morphology alteration and lattice strain alterations. Based on this, we explore and demonstrate the potential of applying DCT as a cost-effective and sustainable multi-property manipulation process for Nd-Fe-B based permanent magnets, aimed particularly at manipulating the extrinsic magnetic properties and corrosion resistance of the entire material.

