Micostructure-coercivity relationship in Nd-rich Ga-doped Nd-Fe-B sintered magnets

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Recent trend in coercivity improvement of Nd-Fe-B sintered magnet is to refine the grains size. However, magnetic alignment of fine particles of less than 3 μm is difficult in a large-scale industrial production process. Recently, Hasegawa et al. reported that a high coercivity ($\mu_0 H_c$) of 1.8 T can be achieved even for the sintered magnets with an average grain size of 6 μm. This opened up the realistic approach in achieving high coercivity in industrially viable Nd-Fe-B sintered magnets [1,2]. The alloy contains an excess amount of Nd and a small amount of Ga-dopant and the high coercivity was attributed to the formation of $Nd_6Fe_{13}Ga$ phase, and non-ferromagnetic grain boundary phase separating $Nd_2Fe_{14}B$ grains, both of which are rarely observed in standard commercial Nd-Fe-B sintered magnets. In this work, we analyzed the structure and chemical composition of the constituent phases at grain boundaries and triple junctions in the Nd-rich Ga-doped Nd-Fe-B sintered magnet annealed at various temperatures, and clarified the role of Ga on the substantial coercivity increase.

Two samples were used in this study. One is Nd-rich Ga-doped sintered magnet with the chemical composition of Fe-24.6Nd-7.87Pr-0.85B-0.13Cu-0.92Co-0.35Al-0.53Ga (wt.%), and the other is Ga-free magnet with the chemical composition of Fe-24.6Nd-7.87Pr-0.85B-0.13Cu-0.92Co-0.35Al (wt.%). Hereafter, these samples are denoted as Ga-doped sample and Ga-free sample, respectively. The sintered samples were post-sinter annealed at various temperatures for 1 h in a vacuum atmosphere. The microstructures of the samples were analyzed by scanning electron microscope (SEM, Carl-Zeiss Cross Beam 1540EsB), transmission electron microscope (TEM, FEI Titan G2 80-200).

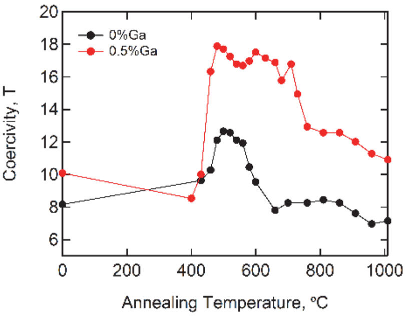

Figure 1: Variations in coercivity ($\mu_0 H_c$) as functions of annealing temperature for Ga-doped and Ga-free samples.

Figure 1 shows the variations in the coercivity ($\mu_0 H_c$) as functions of post-sinter annealing temperature. Ga-doped samples exhibit higher coercivity compared to the Ga-free samples, and the temperature range to achieve high coercivity in the Ga-doped sample is much wider compared to the Ga-free sample. Figure 2 shows backscattered electron SEM images of as-sintered samples and the samples annealed at 480, 600 and 750 ° C. In all samples, Nd-rich phases are present at grain boundary triple junctions. The variation in the areal fraction of the $Nd_6Fe_{13}Ga$ phase is consistent with the change in coercivity. Thick non-ferromagnetic grain boundary phase is formed between neighboring $Nd_2Fe_{14}B$ grains in the samples annealed at 480 and 600°C. Therefore, the main reason for the substantial coercivity increase can be attributed to the formation of non-ferromagnetic grain boundary phase. Based on these results, the effect of $Nd_6Fe_{13}Ga$ phase on coercivity will be discussed.

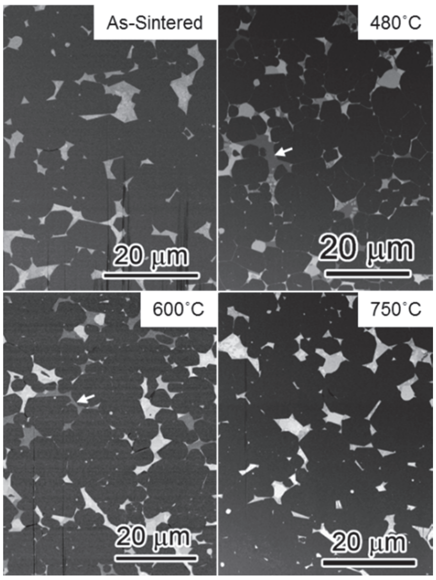

Figure 2: BSE SEM images of as-sintered sample and the samples annealed at 480, 600 750°C. $Nd_6Fe_{13}Ga$ phase is indicated by arrows.

References
1) Hasegawa et al., Abstract for Annual meeting, Japan Society of powder and powder metallurgy, 202 (2013)
2) Yamasaki et al., Abstract for Annual spring meeting, Japan Institute of Metals, S7・21 (2014)