DOI: 10.24425/amm.2021.136404

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EFFECT OF DEFORMATION TEMPERATURE ON THE MAGNETIC PROPERTIES OF PrFeB ALLOY FABRICATED BY GAS ATOMIZATION

To form the fine micro-structures, the $Pr_{17}Fe_{78}B_5$ magnet powders were produced in the optimized gas atomization conditions and it was investigated that the formation of the textures, microstructures, and the changes in the magnetic properties with increasing the deformation temperatures and rolling directions. Due to the rapid cooling system than the casting process, the homogenous microstructures were composed of the Pr-rich and $Pr_2Fe_{14}B$ without any oxides and α-Fe and enables grain refinement. The pore ratios were 2.87, 1.42, and 0.22% at the deformation temperatures of 600, 700, 800°C, respectively in the rolled samples to align the c-axis which is the magnetic easy axis. Because Pr-rich phase cannot flow into the pore with a liquid state at low temperature, the improvement of pore densification was gradually observed with increasing deformation temperature. To confirm the magnetic decoupling effects of $Pr_2Fe_{14}B$ phases by Pr-rich phases, the magnetic properties were investigated in rolled samples produced at the deformation temperature of 800°C. Although the remanent field is slightly decreased by 30%, the coercivity fields increased by about 2 times than that previous casted ingot. It is suggested that the gas atomization method can be suitable for fabricating grain refined and pure PrFeB magnets, and the plastic deformation conditions and rolling directions are a critical role to manipulate microstructure and magnetic properties.

Keywords: PrFeB alloy, Gas atomization, Plastic deformation, Rolling, REFeB magnet

1. Introduction

Because of the excellent magnetic properties, RE-based anisotropic sintered magnets (RE = Rare earth) are widely used in the various fields, such as Magnetic resonance imaging (MRI), speakers, Electric vehicles (EVs), and wind generators [1-3]. Traditionally, the magnets have been fabricated by strip casting or melt spinning; however, it has shown the disadvantages which are the complex processes and the fabricated powder easily oxidized owing to a large surface. Therefore, many studies have been conducted to simplify and to improve productivity the magnet manufacturing process, and plastic deformation using gas atomized powder is one of the candidates among the studies [4-6]. It is known that the magnetic properties of PrFeB magnets can be enhanced by the plastic deformation via ingot casting owing to the c-axis alignment and grain refinement [7-10]. However, the ingot fabricated by casting consists of large grains and a high fraction of α-Fe phase due to a low cooling rate, limiting its magnetic properties [10].

Gas atomization is well known for fabrication of metal powder and its cooling rate is faster than that in ingot casting. Therefore, a fine microstructure can be obtained. The fabrication of a PrFeB magnet using gas atomization and plastic deformation enhances its magnetic properties owing to the formation of fine microstructure and hard magnetic phase as a result of rapid cooling.

In this study, the possibility of PrFeB magnet fabrication is investigated based on the plastic deformation of gas-atomized powder at the deformation temperature. Initially, the PrFeB powder is fabricated by gas atomization. Then, it is pre-compacted and plastic-deformed by rolling. The microstructure and magnetic properties of the PrFeB billets were investigated after plastic deformation.

2. Experimental

The composition of the PrFeB alloy was fixed to $Pr_{17}Fe_{78}B_5$ for comparison with ingot casting. The PrFeB alloy was placed

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in an Al_2O_3 crucible and it was melted under Ar in an induction melting furnace. The PrFeB powder was fabricated by gas atomization (Model: DTIH-0050MF, Dongyang Induction Melting Furnace Corporation, Korea) at 1500°C under a pressure of 50 bar. The atomized powder was sieved to a powder size of 53 µm, and the powder was pre-compacted at 680°C under a pressure of 20 MPa in 22Φ graphite mold using a spark plasma sintering equipment (Model: SPS-20, Welltech Co., Korea). The pre-compacted billet was encased in SUS 304 sheath to prevent oxidization and protect the pre-compacted billet from the fracture of PrFeB billet. Then, the encased billet was sealed by welding and de-gassed under vacuum. The pre-compacted PrFeB billets were rolled at 600, 700, and 800°C at a reduction ratio of 50%, and the specimens were fabricated by grinding. The microstructures of the specimens were observed by FE-SEM (Model: JEOL, JSM-7100F, Japan). The pore ratio and Pr-rich size were measured using Image J software. The magnetic properties of the specimens were measured using a B-H tracer (Model: REMAGRAPH C-500, Magnet-Physik, Germany).

3. Results and discussion

The microstructure of the cross section of the gas-atomized $Pr_{17}Fe_{78}B_5$ powder is shown in Fig. 1. The microstructure of the gas-atomized powder consists of Pr-rich (white) and $Pr_2Fe_{14}B$ (gray) phases and α-Fe phase was not observed. The grain size of PrFeB gas atomized powder was measured using an image analyzer, and the average grain size of $Pr_2Fe_{14}B$ was 1.12 µm. The fraction of α-Fe phase and grain size of the $Pr_2Fe_{14}B$ phase of the casted ingot with same composition from previous research [10] are shown in Table 1. In the casted ingot, the fraction of α-Fe and grain size decreased with the decreasing thickness of the mold due to increasing cooling rate. The fraction of the α-Fe phase and grain size dramatically decreased compared to the casted ingot because of the high cooling rate of atomization ($\sim 10^{2\sim6}$ K/s) [11].

To confirm the effect of the plastic deformability of PrFeB alloy, $Pr_{17}Fe_{78}B_5$ powder was pre-compacted and then rolled at

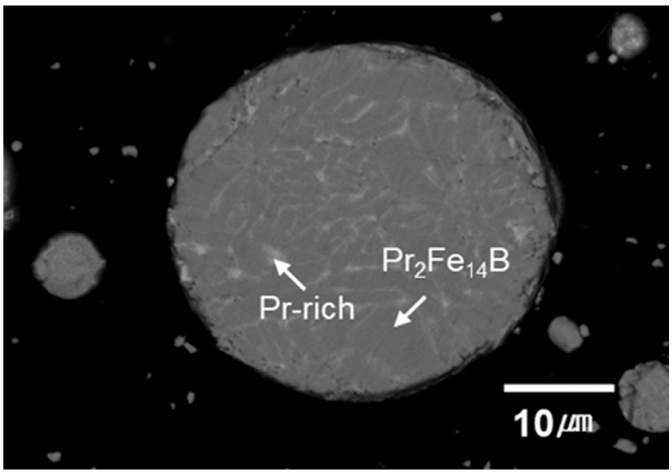

Fig. 1. Microstructure of $Pr_{17}Fe_{78}B_5$ gas-atomized powder

TABLE 1

Fraction of α-Fe phase and grain size with fabrication method

Fabrication method	Thickness of mold	Fraction of α-Fe phase (%)	Grain size (µm)
Casting [10]	7 mm	22.14	10.5
	30 mm	31.9	18
Gas atomization		—	1.12

the deformation temperature. The microstructure of the PrFeB alloy after rolling at the deformation temperature is shown in Fig. 2(a-c). When the deformation temperature was 600°C (Fig. 2(a)), many pores were observed between the powder boundaries. These pores decreased with increasing deformation temperature. From image analysis, the pore ratios at the deformation temperatures of 600, 700, and 800°C were 2.87, 1.42, and 0.22%, respectively. Moreover, the average Pr-rich size increased to 1.99, 2.90, and 4.4 μm^2 with the increasing deformation temperatures of 600, 700, and 800°C, respectively. Before plastic deformation, the pre-compacted billet had an apparent density of approximately 88%. Many pores existed even after 600°C of the plastic deformation temperature. However, most of the pores were removed at 800°C of the deformation temperature. The average grain size was 6.13, 5.15 and 2.54 µm with deformation temperature at 600, 700 and 800°C respectively. It can be explained that the melting point of the Pr-rich phase is approximately 680°C [12], Thus, the Pr-rich phase cannot flow into the pore in liquid state at the deformation temperature of 600°C. In addition, it was confirmed that grain refinement and pore densification were improved at a deformation temperature of 700°C or higher than at a deformation temperature of 600°C, but did not proceed compared to the deformation temperature of 800°C. It seems to the Pr-rich phase could not effectively enter the pores or grain boundaries because the temperature was closed to melting point at the deformation temperature of 700°C. However, at the deformation temperature of 800°C, the Pr-rich phase exists in liquid state because the temperature exceeds its melting point, and the pore and grain boundaries easily fill.

Owing to the variation of the Pr-rich size with deformation temperature, a liquid-state Pr-rich phase is used to fill the pore and grain boundaries, and then it grows with processing at a high temperature. In addition, at the deformation temperature of 800°C, it is observed that the Pr-rich phase exists between the grain boundaries. Thus, enhanced magnetic properties owing to the decoupling effect of the hard magnetic ($Pr_2Fe_{14}B$) phase are predicted at the deformation temperature of 800°C.

The magnetic properties of the $Pr_{17}Fe_{78}B_5$ alloy after the plastic deformation at deformation temperature were measured using a B-H analyzer, and the values of Br and iHc in the normal direction (ND) direction are shown in Fig. 3. These magnetic properties were low owing to the unfilled pores at the deformation temperature of 600°C, but they increased with the increase in deformation temperature. At the deformation temperature of 800°C, the coercivity was higher than that reported with the casted ingot (6.9 kOe) of the same composition [10]. In the case

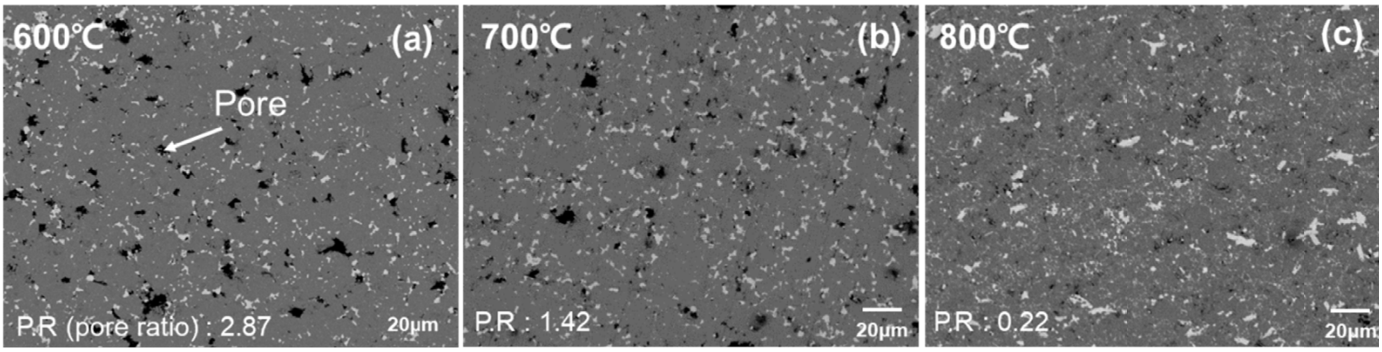

Fig. 2. Microstructure of $Pr_{17}Fe_{78}B_5$ billet after plastic deformation at the deformation temperature of (a) 600, (b) 700, and (c) 800°C

of the atomized powder, the grain size was smaller and finer grains were observed than those of the casted ingots (Table 1). Thus, the coercivity can be enhanced with the same composition. However, the remanent flux density (B_r) was lower than that reported with the casted ingot (10.6 T) [10]. May be the pores existed even after the deformation, and cracks occurred during the rolling. It is well known that B_r is related to the density, so if the density is increased by annealing after the deformation, the B_r can be enhanced [1].

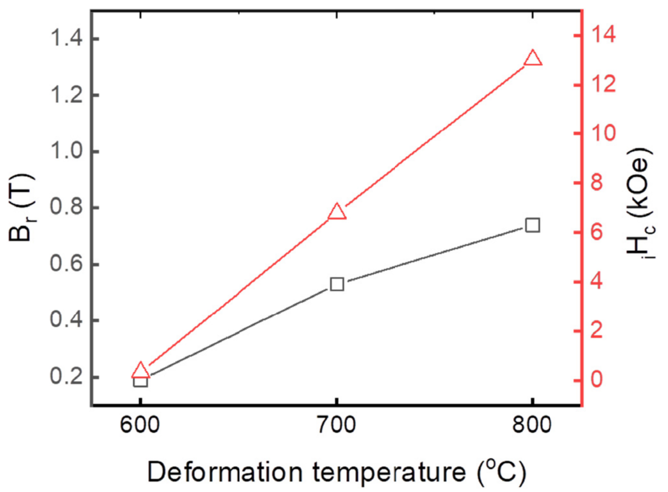

Fig. 3. Deformation temperature dependence of magnetic properties in plastic deformed $Pr_{17}Fe_{78}B_5$ magnet

The curves of demagnetization of the $Pr_{17}Fe_{78}B_5$ alloy in the rolling direction at 800°C are shown in Fig. 4. This indicates that the alloy rolled in the ND direction has a higher value than the alloy rolled in the transverse direction (TD) direction. In the case of the PrFeB casting magnet, the magnetic easy axis (c-axis) of the $RE_2Fe_{14}B$ phase is aligned in the forced direction after the plastic deformation, so ND has a higher value than TD owing to the alignment to the c-axis after rolling. Mechanism of anisotropy formation by plastic deformation of $Pr_2Fe_{14}B$ hard magnetic phase can be indicated as follows, during plastic deformation, slip is occurred along basal plane by elastic moduli between a and c axis and then, c axis is aligned to load direction as the load is continuously applied. Finally, anisotropy is formed [13].

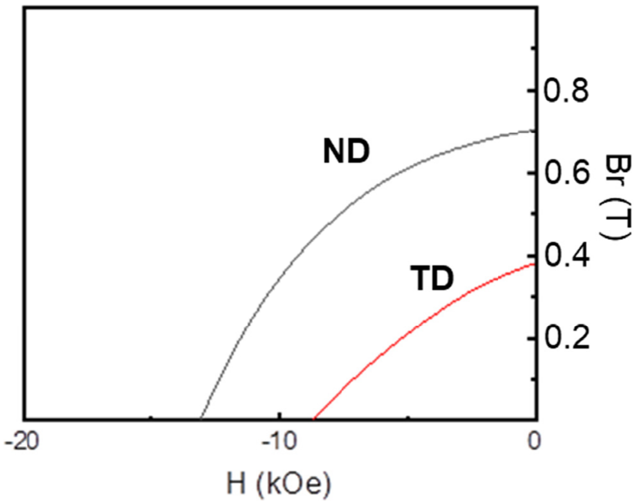

Fig. 4. Demagnetization curves of plastic deformed $Pr_{17}Fe_{78}B_5$ magnet with rolling direction

4. Conclusions

In this study, the magnetic properties of the gas-atomized $Pr_{17}Fe_{78}B_5$ powder after plastic deformation at the deformation temperature were investigated. After the plastic deformation, the pore fraction of the PrFeB billet decreased with increasing deformation temperature, and the size of the Pr-rich phase increased with increasing deformation temperature owing to the flow of the liquid state Pr-rich phase into the pore and grain boundaries. Hence, more fine grains exist at a deformation temperature of 800°C than other conditions. In addition, the curves of demagnetization are the evidence of anisotropy in the rolling direction at the deformation temperature of 800°C.

Thus, we can confirm that, the grain refinement of the hard magnetic phase and the alignment to c-axis occurs after the plastic deformation. Thus, the coercivity of the $Pr_{17}Fe_{78}B_5$ alloy is higher than that of the casted ingot with the same composition. However, the remanent flux density is lower than that of the casted ingot owing to the residual pores and cracks that occur during the plastic deformation. This suggests that the magnetic properties can be improved either by annealing or grain boundary diffusion after the plastic deformation.

Acknowledgments

This research was supported by a grant from the project “Development of environment friendly pyrometallurgy process for high purity HREE and materialization” by the Korea Evaluation Institute of Industrial Technology (KEIT), Republic of Korea.

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