Magnetic properties of DyF₃-doped Nd-Fe-B-type hot-deformed magnet

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1. INTRODUCTION

One of the critical issues of recent Nd-Fe-B-type magnet industry is an enhancement of coercivity for high temperature applications, such as the traction motors in hybrid and electrical vehicles and wind turbine generators. It has been generally accepted that the key requirements for achieving high coercivity in Nd-Fe-B-type magnet are having a fine Nd₂Fe₁₄B grain structure, ideally the size comparable to the critical single domain size of the Nd₂Fe₁₄B phase (~300 nm). As the hot-deformed (die-upset) magnet is prepared from starting melt-spun material with ultra-fine grain structure, the hot-deformed magnet is featured with fine microstructure consisting of nano-scale grains. However, even with such a fine grain structure the hot-deformed magnet still has rather low coercivity. As an effective means for enhancing the coercivity, grain boundary diffusion process using heavy rare earth salt (DyF₃, TbF₃...) has been widely investigated. For the Nd-Fe-B-type hot-deformed magnet, DyF₃ doping has been extensively exploited to improve the high temperature performance by taking an advantage of enhancing the coercivity and electrical resistivity. In this study, decomposition of DyF₃ and its effect on the magnetic performance of the hot-pressed magnet and hot-deformed magnet of melt-spun Nd-Fe-B-type material were investigated.

2. EXPERIMENTAL WORK

Starting material used in the present work was commercial melt-spun flakes (MQU-F : $Nd_{13.6}Fe_{73.6}Co_{6.6}Ga_{0.6}B_{5.6}$) supplied by the Magnequench (Molycorp). Crushed flakes (150 µm – 300 µm) were mixed with 1.6 wt% DyF₃ in cyclohexane to avoid oxidation, and then hot-pressed with 100 MPa at the temperature ranging from 610 °C to 735 °C in a vacuum to prepare fully dense compact. The prepared hot-pressed compacts were hot-deformed at 735 °C with 75 % height reduction (strain = 0.75) with strain rate of 1.25 x 10⁻²/s in a vacuum. Magnetic characterization was performed along the pressing direction using SQUID (max. field = 5 T) and VSM.

3. RESULTS AND DISCUSSION

DyF₃ was thermally decomposed above 660 $^{\circ}$ C, and this decomposition was linked closely to the coercivity enhancement. When the DyF₃ doped flakes were hot-pressed above the decomposition temperature of DyF₃ the diffusion of Dy into the flakes was promoted, and leading to profound coercivity enhancement. Coercivity of the hot-pressed magnet was further enhanced by post-hot-press annealing, and coercivity as high as 24.5 kOe was obtained after the optimum annealing. The DyF₃ doped hot-deformed magnet exhibited enhanced magnetic performance (iHc = 17.5 kOe, Br = 12.8 kG, (BH)max = 37.6 MGOe) with respect to the un-doped magnet without sacrificing significant remanence. Coercivity was improved by 30 %. In magnet in which the decomposition of DyF₃ and Dy diffusion were fully accomplished the region originally occupied by added DyF₃ was completely replaced by NdF₃.