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Application News

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Electron Probe Microanalyzer

Analysis of Neodymium Sintered Magnets Produced by Grain Boundary Diffusion Process

Introduction

Neodymium magnets display the strongest magnetic characteristics among all rare earth magnets. Discovered in 1982, they are now used in a wide range of applications, including not only the speakers of mobile phones and hard disk drives of notebook computers, but also motors in hybrid automobiles, electric vehicles, and household electrical appliances such as refrigerators and air-conditioners. Accompanying recent requirements for downsizing and weight reduction in electronic equipment for energy conservation, both demand for neodymium magnets and their range of applications have grown rapidly, and applications extend even to medical MRI systems. For this reason, more advanced technologies for achieving higher heat resistance and coercivity in neodymium magnets are demanded, and research and development are progressing.

This article introduces an example of analysis of grain boundary diffusion-treated neodymium sintered magnets using a Shimadzu EPMA™ electron probe microanalyzer (EPMA-8050G, hereinafter, EPMA).

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Analysis of Neodymium Sintered Magnets

The main component elements of neodymium sintered magnets are neodymium (Nd), iron (Fe), and boron (B), and the representative composition is $Nd_{15}Fe_{77}B_{8}$. The main phase of $Nd_{2}Fe_{14}B$ (ferromagnetic phase) is surrounded by a B-rich phase of $Nd_{1.1}Fe_{4}B_{4}$ (nonmagnetic phase) and Nd-rich phases (nonmagnetic phase) with high contents of Nd. The Nd-rich phases are a thin phase which exists on the grain boundaries of the main phase and an oxide phase (oxidized in the atmosphere) which exists at main phase grain boundary triple junctions. In order to increase the heat resistance of neodymium sintered magnets, coercivity is improved by substituting part of the Nd in the $Nd_{2}Fe_{14}B$ main phase with dysprosium (Dy), terbium (Tb), or other heavy rare earth elements (HREEs). Although Tb is more effective than Dy for improving coercivity in high temperature environments around 200 °C, Dy is mainly used, as it is somewhat more abundant in the Earth's crust and thus is less expensive. Addition of slightly less than 10 wt% of Tb, Dy, or other HREEs to neodymium sintered magnets improves coercivity by increasing magnetocrystalline anisotropy near the main phase grain boundaries, but due to the high sintering temperature of approximately 1,100 °C, HREEs diffuse into the interior of the main phase, and as a result, reduced remanence becomes a problem. Moreover, since production of Tb and Dy is unevenly distributed, being limited to only ion adsorption-type deposits in southern China, the abundance of these elements is small and supply stability is a problem. For these reasons, active research aimed at realizing high coercivity while reducing the use of Tb and Dy is now underway.

Fig. 1 shows the results of a mapping analysis of a Tb-containing neodymium sintered magnet. It can be understood that Tb is distributed at the main phase grain boundaries, and Co, Cu, and Ga, which contribute to promoting high coercivity, are distributed in the vicinity of Nd-rich phases.

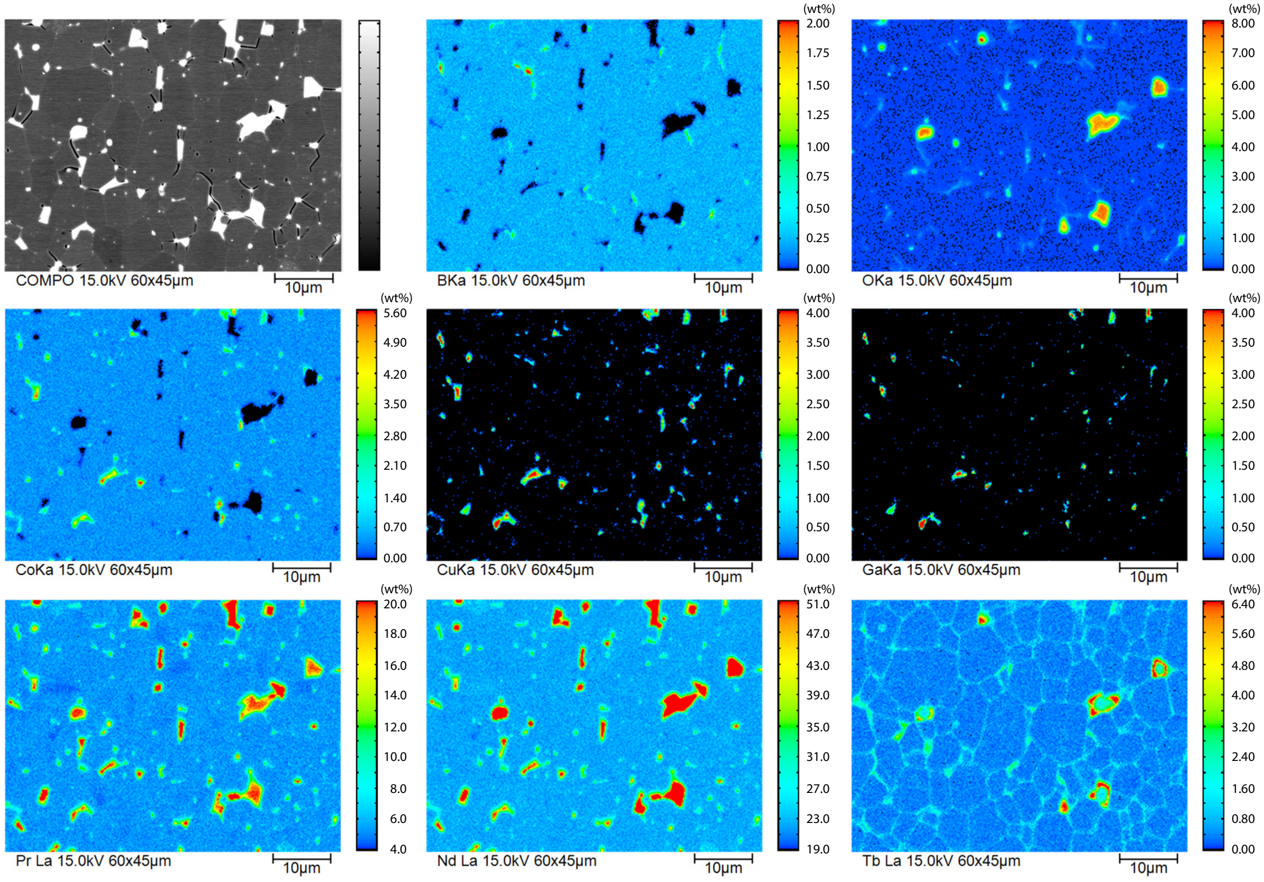

Fig. 1 Mapping Analysis of Neodymium Sintered Magnet

Grain Boundary Diffusion Treatment of Neodymium Sintered Magnets

Sintered magnets are formed by the two alloys mixed process or the grain boundary diffusion process. Grain boundary diffusion processes include a sputtering method which is used to deposit Tb and Dy on the magnet surface after sintering, followed by heat treatment for grain boundary diffusion, and a method in which compounds of Tb, Dy, and other HREEs are coated and then heat-treated at a temperature lower than the sintering temperature, among others.

In grain boundary diffusion processes in which fluorides or oxides of Tb or Dy are coated on the sintered magnet, the Nd-rich phase dissolves during heat treatment, part of the Nd diffuses to the surface of the sintered magnet, and Tb/Dy is taken into the magnet by substitution of Nd with Tb/Dy. As a result, it is possible to enhance coercivity and suppress the reduction of remanence by forming a thin, uniform, continuous Tb/Dy enriched layer near the grain boundaries of the $Nd_2Fe_{14}B$ main phase.

Fig. 2 shows the results of an analysis of a Tb-added and grain boundary diffusion-treated neodymium magnet from the surface to the center, indicating that Tb has diffused from the magnet surface through the main phase grain boundaries, reaching the region approximately 150 µm from the surface. In the line analysis display (width of 8 wt% for all elements) on the COMPO image of the mapping analysis results, Nd and Pr have been replaced with Tb, and the Tb concentration decreases slightly in the direction of the central region.

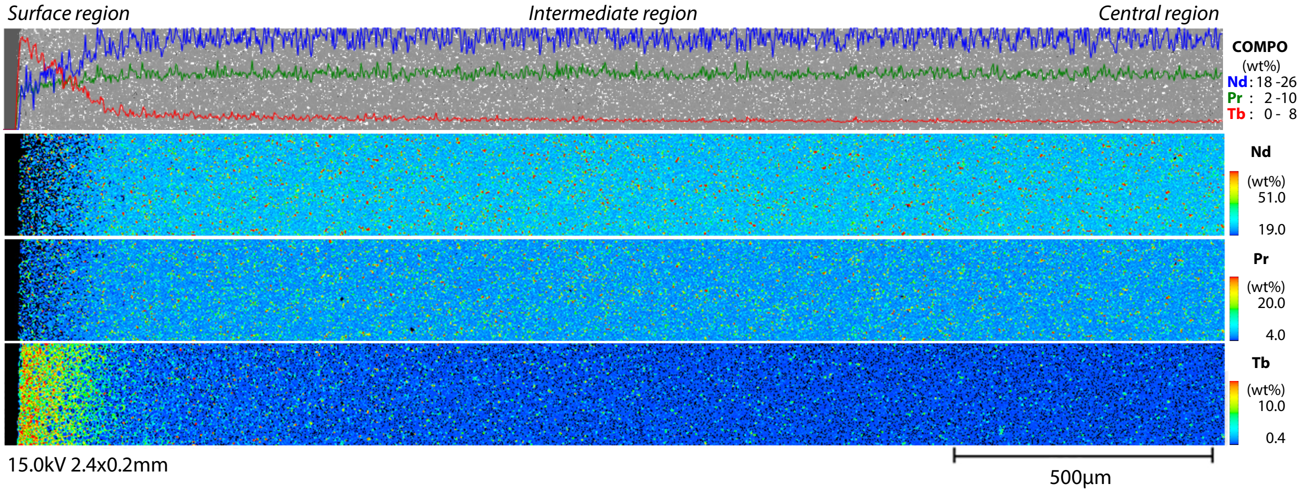

Fig. 2 Wide-Area Mapping Analysis of Tb Grain Boundary Diffusion-Treated Neodymium Magnet

Microstructure of Neodymium Sintered Magnet

After sintering, the structure of the neodymium magnet consists of a main phase of $Nd_2Fe_{14}B$ (ferromagnetic phase) covered by Nd-rich phases (nonmagnetic phase), and reversed magnetic domains form easily near the grain boundaries of the main phase. Therefore, heat resistance is achieved by suppressing formation of the reversed magnetic domains at high temperature by diffusing Tb to the grain boundaries.

In an enlarged mapping analysis of the surface region (Fig. 3(a)), intermediate region (Fig. 3(b)), and central region (Fig. 3(c)) of the Tb grain boundary diffusion-treated neodymium magnet, the crystal grains of the $Nd_2Fe_{14}B$ main phase are polygonal in shape and have a grain diameter of approximately 5 µm. In comparison with the central region, in the surface region, the Tb concentration at the grain boundaries is higher than the concentration in the main phase, and in the internal regions, i.e., the intermediate and central regions, Tb is unevenly distributed near the main phase grain boundaries, and a thin, uniform, continuous Tb enriched layer has formed.

As illustrated here, improvement of heat resistance and coercivity characteristics can be evaluated by investigating the changes in the magnet microstructure.

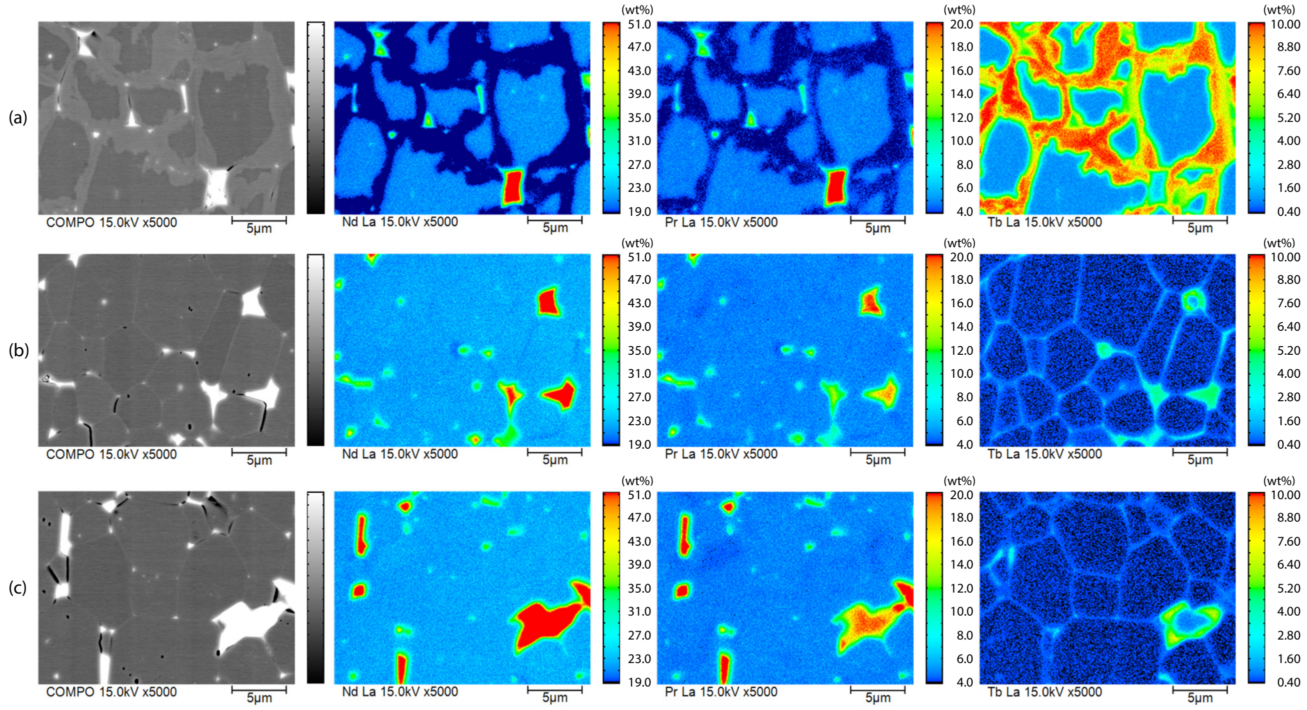

Fig. 3 Enlarged Mapping Analysis of (a) Surface Region, (b) Intermediate Region, and (c) Central Region of Tb Grain Boundary Diffusion-Treated Neodymium Magnet

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