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Microstructures and magnetic domain structures in $Sm_2(Fe,Mn)_{17}N_\delta$ powders studied by analytical electron microscopy and Lorentz microscopy

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Abstract

Microstructures and magnetic domain structures of $Sm_2(Fe,Mn)_{17}N_\delta$ ($\delta = 0$, 3.4, 4.2, 5.0) powders are investigated by analytical electron microscopy and Lorentz microscopy, respectively. It is found that amorphous phases form with the addition of nitrogen and the area of the amorphous phases increases with the increase in nitrogen content. By elemental mapping with electron energy-loss spectroscopy (EELS), it is elucidated that the amorphous phase is enriched with N and Mn, while the crystalline phase is enriched with Fe. The analysis with Lorentz microscopy reveals that the size of the magnetic domains decreases with the increase in nitrogen content. Further, it is clarified that the domain walls exist on the Mn-enriched amorphous phases. Finally, the domain wall pinning in the amorphous boundary regions is considered to result in a large coercivity (1.04 MA/m) in $Sm_2(Fe,Mn)_{17}N_{5.0}$ powder.

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Keywords: EELS elemental mapping; TEM; Lorentz microscopy; Domain wall pinning; Coercivity mechanism; Reduction and diffusion method

1. Introduction

$Sm_2(Fe,Mn)_{17}N_\delta$ coarse powders have attracted considerable attention as next generation bonded magnets because of their good oxidation resistance and thermal stability of coercivity as compared to $Sm_2Fe_{17}N_\delta$ fine powders developed in 1990 [1–4]. It is well known that the fine powder gives rise to poor oxidation resistance and thermal instability of coercivity as well as a need for high compaction pressure during application [3,4]. Therefore, several

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efforts have been made to obtain high coercivity in the coarse powders by the addition of elements such as Ta, Co, W, and Mn in $Sm_2Fe_{17}N_\delta$ alloys [4–7]. However, their magnetic properties were insufficient for their usage as commercial bonded magnets. Recently, $Sm_2(Fe,Mn)_{17}N_\delta$ powders that surpass $(BH)_{max}$ 175 kJ/m^3 and show good oxidation resistance and high coercivity have been developed by Iseki et al. [8].

In order to further improve the magnetic properties of $Sm_2(Fe,Mn)_{17}N_\delta$ powders, understanding their microstructures and magnetic domain structures is of vital importance. Thus far, microstructures in $Sm_2(Fe,Mn)_{17}N_\delta$ powders have been investigated by a few research groups, using scanning electron microscopy and transmission electron microscopy (TEM) [3,4]. However, the detailed distribution of a light element N in the constituent phases through an elemental mapping technique has not yet been analyzed. Furthermore, the observation of magnetic domain structures has not yet been carried out, although it is requisite for both fundamentals and applications of $Sm_2(Fe,Mn)_{17}N_\delta$ powders.

In this paper, analytical electron microscopy and Lorentz microscopy have been performed to investigate the microstructures and magnetic domain structures of $Sm_2(Fe,Mn)_{17}N_\delta$ powders, respectively. By electron energy-loss spectroscopy (EELS), elemental mapping is carried out to explore the detailed distribution of additive elements such as N, Mn, and Fe. With an increase in nitrogen content, the change in magnetic domain structures was observed by Lorentz microscopy, and the correlation between the microstructures and magnetic domain structures was briefly discussed.

2. Experimental procedure

$Sm_2(Fe_{0.95},Mn_{0.05})_{17}N_\delta$ ($\delta = 0$, 3.4, 4.2, 5.0) coarse powders studied in the present work were produced by RD (reduction and diffusion) process, the details of which can be found in Ref. [8]. Some of their magnetic properties, i.e., the saturation magnetization $4\pi I_s$, remanence $4\pi I_r$, coercivity ${}_iH_c$, and magnetic field H_k at $0.9 \times 4\pi I_r$, are shown in Fig. 1. It is noted that the coercivity ${}_iH_c$ increases monotonously with the increase in nitrogen content, while the saturation magnetization $4\pi I_s$ and remanence $4\pi I_r$ gradually decrease with the increase in nitrogen content. Thin foil specimens for TEM observations were prepared by a crushing method or a focused ion-beam method (FB-2000A, FB-2100) with a microsampling technique. EDS and EELS analyses were carried out using a JEM-2100F/STEM operated at 200 kV, equipped with a field emission gun, an EDS system (JED-2300 T), and a Gatan imaging filter (GIF TRIDIEM). Using an electron probe of 0.7 nm in diameter, elemental mapping images with the spectrum imaging method were obtained to analyze the distribution of additive elements. Magnetic domain structures were investigated by Lorentz microscopy using a JEM-3000F TEM, to which a field emission gun was installed. This microscope also has a special polepiece designed for observing the magnetic domains, in other words, the magnetic field at the specimen position can be reduced to less than 2 mT [9].

3. Results and discussion

Fig. 2 shows HREM images of $Sm_2(Fe_{0.95}, Mn_{0.05})_{17}$, $Sm_2(Fe_{0.95},Mn_{0.05})_{17}N_{4.2}$, and $Sm_2(Fe_{0.95}, Mn_{0.05})_{17}N_{5.0}$ powders. As shown in Fig. 2(a),

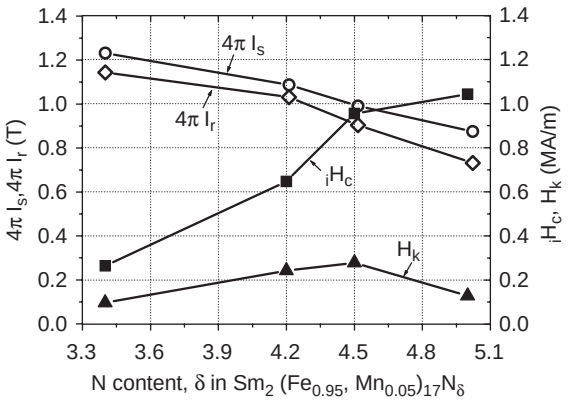

Fig. 1. Nitrogen content dependence of magnetic properties of $Sm_2(Fe_{0.95},Mn_{0.05})_{17}N_\delta$ powders. $4\pi I_s$ and $4\pi I_r$ indicate the saturation magnetization and remanent magnetization, respectively, while ${}_iH_c$ and H_k indicate the coercivity and the magnetic field at $0.9 \times 4\pi I_r$, respectively.