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Extraction of Sm from Sm–Fe–N magnets by the glass slag method

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Abstract

Sm–Fe–N alloys were successfully separated into the metallic component and the slag component by the glass slag method using boron trioxide. X-ray diffraction and magnetic measurements revealed that the metallic component consisted of α-Fe phase and that the slag component consisted of BN and Sm-containing amorphous materials. The chemical analyses confirmed that the Sm content in the metallic component was very limited and the slag component contained a large amount of samarium. The formation of the BN phase was believed to be the reaction between SmN, decomposed from $Sm_2Fe_{17}N_3$ phase at high temperatures, and boron trioxide during the glass slag process.

Keywords: Rare earth; Hard magnets; Recycling; Solidification

1. Introduction

The production of rare-earth magnets has significantly increased mainly due to their superior maximum energy products. These magnets have been widely used for various electromechanical and electronic devices [1,2]. On the other hand, large amounts of scrap from the rare-earth magnets are being stockpiled due to the lack of a cost-effective recycling process. Thus, it is crucial to develop a recycling process for the rare-earth magnets. Several attempts have been made to recycle rare-earth permanent magnet materials [3–7]. We have been studying for a dry recycling process, the so-called glass slag method, and have succeeded in the recovery of Nd from Nd–Fe–B alloys and Sm from Sm–Co and Sm–Fe alloys by this method [8–10].

In this study we investigated the possibility of extracting rare earth from the Sm–Fe–N magnets by the glass slag method, which are known as the latest type of rare earth magnets. However, the magnetic $Sm_2Fe_{17}N_3$ phase in the Sm–Fe–N magnets is not stable at high temperature and decomposes into α-Fe and SmN above 873 K [11]. The decomposition of the $Sm_2Fe_{17}N_3$ phase may affect the extraction of Sm from Sm–Fe–N magnets by the glass slag method. The degree of extraction of Sm from Sm–Fe–N magnets was evaluated by structural and magnetic analyses of the alloys processed by the glass slag method.

2. Experimental

A small amount of the $Sm_2Fe_{17}N_3$ alloy powder was cold-pressed into a green compact and then placed in a BN crucible together with pieces of boron trioxide. The experimental set-up for the glass slag method has been described in detail elsewhere [12]. The green compact was preheated slowly to just above the melting temperature of the slag material and then cooled down to room temperature in the furnace that had been evacuated and backfilled with argon. The resultant green compact was encapsulated in the boron trioxide. The green compact was heated to the reaction temperature of 1833 K (about 100 K above the melting temperature of Sm_2Fe_{17} compound) for 1 h and then cooled down to room temperature in an argon atmosphere.

After the removal of the surrounding slag materials, the specimens were cut by a low-speed diamond wheel for

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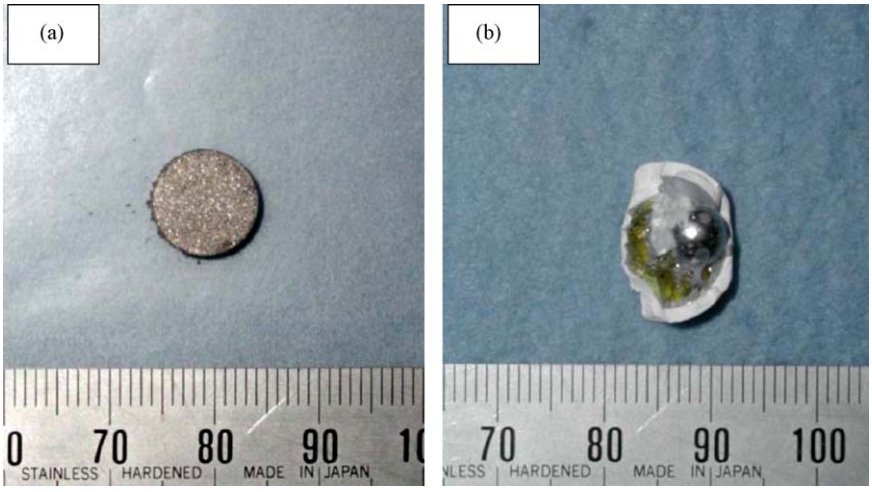

Fig. 1. External appearances of (a) green compact of the Sm–Fe–N powder and (b) those processed by the glass slag method. The spherical metallic specimen was surrounded by the yellowish slag material in the crucible.

structural and property measurements. The phases in the specimens were identified by X-ray diffraction (XRD) using Cu Kα radiation. The composition of the specimens was determined by chemical analyses using inductively coupled plasma (ICP). The magnetization of the specimens was examined at a heating rate of 0.16 Ks^{-1} in a helium atmosphere using a vibrating sample magnetometer (VSM) at an applied field of 40 kAm^{-1}. The hysteresis loops of the specimens were measured at room temperature by VSM with a maximum applied field of 1.2 MAm^{-1}. The VSM was calibrated using a pure nickel sphere.

3. Results and discussion

Fig. 1 shows the external appearances of the green compact of the Sm–Fe–N powder and those processed by the glass slag method. The specimen produced by the glass slag method had a spherical shape. Because of the high oxidation tendency of rare-earth-containing alloys, the surfaces of such alloy ingots prepared by melting and solidification are normally not smooth. However, the surfaces of the specimens obtained had a very smooth metallic surface. On the other hand, the boron trioxide used for the glass slag method became yellowish. This is most probably due to the Sm^{3+} ion, whose color is yellow. The existence of this effect implies that the rare earth, in this case Sm, is extracted from the Sm–Fe–N powder by the surrounding boron trioxide. The metallic specimen and resultant yellowish glass slag materials were examined to evaluate the degree of extraction of Sm from Sm–Fe–N powder by the glass slag method.

Fig. 2 shows XRD patterns of the metallic specimen produced by the glass slag method and of the original Sm–Fe–N powder. The XRD pattern of the Sm–Fe–N powder is indexed to the $Sm_2Fe_{17}N_3$ phase. On the other hand, no diffraction peaks of the $Sm_2Fe_{17}N_3$ phase are found in the XRD pattern of the metallic specimen produced by the glass slag method. The diffraction peaks are well indexed to the α-Fe in the XRD pattern, suggesting that the metallic specimen consists of the α-Fe phase. It is known that the $Sm_2Fe_{17}N_3$ phase in the Sm–Fe–N magnets decomposes into α-Fe and SmN above 873 K [11]. However, no diffraction peaks of the other phase are seen in the XRD pattern. It has been reported that the molten boron trioxide dissolves impurities from molten metals [13]. The SmN phase formed by the decomposition of the $Sm_2Fe_{17}N_3$ phase might have been extracted by the surrounding molten boron trioxide during the melting and solidification of the Sm–Fe–N powder.

Fig. 3 shows XRD patterns of the boron trioxide before and after the glass slag experiments. The XRD pattern of the boron trioxide before the glass slag experiment shows halo-like peaks together with the small peaks of the crystalline

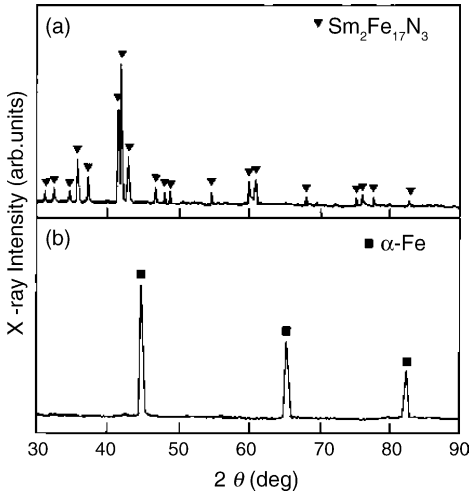

Fig. 2. XRD patterns of (a) the original Sm–Fe–N powder and (b) those processed by the glass slag method.