Magnetic properties of HDDR powders from precursors by atomization

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In conventional atomization approach of producing permanent magnetic powders for HDDR process, only powders with small size, below 200 μm for example, can be used in order to guarantee good magnetic properties of the products. In the present presentation, we utilized large powders greater than 200 μm in diameter selected from atomization to produce permanent magnetic powders by HDDR treatment. It is demonstrated that the cylindric grains of atomized powders can be changed to fine structure with coaxial grains and good magnetic properties can be achieved. It is also found that after HDDR treatment, an annealing is necessary to further improvement coercivity enhancement of the HDDR powders.

Key words: HDDR, atomization powders, Nd-Fe-B magnet, coercivity.

1. Introduction

Nd-Fe-B magnetic powders produced by inert gas atomization with following hydrogenation, disproportionation, desorption and recombination (HDDR) treatment have been studied recently. The powders used in these studies are spherical in shape and have size below 200 μm in diameter. It is found that after HDDR treatment the powders with coaxial-grain structure exhibit magnetic anisotropy.^{1,2} However, the proportion of the powders with the size larger than 200 μm reaches 15% of the total atomized powders. This presentation reported the magnetic properties of the powders by HDDR treatment of large atomized powders by atomization. It is indicated that atomized powders in spherical shape are favorable to the hydrogenation and desorption process in the HDDR treatment, and the HDDR powders exhibit higher magnetic properties than that of atomized powder in the size of below 200 μm . Coarse cylindric grains in atomized powder before HDDR process converted to fine coaxial grains after HDDR treatment.

2. Experimental

The alloy with composition of $\text{Nd}_{12.8}\text{Fe}_{70.5}\text{Co}_{10}\text{Ga}_{0.5}\text{B}_6$ (at.%) was chosen to carried out the HDDR treatment. The alloy was melted in a induction furnace and then atomized under high pressure in Ar atmosphere. After atomization, the powders with the size above 200 μm in diameter were selected to conduct a HDDR treatment. For the purpose of comparison, the same treatment was also performed to the atomized powders below 200 μm in diameter. The powders to be

treated were put into a container which was evacuated and then filled with hydrogen of 2-5 atm. After elevating temperature to 200°C and keeping this temperature for 1-1.5 hour, then raised temperature to 800-850°C and filled the container with hydrogen of 5-8atm. After keeping temperature for 5-10 min., the hydrogen pressure was decreased to 2-4atm and keep temperature for 2-4h for disproportionation. After decrease in temperature to 700-750°C, the hydrogen was evaluated for desorption. After HDDR process, an annealing at approximately 600°C for 1h is performed in order to further increase the coercivity of the HDDR powders. The morphology and the microstructure were characterized by scanning electron microscopy and transmission electron microscopy equipped with energy dispersive analysis system.

3. Results and discussion

Figure 1 shows a size distribution of atomized powders. The as-atomized powders present spherical shape with smooth surface, as shown in Figure 2. The characterization by transmission electron microscopy shows that the powders exhibit amorphous, coaxial or cylindric microcrystalline structure, with powders above 200 μm indicating cylindric microstructure. The microstructure of the atomized powders are characterized by coarse cylindric grains with cross section of 2-4 μm . It has been established that particles with a few micrometers fall into single magnetic domain category.

The powders spherical in shape are suitable for treatment of HDDR. In the hydrogenation process.

Fig. 1 The distribution of as-atomized powders.

hydrogen penetrates the powders along the crystal interface and reacts with Nd-rich phases first and then reacts with $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase.³ After hydrogenation, the powders were broken into small particles. These small particles still composed of polycrystals. These polycrystals are not cylindric but coaxial in shape, as shown in Figure 3.

With addition of appropriate amount of Ga, the remanence of the HDDR powders was found to be enhanced significantly. The reason for this can be attributed to the favorable orientation of recrystallized $\text{Nd}_2\text{Fe}_{14}\text{B}$ grains in dehydrogenation.⁴ In subsequent recombination, the products formed in disproportionation process return to initial alloy with a structure of very fine grains. Therefore, a enhancement of remanence (Br) appeared.

Fig. 2 The morphology of as-atomized powders

Fig. 3 The morphology of powders after HDDR treatment

The magnetic properties of permanent magnets, especially the coercivity iH_c is closely related to their microstructure. The microstructure of the HDDR powders depends on the HDDR treatment process. It is demonstrated that magnetic properties, especially coercivity, is dependent on the microstructure of magnets. The present experimental results indicated that the HDDR treatment is important for the improvement of coercivity of the product powders. As shown above, the atomized powders with large size exhibit coarse cylindric grain structure. Magnetic measurement shows that the coercivity of the powders decrease with the increase in the

powder size, as shown in Figure 4 (line a), in which the data were also given for the powders below. After hydrogenation and disproportion process of $200\mu\text{m}$ for comparison. One can see that the coercivity of the as-atomized powders greater than $200\mu\text{m}$ is very low. HDDR treatment, the microstructure of the powders changed from coarse cylindric grains to homogeneous-distributed fine coaxial grains; and in the desorption, the formation of fine grains in the regime of single domain size will result in prominent enhancement of coercivity (iH_c), as shown in Figure 4 (line b). The coercivity of powders by HDDR treatment is independent on the initial atomized powder size. In addition, one can see from Figure 4 (line c) that the coercivity can be further improved upon an annealing treatment. This may be ascribed to the homogenization of the microstructure by growth of too small grains and elimination of residual stress formed during the HDDR treatment.

Fig. 4 The relationship between powder size and coercivity before and after HDDR treatment. a) as-atomized; b) HDDR treatment; c) HDDR + annealing.

4. Conclusions

Large powders (above $200\mu\text{m}$) selected after atomization can be utilized to produce permanent magnetic powders with good magnetic properties by HDDR treatment. In the treatment, the cylindric grains of atomized powders can be changed to fine structure with coaxial grains. After HDDR treatment, an annealing is favorable to further improvement of magnetic properties of the HDDR powders by coercivity enhancement.

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