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The Effects of Hydrogen Reduction on Magnetic Properties of Barium Ferrite Particles

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Abstract. A reduction process of the substituted Ba-ferrite has been found to be divided into three steps. At the first and second steps, the magneto-plumbite structure maintains and the coercivity increases. When the reduced-substituted Ba-ferrite particles are reoxidized, the coercivity is reversible in the first step but irreversible in the second step. During the third step, the magneto-plumbite structure was collapsed with formation of α -Fe and BaFeO_{3-x} phases and consequently the coercivity distribution was broad and the coercivity irreversible. The coercivity and saturation magnetization decreases and increases up to 130 emu/g respectively. In this study, it is found that the substituted cations prevent the magneto-plumbite structure from collapse during the reduction process and furthermore migrate from the magnetic sites of $4f_{IV}+2a$, $2b$, and $12k$ to $4f_{VI}$ and $12k'$. An increase in the coercivity before the collapse of magneto-plumbite structure is attributed to the migration of substituted cations in hexagonal Ba-ferrite structure.

1. INTRODUCTION

It has been known that the Ba-ferrite is one of the best candidate materials for high density magnetic recording. The Ba-ferrite particles have smaller magnetic moment than currently used needle-shaped particles. To overcome disadvantages of low magnetic moment, methods such as an improvement of Ba-ferrite's magnetic moment [1], double layer structure of tape with underlayer of $\text{Co-}\gamma\text{-Fe}_2\text{O}_3$ or metal particle [2], and an improvement of magnetic head's sensitivity [3] have been proposed. In order to increase magnetic moment of Ba-ferrite particles, cation substitution method [4], epitaxial growing of spinel ferrite on Ba-ferrite surface [5], and creation of α -Fe phase by reduction [6] have been studied. During the reduction process, magnetic moment increases with an amount of α -Fe phase created, and coercivity increases with reduction temperature unlike the behaviors of pure Ba-ferrite particles. Therefore, the reduction behaviors of both substituted and pure Ba-ferrite particles have been studied to understand the reason why an increase in coercivity with the reduction temperature occurs.

2. RESULTS AND DISCUSSION

The Ba-ferrite particles were prepared by glass-recrystallization process, which is described in details elsewhere. [7] The coercivity of pure Ba-ferrite particles decreases monotonically with reduction temperature, while that of substituted Ba-ferrite particles shows a peaking effect of coercivity as shown in Figure 1. The specific saturation magnetization decreases slightly for both substituted and pure Ba-ferrite particles up to 350 °C and does rapidly for the pure Ba-ferrite particles at 450 °C because of phase transformation from magneto-plumbite structure to amorphous phase. But the substituted Ba-ferrite retains magneto-plumbite structure with small amount of α -Fe phase up to 450 °C. The α -Fe phase is a dominant phase at 520 °C for both

pure and substituted Ba-ferrite particles with insignificant amount of BaFeO_{3-x} phase. An appearance of α -Fe phase at 520 °C enhances the specific saturation magnetization and decreases the coercivity. After complete reduction the specific saturation magnetization reaches 130 emu/g at 520 °C. A coercivity distribution becomes broad from 350 °C before formation of α -Fe.

Figure 2 shows TG and DTG curves for study of reduction behaviors of the substituted Ba-ferrite particles. The reduction process is found to be divided into three steps. Weight loss up to 700 °C is 24.1 %, which indicates 88 % of the particle is reduced judging from 27.3 % oxygen content of pure Ba-ferrite. During the first step at 30 °C to 280 °C, the weight loss is small, 1.2 %, while the weight loss increased to 7.8 % in the second step at 280 °C to 410 °C. The coercivity is reversible by the re-oxidation for the first step, while during the second step the switching field distribution becomes broad with irreversible coercivity. This is because Ba-ferrite was reduced above thickness of oxygen deficient layer which oxygen transfers reversely, the surface layer became oxidized but when it was re-oxidized the inner layer didn't. In the third step the weight loss is 15.1 %, which is attributed to reduction of Ba-ferrite to α -Fe and a small amount of BaFeO_{3-x} . An exposure of this reduced Ba-ferrite particles to the air is immediately reoxidized.

Fig. 1 Change in coercivity and specific saturation magnetization of pure and substituted Ba-ferrite particles with reduction temperature.

Fig. 2 TG and DTG curves of substituted Ba-ferrite particles with reduction temperature. (20 °C/min., $\text{H}_2:\text{N}_2=1:1$)

Mössbauer spectra of substituted Ba-ferrite particles reduced at the various temperature were taken for a study on an increase in coercivity. The substituted Ba-ferrite particles retain a single phase up to 350 °C, at which reduction temperature the coercivity increases sharply. The substituted cations migrated from the sites of $4f_{IV}+2a$, $2b$, and $12k$ to $4f_{VI}$ and $12k'$ during the reduction process. It is speculated that oxygen deficiency of Ba-ferrite, occurred during reduction, enhances the substituted cations to migrate to thermodynamically stable sites. Therefore the increased coercivity of single phase Ba-ferrite particles reduced up to 350 °C is not enhanced by a strain induced anisotropy, which occurred due to nucleation of α -Fe phase in Ba-ferrite structure [6], but by nucleation of highly anisotropic phase in the structure due to the migration of substituted cations.

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