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Preparation and Magnetic Properties of Acicular Ba-Ferrite Powder

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Acicular α-FeOOH and $Ba(OH)_2 \cdot 8H_2O$ are starting materials in this study. This paper presents the characteristics of the contents of citric acid and heating condition for preparing acicular barium ferrite powder. They control particle shape, crystalline phase, magnetic properties of acicular barium ferrite powder. So the effects of the contents of citric acid and heating condition are studied. The experimental condition for starting materials were 800~1000 °C in firing and 0~40 wt.% citric acid, respectively. Ba-ferrite particles fired at the range of 800 °C to 900 °C were maintained as acicular particle shape, but there were mixed particles of acicular and round shape after fired at 950 °C. Ba-ferrite powder of the single phase was obtained in firing at 900~1000 °C and with 20 wt.% citric acid. There were unreacted phase of α-Fe_2O_3 and $BaFe_2O_4$ phases as a second phase in case of sintering at below 850 °C. Acicular barium ferrite powder of single phase was also produced in firing at 900 °C with 20 wt.% citric acid. The saturation magnetization of single phase of acicular $BaFe_{12}O_{19}$ powder was about 51 emu/g and coercivity was about 4200 Oe.

1. Introduction

The synthesis of complex oxides using a flux has been one of the useful processing of obtaining particles with controlled shapes of particles [1]. In case of application as a magnetic recording media, it is also crucial to improve the particle shape-controlled techniques to produce the functional powders for magnetic applications.

Recently, Ba-ferrite fine powder with an irregular shape has been widely used as the magnetic recording media for transportation tickets in applications. Platlets of Ba-ferrite particles prepared by reaction in an autoclave [2] or precipitation from a melt [3] have been studied for use in perpendicular magnetic recording media. When the particles were applied to magnetic recording media, a high recording density should be requested and a good orientation to the applied field is also crucial to the performance of recording devices. Therefore, the improved preparation for acicular hexagonal ferrite particles with high anisotropy may definitely enhance the properties of the magnetic recording media.

An important focusing in this study is that the acicular Ba-ferrite powder can be formed at a relatively high temperature without a change of needle-shaped particle. Goethite is used as a precursor for magnetic iron oxide particles for recording media [4]. A smaller particle size with a higher aspect ratio has always been the target of goethite precipitation technology because of superiority of magnetic properties. There are many methods for synthesizing goethite, for example, hydrolysis of the ferric hydroxide precipitates. One of the most popular methods is the oxidative precipitation of hydrolysis iron (II) oxides from aqueous ferrous salt solutions [5, 6].

The main purpose of this work is aimed at the preparation of acicular Ba-ferrite powder by citrate process [7, 8] and keeping its needle-like shape after firing to get a single phase of $BaFe_{19}O_{12}$. Among various parameters responsible for the reaction scheme, the flux species and particle size of starting materials were chosen as citric acid, α-FeOOH, respectively. Their effects on the particle morphology of the reaction products of Ba-ferrite were investigated.

2. Experiment

Acicular Ba-ferrite particles were prepared using acicular goethite (α-FeOOH) synthesized, colloidal $Ba(OH)_2 \cdot 8H_2O$, $C_6H_8O_7 \cdot H_2O$. Goethite was prepared by wet synthesis [9]. Ferrous hydroxide was obtained from the reaction of iron (II) sulfate solution with the concentration on NaOH, and the equivalent ratio R ($2NaOH/FeSO_4 \cdot 7H_2O$) was 13. This was followed by air oxidation in a vessel equipped with thermocouples, a pH-meter, an agitator and reacted at 50 °C for 4 h. The precipitated goethite particles were washed, filtered, and dried at 80 °C.

The mole ratio of Ba/12Fe was selected in a range of 1.0~2.0 and amount of citric acid as an additives was 0~40 wt.%. The mixture of starting materials was milled in distilled water and dried for 24 h. The dried powder was fired at 800~1000 °C and obtained Ba-ferrites.

XRD analysis was carried out to identify the crystalline

phase and the morphology of particle was observed by SEM. The magnetic properties were measured by VSM.

3. Results and Discussion

Fig. 1 shows the observation of SEM for morphology of acicular α-FeOOH particles synthesized by wet process with length of 1.5~2.0 μm in average. In terms of the powder morphology, the acicular shape of particles can be provided to improve the magnetic recording properties such as recording density, noise, demagnetization loss of remanence and switching field distribution [10]. In addition, fine acicular particles of metal α-Fe have been considered as the other candidate for high density recording materials [11].

In Fig. 2 the relative XRD intensity ratio (I_R) of synthesized Ba-ferrites powders with amount of citric acid as an additives was presented to ensure the crystalline phase of Ba-ferrites. From this result, we found that the single phase of Ba-ferrite was obtained at 20 wt.% of citric acid. When excess amount of citric acid was added, I_R of α-Fe_2O_3 and $BaFe_2O_4$ values were expected to increase due to low reactivity of starting materials from those voids provided by burning-out citric acid in firing.

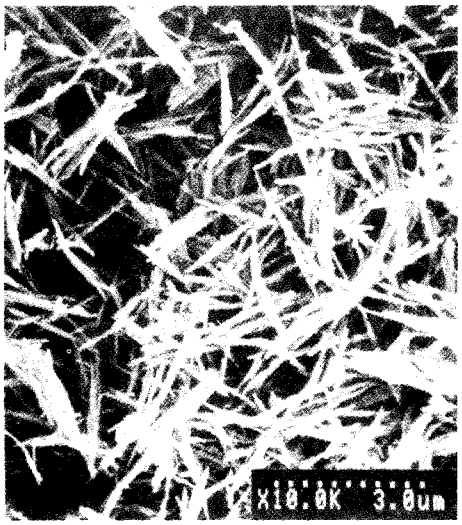

Fig. 1. SEM micrograph of acicular α-FeOOH particles as starting materials in this study.

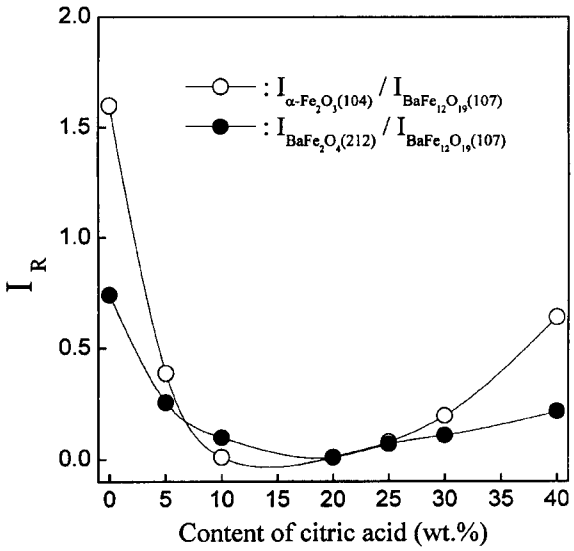

Fig. 2. Relative intensity ratio (I_R) of XRD patterns for Ba-ferrite powders fired at 900 °C for 5 h with content of citric acid.

Fig. 3 shows the results of saturation magnetization (M_S) and coercivity (H_C) of Ba-ferrites with amount of citric acid fired at 900 °C for 5h. As based on the result of Fig. 2, the magnetic properties of M_S and H_C were enhanced with citric acid added to help the reactivity between the particles of starting material. This processing was effective to prevent the particles from aggregating and in obtaining a homogeneous reaction.

Citric acid is generally useful to enhance the reactivity in compositions of alkali metal ions as an additive [12]. Therefore, it may reduce temperature for phase formation of final product in this work. As shown in Fig. 4, the single phase of $BaFe_{12}O_{19}$ was formed over 900 °C. In solid-state reaction of formation for Ba-ferrites, the firing temperature for single phase is normally much higher than 900 °C. In case of firing at 850 °C, even though a tiny peak of $BaFe_2O_4$ was detected but the single phase of $BaFe_{12}O_{19}$ was appeared at 900 °C.

In order to figure out the particle shape of Ba-ferrite in this work, SEM micrographs of Ba-ferrite powders added 20 wt.% citric acid with firing temperature were presented in Fig. 5. It is shown that the acicular shape is kept as formed up to 950 °C in firing but its acicular tip was collapsed at 1000 °C and rounded because of its interparticular sintering phenomena with increasing the firing temperature.

The requirements for enhancements of these acicular Ba-ferrite powders is to prepare a stable morphology of particle shape and maintain their high performance of magnetic properties, which should be increasingly important to use as

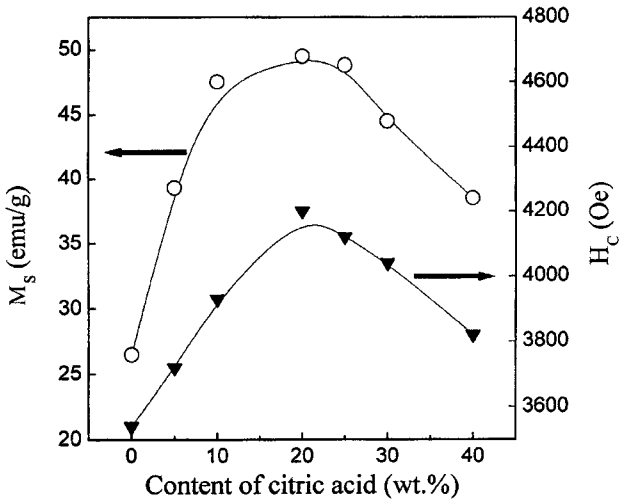

Fig. 3. Saturation magnetization (M_S) and coercivity (H_C) of Ba-ferrite powders with content of citric acid fired at 900 °C for 5 h.

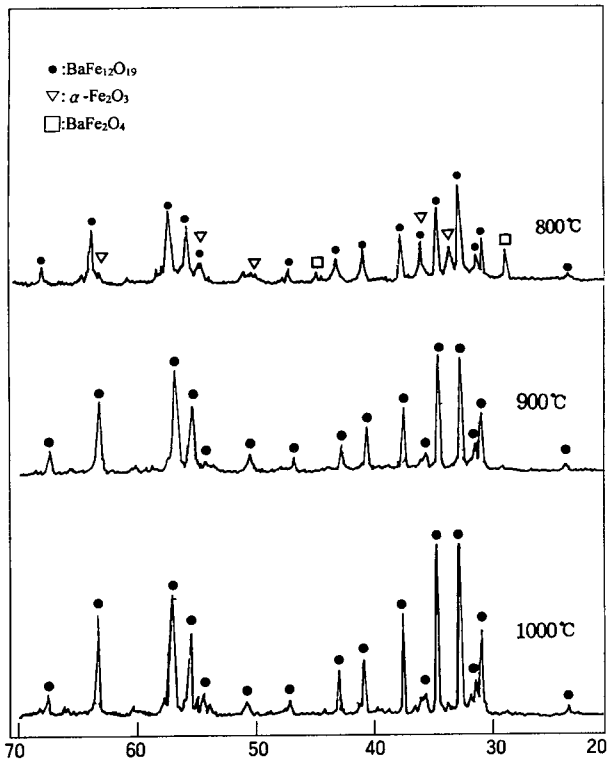

Fig. 4. XRD patterns of Ba-ferrite powders with addition of 20 wt.% citric acid fired at various temperature for 5 h.

magnetic recording particulate media. Fig. 6 shows the variation of M_S and H_C with firing temperature of Ba-ferrites as synthesized. The saturation magnetization was jumped up to about 48 emu/g for sample fired at 850 °C and steadily increased up to about 52 emu/g over 850 °C. On the other

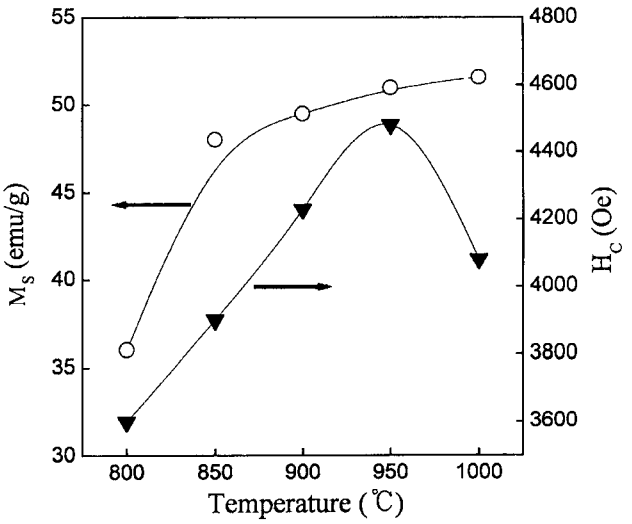

Fig. 6. Saturation magnetization (M_S) and coercivity (H_C) of Ba-ferrite powders with addition of 20 wt.% citric acid fired at 900 °C for 5 h.

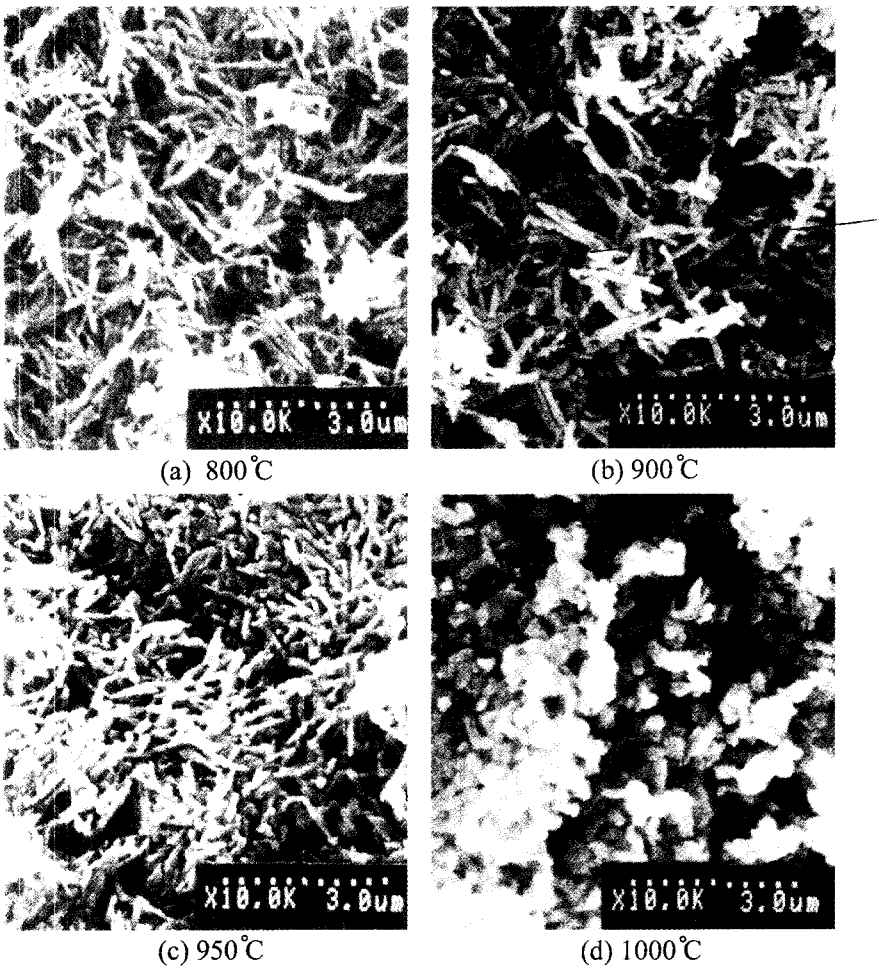

Fig. 5. SEM micrographs of acicular Ba-ferrite particles with addition of 20 wt.% citric acid fired at various temperatures for 5 h.

hand, it is found that the coercivity was sensitively affected by firing temperature, which had relatively strong dependence on firing condition, but tended to decrease at higher temperature over 950 °C. Any decrease in saturation magnetization or coercivity of Ba-ferrite in this study should be mainly due to its unreacted and second phase such as α-Fe_2O_3 and $BaFe_2O_4$ with amount of citric acid existed in the matrix of $BaFe_{19}O_{12}$.

4. Conclusions

The single phase of acicular Ba-ferrite powder were successfully prepared by citrate process with optimum condition of preparation. The magnetic properties of acicular Ba-ferrite powders were dominantly dependent on the amount of citric acid rather than firing temperature in this study. Those results may provide the possibility to obtain fine particles of any acicular ferrites for the purpose of application to the new functional materials including magnetic recording media that can be formed from the starting materials of acicular goethite particles.

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