

Barium ferrite powders prepared by microwave-induced hydrothermal reaction and magnetic property

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ABSTRACT

We have prepared barium ferrite particles by the microwave-induced hydrothermal method. The crystallization of the barium ferrite particles is promoted within a short time by microwave irradiation because the seeds of barium ferrite having large permeability are rapidly heated through the interaction of barium ferrite with the magnetic component of the microwaves. Crystals having unusually large thickness were obtained compared with the conventional hydrothermal method. The magnetic properties of barium ferrite particles are discussed.

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1. Introduction

Some ferrite ceramics with magnetic properties have been extensively studied for use as permanent magnetic materials, electromagnetic wave absorption materials and for use as advanced recording media [1–4]. Hexagonal barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$) has frequently been used as a hard ferrite ceramic due to its large saturation magnetization, large uniaxial magnetic anisotropy in a longitudinal direction (c -axis), high Curie temperature and high chemical stability [5–8]. However, particles with large size suffer from demagnetization due to multiple magnetic domain structures [9]. The direction of magnetization in the crystals is mobile, and can rotate around the applied magnetic field counter to the magnetization direction of a permanent magnet. Therefore, barium ferrite for technological application needs to have not only fine particle size with narrow size

distribution but also chemical homogeneity in its microscopic structures [10–12]. Moreover, all the particles should be arrayed in a plane for coating of magnetic media in applied devices, so that the c -axis of the particles is aligned perpendicular to the film plane. Therefore, barium ferrite particles having a flat plane in shape with thin and small hexagonal crystals are desirable.

The solid-phase reaction for preparing barium ferrite requires high-temperature annealing at over 1273 K, which induces agglomeration of the particles. For that reason, a milling process for the agglomerates is absolutely imperative, but this causes broad particle size distribution of barium ferrite and introduces impurities during the processing.

Several solution phase reactions, such as co-precipitation [13], glass crystallization [14], microemulsion synthesis [15] and sol-gel synthesis [16], have been studied to produce fine and homogeneous particles. These methods have been applied to preparation of the precursors of ultrafine barium ferrite particles under low temperatures (below 500 K). However, these methods typically lead to the partial formation of residual intermediates such as $\alpha\text{-Fe}_2\text{O}_3$, $\gamma\text{-Fe}_2\text{O}_3$, BaCO_3 and BaFe_2O_3 with barium ferrite. High-temperature annealing is needed to obtain pure phase

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BaFe₁₂O₁₉, which results in significant increase in particle size [17]. Some other methods have succeeded in the growth of single-phase BaFe₁₂O₁₉ with particle sizes in the single domain limit [18–21]. However, a long reaction time was needed.

In the case of hydrothermal reaction, pure barium ferrite powder is obtained without the formation of these intermediates at a relatively low temperature (573 K) [22]. Ponton et al. [23] have reported on the hydrothermal synthesis of barium ferrite by conventional heating with an autoclave. In this hydrothermal method, particles of a flat plane, in shape perpendicular to the *c*-axis, were obtained. Ponton's idea, i.e., employing the hydrothermal reaction, should be taken as a strategy for controlling the morphology of barium ferrite particles.

Recently, our group has reported on preparation of mono-dispersed Ag and Cu nanoparticles with narrow size distribution under microwave irradiation [24–26]. For microwave synthesis, reactants were directly heated by microwave irradiation because microwaves have a characteristic heating mode through the interaction of oscillating electric and magnetic fields with substances. Furthermore, specific effects of microwave irradiation on chemical reaction have been demonstrated, i.e. enhancements in the reaction rates, yields and selectivities [27–30].

The microwave-induced hydrothermal methods were studied by Komarneni et al. [31–34] on various spinel ferrites. The mechanism of the formation of hexagonal barium ferrite is different from that of spinel ferrites. Barium ferrite is formed through the intermediate phases of α -Fe₂O₃ and BaFe₂O₃, while spinel ferrite is formed by one step without any intermediates. Therefore, pure barium ferrite particles are more difficult to prepare than spinel ferrite. Precise control of the particle size and shape of barium ferrite particles requires different information and strategies compared with spinel ferrites.

Chikata and Katoh [35] prepared fine particles of barium ferrite using the hydrothermal method under microwave irradiation. We believe that it is worth offering more information about this method for the preparation of barium ferrite. This article reports on the structure and magnetic properties of fine barium ferrite particles prepared by the microwave-induced hydrothermal method at low temperature within a short time.

2. Experimental details

The sizes and morphologies were characterized by a transmission electron microscope (TEM) at 200 kV with Hitachi H-800 (Hitachi High-Technologies Co.). The crystal phase of the powder was analyzed with powder X-ray diffraction (XRD). XRD measurements were taken using a MultiFlex (Rigaku Co.) with a Cu-K α radiation source in the range of the 2θ Bragg angles = 20–90° at 40 kV and 40 mA. Magnetic susceptibility data were obtained at 300 K at various applied fields between –20 and 20 kOe by using a SQUID susceptometer with MPMS-5S (Quantum Design Co.).

The starting materials FeCl₃·6H₂O, BaCl₂·6H₂O and NaOH were purchased from Wako Pure Chemical Industries Ltd. FeCl₃·6H₂O (5 mmol) and BaCl₂·6H₂O (0.625 mmol), [Fe³⁺]/[Ba²⁺] = 8, were dissolved completely in deionized water (40 ml). The coprecipitate was produced by adding NaOH (40 mmol) to the solution. The resultant solution in a sealed tetrafluorometoxil (TFM) vessel was heated with a 2.45 GHz microwave apparatus Ultra Clave (Milestone Co.) at a rate of temperature increase of 10 K/min, then kept at 473 K for 30 min, and then cooled to room temperature. The reaction pressure under initial Ar atmosphere (5 MPa) went up to 10 MPa by microwave heating. The precipitate was collected by filtration and washed with deionized water three times. The precipitate was dried under vacuum at 334 K for 4 h. Rufous barium ferrite

powder was obtained (denoted as A). The barium ferrite was heat-treated at 1273 K for 1 h in air (denoted as B).

3. Results and discussion

The powder XRD patterns of samples A and B are shown in Fig. 1. The XRD pattern of sample A produced at low temperature by microwave irradiation corresponded to the pure phase of the hexagonal BaFe₁₂O₁₉ with high crystallinity, including no intermediates such as α -Fe₂O₃, γ -Fe₂O₃, BaCO₃ or BaFe₂O₃. The difference in the intensity of (107) peaks between samples A and B indicates the preferential growth of certain crystallographic planes of the particles. The lattice parameters ($a = 5.890 \text{ \AA}$, $c = 23.24 \text{ \AA}$) calculated from the XRD for sample A were in good agreement with general bulk hexagonal barium ferrite. The TEM images of samples A and B are shown in Fig. 2. The particles possessed flat planes in shape. The average size, thickness and aspect ratio were measured from the TEM images and are given in Table 1. The average particle sizes were about 500 nm for sample A and about 1100 nm for sample B. Regarding thickness, sample A was very thin. As a result, the aspect ratio was

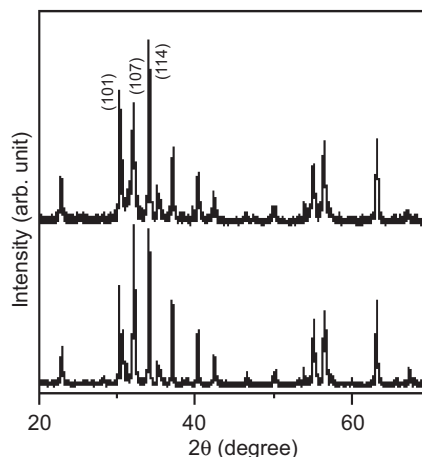


Fig. 1. X-ray diffraction patterns of samples A (above) and B heat-treated at 1273 K for 1 h (below).

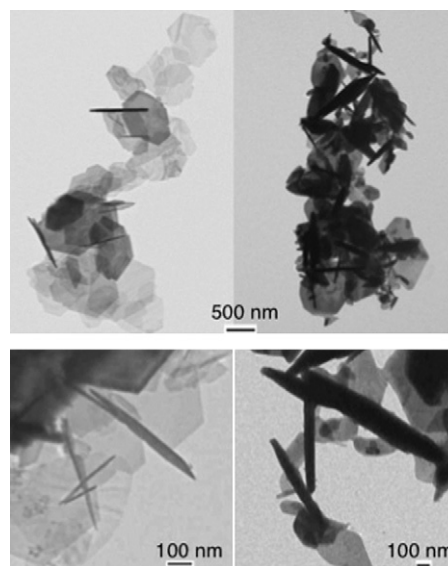


Fig. 2. Transmission electron images of samples A (left) and B (right).

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