Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 27
ISSUE 1
March
2008

Microstructure and magnetic properties of $BaFe_{12}O_{19}$ powder

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Received 04.01.2008; published in revised form 01.03.2008

Methodology of research

ABSTRACT

Purpose: Analysis of microstructure and magnetic properties of $BaFe_{12}O_{19}$ powder obtained by milling and annealing of Fe_2O_3 and $BaCO_3$ precursors.
Design/methodology/approach: The mixture of iron oxide (Fe_2O_3) and barium carbonate ($BaCO_3$) powders was used to obtain $BaFe_{12}O_{19}$ powder by using high-energy ball milling and heat treatment processes. The X-ray diffraction methods were used for qualitative, quantitative phase analyses and for crystallite size and lattice distortion determination. The thermal properties of the studied powders were analyzed using the differential thermal analysis (DTA). The magnetic properties of examined powder material were studied by resonance vibrating sample magnetometer (R-VSM). The size of powder particles was determined by a laser particle analyzer.
Findings: The milling process of iron oxide and barium carbonate mixture causes decrease of the crystallite size of involved phases. The X-ray diffraction investigations of Fe_2O_3 and $BaCO_3$ mixture milled for 50 hours and annealed at 850, 900, 950 and 1000°C enabled the identification of hard magnetic $BaFe_{12}O_{19}$ phase and also small amount of Fe_2O_3 phase. The magnetic properties of studied powders are dependent on temperature of their annealing. The sample annealed at 1000°C has the best hard magnetic properties from all studied samples. The content changes of hard magnetic phase ($BaFe_{12}O_{19}$) with the increase of annealing temperature results in the improvement of hard magnetic properties.
Practical implications: The $BaFe_{12}O_{19}$ powder can be suitable component to produce sintered hard magnetic materials.
Originality/value: The study results of $BaFe_{12}O_{19}$ powders confirm the utility of applied investigation methods in the microstructure and magnetic properties analysis of powder materials.
Keywords: X-ray phase analysis; R-VSM; High-energy ball milling; Barium ferrite

1. Introduction

Barium ferrites are well known hard magnetic materials, which are based on iron oxides. They are also called as ferrite magnets and could not be easily replaced by any other magnets [1-4]. Barium ferrites are scientifically and technologically attractive, because of their relatively high Curie temperature, high coercive force and high magnetic anisotropy field, as well as their excellent chemical stability and corrosion resistivity [5,6].

Ferrites are still widely used although they have less magnetic strength than rare earth magnets [7]. Due to these properties,

many methods of synthesis have been developed to obtain a low production cost of powder particles of barium ferrite [8].

Barium ferrites are usually produced by the conventional mixed oxide ceramic method, which involves the calcination of a mixture of $BaCO_3$ and Fe_2O_3 at 1200°C. More recently, they have been fabricated by milling and heat treatment process [9-11].

The aim of this paper is the microstructure analysis and magnetic properties characterization of $BaFe_{12}O_{19}$ powder obtained by milling and heat treatment of Fe_2O_3 and $BaCO_3$ precursors.

2. Material and research methodology

The $BaFe_{12}O_{19}$ powders were obtained from the mixture of iron oxide Fe_2O_3 (99% purity) and barium carbonate $BaCO_3$ (99% purity) powders ($1.1BaCO_3 + 6Fe_2O_3$) by using high-energy ball milling and heat treatment processes.

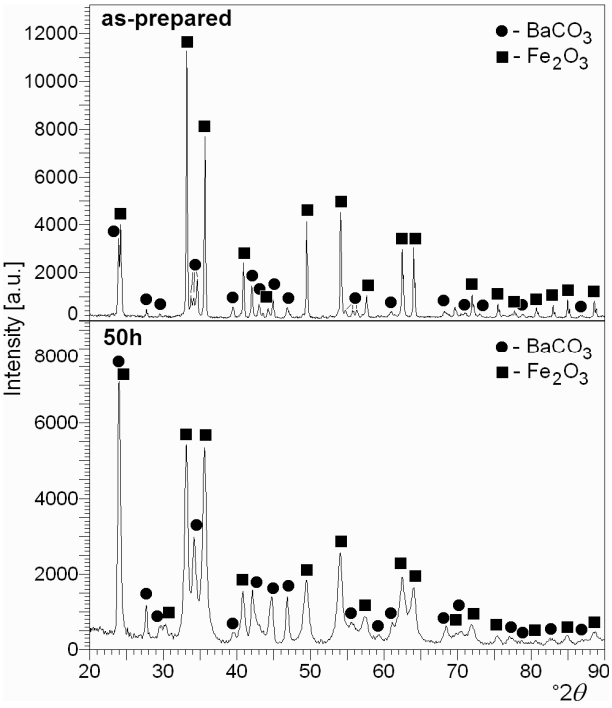

Fig. 1. X-ray diffraction patterns of as-prepared Fe_2O_3 and $BaCO_3$ mixture and after 50 hours of milling process

Ball milling process was carried out in a vibratory mill (SPEX 8000 CertiPrep Mixer/Mill) for 50 hours under argon atmosphere. The weight ratio of balls to milled material was 5:1. After milling process the powders were annealed in the electric chamber furnace (Thermolyne 6020C) at 850, 900, 950 and 1000°C in the air under atmosphere pressure for 1 hour.

Phase analysis was carried out using the X-Pert Philips diffractometer equipped with curved graphite monochromator on diffracted beam and a tube provided with copper anode. The X-ray diffraction patterns were recorded by "step-scanning" method in 2θ range from 20° to 140° and 0.05° step.

The Rietveld analysis was performed applying DBWS-9807 program that is an update version for Rietveld refinement with PC and mainframe computers. The pseudo-Voigt function was used in the describing of diffraction line profiles at Rietveld refinement. The phase abundance was determined using the relation proposed by Hill and Howard [12-14]. The crystallite sizes and lattice distortions for Fe_2O_3 and $BaCO_3$ phases were estimated using Williamson-Hall method [15].

The thermal properties of the tested powder were determined by the Mettler thermoanalyser using the differential thermal analysis (DTA) at a heating rate of 15 K/min under an argon atmosphere.

The magnetic hysteresis loops of obtained powder material were measured by the Resonance Vibrating Sample Magnetometer (R-VSM). The idea of R-VSM is based on the Faraday induction law and the original Foner solution.

The diameter sizes of examined powder particles were determined using Fritsch Particle Sizer "Analysette 22" in measuring range from 0.1 µm to 1180 µm.

3. Results and discussion

The diffraction patterns of Fe_2O_3 and $BaCO_3$ mixture (Fig.1) shows the broadening of diffraction lines for milled samples. These effects indicate that ball milling causes decrease of the crystallite size of tested phases and involved to homogenization of the milled mixture. As the result of milling process the crystallite size (D) of Fe_2O_3 and $BaCO_3$ phases diminishes to 15 nm and 35 nm, respectively of the milling time up to 50 hours.

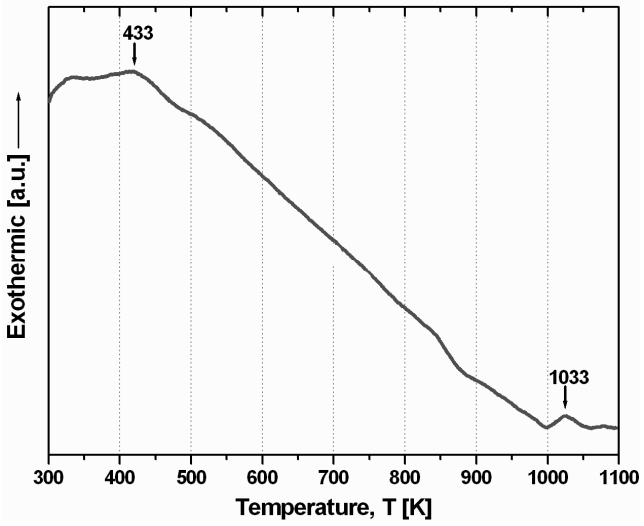

Fig. 2. DTA pattern of Fe_2O_3 and $BaCO_3$ mixture after 50 hours of milling process

After 50 hours milling process the lattice distortions ($<\Delta a/a>$) are 0.12% and 0.042% for Fe_2O_3 and $BaCO_3$, respectively.

The differential thermal analysis showed exothermic reaction peaks at 433°C and at 1033°C (Fig. 2). The first peak probably corresponds to formation of the barium hexaferrite phase. The

hysteresis loop of Fe_2O_3 and $BaCO_3$ mixture after milling is shown in Figure 3. The hard magnetic properties of examined mixture are very low, because of lack of hard magnetic phase.

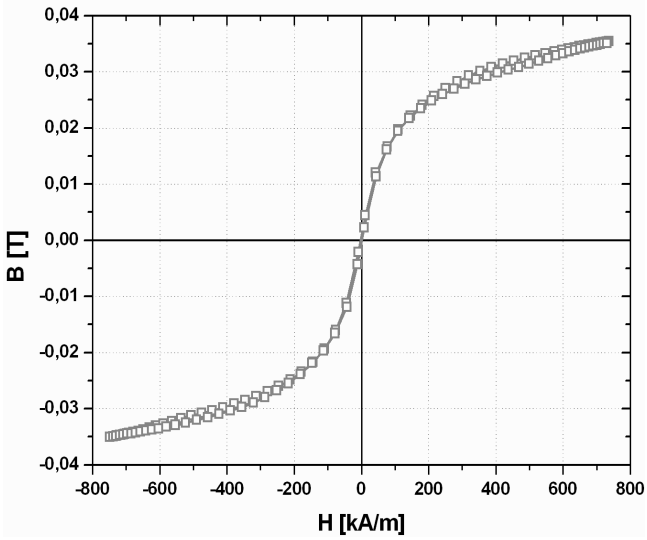

Fig. 3. Hysteresis loop of Fe_2O_3 and $BaCO_3$ mixture after 50 hours of milling process

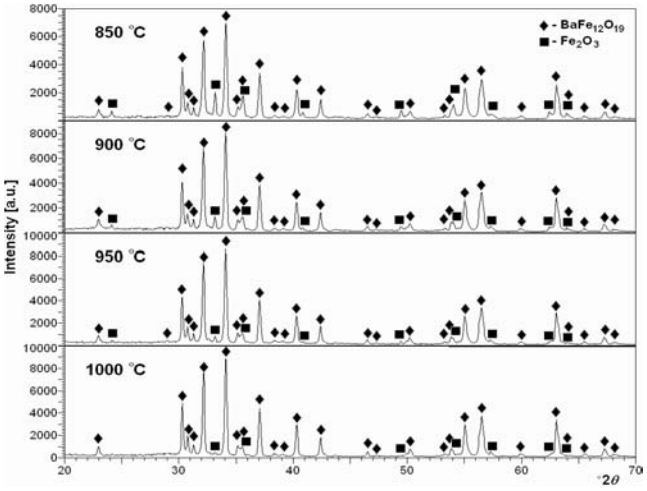

Fig. 4. X-ray diffraction patterns of Fe_2O_3 and $BaCO_3$ mixture after 50 hours of milling and annealing at 850, 900, 950 and 1000°C

The X-ray diffraction investigations of Fe_2O_3 and $BaCO_3$ mixture milled for 50 hours and annealed at 850, 900, 950 and 1000°C enabled the identification of hard magnetic $BaFe_{12}O_{19}$ phase (Fig. 4). Moreover, the X-ray analysis revealed also the presence of Fe_2O_3 phase in studied materials.

The effect of annealing process on content of $BaFe_{12}O_{19}$ and Fe_2O_3 phases for different temperatures is presented in Figure 5 and 6. The annealing process causes increase of content of hard magnetic phase ($BaFe_{12}O_{19}$) obtained after heat treatment and decrease of Fe_2O_3 phase. After annealing at 1000°C the contents of $BaFe_{12}O_{19}$ and Fe_2O_3 phases are equal to 98.4 and 1.6 wt.%.

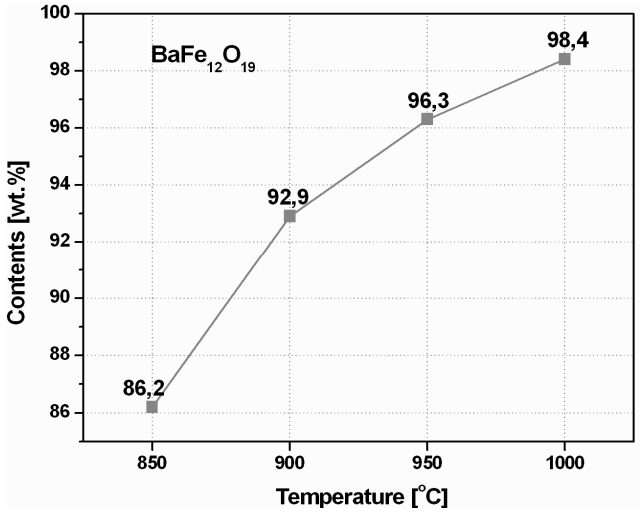

Fig. 5. Contents of $BaFe_{12}O_{19}$ phase after 50 hours of milling and annealing at 850, 900, 950 and 1000°C

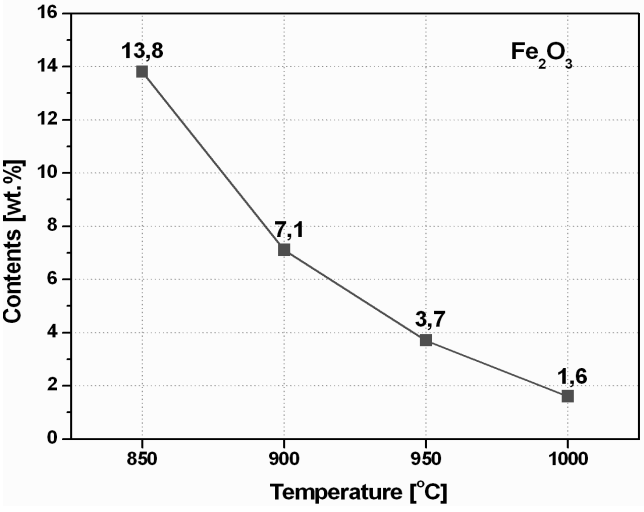

Fig. 6. Contents of Fe_2O_3 phase after 50 hours of milling and annealing at 850, 900, 950 and 1000°C

The effect of annealing temperature on magnetic properties of Fe_2O_3 and $BaCO_3$ mixture is presented in Figure 7. Table 1 contains chosen magnetic properties measured in the field 800 kA/m. The magnetic properties of studied powders are dependent on temperature of their annealing. For example, the sample annealed at 1000°C has the best hard magnetic properties from all studied samples. The remanence (B_r) of that material has a value of 0.116 T, the saturation magnetization (B_s) is 0.189 T and coercive force (H_C) for sample annealed at 1000°C is 266 kA/m.

The high values of remanence, saturation magnetization and coercivity are certainly associated with the microstructure and phase composition of investigated powders after annealing process. The content increase of hard magnetic phase ($BaFe_{12}O_{19}$) with the increase of annealing temperature results in improvement of hard magnetic properties.

Table 1.
Magnetic properties of Fe_2O_3 and $BaCO_3$ mixture after 50 hours of milling and different annealing temperature (field 800 kA/m)

Annealing temperature [°C]	Coercive force H_c [kA/m]	Remanence, B_r [T]	Saturation magnetization, B_s [T]
850	253	0.099	0.164
900	279	0.102	0.168
950	219	0.104	0.172
1000	266	0.116	0.189

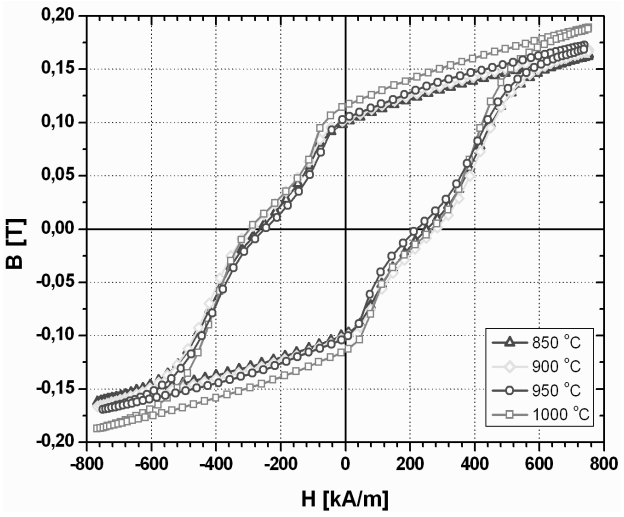

Fig. 7. Hysteresis loops of Fe_2O_3 and $BaCO_3$ mixture after 50 hours of milling and annealing at 850, 900, 950 and 1000°C

The diameter arithmetic mean of examined population of Fe_2O_3 and $BaCO_3$ powders after 50 hours of milling is 7.64(6) µm. Moreover, the size of powders, which are the most probable (mode) is 15.11(7) µm. The representative diameter of examined powders (median) is equal to 4.37(5) µm.

4. Conclusions

The investigations performed on the Fe_2O_3 and $BaCO_3$ mixture after milling and heat treatment allowed to formulate the following statements:

- high-energy milling of studied composite powder for 50 hours results in the decrease of Fe_2O_3 and $BaCO_3$ crystallite size to 15 nm and 35 nm, respectively,
- the barium ferrite phase appeared to be the main component of milled for 50 hours and annealed at 1000°C sample (98.4 wt.%), whereas the content of Fe_2O_3 phase is the much lower (1.6 wt.%),
- the magnetic properties of studied powders is dependent on temperature of their annealing,
- the best magnetic properties were obtained for powder annealed at temperature of 1000°C,
- the diameter arithmetic mean of Fe_2O_3 and $BaCO_3$ mixture powder particles after 50 hours of milling is about 8 µm.

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