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**Citation style:** Nowosielski Ryszard, Babilas R., Dercz Grzegorz, Pająk Lucjan. (2007). Barium ferrite powders prepared by mechanical alloying and annealing. "Journal of Achievements in Materials and Manufacturing Engineering" (Vol. 22, iss. 1 (2007), s. 45-58).

Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 22
ISSUE 1
May
2007

# Barium ferrite powders prepared by milling and annealing

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Received 12.03.2007; published in revised form 01.05.2007

**Methodology of research**

## ABSTRACT

**Purpose:** Microstructure and magnetic properties analysis of barium ferrite powder obtained by milling and heat treatment.

**Design/methodology/approach:** The milling process was carried out in a vibratory mill, which generated vibrations of the balls and milled material inside the container during which their collisions occur. After milling process the powders were annealed in electric chamber furnace. The X-ray diffraction methods were used for qualitative phase analysis of studied powder samples. The distribution of powder particles was determined by a laser particle analyzer. The magnetic hysteresis loops of examined powder material were measured by resonance vibrating sample magnetometer (R-VSM).

**Findings:** The milling process of iron oxide and barium carbonate mixture causes decrease of the crystallite size of involved phases. The X-ray investigations of tested mixture milled for 30 hours and annealed at 950 °C enabled the identification of hard magnetic $BaFe_{12}O_{19}$ phase and also the presence of $Fe_2O_3$ phase in examined material. The $Fe_2O_3$ phase is a rest of $BaCO_3$ dissociation in the presence of $Fe_2O_3$, which forms a compound of $BaFe_{12}O_{19}$. The best coercive force (HC) for mixture of powders annealed at 950 °C for 10, 20 and 30 hours is 349 kA/m, 366 kA/m and 364 kA/m, respectively. The arithmetic mean of diameter of $Fe_2O_3$ and $BaCO_3$ mixture powders after 30 hours of milling is about 6.0 µm.

**Practical implications:** The barium ferrite powder obtained by milling and annealing can be suitable components to produce sintered and elastic magnets with polymer matrix.

**Originality/value:** The results of tested barium ferrite investigations by different methods confirm their utility 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 and cannot be easily replaced by any other magnetic materials [1-4]. Hexagonal barium ferrite having the chemical formula of $BaFe_{12}O_{19}$ are widely used in magnetic recording media, microwave devices and electromagnetic shielding fields [5,6]. Barium ferrite possesses relatively high Curie temperature, coercive force and magnetic anisotropy field, as well as its excellent chemical stability and corrosion resistivity [7,8].

Ferrite magnets are still widely used although they have less magnetic strength than rare earth magnets. Nevertheless, many methods of synthesis have been developed to obtain a low production cost of powder particles of barium ferrite [9]. Barium ferrites are usually produced by the conventional mixed oxide ceramic method, which involves the calcination of the mixture of $BaCO_3$ and $Fe_2O_3$ at 1200˚C [6].

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

## 2. Material and research methodology

For synthesis of $BaFe_{12}O_{19}$, mixture of iron oxide $Fe_2O_3$ (99% purity) and barium carbonate $BaCO_3$ (99% purity) powders was used with composition $1.1BaCO_3 + 6Fe_2O_3$ (Fig.1.).

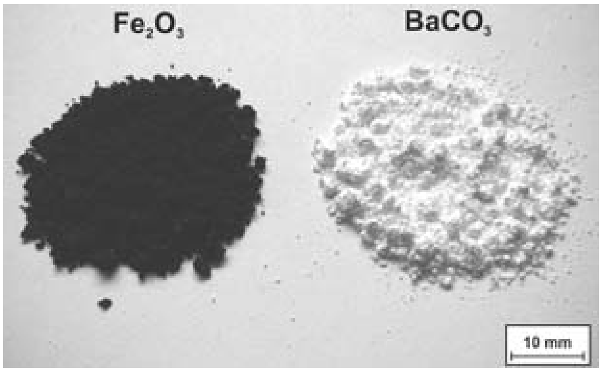


Fig. 1. External morphology of $Fe_2O_3$ and $BaCO_3$ powders used for milling process

Ball milling process was carried out in a vibratory mill type SPEX 8000 CertiPrep Mixer/Mill for 10, 20 and 30 hours under argon atmosphere. The weight ratio of balls to milled material was 5:1. The mill generated vibrations of balls and the material inside the container during which their collisions occur [10,11].

After milling process the powders were annealed in the electric chamber furnace THERMOLYNE 6020C in 900, 950 and 1000 °C in the air at 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 [12-13]. It was supplied by current intensity of 30 mA and voltage of 40 kV. The length of radiation ($\lambda Cu_{K\alpha}$) was 1.54178 Å. The data of diffraction lines were recorded by "step-scanning" method in $2\theta$ range from 10° to 90° and 0.05° step.

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 [15]. As distinct from Foner's VSM in the R-VSM sample oscillation are forced by piezoelectric transducer. Sample oscillates parallely to the direction of external magnetic field and configuration of pick-up coils in the form of small Smith coils were applied.

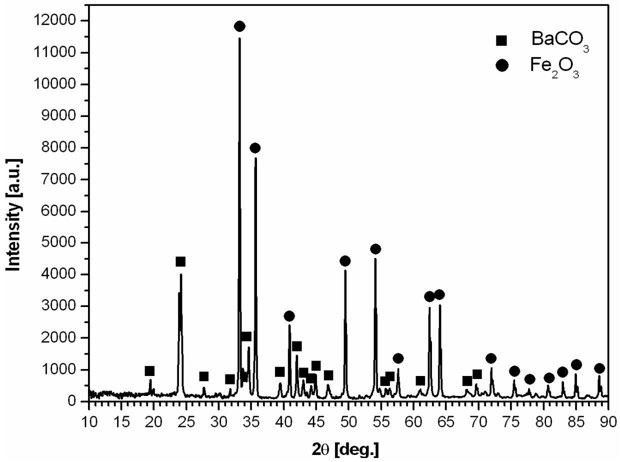


Fig. 2. X-ray diffraction pattern of $Fe_2O_3$ and $BaCO_3$ mixture as a starting material for milling

Moreover, 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

Comparison of diffraction patterns of $Fe_2O_3$ and $BaCO_3$ mixture before milling (Fig. 2) and after 30 hours of milling (Fig. 3) shows the broadening of diffraction lines and decrease of their intensity. These effects indicate that ball milling causes decrease of the crystallite size of tested phases and leads to homogenizing the milled mixture. The diffraction patterns of milled powders at different times are shown in Figure 4.

The X-ray diffraction investigations of $Fe_2O_3$ and $BaCO_3$ mixture milled for 30 hours and annealed at 950 °C enabled the identification of hard magnetic $BaFe_{12}O_{19}$ phase (Fig. 5). Moreover, the X-ray analysis also revealed presence of $Fe_2O_3$ phase in examined material. The iron oxide ($Fe_2O_3$) is a rest of $BaCO_3$ dissociation in the presence of $Fe_2O_3$, which forms a compound of $BaFe_{12}O_{19}$. The X-ray diffraction patterns of $Fe_2O_3$ and $BaCO_3$ mixture after different times of milling and annealing at 950 °C are presented in Figure 6.

The hysteresis loops of $Fe_2O_3$ and $BaCO_3$ mixture after different times of ball milling is shown in Figure 7. The hard magnetic properties of tested mixture are very low, because of lack of hard magnetic phase. The effect of annealing temperature (950 °C) on magnetic properties of $Fe_2O_3$ and $BaCO_3$ mixture for different times of milling is presented in Figure 8.

Sample annealed at 950 °C have high value of coercivity. The best coercive force ($H_C$) for mixture of powders annealed at 950 °C is 349 kA/m, 366 kA/m and 364 kA/m after 10, 20 and 30 hours of milling, respectively. The high values of coercivity are certainly associated with the microstructure of investigated powder samples.

The coercivity decreases with increasing temperature of annealing above 950 °C, as shown in Figure 9. This effect is related with increasing of the crystallite size at higher temperatures.

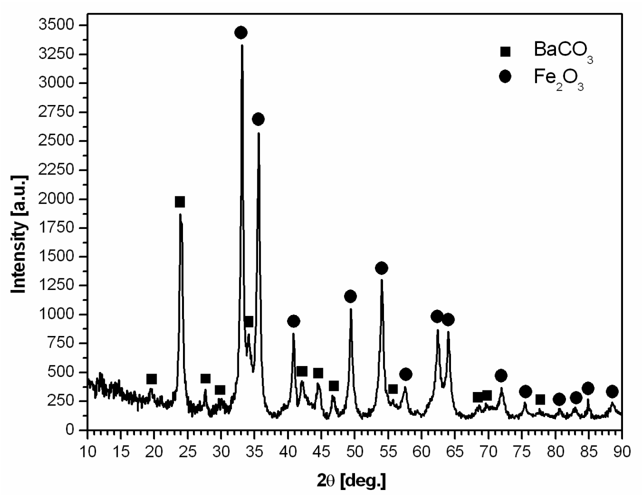


Fig. 3. X-ray diffraction pattern of $Fe_2O_3$ and $BaCO_3$ mixture after 30 hours of milling

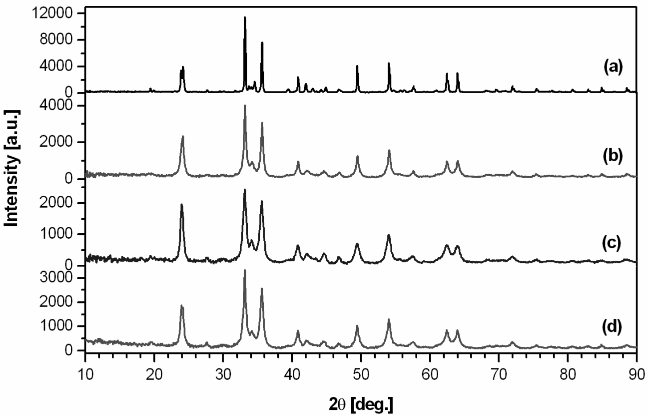


Fig. 4. X-ray diffraction patterns of $Fe_2O_3$ and $BaCO_3$ mixture after different times of milling: a) 0 h, b) 10 h, c) 20 h, d) 30 h

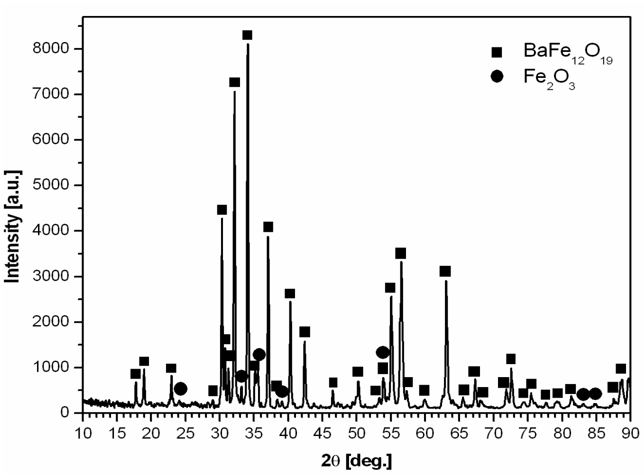


Fig. 5. X-ray diffraction pattern of $Fe_2O_3$ and $BaCO_3$ mixture after 30 hours of milling and annealing at 950 °C

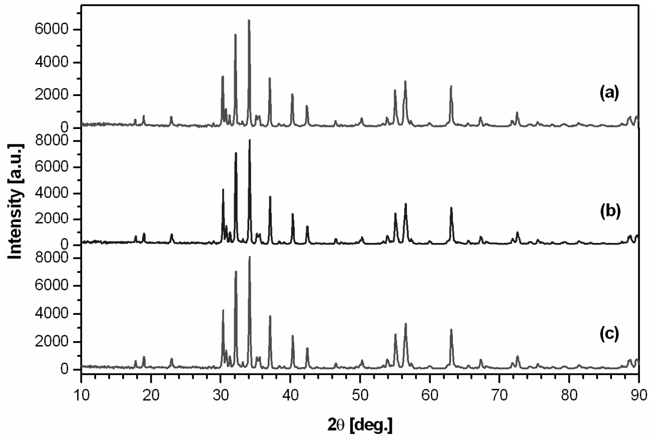


Fig. 6. X-ray diffraction patterns of $Fe_2O_3$ and $BaCO_3$ mixture after: a) 10 h, b) 20 h, c) 30 h of milling and annealing at 950 °C

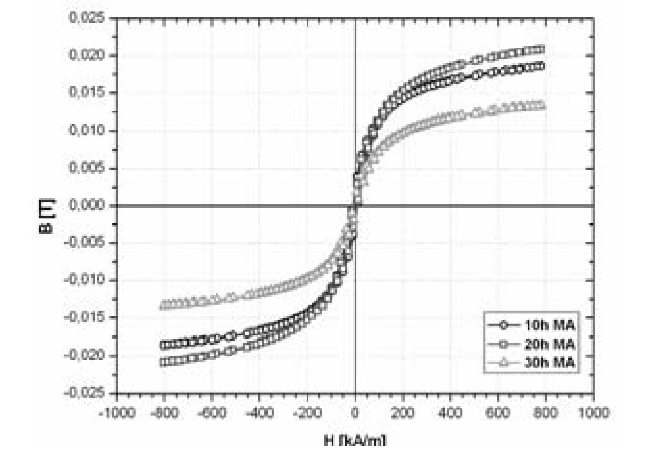


Fig. 7. Hysteresis loops of $Fe_2O_3$ and $BaCO_3$ mixture after different milling times

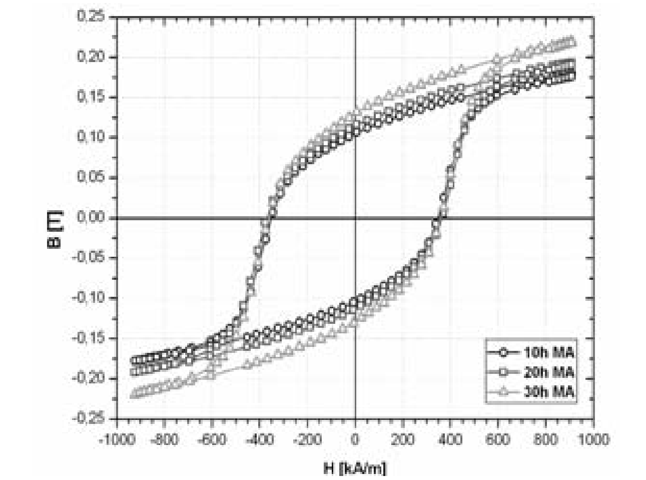


Fig. 8. Hysteresis loops of $Fe_2O_3$ and $BaCO_3$ mixture after different milling times and annealing at 950 °C

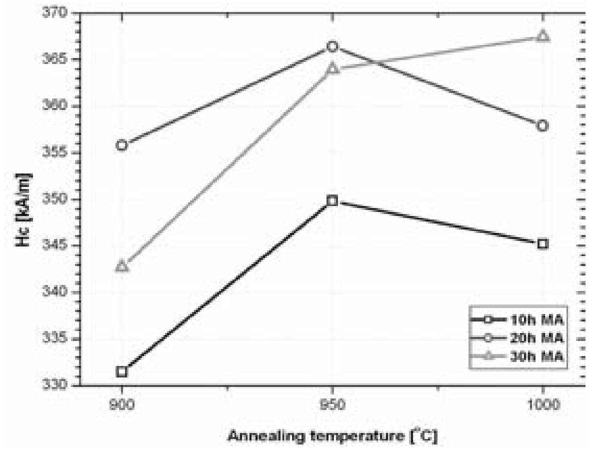


Fig. 9. Coercive force ($H_C$) as a function of annealing temperature of $Fe_2O_3$ and $BaCO_3$ mixture for different milling times

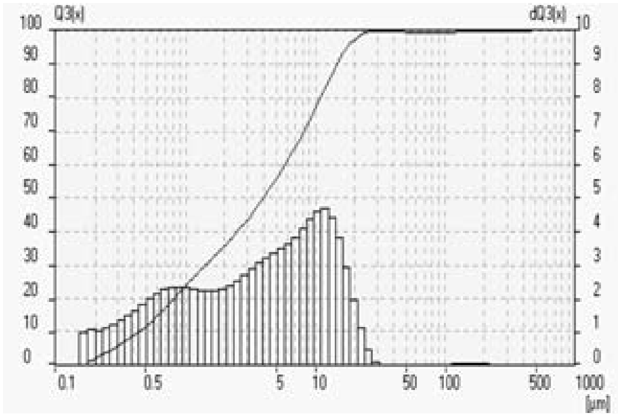


Fig. 10. Distribution and summary curve of powder particle size of $Fe_2O_3$ and $BaCO_3$ mixture after 30 hours of milling process

The distribution of powder particles size of examined powder is presented in Figure 10. The arithmetic mean of powder diameter population is 6.025 µm. The size of powders, which are the most probable (mode) is 11.731 µm. The representative diameter of examined powders (median) is a value of 3.956 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:

- the milling process of $Fe_2O_3$ and $BaCO_3$ mixture leads to crystallite refinement,
- the X-ray diffraction investigations of tested mixture milled for 30 hours and annealed at 950 °C enabled the identification of $BaFe_{12}O_{19}$ and $Fe_2O_3$ phases,
- the coercive force of studied powders is dependent on time of ball milling and temperature of their annealing,
- the best coercive force was obtained for powders annealed at temperature of 950 °C,
- the arithmetic mean diameter of $Fe_2O_3$ and $BaCO_3$ mixture powders after 30 hours of milling is about 6 µm.

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