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Microstructure and magnetic properties of commercial barium ferrite powders

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Methodology of research

ABSTRACT

Purpose: Microstructural and magnetic properties analysis of commercial barium ferrite powder $BaFe_{12}O_{19}$.

Design/methodology/approach: The X-ray diffraction methods were utilized not only for qualitative and quantitative phase analysis of studied powder sample, but also for the determination of lattice parameters, crystallite size and the lattice distortion. The Rietveld method was used in the verification of the qualitative phase composition and in the determination of phase abundance. Hill and Howard procedure was applied for quantitative phase analysis. The parameters of the individual diffraction line profiles were determined by PRO-FIT Toraya procedure. The morphology of barium ferrite powders was analyzed using the scanning electron miroscopy (SEM) method. The distribution of powder particles was determined by a laser particle analyzer. Moreover, the magnetic hysteresis loop of examined powder material were measured by resonance vibrating sample magnetometer (R-VSM).

Findings: The X-ray diffraction analysis revealed the presence of hexagonal $BaFe_{12}O_{19}$ and rhombohedral Fe_2O_3 phases in examined powder samples. The barium ferrite phase appeared to be the main component of the samples (97.8 wt.%). The crystallite size of $BaFe_{12}O_{19}$ phase is above 100 nm. The size of studied powders is in the range from 0.2 μm to 40.5 μm. The arithmetic mean diameter of $BaFe_{12}O_{19}$ powders population is 10.335 μm. The SEM images showed irregular shape and size of powder particles. The coercive force (HC) obtained from hysteresis loop has a value about 159 kA/m.

Practical implications: Structure analysis of commercial barium ferrite powder is helpful to prepare this material by laboratory methods.

Originality/value: The obtained results of investigations by different methods of structure characterization confirm their utility in the microstructure analysis of powder materials.

Keywords: X-ray phase analysis; Rietveld method; SEM; R-VSM

1. Introduction

Barium ferrite is a well known permanent magnet material [1,2]. Hexagonal barium ferrite having the chemical formula of $BaFe_{12}O_{19}$ (BaM) are widely used in magnetic recording media, microwave devices and electromagnetic shielding fields [3,4]. Barium ferrite is scientifically and technologically attractive, because of its relatively high Curie temperature, high coercive force and high magnetic anisotropy field, as well as its 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 mechanical alloying prosess [4].

The aim of the this paper is the microstructure analysis and magnetic property characterization of commercial $BaFe_{12}O_{19}$ powder.

2. Material and research methodology

The investigations were realized on the samples of commercial powders of barium ferrite. The tested material samples were prepared by putting the $BaFe_{12}O_{19}$ powder on glassy slides. The slides have square form about 5 mm lenght and 5 mm width (Fig.1.). The thickness of samples is about 1 mm.

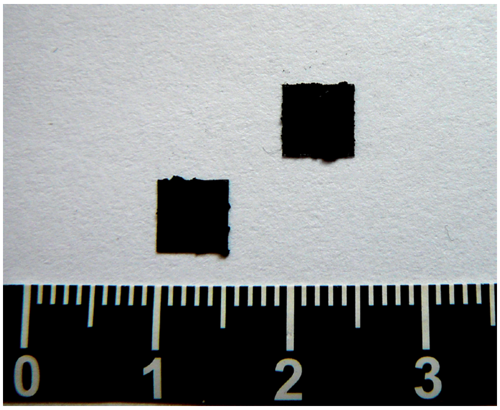

Fig. 1. External morphology of the studied barium ferrite powders in form of square samples

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. 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 2θ range from 15° to 85° and 0.05° step.

The profile parameters of individual diffraction lines were determined using Toraya PRO-FIT procedure, which applies Pearson VII function for the description of line profiles. PRO-FIT Toraya procedure enables the determination of profile parameters of the individual diffraction line.

The Rietveld analysis was performed applying DBWS-9807 program that is an update version of the DBWS programs 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 R_{wp} (weighted-pattern factor) and S (goodness-of-fit) parameters were used as numerical criteria of the quality of the fit of calculated to experimental diffraction data. Furthermore, the R_{exp} is a determinant of measurement quality realization. The phase abundance was determined using the relation proposed by Hill and Howard. The crystallite sizes and lattice distortions of $BaFe_{12}O_{19}$ phase were estimated using Williamson-Hall method.

The Rietveld method [9] and Toraya [10] procedure were applied as the usful tools of XRD patterns analysis in the verification of the qualitative phase composition [11,12].

The morphology of the barium ferrite powders and a fracture surface of the composite material was carried out using the OPTON DS540 scanning electron microscope provided at the magnification of 1000 – 5000x with the ISIS software for the computer recording of images.

Moreover, the diameter sizes of examined powder particles were determined using Fritsch Particle Sizer "Analysette 22" in measuring range from 0,1 µm to 1181,86 µm. The device consists of helium-neon laser, optical system, measuring container and steer module. Diameters of powder particles are estimated by computer unit on the ground of geometric parameters.

The magnetic hysteresis loop of studied powder material were measured by a Resonance Vibrating Sample Magnetometer (R-VSM) [13]. The idea of R-VSM is based on the Faraday induction law and the original Foner solution [14]. As distinct from Foner's VSM in R-VSM sample oscillation (with maximum amplitude of 1 mm) are forced by piezoelectric transducer. Moreover, sample oscillates parallely to the direction of external magnetic field and configuration of pick-up coils in the form of small Helmholtz was applied.

3. Results and discussion

The X-ray diffraction investigations enabled the identification of hexagonal $BaFe_{12}O_{19}$ ($P6_3/mmc$) and rhombohedral Fe_2O_3 ($R\bar{3}c$) phases (Fig. 2).

The values of lattice parameters determined by Rietveld method (the accuracy in their determination found using alumina plate SRM 1976 standard is ±0.015%) and these found in ICDD files for all concerned phases are also given in Table 1.

The barium ferrite phase appeared to be the main component of the sample (97.8 wt.%). On the other hand the content of Fe_2O_3 phase is much lower (2.2 wt.%).

The values of fitting parameters: R_{wp}, R_{exp}, and goodness-of-fit S obtained for powder samples are in the range of: R_{wp} = 7.95–8.36%, R_{exp} = 5.03–5.19%, goodness-of-fit S = 1.58–1.61. The crystallite size (D) of $BaFe_{12}O_{19}$ phase is above100 nm and the lattice strain ($<\Delta a/a>$) is 0.033%.

The hysteresis loop of tested powder sample, measured at room temperature in the field 800 kA/m, is shown in Figure 4. The coercive force (H_C) is about 159 kA/m.

Statistical means and diameter sizes of examined powder are presented in Table 2. Moreover, the distribution of powder particle size is shown in Figure 3. The size of studied barium ferrite powders is contained in range from 0,2 µm to 40,5 µm. The arithmetic mean diameter of $BaFe_{12}O_{19}$ powders population is 10,335 µm and the geometric mean diameter is 8,538 µm. The standard deviation has a value of 3,215 µm and the mean square deviation is 4,571 µm. The size of powders, which are the most probable (mode) is 10,564 µm. The representative diameter of examined barium ferrite powders (median) has a value of 9,415 µm.

Table 1.
Lattice parameters and the contents of barium ferrite powder sample [15]

Phase	Space group	Lattice parameters [nm]		Contents [wt.%]
		Rietveld	ICDD	
$BaFe_{12}O_{19}$	$P6_3/mmc$	$a_0 = 0.58910(9)$	$a_0 = 0.58920(1)$	97.8
		$c_0 = 2.3213(2)$	$c_0 = 2.3183(1)$	
Fe_2O_3	$R\bar{3}c$	$a_0 = 0.50343(8)$	$a_0 = 0.5034$	2.2
		$c_0 = 1.37607(2)$	$c_0 = 1.3747$	

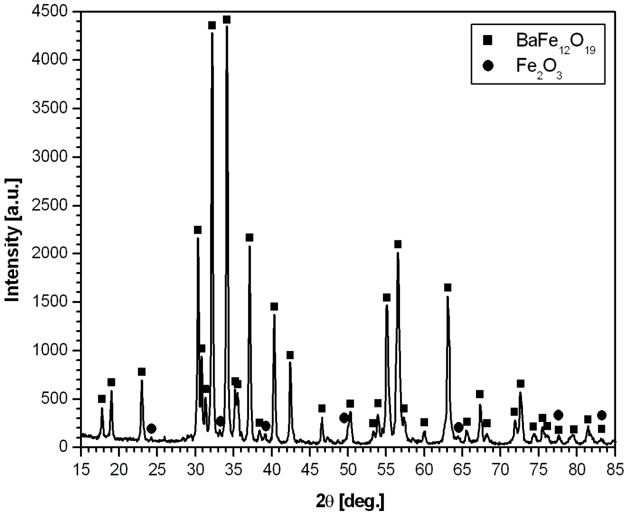

Fig. 2. X-ray diffraction pattern of barium ferrite powder sample [15]

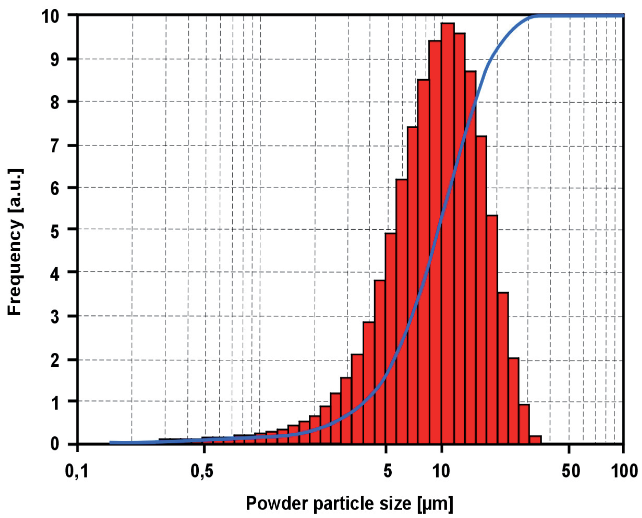

Fig. 3. Distribution and summary curve of powder particle size of studied barium ferrite powder

Table 2.
Main statistical parameters and diameter sizes of studied $BaFe_{12}O_{19}$ powder particles

No.	Powder size	Value [µm]
1.	Arithmetic mean diameter	10,335
2.	Geometric mean diameter	8,538
3.	Standard deviation	3,215
4.	Mean square deviation	4,571
5.	Mode	10,564
6.	Median	9,415

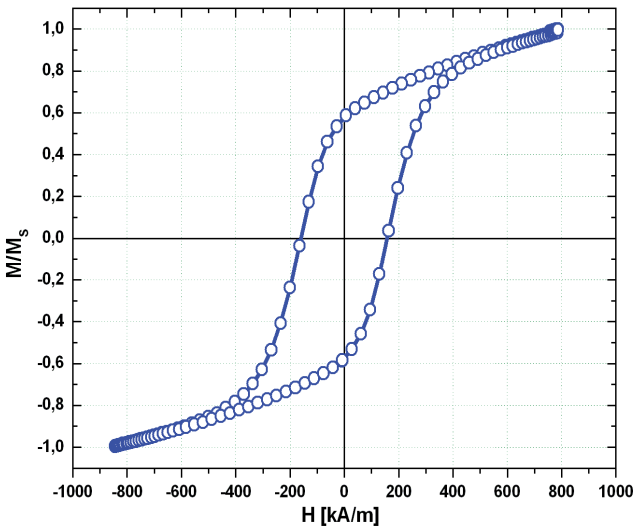

Fig. 4. Hysteresis loop of studied barium ferrite powder

Figure 5 shows the morphology of $BaFe_{12}O_{19}$ powder and observed by the scanning electron microscope. The morphology images of barium ferrite powder confirm the results determined by a laser particle analyzer and showed that studied powder particles have irregular shape and size.

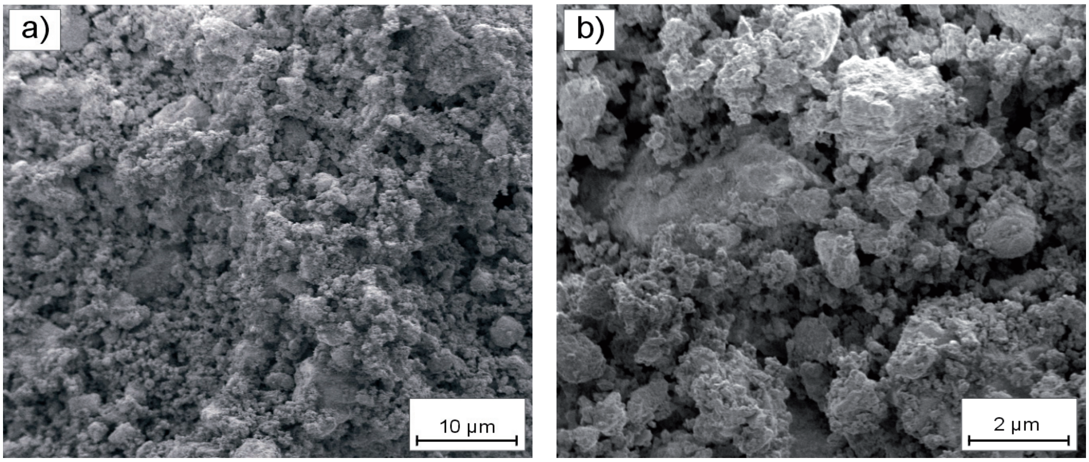

Fig. 5. SEM images of $BaFe_{12}O_{19}$ powder as-prepared samples

4. Conclusions

The investigations performed on the samples of barium ferrite powder allowed to formulate the following statements:

- the lattice parameters of the barium ferrite powder are a_0 = 0.58910(9) nm, c_0 = 2.3213(2) nm,
- the crystallite size of $BaFe_{12}O_{19}$ phase is above 100 nm and the lattice strain (<*Δa/a*>) is 0.033%,
- a good agreement of lattice parameters determined by Rietveld refinement method and from ICDD files was obtained for all involved phases,
- the coercive force (H_C) of studied powder has a value about 159 kA/m,
- the morphology images showed that powder particles are of irregular shapes and sizes,
- the arithmetic mean diameter of $BaFe_{12}O_{19}$ powders population is about 10.335 μm.

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