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Microstructure of composite material with powders of barium ferrite

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Materials

ABSTRACT

Purpose: The aim of the present work is the microstructure characterization of commercial powder $BaFe_{12}O_{19}$ (as-prepared) and composite material with $BaFe_{12}O_{19}$ powders and polymer matrix, using XRD (X-Ray Diffraction) and SEM (Scanning Electron Microscopy) methods.
Design/methodology/approach: The morphology of barium ferrite powders and a fracture surface of the examined composite material was realized by using the scanning electron microscope. The methods of X-ray diffraction were used for the qualitative phase analysis. The parameters of diffraction line profiles were determined by PRO-FIT Toraya procedure.
Findings: The X-ray diffraction analysis permitted on identification the $BaFe_{12}O_{19}$ and Fe_2O_3 phases in an examined material. Basing on Toraya method is determination of: lattice parameters, crystallite size (D) and the lattice distortion ($<\Delta a/a>$). Distribution of powders of barium ferrite in polymer matrix is irregular and powder particles have irregular shapes and dimensions.
Research limitations/implications: For future research the X-ray analysis should be performed by the Rietveld method, which allows to characterization the microstructure of tested material and verification of its qualitative phase composition.
Originality/value: The applied Toraya method of structure analysis appeared to be very useful in the microstructure analysis.
Keywords: Composites; X-ray phase analysis; Toraya procedure; Barium ferrite

1. Introduction

Composite materials are practically used in all branches of industry. Ferrite-based materials are the most widely used as magnetic materials. They have excellent chemical stability and are relatively cheap to produce. Ferrite magnets such as barium ferrites have applications as accumulating and processing of information [1-7].

Barium ferrites are sintered from barium carbonates and iron oxides. They was produced by methods of powders metallurgy. First stage of process of receiving the barium ferrites is called as ferritization. The aim of that process is producing magnetic materials – ($BaFe_{12}O_{19}$), which grains are milled and oriented during forming in magnetic field [3,8].

Preparation process of barium ferrite includes a stage of mixing barium carbonates and iron oxides and also annealing of the mixture in temperature of 1350°C. At the temperature of 600°C, it begins process of dissociation $BaCO_3$, what in presence of Fe_2O_3 leads to rise the monoferrite $BaFe_2O_4$. Afterwards the monoferrite reacts with Fe_2O_3 and creates at last a compound of $BaFe_{12}O_{19}$. Prepared in this way ferrite is grinded in ball mills to reach size of grain about 1 μm, pressed to form and sintered in temperature of 1200°C [3,4,9].

Barium ferrites are used to produce elastic magnets with polymer matrix. That type of materials is produced by mixing polymer material with powders of ferrite and then rolling to reach a form of a ribbon or a foil. Magnetic properties of that magnets depend on a content of ferrite powders in polymer matrix. They also have low weight, cost, good corrosion resistance and they are relatively cheap [4,5,10].

2. Material and research methodology

The aim of the present work is the microstructure characterization of commercial powder $BaFe_{12}O_{19}$ (as-prepared) and composite with $BaFe_{12}O_{19}$ powders and polymer matrix, using XRD (X-Ray Diffraction) and SEM (Scanning Electron Microscopy) methods. In this work is used a Toraya method for a structure determination of commercial materials – barium iron oxide. Basing on above methods is determination of: lattice parameters, crystallite size (*D*) and the lattice distortion ($<\Delta a/a>$).

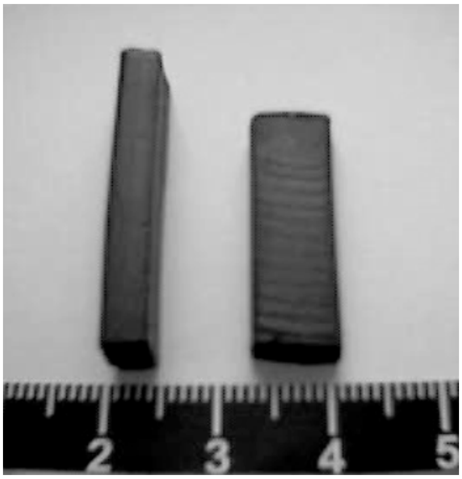

Fig.1 External morphology of the tested composite material with powders of barium ferrite in a cuboid form (commercial material)

The investigations were realized on samples of commercial composite with polymer matrix (polyvinyl chloride), which contains powders of barium ferrite. The tested material has a cuboid form about thickness 4 mm and width 7 mm (Fig. 1).

Phase analysis was carried out by 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 was recorded by "step-scanning" method with the range of 10° to 140° 2θ 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 [11]. The Toraya procedure is a usefull tool for microstructure characterization, so it was used for multicomponent powder analysis [12-14].

For materials with metallic phases it may be assumed that the X-ray diffraction lines are broadened mainly due to (a) instrumental effect, (b) small crystallite size, and (c) lattice distortion. The NIST SRM660a (LaB_6 powder) standard was used for instrumental broadening determination. The crystallite sizes and lattice distortions of $BaFe_{12}O_{19}$ phase were estimated using Williamson-Hall method [15].

The values lattice parameters calculated by the classical method applying the extrapolation of the Nelson-Riley function. The accuracy of lattice parameter determination found using alumina plate SRM 1976 standard is ± 0.02%.

The content of the barium ferrite powders in tested composite material is 90,6 wt.% and 65,2 vol.%. The values of mass and volumetric contents are presented in Table 1.

Table 1.
Mass, volumetric contents of powders bounded with polyvinyl chloride in tested composite

No.	Characteristic	Phase	Unit	Value
1.	Mass contents	$BaFe_{12}O_{19}$ + Fe_2O_3	wt.%	90,6
		PVC	wt.%	9,4
2.	Volumetric contents	$BaFe_{12}O_{19}$ + Fe_2O_3	vol.%	65,2
		PVC	vol.%	34,8

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 100x – 5000x with the ISIS software for the computer recording of images.

3. Results and discussion

The X-ray diffraction investigations permitted on identification the $BaFe_{12}O_{19}$ (ICDD PDF 43-0002) and the Fe_2O_3 (ICDD PDF 85-0987) phases in tested composite (Fig. 2).

The values of lattice parameters, their accuracy and these found in ICDD files for $BaFe_{12}O_{19}$ phase are also given in Table 2. The crystallite size (*D*) of $BaFe_{12}O_{19}$ phase is above 100 nm and the lattice strain ($<\Delta a/a>$) is 0.033%.

Table 2.
Phase lattice parameters for powder of barium ferrite

Phase	Lattice parameters [nm]	
	Toraya	ICDD file
$BaFe_{12}O_{19}$	a_0 = 0.5889(1) c_0 = 2.321(4)	a_0 = 0.58920(1) c_0 = 2.3183(1)

Comparison of received lattice parameters of examined phase ($BaFe_{12}O_{19}$) and lattice parameters from the ICDD card files for model materials allows to formulate following statement. The elementary cell is elongated in a direction of Z axis and shortened in a direction of X and Y axis.

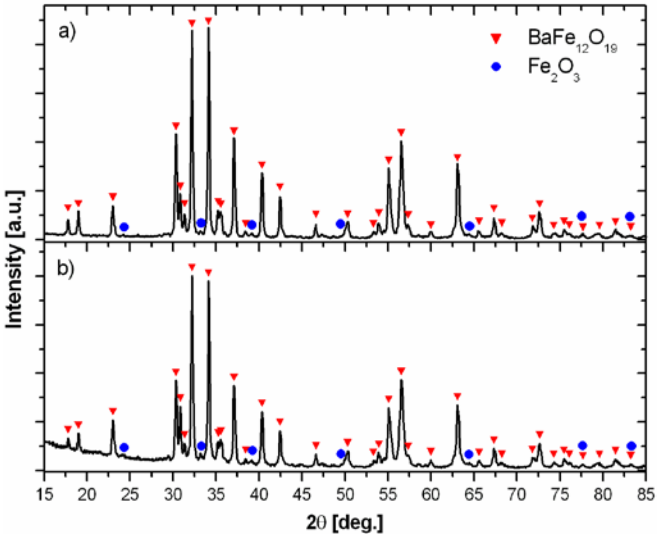

Fig.2 XRD pattern of the tested material: a) powder sample, b) composite sample with polymer matrix

That results might be happened by modification of atoms position in investigated elementary cell. That fact will be analyse by using the Rietveld method and present in the next papers.

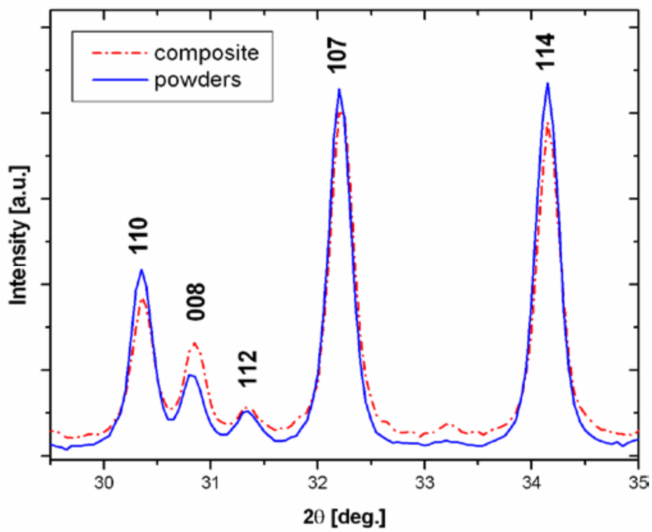

Fig.3 Comparison of XRD patterns for powders of barium ferrite and a composite material with powders of $BaFe_{12}O_{19}$

Comparison of fragment of XRD patterns for powders of barium ferrite and the composite material is presented in Figure 3. Difference in the intensity of (008) (2θ = 30.830) diffraction line for composite and powders induced axial texture on direction [001] in composite material.

Technological process of forming the tested composite material by mixing polymer material with powders of barium ferrite and next by rolling to form of a ribbon caused a formation of the axial texture. The intensity change of the other diffraction lines is not observed.

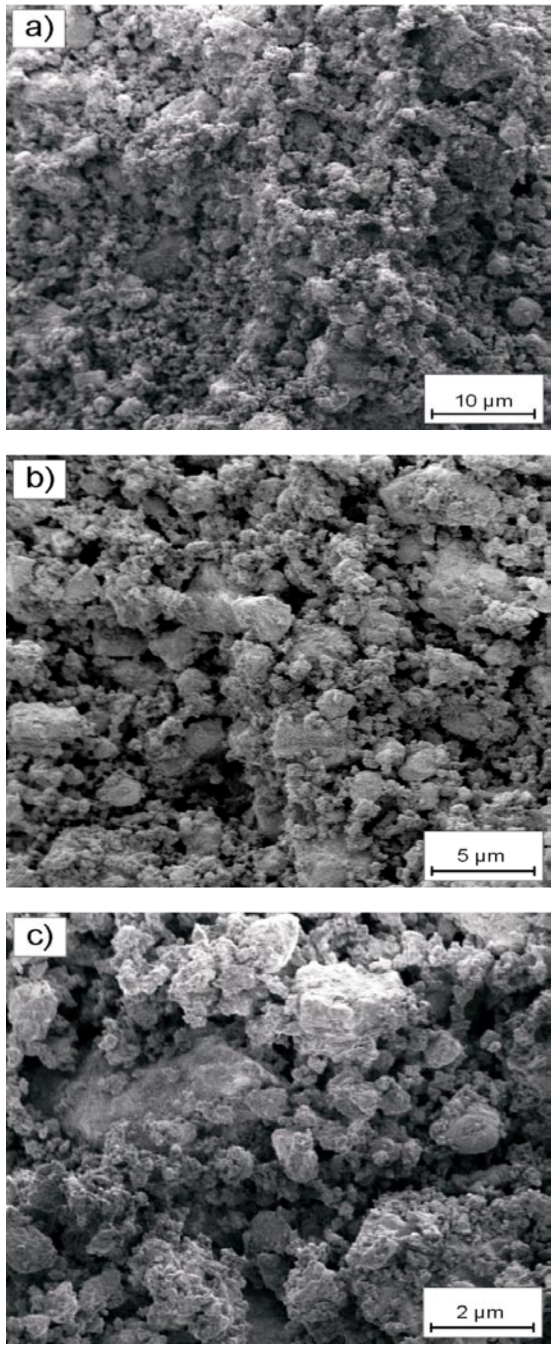

Fig.4 Morphology of a barium ferrite ($BaFe_{12}O_{19}$) powders used as a component in the investigated composite material

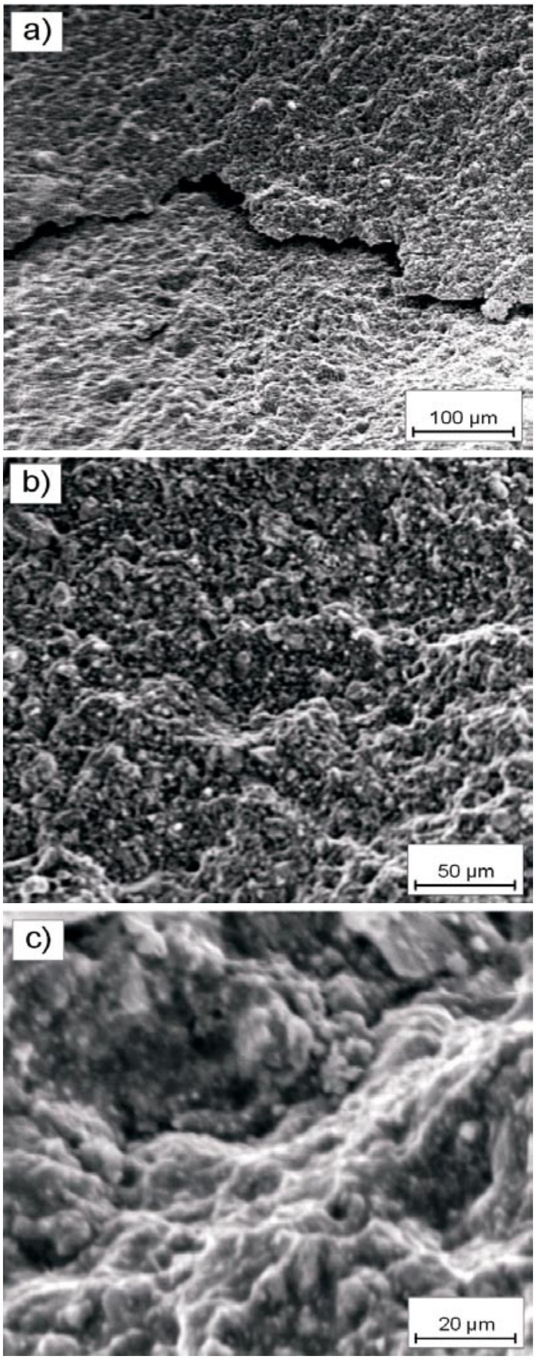

Fig.5 Morphology of a fracture surface of the composite material

Figure 4 shows the morphology of $BaFe_{12}O_{19}$ powder observed by the scanning electron microscope. Moreover, the morphology of a fracture of the tested composite is presented in the Figure 5. During metallographic observation of morphology of powders and fracture's surfaces of the composite's samples it was observed that the distribution of powders of barium ferrite in matrix is irregular. Powder particles have irregular shape and size.

4. Conclusions

The investigations of the barium ferrite powders and tested composite material allowed to formulate the following statements:

- analysis of the XRD pattern revealed the presence of two phases: $BaFe_{12}O_{19}$ and Fe_2O_3;
- the lattice parameters are a_0=0.5889(1) nm, c_0=2.321(4) nm;
- the crystallite size of $BaFe_{12}O_{19}$ phase is above 100 nm and the lattice strain ($\langle \Delta a/a \rangle$) is 0.033%;
- the SEM images showed that distribution of powders of barium ferrite in polymer matrix is irregular and powder particles have irregular shapes and sizes.

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