Synthesis and X-Ray Analysis of Complex Ferrites

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Abstract: Ferrite with mixed composition $YbBiNaFeO_5$ was synthesized by high temperature solid state reaction. There were made complex studies including thermal analysis, X-ray powder analysis, and electron microscopy. The structure of the ferrites, type of syngony, parameters of the unit cells, radiographic and pycnometric densities were determined by X-ray phase analysis for a first time: a = 10,81 Å, $V_{un.cell}$ = 1263,2 Å^3, ρ_{rad} = 9,31 g/cm^3, ρ_{picn} = 9,33 g/cm^3. It is determined that synthesized phase crystallizes in cubic syngony. A comparative analysis of the relationship between crystal lattice parameters with parameters of the crystal lattice of initial oxides and complex ferrites has been performed.

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1. Introduction

Semiconductor materials, which functionality is stipulated by electron charge are generally used in modern electronics. Increasing requirements to electronic devices expose the problem of search and implementation of alternative materials, working on non-classical principles. The basis of future electronic can become spintronics devices that use both electron charge and its spin. [1].

Spintronics became known after opening the effect of discovery of "giant" magnetoresistance (GMR), which is stipulated by different scattering on ferromagnetic impurities of two groups of electrons' spins with "up" and "down". This selection requires the significant difference between the average lengths of free path of the electrons with "up" and "down" directions of the spins. This occurs in ferromagnetic materials characterizing by differences in the density of the free states of electrons caused by exchange splitting of 3d-zone. This principle lays in the basis of magnetoresistive devices implementing the effects of the giant and tunnel magnetoresistance [2].

Literature data analysis shows that ortoferrites $BiFeO_3$ or so-called multiferroics are the most studied of ferrities that simultaneously have the electric polarization and magnetic ordering. The ortoferrites are prospective for implementation as the working environment in the data storage and processing devices. At the present time, the search of new materials with ferroelectric properties and specific electronic and magnetic structures is performed using the most famous multiferroic for. Substituted perovskites based on the bismuth ferrites often combine ferroelectric and weak ferromagnetic properties with the dominant antiferromagnetic ordering [3, 4].

This study is to investigate the conditions for obtaining and X-ray features of new classes of complex mixed bismuth ferrites, in which Bi^{+3} is partially substituted by the ions of rare-earth elements and sodium.

2. Materials and methods. New polycrystalline complex of bismuth ferrites were synthesized by ceramic processing technology. Bismuth oxide (III) (mark "chemically pure"), sodium carbonate (mark "highly-pure"), ytterbium oxide (mark "highly-pure"), and iron oxide (III) (mark "chemically pure") were used as the initial components. Solid-phase synthesis was carried using the thermal data of the initial components taking into account Tamman conditions for ceramic reactions [5, 6]. Stoichiometrically calculated mixtures of initial components preliminary annealed in a muffle furnace at 400ºC for one hour, were thoroughly stirred and the calculated mixture of the starting components are thoroughly mixed and grinded in an agate mortar, placed in an alundum crucibles, and annealed in a silite furnace. Annealing was performed by two stages: first stage at 600ºC for 48 h and second stage at 800ºC for 20 h [7].

Formation of new phases was controlled by X-ray scattering analysis performed using the radiologic diffractometer X'Pert MPD PRO (PANalytical). The conditions of analysis included: CuK_a – radiation, Ni – filter, U=30 kV, I=10 mA, rotation rate – 1000 impulses/s, and time constant τ = 5 s, $2\theta = 10° – 90°$. Diffraction peaks were evaluated using hundred-point scale. Radiographs of the

synthesized polycrystalline powders were indexed by the homology method (homologue is distorted structure type of perovskite) [8]. Pycnometric density of manganites was determined by the method described in [9]. Toluene served as indifferent liquid. The density of each ferrite was measured 4 – 5 times and the data were averaged. Table below shows the results of indexing of radiographs of ferrites.

3. Results and discussion. New polycrystalline complex of bismuth ferrites were synthesized by ceramic processing technology. Bismuth oxide (III) (mark "chemically pure"), sodium carbonate (mark "highly-pure"), ytterbium oxide (mark "highly-pure"), and iron oxide (III) (mark "chemically pure") were used as the initial components. Solid-phase synthesis was carried using the thermal data of the initial components taking into account Tamman conditions for ceramic reactions [5, 6]. Stoichiometrically calculated mixtures of initial components preliminary annealed in a muffle furnace at 400ºC for one hour, were thoroughly stirred and the calculated mixture of the starting components are thoroughly mixed and grinded in an agate mortar, placed in an alundum crucibles, and annealed in a silite furnace. Annealing was performed by two stages: first stage at 600ºC for 48 h and second stage at 800ºC for 20 h [7].

After x-ray scattering of synthesized phases, the morphological peculiarities of ceramic surface were investigated in the contact mode of the electron microscope JED-2300 (JEOL, Japan). The microstructure of polycrystalline phase of $YbBiNaFeO_5$ is shown in Fig.1.

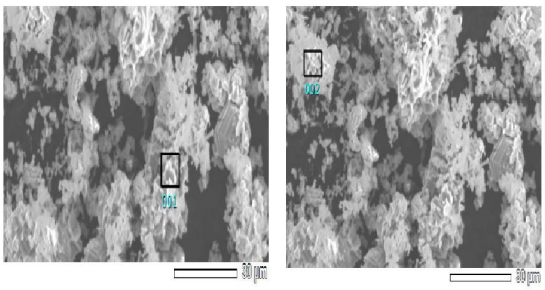

Fig. 1. Microdiffraction pattern of $YbBiNaFeO_5$

The experiments revealed the different morphological structures in the electron-microscopic images of complex bismuthites. The morphological structures were conditionally classified according to morphological features and crystal form of particles was determined as porous structures of rounded shape.

Quantitative results of Specific x-ray spectra of electron transitions at the internal levels of atoms between neighboring electron orbits along directions of the layers (001) are shown in Table 1.

Elements	kV	Mass	Error	Atom	Elements	kV	Mass	Error	Atom
C	0,277	4,93	0,15	12,82	C	0,277	5,53	0,26	12,72
O	0,525	18,37	0,13	35,86	O	0,525	25,43	0,23	43,90
Na	1,041	10,11	0,19	13,73	Na	1,041	12,31	0,32	14,78
Yb	3,690	0,23	0,25	0,18	Yb	3,690	0,82	0,44	0,56
Bi	5,894	34,70	0,68	19,72	Bi	5,894	46,95	1,20	23,60
Fe	6,398	31,66	0,75	17,70	Fe	6,398	8,97	1,33	4,43

Table 1. Quantitative results

Phase analysis of X-ray pattern of the sample has showed the individuality of each line of reflection. Line intensity was assessed using a hundred-point scale of most intensive reflection line. Indexing of x-ray pattern of $YbBiNaFeO_5$ powder was carried out by the homology as distortion of the original structure of δ–Bi_2O_3.

№	h	k	l	d [A]	2Theta[deg]	I [%]
1	1	1	0	7,20080	12,282	0,1
2	2	0	0	5,09180	17,403	1,6
3	2	1	1	4,15740	21,355	2,6
4	2	2	0	3,60040	24,708	18,4
5	3	1	0	3,22030	27,679	100,0
6	2	2	2	2,93970	30,382	25,2
7	3	2	1	2,72160	32,882	65,3
8	4	0	0	2,54590	35,223	2,9
9	3	3	0	2,40030	37,437	4,7
10	0	2	4	2,27710	39,544	6,2
11	3	3	2	2,17110	41,562	9,9
12	4	2	2	2,07870	43,501	9,5
13	1	3	4	1,99720	45,373	13,6
14	5	2	1	1,85920	48,953	4,5
15	4	4	0	1,80020	50,668	0,1
16	0	3	5	1,74650	52,342	29,4
17	6	0	0	1,69720	53,984	14,8
18	5	3	2	1,65200	55,587	21,5
19	6	2	0	1,61020	57,160	0,3
20	5	4	1	1,55714	58,707	2,0
21	6	2	2	1,53520	60,233	1,9
22	6	3	1	1,50150	61,730	14,4
23	4	4	4	1,46990	63,209	1,3
24	3	4	5	1,44020	64,668	6,7
25	0	4	6	1,41220	66,112	1,4
26	7	2	1	1,38580	67,539	3,3
27	2	4	6	1,36080	68,952	1,4
28	0	3	7	1,33720	70,347	1,0
29	6	5	1	1,29330	73,112	2,8
30	8	0	0	1,27290	74,480	0,3
31	1	4	7	1,25350	75,834	1,7
32	8	2	0	1,23490	77,185	0,7
33	3	5	6	1,21720	78,521	8,8
34	6	6	0	1,20010	79,862	8,2
35	1	3	8	1,18380	81,189	6,8
36	6	6	6	1,16810	82,515	0,3
37	2	5	7	1,15300	83,839	1,7
38	8	4	0	1,13860	85,147	0,6
39	9	1	0	1,12460	86,464	1,6
40	8	4	2	1,11110	87,780	2,4

Table 2. Indexing of radiographs of $YbBiNaFeO_5$ powder

Synthesized phase is crystallized in cubic space-centered lattice. The reliability of the results confirms the even sums of Miller indices (hkl) and the satisfactory coincidences of the experimental and theoretical values of the inverse squares of the interplanar distances. The unit cell parameter a is 10,81 Å if the meaning of explicit units is 1 that is confirmed by the satisfactory correlation of the values of the x-ray (9,31 g/cm^3) and pycnometric (9,332 g/cm3) densities.

The consistency of the results of indexing of the X-ray pattern for ferrite is confirmed by the

coherence of values of X-ray and pycnometric densities.

4. Conclusions. $YbBiNaFeO_5$ is crystallized in cubic space-centered syngony. The analysis of the relation between the parameters of crystal lattice of the original δ-Bi_2O_3 shows the increase in the value of the parameters by two times of previously synthesized ferrites $BiCaFe_2O_{5,5}$ and $BiSrFe_2O_{5,5}$ crystallized in the cubic crystalline lattice. There was an increase of parameter *a* compared with the initial crystalline lattice of δ-Bi_2O_3 from 5,68 to 11,1 Å [10, 11]. Close values of the parameters of unit cell proves the isostructure of complex ferrites.

Thus, complex oxides $YbBiNaFeO_5$ were synthesized for the first time. The types of syngony and parameters of unit cells were determined by X-ray scattering analysis.

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