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CRYSTAL STRUCTURE, MAGNETIC AND ELECTRICAL PROPERTIES AND THERMAL EXPANSION OF FERRITES OF THE SYSTEM $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ (x = 0–0.5)

High-coercivity ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ (x = 0–0.5) with magnetoplumbite structure were prepared from oxides Fe_2O_3, Sm_2O_3, ZnO and carbonate $SrCO_3$ by solid-state ceramic method, the dependence of the unit cell parameters a and c on the value of x was determined. It was determined that samples of $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ were single-phased up to $x = 0.2$, and also contained α-Fe_2O_3 for $x \geq 0.3$ phase, quantity of which gradually increased with increasing x up to 0.5, and small quantities of phases $ZnFe_2O_4$ and $SmFeO_3$ were present in the samples with $x = 0.4$ and 0.5. The magnetic, electrical properties and thermal expansion of these ferrite samples were studied, the values of specific saturation magnetization (σ_s) were determined by magnetic hysteresis loops at 5 and 300 K. It was found that the solid solution $Sr_{0.9}Sm_{0.1}Fe_{11.9}Zn_{0.1}O_{19}$ at 300 K has specific saturation magnetization (σ_s) and coercive force ($_\sigma H_c$) respectively by 0.4 and 9.7% higher than the base ferrite $SrFe_{12}O_{19}$.

Introduction Strontium ferrite $SrFe_{12}O_{19}$ with magnetoplumbite structure is a hard-magnetic material, which is used for the manufacture of ceramic permanent magnets widely used in various fields of science and technology [1]. Since the 90th of the last century and hitherto the study of solid solutions based on $SrFe_{12}O_{19}$ in which ions $Sr^{2+}$are partially replaced by ions of rare earth element Ln^{3+} (Ln – La, Nd, Pr, Sm), and an equivalent amount of ions Fe^{3+} is substituted with ions M^{2+} (M – Zn, Co, Mn, Cu) is considered to be a promising direction to search for new hard-magnetic materials. In work [2] it has been found that in the system $Sr_{1-x}La_xFe_{12-x}Zn_xO_{19}$ the increasing of substitution degree x up to 0.3 leads to a gradual increase of magnetization and to coercive force reduction, and permanent anisotropic magnet made from solid solution $Sr_{0.7}La_{0.3}Fe_{11.7}Zn_{0.3}O_{19}$ has a value of energy product $(BH)_{m.x}$ = 41 кJ/m^3.In work [3] it is shown that in the system $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$ the partial substitution of strontium ions Sr^{2+} with ions La^{3+} and ions Fe^{3+} with ions Co^{2+} to $x = 0{,}2$ causes a decrease of magnetization, but at the same time there is an increase of the anisotropy field, which allows to produce anisotropic permanent magnets with magnitude $(BH)_{max}$ = 38,4 кJ/m^3 from the solid solution $Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O_{19}$.

In recent years a series of works was published in which the crystal structure, Messbauer spectra and to a lesser extent the magnetic properties of ferrites of systems $Sr_{1-x}Ln_xFe_{12-x}M_xO_{19}$ (Ln – Nd, Pr, Sm; M – Zn, Co) were investigated. [4–6]. The synthesis of the ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ (x = 0; 0.1; 0.2; 0.3; 0.4; 0.5) with magnetoplumbite structure was carried out, their crystal structure, the saturation magnetization, coercive force, DC conductivity and thermal expansion were studied in this paper.

Experimental technique. Ceramic samples of ferrites of the system $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ (x = 0; 0.1; 0.2; 0.3; 0.4; 0.5) were synthesized by solid-phase method from samarium oxides (Sm_2O_3)), iron (Fe_2O_3), zinc (ZnO), strontium carbonate ($SrCO_3$). All reagents were of analytical grade qualification. Before weighing samarium oxide had been calcined at the 1273 K for 3 h. Stirring and milling of basic materials, taken in the necessary ratio was carried out in the planetary ball mill Puluerizette 6 of Fritsch company with the addition of ethanol. The resulting batch (with the addition of ethanol for mold ability upgrading) were compressed under pressure of 50-75 MPa into tablets with a diameter of 19 mm and a height of 5-7 mm, which were then dried at 373 K for 4 hours and fired on substrates from alumina at a temperature of 1523 K in air. After prebaking tablets were crushed, milled, compressed to form tablets of 9 mm diameter, 2-3 mm in height and parallelepipeds of the size 5×5×30 mm^3, which were calcined at 1523 K for four hours in air.

X-ray diffractograms of ferrite samples were recorded on a X-ray diffractometer Bruker D8 (radiation CuK α) at room temperature. Unit cell parameters (a and c) of magnetoplumbite hexagonal structure were calculated using the full-profile analysis by the Rietveld method (program FullProf). Dependences of the specific magnetization on the temperature and the magnitude of ferrite magnetic field $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ were obtained in the Scientific and Practical Center of NASB for Material sciences. The specific magnetization (σ sp) of ferrite powders in a magnetic field of 8.6 kOe was measured by the Faraday method in the temperature range 77–900 K. The specific saturation magnetization and hysteresis loop parameters of specific magnetization of cylindrical shape

ferrite samples 5.0-5.4 mm long and 1.0-1.2 mm in diameter were determined by the oscillation method in a magnetic field up to 14 T at temperatures of 5 and 300 K. The samples conductivity was measured at a constant current in air in the temperature range 300-1100 K. The thermal expansion of the ceramic samples was investigated by the dilatometric method using a quartz dilatometer with a dial indicator in the temperature range 300–1100 K.

Results and their discussion. Analysis of X-ray diffraction patterns (Fig. 1) showed that the obtained samples of ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ were single phase ones. On X-ray diffraction patterns of ferrite samples with x ≥ 0.3, except for the X-ray lines belonging to the magnetoplumbite hexagonal structure, were also the most intense lines of α-Fe_2O_3 and $SmFeO_3$. On X-ray diffraction patterns of ferrite samples with x = 0.4 and 0.5, along with the lines of impurity phase α-Fe_2O_3, $SmFeO_3$, was the line $ZnFe_2O_4$, and in the sample with x = 0.5 – the hardly noticeable line Sm_2O_3.

Table 1 shows the values of the crystal lattice parameters *a, c* and of volume *V* of unit cell ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$.

It is obvious that increasing of the substitution degree *x* 0.3 leads to a gradual increase in the crystal lattice parameter *a* from the value of 5.8791 Å for $SrFe_{12}O_{19}$ to 5.8823 Å for the sample with *x* = 0.3 (Fig. 2, curve 1).

The crystal lattice parameter *c* with increasing of substitution degree *x* from 0 to 0.3 is gradually reduced from the value 23.070 Å for $SrFe_{12}O_{19}$ to 22.997 Å for the sample with *x* = 0.3, and with a further increase in the substitution degree *x* to 0.5 it increases (Fig. 2, curve 2).

The data analysis presented in Table 1 and in Fig. 2 shows that the maximum substitution degree of ions Sr^{2+}, Fe^{3+} in the ferrite $SrFe_{12}O_{19}$ with ions Sm^{3+}, Zn^{2+} at the temperature of 1523 K to form solid solutions $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ corresponds to the value *x* ≈ 0.3.

For basic strontium ferrite $SrFe_{12}O_{19}$ the parameters *a* and *c* defined in the present study (Table 1), are consistent with the values of *a* and *c* (5.8844 and 23.05 Å) for $SrFe_{12}O_{19}$, given in [2].

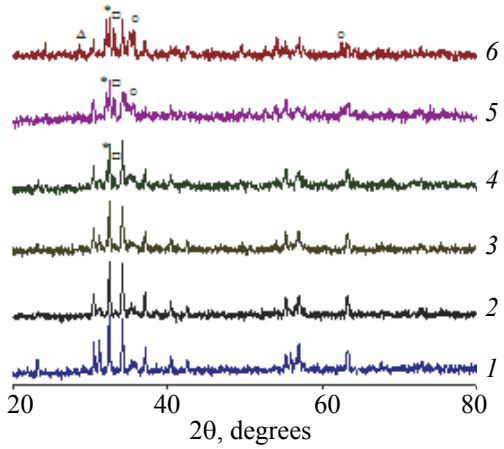

Fig. 1. X-ray diffractograms of ferrite samples of system $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ at *x*: *1* – 0; *2* – 0.1; *3* – 0.2; *4* – 0.3; *5* – 0.4; *6* – 0.5; Δ – Sm_2O_3; * – $SmFeO_3$; □ – α-Fe_2O_3; ○ – $ZnFe_2O_4$

Table. 1 shows the values of X-ray sructural density, found by the formula:

$$\rho_{xray} = \frac{2M}{N_A V}, \quad (1)$$

where *M* - molar mass of ferrite; N_A - Avogadro number, as well as apparent density calculated using the weight and volume of samples, and the relative density calculated by the following formula:

$$\rho_{rel} = \frac{\rho_{app}}{\rho_{xray}}, \quad (2)$$

for the samples of ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ with the substitution degree *x* ≤ 0.2.

It is evident that in the single-phase X-ray structural density rises with increasing of substitution degree *x*, and the apparent and relative densities decrease. This indicates the deterioration of the sinterability of the samples with increasing of substitution degree *x*.

Fig. 3 shows that the majority of crystallites ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ (0 < x ≤ 0.5) does not have a clear-cut faceting, and their size is 2-5 μm. However the photo of ferrite $SrFe_{12}O_{19}$ (Fig. 3*a*) shows that it contains individual crystallites that are larger than 10 μm.

Table 1

Values of the parameters *a, c* and *V* of the crystal lattice ferrites $Sr1_{-x}Sm_xFe_{12-x}Zn_xO_{19}$ (0 ≤ *x* ≤ 0,5) with magnetoplumbite structure. X-ray structural (ρ Xray) apparent (ρ app) and relative density (ρ rel) of solid solutions of ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ (0 ≤ *x* ≤ 0,2)

x	*a*, Å	*c*, Å	*V*, Å^3	ρ_{xray}, g/cm^3	ρ_{app} g/cm^3	ρ_{rel}, %
0	5.8791	23.070	690.544	5.103	4.093	80.21
0.1	5.8805	23.055	690.431	5.142	4.028	78.34
0.2	5.8814	23.029	689.873	5.186	3.901	75.23
0.3	5.8823	22.997	689.128	–	–	–
0.4	5.8773	23.057	689.738	–	–	–
0.5	5.8749	23.048	688.915	–	–	–

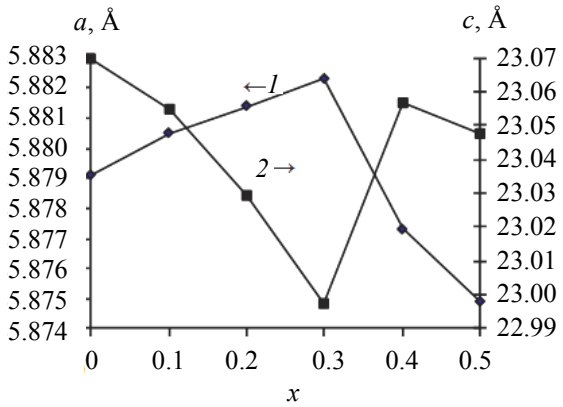

Fig 2. The parameters dependences of crystal lattice *a* (*1*) and *c* (*2*) of solid solutions of ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ (x = 0–0.5) on the substitution degree x

Fig. 4 shows the temperature dependences of the specific magnetization measured in the magnetic field of 8.6 kOe for ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ ($0 \le x \le 0.5$), due to which the Curie temperatures (T_c), are defined for these ferrites and listed in Table. 2.

Table 2

Curie temperature (T_c), specific magnetization at temperatures 77 and 298 K (σ_{77}, σ_{298}) of ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$

x	T_c, K	σ_{77}, G · cm^3/g	σ_{298}, G · cm^3/g
0	729	84.79	61.30
0.1	724	84.84	62.06
0.2	720	75.04	55.16
0.3	718	67.46	49.11
0.4	715	56.31	41.00
0.5	712	48.76	33.89

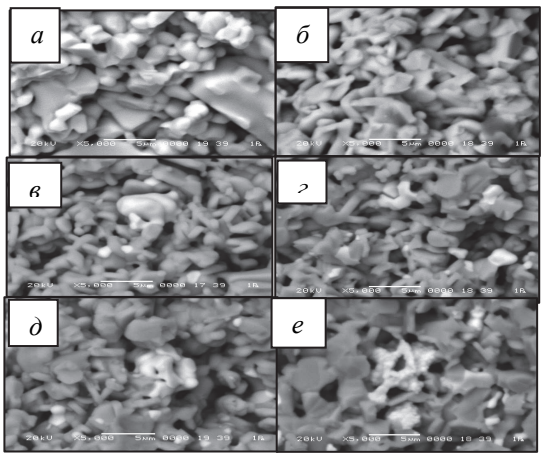

Fig. 3. Electron microscopic pictures of ferrites of system $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ at x: *a*- 0, *b* - 0.1, *c* - 0.2, *d* - 0.3, e- 0.4, f - 0.5 (× 5000 times)

The values of specific magnetization at temperatures of 77 K (σ_{77}) and 298 K (σ_{298}) are indicated in the same table. It should be noted that the specific magnetization σ_{77}, σ_{298} for a solid solution with x = 0.1 is slightly higher than for the ferrite $SrFe_{12}O_{19}$, but at further increase of x from 0.2 to 0.5 the values σ_{77}, σ_{298} drastically reduce.

This is probably due to the fact that the ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ with $0.3 \le x \le 0.5$ contain nonmagnetic phases α-Fe_2O_3, $ZnFe_2O_4$ and the phase of submagnetic samarium ferrite of a $SmFeO_3$.

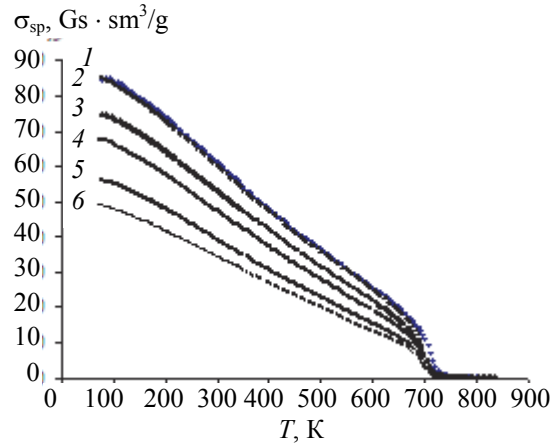

Fig. 4. Temperature dependences of the specific magnetization σ s of ferrite samples of the system $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ at x: 1 – 0; 2 - 0.1; 3 - 0.2; 4 - 0.3; 5 - 0.4; 6 - 0.5

These data suggest that increasing the substitution degree x leads to a gradual reduction of the Curie temperature from 729 K for $SrFe_{12}O_{19}$ to 712 K for the sample $Sr_{0.5}Sm_{0.5}Fe_{11.5}Zn_{0.5}O_{19}$.

Fig. 5 shows as an example the hysteresis loop magnetization for $SrFe_{12}O_{19}$ at temperatures 5 and 300 K. It is obvious that the saturation magnetization of this ferrite is achieved in the fields of about 1 T (10 kOe), above which there is a slight increase in the anhysteretic magnetization due to paraprocess.

Similar hysteresis loops magnetization at temperatures of 5 and 300 K were obtained for all the investigated ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ ($0 < x \le 0.5$), and it allowed to identify the specific saturation magnetization (σ_s), the specific remanent magnetization (σ_r) and coercivity ($_\sigma H_f$).

The saturation magnetization (n_s), expressed in Bohr magnetons per formula unit of ferrite $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ with $x \le 0.2$ is calculated:

$$n_s = \frac{\sigma_s M}{5585}, \quad (3)$$

where M - molar mass of the respective ferrite; 5,585 - a quantity equal to the product of the Bohr magneton (μ_B) by Avogadro's number.

Using the quantities of specific remanent magnetization were calculated the values of remanent magnetization (n_r), expressed in Bohr magnetons. The obtained values of n_s, n_r, $_\sigma H_f$ for ferrites of the system $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ at 5 and 300 K are given in Table. 3.

Table 3

The specific saturation magnetization (σ_s), the saturation magnetization of one formula unit (n_s), the specific remanent magnetization (σ_r), coercive force ($_\sigma H_f$) of ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ at 5 and 300 K

x	T = 5 K						T = 300 K					
	σ_s, G · cm^3/g	n_s, μ_B	σ_r, G · cm^3/g	n_r, μ_B	$_\sigma H_f$, Oe	σ_r / σ_s	σ_s, G · cm^3/g	n_s, μ_B	σ_r, G · cm^3/g	n_r, μ_B	$_\sigma H_f$, Oe	σ_r / σ_s
0	97.93	18.62	24.36	4.63	653	0.25	64.71	12.30	27.31	5.19	1885	0.42
0.1	94.28	18.05	20.85	3.99	422	0.22	64.99	12.44	28.94	5.54	2068	0.45
0.2	97.62	18.81	20.20	3.89	414	0.21	66.75	12.86	27.61	5.32	1808	0.41
0.3	81.07	–	16.82	–	1418	0.21	54.69	–	21.81	–	2058	0.40
0.4	78.98	–	11.44	–	399	0.15	53.87	–	12.67	–	1000	0.29
0.5	59.49	–	12.99	–	560	0.22	39.86	–	15.19	–	2184	0.38

Analysis of the magnetic parameters of ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ shows that at 300 K the increase of the substitution degree x to 0.2 leads to an increase of the specific saturation magnetization (σ_s) from the value of 64.71 G · cm^3 / g for $SrFe_{12}O_{19}$ to 66.75 g · cm^3 / g for the solid solution with x = 0.2.

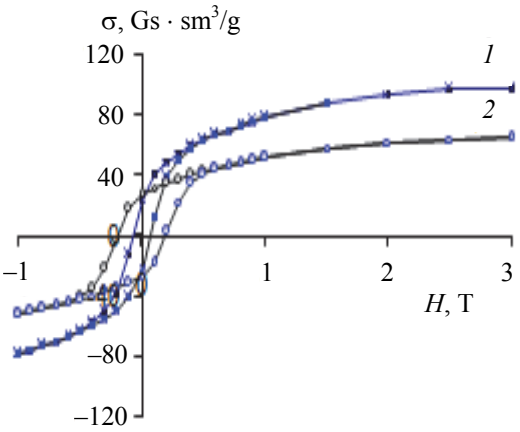

Fig. 5. Hysteresis loop of specific magnetization at temperatures 5 K (*1*) and 300 K (*2*) for $SrFe_{12}O_{19}$

Further increase of the substitution degree x from 0.2 to 0.5 causes a gradual decrease of the specific saturation magnetization (σ_s) to G · 39.86 cm^3 / g for the ferrite sample with x = 0.5.

From the data given in Table. 3, it follows that the specific saturation magnetization (σ_s) and coercive force ($_\sigma H_f$) of the solid solution sample $Sr_{0.9}Sm_{0.1}Fe_{11.9}Zn_{0.1}O_{19}$ at 300 K are 0.4 and 9.7%, greater respectively than the values of these parameters for the base ferrite $SrFe_{12}O_{19}$.

In Fig. 6 dependences of the specific conductivity (æ) on temperature T and ln æ from T-1 for ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ show that the electrical conductivity of all the investigated samples of ferrites rises with increasing temperature and is a semi-conductor one.

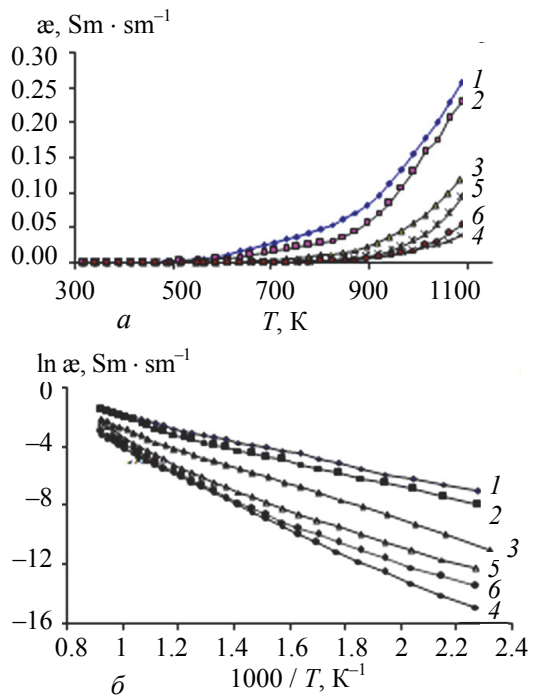

Fig. 6 Dependences of the specific conductivity (æ) on the temperature T (*a*) and ln æ from T-1 (b) of ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ at x: *1* – 0; *2* – 0.1; *3* – 0.2; *4* – 0.3; *5* – 0.4; *6* – 0.5

Increasing of the substitution degree x 0.3 leads to a decrease of the specific conductivity at the same temperatures, and at a further increase of x to 0.4, 0.5, its increase is observed. On linear dependences ln æ obtained for all of the studied ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ from T^{-1} a knee of curve is observed at a temperature T_k, which is about 100 K above the Curie temperature for the corresponding ferrite $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$.

In this case, as in the system $Sr_{1-x}La_xFe_{12-x}Co_xO_{19}$ [7], inclination of dependence lines ln æ from T^{-1} in the temperature range $T > T_k$ is greater than for temperatures $T < T_k$. This indicates a higher value of activation energy of ferrite conductivity $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ for temperatures $T > T_k$ (E_{A2}), than for temperatures $T < T_k$ (E_{A1}). The obtained results (Table 4) indicate that increasing of the substitution degree x of ions Sr^{2+} in the ferrite $SrFe_{12}O_{19}$ with ions $Sm^{3+}$and ions Fe^{3+} with ions Zn^{2+} from 0 to 0.3 leads to an increase of activation conductivity energy E_{A1}, E_{A2}: from the values of 0.32, 0.44 e V, respectively for the ferrite $SrFe_{12}O_{19}$ to the values 0.76, 0.82 eV for the solid solution $Sr_{0.7}Sm_{0.3}Fe_{11.7}Zn_{0.3}O_{19}$. The difference between E_{A2} and E_{A1} ($\Delta E = E_{A2} - E_{A1}$) varies slightly with increasing x from 0.12 eV for $SrFe_{12}O_{19}$ to 0.17; 0.13 eV for solid solutions $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ with $x = 0.1$; 0.2 respectively.

Table 4

Activation conductivity energy lower (E_{A1}) and above (E_{A2}) temperature Tk and the value $\Delta E = E_{A2} - E_{A1}$ for ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$

x	T_k, K	E_{A1}, eV	E_{A2}, eV	ΔE, eV
0	822	0.32	0.44	0.12
0.1	799	0.35	0.52	0.17
0.2	806	0.49	0.62	0.13
0.3	869	0.76	0.82	0.06
0.4	823	0.63	0.89	0.26
0.5	823	0.72	0.86	0.14

Dependences of elongation on the temperature (Fig. 7) for all ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ ($x = 0.1$–0.5) are practically linear, indicating the absence of other phase transitions in these samples in the test temperature range, except for the magnetic transition at T_c.

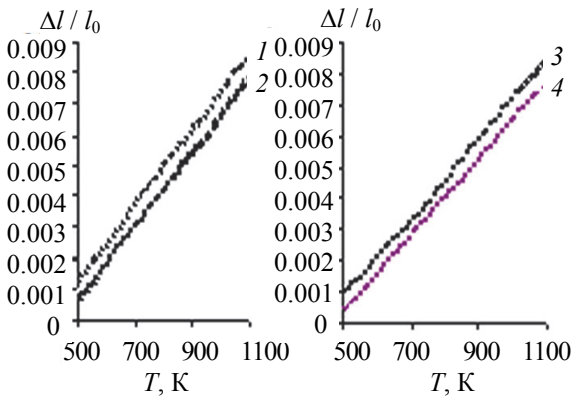

Fig.7 Temperature elongation $\Delta l / l_0$ dependences of ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ at x: *1* – 0.1; *2* – 0.2; *3* – 0.3; *4* – 0.4

Calculations showed that the linear thermal expansion coefficient (α) for ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ with increasing substitution degree of x slightly decreases: from $1.22 \cdot 10^{-5}$ K^{-1} for $SrFe_{12}O_{19}$ to $1.12 \cdot 10^{-5}$ K^{-1} for $Sr_{0.5}Sm_{0.5}Fe_{11.5}Zn_{0.5}O_{19}$.

Conclusion. The ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ ($x = 0$–0.5) were synthesized by solid-phase method in air at a temperature of 1523 K.

XRD showed that the samples with $x \le 0.2$ are single phase ones, and when $x \ge 0.3$ they contained the phase α-Fe_2O_3, the amount of which is gradually increased with growth of x to 0.5, in the samples with x = 0.4; 0.5 phases $ZnFe_2O_4$ and $SmFeO_3$ were present in a small amount.

It has been established that the increase of the substitution degree of x up to 0.3 increases a crystal lattice parameter a but reduces the crystal lattice parameter c.

It has been found that the Curie temperature of ferrite samples $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ ($x = 0$–0.5) gradually decreases with increasing of substitution degree x from 729 K for $SrFe_{12}O_{19}$ to 712 K for the sample $Sr_{0.5}Sm_{0.5}Fe_{11.5}Zn_{0.5}O_{19}$.

Specific magnetization measurements carried out at temperatures of 5 and 300 K in the magnetic field up to 14 T showed that the saturation magnetization of the ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ is achieved in the field about 1 T.

It has been found experimentally that the specific saturation magnetization (σ_s) and coercive force ($_\sigma H_f$) of the solid solution sample $Sr_{0.9}Sm_{0.1}Fe_{11.9}Zn_{0.1}O_{19}$ at 300 K are 0.4 and 9.7% greater respectively, than the values of these parameters for the base ferrite $SrFe_{12}O_{19}$.

The using of the ferrite $Sr_{0.9}Sm_{0.1}Fe_{11.9}Zn_{0.1}O_{19}$ proves to be promising for the manufacture of permanent magnets with high value of energy product $(BH)_{max}$

It is shown that the dependence of specific conductivity on the temperature for all the investigated ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ ($x = 0$–0.5) is a semiconductor one.

Activation conductivity energy, calculated using the linear dependence sections ln æ from 1 / T, in the temperature range above the knee temperature (T_k) is 0.06-0.26 eV higher than the activation conductivity energy at temperatures below T_k, which at about 100 K are higher the Curie temperature (T_c).

Linear thermal expansion coefficient (α) for the ferrites $Sr_{1-x}Sm_xFe_{12-x}Zn_xO_{19}$ slightly decreases with increasing substitution degree of x: from $1.22 \cdot 10^{-5}$ K^{-1} for $SrFe_{12}O_{19}$ to $1.12 \cdot 10^{-5}$ K^{-1} for $Sr_{0.5}Sm_{0.5}Fe_{11.5}Zn_{0.5}O_{19}$.

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