PHASE FORMATION IN THE FeSb₂S₄-FeLn₂S₄ SYSTEM, SYNTHESIS AND PROPERTIES OF COMPOUNDS OF THE FeLnSbS₄ (Ln=Nd, Er) TYPE

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Abstract: *Phase formation in the FeSb₂S₄-FeNd₂S₄ and FeSb₂S₄-FeEr₂S₄ systems were studied by means of differential thermal analysis, X-ray diffraction technique, microstructural analysis and microhardness measurement. Phase diagrams of the title systems have been constructed and the formation of a quaternary sulfosalt with the FeLnSbS₄ composition has been established. The compounds FeNdSbS₄ and FeErSbS₄ melt congruently at 960 and 1330 K, respectively, and form continuous substitutional solid solutions with FeSb₂S₄. Sulfosalts FeLnSbS₄ crystallize in the orthorhombic structure with following unit cell parameters: a=11.392, b=14.132, c=3.746Å, sp.gr. Pbam, z=4, density ρ=4.88 g/cm³ (for FeNdSbS₄); a=11.360, b=13.842, c=3.600 Å, sp. gr. Pbam z=4, ρ=5.17 g/cm³ (for FeErSbS₄). The FeLnSbS₄ type sulfosalts is classified under the FeSb₂S₄ berthierite structure.*
Key words: *system, phase diagram, sulfosalt, solid solutions, unit cell parameters, berthierite*
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Introduction

Obtaining and studying the fundamental physical properties of crystals with valuable, practically important characteristics is one of the main problems of modern science. The production of synthetic crystals largely determines the development of such important fields of technology as radio electronics, semiconductor and quantum electronics, technical optics, acoustics, etc. [1-5].

Here we study phase equilibria in the $FeSb_2S_4$-$FeNd_2S_4$ and $FeSb_2S_4$-$FeEr_2S_4$ quasiternary systems. The initial ternary sulphides are formed in the systems FeS - Sb_2S_3 and FeS - Ln_2S_3 (Ln = Nd, Er) quasibinary systems and have been studied in detail in the literature [6, 8]. The compounds $FeNd_2S_4$ and $FeEr_2S_4$ melt congruently and crystallize in the hexagonal syngony [1, 8].

The $FeSb_2S_4$ compound occurs in nature in the form of the mineral berthierite [9], and crystallizes in the orthorhombic structure with lattice parameters $a = 11.44$, $b = 14.12$, $c = 4.76$ Å, $z = 4$, Sp. gr. *Pbam*. In the structure of $FeSb_2S_4$, there are two different kinds of Sb atoms, and each one is close to a sulfur atom (Sb - S distance = 2.5 Å). The Fe atoms are surrounded by six sulfur atoms in a distorted octahedron; octahedra have common edges and form chains parallel to the "c" axis [9]. It should be mentioned that garavellite, or $FeSbBiS_4$, is a naturally occurring mineral. A structural study of garavellite has not been carried out, but it has been determined that it crystallizes in the orthorhombic lattice with parameters: $a = 11.439$, $b = 14.093$, $c = 3.754$ Å, z = 4; Sp. gr. *Pbam* [10]. This compound can be represented as the replacement of one antimony atom with a bismuth atom in the structure of berthierite $FeSb_2S_4$.

Complex compounds of rare earth elements and solid solutions based on them draw attention as magnetic, luminescent, acousto-optical, and other materials and employed in contemporary microelectronics

[11-16]. Previously, we [17-19] studied synthetic analogues of the berthierite mineral $FeSb_2S_4$ with "light" lanthanides.

The purpose of this work is to study phase formation in the $FeSb_2S_4 – FeNd_2S_4$ ($FeEr_2S_4$) systems and obtain single crystals of compounds of the $FeLnSbS_4$ (Ln=Nd, Er) type for physical measurements.

Experimental part

Quaternary alloys $(FeSb_2S_4)_{1-x}(FeLn_2S_4)_x$ were obtained by fusing the original ternary compounds. Triple sulfides $FeSb_2S_4$ and $FeLn_2S_4$ (Ln=Nd, Er) were synthesized by the ampoule method from highly pure elements (iron carbonyl, antimony grade Su-3, lanthanide grade LnM-O and sulfur grade B5). The synthesis of compounds ($FeSb_2S_4$, $FeNd_2S_4$ and $FeEr_2S_4$) was carried out in evacuated 10^{-2} Pa quartz ampoules at a temperature of 1100-1150 K. Then, homogenizing annealing of samples was carried out at 950 K for 2 weeks synthesis. Obtained compounds were used in the production of quaternary alloys.

Quaternary alloys of the $FeSb_2S_4$-$FeNd_2S_4$ and $FeSb_2S_4$-$FeEr_2S_4$ systems were synthesized in single-section furnaces at 1250-1275 K. After holding (1 hour) at this temperature, the furnace was cooled to 650 K with a rate of 50°/hour, and homogenizing annealing was carried out in this mode for 470-480 hours. Details of the synthesis procedure are given in [20].

The alloys were studied by differential-thermal analysis (DTA) in NTR-73 device (heating rate was 10°/min, alumel-chromium thermocouple is used, Al_2O_3 served as the standard) and X-ray diffraction technique (XRD) in D2 PHASER diffractometer from Bruker with $CuK\alpha$ radiation and Ni-filter, as well as microhardness and density measurement (in PMT-3 device). The melting point of the samples was determined with an accuracy of ±5 K, and the lattice parameters were calculated with an accuracy of ±0.001 Å. The electrical properties of the obtained sulfosalts were carried out by the compensation method using a direct current probe described in [20].

Results and its discussion

Phase diagrams of the $FeSb_2S_4$-$FeNd_2S_4$ and $FeSb_2S_4$-$FeEr_2S_4$ systems, constructed according to DTA, X-ray diffraction data, microhardness and density measurements are presented in Fig. 1. (a; b). As can be seen, a complex interaction occurs between the initial components with the formation of the intermediate phase $FeLnSbS_4$ (Ln=Nd, Er) and solid solutions based on $FeSb_2S_4$. Quaternary compounds $FeNdSbS_4$ and $FeErSbS_4$ melt congruently at 960 and 1330 K, respectively, and divide each system into two subsystems: $FeSb_2S_4$ - $FeNdSbS_4$ and $FeNdSbS_4$ - $FeNd_2S_4$ ($FeSb_2S_4$ - $FeErSbS_4$ and $FeErSbS_4$ - $FeEr_2S_4$).

As can be seen, in both cases, $FeSb_2S_4$ and quaternary compounds ($FeNdSbS_4$ and $FeErSbS_4$) completely dissolve in each other at any ratio, both in liquid and solid states.

The presence of a continuous series of solid solutions in the corresponding subsystems was confirmed by the X-ray method. Fig. 2 shows X-ray diffraction patterns of the compounds $FeNdSbS_4$ and $FeErSbS_4$, and Table. 1 listed X-ray diffraction patterns data of quaternary and initial sulfosalts for comparison. The compounds $FeNdSbS_4$ and $FeErSbS_4$ are isostructural with $FeSb_2S_4$, crystallize in the orthorhombic system with the following unit cell parameters for $FeNdSbS_4$: a=11.392, b=14.132, c=3.76 Å, V=603.566 Å, z=4, Sp. gr. *Pbam* ρ=4,88 g/cm^3; and for $FeErSbS_4$: a=11.360, b=13.842, c=3.600 Å, z=4, Sp. gr. *Pbam*, ρ=5.17 g/cm^3.

The state diagram of the $FeSb_2S_4$-$FeNdSbS_4$ system corresponds to the first type according to Rosebohm, i.e. the liquidus temperature constantly increases from a low-melting ($FeSb_2S_4$) to a high-melting component. Unlike the first subsystem, the state diagram of the $FeSb_2S_4$–$FeErSbS_4$ subsystem belongs to the third type according to Rosebohm.

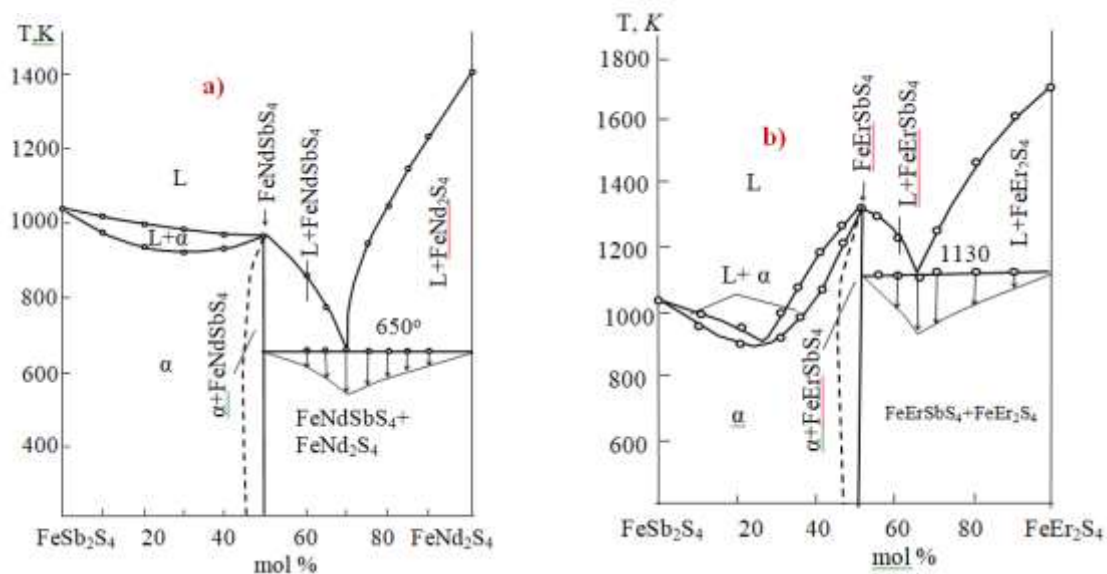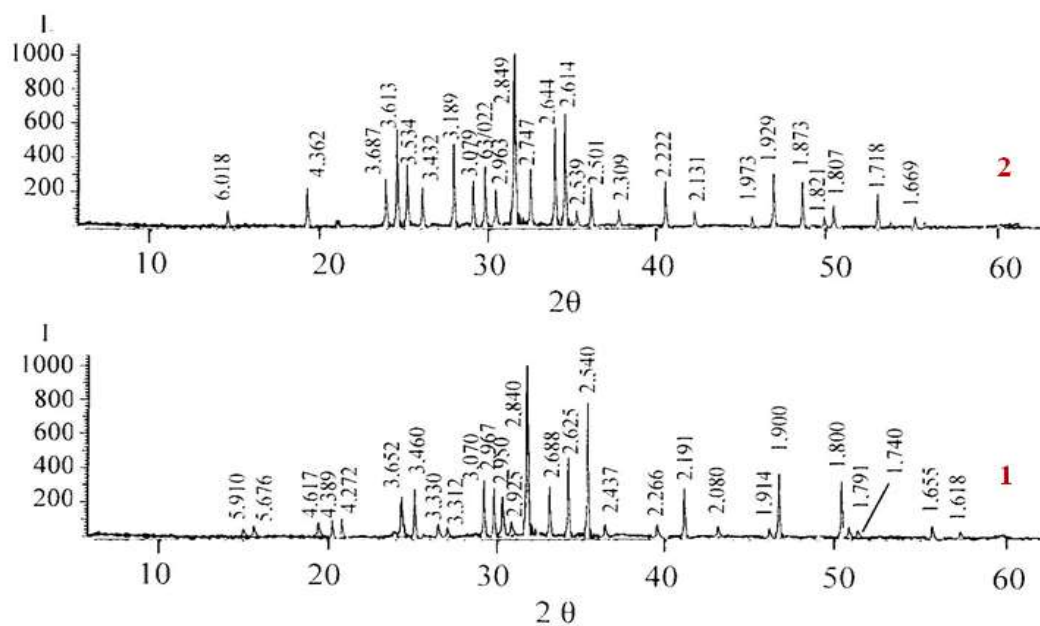**Fig. 1.** Phase diagrams of the $FeSb_2S_4$-$FeNd_2S_4$ (a) and $FeSb_2S_4$-$FeEr_2S_4$ (b) systems

Fig.2. Diffraction patterns of the FeErSbS4 (1) and FeNdSbS4 (2) compounds

Table 1. Interplanar distances and line intensities for the $FeSb_2S_4$, $FeNdSbS_4$, $FeErSbS_4$, $FeNd_2S_4$ and $FeEr_2S_4$ compounds

$FeSb_2S_4$		$FeNdSbS_4$		$FeErSbS_4$		$FeNd_2S_4$		$FeEr_2S_4$	
d_{exp}	I/I_0	d_{exp}	I/I_0	d_{exp}	I/I_0	d_{exp}	I/I_0	D_{exp}	I/I_0
4.300	80	6.018	10	5.910	5	8.201	5	2.916	20
3.620	90	4.362	25	5.676	8	7.053	8	5.007	10
3.350	60	3.687	30	4.617	10	6.904	45	3.992	100
3.150	90	3.613	60	4.389	15	6.514	5	3.513	50
3.010	60	3.534	40	4.272	15	5.802	10	3.201	5
2.830	90	3.432	25	3.652	25	5.593	10	3.142	5
2.600	100	3.189	50	3.460	35	4.471	10	2.629	25
2.510	60	3.079	30	3.330	10	5.196	40	2.377	70

3.230	40	3.022	40	3.312	8	7.784	10	2.314	40
2.155	60	2.963	20	3.070	35	4.600	100	2.262	10
2.035	60	2.849	100	2.967	30	4.487	15	2.199	20
1.990	70	2.747	35	2.950	25	4.268	10	2.051	20
1.900	60	2.644	60	2.925	12	4.100	100	1.993	5
1.870	90	2.614	70	2.840	100	3.930	10	1.969	10
1.785	60	2.539	10	2.688	30	3.692	5	1.920	15
1.760	60	2.501	20	2.625	50	3.573	10	1.855	10
1.690	40	2.309	10	2.540	80	3.525	5	1.761	5
1.660	40	2.222	30	2.437	10	4.493	5	1.744	15
1.630	5	2.131	10	2.266	10	4.463	10	1.681	5
1.585	60	1.973	5	2.191	30	3.276	5	1.647	10
1.495	20	1.929	35	2.080	8	3.734	20	1.580	10
1.415	40	1.873	30	1.914	8	2.680	15	1.553	5
1.365	40	1.821	5	1.900	40	2.579	14	1.534	10
1.335	30	1.807	15	1.800	35	2.343	15	1.492	15
1.318	20	1.718	20	1.791	8	2.075	8	1.463	5
1.255	30	1.669	5	1.740	5	2.050	10		
1.079	70			1.655	10	1.972	5		
1.059	70			1.618	5	1.866	10		
1.045	50					1.475	10		

Table 2. Parameters of crystal lattices of solid solutions $(FeSb_2S_4)_{1-x}(FeNdSbS_4)_x$

Composition	Parameters of the elementary cell, Å			Melting point, К	ρ, g/cm^3	H_{μ}, MPa
	a	b	c			
$FeSb_2S_4$	11.44	14.10	3.76	1030	4.65	1450
$FeSb_{1.9}Nd_{0.1}S_4$	11.438	14.10	3.758	770-1020	4.69	1600
$FeSb_{1.8}Nd_{0.2}S_4$	11.432	14.098	3.754	920-1005	4.73	1730
$FeSb_{1.7}Nd_{0.3}S_4$	11.430	14.095	3.752	930-980	4.75	1830
$FeSb_{1.6}Nd_{0.4}S_4$	11.427	14.094	3.750	945-970	4.80	1900
$FeNdSbS_4$	11.392	14.132	3.746	960	4.88	1900

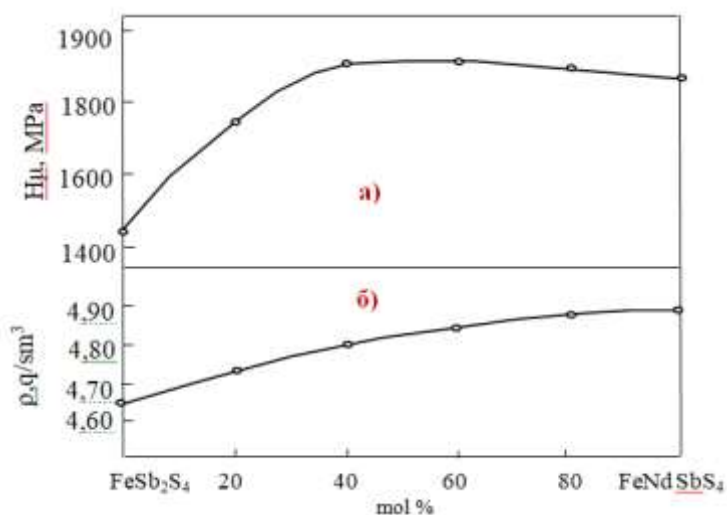**Fig. 3.** Dependence of microhardness (a) and density (b) of solid solutions $(FeSb_2S_4)_{1-x}(FeNdSbS_4)_x$ on composition.

Solid solutions $(FeSb_2S_4)_{1-x}(FeNdSbS_4)_x$ and $(FeSb_2S_4)_{1-x}(FeErSbS_4)_x$ crystallize in the orthorhombic system and belong to the substitution type. The lattice parameters of solid solutions vary linearly with composition change (Vegard's law), which also proves the formation of continuous solid solutions (see Table 2). In the FeSb$_2$S$_4$-FeNdSbS$_4$ and FeSb$_2$S$_4$-FeErSbS$_4$ systems, the occurrence of continuous solid solutions was also established by measuring microhardness and density depending on the composition (Fig. 3). As can be seen, the curves of microhardness and pycnometric density of solid solutions $(FeSb_2S_4)_{1-x}(FeNdSbS_4)_x$ vs.

composition pass through a maximum.

The temperature dependence of the electrical conductivity (Fig. 4, a) and thermo-emf of the FeNdSbS$_4$ and FeErSbS$_4$ quaternary sulfosalts was measured in the 293-900 K temperature range (Fig. 4, b). As can be seen, the inflection in the temperature dependence of electrical conductivity at 400-500 K corresponds to the region of impurity conductivity. With increasing temperature at 550-560 K the intrinsic conductivity appears. The band gap calculated from the intrinsic conductivity region is $\Delta E=1.36$ and $\Delta E=1.45$ eV for FeNdSbS$_4$ and FeErSbS$_4$, respectively.
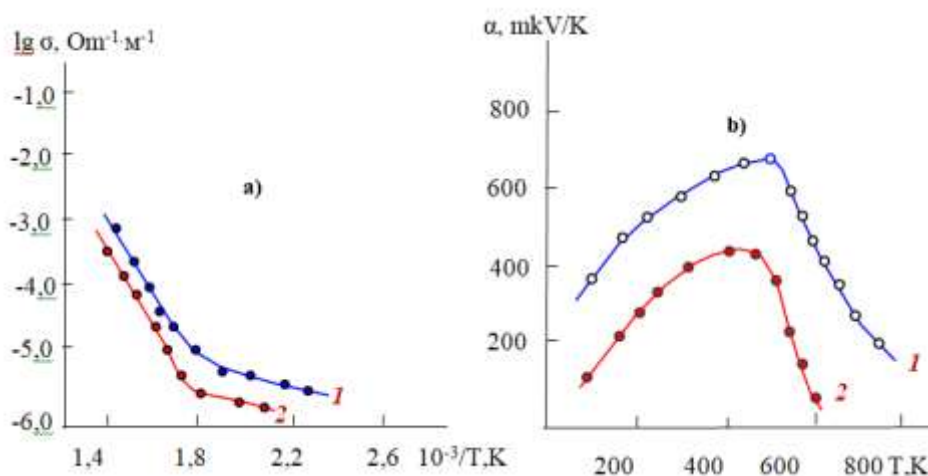

Fig. 4. Temperature dependence of electrical conductivity (a) and thermo-emf (b) on the composition for the FeNdSbS$_4$ (1) and FeErSbS$_4$ (2) compounds

The thermo-emf coefficient from room temperature to the temperature of transition to intrinsic conductivity increases, and in its own region it naturally decreases. The temperature change in the thermo-emf coefficient is in good agreement with the change in the electrical conductivity of these compounds. In the two-phase model, thermo-emf is expressed by the formula:

$$\alpha = \frac{\alpha_{\rho_1} b \cdot \rho_1 + \alpha_{\rho_2} \rho_2}{b \cdot \rho_1 + \rho_2}, \quad (1)$$

where $\alpha\rho_1$ and $\alpha\rho_2$ are thermo-emfs arising from the presence of light and heavy holes, respectively. If the electrical conductivity of heavy holes is small compared to the electrical conductivity of light holes, then the value of ρ_2 in the denominator can be neglected. If $\alpha_{p2}P_2 \ll \alpha_{p1}P_1$ then the total thermo-emf is approximately equal to the thermo-emf of light holes. In the case of a simple parabolic band structure and a power-law dependence of the mean free path on energy, the expression for thermo-emf at an arbitrary degree of degeneracy has the form:

$$\alpha = \frac{k}{e}\left[\frac{(r+2)F_{r+1}(\eta^*)}{(r+2)F_r(\eta^*)}\right] \quad (2)$$

$$F_r(\eta^*) = \int\limits_0^0 \frac{x^2 dx}{e^x - \eta^* + 1} \qquad (3)$$

In the case of a non-degenerate electron gas, expression (2) is simplified:

$$\alpha = \frac{k}{e}\left[r + 2 + \lg \frac{2(2\pi mKT)^{3/2}}{h^3 n} \right] \qquad (4)$$

where m is the effective mass of the density of states:

$$m = N^{3/2}(m_1 m_2 m_3)^{1/3}$$

where N is the number of ellipsoids; m_1, m_2, m_3 principal value of the effective mass tensor, η-reduced chemical potential.

Thus, the $FeSb_2S_4$-$FeLn_2S_4$ (Ln=Nd, Er) systems have been studied and their phase diagrams have been constructed. The formation of quaternary $FeNdSbS_4$ and $FeErSbS_4$ compounds with melting points 960 and 1330 K, respectively, has been established. It was shown that $FeLnSbS_4$ divides the $FeSb_2S_4$-$FeLn_2S_4$ system into two $FeSb_2S_4$-$FeLnSbS_4$ and $FeLnSbS_4$-$FeLn_2S_4$ subsystems. In the first subsystem, continuous solid solutions are formed, and the second subsystem is of the eutectic type. Coordinates of eutectic points: 30 mol% $FeSb_2S_4$ and 650 K (for the $FeSb_2S_4$-$FeNd_2S_4$) 35 mol% $FeSb_2S_4$ and 1130K (for the $FeSb_2S_4$-$FeEr_2S_4$).

Conclusion

Phase equilibria in the $FeSb_2S_4$-$FeNd_2S_4$ and $FeSb_2S_4$-$FeEr_2S_4$ systems were studied by physicochemical methods of analysis: differential thermal analysis, X-ray diffraction technique, microstructural analysis, as well as measurements of density and microhardness. The hase diagrams of the studied quasibinary systems were constructed. It has been established that in these systems sulfosalts $FeNdSbS_4$ and $FeErSbS_4$ are formed and melt congruently at 960 and 1330 K, respectively. These compounds form solid solutions of the substitution type in a wide range with $FeSb_2S_4$. Lanthanide-containing sulfosalts crystallize in the orthorhombic system.

The temperature dependences of electrical conductivity and thermo-EMF of quaternary sulfosalts $FeNdSbS_4$ and $FeErSbS_4$ in the temperature range 230-900 K have been studied. The band gap calculated from the intrinsic conductivity region is $\Delta E = 1.36$ and $\Delta E = 1.45$ eV for $FeNdSbS_4$ and $FeErSbS_4$, respectively.

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$FeSb_2S_4$ - $FeLn_2S_4$ SİSTEMİNDƏ FAZAƏMƏLƏGƏLMƏ, $FeLnSbS_4$ (Ln=Nd, Er) TİPLİ BİRLƏŞMƏLƏRİN SİNTEZİ VƏ XASSƏLƏRİ

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Xülasə: $FeSb_2S_4$ - $FeNd_2S_4$ и $FeSb_2S_4$ - $FeEr_2S_4$ sistemlərində fazaəmələgəlmə diferensial-termiki, rentgenfaza, mikroquruluş və mikrobərklik analiz metodları vasitəsilə tədqiq edilərək, $FeLnSbS_4$ tərkibli dördlü sulfoduzun alınması müəyyən edilmişdir. $FeNdSbS_4$ və $FeErSbS_4$ birləşmələri uyğun olaraq 960 və 1330 K temperaturda əriyir və $FeSb_2S_4$ ilə əvəzolunan tipli bərk məhlullar əmələ gətirir. $FeLnSbS_4$ tipli sulfoduzlar rombik sinqoniyada kristallaşır, elementar qəfəs parametrləri:

FeNdSbS$_4$ -a=11,392, b=14,132, c=3,746Å, fəza qr. Pbam, z=4, sıxlıq ρ=4,88 q/sm^3, FeErSbS$_4$ - a=11,360, b=13,842, c=3,600 Å, fəza qr. Pbam, z=4, ρ=5,17 q/sm^3-dir. FeLnSbS$_4$ tipli sulfoduzlar berterit quruluşlu FeSb$_2$S$_4$ tipinə aiddir.

Açar sözlər: sistem, faza diaqramı, sulfoduz, bərk məhlul, elementar qəfəs parametrləri, berterit

ФАЗООБРАЗОВАНИЕ В СИСТЕМЕ FeSb₂S₄ - FeLn₂S₄ , СИНТЕЗ И СВОЙСТВА СОЕДИНЕНИЙ ТИПА FeLnSbS₄ (Ln=Nd, Er)

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Резюме: Фазообразование в системах FeSb$_2$S$_4$ - FeNd$_2$S$_4$ и FeSb$_2$S$_4$ - FeEr$_2$S$_4$ были изучены методами дифференциально-термического, рентгенофазового, микроструктурного анализа и измерением микротвердости. Впервые построены диаграммы состояния этих систем и установлено образование четверных сульфосолей FeNdSbS$_4$ и FeErSbS$_4$, плавящихся конгруэнтно при 960 и 1330К, соответственно. Эти соединения образуют с FeSb$_2$S$_4$ непрерывные области твердых растворов типа замещения. Сульфосоли FeLnSbS$_4$ кристаллизуются в ромбической сингонии с параметрами элементарный ячейки: FeNdSbS$_4$-a=11,392, b=14,132, c=3,746 Å, пр.гр. Pbam, z=4, плотность ρ=4,88 г/см^3, FeErSbS$_4$-a=11,360, b=13,842, c=3,600 Å, пр.гр. Pbam, z=4, ρ=5,17 г/см^3. Сульфосоли типа FeLnSbS$_4$ относятся к структурному типу бертьерита FeSb$_2$S$_4$.

Ключевые слова: система, фазовая диаграмма, сульфосоль, твердые растворы, параметры элементарной ячейки, бертьерит.