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FeS- FeGa₂S₄- FeGaInS₄ SYSTEM**F.M. Mammadov**

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Abstract: *The phase equilibria in the FeS-FeGa₂S₄-FeGaInS₄ system were studied using the differential thermal and X-ray diffraction phase analysis. The projection of the liquidus surface and the isothermal sections of the phase diagram at 1200 and 900 K were constructed. It revealed that it belongs to quasi-ternary systems with monovariant eutectic and peritectoid equilibria. At 1200 K, the system was characterized by the presence of a continuous series of solid solutions along the boundary system FeGa₂S₄-FeGaInS₄ (β -phase) as well as solid solutions based on the high-temperature modification FeS (α -phase) which formed a wide two-phase region $\alpha + \beta$. The FeS-FeGa₂S₄ boundary system was characterized by the formation of the Fe₂Ga₂S₅ ternary compound at 900 K. This compound formed Fe₂Ga₂S₅+ β and Fe₂Ga₂S₅+ $\alpha+\beta$ heterogeneous regions below this temperature which significantly narrowed the two-phase region $\alpha + \beta$.*

Keywords: *FeGa₂S₄, FeS, FeGaInS₄, phase diagram, liquidus, solid solutions*

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Introduction

Compounds of AB₂X₄- type (A-Mn, Fe, Co, Ni, Ge, Sn, Pb, B-p¹ or p³-element, X-chalcogen) and phases based on them are valuable functional materials with thermoelectric, photoelectric, optical, magnetic and other properties [1-8]. In recent years, it has been established that some compounds of this type with a layered structure such as tetradimite are topological insulators and extremely promising for practical applications, ranging from spintronics to safety systems and medicine [9-13]. It is known that the intensive development of a new scientific field - spintronics is associated with the possibility of transferring the oriented electron spin from a ferromagnet to a semiconductor. The above compounds also apply to materials promising for the specified area [14-16]. In [17, 18] published recently, the MnBi₂Te₄ compound, the first antiferromagnetic topological insulator, was described.

The development and optimization of the synthesis of new complex phases are based on data on phase equilibria and thermodynamic

characteristics of the corresponding systems [19-24].

The paper explores phase relations in the FeS- FeGa₂S₄-FeGaInS₄ system. The initial compounds of this system were studied thoroughly.

The FeS compound melt congruently at 1461 K and undergo polymorphic transitions at 411 and 588 K [25]. The high-temperature modification of FeS crystallized in a tetragonal structure (Sp.gr.P4/nmm) with lattice parameters $a = 0.3768$ nm, $c = 0.5039$ nm [26] or $a = 0.36735$ nm, $c = 0.50328$ nm [27], while the low-temperature modification has a hexagonal structure: $a = 0.34436$ (1) nm, $c = 0.57262$ (2) nm [28].

According to [29], the ternary compound FeGa₂S₄ melts congruently at 1418 K. According to [30], it is formed by the peritectic reaction at 1343 K and undergoes a polymorphic transformation at 1283 K. In [31] it was shown that FeGa₂S₄ crystallizes in a rhombic structure of the ZnAl₂S₄ type with lattice parameters $a = 1.289$ nm; $b = 0.751$; $c =$

0.609 nm. According to [32], this compound exists in two crystalline modifications: the low-temperature compound has a trigonal one (Sp.gr. P3ml, $a = 0.3654$ (2) nm; $c = 1.20556$ nm), and the high-temperature - rhombic ($a = 1.289$; $b = 0.751$; $c = 0.609$ nm) structure.

The FeGaInS_4 compound also melts congruently (1375 K) and has a trigonal structure of the ZnAl_2S_4 type (Sp.gr.P-3m1) with parameters: $a = 0.37765$, $c = 1.22257$ nm [33, 34].

The boundary quasi-binary components of the $\text{FeS-FeGa}_2\text{S}_4\text{-FeGaInS}_4$ system were studied in [29, 33]. According to [29], the $2\text{FeS} - \text{FeGa}_2\text{S}_4$ system is eutectic type. The eutectic

had a composition of ~ 53 mol% FeGa_2S_4 and crystallized at 1328 K. The $\text{Fe}_2\text{Ga}_2\text{S}_5$ compound is formed by a solid-phase reaction at 1043 K. According to [33], the $2\text{FeS} - \text{FeGaInS}_4$ system also has a eutectic type phase diagram. The eutectic coordinates are 63 mol% FeGaInS_4 and 1310 K. The $\text{FeGa}_2\text{S}_4\text{-FeGaInS}_4$ system is characterized by the formation of a continuous series of solid solutions between FeGaInS_4 and the low-temperature modification of FeGa_2S_4 [33]. It is assumed that the formation of solid solutions leads to a sharp increase in the temperature of the polymorphic transition FeGa_2S_4 (1327 K) and the establishment of peritectic equilibrium at ~ 1410 K.

Experimental part

2.1. Materials and synthesis

The initial compounds FeS , FeGa_2S_4 and FeGaInS_4 were synthesized using high-purity iron (99.995%), indium (99.999%), gallium (99.999%) and sulfur (99.99%) purchased from Alfa Aesar.

Stoichiometric mixtures of elements were placed in a quartz ampoule which was evacuated to a residual pressure of $\sim 10^{-2}$ Pa. The syntheses were carried out in a two-zone furnace. In the case of FeS , the lower "hot" zone was slowly heated from a room temperature to a temperature of 1400 K, and for FeGa_2S_4 and FeGaInS_4 compounds it was 30–50 K above their melting points, and part of the ampoule outside the furnace was cooled with water ("cold" zone). The interaction of the components occurs in the "hot" zone, and in the "cold" zone, the chalcogen condenses and returns to the interaction zone. As a result of the reaction in the "cold" zone, the mass of chalcogen gradually decreased and within 1-2 hours it is consumed almost completely. After that, the ampoule was completely placed in the oven and kept at the indicated temperature for 2-3 hours. The obtained samples were subjected to heat treatment at 800 K for 100 hours in order to increase the crystallinity of the synthesized compounds.

The alloys of the studied system were prepared through melting pre-synthesized compounds in evacuated quartz ampoules. The

alloys were first annealed at 1000 K (100 h), then at 900 K (300 h) and cooled in a switched off the furnace. Some alloys, after heat treatment at 1200 K or 900 K, were quenched in cold water.

2.2. Methods

The differential thermal analysis (DTA) and X-ray powder diffraction technique (XRD) were used for the analysis of samples. DTA was carried out using the NETZSCH 404 F1 Pegasus system from room temperature to ~ 1450 K, depending on the composition of the alloys at a heating rate of $10 \text{ K}\cdot\text{min}^{-1}$. Thermal effects temperatures were determined mainly from heating curves. The temperature accuracy was ± 2 K.

Powder X-ray diffraction patterns were recorded on a Bruker D2 Phaser diffractometer using $\text{Cu K}\alpha_1$ radiation at room temperature.

According to DTA, the synthesized compounds melt at 1460 K (FeS), 1420 K (FeGa_2S_4) and 1375 K (FeGaInS_4). Our data are consistent with published data [25, 29, 33]

X-ray diffraction patterns of the synthesized compounds also showed the formation of single-phase materials. The calculated lattice parameters of hexagonal FeS ($a = 0.34440$ nm, $c = 0.57260$ nm), trigonal FeGa_2S_4 ($a = 0.36543$; $c = 1.20558$ nm) and FeGaInS_4 ($a = 0.37765$, $c = 1.22257$ nm) are good consistent with published data [28, 30, 34].

Results

Combined analysis of the obtained experimental data allowed us to obtain a general scheme of the phase equilibria, including isothermal sections at 1200 K and 900 K, polythermal sections 2FeS- [A], FeGa₂S₄- [B] ([A] and [B] alloys with “FeGa_{1.5}In_{0.5}S₄” and “Fe_{1.5}Ga_{0.5}In_{0.5}S₃” compositions, respectively) of the phase diagram as well as the projection of the liquidus surface (Figs. 1–4).

According to the data obtained, the FeS-FeGa₂S₄-FeGaInS₄ system refers to quasi-

ternary systems with monovariant eutectic and peritectoid equilibria.

Isothermal sections at 1200 K and 900 K. Continuous solid solutions along the boundary system FeGa₂S₄-FeGaInS₄ (β -phase) are in connod tie with solid (α) solutions based on the high-temperature modification of FeS and form a two-phase region $\alpha+\beta$, which at 1200 K covers the entire area of the concentration triangle (Fig. 1a).

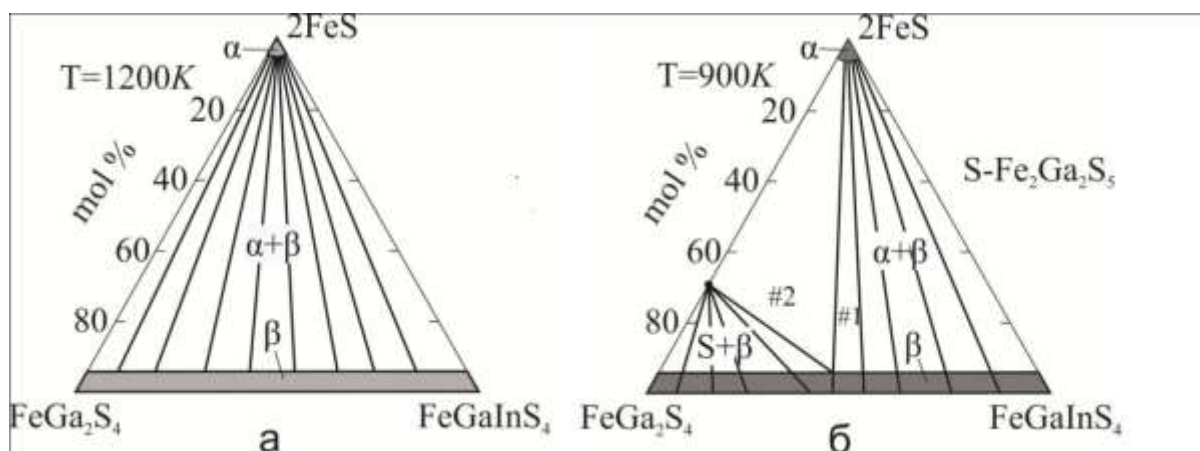


Fig.1. Isothermal sections of the FeS-FeGa₂S₄-FeGaInS₄ system at 1200 K (a) and 900 K(b).

Below 900 K, in the FeS-FeGa₂S₄ boundary system, there is a ternary compound Fe₂Ga₂S₅ formed by the solid-phase reaction between FeS and FeGa₂S₄. This leads to the formation of Fe₂Ga₂S₅+ β and $\alpha+\beta$ +Fe₂Ga₂S₅ phase regions. The isothermal section at 900 K clearly demonstrates this (Fig. 1b).

Phase fields on both isothermal sections are confirmed by powder XRD analysis. As an example, Fig. 2 presents powder diffraction patterns of two annealed alloys (Fig. 1b, samples # 1 and # 2) with the reflection lines of the phases coexisting in them. As can be seen, alloy # 1 consists of a two-phase mixture of FeS (low-temperature modification) and the β -phase. Sample # 2 is three-phase - FeS +

Fe₂Ga₂S₅+ β . A comparison of the reflection angles with the diffraction patterns of alloys of the FeGa₂S₄- FeGaInS₄ side system showed that the β - phase in both samples has a composition of ~50 mol% FeGaInS₄, which is in accordance with Fig. 1b.

The liquidus surface (Fig. 3) consists of three areas corresponding to the primary crystallization of the α , β and β' - phases (β' - solid solutions based on the high-temperature modification of FeGa₂S₄). The last two areas are delimited by us conditionally (P_1P_2 dashed line). P_1P_2 curve refers to monovariant peritectic equilibrium



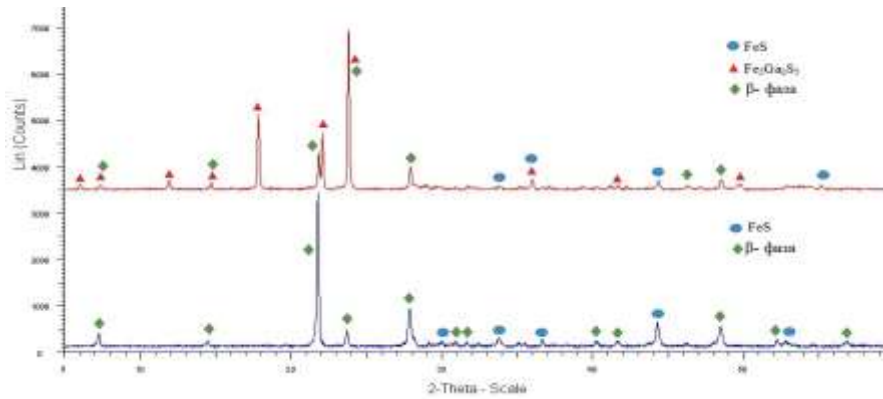


Fig. 2. Powder diffraction patterns of alloys # 1 and # 2 showed on Fig.1

The fields of primary crystallization of α - and β - phases are separated by the e_1e_2 eutectic

curve with the monovariant equilibrium $L \leftrightarrow \alpha + \beta$ (2)

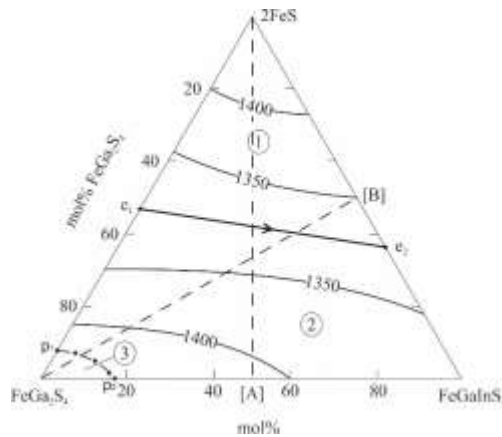


Fig.3. Liquidus surfaces projection of the FeS- FeGa₂S₄- FeGaInS₄ system

Polythermal section FeGa₂S₄ - [B] (Fig. 5). The liquidus of this section consists of three curves, which from left to right correspond to the primary crystallization of β' -, β - and α - solid solutions. The formation of β' - and β - phases is accompanied by a sharp increase in the temperature of the polymorphic transition FeGa₂S₄ (from 1330 K to ~ 1410 K) and the establishment of peritectoid equilibrium (1).

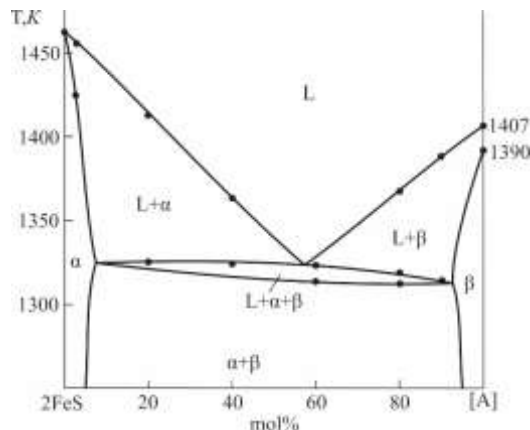


Fig. 4. Polythermal section 2FeS- [A] of the FeS- FeGa₂S₄- FeGaInS₄ system

Polythermal section FeGa₂S₄- [B] (Fig. 5). The liquidus of this section consists of three curves, which from left to right correspond to the primary crystallization of β' -, β - and α - solid solutions. The formation of β' - and β - phases is accompanied by a sharp increase in the temperature of the polymorphic transition FeGa₂S₄ (from 1330 K to ~ 1410 K) and the establishment of peritectoid equilibrium (1).

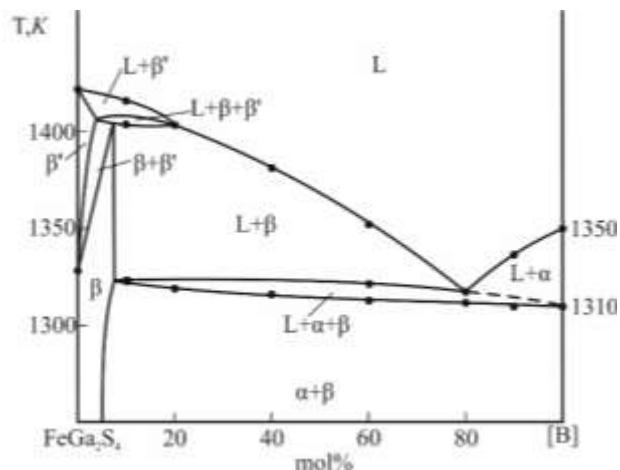


Fig. 4. Polythermal section FeGa₂S₄- [B] of the FeS- FeGa₂S₄- FeGaInS₄ system

The intersection point of liquidus curves of α and β - phases corresponds to the beginning of a monovariant eutectic reaction (2). During this reaction, a three-phase region $L+\alpha+\beta$ is formed, and upon its completion, a two-phase region $\alpha+\beta$ is formed.

According to our data (Figs. 1, 4, 5), the homogeneity regions of the α and β - phases of the above sections are maximum at the eutectic temperature (1310–1330 K) and amount to 6–7 mol%.

Conclusion

Based on the experimental data, we obtained a general scheme of phase equilibria in the FeS-FeGa₂S₄-FeGaInS₄ system, including the projection of the liquidus surface, two polythermal sections, and isothermal sections of the phase diagram at 1200 and 900 K. At 1200 K, the system is characterized by the presence of a continuous series of solid solutions along the side system

FeGa₂S₄-FeGaInS₄ (β -phase) and α -solid solutions based on the high-temperature modification of FeS, which form a wide two-phase region $\alpha + \beta$. At 900 K, the Fe₂Ga₂S₅ compound is involved in phase equilibria, which leads to the formation of heterogeneous regions Fe₂Ga₂S₅+ β и FeS+Fe₂Ga₂S₅+ β . The results can be used to develop new magnetic materials based on β -solid solutions.

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FeS- FeGa₂S₄- FeGaInS₄ SİSTEMİ

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Differensial termiki və rentgenfaza analiz metodları ilə FeS-FeGa₂S₄-FeGaInS₄ sistemində faza tarazlığı tədqiq edilmişdir. Sistemin likvidus səthinin proeksiyası, 1200 və 900 K temperaturlarda izotermik kəsiklərin faza diaqramları qurulmuşdur. Müəyyən edilmişdir ki, sistem monovariant evtektik və peritektoid tarazlığı olan kvaziüçlü sistemə aiddir. Sistem 1200 K-də FeGa₂S₄-FeGaInS₄

(β - faza) yan kəsiyi boyunca fasiləsiz bərk məhlulların mövcudluğu və FeS (α -) yüksək temperaturlu modifikasiyası əsasında həllolma sahəsinin olması ilə xarakterizə olunur. Üçlü sistemin FeS-FeGa₂S₄ yan kəsiyində 900 K temperaturda Fe₂Ga₂S₅ üçlü birləşməsi əmələ gəlir. Bu birləşmə Fe₂Ga₂S₅ + β və Fe₂Ga₂S₅ + α + β heterogen sahələri əmələ gətirir ki, bunun da təsirindən α + β iki fazalı sahə əhəmiyyətli dərəcədə kiçilir.

Açar sözlər: FeGa₂S₄, FeS, FeGaInS₄, faza diaqramı, likvidus, bərk məhlullar.

СИСТЕМА FeS- FeGa₂S₄- FeGaInS₄

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Методами дифференциально-термического и рентгенофазового анализов исследованы фазовые равновесия в системе FeS-FeGa₂S₄-FeGaInS₄. Построены проекция поверхности ликвидуса и изотермические сечения фазовой диаграммы при 1200 и 900 К. Установлено, что она относится к квазитройным системам с моновариантными эвтектическим и перитектоидным равновесиями. При 1200 К система характеризуется наличием непрерывного ряда твердых растворов вдоль боковой системы FeGa₂S₄-FeGaInS₄ (β -фаза) и твердых растворов на основе высокотемпературной модификации FeS (α -), которые образуют широкую двухфазную область α + β . При 900 К в боковой системе FeS-FeGa₂S₄ образуется тройное соединение Fe₂Ga₂S₅, которое формируя гетерогенные области Fe₂Ga₂S₅+ β и Fe₂Ga₂S₅+ α + β ниже этой температуры значительно сужает двухфазную область α + β .

Ключевые слова: FeGa₂S₄, FeS, FeGaInS₄, фазовая диаграмма, ликвидус, твердые растворы.