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The study of the quasi-triple system $FeS–Ga_2S_3–Ag_2S$ by a $FeGa_2S_4–AgGaS_2$ section

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Abstract

The interest in the study of systems containing sulphides with the formula $A^IB^{III}C^{VI}_2$ is generated in particular by emerging opportunities for their practical use in the production of non-linear optical devices, detectors, solar cells, photodiodes, luminophors, etc. Therefore, taking into account the search for new promising materials based on silver and iron thiogallates, the goal of this work is to study the quasi-binary section $FeGa_2S_4–AgGaS_2$ of the quaternary system Fe–Ag–Ga–S.

The alloys of the $AgGaS_2$-$FeGa_2S_4$ system were synthesised from high-purity base metals: iron – 99.995 %, gallium – 99.999 %, silver – 99.99 %, and sulphur – 99.99 %. The alloys were studied using differential thermal analysis, X-ray phase analysis, and microstructural analysis as well as microhardness measurement and density determination.

Using the methods of physicochemical analysis, a *T-x* phase diagram of the $AgGaS_2$-$FeGa_2S_4$ section, which is the internal section of the quasi-triple FeS–Ga_2S3–Ag_2S system, was studied and constructed for the first time. It was established that this system is of the simple eutectic type. The composition of the eutectic point is 56 mol% $FeGa_2S_4$ and T = 1100 K. The solid solution ranges were determined on the basis of the source components. Based on $FeGa_2S_4$ and $AgGaS_2$ at the eutectic temperature the solubility stretches to 10 and 16 mol% respectively. With decreasing temperature, the solid solutions narrow and, at room temperature, comprise 4 mol% $AgGaS_2$ based on iron thiogallate ($FeGa_2S_4$) and 11 mol% $FeGa_2S_4$ based on silver thiogallate ($AgGaS_2$).

Keywords: phase diagram, solid solution, $FeGa_2S_4$, $AgGaS_2$, quasi-triple system, eutectic, X-ray analysis, $FeS–Ga_2S_3–Ag_2S$.

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1. Introduction

The interest in the study of systems containing sulphides with the formula $A^IB^{III}C^{VI}_2$ is generated in particular by emerging opportunities for their practical use in the production of non-linear optical devices, detectors, solar cells, photodiodes, luminophors, etc. [1–17].

The reported data shows that multicomponent sulphide compounds, especially those containing magnetic ($FeGa_2S_4$, $Fe_2Ga_2S_5$, $FeIn_2S_4$, etc.) ions, are functional materials and are used in the production of magneto-optical devices, photodetectors, lasers, light modulators, etc. [18–25].

The source components comprising the quaternary system Ag–Fe–Ga–S were thoroughly studied in [26–42]. Compositions $AgGaS_2$, Ag_9GaS_6, and $Ag_2Ga_{20}S_{31}$ were established during the study of the $Ag_2S–Ga_2S_3$ binary system [26, 30, 31]. $Ag_2Ga_{20}S_{31}$ is formed from them by a peritectic reaction at 1268 K while $AgGaS_2$ and Ag_9GaS_6 melt congruently at 1270 and 1063 K respectively. $AgGaS_2$ crystallises within the chalcopyrite-type structure (a = 5.7544, c = 10.299 Å space group I42d) [27] and is a p-type semiconductor with a band gap of ΔE = 2.75 eV [32].

The phase diagram of the section Ga_2S_3–FeS was studied in [33–42]. The authors established

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that triple compounds $FeGa_2S_4$ and $Fe_2Ga_2S_5$ are formed in the system Ga_2S_3–FeS [38, 42].

The microhardness of the $FeGa_2S_4$ and $Fe_2Ga_2S_5$ compounds are 4000±5 and 3500±5 MPa respectively [42].

The $FeGa_2S_4$ compound melts congruently at 1418 K [38], although, according to [39], $FeGa_2S_4$ is formed by a peritectic reaction at 1343 K and undergoes polymorphous transformation at 1283 K. $FeGa_2S_4$ crystallises in a rhombic crystal system of the $ZnAl_2S_4$ type with the following parameters: a = 1.289 nm, b = 0.751, c = 0.609 nm [40]. According to [41], this compound has two crystalline modifications: low-temperature trigonal P3ml: a = 0.3654 nm, c = 1.2056 nm; and high-temperature rhombic: a = 1.289, b = 0.751, c = 0.609 nm.

The goal of this work is to study the quasi-binary section $FeGa_2S_4$–$AgGaS_2$ of the quaternary system Fe–Ag–Ga–S.

2. Experimental

Synthesis of the alloys of the $AgGaS_2$–$FeGa_2S_4$ system were conducted from base metals. The base metals ($AgGaS_2$ and $FeGa_2S_4$) were synthesised using high-purity: iron – 99.995 %, gallium – 99.999 %, silver – 99.99 %, and sulphur – 99.99 %. Stoichiometric mixtures of the elements were placed in vacuum quartz ampoules (17 cm long and 1.5 cm in diameter) with residual pressure ~0.133 Pa [43]. Then the ampoule was placed in a two-zone furnace. The furnace was slowly heated from room temperature to the fusion temperature of the compound $FeGa_2S_4$. Sulphur becomes condensed in the cold region and returns to the interaction zone. The alloys in a liquid state were stirred at regular intervals. The outer part of the ampoule was cooled with water. After 1.5–2 hours in the cold region, the mass of the sulphur decreased. After that, the whole ampoule was placed in the furnace and held at a temperature of 1450 K for 2.5 hours. The process of synthesis lasted for at least 4 hours. Then the obtained samples were homogenised at a temperature of 800 K for 150 h. The alloys were studied using differential thermal analysis (DTA), X-ray phase analysis (XRD), and microstructural analysis (MSA) as well as microhardness measurement and density determination. XRD was performed on a D2 PHASER using Ni-filtered CuK_α radiation.

DTA of the alloys of the system was conducted on an HTP-73 device with a heating rate of 10 degrees per minute. Calibrating chromel-alumel thermocouples were used with Al_2O_3 as a reference standard. An etchant of the composition NH_4NO_3(3–8 wt%)+ $K_2Cr_2O_7$(0.02–0.5 wt%) + conc. H_2SO_4 was used during the study of the microstructure of the alloys with the etching time of 20 sec. The microhardness of the alloys was measured on a microhardness tester PMT-3 at the loads 0.01 and 0.02 N. MSA of the system alloys was performed on a MIM-8 metallographic microscope on preliminarily etched sections polished with the paste.

3. Results and discussion

Based on the results of physicochemical analysis (DTA, XRD, MSA, and density determination), a phase diagram of the system $AgGaS_2$–$FeGa_2S_4$ was developed. The DTA results showed that all thermograms of the system alloys (90–10 mol% $AgGaS_2$) have three endoeffects each, except for the alloy containing 56 mol% $FeGa_2S_4$ while the alloys containing 90 and 10 mol% $AgGaS_2$ demonstrate two and four endoeffects each respectively (Table 1). The effects at 905 K correspond to the phase transition α-$FeGa_2S_4$ ↔ β-$FeGa_2S_4$.

As Fig. 1 shows, the phase diagram of the system $AgGaS_2$–$FeGa_2S_4$ belongs to the eutectic type with limited component solubility in solid state. The solubility at 300 K based on $AgGaS_2$ is

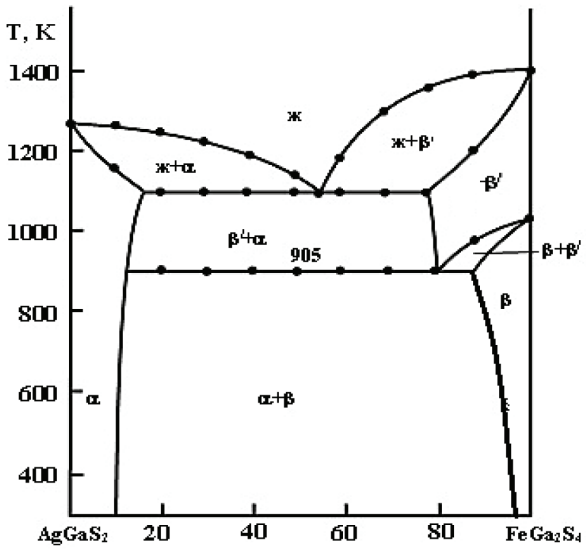

Fig. 1. Phase diagram of the $AgGaS_2$-$FeGa_2S_4$ system

Table 1. Composition, results of the DTA of alloys of the $AgGaS_2–FeGa_2S_4$ system

Composition mol% $FeGa_2S_4$	Thermal effects, K
100	1420
90	905, 980, 1235, 1405
80	905, 1100, 1375
70	905, 1100, 1310
60	905, 1100, 1175
56	1100 (eutectic)
50	905, 1100, 1145
40	905, 1100, 1195
30	905, 1100, 1230
20	905, 1100, 1250
10	1175, 1260
0.0	1270

11 mol% $FeGa_2S_4$ and 4 mol% $AgGaS_2$ based on $FeGa_2S_4$. The solubility at the eutectic temperature stretches to 16 and 10 mol% respectively. The eutectic has a composition of 56 mol% $FeGa_2S_4$ and crystallises at 1100 K.

The liquidus of the system $AgGaS_2–FeGa_2S_4$ consists of the primary crystallisation branches α and β solid solutions crossing at 56 mol% $FeGa_2S_4$ and T = 1100 K. The temperature of the phase transition $\beta(FeGa_2S_4)\leftrightarrow\beta'(FeGa_2S_4)$ is reduced to 905 K under the influence of the second component. MSA of the annealed alloys showed that the alloys of the system $AgGaS_2–FeGa_2S_4$ are single-phase except for the alloys containing 11–96 mol% $FeGa_2S_4$.

Below solidus, α and β solid solutions co-crystallise. The solubility regions based on source components are narrow: 11 mol% $FeGa_2S_4$ based on $AgGaS_2$ and 4 mol% $AgGaS_2$ based on the second component. Solubility limits were determined using the XRD and MSA of the alloys annealed and quenched at the temperature 700 K.

To determine the limits of the regions of solid solutions of the source components ($AgGaS_2$ and $FeGa_2S_4$), 98, 96, 95, 93, 91, 90, 89, 88 mol% were additionally synthesised from both sides. These alloys were annealed at 650 and 800 K with the annealing duration of 1 month (Table 2).

After the annealing, a microstructural analysis of the alloys was conducted that showed that there are limited solubility regions near $AgGaS_2$ and $FeGa_2S_4$. Solid solutions based on $AgGaS_2$ belong to the Ag_2GeS_3 structural type and

Table 2. Annealing of the alloys of the $AgGaS_2–FeGa_2S_4$ system at temperatures of 650 and 800 K

Composition mol%		650 K phase count	800 K, phase count
$AgGaS_2$	$FeGa_2S_4$		
0.0	100	α	α
2.0	98	α	α
4.0	96	α+β	α
5.0	95	α+β	α+β
7.0	93	α+β	α+β
9.0	91	α+β	α+β
10	90	α+β	α+β
11	89	α+β	α+β
12	88	α+β	α+β
100	0.0	β	β
98	2.0	β	β
96	4.0	β	β
95	5.0	β	β
93	7.0	β	β
91	9.0	β	β
90	10	α+β	β
89	11	α+β	β
88	12	α+β	α+β

crystallise in the monoclinic syngony. Within the solubility limits, the parameters of the crystal lattice increase: a = 0.627÷0.748, b = 0.580÷0.664, c = 1.318÷1.386 nm, β = 93.27÷93°61.

The results of the X-ray phase analysis are in good agreement with the data of the microstructural analysis and confirm the formation of solid solutions based on the source components in the system $AgGaS_2–FeGa_2S_4$.

The data of the X-ray powder patterns of the alloys of the system $AgGaS_2–FeGa_2S_4$ showed that the samples of the compositions 0–11 and 95–100 mol% $FeGa_2S_4$ are single-phase. Their diffraction lines are identical to the diffraction patterns of the source components (silver thiogallate and iron thiogallate). The diffraction pattern of the alloys containing 11–96 mol% $FeGa_2S_4$ is two-phase (Fig. 2).

4. Conclusions

1. Using the methods of physicochemical analysis (XRD, DTA, MSA), a phase diagram of the system $AgGaS_2–FeGa_2S_4$ was studied and constructed for the first time. It was established that the system is a quasi-binary cross-section of the $FeS–Ga_2S_3–Ag_2S$ quasi-triple system and belongs to the simple eutectic type.

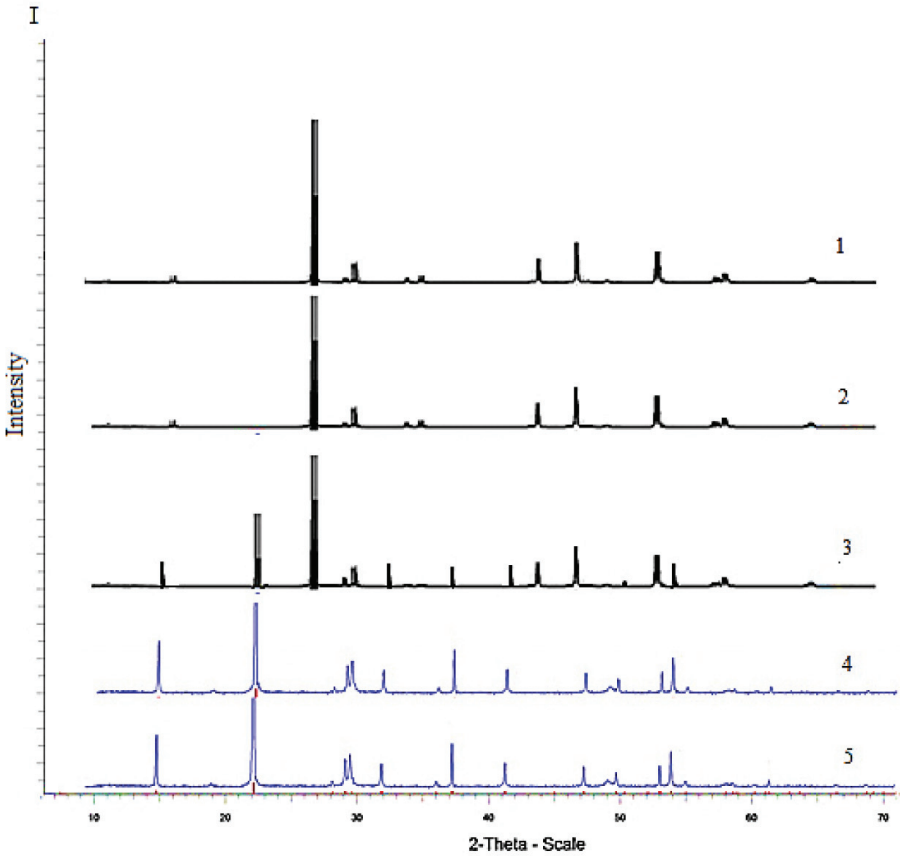

Fig. 2. Diffraction pattern of the alloys of the $AgGaS_2$–$FeGa_2S_4$ system: *1* – $AgGaS_2$; *2* – 11 mol% $FeGa_2S_4$; *3* – 40 mol% $FeGa_2S_4$; *4* – 96 mol% $FeGa_2S_4$; 5 – $FeGa_2S_4$

2. The formation of solid solutions based on their source components was found in the $AgGaS_2$–$FeGa_2S_4$ system. The solubility based on iron thiogallate at room temperature is 4 mol% $AgGaS_2$ and the solubility based on silver thiogallate is 11 mol% $FeGa_2S_4$.

Conflict of interests

The author declares that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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