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Ag_2S-Ga_2S_3-PbS QUASI-TERNARY SYSTEM

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Abstract

Multicomponent chalcogenides of copper and silver are scientifically and practically prospective materials, widely used in microelectronics. Thus, these materials posses unique properties such as optical, electrical, ferroelectric and ion conductivity. One of the urgent problems in the solar energy is the development of easily producable, environmentally friendly materials, capable of efficiently converting solar energy into electricity and other energy types. From this point of view, the search for chalcopyrite-structured compounds, such as $AgGaS_2$, $CuInS_2$ and $PbInS_2$, is of great importance. The synthesis of quaternary alloys was carried out in an evacuated quartz ampoule using pre-synthesized melts and high-purity elements at a temperature of 1100–1400 K. After melting the samples were gradually cooled down to 650–800 K at a rate of 10–15 degrees per hour, and then homogenization was carried out under the condition of 300–400 seconds.The state of equilibrium in the quasi-ternary Ag_2S-Ga_2S_3-PbS system has been studied for the first time using modern methods of physicochemical analysis. The study has found that the four sections of the quasi-ternary system are quasi-binary, and based on them, the quasi-ternary system is triangulated into five pseudo-ternary subsystems. It has been proved that in the ternary system a four-component compound $AgPb_2GaS_4$ is formed and there are limited solubility regions based on the initial components.

Keywords: system; quasi-ternary system; triangulation; pseudoternary; compound; eutectic.

КВАЗІПОТРІЙНА СИСТЕМА Ag_2S-Ga_2S_3-PbS

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Анотація

Багатокомпонентні суміші халькогенідів міді та срібла є науково та практично перспективними матеріалами, які широко використовуються в мікроелектроніці. Ці матеріали мають унікальні властивості, такі як оптична, електрична, сегнетоелектрична та іонна провідність. Однією з актуальних проблем сонячної енергетики є розробка легко вироблюваних, екологічно чистих матеріалів, здатних ефективно перетворювати сонячну енергію в електричну та інші види енергії. З цієї точки зору пошук сполук зі структурою халькопіриту, таких як $AgGaS_2$, $CuInS_2$ та $PbInS_2$, має велике значення. Синтез четвертинних сплавів проводили у вакуумованій кварцовій ампулі з використанням попередньо синтезованих розплавів і елементів високої чистоти за температури 1100–1400 К. Після плавлення зразки поступово охолоджували до 650–800 К зі швидкістю 10–15 градусів на годину з наступною гомогенізацією в умовах 300–400 секунд. Вперше досліджено фазову рівновагу в квазіпотрійній системі Ag_2S-Ga_2S_3-PbS із застосуванням сучасних методів дослідження фізико-хімічного аналізу. В результаті дослідження встановлено, що чотири розділи квазіпотрійної системи є квазібінарними, і на їх основі квазіпотрійна система розбита на п'ять псевдопотрійних підсистем. Доведено, що потрійна система має обмежені зони розчинності на основі чотирикомпонентної сполуки складу $AgPb_2GaS_4$ та первинних компонентів.

Ключові слова: система; квазіпотрійна система; тріангуляція; псевдопотрійна система; сполука; евтектика.

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Introduction

Multicomponent chalcogenides of copper and silver are scientifically and practically prospective materials, widely used in microelectronics. Thus, these materials posses unique properties such as optical, electrical, ferroelectric and ion conductivity [1–9]. The search for new less-studied quaternary chalcogenide semiconductos of copper and sulfur is one of the relevant issues both in semiconductor physics and solid state physics. One of the urgent problems in the solar energy is the development of easily productable, environmentally friendly materials, capable of efficiently converting solar energy into electricity and other energy types. From this point of view, the search for chalcopyrite-structured compounds, such as $AgGaS_2$, $CuInS_2$ and $PbInS_2$ is of great importance [10–14]. Searching for and obtaining promising materials with new functional properties, especially growingtheir high-purity mono crystals, as well as studying the temperature-composition dependence of the relevant systems are crucial from the technological point of view [15]. In this aspect, the study of Ag_2S-Ga_2S_3-PbS quasi-ternary system is highly relevant, it can contribute to the development of photosensitive luminescent materials.

Side systems (Ag_2S-PbS, Ag_2S-Ga_2S_3, PbS-Ga_2S_3) and initial components that make up the Ag_2S-Ga_2S_3-PbS quasi-ternary system have been studied sufficiently in literature sources [16–20].

The Ag_2S-PbS system is a quasi-binary section of the Ag_2S-Ga_2S_3-PbS quasi-ternary system and belongs to the simple eutectic type. The eutectic point coordinates are 26 mol% PbS and T = 870 K. Under the influence of lead sulfide, the temperature of $(Ag_2S)_I \leftrightarrow (Ag_2S)_{II}$ and $(Ag_2S)_{II} \leftrightarrow (Ag_2S)_{III}$ phase transitions decreases (with eutectoid character) and occurs at 735 and 451 K, respectively. Based on Ag_2S and PbS, 1.0 and 2 mol% solid solution regions are formed, respectively [16].

The Ag_2S-Ga_2S_3 system was thoroughly studied by the authors [17–20]. The researches have determined that the Ag_2S-Ga_2S_3 system is quasibinary and characterized by the formation of ternary thiogallate compounds: Ag_9GaS_6, $AgGaS_2$ and $Ag_2Ga_{20}S_{31}$ [19; 20]. Ag_9GaS_6 melts congruently at 1063 K, forms eutectic equilibria with Ag_2S and $AgGaS_2$: $L \leftrightarrows \alpha\text{-}Ag_2S + Ag_9GaS_6$ and $L \leftrightarrows Ag_9GaS_6 + AgGaS_2$. The eutectic point coordinates are: 5 mol% Ga_2S_3 and T=1018 K, 15 mol% Ga_2S_3 and T = 1038 K. High-temperature modification of Ag_9GaS_6 compound crystallizes in a cubic structure (a = 10.798A^0), whereas low-temperature modification crystallizes in an orthorhombic structure (a = 10.777, b = 7.706, c = 7.605 A^0) [19].

The $AgGaS_2$ compound also melts without decomposition at 1268 K and crystallizes in a hexagonal syngony (a = 5.7544, c = 10.299 A^0, space group $I\bar{4}2d$). The third compound in the system $Ag_2Ga_{20}S_{31}$ is formed by the peritectic reaction at 1268K $L + Ga_2S_3 \leftrightarrows Ag_2Ga_{20}S_{31}$ and together with $AgGaS_2$ creates the eutectic at 1188 K [20].

The PbS-Ga_2S_3 system represents a quasibinary section of the Pb-Ga-S ternary system and is characterized by the formation of $PbGa_2S_4$ and $Pb_2Ga_2S_5$ compounds. Both compounds melt congruently at 1163 and 1063 K, respectively. Both ternary compounds crystallize in the orthorhombic system.

Experimental part

The synthesis of quaternary alloys was carried out in an evacuated quartz ampoule using pre-synthesized melts (Ag_2S, PbS, Ga_2S_3) and high-purity elements at a temperature of 1100–1400 K. After melting the samples were gradually cooled down to 650–800 K at a rate of 10–15 degrees per hour, and followed by homogenization under the condition of 300–400h. After that, physicochemical analysis methods were applied to the studied samples.

The homogenized alloys of the Ag_2S-Ga_2S_3-PbS quasi-ternary system were studied using differential thermal analysis (DTA), X-ray fluoresence (RFA) and microstructure analysis (MSA), along with methods for determining the density and microhardness of the alloys. The DTA analysis of the alloys was performed using a NETZSCH 404 F1 Pegasus device with a heating rate of 10 K/min, up to maximum temperature of ~1400 K. Powder X-ray difraction measurements were taken on a D2 Phaser radiometer (CuK_α lamp, Ni filter), microhardness was measured with PMT-3 device, and microstructural studies were conducted using a MIM microscope. The density was determined by the pycnometric method with toluene as a filling ligating fluid.

Results and discussion

It should be noted in advance that there is no available literature data on the Ag_2S-Ga_2S_3-PbS quasi-ternary system and it has been studied for the first time by us. Below is an overview of the quasi-binary and partially quasi-ternary section of the system.

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The $AgGaS_2$-$PbGa_2S_4$ section is a quasi-binary section of the quasi-ternary system with the eutectic characteristics [21]. The liquid of the system consists of solid solutions formed on he basis of $AgGaS_2$ and $PbGa_2S_4$ which crystallize along the initial crystallization curves α and β and solidify at the eutectic point. (Fig. 1.). The eutectic point coordinates are 45 mol% $AgGaS_2$ 1100 K. The lattice parameters of solid solutions formed on the basis of $AgGaS_2$ vary as follows: a = 5.754–6.042, c = 10.26÷10.34 A^0, d = 4.60÷4.75 g/cm^3.

σ-Solid solutions based on $PbGa_2S_4$ cover the solidity interval of 82–100 mol% $PbGa_2S_4$. These solid solutions crystallize in the rhombic structure: a = 20.44÷20.72, b = 20.64÷20.90, c = 12.09÷12.24 A^0 f.q. Fddd2, Z = 32, d = 4.94÷4.80g/cm^3.

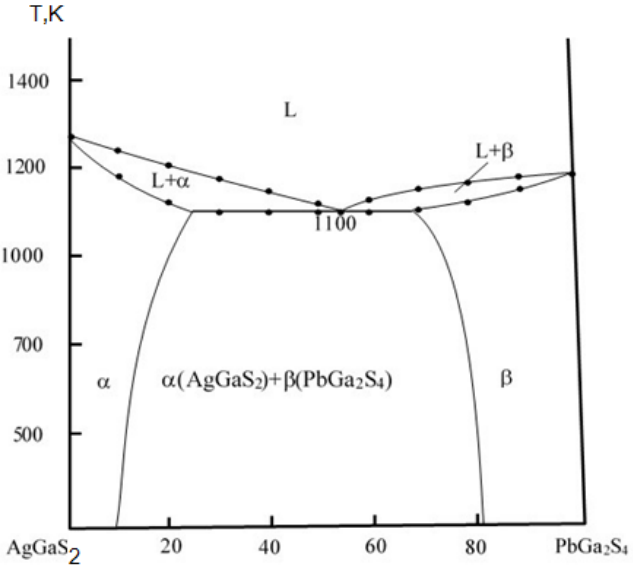

Fig. 1. Phase diagram of the $AgGaS_2$-$PbGa_2S_4$ system [21]

The $AgGaS_2$-$Pb_2Ga_2S_5$ section is quasi-binary, simple eutectic type. Coordinates of the eutectic point are 45 mol% $AgGaS_2$, T=950 K [22] (Fig. 2.). According to the results of the microstructure and X-ray analysis, 12 mol% of $AgGaS_2$ (ε-solid solutions) and 13 mol% of $Pb_2Ga_2S_5$ (σ-solid solutions) are formed. Phases with ε-type variable composition crystallize in chalcopyrite type (a = 5.754÷5.804; c = 10.299÷10.328 A^0). $(Pb_2Ga_2S_5)1\text{-}x(AgGaS_2)x$ type σ-solid solutions crystallize in rhombic syngonia (a = 12.38÷12.30; b = 11.90÷11.84; c = 11.03÷10.90 A^0) .

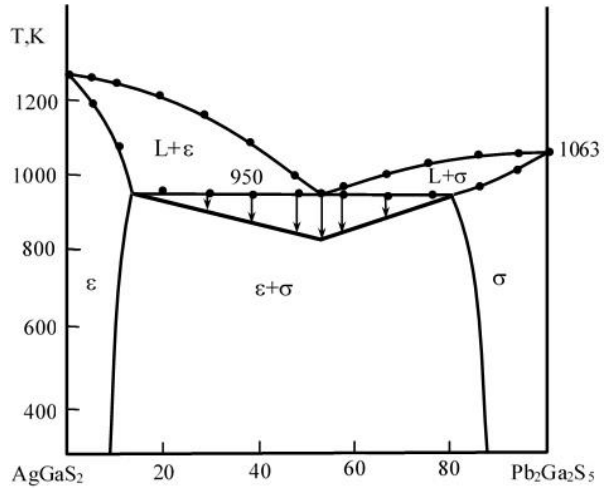

Fig. 2. Phase diagram of the $AgGaS_2$-$Pb_2Ga_2S_5$ system [22]

The $AgGaS_2$-PbS section is characterized by the formation of a quadruple compound with $AgPb_2GaS_4$ composition (Fig. 1). The $AgPb_2GaS_4$ compound is formed by the peritectic reaction L+β(PbS)⇆$AgPb_2GaS_4$ and melts decomposing at 1225 K temperature (Fig. 3.).

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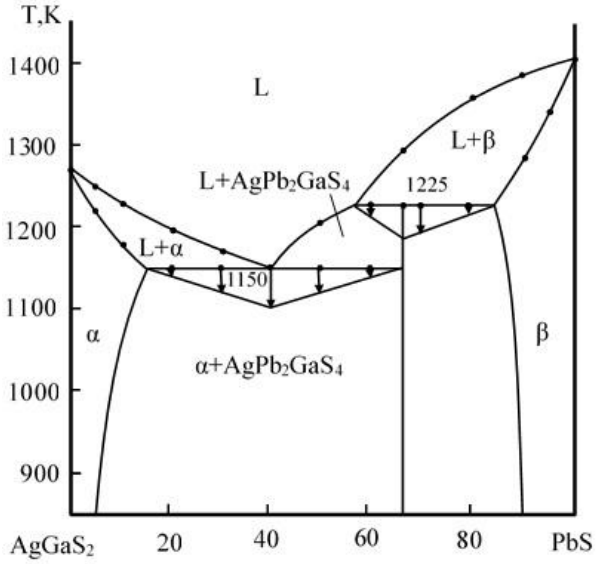

Fig. 3. Phase diagram of $AgGaS_2$-PbS system

The $AgPb_2GaS_4$ compound silver thiogallate is $AgGaS_2$ eutectic equilibria with (40 mol% PbS, T = 1150 K). 5 mol% PbS based on $AgGaS_2$ and 10 mol% $AgGaS_2$ based on PbS form a variable composition phase. According to the results of radiographic analysis, $AgPb_2GaS_4$ compound crystallizes in orthorhombic syngonia (a = 8.20, b = 6.84, c = 6.62 A^0).

The Ag_9GaS_6-$AgPb_2GaS_4$ cross section is a partial quasi-binary cross section of the quasi-ternary system [23]. Since the $AgPb_2GaS_4$ compound melts by decomposing, it behaves as a non-quasi-binary section in the range of 70–100 mol% $AgPb_2GaS_4$ and at temperatures up to 1180 K (Fig. 4.).

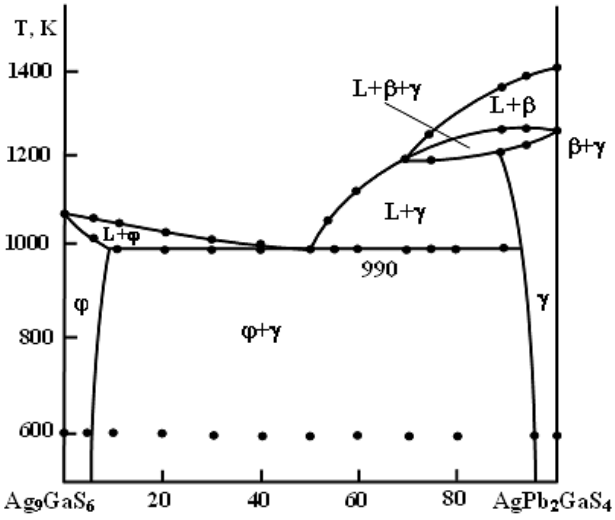

Fig. 4. Phase diagram of the Ag_9GaS_6-$AgPb_2GaS_4$ system [23]

Three-phase alloys (L+β+γ) crystallize together in the subsolidus in the indicated interval of the system. Two phases (φ+γ) crystallize together in the solidus of the system. Eutectic equilibrium occurs at 50 mol% Ag_9GaS_6 and T = 990 K. Substitution-type solid solutions are formed based on the component in a limited interval.

The Ag_9GaS_6-PbS crosssection is a quasi-binary of the Ag_2S-Ga_2S_3-PbS quasi-ternary system, of eutectic type [23]. The eutectic point coordinates are : 45 mol% PbS and T = 950 K. based on Ag_9GaS_6. 5mol%, on PbS – 8.5 mol% solid solution is formed. (Fig. 5.).

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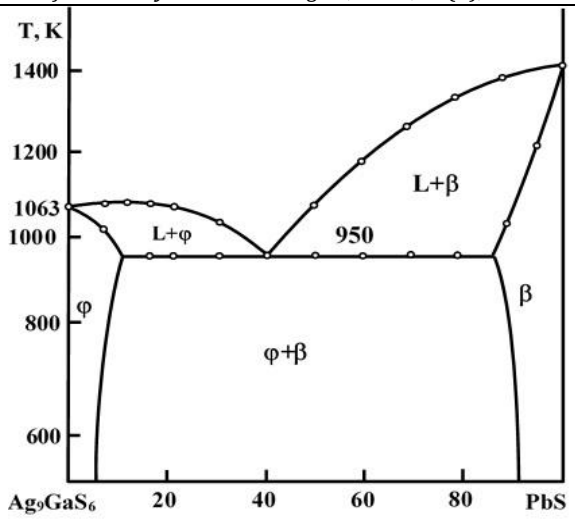

Fig. 5. Phase diagram of Ag_9GaS_6-PbS system [23]

The $AgPb_2GaS_4$-$Pb_2Ga_2S_5$ system being a partial quasi-binary section of the quasi-ternary system behaves as a quasi-binary, in the solidity interval of 100–42 mol% $Pb_2Ga_2S_5$ and below 970 K (Fig. 6.).

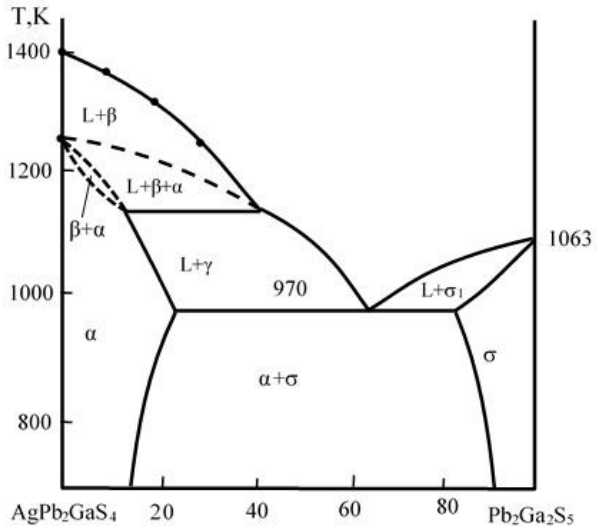

Fig. 6. Phase diagram of the $AgPb_2GaS_4$-$Pb_2Ga_2S_5$ system

The system is non-quasi-binary above 1125 K and in the range of 0–42 mol% $Pb_2Ga_2S_5$. In this part, first L+β, L+β+α, and then β+α and L+γ phases crystallize. Two phases α+σ crystallize together in solidus (Fig. 6.). The liquidus of the system β(PbS) consists of initial crystallization curves, α($AgPb_2GaS_4$) and σ_1($Pb_2Ga_2S_5$) phases, intersecting at 65 mol% $Pb_2Ga_2S_5$ content and temperature of 970 K. Based on $AgPb_2GaS_4$ 14 mol% and based on $Pb_2Ga_2S_5$ 10 mol% a variable composition phase is formed.

The $Ag_2Ga_{20}S_{31}$-$PbGa_2S_4$ section is also a partial quasi-binary section, characterized by obtaining a limited solubility area based only on the initial component $Ag_2Ga_{20}S_{31}$ (Fig. 7.). The section is quasi-binary only at the solidus. The peritectic reaction L+μ(Ga_2S_3)⇆æ($Ag_2Ga_{20}S_{31}$) occurs at temperatures above 1085 K. The eutectic e_{14} contains 65 mol% $PbGa_2S_4$ and crystallizes at 1085 K. æ($Ag_2Ga_{20}S_{31}$) and σ_2 ($PbGa_2S_4$) phases crystallize together in solidus in the range of 10–100 mol% $PbGa_2S_4$. A 10 mol% solid solution is formed based on $Ag_2Ga_{20}S_{31}$. The solution limit is not set on the basis of PbS.

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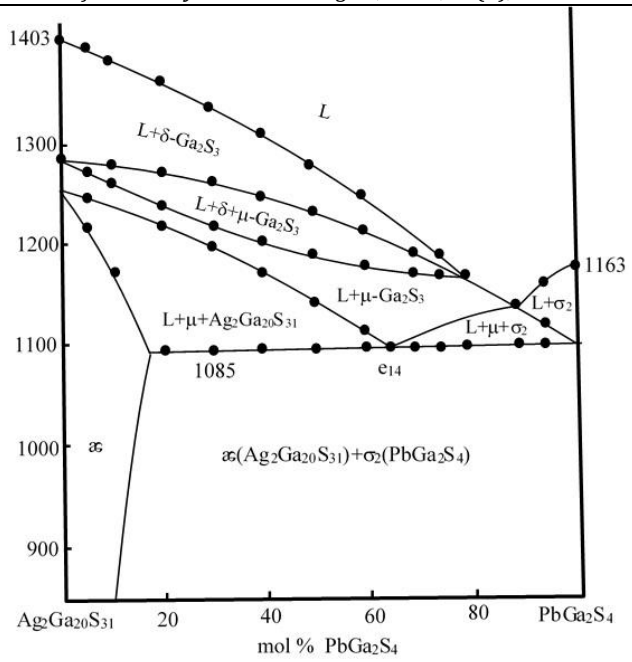

Fig. 7. Phase diagram of the $Ag_2Ga_{20}S_{31}$-$PbGa_2S_4$ system

The coordinates of invariant points in the indicated systems are presented in the table.

Table

Invariant points in the quasi-binary and partially quasi-binary sections of the Ag_2S-Ga_2S_3-PbS quasi-ternary system

Section	Invariant point	T,K	Reaction
$AgGaS_2$-$PbGa_2S_4$	eutectic	1100	L⇆α+β
$AgGaS_2$-$Pb_2Ga_2S_5$	eutectic	950	L⇆ε+σ
$AgGaS_2$-PbS	peritectic	1225	L+PbS⇆$AgPb_2GaS_4$
	eutectic	1150	L⇆ε+γ($AgPb_2GaS_4$)
Ag_9GaS_6- $AgPb_2GaS_4$	eutectic	990	L⇆φ+Υ($AgPb_2GaS_4$)
Ag_9GaS_6-PbS	eutectic	950	L⇆ φ +β
$AgPb_2GaS_4$- $Pb_2Ga_2S_5$	eutectic	970	L⇆γ+σ
$Ag_2Ga_{20}S_{31}$ - $PbGa_2S_4$	eutectic	1085	L⇆æ+$σ_2$

4 quasi and 3 partial quasi-binary sections were studied on the Ag_2S-Ga_2S_3-PbS quasi-ternary system. Based on quasi-binary sections a quasi-ternary system is triangulated. (Fig. 8.)

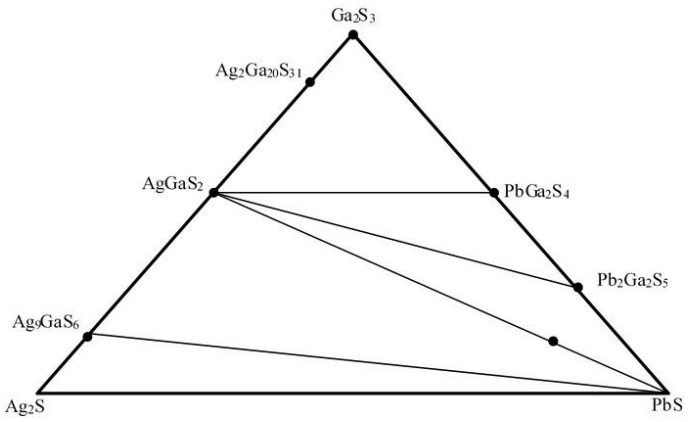

Fig. 8. Triangulation of the Ag_2S-Ga_2S_3-PbS ternary system

It has been established that the quasi-ternary system is triangulated into 5 subsystems:$AgGaS_2$-Ga_2S_3-$PbGa_2S_4$, $AgGaS_2$-$PbGa_2S_4$-$Pb_2Ga_2S_5$, $AgGaS_2$-PbS-$Pb_2Ga_2S_5$, Ag_9GaS_6-PbS-$AgGaS_2$, Ag_2S-Ag_9GaS_6-PbS.

The triangulation of the quasi-ternary system and its quasi-binary sections allowed to determine the composition of the alloy to grow mono crystals of high-temperature melting compounds such as PbS, Ag_2S, Ga_2S_3, $AgGaS_2$, $AgPb_2GaS_4$.

Conclusion

The phase equilibrium in the Ag_2S-Ga_2S_3-PbS quasi-ternary system was studied for the first time using the research methods of complex physicochemical analysis. As a result of the research, it was determined that the four sections of the quasi-ternary system are quasi-binary, and their T-x phase diagrams were constructed. It was found that the ternary system has limited solubility areas based on the four-component compound of the composition $AgPb_2GaS_4$ and initial components.

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