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PHASE EQUILIBRIUM IN QUASI-TERNARY SYSTEM

 $\text{InAs}_2\text{SSe}_3\text{--InAs}_2\text{S}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$

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Phase equilibrium in the systems $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{SSe}_3$, $\text{InAs}_2\text{SSe}_3\text{--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $\text{InAs}_2\text{S}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $\text{InAs}_2\text{S}_2\text{Se}_2\text{--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $(\text{InAs}_2\text{S}_3\text{Se})_{0.70}(\text{In}_3\text{As}_2\text{S}_3\text{Se}_3)_{0.30}$ (e_4)– $(\text{InAs}_2\text{SSe}_3)_{0.60}(\text{In}_3\text{As}_2\text{S}_3\text{Se}_3)_{0.40}$ (e_3) were studied using the methods of physico-chemical analysis (DTA, X-ray phase, MSA, measurement of microhardness and determination of density) and state diagram was plotted. It was established that except the latter, all are quasi-binary and belong to eutectic type. The system $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{SSe}_3$ is characterized by the formation of a new quaternary compound $\text{InAs}_2\text{S}_2\text{Se}_2$ melting congruently at 615 K. Liquidus surface projection of quasi-ternary system $\text{InAs}_2\text{SSe}_3\text{--InAs}_2\text{S}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$ was constructed. Primary crystallization fields and coordinates of non- and monovariant equilibrium were determined.

Keywords: *system, liquidus, solidus, quasi-ternary, eutectics.*

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Layered crystals of indium selenide, sesquialteral sulfides and arsenous selenide (As_2S_3 , As_2Se_3) are prospective semiconducting compounds with photosensitive and acousto-optic properties [1–15]. Complexity of the composition of chalcogenides may considerably improve their physical characteristics. Therefore studying phase equilibrium in quasi-ternary system $\text{InAs}_2\text{SSe}_3\text{--InAs}_2\text{S}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$ is of not only scientific, but also practical interest. We have earlier studied quasi-binary sections of quaternary system As–In–S–Se [16–19].

Compounds $\text{InAs}_2\text{S}_3\text{Se}$ and $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$, found while studying quasi-binary section $\text{As}_2\text{S}_3\text{--InSe}$ melt congruently at 643 and 1148 K correspondingly [20]. $\text{InAs}_2\text{S}_3\text{Se}$ is crystallized in monoclinic syngony ($a = 12.31$, $b = 11.43$, $c = 6.62$ Å, $\beta = 107^\circ$, $Z = 3$, $\rho = 4.66$ g/cm³, $H_\mu = 1240$ MPa), but $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ is crystallized in tetragonal syngony ($a = 9.36$, $c = 6.15$ Å, $Z = 2$, $\rho = 4.75$ g/cm³, $H_\mu = 850$ MPa) [21].

The work is aimed at constructing liquidus surface projection of quasi-ternary system $\text{InAs}_2\text{SSe}_3\text{--InAs}_2\text{S}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$ and studying physico-chemical properties of compounds and phases of variable composition.

Methods

We used high-purity reagents for synthesis: arsenic – B3, indium – In-000, sulphur "High Purity" 16-5, selenium "High Purity" B5. Quaternary alloys were synthesized from addition alloys $\text{InAs}_2\text{SSe}_3$, $\text{InAs}_2\text{S}_3\text{Se}$ and

$\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$, preliminarily produced from binary chalcogenides InSe, As_2S_3 and As_2Se_3 in evacuated and sealed quartz ampoules at 600–1000 K depending on the composition. After completion of synthesis cast samples were annealed at 450–500 K for two weeks.

Annealed and homogenized alloys were studied using the methods of DTA (NTR-73 with accuracy of 3–5 K, chromel-alumel thermocouple; calcined Al_2O_3 was used as standard, heating rate is 9 grade/min), X-ray phase analysis (D2 PHASER Bruker, CuK_α -radiation, Ni-filter), MSA (MIM-7, mixture of conc. $\text{KOH} + \text{C}_2\text{H}_5\text{OH} = 2:1$ as an etching agent, etching time 10–15 s). Microhardness of phases was measured on microhardness tester PMT-3 with accuracy of 5%, density of samples was determined using picnometer method.

Results and discussion

Phase diagram of the system $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{SSe}_3$ constructed according to the data of physico-chemical analysis is presented in Figure 1. As Figure 1 shows, the section $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{SSe}_3$ is quasi-binary section of quasi-ternary system $\text{InSe--As}_2\text{S}_3\text{--As}_2\text{Se}_3$ and is characterized by the formation of congruently melting compound of the composition $\text{InAs}_2\text{S}_2\text{Se}_2$, as well as limited solubility based on primary compounds.

Compound $\text{InAs}_2\text{S}_2\text{Se}_2$ divides the system $\text{InAs}_2\text{SSe}_3\text{--InAs}_2\text{S}_3\text{Se}$ into two secondary systems: $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{S}_2\text{Se}_2$ and $\text{InAs}_2\text{S}_2\text{Se}_2\text{--InAs}_2\text{SSe}_3$.

Fig.1. Phase diagram of the system $\text{InAs}_2\text{S}_3\text{Se}-\text{InAs}_2\text{S}_2\text{Se}_3$.

Fig.2. Phase diagram of the system $\text{InAs}_2\text{S}_2\text{Se}_3-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$.

As Figure 1 shows, the secondary systems belong to eutectic type.

Coordinates of eutectic points are 45 mol% $\text{InAs}_2\text{S}_2\text{Se}_3$, $T=580$ K and 60 mol% $\text{InAs}_2\text{S}_2\text{Se}_3$, $T=548$ K. Solubility based on $\text{InAs}_2\text{S}_3\text{Se}$ at 400 K is found to be 5 mol% $\text{InAs}_2\text{S}_2\text{Se}_3$, on the basis of $\text{InAs}_2\text{S}_2\text{Se}_3$ it is 10 mol% $\text{InAs}_2\text{S}_3\text{Se}$. Compound $\text{InAs}_2\text{S}_2\text{Se}_2$ melts at 608 K congruently and is formed due to partial substitution of selenium in InAs_2Se_4 with sulphur atoms. Liquidus of the system $\text{InAs}_2\text{S}_3\text{Se}-\text{InAs}_2\text{S}_2\text{Se}_3$ consists of three branches of primary crystallization of phases. α -phase (solid solutions based on $\text{InAs}_2\text{S}_3\text{Se}$), $\text{InAs}_2\text{S}_2\text{Se}_2$ and β -phase (solid solutions based on $\text{InAs}_2\text{S}_2\text{Se}_3$).

System $\text{InAs}_2\text{S}_2\text{Se}_3-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$. Primary compounds melt congruently at 1118 and 1148 K, correspondingly. Compound $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ is crystallized in tetragonal syngony with lattice parameters of $a=9.36$. $c=6.16$ Å, $Z=2$, $\rho=4.75$ g/cm³, compound $\text{InAs}_2\text{S}_2\text{Se}_3$ has a density of $\rho=5.46$ g/cm³, microhardness $H\mu=1040$ MPa.

According to physico-chemical analysis $T-x$ diagram of the system $\text{InAs}_2\text{S}_2\text{Se}_3-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ (Figure 2) was constructed. It was defined that it belongs to eutectic type. Coordinates of eutectic point: 40 mol% $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ and $T=715$ K.

Crystallization of alloys of the composition 45–100 mol% $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ begins with

extraction of β -solid solution basis on the of $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$. Primary crystallization region of α – solid solutions on the basis of $\text{InAs}_2\text{S}_2\text{Se}_3$ corresponds to 0–45 mol% $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$. Lines of primary crystallization cross at 40 mol% $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$. Solubility on the base of $\text{InAs}_2\text{S}_2\text{Se}_3$ makes up 6 mol%, and on the basis of $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ reaches 13 mol%.

According to the data of MSA and X-ray phase analysis in concentration range of 6–87 mol% $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ two phases are crystallized (α and β).

The system $\text{InAs}_2\text{S}_3\text{Se}-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ is also quasi-binary and belongs to a simple eutectic type (Figure 3). Coordinates of eutectic point were defined by Tamman triangle construction: 30 mol. % $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ and $T=550$ K. Solubility on the basis of $\text{InAs}_2\text{S}_3\text{Se}$ at 400 K make up 5 mol% $\text{InAs}_2\text{S}_3\text{Se}$, but on the basis of $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ solubility reaches 15 mol% $\text{InAs}_2\text{S}_3\text{Se}$. According to the data of X-ray phase analysis β -solid solutions are crystallized in tetragonal syngony. Lattice cell parameters of β -solid solutions in homogeneity region changes in the range of $a = 9.36-9.54$; $c = 6.156-6.22$ Å, $Z=2$, $\rho=4.75-4.60$ g/cm³, $H\mu=850-975$ MPa.

Section $\text{InAs}_2\text{S}_2\text{Se}_2-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ (Figure 4) is a quasi-binary section of quasi-ternary system $\text{InAs}_2\text{S}_2\text{Se}_3-\text{InAs}_2\text{S}_3\text{Se}-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ and belongs to a simple eutectic type. Branches of

primary crystallization of α - and β -solid solutions cross at 25 mol% $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ and at 540 K (e_5). According to MSA data all alloys except 0–3 and 89–100 mol% $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$, are diphasic ones.

Section $(\text{InAs}_2\text{S}_3\text{Se})_{0.70}(\text{In}_3\text{As}_2\text{S}_3\text{Se}_3)_{0.30}$ (e_4)– $(\text{InAs}_2\text{S}_3\text{Se})_{0.60}(\text{In}_3\text{As}_2\text{S}_3\text{Se}_3)_{0.40}$ (e_3) is non-quasi-binary, crosses crystallization fields of two secondary systems: $\text{InAs}_2\text{S}_3\text{Se}$ – $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ – $\text{InAs}_2\text{S}_2\text{Se}_2$ (I) and $\text{InAs}_2\text{S}_2\text{Se}_2$ – $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ – $\text{InAs}_2\text{Se}_3\text{S}$ (II) (Figure 5). In the concentration range of 0–50 mol% $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ the section crosses secondary system (1), on three sides

eutectic transformation takes place, therefore alloys of this ternary system harden at temperature of ternary eutectics 500 K by the reaction $L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se} + \text{InAs}_2\text{S}_2\text{Se}_2 + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3$.

The composition 50 mol% $\text{InAs}_2\text{S}_2\text{Se}_2$ responds to the cross point of the section e_3 – e_4 with quasi-binary section $\text{InAs}_2\text{S}_2\text{Se}_2$ – $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$. Then the section passes through ternary system $\text{InAs}_2\text{S}_2\text{Se}_2$ – $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ – $\text{InAs}_2\text{Se}_3\text{S}$, non-variant eutectic reaction occurs in this part of the section at 515 K:

Fig.3. Phase diagram of the system $\text{InAs}_2\text{S}_3\text{Se}$ – $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$.

Fig.4. Phase diagram of the system $\text{InAs}_2\text{S}_2\text{Se}_2$ – $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$.

Fig.5. Phase diagram of the system e_4 – e_3 .

Liquidus of the section of this part of diagram consists of two primary crystallization branches of phases $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ and $\text{InAs}_2\text{S}_3\text{Se}_3$.

Liquidus surface projection of subsystem $\text{InAs}_2\text{S}_3\text{Se}-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3-\text{InAs}_2\text{S}_3\text{Se}_3$ was constructed according to state diagram (Figure 6). Liquidus surface projection of quasi-ternary system consists of 4 primary crystallization fields of phases. The most extensive regions of liquidus surface are the fields of $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ (3), $\text{InAs}_2\text{S}_3\text{Se}_3$ (1) and $\text{InAs}_2\text{S}_3\text{Se}$ (4).

In quasi-ternary system $\text{InAs}_2\text{S}_3\text{Se}-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3-\text{InAs}_2\text{S}_3\text{Se}_3$ there are 5 curves of monovariant equilibrium and 7 non-variant equilibrium points, two of which are the points of ternary eutectics 5 (Table).

Thus, liquidus surface projection of quasi-ternary system $\text{InAs}_2\text{S}_3\text{Se}-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3-\text{InAs}_2\text{S}_3\text{Se}_3$ was constructed for the first time. Formation of one ternary compound of anion-substituted type and solid solution regions based on primary components was established.

Fig. 6. Liquidus surface projection of the $\text{InAs}_2\text{S}_3\text{Se}-\text{InAs}_2\text{S}_3\text{Se}_3-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$ quasi-ternary system. Primary crystallization regions: 1 – $\text{InAs}_2\text{S}_3\text{Se}_3$, 2 – $\text{InAs}_2\text{S}_2\text{Se}_2$, 3 – $\text{In}_3\text{As}_2\text{S}_3\text{Se}_3$, 4 – $\text{InAs}_2\text{S}_3\text{Se}$.

Mono- and nonvariant equilibria in the $\text{InAs}_2\text{S}_3\text{Se}-\text{In}_3\text{As}_2\text{S}_3\text{Se}_3-\text{InAs}_2\text{S}_3\text{Se}_3$ quasi-ternary system

Equilibrium curve and point	Reaction	Temperature, K
e_1	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se} + \text{InAs}_2\text{S}_2\text{Se}_2$	580
e_2	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se}_2 + \text{InAs}_2\text{S}_3\text{Se}_3$	548
e_3	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se}_3 + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3$	600
e_4	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se} + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3$	550
e_5	$L \leftrightarrow \text{InAs}_2\text{S}_2\text{Se}_2 + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3$	540
E_1	$L \leftrightarrow \text{InAs}_2\text{S}_2\text{Se}_2 + \text{InAs}_2\text{S}_3\text{Se} + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3$	500
E_2	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se}_3 + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3 + \text{InAs}_2\text{S}_2\text{Se}_2$	515
e_3E_2	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se}_3 + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3$	600–515
e_2E_2	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se}_3 + \text{InAs}_2\text{S}_2\text{Se}_2$	548–515
$E_2e_5E_1$	$L \leftrightarrow \text{InAs}_2\text{S}_2\text{Se}_2 + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3$	515–540–500
e_4E_1	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se} + \text{In}_3\text{As}_2\text{S}_3\text{Se}_3$	550–500
e_1E_1	$L \leftrightarrow \text{InAs}_2\text{S}_3\text{Se} + \text{InAs}_2\text{S}_2\text{Se}_2$	580–500

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$\text{InAs}_2\text{SSe}_3\text{--InAsS}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$ KBAZIÜÇLÜ SİSTEMİNDƏ FAZA TARAZLIĞI

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Fiziki-kimyəvi analiz metodları (DTA, RFA, MSA, mikrobərkliyin və sıxlığın ölçülməsi) ilə $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{SSe}_3$, $\text{InAs}_2\text{SSe}_3\text{--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $\text{InAsS}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $\text{InAs}_2\text{S}_2\text{Se}_3\text{--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $(\text{InAs}_2\text{S}_3\text{Se})_{0.70}(\text{In}_3\text{As}_2\text{S}_3\text{Se}_3)_{0.30}$ (e_4)– $(\text{InAs}_2\text{SSe}_3)_{0.60}(\text{In}_3\text{As}_2\text{S}_3\text{Se}_3)_{0.40}$ (e_3) sistemlərində faza tarazlığı öyrənilmiş və hal diaqramı qurulmuşdur. Müəyyən edilmişdir ki, sonuncu istisna olmaqla, hamısı kvazibinar olub, evtektik tiplidirlər. $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{SSe}_3$ sistemi 615 K temperaturda konqruent əriyən və tərkibi $\text{InAs}_2\text{S}_2\text{Se}_2$ olan yeni dördlü birləşmənin əmələ gəlməyi ilə xarakterizə olunur. $\text{InAs}_2\text{SSe}_3\text{--InAsS}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$ kvaziüçlü sisteminin likvidus səthinin proyeksiyası qurulmuş, fazanın ilkin kristallaşma sahələri, mono- və nonvariant tarazlıqların koordinatları təyin edilmişdir.

Açar sözlər: sistem, likvidus, solidus, kvaziüçlü, evtektika.

ФАЗОВЫЕ РАВНОВЕСИЯ В КВАЗИТРОЙНОЙ СИСТЕМЕ $\text{InAs}_2\text{SSe}_3\text{--InAs}_2\text{S}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$ **Р.С.Магомедрагимова, И.И.Алиев, О.М.Алиев, К.Н.Бабанлы**

Методами физико-химического анализа (ДТА, РФА, МСА, измерением микротвердости и определением плотности) изучены фазовые равновесия в системах $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{SSe}_3\text{S}$, $\text{InAs}_2\text{SSe}_3\text{--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $\text{InAs}_2\text{S}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $\text{InAs}_2\text{S}_2\text{Se}_2\text{--In}_3\text{As}_2\text{S}_3\text{Se}_3$, $(\text{InAs}_2\text{S}_3\text{Se})_{0.70}(\text{In}_3\text{As}_2\text{S}_3\text{Se}_3)_{0.30}$ (e_4)– $(\text{InAs}_2\text{SSe}_3)_{0.60}(\text{In}_3\text{As}_2\text{S}_3\text{Se}_3)_{0.40}$ (e_3) и построены диаграммы состояния. Установлено, что кроме последнего, все они квазибинарные и относятся к эвтектическому типу. Система $\text{InAs}_2\text{S}_3\text{Se--InAs}_2\text{SSe}_3$ характеризуется образованием нового четверного соединения $\text{InAs}_2\text{S}_2\text{Se}_2$, плавящегося конгруэнтно при температуре 615 К. По полученным данным построена проекция поверхности ликвидуса квазитройной системы $\text{InAs}_2\text{SSe}_3\text{--InAs}_2\text{S}_3\text{Se--In}_3\text{As}_2\text{S}_3\text{Se}_3$. Определены поля первичной кристаллизации и координаты моно- и невариантных равновесий.

Ключевые слова: система, ликвидус, солидус, квазитройная, эвтектика.