UDC 541.123/.123.8/9:546.57'81'86/23

PHASE EQUILIBRIA IN THE Ag_2Se-PbSe-$AgSbSe_2$ SYSTEM

[1]Sh.H. Mansimova, [2]K.N. Babanly, [2]L.F. Mashadiyeva, [1]R.J. Mirzoyeva, [2]M.B. Babanly

[1]Baku State University
Z. Khalilov, 23, AZ-1148, Baku, Azerbaijan
[2]Institute of Catalysis and Inorganic Chemistry National Academy of Sciences of Azerbaijan
113, H.Javid Ave., AZ-1143, Baku, Azerbaijan; e-mail: leylafm76@gmail.com

Received 16.12.2018

The work presents results of the study into phase equilibriums in the Ag_2Se-PbSe-$AgSbSe_2$ through the methods of differential thermal and X-ray diffraction analyses. Based on the experimental data, polythermic sections of $AgSbSe_2$-$(PbSe)_{0,5}(Ag_2Se)_{0,5}$ and Ag_2Se-$(PbSe)_{0,5}(AgSbSe_2)_{0,5}$, isothermal section at 300 K of the phase diagram, as well as projection of the liquidus surface were constructed. It showed that the system cited above is a quasi-ternary plane of the Ag-Pb-Sb-Se quaternary system characterized by the presence of non-invariant transition equilibrium. The fields of primary crystallization of phases, types and coordinates of non- and monovariant equilibria were determined. A wide range (~80 mol%) of solid solutions based on $AgSbSe_2$ along the PbSe-$AgSbSe_2$ section was revealed.

Keywords: *phase diagram, solid solutions, liquidus surface, silver-antimony selenide, lead monoselenide.*

Doi.org/10.32737/2221-8688-2019-1-41-49

INTRODUCTION

In recent decades, the study of thermoelectric materials has attracted increasing attention both from an energy and environmental point of view. In addition, the development of highly efficient thermoelectric devices for heat waste recovery systems can bring enormous economic benefits [1-5]. Recent studies have shown that Ag-B^V-X and Ag-A^{IV}-B^V-X (where A^{IV}-Sn, Pb; B^V-Sb, Bi; X-S, Se, Te) alloys show high ZT values of thermoelectric figure of merit [6-10]. In particular, ternary compounds with the general formula AgB^VX_2 attract the attention of researchers due to their thermoelectric, optical, and electronic properties [11–15]. The functional properties of such materials can be improved using such processes as doping, obtaining of related solid solutions and composites. And this, in turn, is based on the study of phase equilibria in the corresponding systems [16-18].

Earlier, we carried out complex studies of phase equilibria and thermodynamic properties of the Ag_2Se–PbSe-Bi_2Se_3 [19], Ag_2Te–PbTe-Bi_2Te_3 [20], Ag_2Te–SnTe-Bi_2Te_3 [21] and Ag_2Te–SnTe-Sb_2Te_3 [22, 23] systems where wide areas of solid solutions along $A^{IV}X$-AgB^VX_2 sections are revealed.

This paper presents new experimental data on phase equilibria in the quasi-ternary Ag_2Se-PbSe-$AgSbSe_2$ system.

All the initial components of the title system, which are semiconductors [24], have been studied quite well. Lead monoselenide melts congruently at 1354 K [25] and crystallizes in a NaCl-type crystal lattice (Sp.gr. *Fm*3*m*) with the unit cell parameter a = 6.1243Å [26].

Silver selenide is characterized by polymorphism and mixed electron-ion conductivity [27]. This compound melts congruently at 1170 K [28]. According to the authors of [29], the α→β phase transition in Ag_2Se occurs at 407.7±0.5 K. The low-temperature β-Ag_2Se phase crystallizes in an orthorhombic structure (Sp.gr. $P\ 2_12_12_1$) with cell parameters: a = 4.3359 Å, b = 7, 0700 Å, c = 7.7740 Å, Z = 4 [30]. The high-temperature

α-Ag_2Se phase forms crystals of the cubic system (Sp.gr. *I m3m*) with cell parameters *a* = 5.043 Å, Z = 2 [31].

The $AgSbSe_2$ compound also melts congruently at 908 K [32] and forms a cubic crystal lattice of the NaCl type (Sp.gr. *Fm3m*) with the parameter a = 5.786 Å [33].

The boundary quasi-binary systems of Ag_2Se-PbSe, Ag_2Se-$AgSbSe_2$ and PbSe-$AgSbSe_2$ have been studied in a number of works. The Ag_2Se-PbSe system has a simple eutectic type phase diagram. Eutectics melts at 933 K and contains 25 mol. % PbSe [34] (22 mol.% PbSe according to [35]).

The Ag_2Se-$AgSbSe_2$ system forms an eutectic-type phase diagram [31]. The eutectic melt contains 46 mol. % $AgSbSe_2$ and crystallizes at 813 K.

The PbSe-$AgSbSe_2$ system was recently studied in [36] and its phase diagram was constructed. It is shown that the system is quasi-binary and belongs to the peritectic type (Fig. 1). Peritectics has coordinates of 18 mol.% $AgSbSe_2$ and 1220 K. Solubility on the basis of $AgSbSe_2$ and PbSe at the peritectic temperature is 87 (γ-phase) and 5 mol% (β-phase), and at room temperature 80 and ~2 mol%, respectively. There is a minimum point (M) on the curves of the liquidus and solidus.

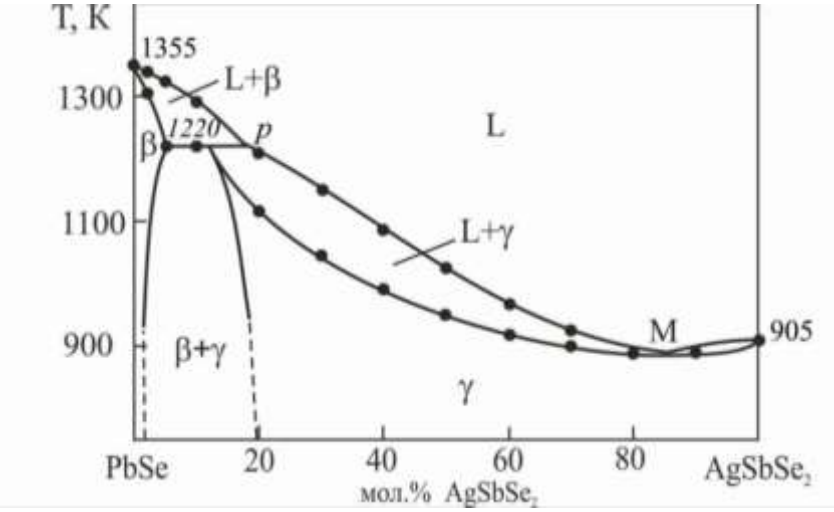

Fig.1. Phase diagram of the PbSe-$AgSbSe_2$ system [36]

EXPERIMENTAL PART

For the experiments, the initial compounds of the studied system of Ag_2Se, PbSe and $AgSbSe_2$ were obtained. The synthesis was carried out by fusing the corresponding elementary components in stoichiometric ratios in evacuated to ~10^{-2} Pa and sealed quartz ampoules at temperatures 50° above the melting points of the compounds. Simple substances from the company EVOCHEM ADVANCED MATERIALS GMBH (Germany) of high purity were used for the synthesis: silver in granules (Ag-00047; 99.999%), antimony in granules (Sb-00002; 99.999%), lead in granules (Pb-00005; 99.9995%), selenium in granules (Se-00002; 99.999%). Taking into account the high vapor pressure of selenium at the melting temperatures of binary selenides of Ag_2Se and PbSe, the synthesis of both compounds was performed in the two-zone mode. The hot zone temperature was 1150 K for Ag_2Se and 1390 K for PbSe, and the cold zone was 900 K, which is slightly below the boiling point of selenium (958 K) [37]. According to the recommendations of

the authors of [38], the ampoule with Ag_2Se melt was quenched from a temperature of 1100 K into cold water after the synthesis in order to obtain a uniform stoichiometric composition of this compound. All the synthesized compounds were checked by the methods of DTA and PXRD. The obtained values of melting points and lattice parameters for all synthesized compounds were close to the above literature data within the error (±3 K and ±0,0003 Å**).**

For experiments, a series of alloys along the $AgSbSe_2$-[A] and Ag_2Se-[B] sections (where [A] and [B] are alloys with composition $(PbSe)_{0,5}(Ag_2Se)_{0,5}$ and $(PbSe)_{0,5}(AgSbSe_2)_{0,5}$, respectively), as well as a number of additional alloys outside of them were prepared by melting the initial compounds under vacuum. Cast non-homogenized samples were annealed at 770 K (500 h) in order to achieve a state closest to the equilibrium state in alloys.

DTA was performed in range from room temperature to 1400 K with a heating rate of 10 K· min^{-1} on a NETZSCH 404 F1 PEGASUS SYSTEM differential scanning calorimeter. The measurement results were processed using the NETZSCH Proteus Software. The accuracy of temperature measurement was within ±2 K.

X-ray phase analysis was carried out at room temperature on a BRUKER D8 ADVANCE diffractometer with $CuK\alpha_1$ radiation. X-ray patterns were indexed using BRUKER TOPAS V3.0 Software.

RESULTS AND DISCUSSION

By means of joint processing of obtained experimental results and literature data on the boundary quasi-binary systems of Ag_2Se-PbSe [34,35], Ag_2Se-$AgSbSe_2$ [32] and PbSe-$AgSbSe_2$ [36], we obtained a mutually agreed picture of phase equilibria in the Ag_2Se-PbSe-$AgSbSe_2$ system.

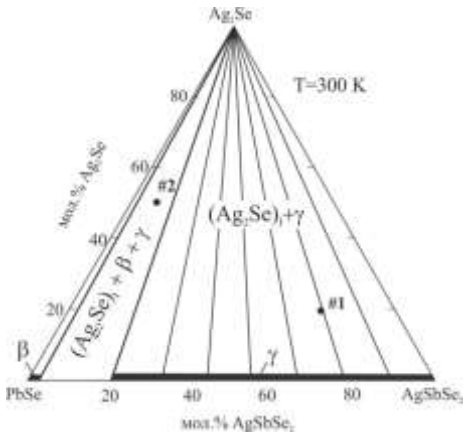

Fig. 2. Isothermal section at 300 K of the phase diagram of the Ag_2Se-PbSe-$AgSbSe_2$ system. # 1 and # 2 - alloys with powder X-ray patterns are presented in Fig.3.

Phase equilibria at room temperature. The obtained diagram of solid-phase equilibria (Fig. 2) in the Ag_2Se-PbSe-$AgSbSe_2$ system clearly shows the location of phase regions at 700 K. As can be seen, Ag_2Se forms connodes with the β- and γ-phases based on PbSe and $AgSbSe_2$, respectively. As a result, the concentration triangle is divided into two two-phase and one three-phase areas. The phase compositions of the alloys are confirmed by PXRD technique. For example, Fig. 3 shows powder diffraction patterns of two alloys: # 1 with a composition of 20

mol.% Ag_2Se, 62 mol.% $AgSbSe_2$, 18 mol.% PbSe and # 2 with a composition of 50 mol.% Ag_2Se, 6 mol.% $AgSbSe_2$, 44 mol.% PbSe. As can be seen from Figure 3, alloy # 1 consists of a two-phase mixture $(Ag_2Se)_I+\gamma$, and alloy # 2 consists of a three-phase mixture $(Ag_2Se)_I+\beta+\gamma$.

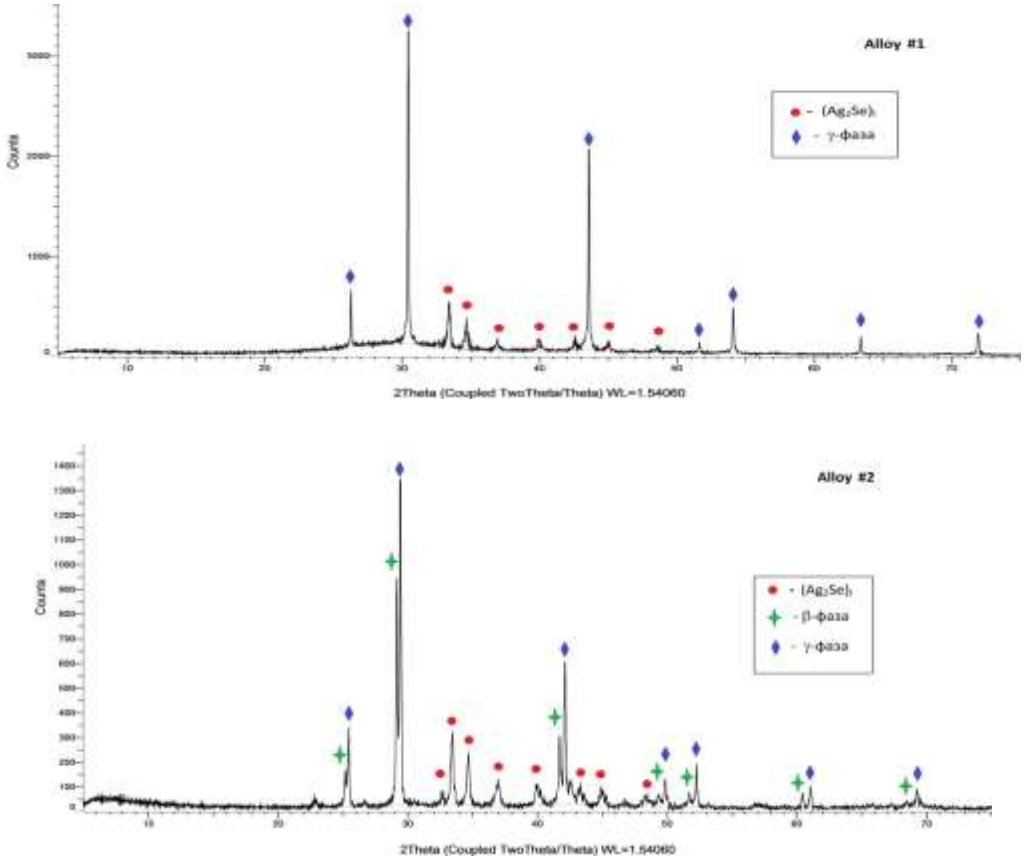

Fig. 3. Powder X-ray patterns of alloys # 1 (20 mol.% Ag_2Se, 62 mol.% $AgSbSe_2$, 18 mol.% PbSe) and # 2 (50 mol.% Ag_2Se, 6 mol.% $AgSbSe_2$, 44 mol.% PbSe)

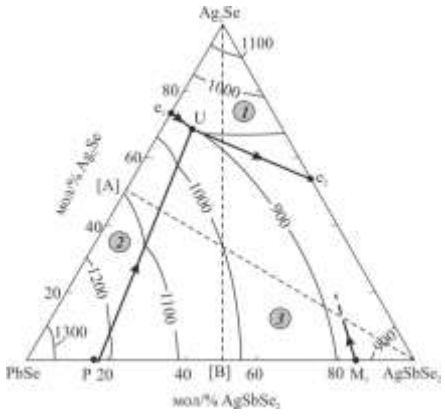

Fig. 4. Projection of the liquidus surface of the Ag_2Se-PbSe-$AgSbSe_2$system. Primary crystallization fields: 1 - α, 2 - β, 3 - γ. Dotted lines - cuts $AgSbSe_2$-[A] and Ag_2Se-[B]

Liquidus surface. The liquidus of the Ag_2Se-PbSe-$AgSbSe_2$ system (Fig. 4) consists of three fields of the primary crystallization of the α-, β- and γ-phases (respectively, fields 1, 2 and 3 on Fig. 4). The liquidus surface of the γ-phase ($AgSbSe_2$) has the greatest width. The fields of primary crystallization of phases are separated by one peritectic (*pU*) and two eutectic (*e_1U, Ue_2*) curves. All three curves converge at a transition point *U*, corresponding to the composition of the melt that is in invariant equilibrium L+β↔α+γ.

The types and coordinates of invariant equilibria, as well as temperature ranges of monovariant equilibria are listed in the Table.

Table. Non- and monovariant equilibria in the Ag_2Se-PbSe-$AgSbSe_2$ system

Point or curve in Fig.4	Equilibrium	Composition, mol%			T, *K*
		Ag_2Se	$AgSbSe_2$	PbSe	
e_1	L↔α+β	73	-	27	933
e_2	L↔α+γ	54	46	-	793
P	L+β↔γ	-	82	18	1220
M	L↔γ	-	85	15	890
U	L+β↔α+γ	70	8	22	910
e_1U	L↔α+β				933-910
pU	L+β↔γ				1220-910
e_2U	L↔α+γ				793-910

Polythermal sections. We analyzed and constructed two polythermal sections $AgSbSe_2$-[A] and Ag_2Se-[B].

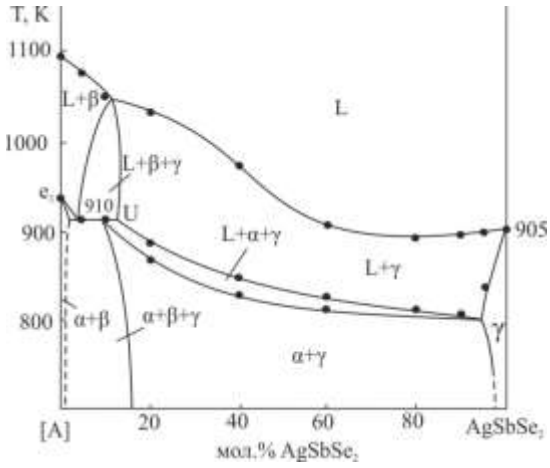

Fig. 5. Polythermal section $AgSbSe_2$-[A] of the phase diagram of the system Ag_2Se-PbSe-$AgSbSe_2$.

The $AgSbSe_2$-[A] section (Fig.5). The liquidus of this section consists of two branches corresponding to the primary crystallization of the β-phase based on PbSe and γ-phase based on $AgSbSe_2$. A flat minimum is observed on the liquidus curve of the γ-phase which is probably due to the presence of a minimum point on the PbSe–$AgSbSe_2$ boundary system. Curves below the liquidus correspond to monovariant peritectic

(*pU*) and eutectic (*e_1U; e_2U*) equilibria (Table, Fig.4). The horizontal line at 910 K characterizes the nonvariant transition equilibrium L+β↔α+γ (point *U* in Fig. 4). After completion of this process a three-phase

The Ag_2Se-[B] section (Fig. 6). This section passes through the primary crystallization fields of the α- and γ-phases. Then joint secondary crystallization of these phases occurs and as a result, a two-phase α+γ area is formed in a sub-solidus. The horizontal line at 397 K corresponds to the phase transition of the α-phase.

α+β+γ area is formed at the excess of β-phase. As for the excess of liquid, the transition reaction finished by the formation of the three-phase L+α+γ region.

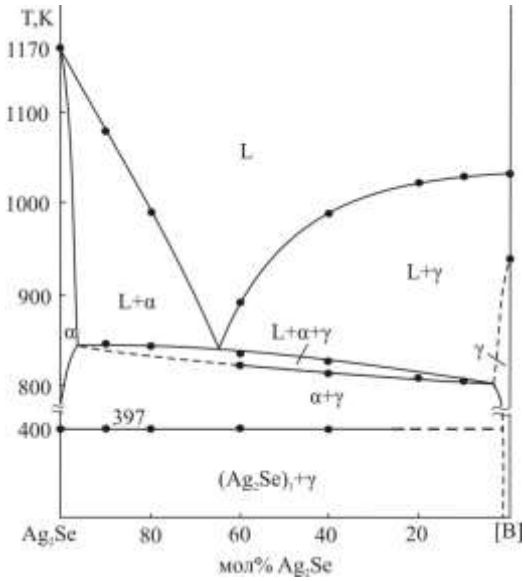

Fig. 6. Polythermal section Ag_2Se-[B] of the phase diagram of the Ag_2Se-PbSe-$AgSbSe_2$ system.

CONCLUSION

New experimental data on phase equilibria in the quasi-ternary Ag_2Se-PbSe-$AgSbSe_2$ system were obtained. Two polythermal sections, an isothermal section at 300 K of the phase diagram, and a liquidus surface projection were constructed. Broad range of solid solutions based on $AgSbSe_2$ (γ-phase) and insignificant solubility on the basis of Ag_2Se (α-phase) and PbSe (β-phase) were revealed in the system, and the fields of primary crystallization of these phases determined.

Acknowledgments

This work was supported by the Science Development Foundation under the President of the Republic of Azerbaijan – **Grant № EİF-BGM-4-RFTF-1/2017-21/11/4-M-12.**

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Ag_2Se-PbSe-$AgSbSe_2$ SİSTEMİNDƏ FAZA TARAZLIQLARI

[1]Ş.H. Mənsimova, [2]L.F. Məşədiyeva, [2]K.N. Babanlı[2],
[1]R.C. Mirzəyeva, [2]M.B. Babanlı

[1]Bakı Dövlət Universiteti
Az 1148 Bakı, Z.Xəlilov küç, 23; e-mail: info@bsu.az
[2]AMEA akademik M.Nağıyev adına Kataliz və Qeyri-üzvi Kimya İnstitutu
AZ 1143 Bakı, H.Cavid pr.113; e-mail: leylafm76@gmail.com

İşdə Ag_2Se-PbSe-$AgSbSe_2$ sistemində faza tarazlıqlarının differensial-termiki və rentgenfaza analizi üsulları ilə tədqiqinin nəticələri verilir. Təcrübi nəticələr əsasında sistemin faza diaqramının $AgSbSe_2$-$(PbSe)_{0,5}(Ag_2Se)_{0,5}$ və Ag_2Se-$(PbSe)_{0,5}(AgSbSe_2)_{0,5}$ politermik kəsikləri, 300 K-də izotermik kəsiyi və likvidus səthinin proyeksiyası qurulmuşdur. Göstərilmişdir ki, o, Ag-Pb-Sb-Se dördkomponentli sisteminin kvaziüçlü müstəvisidir və nonvariant keçid tarazlığı ilə xarakterizə olunur. Sistemdə fazaların ilkin kristallaşma sahələri, non- və monovariant tarazlıqların tipləri və koordinatları təyin edilmişdir. $AgSbSe_2$ birləşməsi əsasında PbSe-$AgSbSe_2$ kəsiyi boyunca geniş (~80 mol%) bərk məhlul sahəsi aşkar olunmuşdur.

Açar sözlər: *faza diaqramı, bərk məhlullar, likvidus səthi, gümüş-stibium selenidi, qurğuşun monoselenid.*

ФАЗОВЫЕ РАВНОВЕСИЯ В СИСТЕМЕ Ag_2Se-PbSe-$AgSbSe_2$

[1]Ш.Г. Мансимова, [2]Л.Ф. Машадиева, [2]К.Н. Бабанлы,
[1]Р.Дж. Мирзоева, [2]М.Б. Бабанлы

[1]Бакинский Государственный Университет
AZ 1073 Баку, ул. З. Халилова 23, e-mail: info@bsu.az
[2]Институт Катализа и Неорганической Химии им. акад. М. Нагиева
Национальной АН Азербайджана,
AZ 1143 Баку, пр.Г. Джавида,113; e-mail: leylafm76@gmail.com

В работе представлены результаты исследования фазовых равновесий в системе Ag_2Se-PbSe-$AgSbSe_2$ методами дифференциального термического и рентгенофазового анализов. На основании экспериментальных данных построены политермические сечения $AgSbSe_2$-$(PbSe)_{0,5}(Ag_2Se)_{0,5}$ и Ag_2Se-$(PbSe)_{0,5}(AgSbSe_2)_{0,5}$, изотермическое сечение при 300 K фазовой диаграммы, а также проекция поверхности ликвидуса. Показано, что данная система является квазитройной плоскостью четверной системы Ag-Pb-Sb-Se и характеризуется наличием нонвариантного переходного равновесия. Определены поля первичной кристаллизации фаз, типы и координаты нон- и моновариантных равновесий. Выявлена широкая область (~80 мол%) твердых растворов на основе $AgSbSe_2$ вдоль разреза PbSe-$AgSbSe_2$.

Ключевые слова: *фазовая диаграмма, твердые растворы, поверхность ликвидуса, селенид серебра-сурьмы, моноселенид свинца.*