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INFLUENCE OF PbTe ADDITION ON THE ELECTRIC CHARGE AND HEAT TRANSPORT IN AgSbSe$_2$

A.A. Saddinova[1], **R.I. Selim-zadeh**[1], **A.E. Babayeva**[1], **A.A. Orujova**[2]
[1]*Institute of Physics Ministry of Science and Education Republic of AzerbaijanAZ-1143, Azerbaijan*
[2]*Mingachevir State University, Az-4500, Azerbaijan*
**Corresponding Author email: a.saddinova@physics.science.az*

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The aim of this study is to investigate the influence of PbTe addition on the thermoelectric properties and band parameters of AgSbSe$_2$ at temperatures below room temperature. For this purpose, the polycrystalline (AgSbSe$_2$)$_x$(PbTe)$_{1-x}$ (x = 1; 0.9; 0.85) samples were prepared by direct fusion method. The temperature dependences of electrical conductivity, Seebeck coefficient, and thermal conductivity of (AgSbSe$_2$)$_x$(PbTe)$_{1-x}$ (x = 1; 0.9; 0.85) samples were investigated in the temperature range 80-350K. The value of electrical conductivity of PbTe added samples decreased compared to AgSbSe$_2$. Simultaneously, the inversion of the sign of Seebeck coefficient (*n→p*) in (AgSbSe$_2$)$_x$(PbTe)$_{1-x}$ (x=0.9; 0.85) solid solutions at temperatures of T > 110K was observed. It was determined that the effective mass of holes value in (AgSbSe$_2$)$_x$(PbTe)$_{1-x}$ (x=0.9; 0.85) solid solutions increased compared to AgSbSe$_2$. It has been found that the lattice thermal conductivity of (AgSbSe$_2$)$_x$(PbTe)$_{1-x}$ (x=0.9; 0.85) solid solutions decreases significantly with increasing PbTe amount in AgSbSe$_2$.

Keywords: *Thermoelectric material;Electrical conductivity; Seebeck coefficient;Thermal conductivity;Effective mass*
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INTRODUCTION

Binary silver selenide and related ternary semiconductor materials have a number of valuable functional properties and are effective, environmentally friendly compounds. Based on these properties, these materials are intensively studied by many researchers [1-5].

A number of important functional properties of compounds with the general formula $A^{I}B^{IV}X^{VI}$ (A-Ag, Cu; B-Si, Ge, Sn; X-Se, S) have been extensively studied. In particular, numerous studies have been conducted on the photo-electrical properties of chalcogenide glasses containing Ag such as photodoping, photovoltage, and photochemical modification [6-9].

Among these functional properties, one of the most interesting is ionic conductivity. As is known, ionic conductors are promising materials for the preparation of ion batteries. Possible crystallochemical conditions for the formation of the superionic state have been shown in studies [10-18]. Many articles have provided detailed information on the ionic conductivity of binary compounds formed with Ag^+ and Cu^+ chalcogenides [19-21].

The ternary semiconductor AgSbSe$_2$ is considered a promising thermoelectric materials due to its thermoelectric properties, and particularly its very low lattice thermal conductivity [22, 23]. AgSbSe$_2$ features a strong anharmonic bonding arrangement associated with the Sb 5s^2 lone pair, which leads to strong phonon-phonon interactions that reduce the lattice thermal conductivity [24]. Additionally, AgSbSe$_2$ is a narrow-bandgap semiconductor exhibiting p-type conductivity with a favourable valence-band structure composed of multiple flat valleys, potentially leading to a high effective mass of holes and thus the high value of the Seebeck coefficient. The band gap values of AgSbSe$_2$ measured by various methods are in the range of 0.3-1.1 eV [25-27]. This intrinsically low thermal conductivity and the appropriate electronic band structure make AgSbSe$_2$ a promising candidate for thermoelectric applications in the intermediate temperature range (500-700K) [28-30]. On the other hand, among the traditional thermoelectric materials, PbTe and its alloys are considered as excellent thermoelectric materials that operate in the medium and high temperature range (400 K - 800K) [31]. The crystal structure of I-V-VI$_2$ group compounds (AgSbSe$_2$, AgSbTe$_2$ etc.) is very similar to that of IV-VI group compounds (PbTe, PbSe, etc.) and all these compounds crystallize in NaCl structure. Therefore, these materials may be used to prepare solid solutions based on I-V-VI$_2$ group compounds [32, 33].

Investigations of AgSbSe$_2$ were mainly carried out above room temperature to enhance its thermoelectric properties [28-30]. Also, the kinetic properties of AgSbSe$_2$ and its solid solutions, their electronic structure, mechanisms of electric charge and heat transport have been little studied at low temperatures [34]. In this study, to address these questions experimentally, we synthesized (AgSbSe$_2$)$_x$(PbTe)$_{1-x}$ (x = 1; 0.9; 0.85) samples and investigated the influence of PbTe on the thermoelectric properties and band parameters of AgSbSe$_2$ at the temperature range of 80-350K.

EXPERIMENTAL METHOD

Synthesis. Polycrystalline (AgSbSe$_2$)$_x$(PbTe)$_{1-x}$ (x = 1; 0.9; 0.85) bulk samples were prepared using a direct fusion method together with mechanical pressing. The constituent elements Ag, Sb, Se, Pb, Te (99,99% purity) in stoichiometric ratios were sealed in evacuated quartz tubes under a pressure value of 10^{-4} Torr. The tubes were placed in furnace and

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heated slowly (1 K/min) up to 1000 K ($AgSbSe_2$ melting point: 908 K) over a period of 12 hours. Then tubes were maintained at this temperature for 10 hours and gradually cooled to room temperature at the same rate. The obtained ingots were ground into fine powders to eliminate pores. Then the powders were pressed (under a pressure of 500 MPa) to form bulk samples with dimensions of 12×5.2×2.4 mm^3.

Powder X-ray diffraction. Powder X-Ray diffraction (PXRD) analysis was conducted using a Bruker D8 Advance diffractometer with a CuKα anode (λ = 0.1542 nm), operating at 40 kV and 40 mA. The lattice constants were calculated using the Rietveld method with EVA and TOPAS software.

Carrier concentration. The values of carrier concentration and Hall mobility at room temperature were derived from Hall coefficient measurements using by the Van der Pauw method (in direct current and 0,32 T magnetic field) on the HL5500PC Hall Effect Measurement System.

Electrical transport. The thermoelectric properties were measured simultaneously from liquid nitrogen temperature to 350K in direct current using four-probe potentiometric method.

RESULTS AND DISCUSSION

Structural characterization

Figure 1 displays the PXRD patterns for $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0.9; 0.85) samples. The results of X-ray analysis revealed that all samples were single-phase with a cubic NaCl-type structure (Fm3m), with no impurity phases observed.

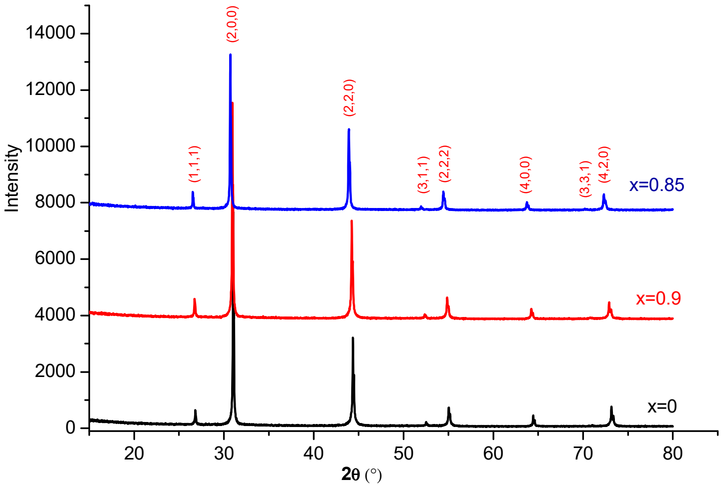

Figure 1. Powder XRD patterns of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0.9; 0.85)

As seen in Figure 2, the linear expansion in the lattice constants with increasing PbTe concentrations follows the Vegard's law for the $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0.9; 0.85) samples. The ionic radius of Pb (Te) is larger than of Sb. Therefore, as larger Pb (Te) is introduced in the place of smaller Sb, the unit cell undergoes a systematic expansion, leading to an increase in the lattice parameter [29].

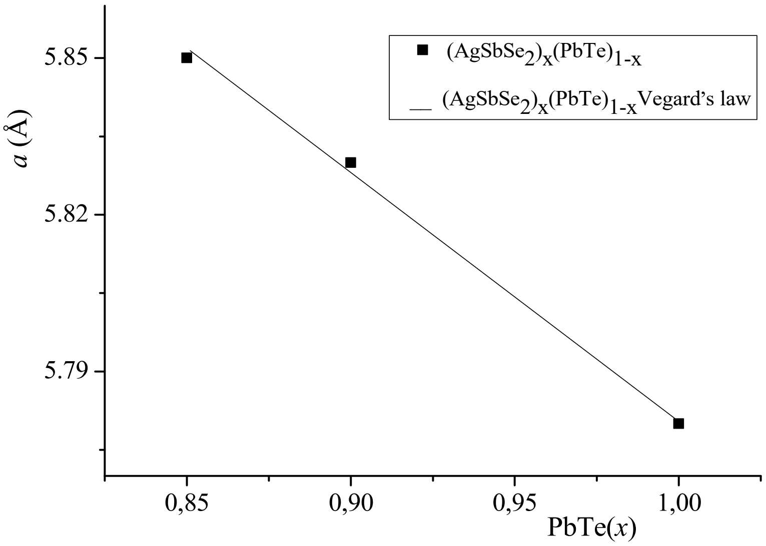

Figure 2. Composition (x) dependence of lattice constants for $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0.9; 0.85), and a straight line indicates Vegard's law for solid solution

Electrical transport properties

The temperature dependence of electrical conductivity (σ) of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=1; 0.9; 0.85) samples over the 80-350 K range is presented in Figure 3.

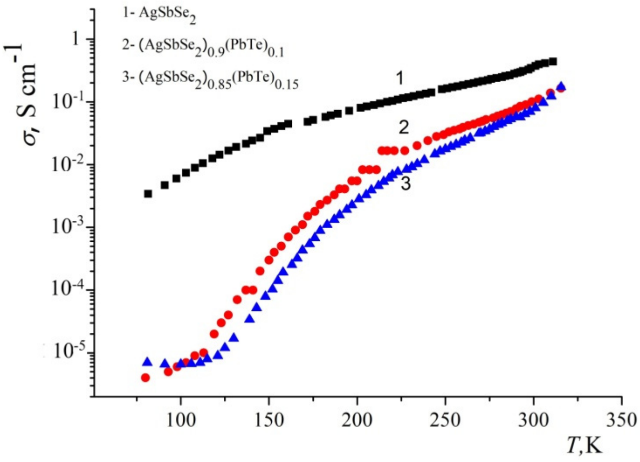

Figure 3. Temperature dependence of the electrical conductivity of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0.9; 0.85) solid solutions

As observed in Fig. 3, the value of σ at below room temperatures is relatively small and exponentially increases with temperature. However, the value of electrical conductivity of all samples increases with rising temperature. Additionally, we observed a decrease in the electrical conductivity of PbTe added samples compared to AgSbSe$_2$. At room temperature, the measured values of σ for $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0.9; 0.85) solid solutions are 0.4 S·cm^{-1}, 0.11 S·cm^{-1} and 0.09 S·cm^{-1}, respectively.

The addition of PbTe to AgSbSe$_2$ lead to partial compensation of electroactive acceptor centers, resulting in a decrease in hole concentration p. Hall measurements at room temperature indicate positive sign of the Hall coefficient (R_H) for all samples. This indicates that the majority carriers are holes and the samples exhibit p-type conduction. The carrier concentrations of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=1; 0.9; 0.85) solid solutions, estimated from measurements of the R_H ($p=1/eR_H$, where e is the elementary charge) at room temperature, are $5.6\cdot10^{17}$ cm^{-3}, $4.8\cdot10^{16}$ cm^{-3} and $3.7\cdot10^{16}$ cm^{-3}, respectively. Furthermore, the addition of PbTe to AgSbSe$_2$most likely increases the number of defects in the crystal lattice. This leads to an increase in the scattering centers of charge carriers and, as a result, to a decrease in the Hall mobility μ ($\mu=\sigma/pe$). The Hall mobilities of $(AgSbSe_2)_x(PbTe)_{1-x}$(x=1; 0.9; 0.85) solid solutions at room temperature are 4.2 cm^2/V·s, 3 cm^2/V·s and 2.3 cm^2/V·s, respectively. Thus, we assume that these two factors together cause a decrease in the electrical conductivity of $(AgSbSe_2)_x(PbTe)_{1-x}$(x=0.9; 0.85) solid solutions.

The temperature dependence of the Seebeck coefficient (S) of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=1; 0.9; 0.85) solid solutions in the 80-350K range is presented in Figure 4.

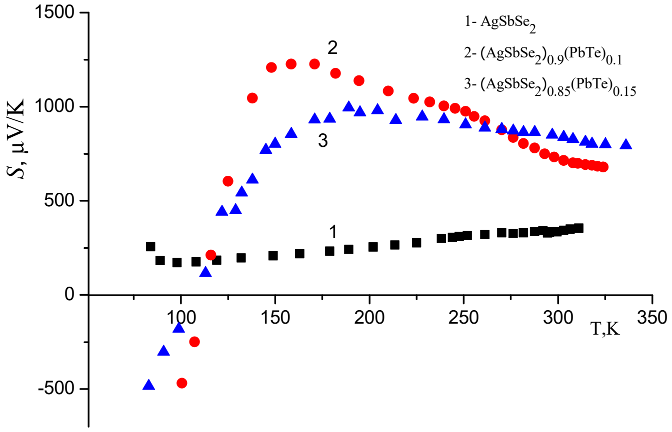

Figure 4. Temperature dependence of the Seebeck coefficient in $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0.9; 0.85) solid solutions

As is known, AgSbSe$_2$ is a p-type semiconductor [22-24]. As can be seen in Fig. 4, AgSbSe$_2$ exhibits p-type conductivity in the temperature range of 80-350K. However, in the $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0.9; 0.85) solid solutions, the sign of the Seebeck coefficient is negative at temperatures below 110 K, indicating n-type conductivity. It is possible that the addition of PbTe to the AgSbSe$_2$ leads to the formation of shallow donor levels. The ionization of these donor centers leads to a significantly increase in the contribution of electrons in the Seebeck coefficient compared to AgSbSe$_2$.

Consequently, for $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0.9; 0.85) solid solutions, the sign of Seebeck coefficient is negative at temperatures below 110K (Fig. 4). At T>110K the Seebeck coefficient changes its sign ($n \rightarrow p$) and becomes positive. Such a change of sign is also manifested at temperature dependence of Hall coefficient. The increase in hole concentration, most likely resulting from the ionization of deep acceptor centers as temperature rises, leads to the change in the sign of the Seebeck coefficient. Moreover, the values of Seebeck coefficient in $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0.9; 0.85) solid solutions are larger than $AgSbSe_2$ (350 μV/K), at room temperature, and the values of S are 701 μV/K and 828 μV/K, respectively. The increase in the Seebeck coefficient for $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=0.9; 0.85) solid solutions compared to $AgSbSe_2$ most probably may be explained by the increased density of states effective mass for holes $m^*_{h,dos}$. Assuming a single parabolic band model with acoustic phonon scattering (r=0, where r is scattering factor), we calculated the values of effective mass of holes m^*_p using the following equations (1)-(3) based on the values of S and hole concentrations p [35]:

$$p = \frac{\left(2m^*_p k_B T\right)^{3/2}}{3\pi^2\hbar^3} F_{3/2}(\eta^*) \tag{1}$$

$$m^*_p = \frac{\left(3\pi^2\hbar^3\right)^{3/2}}{2k_B T\left[F_{3/2}(\eta^*)\right]^{3/2}} \tag{2}$$

$$F_r(\eta) = \int_0^{\infty}\left(-\frac{\partial f_0}{\partial x}\right)x^r dx \tag{3}$$

where, η^* is the reduced Fermi energy, $F_r(\eta)$ - the r^{th}- order Fermi integral, k_B- the Boltzmann constant, $\hbar$ - the reduced Planck's constant ($\hbar = h/2\pi$), e- elementary charge.

From these calculations, the value of effective mass for holes m^*_p at room temperature for $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=1; 0.9; 0.85) solid solutions were found to be $m^*_p = 0.13m_0$, $m^*_p = 3.82m_0$, and $m^*_p = 3.98m_0$, respectively. We suppose that with increasing temperatures, the high-density charge carriers resulting from the addition of PbTe into $AgSbSe_2$ may be redistributed among multiple energy states within the valence band. This could potentially lead to changes in the electronic band structure that could affect the material's properties. In work [36] the authors suggest that such redistribution of the density of states of charge carriers leads to an increase in the effective coverage of bands. An increase in the number of charge carriers in the multiple degenerate valence bands can cause a higher value of their density of states effective mass m^*. This, is in turn, lead to an increase in the Seebeck coefficient, since S is directly related to m^* by the equation of $S = \frac{8\pi^2 k_B^2}{3eh^2} m^* T \left(\frac{\pi}{3n}\right)^{2/3}$ [37]. Additionally, the increase in the values of m^*_p due to the amount of PbTe also results in decreased Hall mobility, leading to a decrease in the value of electrical conductivity (σ) in the $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0.9; 0.85) solid solutions compared to the pristine $AgSbSe_2$ sample. This confirms earlier discussed results on electrical conductivity and Hall mobility.

We have also investigated the thermal conductivity (k) of the $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0.9; 0.85) solid solutions in the temperature range of 80-350K. The obtained result is shown in Figure 5.

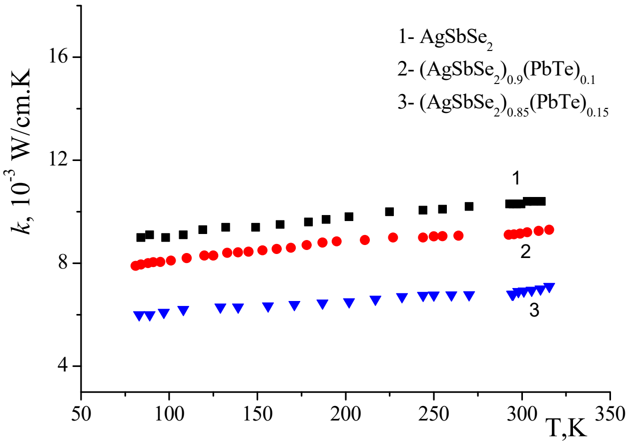

Figure 5. Temperature dependence of thermal conductivity of $AgSbSe_2$ and $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0.9; 0.85) solid solutions

As can be seen from Fig 5, the values of thermal conductivity of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0.9; 0.85) significantly decreased compared to $AgSbSe_2$. We have calculated the electronic contribution in thermal conductivity k_{el} using the Wiedemann-Franz law: $k_{el} = L_0\sigma T$, where $L_0 = 2.44\cdot10^{-8}$ W·Ω/K^2 is Lorentz number [25], and, for all three samples, k_{el} is not more than 2%. The obtained lattice thermal conductivity k_{lat} values of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=1; 0.9; 0.85) are low and 0.85 W/m·K, 0.78 W/m·K and 0.57 W/m·K, respectively.

The main reason for the low thermal conductivity of $AgSbSe_2$ and other compounds of the I-V-VI$_2$ group is most likely the disorder of their structure [38]. Moreover, the point defects which exist in the crystal lattice and structural components may cause low thermal conductivity of $AgSbSe_2$. As mentioned above, $AgSbSe_2$ crystallize in disordered

NaCl type structure. In such structure, the random distribution of non-chalcogenide atoms may cause energetically favorable (Ag^+–Sb^{3+}) and unfavorable (Ag^+–Ag^+ and Sb^{3+}–Sb^{3+}) states of Ag^+ and Sb^{3+} ions in the Ag/Sb sublattice [39, 40]. Therefore, the presence of regions with orderly and disorderly distributions of Ag and Sb atoms in $AgSbSe_2$, high anharmonicity of the Sb-Se bonding arrangement [29], displacement of atoms and deformation of crystal lattice leads to the formation of phonon scattering centers. Consequently, the k_{lat} value decreases due to increasing phonons scattering.

Furthermore, it is known that silver chalcogenides are high-ionic-conductivity materials [41, 42]. The silver ions leave their positions as the temperature increases, and intensifying more energetically favorable state of the crystal lattice. As a result, the defects in the crystal structure insignificantly decrease and thermal conductivity slightly increase with rising temperature. Simultaneously, also this factor causes the distortion of the crystal lattice and as a result, the k_{lat} value decreases caused by increasing phonons scattering.

Thus, all mentioned above factors most probably collectively lead to a decrease of the k_{lat} value of $AgSbSe_2$ due to increasing phonons scattering. The addition of PbTe to $AgSbSe_2$ increases the disorder of the crystal lattice, which ultimately leads to a significant decrease in the lattice thermal conductivity in $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=0.9; 0.85) solid solutions.

CONCLUSIONS

In summary, we observed that the electrical conductivity of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0.9; 0.85) solid solutions decreased compared to $AgSbSe_2$ in the 80-350 K temperature range. Simultaneously, the inversion of the sign of Seebeck coefficient ($n \rightarrow p$) of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=0.9; 0.85) at temperatures of T>110K occurred most likely due to the ionization of deep acceptor centers. Additionally, we observed higher values of the hole effective mass in $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=0.9; 0.85) compared to $AgSbSe_2$, which is due to the increase in redistribution of the density of states of charge carriers in the multiple energy states of the valence band. This enhances the Seebeck coefficient value of $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=0.9; 0.85) solid solutions. Moreover, the strong phonon-phonon interactions due to the high anharmonicity in the Sb-Se bonding arrangement, effective scattering of phonons by the disordered crystal lattice and point defects lead to the low lattice thermal conductivity k_{lat} of the $(AgSbSe_2)_x(PbTe)_{1-x}$ (x=0.9; 0.85) solid solutions compared to pristine $AgSbSe_2$.

The results obtained in this study provide directions for future research- including nanostructuring, doping, band convergence, and defect engineering to enhance the properties of thermoelectric materials.

Declarations

- Funding: The authors did not receive support from any organization for the submitted work.
- Financial interests: The authors have no relevant financial or non-financial interests to disclose.
- Conflict of interest: The authors declare that they have no conflict of interest.
- Author contribution: The authors contributed equally to this work.

Data Availability Statement

Due to privacy and ethical restrictions, the data used in this study cannot be shared publicly. However, the analyzed data are available to the researcher from the corresponding author on reasonable request with appropriate ethical approval.

ORCID

A.A. Saddinova, https://orcid.org/0009-0005-9967-3575; **R.I. Selim-zadeh,** https://orcid.org/0009-0007-3649-5390
A.E. Babayeva, https://orcid.org/0009-0001-0286-5783; **A.A. Orujova,** https://orcid.org/0009-0007-7254-9499

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ВПЛИВ ДОДАВАННЯ PbTe НА ЕЛЕКТРИЧНИЙ ЗАРЯД ТА ТЕПЛОПЕРЕНОС В AgSbSe$_2$

А.А. Саддинова[1], Р.І. Селім-заде[1], А.Є. Бабаєва[1], А.А. Оруджова[2]
[1]Інститут фізики Міністерства науки і освіти Азербайджанської Республіки, AZ-1143, Азербайджан
[2]Мінгачевірський державний університет, Az-4500, Азербайджан

Метою цього дослідження є вивчення впливу додавання PbTe на термоелектричні властивості та параметри зон AgSbSe$_2$ за температур нижче кімнатної. Для цього методом прямого сплавлення було виготовлено полікристалічні зразки $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0,9; 0,85). Досліджено температурні залежності електропровідності, коефіцієнта термоЕРС та теплопровідності зразків $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 1; 0,9; 0,85) в діапазоні температур 80-350 K. Значення електропровідності зразків з додаванням PbTe зменшилося порівняно з AgSbSe$_2$. Одночасно спостерігалася інверсія знака коефіцієнта термоЕРС (n→p) у твердих розчинах $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0,9; 0,85) за температур $T>110K$. Було визначено, що значення ефективної маси дірок у твердих розчинах $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0,9; 0,85) збільшується порівняно з AgSbSe$_2$. Було виявлено, що ґраткова теплопровідність твердих розчинів $(AgSbSe_2)_x(PbTe)_{1-x}$ (x = 0,9; 0,85) значно зменшується зі збільшенням кількості PbTe в AgSbSe$_2$.

Ключові слова: *термоелектричний матеріал; електропровідність; коефіцієнт Зеєбека; теплопровідність; ефективна маса*