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GALVANOMAGNETIC PROPERTIES OF $Gd_xSn_{1-x}Se$ SOLID SOLUTIONS

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ARTICLE INFO	ABSTRACT
Article history:	The nature of the interaction in the SnSe – GdSe quasibinary section was
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Received in revised form: 2025-02-10	$Gd_xSn_{1-x}Se$ solid solutions were obtained, complex physicochemical
Accepted: 2025-02-10	analyses were carried out and crystal parameters of the sample were
Available online	determined. The specific electrical conductivity, Hall coefficient, and
Keywords: solid solution,	magnetic resistance of $Gd_xSn_{1-x}Se$ solid solutions were studied in the
crystal parameters, specific electrical	temperature range of 300-650 K, and the charge transport mechanisms were
conductivity, Hall coefficient,	investigated. The dependence of magnetic resistance on composition,
magnetoresistance,	temperature, and magnetic field intensity was studied. The chemical bond
zone structure	and crystallization type structure in Tin monoselenide has also been
	investigated. Using a special X-ray diffractometer, the X-ray structural
	analysis was carried.

1. Introduction

Interest in semiconductor compounds of the A^4B^6 type is due to the prospects for their use in semiconductor instrumentation. For example, *SnS* and *SnSe* are used as a base material for creating active elements that operate in the infrared region of the optical spectrum, in thermodynamic converters [1-2], an absorbing layer in thin-film solar energy converters [3-4], and also as photoconductors [5], semiconductor sensors [6], micro-batteries [7]. The fundamental characteristics of these compounds a small band gap, high permittivity, relatively high radiation resistance, and the predominance of ionic bonding - further expand the possibilities of their application [8]. In recent years, much effort has been directed at creating photovoltaic devices from cost-effective, low-toxic, low-cost materials with a simple production technology. In this regard, SnSe semiconductor compounds have proven to be very promising due to the low cost of the material, which is due to the widespread occurrence of tin and selenium in nature.

Tin monoselenide has a complex ionic-covalent chemical bond and crystallizes in the *NaCl* –type structure. One of the main features of SnSe is the presence of defects to a certain extent [9]. It was found that the presence of vacancies in both sublattices and their interaction is

caused by antistructural $\sim 10^{17} sm^{-3}$ defects. A high concentration of these defects in *SnSe* leads to the formation of p-type conductivity. Tin selenide is a base material for creating active elements that operate in the infrared region of the optical spectrum, as well as in thermodynamic converters [10,11].

Doping them with impurities that create quasi-local levels expands the possibilities of their practical application. The introduction of rare earth metal (REM) elements into tin monoselenide causes the emergence of a number of physical properties associated with the nature of the defect and the interaction of defects. The behavior of REM impurities in semiconductors is characterized by some distinctive features, for example, a combination of low solubility and the ability of REM to "purify" the material, which makes it possible to significantly reduce the concentration of background impurities and defects [12].

The corresponding studies are of particular importance in fundamental physics, as well as from the point of view of their application. Substances obtained with the participation of rare earth metals (REM) are widely used in the manufacture of some converters of electrical energy, various types of thermistors resistant to radiation, pressure and humidity. With incomplete filling of the electronic structure of REM level 4f, an easy transition to 4f - 5d - 6s occurs, and the creation of a variable valence due to mobile electrons of atoms of level 4f makes the materials obtained with their participation an interesting object of study. From this point of view, the possibility of obtaining new promising materials with the required physical properties based on alloys and compounds with the participation of REM makes their study especially important [13, 14]. Therefore, the study of the interaction between chalcogenides *SnSe* and *GdSe*, as well as a comprehensive study of the galvanomagnetic properties of the formed solid solutions is of scientific and practical interest.

2. Experimental procedure

The initial components of tin macri "B4-000", selenium grade "OC417-4" and chemically pure prosedium (99.98%) were used to synthesize the SnSe – GdSe alloys. The synthesis was carried out in evacuated quartz ampoules, at a pressure of 0.1333 Pa, by the method of direct melting of the components in two stages. At the initial stage, the ampoule together with the substance was heated to the melting point of selenium at a rate of 4 - 5 degrees / minute and maintained at this temperature for 3 - 4 hours, then the temperature was gradually increased depending on the composition to 950 - 1000 °C, and maintained for 8 - 9 hours. The synthesized samples for complex physicochemical analysis and electrophysical studies were annealed for 140 - 210 *hours* depending on the composition: the annealing time was increased with an increase in the gadolinium content. Homogenizing annealing of the obtained single-phase samples was carried out in a spectrally pure argon environment.

The interaction in the *SnSe* – *GdSe* system was studied by differential thermal analysis (DTA), X-ray phase analysis (XPA), microstructural analysis (MSA), as well as by measuring microhardness and determining the density. To determine the thermal effects of the obtained samples and phase transitions, DTA was carried out on a Perkin Elmer Sinintltaneons Thermal Analgiyzer, STA 6000 (USA). Nitrogen was used as the working gas with a feed rate of 20 *ml/sec*, the sample is heated to melting at a heating rate of 5 °C/*min*.

X-ray structural analysis was carried out on a Miniflex X-ray diffractometer by Rigaku Corporation, operating at 30 kV, 10 mA, and $CuK\alpha$ radiation ($\lambda = 1.5406$ Å). Diffraction

reflections were observed at a displacement angle of 2q in the range of 0–80°. A Japanese JEOL JSM6610-LV scanning electron microscope was used to study the morphology and microcomposition of the sample surface. The galvanomagnetic properties of samples of different compositions obtained in the field of solid solutions of the quasi-binary compound $(SnSe)_{1-x} - (GdSe)_x$ were studied in a wide temperature range, and an analysis of the general pattern was carried out. The specific electrical conductivity (σ), Hall coefficient (R), and relative magnetic resistance ($\Delta \rho / \rho$) were measured in the temperature range of 300 ÷ 650K in a constant magnetic field in the mode direct current [15]. The experimental error did not exceed 4.2%.

3. RESULTS AND DISCUSSION

3.1. Physicochemical analysis

Sharp peaks corresponding to melting and solidification are observed on the heating and cooling curves in the thermograms of the obtained $Gd_xSn_{1-x}Se$ alloy system. This allows us to state that congruently melting alloys are formed during the synthesis. In the binary compound *SnSe*, partial replacement of *Sn* atoms by *Gd* atoms contributes to a decrease in the melting temperature, which apparently causes REE in these crystals to act as softeners.



Fig. 1. X-ray diffraction spectrum of crystals Gd_xSn_{1-x}Se: 2- x=0,005

An analysis of the intensities of X –ray reflections shows the presence of a predominant direction in the crystal, and also that the studied sample consists of a single phase. Indexing of X –ray patterns shows that in the solubility region of Gd based on SnSe, the studied alloys crystallize in the orthorhombic syngony with the space group $D_{2h}^{16}-P_{cmn}$ (Fig. 1). In the X-ray diffraction patterns in the range $0 \le x \le 0.02$, no shifts of diffraction lines are observed, and only a change in their intensity in the specified range indicates that solid solutions based on SnSe have formed. In the binary compound SnSe in the dissolution region, due to the partial replacement of Sn atoms by rare-earth metal atoms of larger radius, the intensity of reflections decreases and the parameters of the orthorhombic unit cell of the lattice increase additively. In this region, the growth of the lattice parameters is linear and no deviation from Vegard's law is observed.

From the analysis of X –ray structural data it follows that with the introduction of gadolinium selenide an increase in the parameters of the unit cell of *SnSe* is observed as the

concentration of *Gd* increases, as well as intense scattering of carriers by lattice "distortions" coinciding with studies of the thermal conductivity of alloys [16]. With an increase in the *Gd* content in the alloy, the density of $Gd_xSn_{1-x}Se$ does not change significantly. This confirms that the Gd atoms introduced by us into the $Gd_xSn_{1-x}Se$ alloy primarily occupy the interstitial vacant place in the crystal, i.e. defect formation occurs according to Frenkel [17].



Fig. 2. X-ray microanalysis of the crystal surface GdxSn1-xSe: 2- x=0,005

The observed growth of the lattice parameters, good agreement of partial substitution of Sn atoms by Gd atoms of larger radius and observance of Vegard's law allow us to speak about the formation of solid substitution solutions based on *SnSe*. A study of the dependence of the microstructure, microhardness and density of the composition, also revealed by X –ray and pycnometric methods, shows that the region of dissolution of *GdSe* in *SnSe* at room temperature is limited to 2 *mol*%.

The results of the conducted complex physicochemical analyses show that the $Gd_xSn_{1-x}Se$ alloy system, like the main substance *SnSe*, crystallizes in the orthorhombic syngony. With an increase in the percentage of *GdSe* in the composition, a weak increase in the parameters of the unit cell of the crystal lattice, density and microhardness is observed, and the thermal effects shift to the region of relatively lower temperatures.

Since the atoms of *Sn* and *Gd* belong to different subgroups, differ greatly in electron configurations, the sizes of the atoms of the soluble element differ from the sizes of the solvent atom, therefore, when a solid substitution solution of $Gd_xSn_{1-x}Se$ is formed, the *SnSe* crystal lattice is slightly distorted, without losing its basic structure.

Quantitative X –ray microanalysis determined the phase composition and distribution of chemical elements on the surface of the sample under study (Fig. 2). Analysis of the results obtained shows surface homogeneity, but with a change in stoichiometry within the homogeneity region of SnSe on the side of excess selenium.

3.2. Electrical conductivity in solid solutions $Gd_xSn_{1-x}Se$

With an increase in the amount of *Gd* in small concentrations in the $Gd_xSn_{1-x}Se$ alloy system, the specific electrical conductivity decreases, and at x > 0.01, an increase in electrical conductivity is observed. At *Gd* concentrations $0 \le x \le 0.01$, filling of tin vacancies and a change in chalcogen antistructural defects leads to a decrease in the values of the charge carrier concentration and specific electrical conductivity. It can be said that with a subsequent increase in the amount of *GdSe*, antistructural defects do not occur. It is very likely that a partial increase

in specific electrical conductivity is associated with the appearance of the second type of charge carriers as a result of the action of donor *Gd* atoms.



The temperature dependence of the electrical conductivity of $Gd_x Sn_{1-x}Se$ crystals is shown in Figure 3. As can be seen from the graphs plotted on the scale $\sigma \propto f(10^3/T)$, the value of electrical conductivity (σ) at a given temperature proportionally decreases upon moving from the *SnSe* compound to solid solutions based on it. On the other hand, the $\sigma(T)$ dependence changes according to a law close to exponential. In the temperature range $T = 300 \div 400K$, the electrical conductivity changes according to a weak and monotonic law. In the temperature range $T = 400 \div 480K$, the σ value remains relatively constant. At $T \ge 420K$, the graph passes through a minimum and partially increases, and at T = 560K, a monotonic, sharper increase occurs again. In the sample with composition x = 0.01, this anomalous change is partially weak, and at temperature T > 620 K the change in $\sigma(T)$ occurs with the same intensity in both compositions.

For the studied samples, the thermal activation energy of charge carriers was determined. It was found that in the temperature range $T = 300 \div 400K$ in sample 2, containing x = 0.005: $E_A = 0.27 \ eV$, and for sample 3 (x = 0.01) $E_A = 0.02 \ eV$ [18].

On the other hand, the strengthening of the $\sigma(T)$ dependence at temperatures T > 500K indicates that "heavy" charge carriers also participate in conductivity due to the activation of additional impurity energy levels located deep [19].

3.3. Hall coefficient in solid solutions $Gd_xSn_{1-x}Se$

The temperature dependence of the Hall coefficient of the obtained samples of the composition (x = 0.0, 0.005 and 0.01) was studied and the results are presented in Figure 4. As can be seen from the graphs, the temperature dependence of the Hall coefficient R(T) corresponds to the dependence of electrical conductivity $\sigma(T)$. As in the electrical conductivity mechanism, the Hall coefficient in the region of impurity conductivity is weak, monotonically decreasing. This decrease corresponds to an increase in the concentration of charge carriers as a result of thermal excitation with increasing temperature. With a subsequent increase in temperature, the Hall coefficient increases. This anomaly is explained by the presence of traps in

the forbidden zone for electrons or the formation of additional acceptors with an increase in temperature [20].

With a subsequent increase in temperature (T > 560K), it passes through a weak extremum, sharply decreases and approaches the temperature characterizing the intrinsic conductivity. The decrease in the R(T) dependence, although insignificant, after passing the extremum, shows that the band structure of these samples is complex. This type of dependence is found in the base compound *SnSe*, as well as in its structural analogs. The reason for this is the complex nature of the band structure of these compounds, the state of the valence band from two lower bands and is explained by the existence of light and heavy holes, respectively.

In the presence of light and heavy holes, due to the weak dependence of the ratio of their mobility on temperature, the temperature dependence of the Hall coefficient is determined by the distribution of charge carriers in the valence subbands. Due to the weak mobility of holes in the second valence band, their share in the Hall effect is weak, due to the passage of holes into the second zone, the effective concentration decreases, and the Hall coefficient increases.

Experiments show that the electrical conductivity and Hall coefficient of the samples in the temperature range of our study change according to the regularity of semiconductor materials. Depending on the sign of the thermo EMF and the Hall coefficient in the samples, it was found that at room temperature the sample with the content of x = 0.005 has p –type conductivity, and the sample with the content of x = 0.01 has n –type conductivity. The Hall coefficient of the sample with the composition x = 0.005 changes sign from positive to negative at a temperature of T = 455 K. With a further increase in temperature, the sign of the Hall coefficient becomes negative.

A change in the sign of the Hall coefficient when passing the inversion point can occur for two reasons. The first reason: due to an increase in temperature due to the activation of charge carriers at partially localized energy levels, the concentration of electrons can increase, and in the second case this can be due to the high mobility of electrons participating in conductivity. The obtained sample compositions are partially compensated semiconductor materials; three types of charge carriers participate in conductivity simultaneously.



Fig. 4. Temperature dependence of the Hall coefficient in solid solutions GdxSn1-xSe: 1) x=0,000; 2) x=0,005; 3) x=0,010

The temperature dependence of the Hall coefficient for the sample with x = 0.01 is shown on the 3rd curve. This sample has n –type conductivity, and the absolute value of the Hall coefficient slightly increases with temperature. It decreases, passing through a weak maximum at T = 525K. Such an anomalous change in the R(T) dependence can occur for two reasons. The first reason can be associated with the depletion of the number of free electrons participating in conductivity from donor energy levels at this temperature (T = 525K), and the second reason can be associated with the complex structure of the energy band. The complexity of the band structure leads to the fact that excited electrons at this temperature become "heavy" electrons and create exciton pairs. For this reason, the value of the total electrical conductivity decreases. The temperature dependence of the Hall coefficient R(T) indicates that the scattering of charge carriers from different centers is quite complex [21].

3.3. Magnetoresistance in $Gd_xSn_{1-x}Se$ solid solutions

Figure 5 shows the temperature dependence of the magnetoresistance of the $Gd_xSn_{1-x}Se$ melt system (x = 0; 0.005; 0.01). As can be seen from the graph, magnetic resistance is observed in all the studied samples and at a temperature of $T = 300K \Delta \rho / \rho$ has a maximum value. With increasing temperature, due to the $\Delta \rho / \rho_0$ of charge carriers of the second kind, i.e., minor charge carriers, and their change in the magnetic field, is the cause of a different increase in magnetic resistance.



Fig.5. Temperature dependence of the change in the relative magnetoresistance of the solid solution of the Gd_xSn_{1-x}Se system: 1-x=0.0; 2-x=0.005; 3-x=0.01 1) x=0,000; 2) x=0,005; 3) x=0,010

The magnetic resistance in the binary compound *SnSe* at room temperature is 3%. With increasing temperature, the absolute value of the magnetic resistance decreases and after 420 K, having performed an inversion, changes sign from positive to negative (Figure 3, curve 1). With an increase in the amount of *Gd* element in the composition, the absolute value of magnetic resistance ($\Delta \rho / \rho$) increases, but its temperature dependence changes similarly to a binary compound.

The inversion points in solid solutions of *SnSe* with the element *Gd* shifts to the region of higher temperature: for samples of composition x = 0.005 the inversion point occurs at temperature T = 460K; and for samples of composition x = 0.01 at temperature T = 470K. As a

result, due to activation of charge carriers of the second kind, the magnetic resistance created by them is of great importance.

With further increase in temperature the sign of $(\Delta \rho / \rho)$ is negative and in the temperature range T = 530 - 560K, having passed through the maximum, decreases. The change in the sign of magnetic resistance in this temperature range also shows a change in the mechanism of scattering of charge carriers. A large absolute value of $(\Delta \rho / \rho)$ shows the presence of charge carriers of the second kind in the composition [22].

Thus, it was determined that the change in the mechanism of scattering of charge carriers also involves carriers of the second kind of this composition. The dependence of the magnetic resistance on the magnetic field strength (H) near this inversion point at a temperature of T = 420 K was investigated (Fig. 6).



Fig.6. Dependence of the magnetoresistance of Gd_xSn_{1-x}Se on the magnetic field strength: 2- x=0.005; 3- x=0.01; 4- x=0.02.

The dependence of the magnetoresistance on the magnetic field strength is given in the coordinates $(\Delta \rho / \rho) = f(H^2)$. It is evident from the figure that the magnetic resistance $(\Delta \rho / \rho)$ does not change linearly from the magnetic field strength. This is explained by the expenditure of additional energy on the direction of spin ordering of "light" and "heavy" electrons.

The study of the dependence of magnetic resistance on the magnetic field strength shows that at low values of the magnetic field strength in the direction of the field H, energy is spent on the direction of spin ordering of both free and localized microcenters. Due to the increase in the magnetic field strength and spin, the number of shared electrons increases, and then at a certain value of H, the magnetic resistance increases linearly [23].

At a relatively large field value, with an increase in H, the increase in the number of shared electrons along the spin is observed linear dependence $\Delta \rho / \rho$) = $f(H^2)$. Here there is a probability of the transition of partially localized microferromagnetic centers to paramagnetic ones.

From the analysis of the literature, it was determined that if magnetic resistance and its change are observed in these samples, then such substances have a complex structure of the energy band. In general, the observation of an arbitrary composition of magnetic resistance and its high value shows both the complex structure of the energy band of the substance and the presence of charge carriers of the second kind. Due to the activation of charge carriers of the second kind, the emergence of a large value of magnetic resistance in the samples is observed.

4. Conclusion

The results of the complex physicochemical analyses show that the dissolution region of *GdSe* in *SnSe* at room temperature is limited to 2 mol%. The $Gd_xSn_{1-x}Se$ alloy system, like the main substance SnSe, crystallizes in the orthorhombic syngony. With an increase in the percentage of GdSe in the composition, a slight increase in the parameters of the unit cell of the crystal lattice, density and microhardness is observed, and thermal effects shift to the region of relatively lower temperatures.

In solid solutions of $Gd_x Sn_{1-x}Se$, impurities are compensated by intrinsic defects, with moderate compensations, the concentrations of current carriers decrease accordingly and with an increase in the concentration of hodolinium in solid solutions (x = 0.005), a change in the sign of p to n type conductivity is observed. From the dependence $\sigma(T)$, the activation energies of levels located in the forbidden zone are determined, approximately 0.2 *eV* below the bottom of the conduction band, and the degree of compensation slightly affects the activation energy. Filling of tin vacancies and change of chalcogen antistructural defects leads to decrease of specific conductivity, concentration of charge carriers, activation energy and width of forbidden zone. Temperature dependence of Hall coefficient is well explained on the basis of two-zone model.

As temperature increases, absolute value of magnetic resistance decreases and inversion changes sign from positive to negative. As amount of *Gd* element in composition increases, absolute value of magnetic resistance increases and inversion point shifts to the region of higher temperature. From magnetic field dependences of resistance in $Gd_xSn_{1-x}Se$ samples, it follows that magnetoresistance of samples is typical for semiconductors, in weak magnetic fields standard, quadratic growth is observed.

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