

The model of impurity band reconstruction has been proposed for n-ZrNiSn intermetallic semiconductor doped with acceptor impurities. The hardening of heavily doped n-ZrNiSn specimens, in contrast to the tempering procedure, has been found to result in a substantial shift of the Fermi level and a considerable variation of the majority and minority charge carrier concentrations, provided that the concentrations of acceptor impurities were identical in both cases. This phenomenon can be caused, besides other reasons, by different degrees of local deformations in polycrystalline semiconductor specimens.

# 1. Introduction

In this work, we expound our understanding of the problem which nowadays is dealt with and discussed in a good many laboratories, but a consensus has not been reached yet. The matter concerns the origin of the dependences of the physical properties of semiconductors with the structure of the MgAgAs type on the conditions of homogenizing annealing. Since these semiconductors are promising thermoelectric materials, the stability of their electrophysical parameters has to be provided in a wide temperature interval, and the factors that can change semiconductor parameters in an uncontrollable way have to be determined.

The experimental and theoretical researches of the influence of acceptor and donor impurities with various concentrations on the magnetic and kinetic phenomena

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in *n*-ZrNiSn and *p*-TiCoSb semiconductors allowed us to propose an approach for the analysis of the transfer phenomena in heavily doped ( $N_A$  and  $N_D \sim 10^{19} \div$  $10^{21} \text{ cm}^{-3}$ ) and compensated semiconductors with the structure of the MgAgAs type [1-6], the essence of which is taking the features of heavily doped and compensated semiconductors [7], on the one hand, and local irregularities of the crystalline structure [8], on the other hand, into consideration simultaneously. This enabled us to determine, for the first time, the role of impurity bands in the conductivity of the indicated semiconductors, to suggest models which describe the reconstruction of impurity bands for impurities of various types when the concentration of those impurities varies, and to observe the insulator-metal transitions which were predicted by theoretical calculations and are attributed by us to the transitions of the Anderson type.

Notice that, in the works cited above, acceptor impurities were introduced into n-ZrNiSn through the substitution of  $Zr(4d^25s^2)$  atoms in its crystal structure by  $Sc(3d^14s^2)$  ones, and donor impurities were introduced into p-TiCoSb through the substitution of  $Co(3d^74s^2)$  atoms in its crystal structure by Ni $(3d^84s^2)$  ones.

The researches of the kinetic, galvano-magnetic, structural, and metallographic properties and the electron paramagnetic resonance spectra of pure and doped ZrNiSn and TiCoSb were carried out, in particular, in works [9–16].

In work [17], the assumption has been put forward that the duration of the homogenizing annealing of ZrNiSn affects the reciprocal arrangement of Zr and Sn atoms which are close by their dimensions. Therefore, the composition of the ZrNiSn compound should be represented taking into account the distribution of Zr and Sn atoms in it, namely, by the formula  $(\operatorname{Zr}_x \operatorname{Sn}_{1-x})\operatorname{Ni}(\operatorname{Sn}_x \operatorname{Zr}_{1-x})$ . The distribution of Zr and Sn atoms is completely stochastic at x = 0.5 (the structure of the  $CaF_2$  type is realized) and completely regular at x = 1 (with the structure of the MgAgAs type). The authors of work [13], while studying the influence of annealing on the kinetic and galvano-magnetic properties of ZrNiSn, HfNiSn, and Zr<sub>0.5</sub>Hf<sub>0.5</sub>NiSn pure and doped with acceptor (In) or donor (Sb) impurities, - observed that HfNiSn manifested much larger variations of the electrical resistance, the heat conductivity, and the concentration and mobility of charge carriers than ZrNiSn and Zr<sub>0.5</sub>Hf<sub>0.5</sub>NiSn did. In so doing, the difference between the dimensions of Hf and Sn atoms is much larger than that between Zr and Sn ones. Therefore, the conclusion that the substitution of Zr and Hf positions, on the one hand, and Sn ones, on the other hand, is improbable and not crucial for the variation of the electrophysical parameters of those semiconductors seems reasonable. The transitions insulator-metal, which were observed in work [13] on the doping of semiconductors, were associated with the dependence of the energy gap width on the impurity concentration. It was done taking no account of such concepts as impurity conduction, impurity bands, and so on, although the authors [13] did form the impurity bands by introducing the significant amounts (about  $10^{20}$  cm<sup>-3</sup>) of impurities. The assumption that the increase of the cerium concentration leads to a reduction of the energy gap width was put forward in work [11] on the basis of the results obtained for the temperature dependences of the magnetic susceptibility of  $M_{1-x}Ce_xNiSn$  (M = Zr, Ti). By the way, Ce is an acceptor impurity with respect to Zr and Ti.

We believe that the authors of works [11, 13] observed the activation of charge carriers from the Fermi level into one of the continuous energy bands (depending on the impurity type) rather than their interband activation. As a result, the diminishing of the activation energy, which accompanies the growth of the impurity concentration, is not resulted from the collapse of the semiconductor energy gap, but is connected with the variation of the compensation factor

and, hence, with the displacement, towards that or another continuous energy band, of the Fermi level, which is fixed in the Coulomb gap of the impurity band. This process is followed by the entering of the Fermi level into these bands, so that the conductivity changes its type from the activation to metallic one [1-8].

In this work, we studied the behaviors of the electrical conductivity, the Seebeck coefficient, the magnetic susceptibility, and the structural characteristics of n-ZrNiSn semiconductor at doping the latter up to various concentrations of acceptor impurities by substituting  $\operatorname{Co}(3d^74s^2)$  atoms for  $\operatorname{Ni}(3d^84s^2)$ ones, as well as the formation of the substitutional solid solution  $ZrNi_{1-x}Co_xSn$ . The concentration of impurities was changed from  $N_A = 8.7 \times 10^{19} \text{ cm}^{-3}$ to  $N_A = 1.7 \times 10^{21} \text{ cm}^{-3}$  at that. We notice that it is within this interval of acceptor impurity concentrations that the maximal values of the thermoelectric power factor of the material  $(Z = S^2 \sigma)$  are observed. Moreover, the significant impurity concentrations allow the features of the kinetic characteristics of semiconductors to be observed at sufficiently high temperatures [7, 8].

# 2. Experimental Method

The specimens of the  $ZrNi_{1-x}Co_xSn$  solid solution were fabricated using the method of electric arc melting of initial elements on a water-cooled copper bottom in a purified argon atmosphere. The alloys were subjected first to the homogenizing annealing in evacuated quartz ampoules at a temperature of 1070 K for 720 h, and, afterwards, they were gradually cooled down to room temperature during 24 h. After carrying out the complex of electrophysical and structural researches, the specimens were subjected again to the homogenizing annealing at 1070 K for 120 h and then quickly quenched following the technique which is applied in crystal chemistry for studying the isothermal cross-sections of multicomponent systems. The x-ray phase analysis was fulfilled making use of DRON-2.0 (Fe $K_{\alpha}$ -emission) and HZG-4A (Cu $K_{\alpha}$ -emission) diffractometers. The calculation of lattice parameters and the more precise determination of the crystalline structure were carried out by means of the program package CSD [18]. The electrical resistance  $\rho$ , the Seebeck coefficient S with respect to copper, and the magnetic susceptibility  $\chi$ (using the Faraday method) were measured in the temperature interval 80-380 K.

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Fig. 1. Temperature dependences of the specific resistance  $\rho$  of the hardened (nonprimed numbers) and tempered (primed numbers) *n*-ZrNiSn specimens with various acceptor impurity concentrations  $N_A$  (in cm<sup>-3</sup> units):  $1 - 8.7 \times 10^{19}$  (x = 0.005),  $2 - 1.7 \times 10^{20}$  (x = 0.01),  $3 - 3.5 \times 10^{20}$  (x = 0.02),  $4 - 7.0 \times 10^{20}$  (x = 0.04),  $5 - 1.1 \times 10^{21}$  (x = 0.06),  $6 - 1.4 \times 10^{21}$  (x = 0.08), and  $\gamma - 1.7 \times 1021$  (x = 0.1)

### 3. Experimental Results and Their Discussion

The X-ray phase and structural analyses of the specimens under investigation confirmed that all the  $\text{ZrNi}_{1-x}\text{Co}_x\text{Sn}$  specimens were single-phase, and the distribution of atoms in them corresponds to the MgAgAs structural type (space group  $F\bar{4}3m$ ): 4 Ni in 4(a) 0 0 0 (Ni +Co), 4 Sn in 4(c)  $\frac{1}{4}$   $\frac{1}{4}$   $\frac{1}{4}$ , and 4 Zr in 4(d)  $\frac{3}{4}$   $\frac{3}{4}$   $\frac{3}{4}$ .

In Figs. 1 and 2, the temperature dependences of the electrical resistance and the Seebeck coefficient are shown for tempered and hardened *n*-ZrNiSn specimens with various concentrations of acceptor impurities. The dependences  $\ln \rho(1/T)$  and S(1/T) obtained for the tempered  $\operatorname{ZrNi}_{1-x}\operatorname{Co}_x$ Sn specimens with  $x \leq 006$  are typical of semiconductors. These dependences allow one to distinguish between high- and low-temperature activation sections, which we associate with the activation of electrons or holes from the Fermi level into the non-local states in the relevant continuous energy bands and with the hopping conduction, respectively. If  $x \geq 0.08$ , the activation sections disappear, and the



Fig. 2. The same as in Fig. 1, but for the Seebeck coefficient  ${\cal S}$ 

dependences acquire metallic character — the transition insulator-metal takes place.

For hardened specimens, the dependence  $\ln \rho(1/T)$ differs fundamentally from the previous case. First, the conductivity of hardened specimens considerably exceeds that of tempered ones by value (but a specimen with x = 0.02). Secondly, these specimens do not reveal the low-temperature activation section which is associated with the hopping conduction. Instead, a reduction of the conductivity is observed as the temperature grows, which is characteristic of metals and is caused by the increasing contribution of charge carrier scattering to conductivity.

For the tempered and hardened specimens, the increase of the acceptor impurity concentration leads to the increase of the Seebeck coefficient by absolute value in the temperature interval where the hopping conduction of the tempered specimens is observed; and the sign of the coefficient testifies that the dominating contribution to the conductivity is made by free holes that were formed on the ionization of acceptor impurities. In addition, at  $x = 0.005 \div 0.06$ , there appear the extremes in the dependences S(1/T), the positions of which are shifted towards higher temperatures as the impurity concentrations increase. The extremes evidence for the existence of at least two competing mechanisms of conductivity: by means of free holes, which emerge

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Fig. 3. Dependences of the Seebeck coefficient S at 80 (1) and 300 K (2<sup>\*</sup>), the electrical conductivity  $\sigma$  at 80 (3) and 300 K (4), and the magnetic susceptibility  $\chi$  (5) on the acceptor impurity concentration for tempered *n*-ZrNiSn specimens. The ordinates of curve 2<sup>\*</sup> are scaled up by a factor of four

owing to the ionization of controllable acceptor impurities in the low-temperature range, and free electrons, which are a result of the ionization of uncontrollable donor impurities at relatively high temperatures. Whence, a conclusion follows that the depth of the donor levels reckoned from the edge of the conduction band is larger than the depth of the acceptor levels reckoned from the edge one.

As the temperature grows, the dependence S(1/T), having reached its maximum, becomes linear, until its sign changes at  $x = 0.005 \div 0.02$ . For the values x >0.04, the Seebeck coefficient is positive in the studied temperature interval. At x > 0.06, the maximum of the dependence S(1/T) lies outside the experimental temperature interval, and the conductivity is determined by free holes only.

The analysis of the dependences  $\ln \rho(1/T)$  and S(1/T) measured for tempered and hardened specimens testifies that the concentration of charge carriers in hardened specimens, which take part in conductivity, considerably exceeds that in tempered ones. The fact that the Seebeck coefficient changes its sign, if the temperature lies in the studied temperature interval, even at the acceptor impurity concentration  $N_A = 3.5 \times 10^{20} \text{ cm}^{-3}$  (x = 0.02) evidences for the availability of a significant concentration of uncontrollable donor impurities in the specimens.



Fig. 4. Dependences of the Seebeck coefficient S at 80  $(1^*)$  and 300 K (2) and the electrical conductivity  $\sigma$  at 80 (3) and 300 K (4) on the acceptor impurity concentration for hardened *n*-ZrNiSn specimens. The ordinates of curve  $1^*$  are scaled up by a factor of five

The dependences of the conductivity, the Seebeck coefficient, and the magnetic susceptibility for the specimen tempered at 300 K, which are exhibited in Figs. 3 and 4, illustrate the mechanisms of conductivity in the investigated *n*-ZrNiSn specimens more neatly. For example, in the dependences S(x) and  $\sigma(x)$  for the hardened specimens, there appear oscillations similar to those observed in the hardened *n*-ZrNiSn semiconductors doped with acceptor impurities by substituting Sc atoms for Zr ones [2, 4].

Similarly to the extremes in the dependences S(1/T), those in the S(x) ones are also a result of several competing processes. For the tempered specimens, the growing S within the interval  $x = 0.005 \div$ 0.02 testifies to the reduction of the free electron contribution to the semiconductor conductivity, owing to the appearance and increase of the free hole one, whereas the diminution of S(x) at x > 0.02 evidences for the dominating contribution of free holes to the total conductivity of semiconductors. This statement agrees with the conclusions following from the analysis of the dependences  $\sigma(x)$  obtained for tempered specimens. Really, since the conductivity of a semiconductor is proportional to the density of states at the Fermi level  $(\sigma \sim N(E_{\rm F}))$ , and  $S \sim k_{\rm B}T/E_{\rm F}$ , the observable synchronism in the behavior of the dependences S(x)and  $\sigma(x)$  at 0.005 < x < 0.02 and x > 0.02 becomes clear. The growth of the S(x) dependence points at a reduction of the density of states at the Fermi level

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which is caused by the variation of the semiconductor compensation factor and, as a consequence, by an insignificant decrease of  $\sigma(x)$  at T = 300 K.

This conclusion is confirmed by the behavior of the magnetic susceptibility  $\chi(x)$  (the investigated semiconductors are Pauli paramagnets, in which  $\chi \sim N(E_{\rm F})$ ). In the range of the acceptor impurity concentrations  $x \leq 0.01$ , where the dependences  $\chi(x)$ fall down, the dependences S(x) grow. If  $x \geq 0.02$ , the monotonous growth of the dependences  $\chi(x)$  and the decrease of the dependence S(x) are observed.

To summarize the aforesaid for the tempered specimens, we note that the conductivity of *n*-ZrNiSn semiconductors with low (x < 0.01) and high (x > 0.07) concentrations of acceptor impurities is governed by electrons in the conduction band and holes in the valence one, respectively. In the intermediate concentration range of acceptor impurities  $(0.01 \le x \le 0.07)$ , the conductivity is determined by at least three mechanisms: the activation of electrons into the conduction band, the activation of holes into the valence band, and the hopping conduction over localized states.

In the case of hardened specimens, the behavior of S(x) correlates well with that of  $\sigma(x)$ , as it was in the tempered specimens. It is evident from Fig. 4 that the maxima of the dependence  $\sigma(x)$  correspond to the minima of the dependences S(x). The oscillatory character of those dependences reflects the variation dynamics of the density of states at the Fermi level and is associated with the reconstruction processes of the donor and acceptor impurity bands of the semiconductor owing to the changes of the compensation factor, the dimensions of the bands, and their arrangement with respect to the tops of the continuous energy bands. The models of a single impurity band in n-ZrNiSn doped with acceptor impurities or *p*-TiCoSb doped with donor ones are not valid in our case because of a high concentration of uncontrollable donor impurities which are supposed to form their own donor impurity band. The qualitative analysis of the mechanisms of activation processes in the heavily doped semiconductors under investigation, which is given below, can serve as an evidence for that. In other words, three questions started with "What", "Where from", and "Where to" are to be answered.

From the dependences

$$S = (k_{\rm B}/e)(\varepsilon/k_{\rm B} + A), \tag{1}$$

where the coefficient A depends on the charge carrier scattering origin and was taken by us to equal unity [8], and

$$\sigma = \sigma_0 \exp(-\varepsilon/k_{\rm B}T),\tag{2}$$

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the activation energies of charge carriers from the Fermi level were determined for the specimens of both types (see the table). The table data demonstrate that, in the case of tempered specimens, the activation energy monotonously decreases as the acceptor impurity concentration grows. If electrons are assumed to be activated from the Fermi level into the conduction band, the reduction of the activation energy means the displacement of the Fermi level towards the conduction band, provided that we introduce the acceptor dopant. Such a situation is typical of semiconductors and is observed in semiconductors with the conductivity of the electron type within the interval of their transition from the case of strong compensation  $(1 - K \ll 1)$  to the case of weak one  $(K = N_A/N_D \ll 1)$ , when the Fermi level drifts from the bottom to the top of the donor impurity band [7].

We pointed out above that the extremes in the dependences S(x) and S(1/T) are associated with, at least, the contributions of free electrons (a result of the donor impurity ionization) and free holes (a result of the acceptor impurity ionization) to the conductivity. Therefore, it is not clear how to explain the drift of the Fermi level towards the top of the impurity valence band in the framework of the single donor band model, if the majority charge carriers at x >0.02 are free holes and the Fermi level, according to the electroneutrality condition, has to be fixed about the midpoint of the energy gap (provided that the concentrations of donor and acceptor impurities are mutually comparable and those impurities are ionized), taking into account that such an explanation has to be consistent with the data for tempered specimens quoted in the table.

While analyzing the behavior of the dependences S(x) and  $\chi(x)$  for tempered specimens in the range of acceptor impurity concentrations  $0 < x \leq 0.02$ , one may assume that if the compensation factor of the semiconductor should change following the mechanism described above [7], the Fermi level would drift from the bottom to the top of the donor impurity band. The

Activation energies for tempered and hardened n-ZrNiSn specimens with various acceptor impurity concentrations

x	$N_A, \mathrm{cm}^{-3}$	Tempered		Hardened	
		$\varepsilon_1^{\rho}$ , meV	$\varepsilon_1^S$ , meV	$\varepsilon_1^{\rho}, \text{meV}$	$\varepsilon_1^S$ , meV
0.005	$8.7 \times 10^{19}$	252	236	145	202
0.01	$1.7 \times 10^{20}$	216	173	91	115
0.02	$3.5 \times 10^{20}$	115	108	116	166
0.04	$7.0 \times 10^{20}$	75	65	45	56



Fig. 5. Model of the impurity band reconstruction in n-ZrNiSn at doping the latter with acceptor impurities

activation energy of electrons in the conduction band would decrease at that (see the table).

Provided that the concentration of acceptor impurities (x > 0.02) exceeds that of uncontrollable donor impurities, the overcompensation of the semiconductor from the n- to the p-type conductivity occurs, and the Fermi level becomes fixed by the acceptor band. The semiconductor becomes strongly compensated, and the Fermi level turns out located near the top of the acceptor impurity band. A further increase of the acceptor impurity concentration brings about the case of weak compensation  $(N_D/N_A \ll 1)$ , and the Fermi level drifts from the top of the acceptor impurity band to its bottom and, at x > 0.08, enters the continuous energy interval in the valence band [1-8]. The observed reduction of the activation energy for specimens with x = 0.02 and 0.04 can be explained in such a way.

The fact that the values of the activation energy, which were deduced from the electrical conductivity dependences for the tempered specimens, exceed those obtained from the corresponding thermopower dependences is quite natural for heavily doped semiconductors, which the hopping conduction is typical of. The activation energy of mobility w(T) contributes to the slope of the straight line ln  $\rho(1/T)$  rather than the S(1/T) one [8].

The consideration above also explains the results concerning the nonmonotonous variation of the activation energy in hardened specimens as the acceptor impurity concentration increases. In particular, these are the illogical, at first sight, jumps of the relevant values at x = 0.01 and 0.02 (the results obtained were

reproduced many times and are not of stochastic origin, as was supposed first). It is evident from Fig. 4 that the maximum of the dependence S(x) is located at  $x \approx 0.02$ , with holes already being the majority charge carriers. That is, the activation energy in the hardened specimens reduces at x = 0.005 and 0.01, following the mechanism described above and owing to the drift of the Fermi level from the bottom to the top of the donor impurity band. On the other hand, the reduction of the activation energy at x = 0.02 and 0.04 stems from the drift of the Fermi level from the top to the bottom of the acceptor impurity band. That is why the monotonous diminution of the activation energy with the variation of the acceptor impurity concentration for tempered specimens is accidental, because the activation proceeds from various impurity levels and happens into various continuous energy bands. The values of the activation energy, which are quoted in the table, can be conditionally split into two groups, and the three questions put above can be answered: the electrons from the donor impurity band, at  $x = 0.005 \div 0.01$ , and the holes from the acceptor impurity band, at  $x = 0.02 \div 0.04$ , are activated into the conduction and the valence band, respectively (see Fig. 5).

In the case of hardened specimens, the values of the activation energy obtained from the electrical conductivity dependences are lower than those deduced from the corresponding thermopower dependences. It may take place owing to the absence of the hopping conduction mechanism (metallization of conductivity in the impurity band [8]), which was paid attention earlier.

The maximum in the dependence S(x) and the corresponding minimum in the dependence  $\sigma(x)$  at  $x \approx$ 

0.07, which were observed for hardened specimens at T = 300 K, are obviously attributed with the fixation of the Fermi level by the mobility edge of the valence band [2–6,8].

Since we investigated the same specimens, which, after the homogenizing annealing, were cooled down in different regimes, it is, in our opinion, the mechanism of cooling of polycrystalline semiconductors with the structure of the MgAgAs type that governs the position of the Fermi level and the concentrations of the majority and minority charge carriers. In addition, the preliminary results of structural researches showed that the degree of local deformations in crystallites of tempered and hardened specimens substantially depends on the mode of polycrystalline specimen cooling.

# 4. Conclusions

From the above discussion, it appears that the suggested model of the reconstruction of the donor and acceptor impurity bands in n-ZrNiSn semiconductor at doping the latter with acceptor impurities adequately describes the mechanisms of conductivity in this semiconductor. The analysis of the corresponding kinetic characteristics testifies that the tempering process makes a smaller contribution to the defect formation than the hardening one does, which may be caused, in particular, by different degrees of local deformations in polycrystalline specimens.

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#### ВПЛИВ РЕЖИМІВ ТЕРМООБРОБКИ НА ПРОВІДНІСТЬ СИЛЬНОЛЕГОВАНИХ НАПІВПРОВІДНИКІВ *n*-ZrNiSn. 1. ЕКСПЕРИМЕНТАЛЬНІ РЕЗУЛЬТАТИ

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Резюме

Запропоновано модель перебудови домішкових зон напівпровідника *n*-ZrNiSn при легуванні акцепторними домішками. Встановлено, що в сильнолегованих інтерметалічних напівпровідниках *n*-ZrNiSn для однакових концентрацій акцепторних домішок гартування зразків у порівнянні з відпуском суттєво змінює положення рівня Фермі, концентрації основних та неосновних носіїв струму і може бути, зокрема, зумовлене різним ступенем локальних деформацій полікристалічних зразків.