EFFECT OF GAMMA RADIATION ON THE ELECTRICAL PROPERTIES OF A SOLID SOLUTION $Bi_{0.85}Sb_{0,15}$ <Te> MODIFIED WITH ZrO_2

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The effects of gamma irradiation on the electrical properties of extruded $Bi_{0.85}$ $Sb_{0.15}$ samples modified with ZrO_2 containing tellurium donor impurities in the temperature range ~77÷300K have been studied. It was found that radiation defects appear during irradiation, which play the role of donor centers, as a result of which the concentration of free electrons n, and, consequently, the electrical conductivity σ increases, and the thermopower coefficient α decreases. These defects, scattering current carriers, reduce their mobility μ. With an increase in the radiation dose, the concentration of defects also increases, and free carriers are captured at the level of the radiation defect. In modified Bi $_{0.85}$ Sb $_{0,15}$<Te> with radiative introduction of acceptor (negatively charged) centers, there is a partial neutralization of the ionic cores, a decrease in the efficiency of impurity scattering of charge carriers and, accordingly, some increase in mobility.

Keywords: Extrusion, electrical conductivity, alloying, modification, radiation, defects.
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INTRODUCTION

Solid solutions of Bi-Sb systems are the most effective materials for creating various low-temperature thermo- and magnetothermoelectric energy converters [1,2]. Particularly promising in this direction are high-strength extruded materials based on these systems [3–5]. The method has a high productivity and open up wide possibilities for profiling thermoelement legs and obtaining a homogeneous matrix microstructure.

Thermoelectric cooling is the best technical solution for the problems of temperature reduction and thermal stabilization of elements of microelectronics, optoelectronics and lighting engineering. Thermoelectric fine-grained materials, which are mechanically stronger than materials obtained by crystallization from a melt, and an increase in thermoelectric efficiency in them can be achieved by reducing the lattice thermal conductivity, as a result of an increase in phonon scattering at grain boundaries [6]. An urgent task of thermoelectricity is the search for appropriate thermoelectric materials, which should have a high thermoelectric figure of merit (figure of merit) $Z=\alpha^2\sigma/\chi$, where σ, α and χ are the coefficients of electrical conductivity, thermoelectric power and thermal conductivity, respectively. To obtain a material with the required parameters, it is necessary to establish the regularities of the influence of the composition, production mode, grain size, doping, charge carrier concentration and the conditions for scattering of electrons and phonons, leading to a sufficiently high ratio of current carrier mobility to lattice thermal conductivity μ/χ_p, which directly affects thermoelectric efficiency of the material. In recent years, the way to increase the efficiency of thermoelectric materials is reduced to the creation of two-dimensional and three-dimensional defects in the crystal structure, the distance between which is commensurate with the mean free path of charge carriers or the wavelength of acoustic phonons responsible for heat transfer. This leads to a change in the parameters of the energy spectrum of charge carriers and to varying degrees affects the processes of scattering of charge carriers and phonons [7]. These assumptions are based on the possibility of creating conditions under which a section of a material with different physical properties scatters thermal vibrations more strongly than electrons and holes. The effect can be achieved, for example, by introducing a finely dispersed second phase into the substance matrix (similar to the introduction of the second phase into the material during its dispersion strengthening) [8].

One of the promising methods is also the development of a technology for increasing the efficiency of thermoelectric material through modification. The essence of the method is the introduction of a scattering phase matrix (modifier) with a thermal expansion coefficient (*TEC*) different from the *TEC* of the semiconductor substance (*SPW*) into a SPW. As a result of the difference in the *TEC* of the semiconductor and the modifier, after cooling, elastically stressed zones are formed in the pressed *SPW*. The creation of such strained zones in the lattice of a thermoelectric matrix leads to the fact that the thermal conductivity of the matrix decreases more than its electrical resistance increases [9].

The use of extruded samples of solid solutions based on the Bi-Sb system for the creation of electronic devices significantly improves the performance of electronic equipment. These devices are often used in radiation conditions. However, the radiation resistance of such electronic devices does not fully meet the needs of modern electronics, which are subject to the requirements of resistance to radiation exposure. The formation of radiation defects in semiconductor materials, affecting their physical properties, changes the parameters of the device based on it [10], and insufficient attention was paid to the study of the effect of gamma radiation on the electrical and thermal properties of solid solutions of Bi-Sb systems.

Taking this into account, in order to elucidate the features of the influence of radiation defects (RD) on the electrical properties of solid solutions of Bi-Sb systems, we obtained extruded samples of $Bi_{0.85}Sb_{0,15}$doped with tellurium and modified with ZrO_2, studied the patterns of changes in electrical conductivity, thermal emf coefficient. with. and Hall in extruded pure and tellurium samples of $Bi_{0.85}Sb_{0,15}$solid solutions modified with high-temperature zirconium oxides (ZrO_2) from a dose of gamma radiation in the temperature range ~77÷300*K*. Non-irradiated samples and samples irradiated with 1 *Mrad*, 10 *Mrad* and 50 *Mrad* with gamma quanta were examined.

EXPERIMENTAL PART

Extruded samples of $Bi_{0.85}Sb_{0,15}$<Te> solid solutions modified with ZrO_2 were obtained in the following technological sequence: composition synthesis from initial components; mechanical grinding of the alloy in a porcelain mortar and selection of a fraction with a particle size of ~0.5*mm;* mechanical mixing of powders of the alloy and modifier ZrO_2 1 wt.%, production from it by cold pressing at room temperature of briquettes with a diameter of ~30*mm* for extrusion; extrusion of fine billets. The introduction of modifier particles into the matrix is carried out during the annealing of the workpiece at temperatures between the liquidus and solidus temperatures of the *PPV* (melting). The modifier is evenly distributed from the liquid part of the volume, the proportion of which can be varied by the annealing temperature in accordance with the state diagram. The melting process has a positive effect on the properties of the thermoelectric material.

The technology under consideration makes it possible to obtain composite materials with dispersed structures - with particles of the second phase uniformly distributed in the bulk of the semiconductor. In order for the distance between the particles to be sufficiently small at a small amount of the dielectric phase in the semiconductor, the particle sizes must also be small. ZrO_2 particles were used as the modifying phase (ZrO_2 powder was obtained by the plasma-chemical method, the average particle diameter is ~50*nm*, the melting point is ~2950*K*).

Since the concentration of charge carriers and their mobility are critical parameters for a thermoelectric material, and modification should not lead to an uncontrolled change in the composition of the semiconductor matrix, it is necessary that the particles of the second phase be stable in contact with the matrix - there is no possibility of dissolution or interaction with the formation of new phases. These requirements are met by some high-temperature compounds, in particular, oxides. Confirmation of the stability of the modifying phases is the invariability of the properties of the material during long-term testing.

Bismuth "*VI-0000*", antimony "*SU-0000*" and tellurium *T-sCh*, distilled (or doubly sublimated) were used as initial components. Impurities and starting components were weighed with an accuracy of ±0.0001*g*. Samples with a concentration of 0.0001 at.% Te were obtained by fusing an appropriate amount of a $Bi_{0.85}Sb_{0,15}$sample doped with 0.1 at.% Te with a pure $Bi_{0.85}Sb_{0,15}$ sample.

The initial components in a stoichiometric ratio were placed in a quartz ampoule, previously etched in a chrompeak solution ($K_2Cr_2O_7$ + H_2SO_4) and washed with distilled water. The synthesis was carried out at ~ 673*K* for 2 hours, by direct fusion of the components, in quartz ampoules evacuated to ~10^{-2}*Pa*. During the synthesis, the ampoule with the substance was constantly subjected to rocking. The ampoule with the synthesized substance was abruptly cooled to room temperature by dipping into water.

Extrusion was carried out on an *MS*-1000 hydraulic press using special equipment. Technological parameters of extrusion were: $T_{ex.}=475 \sim 3K$; $R_{ex.}=480\ MPa$, press travel speed $V_{ts}= 0.02cm/min$, drawing ratio -25.

It was found that polycrystals of Bi-Sb solid solutions obtained by extrusion have high mechanical properties compared to single-crystals of the same composition. In addition, by selecting certain modes of extrusion and subsequent annealing, it is possible to obtain a thermoelectric material with a quality factor close to the quality factor of single crystals, which makes it very promising for use in the field of thermoelectric energy conversion.

The texture and X-ray diffraction patterns were studied at the XR D8 ADVANCE X-ray unit, Bruker, Germany, using a *D2* Phaser diffractometer, Brucker, using CuK radiation from the studied samples by the method described in [11].

The bending strength of the obtained extruded samples of $Bi_{0.85}$ $Sb_{0,15}$ solid solutions is ~3 times higher than the strength of single-crystal samples of this composition, measured by the method described in [12]. After extrusion, the samples were annealed at a temperature of ~503*K* in evacuated quartz ampoules up to ~10^{-1}*Pa*. The samples were irradiated with gamma quanta (gamma radiation) in a ^{60}Co isotope source with different doses (1, 10 and 50 *Mrad*).

The absorbed dose of radiation in the systems under study was determined by comparing the electron densities of the dosimetric systems described in [13]. The electrical and thermal parameters of the samples were measured by the method described in [14] along the length of the sample (rod), i.e. in the direction of extrusion. The errors in the measurements of electrical parameters do not exceed ~3%.

RESULTS AND DISCUSSION

In $Bi_{0.85}Sb_{0,15}$samples doped with tellurium and modified with ZrO_2, gamma radiation in various doses, changing the values of electrical conductivity

(σ) and thermoelectric power (α), does not significantly affect the course of the temperature dependence (except for the sample irradiated with 10 *Mrad*). Irradiation with gamma quanta at low doses increases the electrical conductivity σ, and the thermopower coefficient α almost does not change (fig.1). As the dose increases, σ of modified irradiated samples decreases. Some increase (especially at 50 *Mrad*) of the total thermal conductivity is observed in the irradiated $Bi_{0.85}Sb_{0,15}$<Te> modified ZrO_2 samples compared to the unirradiated sample.

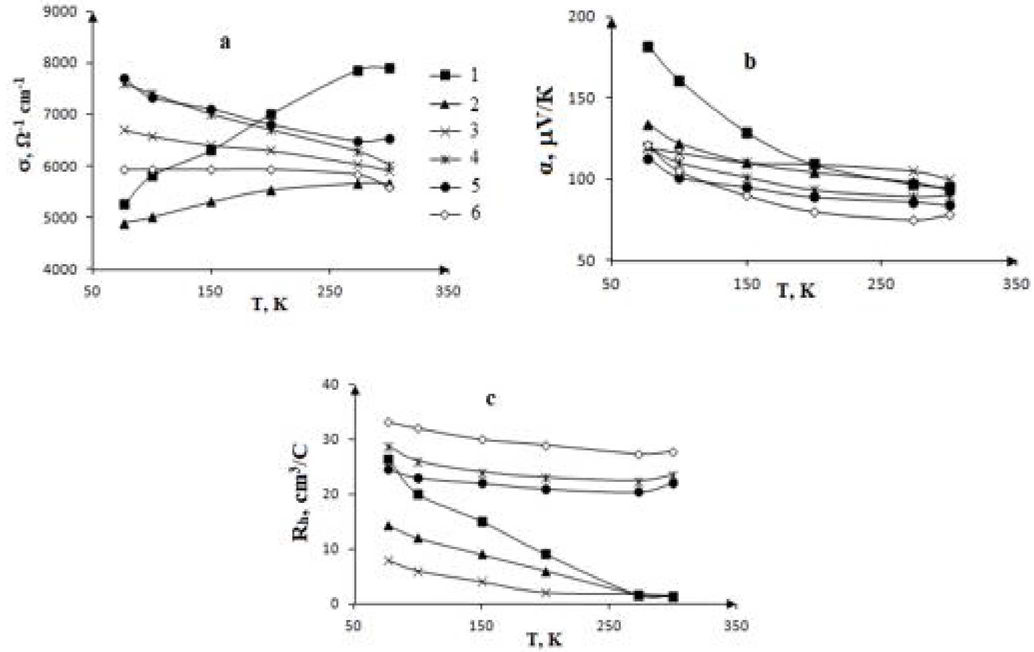

Fig.1. Temperature dependences of electrical conductivity σ (a), thermoelectric coefficients α (b) of Hall (R_H)) (c) extruded samples of $Bi_{0.85}Sb_{0,15}$<Te> modified ZrO_2 solid solution. 1- $Bi_{0.85}Sb_{0,15}$ unmodified and non-irradiated sample; 2- $Bi_{0.85}Sb_{0,15}$+1%ZnO_2 non-irradiated; 3- $Bi_{0.85}Sb_{0,15}$<Te> modified with ZrO_2; 4-6 samples of $Bi_{0.85}Sb_{0,15}$<Te> modified ZrO_2 irradiated with gamma quanta of 1*Mrad*, 10*Mrad* and 50 *Mrad*, respectively.

The thermoelectric figure of merit (Z) of a material depends on the concentration of charge carriers, which is determined by the alloying level of the alloy and the structural state of energetically active self-superstoichiometric and impurity atoms.

Irradiation, as well as plastic deformation during extrusion, especially in the case of composite materials (modified samples), leads to a change in the structural state, i.e. to a change in the parameters of the energy spectrum of charge carriers near the Fermi energy and the electrical properties of intrinsic and impurity defects.

The optimal direction of growth of single crystals of Bi-Sb systems is the crystallographic direction [110] of the rhombohedral cell. However, the highest value of the thermoelectric figure of merit, as well as electrical conductivity, is observed in another crystallographic direction [111], which is perpendicular to the optimal direction of single crystal growth. In these single crystals, the most perfect plane is the [111]plane, along which a split always occurs [15].

During extrusion, due to plastic deformation, some polycrystal grains are oriented so that their trigonal axis becomes parallel to the extrusion axis, i.e. texture is formed. At the same time, as a result of plastic deformation, various defects of the crystal lattice arise in individual grains. In this case, these structural defects are predominantly concentrated between the [111]cleavage planes. The degree of texture will depend on the technological parameters of the extrusion process, on the grain size and post-extrusion heat treatment. During heat treatment, the misorientation of grains due to thermal energy can also occur, i.e. change in the degree of texture of the extruded sample [5].

The introduction of the ZrO_2 modifier into extruded samples increases the dislocation density while increasing the uniformity of its distribution. This leads to a decrease in the scattering of electrons and phonons, i.e. to an increase in the mobility of charge carriers. Modification in all cases increases the proportion of the texture. By creating a microscopically homogeneous distribution of stoppers for moving dislocations, the modification increases the degree of uniformity of the strain distribution.

The effect of modification on the thermoelectric properties of extruded $Bi_{0.85}Sb_{0,15}$samples can be explained both by the processes of structure formation during extrusion and post-formation annealing, and by the properties of the semiconductor matrix.

An analysis of the influence of modifying particles on the deformation textures allows us to say that the determining action of large particles is the blocking of dislocation slips in the basal planes, while small spherical particles activate pyramidal slips and, to a certain extent, block the basal slip. Pyramidal particles activate multiple sliding and, to a lesser extent than others, prevent basic sliding. The difference in the substructure of the deformed material determines the mechanisms of nucleation of recrystallization centers and the mobility of migrating boundaries. The possibility of growth of centers of dynamic recrystallization can be affected by stresses arising in the matrix in the presence of modifier particles due to the difference in their thermal expansion coefficients and deceleration of boundary motion by foreign particles. Scattering of charge carriers by dislocations and dislocation walls is greater along the basal planes than along the principal axis of symmetry in deformed extruded materials.

From the temperature dependence of the Hall coefficient of the modified extruded $Bi_{0.85}Sb_{0,15}$<Te> samples, it can be seen that in all irradiated samples with a modifier, $R_H$exceeds the non-irradiated samples. It is assumed that this is due to the mobility of charge carriers. For all unirradiated samples, especially undoped unmodified samples, a strong change in the Hall coefficient, and hence the concentration of charge carriers, occurs in the temperature range of 77÷200*K*. In the temperature range of 200÷300K, the change in Rx slows down with increasing temperature. For all irradiated tellurium-doped $Bi_{0.85}Sb_{0,15}$samples, the temperature dependence of $R_H$weakens.

Calculated $\mu=\sigma R_H$ from the experimental values of the electrical conductivity and the Hall coefficient, the mobility μ of all irradiated samples is much greater than in non-irradiated modified samples (Table.). At the same time, as the irradiation dose increases, the mobility of charge carriers decreases, except for the sample irradiated with 10 Mrad gamma rays. For all undoped and tellurium-doped $Bi_{0.85}Sb_{0,15}$modified samples, the mobility μ, in the entire range of the studied temperatures, decreases with increasing temperature (fig. 2).

Doping with tellurium leads to a decrease in the absolute value of the mobility relative to the undoped sample. In extruded $Bi_{0.85}Sb_{0,15}$samples, the mobility of charge carriers is largely determined by scattering on structural defects of deformation origin. Modification, creating large amounts of stoppers for moving dislocations, promotes the formation of dislocation structures with a lower concentration of nonequilibrium point defects (spatial inhomogeneity), leads to greater chemical homogeneity in composition, and the degree of deformation during extrusion of composite rods is significantly higher than in the case of deformation of a semiconductor matrix (unmodified) [8].

In samples doped with tellurium, the electrical conductivity increases in magnitude compared to an undoped sample, since the concentration of charge carriers increases

Modification and irradiation itself can be used as a tool for creating heterogeneous materials with desired structural properties.

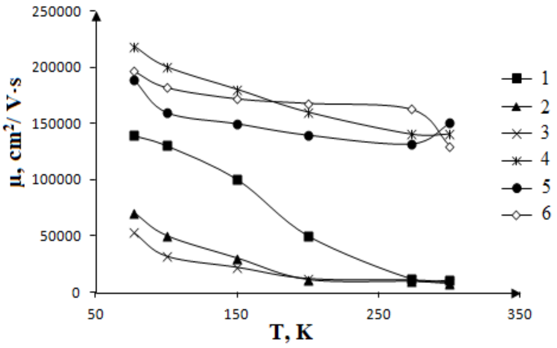

Fig. 2. Temperature dependences of the mobility (μ) of extruded samples of the $Bi_{0.85}Sb_{0,15}$<Te> solid solution modified with ZrO_2. The designations are the same as in Fig. 1

At low doses of irradiation (1*Mrad*), the electrical conductivity σ in the samples increases, while the thermoelectric coefficient α decreases. With an increase in the radiation dose, the concentration of defects also increases, and free carriers are captured at the level of the radiation defect. In this regard, the concentration of carriers of conditioned charged defects n and, consequently, σ of the sample fall, the Fermi level shifts to the depth of the band gap, the thermoelectric coefficient and mobility increase.

The irradiated samples of $Bi_{0.85}Sb_{0,15}$ (semiconductor) are a material with a high degree of compensation. This is what makes it possible to consider the radiation modification of the properties of a semiconductor as a process “reverse” to doping with chemical impurities, as a result of which the initial electrical activity of the material decreases and the degree of its compensation increases.

The effect of radiation on the electrical properties of modified extruded samples of the $Bi_{0.85}Sb_{0,15}$solid solution shows that a highly irradiated semiconductor is always a material with a low concentration of free current carriers, a high concentration of charge bound to defects, and a degree of compensation of radiation donors and acceptors close to unity.

Under irradiation, a process of lowering the initial electrical activity of the material occurs in the sample, as a result of which the Fermi level is shifted from its initial position and is fixed near a certain level position characteristic of a given semiconductor. The electronic parameters of the irradiated material depend on the features of the band spectrum of the semiconductor in the energy range near its minimum band gap, i.e. are determined by the position of the Fermi level relative to the nearest extrema of the conduction band or valence band [16].

In modified samples of the $Bi_{0.85}Sb_{0,15}$solid solution, as in silicon crystals, silicon has the so-called low-dose effect, i.e. anomalous change (increase) in the mobility of current carriers during irradiation with gamma quanta in the region of mixed scattering. The increase in mobility in the modified $Bi_{0.85}Sb_{0,15}$is associated with the radiative introduction of acceptor (negatively charged) centers, which at low doses are generated mainly in the regions of positive ionic cores; as a result, the ionic cores are partially neutralized, which leads to a decrease in the efficiency of impurity scattering of carriers. charge and partially neutralized centers and, accordingly, to some increase in mobility.

Table

Electrical and thermal parameters of extruded samples of $Bi_{0.85}Sb_{0,15}$<Te> solid solution modified with ZrO_2.

Radiation doses	Compositions	At 77*K*				
		σ $\Omega^{-1}cm^{-1}$	*α* *μV/K*	$R_h \cdot 10^{-8}$ cm^3/C	*μ* $cm^2/V \cdot s$	*n* cm^{-3}
0*Mrad*	$Bi_{0,85}Sb_{0,15}$	5250	-182	-26,5	139125	$0,24 \cdot 10^{18}$
	$Bi_{0,85}Sb_{0,15}$+1%ZrO_2	4899	-134	-14,33	70203	$0,44 \cdot 10^{18}$
	$Bi_{0,85}Sb_{0,15}$+0,0001Te +1%ZrO_2	6704	-119	7,9	52962	$0,79 \cdot 10^{18}$
1 *Mrad*	$Bi_{0,85}Sb_{0,15}$+0,0001Te +1%ZrO_2	7598	-120	8,7	218063	$0,22 \cdot 10^{18}$
10*Mrad*	$Bi_{0,85}Sb_{0,15}$+0,0001Te +1%ZrO_2	7698	-113	4,5	188601	$0,26 \cdot 10^{18}$
50*Mrad*	$Bi_{0,85}Sb_{0,15}$+0,0001Te +1%ZrO_2	5937	-121	3,1	96515	$0,19 \cdot 10^{18}$

Irradiation leads to a decrease in the concentration of structural defects resulting from plastic deformation of the crystal lattice in individual grains in extruded $Bi_{0.85}Sb_{0,15}$samples, an increase in the electron mobility and an increase in the prevalence of current carrier scattering on lattice vibrations.

The results of the data obtained indicate that when irradiated with gamma quanta, not only the generation of radiation defects (centers) occurs, but it is also accompanied by their rearrangement. Restructuring significantly depends on the initial level of modification of the ingot, from which the corresponding samples for research were made.

The radiation-stimulated increase in charge mobility (due to the introduction of acceptor-type point defects and local mechanical stresses, which are certainly higher), is probably associated with the specifics of the interaction of radiation centers and with defects arising as a result of plastic deformation of the crystal lattice in individual grains.

Thus, modification makes it possible to obtain composite materials with disperse structures and charge carriers with higher mobility. Irradiation leads to a decrease in the concentration of structural defects arising as a result of plastic deformation, the crystal lattice in individual grains in extruded $Bi_{0.85}Sb_{0,15}$ samples, and an increase in the mobility of current carriers.

CONCLUSION

It was found that, at low doses (1*Mrad*) of irradiation, in unmodified samples of the $Bi_{0.85}Sb_{0,15}$solid solution, radiation defects appear that play the role of donor centers, as a result of which the concentration of free electrons *n* and consequently, the electrical conductivity σ increases, and the thermopower coefficient *α* decreases. These defects, scattering current carriers, reduce their mobility *μ*. With an increase in the radiation dose, the concentration of defects also increases and free carriers are captured at the level of the radiation defect. In modified $Bi_{0.85}Sb_{0,15}$<Te> with radiative introduction of acceptor (negatively charged) centers, which at low doses are generated mainly in the regions of positive ionic cores, the ionic cores are partially neutralized, the efficiency of impurity scattering of charge carriers decreases and, accordingly, some increase in mobility occurs. When irradiated with gamma rays, not only the generation of radiation defects (centers) occurs, but also their rearrangement, which leads to a change in electrical parameters.

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ZrO_2 İLƏ MODİFİKASİYA OLUNMUŞ $Bi_{0,85}Sb_{0,15}$<Te> BƏRK MƏHLULU EKSTRUZİYA NÜMUNƏLƏRİNİN ELEKTRİK XASSƏLƏRİNƏ QAMMA ŞÜALARININ TƏSİRİ

ZrO_2 ilə modifikasiya olunmuş, tellurla aşqarlanmış $Bi_{0,85}Sb_{0,15}$ bərk məhlulu ekstruziya nümunələrinin elektrik xassələrinə qamma şüalarının təsiri ~80÷300*K* temperatur intervalında tədqiq olunmuşdur. Müəyyən olunmuşdur ki, qamma şüalarının təsirindən donor mərkəzləri rolu oynayan radiasiya defektləri yarandığından sərbəst elektronların konsentrasiyası n, uyğun olaraq, elektirikkeçiriciliyi σ artır, termo –e.h.q. əmsalı α isə azalır. Yükdaşıyıcılar bu defektlərdən səpildiyindən onların yürüklüyü μ azalır. Şüalanma dozasının artması ilə defektlərin konsentrasiyası artdığından sərbəst yükdaşıyıcıların rasiyasiya defektlərində tutulması baş verir. Modifikasiya olunmuş $Bi_{0,85}Sb_{0,15}$<Te> nümunələrində radiasiya şüalarının təsirindən akseptor (mənfi yüklü) mərkəzlərinin yaranması, ion "mərkəzlərinin" qismən neytrallaşması, yükdaşıyıcıların aşqarlardan səpilməsinin səmərəliliyinin azalması və müvafiq olaraq yürüklüyün müəyyən qədər artmasına baş verir.

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ВЛИЯНИЕ ГАММА РАДИАЦИИ НА ЭЛЕКТРИЧЕСКИЕ СВОЙСТВА ТВЕРДОГО РАСТВОРА $Bi_{0.85}Sb_{15}$ <Te>, МОДИФИЦИРОВАННЫХ ZrO_2

Исследованы влияния гамма-облучении на электрические свойства экструдированных образцов $Bi_{0,85}Sb_{0,15}$, модифицированных ZrO_2 содержащих донорные примеси теллура в интервале температур ~80÷300*K.* Выяснено, что при облучении возникают радиационные дефекты, играющие роль донорных центров, в результате чего концентрация свободных электронов n, и следовательно, электропроводность σ растет, а коэффициент термоэдс α падает. Эти дефекты, рассеивая носители тока, уменьшают их подвижность μ. *С* ростом дозы облучения растет и концентрация дефектов, происходит захват свободных носителей на уровень радиационного дефекта. В модифицироанны $Bi_{0,85}Sb_{0,15}$<Te> с радиационным введением акцепторных (отрицательно заряженных) центров, происходит частичная нейтрализация ионных остов, снижение эффективности примесного рассеяния носителей заряда и соответственно некоторый рост подвижности.