Influence of gamma radiation on magnetoelectric properties of solid solution Bi₈₅Sb₁₅ modified ZrO₂

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Abstract

The electrical and thermal properties of extruded samples of $Bi_{85}Sb_{15}$ modified with ZrO_2 were investigated depending on the dose of gamma radiation in the temperature range $\sim 77 \div 300$ K and the magnetic field strength (H) $\sim 74 \times 10^4$ A / m. It was found that the increase in the mobility in modified $Bi_{85}Sb_{15}$ is associated with the radiation introduction of acceptor (negatively charged) centers, which at low doses are generated mainly in the regions of positive ionic cores, which, as a result, partial neutralization of the ionic cores occurs, 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. A change in the subsystem of defects in extruded modified samples of the Bi85Sb15 solid solution under the influence of gamma radiation causes a change in the spectrum of localized states and the process of electron scattering, which leads to corresponding changes in the presented electrical and thermal parameters.

Keywords: extrusion, modification, mobility, gamma irradiation, annealing

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1. Introduction

Solid solutions of Bi-Sb systems, especially high-strength extruded materials based on these systems, are the most effective materials for creating various low-temperature energy converters [1-7]. High performance characteristics and great functionality determine the widespread use of Bi-Sb-based devices in various fields of technology under conditions of exposure to ionizing radiation. Ensuring the performance of various energy converters under conditions of exposure to ionizing radiation of natural and artificial origin is becoming an increasingly urgent problem due to the expansion of the field of application of these devices in industrial, special and space objects of systems [8-12]. Thermoelectric figure of merit of thermoelectrics is determined by $Z=\alpha^2 \sigma \chi$, where σ is the specific electrical conductivity, α is the thermoelectric coefficient, χ is the thermal conductivity. The number of ways to maximize the figure of merit of thermoelectric materials is not so great; Most thermoelectrics have already been implemented; therefore, in recent years, optimization will be reduced to varying the current concentration in the material used [13,14]. The way to increase the efficiency of thermoelectric materials is reduced to the creation of structural defects, which leads to a change in the parameters of the energy spectrum of charge carriers near the Fermi energy, which can affect the scattering of charge carriers and phonons to varying degrees.

In recent years, assumptions have been made about the possibility of increasing the thermoelectric figure of merit in materials with two-dimensional and three-dimensional defects of 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. These assumptions are based on the possibility of creating conditions under which a section of a material with different physical properties is characterized by a stronger scattering of thermal vibrations than electrons and holes. The effect can be achieved, for example, by introducing a finely dispersed second phase into the matrix of the substance (similar to the introduction of the second phase into the material during its disperse hardening) [15].

The most realistic and promising is the development of a technology for increasing the efficiency of thermoelectric material through modification. The method consists in introducing a scattering phase (modifier) into a semiconductor substance (PPV) with a coefficient of thermal expansion (CTE) different from the CTE of PPV. As a result of such a difference after cooling from the pressing temperature, elastically stressed zones are formed in the PPV. The creation of such stressed zones in the lattice of the thermoelectric matrix leads to the fact that the thermal conductivity of the matrix decreases more than its electrical resistance increases [16]. Therefore, to obtain a thermoelectric material with the required parameters, it is necessary not only to establish the optimal composition of the material, but also the optimal concentration of charge carriers and the scattering conditions of charge carriers, leading to a sufficiently high ratio of the mobility to the phonon part of the thermal conductivity μ/χ_1 , and develop technologies for obtaining and heat treatment for a given composition [17].

Devices based on Bi-Sb are often used in radiation conditions. The formation of radiation defects affects the physical properties in semiconductors and changes the parameters of a device based on it. Therefore, the study of the effect of radiation defects on the physical properties of Bi-Sb systems acquires a certain scientific and practical interest.

In order to clarify the features of the effect of modification and radiation defects (RD) on the magnetothermoelectric properties of solid solutions of Bi-Sb systems, we obtained extruded samples of $Bi_{85}Sb_{15}$ modified with ZrO_2 and investigated their magnetothermoelectric properties depending on the dose of gamma radiation in the temperature range ~77 \div 300 K and magnetic field strength ~ 74x10⁴ A/m. We investigated unirradiated samples and samples irradiated with 1Mrad, 10Mrad and 50 Mrad with gamma quanta.

2. Experiments

Extruded samples of solid solutions $Bi_{85}Sb_{15} < ZrO_2 >$ were obtained in the following technological sequence: synthesis of the composition from the initial components; mechanical grinding of the alloy in a porcelain mortar and selection of a fraction with a particle size of \leq 0.5 mm; mechanical mixing of powders of the alloy and modifier ZrO_2 1 wt.% (ZrO_2 was obtained by the plasma-chemical method, the average particle diameter is ~ 50 nm, the melting point is ~ 2950 K); production from it by cold pressing at room temperature and a pressure of ~3.5 t/cm² of briquettes with a diameter of ~30 mm for the next stage of the extrusion process; extrusion of fine billets. The introduction of modifier particles into the matrix is carried out in the process of annealing the work piece at temperatures lying between

the temperatures of the liquidus and solidus PPV (submelting). The modifier is evenly distributed with the liquid part of the volume, the proportion of which can be varied by the annealing temperature in accordance with the phase diagram. It has been experimentally established that the best, from this point of view, is annealing at temperatures when the liquid phase is 70-80% of the volume. It was found that the process of submelting itself has a positive effect on the properties of a thermoelectric material.

Bismuth "VI-0000" and antimony "SU-0000" were used as initial components.

The synthesis was carried out by direct fusion of the components. The starting materials in a stoichiometric ratio were placed in a quartz ampoule, preliminarily etched in a chromium peak solution ($K_2Cr_2O_7$) and washed with distilled water. The synthesis was carried out in quartz ampoules evacuated to ~10⁻² Pa at ~ 673 K for 2 hours. During the synthesized substance was sharply cooled to room temperature by immersing it in water. In the process of extrusion, the technological parameters of extrusion (temperature, drawing speed, etc.) were chosen such that the formation of extruded bars took place under superplastic conditions without macro- and micro-damage. The flexural strength of the obtained extruded samples is ~3 times higher than the strength of single-crystal samples of this composition.

Extrusion was carried out on an MS-1000 hydraulic press from a diameter of ~30 mm to a diameter of ~6 mm using special equipment. Technological parameters of extrusion were: $T_{exs.} = 475 \pm 3K$; $P_{exs.} = 480$ MPa, speed of movement of the press $v_{pr} = 0.02$ cm / min, degree of drawing -25.

Using D8 ADVANCE X-ray unit, Bruker, Germany, the texture of extruded samples was investigated by the method described in [18]. X-ray diffraction patterns were recorded at ~ 300 K using a D2 Phaser diffractometer, Brucker, using CuK radiation described in [6]. Using the TOPAS-4.2 program, it was confirmed from the obtained diffractograms that the samples are powders of the Bi₈₅Sb₁₅ solid solution, which crystallizes in the hexagonal system.

The interplanar distances of bismuth, antimony and their compounds along the main lines are close to each other; therefore, the phase composition was determined using a standard. Bi-0000 bismuth was used as a standard.

The Debyegrams and Lauegrams of the $Bi_{85}Sb_{15}$ samples under study exhibit strongly broadened symmetric diffraction peaks for all crystallographic directions, which indicates a large value of microstresses.

The data obtained using X-ray phase analysis are confirmed by electron microscopic studies carried out on a scanning electron microscope. Electron microscopic studies were carried out on thin sections prepared by the usual method using diamond pastes.

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 various doses (1; 10 and 50 Mrad).

The electrical conductivity (σ), thermo-emf (α), Hall (R_h) and thermal conductivity (χ) coefficients of samples were investigated as samples that were annealed after extrusion at ~ 503 K for 2 hours not irradiated with gamma quanta, and the same samples irradiated with gamma - quanta at various doses (1Mrad, 10Mrad and 50 Mrad) in the range of ~77-300 K and magnetic field strength (H) ~ 74x10⁴ A / m.

Samples for measurement were cut from extruded rods by electric spark cutting. In electric spark cutting due to the melting of a semiconductor material and quenching of the liquid phase, the formation of a pulsed field of thermal stresses, etc. a damaged polycrystalline layer is formed on the surface of the samples, heavily contaminated with the products of the electrode and dielectric medium. The thermoelectric parameters of such a layer will differ greatly from the parameters of the initial material. Therefore, after cutting, the surfaces of the samples were treated by electrochemical etching in a KOH + $C_4H_6O_6$ + H_2O solution at ~ 300K. The time of electrochemical etching was 20-25 s, the current density passing through the sample was 0.5 A / cm² [19].

The investigated samples had the shape of a parallelepiped with dimensions (0.2x0.4x1.5) cm³. The contacts were applied to the samples with Wood's alloy (wt%: 25% Bi + 50% Pb + 12.5% Sn + 12.5% Cd) with a melting point of ~ 343 K, using FSKGL flux (CH₅ON₃ + HCl + C₃H₈O₃). The contacts were point-like and had dimensions of 0.5 mm.

The magneto-thermoelectric parameters of the samples were measured by the method described in [20] along the length of the sample (rod), ie. in the direction of extrusion. The error in measuring the electrical parameters was $\sim 3\%$.

3. Results and discussion

The measurement results are shown in Figures 1, 2 and in the table. Irradiation of modified samples at various doses has practically no effect on the behavior of the dependence of electrical conductivity (σ) and thermoelectric power (α) and thermal conductivity (χ) on temperature. Some increase in the overall thermal conductivity of the heterogeneous system due to the thermal conductivity of the modifier particles.



Figure 1. Temperature dependences of electrical conductivity $\sigma(a)$, thermoelectric power coefficients α (b), Hall (R_h) and thermal conductivity χ (d) of extruded samples of Bi₈₅Sb₁₅ solid solution modified with ZrO₂. 1-undoped unirradiated sample; 2- modified unirradiated; 3-5 samples irradiated with gamma quanta of 1Mrad, 10Mrad and 50 Mrad, respectively.

Thermoelectric figure of merit (Z) of a semiconductor material depends on the concentration of charge carriers, which is determined by the degree of deviation of the composition from stoichiometric, the alloying level and the structural state of energetically active intrinsic superstoichiometric and impurity atoms. At a certain concentration of carriers, the thermoelectric efficiency of the material is proportional to μ/χ_1 , where μ is the mobility of

charge carriers, χ_l is the lattice component of thermal conductivity. At ~ 77K, the samples modified and irradiated with 1Mrad gamma quanta have a higher power factor ($\alpha^2 \sigma$) than the samples not irradiated with gamma quanta. Irradiation, like 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. Irradiation simultaneously increases the thermopower (α) and electrical conductivity (σ). This indicates an active influence of dispersed modifier particles opaque for dislocation on the processes of structure formation and mass transfer during extrusion and post-deformation annealing, leading to a redistribution of components between nonequilibrium excess phases and conglomerates of point defects and the main solid solution of nonequilibrium crystallized alloys.

The optimal direction for the growth of single crystals of Bi-Sb systems is the crystallographic direction [110] of the rhombohedral cell. However, the greatest value of the parameter of thermoelectric figure of merit, as well as electrical conductivity, is observed in another crystallographic direction [111], which is perpendicular to the optimal direction of growth of a single crystal. In these single crystals, the most perfect plane is the (111) plane, along which splitting always occurs [21].

During extrusion due to plastic deformation, part of the polycrystal grains is oriented so that their trigonal axis becomes parallel to the extrusion axis, i.e. a texture is formed.

Simultaneously, as a result of plastic deformation, various defects of the crystal lattice appear in individual grains. In this case, these structural defects are mainly concentrated between the cleavage planes (111). 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, grain disorientation can also occur due to thermal energy, i.e. change in the degree of texture of the extruded sample.

The introduction of the ZrO_2 modifier into the 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 and the lattice part of thermal conductivity, which has a positive effect on the thermoelectric properties of these samples. Modification can also affect the electronic component of thermal conductivity. At ~77K, the total thermal conductivity of modified and irradiated samples of 10 and 50 Mrad is greater than that of modified unirradiated samples and irradiated 1 Mrad. Irradiation does not affect the character of the temperature dependences of the studied samples. The thermal conductivity in the irradiated samples (except for the sample irradiated with 1 Mrad gamma quanta) is slightly higher than in the unirradiated samples.

Modification in all cases increases the proportion of the texture. By creating a microscopically uniform distribution of stoppers for moving dislocations, the modification increases the degree of uniformity of the strain distribution.

Analysis of the effect of modifying particles on deformation textures allows us to say that the determining effect of large particles is blocking dislocation slips in the basal planes, small spherical particles activate pyramidal slip and, to a certain extent, block basic slip. Pyramidal particles activate multiple sliding and, to a lesser extent than others, interfere with the basic one. 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 influenced by stresses arising in the matrix in the presence of modifier particles due to the difference in their coefficients of thermal expansion and deceleration of the motion of boundaries by foreign particles.

Irradiation at low doses insignificantly increases the electrical conductivity σ , and the Seebeck coefficient α increases significantly. With an increase in the irradiation dose, the electrical conductivity of the modified irradiated samples decreases. The scattering of charge

Radiation doses	Compositions	At 77 K						At 300 K					
		$\sigma, \Omega^{-1} c m^{-1}$	α, μV/Κ	χ, W/m·K	$R_{\rm h} \cdot 10^{-8}, {\rm cm}^3/C$	μ, cm²/ V·c	n, cm ³	α, Ω ⁻¹ cm ⁻¹	α, μV/Κ	χ, W/m·K	$ m R_h\cdot 10^{-8},cm^{3}/C$	μ, cm²/ V.c	n, cm ³
0 Mrad	Bi ₈₅ Sb ₁₅	5250	-182	3,02	-26,5	139125	0,24.1018	7520	-95	5,96	-1,43	10754	4,4·10 ¹⁸
	$\frac{\text{Bi}_{85}\text{Sb}_{15}\text{+}}{1\%\text{ZnO}_2}$	4899	-134	2,93	-14,33	70203	0,44.1018	5667	-93	5,29	-1,34	7594	4,7·10 ¹⁸
1 Mrad	Bi ₈₅ Sb ₁₅	8481	-121	3,07	-1,26	10686	4,96·10 ¹⁸	6524	-89	5,08	-1,26	8220	5.1018
	$\frac{\text{Bi}_{85}\text{Sb}_{15}+}{1\%\text{ZnO}_2}$	5604	-169	2,50	29,2	163637	0,21.1018	6610	-60	7,56	-21	13173	0,3.1018
10 Mrad	$\mathrm{Bi}_{85}\mathrm{Sb}_{15}$	4240	-161	3,06	-60	254400	0,1.1018	6890	-101	6,32	-17,4	119886	0,36.1018
	$\begin{array}{l} Bi_{85}Sb_{15}+\\ 1\%ZnO_2 \end{array}$	4563	-159	4,37	-37	168831	0,16·10 ¹⁸	5503	-102	5,99	-31,5	173345	0,2.1018
50 Mrad	$\mathrm{Bi}_{85}\mathrm{Sb}_{15}$	4552	-188	4,00	-85	386920	0,07.1018	6448	-90	4,76	-34,3	221166	0,18.1018
	$\begin{array}{l} Bi_{85}Sb_{15}+\\ 1\%ZnO_2 \end{array}$	4703	-161	4,34	-30	141090	0,2.1018	5789	-47	5,41	-21	141876	0,3.1018

carriers by dislocations and dislocation walls is greater along the basal planes than along the main axis of symmetry in deformed extruded materials.

The mobility in all irradiated samples with the modifier exceeds the mobility of the modified unirradiated samples. In the temperature dependence of the Hall coefficient of the modified extruded Bi₈₅Sb₁₅ samples, at ~100 K, maxima are observed, which is assumed to be associated with the mobility of charge carriers. The modifier increases the uniformity of the dislocation density distribution [22], forms a structure with a lower concentration of nonequilibrium point defects, and leads to greater chemical compositional homogeneity.

The calculated Hall mobility μ from the experimental values of electrical conductivity and the Hall coefficient $\mu = \sigma R_h$ of all irradiated samples is much higher than in unirradiated modified samples (table). Simultaneously, with an increase in the radiation dose, the mobility of charge carriers decreases, except for the sample irradiated with 10 Mrad gamma quanta. In the extruded Bi₈₅Sb₁₅ samples, the mobility of charge carriers is largely determined by scattering at structural defects of deformation origin. Modification, creating a large number of stoppers for moving dislocations, promotes the formation of dislocation structures with a smaller scale of 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).

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

Based on the data obtained, it is assumed that at low radiation doses (1 Mrad) in the samples, the electrical conductivity σ increases, and the thermo-emf 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 caused charged defects n and, therefore, σ of the sample decrease, the Fermi level shifts to the depth of the band gap, and the thermoelectric coefficient and mobility increase.

Irradiation with different doses of gamma radiation of modified extruded Bi₈₅Sb₁₅ samples and does not change the temperature dependence of electrical conductivity in the studied temperature range is described by the acoustic scattering mechanism. The electrical conductivity decreases with an increase in the radiation dose, which is associated with a change in the drift mobility of charge carriers. The temperature dependence of the Seebeck coefficient indicates that the materials under study are not strongly degenerate.

Irradiated samples $Bi_{85}Sb_{15}$ (semiconductor) is 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 that is "inverse" 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 $Bi_{85}S_{b15}$ solid solution show 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 compensation degree 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 shifts 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 forbidden band, i.e. are determined by the position of the Fermi level relative to the nearest extrema of the conduction band or valence band.

In the modified samples of the $Bi_{85}Sb_{15}$ solid solution, as in crystals, silicon has the socalled low-dose effect, i.e. an anomalous change (increase) in the mobility of current carriers upon irradiation with gamma quanta in the mixed scattering region [23]. An increase in the mobility in modified $Bi_{85}Sb_{15}$ is associated with the radiation introduction of acceptor (negatively charged) centers, which at low doses are generated mainly in the regions of positive ionic cores, which results in partial neutralization of the ionic cores, which leads to a decrease in the efficiency of impurity scattering of charge carriers and partially neutralized centers and, accordingly, to some increase in mobility.

It can be seen from Figure 2 that at ~ 77 K the magnetic field does not change the course of the σ (H) and α (H) dependences in modified unirradiated and irradiated samples of Bi₈₅Sb₁₅ solid solution with various doses of gamma quanta. However, the magnetoresistance in unirradiated unmodified and modified samples at weak magnetic fields (up to ~ 16x10⁴ A / m) is much higher than in irradiated samples. With an increase in the magnetic field strength, the effect of the magnetic field on σ is almost the same for all samples. In unirradiated and irradiated samples with different doses of gamma quanta, scattering by defects prevails in the scattering of electrons. When a sample is exposed to a magnetic field perpendicular to the direction of motion of electrons and holes, charge carriers are deflected under the action of the Lorentz force. In this case, carriers that scatter weaker and therefore have a longer free path in a magnetic field are deflected more than highly scattering carriers. In a magnetic field in the samples under study, a redistribution of the contributions of various charge carriers to the total current occurs. With an increase in the contribution to the total current of strongly scattering charge carriers, the contribution of weakly scattering carriers decreases.



Figure 2. Dependences of electrical conductivity σ (a), thermoelectric coefficients α (b), Hall (R_h) and thermal conductivity χ (d) on the magnetic field strength (H) of extruded samples of Bi₈₅Sb₁₅ solid solution modified with ZrO₂ at ~ 77K. The designations are the same as in Figure 1.

An insignificant change in α of all the studied irradiated samples of the Bi₈₅Sb₁₅ solid solution shows that irradiation mainly changes the mobility of charge carriers. When the sample is placed in a magnetic field, the contribution to the total current of fast carriers increases, i.e., the average energy of charge carriers in the studied B_{i85}Sb₁₅ and α increases. The concentration of structural defects in unirradiated samples is low; therefore, at ~ 77 K, scattering by acoustic phonons prevails, and the Seebeck coefficient increases strongly in a magnetic field.

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 $Bi_{85}Sb_{15}$ samples, an increase in the electron mobility and an increase in the prevalence of carrier scattering on lattice vibrations. These assumptions are confirmed by the dependence of σ and α on the magnetic field strength.

The transverse magnetoresistance in weak fields is proportional to the square of the magnetic induction B and the square of the carrier mobility μ [24].

$$\Delta \rho / \rho_0 = A \mu^2 B^2$$

where the coefficient A depends on the scattering mechanism of current carriers. The experimental results on the dependence of $\Delta\rho/\rho_0$ on B² are in good agreement with the value of A (A = 1.18) for electron scattering in modified Bi₈₅Sb₁₅. This is evidenced by the regularities and regularities of the dependence of the Hall coefficient on the magnetic field strength in the studied Bi₈₅Sb₁₅ samples. Similar dependences are obtained at high (up to ~

300 K) temperatures. However, due to the decrease in the carrier mobility with increasing temperature, these dependences are somewhat weakened.

The χ values are in good agreement with the above considerations obtained in measurements in a magnetic field.

The results of the data obtained indicate that irradiation with gamma quanta occurs not only the generation of radiation defects (centers), but also accompanied by their rearrangement. The restructuring essentially depends on the initial level of modification of the ingot from which the corresponding samples for research are made.

The radiation-stimulated increase in the 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.

4. Conclusion

Based on the data obtained, it is assumed that, at low doses (1 Mrad) of irradiation in undoped and unmodified samples of the Bi₈₅Sb₁₅ solid solution, radiation defects appear, 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 falls. 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. An increase in the mobility in modified Bi₈₅Sb₁₅ is associated with the radiation introduction of acceptor (negatively charged) centers, which at low doses are generated mainly in the regions of positive ionic cores, which results in partial neutralization of the ionic cores, which leads to a decrease in the efficiency of impurity scattering of charge carriers and partially neutralized centers and, accordingly, to some increase in mobility. The results of the data obtained indicate that irradiation with gamma quanta occurs not only the generation of radiation defects (centers), but also accompanied by their rearrangement. A change in the subsystem of defects in extruded modified samples of the Bi₈₅Sb₁₅ solid solution under the influence of gamma radiation causes a change in the spectrum of localized states and the process of electron scattering, which leads to corresponding changes in the presented electrical and thermal parameters.

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