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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

THE EFFECT OF IONIZING RADIATION ON THE DIELECTRIC AND ELECTRICAL PROPERTIES OF $(TlInS_2)_{1-x}C_x$ SOLID SOLUTIONS

Speciality:	2225.01 – Radiation materials science
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The work was performed at the "Radiation Physics of Disordered Solids" laboratory of the Institute of Radiation Problems of the Ministry of Science and Education of the Republic of Azerbaijan.

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GENERAL DESCRIPTION OF THE WORK

Actuality of the work and work done so far. In recent years, the effects of ionizing radiation on defective semiconductors have spurred significant research advancements across various fields.

The availability of modern measuring devices, which can simultaneously measure multiple parameters of compounds, has made the study of compounds with diverse properties, as well as crystals and solid solutions, particularly relevant.

Low-dimensional chalcogenides with layered and chain-like structures, belonging to the class of $A^3B^3C^6_2$ type compounds, are of particular interest within this context.

These compounds have recently revealed numerous fascinating characteristics related to their electrical, photoelectric, and optical properties, as well as their promising prospects for practical applications.

$A^3B^3C^6_2$ type compounds exhibit ferroelectric properties, including high dielectric permittivity, a substantial piezoelectric modulus, the presence of a dielectric hysteresis loop, and unique electro-optical properties. As a result, these compounds are widely utilized in various modern technologies, including radio engineering, electroacoustics, quantum electronics, and measurement technology. Their application in semiconductors holds significant potential.

One of the intriguing properties of $A^3B^3C^6_2$ type compounds is their pronounced anisotropy. As indicated by a review of the existing literature, interest in materials with anisotropic properties has grown considerably in recent years. In these crystals, the atomic arrangement is such that the movement of electrons is restricted not only in one direction but also in two, resulting in unique physical properties. Additionally, these compounds exhibit a variety of bonding types, including both covalent and molecular (Van der Waals) bonds, which occur on and between surfaces.

This class of crystals simultaneously demonstrates semiconductor and ferroelectric properties, displays photoconductivity, and shows nonlinear behavior in its volt-ampere characteristics.

Currently, there is growing interest in layered-chain crystals belonging to the class of ternary $A^3B^3C^6{}_2$ type compounds, particularly due to the observation of ionic conductivity in these materials at temperatures above room temperature. Layered and chain-like $A^3B^3C^6{}_2$ crystals, along with their conductivity over a wide temperature range, have been studied in the Radiation Physics of Ferroelectrics and Radiation Physics of Disordered Solids laboratories at the Institute of Radiation Problems. These studies have demonstrated that these compounds exhibit both electronic and ionic conductivity. The search for previously identified properties to expand the class of semiconductors of this type has recently attracted the interest of researchers. Some of these compounds and their corresponding solid solutions possess both ferroelectric and semiconducting properties. Studies on the temperature dependence of the dielectric permittivity of $TlInS_2$ crystals, along with the results of differential thermal analysis, indicate that even slight deviations from stoichiometry significantly impact the dielectric properties of ferroelectric $TlInS_2$ crystals. The potential for ferroelectric-semiconductor materials to exhibit both ionic and electronic conductivity has the potential to drive further interest in the discovery and production of materials with novel properties. During the synthesis and growth of materials with novel properties, dopant atoms are introduced (in the form of implantation) to control their electrophysical characteristics, including the temperatures at which ionic phase transitions occur. Studies indicate that the role of defect and impurity subsystems in the mechanisms of structural phase transitions in semiconductor crystals, or other known ferroelectric-semiconductors, differs fundamentally from the "classical" approach. Doping these materials with dopant atoms helps to stabilize the relaxor state, there by defining the boundaries of structural phase transitions. It is well-known that various types of defects are formed in crystals exposed to substitution, the introduction of atoms with different ionic radius, implantation, and gamma radiation. This, in turn, has a strong effect on the dielectric and electrical properties of the material, their temperature dependences, including the ionic conductivity of crystals, and other properties. Consequently, the synthesis of compounds exhibiting these properties and the investigation of their electrophysical characteristics remain highly relevant.

The object and subject of the research. $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ solid solutions obtained by introducing a carbon atom into the semiconductor $TlInS_2$ compound are the object of research. The subject of the research is the study of the effect of ionizing radiation on the dielectric and electrical properties of these compounds.

The goals and objectives of the research is to study the effect of ionizing radiation (γ-rays and H^+ ions) on the structure, dielectric and electrical properties, complex impedance spectra, electron-ion conductivities, ion diffusion, and dielectric relaxation phenomena of $(TlInS_2)_{1-x}C_x$ solid solutions.

To achieve the stated goal, the following tasks are planned to be addressed:

- Synthesis and growth of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds;
- Irradiation of the studied compounds with γ-rays and H^+ (protons);
- Study of the dielectric properties of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds irradiated with γ-rays at doses of $0 \div 0.8$ MGy;
- Study of the electrical conductivities of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds irradiated with γ-rays at doses of $0 \div 0.8$ MGy;
- Study of complex impedance spectra of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds irradiated with γ- rays at doses of $0 \div 0.8$ MGy;
- Study of dielectric relaxation phenomena in $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds irradiated with γ-rays at doses of $0 \div 0.8$ MGy;
- Study of the dielectric and electrical properties and complex impedance spectra of $(TlInS_2)_{0.95}C_{0.05}$ compound before and after H^+ (proton) irradiation;

- Study of distribution of implanted H^+ ions at different depths in $(TlInS_2)_{0.95}C_{0.05}$ compound and the distribution of vacancies in target atoms at different depths;

Research methods. $(TlInS_2)_{1-x}C_x$ compounds were grown by the Bridgman-Stockburger method. Quartz ampoules with a diameter of 0.02 m were used to synthesize the compounds. The components of the synthesized samples were filled into quartz ampoules and placed in a furnace under vacuum conditions The electric furnace was heated to the required temperature at a rate of 0.16-0.2 K/sec, and then the furnace temperature was lowered to room temperature.

$(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds were irradiated in the γ-25 radiation research facility consisting of the Co^{60} isotope, a radiation source operating at the Institute of Radiation Problems. To protect against gamma rays, the sources are kept under water. The gamma ray sources consist of nine radioactive rods connected to the lower and upper handles, tubes and cassettes. Currently, the total dose of the facility is 3500 curies. The radioactive rods, made of the isotope Co^{60}, are arranged symmetrically around the circumference. The size of the irradiated sample is smaller than the size of the chamber, and the doses they receive vary depending on their location.

Ion implantation of the samples was conducted at the “Department of Ion Physics and Implantation” at the Institute of Physics, Maria Curie-Skłodowska University in Lublin, Poland. Implantation was performed using a UNIMAS 79 accelerator at room temperature, with an energy of 150keV, a dose rate of $D=10^{16}$ ions/cm^2sec, and at an angle of 10°.

X-ray studies of $(TlInS_2)_{1-x}C_x$ compounds were carried out using a “D2 Phaser” diffractometer (CuK_a-radiation, Ni-filter) from the German company “Bruker”.

Raman scattering spectra were obtained using a Nanofinder-30 three-dimensional confocal microscope (Tokyo Instr. Japan), with a YAG: Nd laser with a wavelength of $\lambda = 532$ nm as the excitation source.

By measuring the parameters (C tgδ) and (Z φ) of the samples with the help of E7-20 and E7-25 immittance devices, the real and imaginary parts of the dielectric permittivity (ε) as well as the impedance parameters were evaluated. The experiments were conducted in the temperature range of 300÷600K and the frequency range of 25Hz÷1MHz, with a temperature increment of 0.5K.

Defense of the main scientific points:

1. The structures of the compounds $(TlInS_2)_{0.95}C_{0.05}$, $(TlInS_2)_{0.9}C_{0.1}$ were investigated by X-ray diffraction and Combinational Light Scattering methods in comparison with the structure of the $TlInS_2$ crystal, and it was determined that the experimentally obtained oscillation frequencies of these compounds are the same;

2. The fact that the dielectric permittivity ε(T) of the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ above room temperature corresponds to a straight line is characteristic of ionic conductors, and the observation of anomalies in the temperature dependences of tgδ, and the investigation of the effect of γ-rays on these anomalies;

3. The anomalies observed in the temperature dependences of the dielectric permittivity of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds irradiated with γ-rays at doses of 0÷0.8 MGy and the closeness of the Cole-Cole diagrams ($(\varepsilon''(\varepsilon'))$) to a straight line are explained by the presence of a large number of relaxors in them;

4. Determination of the ionic and electronic portion to the total electrical conductivity of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds at doses of 0÷0.8 MGy in a wide temperature range, in a constant electric field, from the σ/σ_0 (t) dependences;

5. To propose equivalent schemes by studying the complex impedance spectra of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds irradiated with γ– rays at doses of 0÷0.8 MGy;

6 Observation of dispersions in the dielectric parameters and complex impedance spectra of the $(TlInS_2)_{0.95}C_{0.05}$ compound over a wide temperature and frequency range of H^+ (proton) irradiation and their comparative investigation to determine the frequency range;

7. Determination and evaluation of the ion and electron portions, diffusion coefficient and relaxation time involved in the complete conductivity of the $(TlInS_2)_{0.95}C_{0.05}$ compound before and after

irradiation with H^+ protons in a constant electric field by determining and evaluating them from the σ/σ0 (t) dependences;

8. In the $(TlInS_2)_{0.95}C_{0.05}$ compound implanted with H^+ ions with an energy of 150 keV, distribution of ions in depth and the distribution of vacancies created by the implanted ions were calculated and the CLS spectra were recorded.

The scientific novelty of the research is as follows:

1) According to X-ray structure and combinational light scattering analyses, the structures of the compounds $TlInS_2$, $(TlInS_2)_{0.95}C_{0.05}$, and $TlInS_2)_{0.9}C_{0.1}$ are similar, which is explained by the placement of graphite (C) atoms in the octahedral voids in the In_4S_{10} complex;

2) The ionic phase transition observed in the temperature dependences of the dielectric permittivity of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds above room temperature and the anomalies in the temperature dependences of tgδ are attributed to Debye-type relaxation processes, the effect of γ– radiation extends the temperature range of transition to the ionic phase by increasing it;

3) The closeness of the Cole-Cole diagrams ($\varepsilon''(\varepsilon')$) in the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ irradiated with γ-rays at doses of 0÷0.8 MGy to a straight line is explained by the presence of a large number of relaxators in them, and the polarization is associated with the displacement of ions over long or short distances as described by the Concher model;

4) The ionic and electronic portion to the total electrical conductivity of the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ irradiated with γ-rays at doses of 0÷0.8 MGy in a constant electric field were determined by the Wagner polarization method and the increase in ionic conductivity is due to the polarization of the volume charges formed in the region near the electrode;

5) Based on the equivalent circuit of the $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds irradiated with γ–rays at radiation doses of 0÷0.8MGy in an alternating electric field, form a semicircle in the diagram, which corresponds to the charge transport process (high-frequency part of the hodograph). This corresponds to a straight line

inclined toward (the low-frequency part of the hodograph), which is attributed to the polarization of volume charges in the region near the electrode.

6) The increase in the polarization of volume charges in a constant electric field leads to a decrease in the electron portion, a reduction in relaxation time and an increase in the ion portion and diffusion coefficient values.

7) The increase in dispersions in the dielectric parameters and complex impedance spectra of the $(TlInS_2)_{0.95}C_{0.05}$ compound before and after irradiation with H^+ ions is due to the formation of interstitial-type defects in this compound as a result of the action of low-energy protons, since the compound has a layered structure.

8) The distribution of vacancies created by implanted ions in the $(TlInS_2)_{0.95}C_{0.05}$ compound before and after H^+ (proton) irradiation was calculated and is related to the homogenity (amorphization) formed in the near-surface region;

Theoretical and practical significance of research:

The results obtained in the dissertation can be widely applied in the development of systems for collecting electrical and optical information, electronic converters, memory elements, and nanoelectronics. In this work, the introduction of atoms with small ionic radius into solids, implantation with H^+ ions and gamma irradiation lead to the control of physical properties, which in turn significantly increases the possibilities of using these compounds in various fields.

Approval of research results:

The results of the dissertation were reported at the following conferences:

-IX International conference Semipalatinsk test site: Legacy and Prospects for Scientific and Technical Potential development (07-09 September 2021. Kurchatov, Republic of Kazakhstan.).

- IX International scientific conference “Actual Problems of Solid State Physics” (22-26 November, 2021, Minsk, Belarus.).

-Radiation Technologies and its application “Scientific and Technical Conference dedicated to the 100th anniversary of the birth of the great leader Heydar Aliyev”. (May 5, 2023, Baku, Azerbaijan.).

-X International Scientific Conference “Actual Problems of Solid State Physics” (May 22-26, 2023, Minsk, Belarus.).

- Radiation Safety Problems: Regional Aspects Scientific and Technical Conference. (October 18-19, 2023, Nakhchivan.)

and also delivered speeches at seminars of the Institute of Radiation Problems of the Ministry of Science and Education of the Republic of Azerbaijan.

The organization where the dissertation work: The presented dissertation work was carried out in the laboratory “Radiation Physics of Disordered Solids” at the Institute of Radiation Problems of the Ministry of Science and Education of the Republic of Azerbaijan.

Applicant's personal contribution to the research conducted. In the dissertation, the applicant justified the relevance of the research based on a review of literature sources, selected appropriate research methods, and applied them to solve scientific problems. The applicant also carried out the planned experiments at all stages of the research and systematized the obtained results. Additionally, the applicant analyzed the experimental findings, participated in discussions of the research at scientific conferences, and prepared scientific articles based on the obtained results.

Published works. 12 scientific works, including 7 articles (2 of the articles were published in Web of Science and Scopus), as well as 5 conference materials were published in republican and international scientific journals related to the topic of the dissertation.

Structure and volume of the dissertation. The dissertation work consists of an introduction, four chapters, results and a list of references. The work includes 44 graphs, 10 pictures, 5 tables and a total of 182400 symbols.

The main content of the work.

The introduction justifies the relevance of the topic of the presented dissertation, outlines the aim, scientific innovations, and practical significance of the work, and presents the main provisions put forward for defense. Additionally, the introduction briefly explains the confirmation of the work's results and summarizes the main content of the chapters. **In Chapter I** of the dissertation, the electrophysical properties of thallium-based chalcogenides, which belong to to the $A^3B^3C^6{}_2$ type compound class, are examined. The chapter also reviews literature data on the effect of proton implantation and gamma rays on these properties. Furthermore, the theoretical foundations of ionic conductivity are provided, based on the analysis of the literature data.

Chapter II of the dissertation describes the synthesis and growth process of the studied materials. Additionally, this chapter outlines the schematic of the devices used for studying the electrical and dielectric properties (the device was constructed in the Laboratory "Radiation Physics of Disordered Solids" at the Institute of Radiation Problems), their operating principles and the methods of irradiation with ionizing radiation.

Chapter III of the dissertation based on X-ray analysis, it was determined that the diffractograms of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ are similar with only a slight difference in the intensities of the reflections, the structures of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds replicate the monoclinic structure of the $TlInS_2$ crystal with the parameters a=10.96 Å; b=10.97 Å; c =15.14 Å; β=100.0°; Z=64, belonging to phase group C2/c. In this work, the Raman spectra of the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ were also recorded and analyzed with comparisons made to the spectra known in the literature for the $TlInS_2$ crystal.

The results from X-ray structural analysis and combinational light scattering experiments show that the structures of the compounds $TlİnS_2$, $(TlInS_2)_{0.95}C_{0.05}$, and $(TlInS_2)_{0.9}C_{0.1}$ do not differ from each other. In other words, this indicates that the graphite (C) atoms are located in the octahedral voids in the In_4S_{10} complex, forming corresponding inclusion solid solutions.

It should be noted that, the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ were synthesized and growth for the first time in this work, and their physical properties are being studied for the first time.

This chapter presents the results of the study of the electrical and dielectric properties of the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ in the temperature range of 300-600K. The temperature dependences of the dielectric permittivity ε'(T) of the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ were studied and are depicted in Fig. 1(a,b). In the graphs, low- and high-intensity peaks were observed in the temperature dependences of the dielectric permittivity ε'(T).

Anomalies were observed in the $(TlInS_2)_{0.95}C_{0.05}$ compound at temperatures T = 370K, T = 415K and T = 532K, and in $(TlInS_2)_{0.9}C_{0.1}$ at temperatures T = 369K, T = 503K and T = 555 K in the ε(T) dependences. The observation of anomalies in these compounds is associated with the presence of a large number of relaxators in them. At low temperatures, there are few freely moving ions, and they move very slowly, resulting in very low ionic conductivity As the temperature increases,the number of interstitial ions also increases. Therefore, the ionic conductivity of the crystal should gradually rise when heated.

It should be noted that the characteristic shape of the ε'(T) graphs corresponds to ionic conductivity, as in the case of ionic conductivity, the value of ε′ increases sharply at temperatures close to the phase transition. In Fig 1. (a,b), the points in the dependences of the dielectric permittivity ln(ε) on 1000/T, at temperatures close to the peaks in the graph above the figure, fall on a straight line, which indicates that the compounds have transitioned into the ionic phase, and the dielectric permittivity follows the exponential law and is expressed by the following equation[1]:

$$\varepsilon = \varepsilon_0 \cdot \exp(-\Delta E_a / k\,T) \qquad (1)$$

where ΔE is the activation energy, k is the Bolsman constant

From the dependence of the dielectric permittivity (1), we can calculate the activation energies of ions above room temperature.

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[1] Лидьярд А. Ионная проводимость кристаллов. Изд-во ИЛ.М., – 1962, с.222.

The activation energies were determined to be ΔEa_1=0.6eV, ΔEa_2 = 0.38eV for $(TlInS_2)_{0.95}C_{0.05}$, and ΔEa_1 = 0.54eV, ΔEa_2 =0.32eV ΔEa_3 =0.22 eV for $(TlInS_2)_{0.9}C_{0.1}$.

It is known that one of the main conditions for the formation of ionic conductivity is the presence of highly polarized cations in the structure (Pb^{2+}, Bi^{3+}, Tl^{1+}, etc.), as well as the presence of large voids that facilitate the mobility of ions. Taking this into account, it can be concluded that the high values of dielectric permittivity at low frequencies in $TlInS_2$, $(TlInS_2)_{0.95}C_{0.05}$, and $(TlInS_2)_{0.9}C_{0.1}$ compounds are based on the mechanism of ionic polarization. This is due to the diffusion of Tl^+ ions through vacancies in the Tl^+ sublattice.

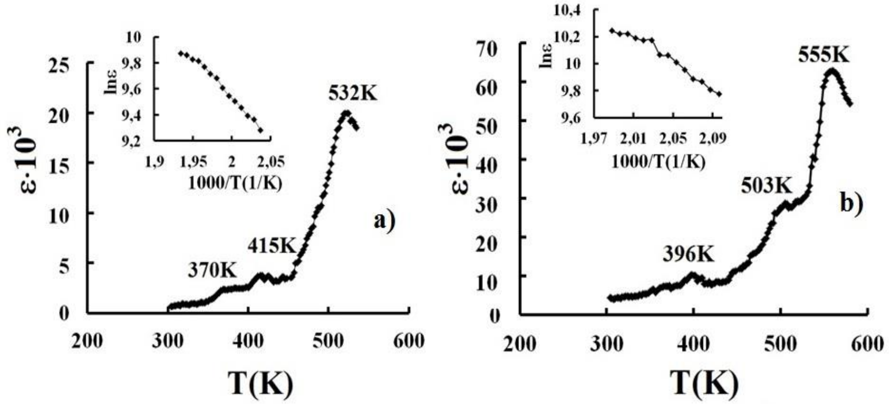

Figure 1. Temperature dependence of dielectric permittivity of compounds (a) $(TlInS_2)_{0.95}C_{0.05}$ and (b) $(TlInS_2)_{0.9}C_{0.1}$ (lnε (1000/T) in the graph above the figure)

From a crystallochemical perspective, it can be stated that the structure of the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ is the particularly favorable for the mobility of highly polarized Tl^+ cations. According to the lattice parameters, carbon atoms can occupy octahedral voids between InS_4 tetrahedra within the $[[In_4S_{10}]]$ tetrahedral complexes of the $TlInS_2$ monoclinic lattice (due to the small size of carbon ions (0.2Å)). As a result, the placement of an electropositive atom in the $(TlInS_2)_{1-x}C_x$ compounds weakens the bond with sulfur and thallium atoms, leading to the formation of a stable relaxator state

Chapter III of the dissertation also presents the results of the study of the frequency dependences of the dielectric permittivity and the tangent of the dielectric loss angle in the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$. The studies were conducted in the temperature range of 300-600 K. Dispersions were observed in the frequency dependences of both the dielectric permittivity and the tangent of the dielectric loss angle. As is known from the literature, such effects correspond to Debye-type relaxation processes[2]. Debye-type relaxation processes processes are characterized by the dispersion of the real and imaginary parts of the dielectric permittivity in an electric field of varying frequency. The characteristic dependence of the peaks of the dielectric quantities on the frequency of the measuring field indicates that the anomaly is of a relaxation nature.

In this case, the cause of the relaxation is the presence of weakly bound ions in the crystal lattice. In the dependences of tgδ(T), peaks were observed at low and high temperatures. As the measurement frequency increased, the peaks of tgδ(T) shifted to higher temperature regions, and the value of tgδ decreased. For the studied compounds, the frequency of charge transport through the barrier was calculated from the dependence of $\lg f_{max}$ on $1/T$. The slope of the straight line obtained from these dependences indicates the energy required for charge transport through the barrier, and this energy was found to be E_{a1}=0.24eV and E_{a2}=0.26eV. By extrapolating the straight line with $1/T \rightarrow 0$, the hopping frequency (relaxor frequency) was determined to be $\nu=8\cdot10^{12}$ Hz for $(TlInS_2)_{0.95}C_{0.05}$ and $\nu = 4\cdot10^{12}$ Hz for $(TlInS_2)_{0.9}C_{0.1}$. The value of the obtained frequency is too low for electronic processes. This frequency corresponds to the phonons frequency in the far infrared spectrum, that is, in turn, relates to the vibrations of the heavy Tl^+ ions.

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[2] Желудов, И.С. Физика кристаллических диэлектриков / И.С. Желудов.– Москва: Наука, – 1968. -463с.

In the third chapter, the Cole-Cole diagrams of these compounds at different temperatures were constructed and studied. It was shown that in the temperature regions where the anomalies ε(T) and tgδ(T) occur, the Cole-Cole diagrams are monotonic in nature, and the dependences ε″(ε′) in the complex plane do not resemble the classical Cole-Cole semicircle. The monotonicity of the dependence ε″(ε′) (i.e., its proximity to a straight line) is possible within the framework of the Jonsher model[3], which is universal for dielectrics based on an energetic approach. In the Jonsher model, polarization is associated with the hopping of ions over long or short distances. The discrete displacement of charges is accompanied by the screening of polarization due to lattice relaxation.

It was determined that the dielectric permittivity of $(TlInS_2)_{0.95}C_{0.05}$ in the "c" axis direction is 30 times greater than that of the $TlInS_2$ crystal (at the same temperature). At the same time, the dielectric permittivity of $(TlInS_2)_{0.9}C_{0.1}$ in the same direction and at the same temperature is 1.15 times higher than that of $(TlInS_2)_{0.95}C_{0.05}$.

In the third chapter, the electrical properties of solid solutions of the $(TlInS_2)_{1-x}C_x$ (x=0, 0.05, 0.1) system were also studied in the temperature range of 300÷600K, and the results are presented in Figure 2(a,b). According to Figure 2, a several-fold increase in the conductivity is observed above room temperature.

It is noted in the literature that the ionic conductivity is determined from the dependence of ln(σ·T) on (1/T) by the following formula. As is known, the temperature dependence of conductivity in compounds with ionic conductivity obeys the Arrhenius law[4].

$$\sigma\cdot T=\sigma_0\cdot\exp(-\Delta E_a/kT) \qquad (2)$$

Here, ΔE_a represents the activation energy, k is Bolsmann constant. Using the formula (2), the activation energies for solid solutions of the system $(TlInS_2)_{1-x}C_x$ (x= 0.05, 0.1) were calculated.

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[3] Jonscher, A.R. Analysis of thermally stimulated depolarisation in "non-debye" dielectrics // Journal of Electrostatics, -1977, 3, -p. 53-68.

[4] Лидьярд А. Ионная проводимость кристаллов. Изд-во ИЛ.М., – 1962, с.222.

For solid solutions containing $(TlInS_2)_{0.95}C_{0.05}$, it is equal to ΔE= 0.3eV, and for $(TlInS_2)_{0.9}C_{0.1}$ solid solution to ΔE= 0.26 eV. It was shown that the jumping increase in the numerical value of electrical conductivity in the temperature range of 450÷600K (Fig 2.(a,b)) is due to the presence of ions with high mobility in the studied compounds, their increase in number with increasing temperature, and the diffusion of these ions over vacancies.

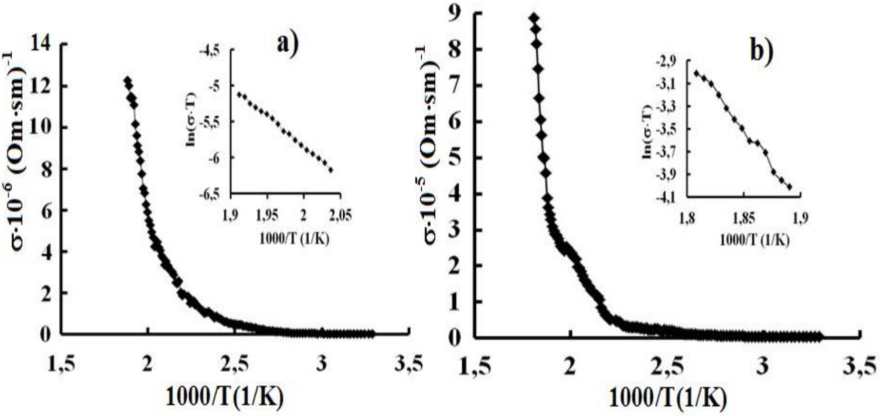

Figure 2. Temperature dependence of conductivity (a) $(TlInS_2)_{0.95}C_{0.05}$ and (b) $(TlInS_2)_{0.9}C_{0.1}$ compounds (the graph above the figure shows the dependences of ln(σ·T) on (1/T))

In the course of the dissertation work, the ionic and electronic components of conductivity at different constant temperatures were also evaluated and the results obtained were presented in this chapter. The electronic and ionic components of conductivity were determined by the Wagner polarization method[5]. According to the Wagner method, a constant electric field is applied to the sample. Under the influence of a constant electric field, mobile ions accumulate near the negatively charged electrode at the sample-electrode interface. When measuring electrical conductivity after removing the electric field, it is observed that the conductivity depends on time. In this case, a polarization process occurs in the

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[5] Wagner, C. Z. Electrochem.Berichte bunsenges // Phys. Chem., -1956, 60, - p. 4.

measuring cell containing the sample, which leads to the formation of a double electric layer at the sample-electrode interface.

Thus, a concentration gradient is created in the sample volume. The presence of concentration gradients of positively charged ions leads to the formation of a diffusion current in the direction opposite to the drift current of ions.

Fig 3.(a,b) shows the time dependences of the electrical conductivity of these compounds in a constant electric field at different constant temperatures. In Fig 3.(a,b), the electrical conductivity initially decreases exponentially and, after a certain time, stabilizes.

The nonlinear decrease in electrical conductivity occurs rapidly at relatively high temperatures. The time-dependent decrease in current in a constant electric field is attributed to the mutual compensation of volume charges near the electrodes. Calculations showed that the polarization of volume charges at the electrode boundary increases in the infra-low frequency region depending on the temperature, and the diffusion coefficient for the $(TlInS_2)_{0.95}C_{0.05}$ solid solution is $(4÷7)\cdot10^{-10}$ (m^2/sec) and for the $(TlInS_2)_{0.9}C_{0.1}$ compound, the diffusion coefficient varies in the interval $(4.5÷9.5)\cdot10^{-10}$ (m^2/sec) depending on the temperature.

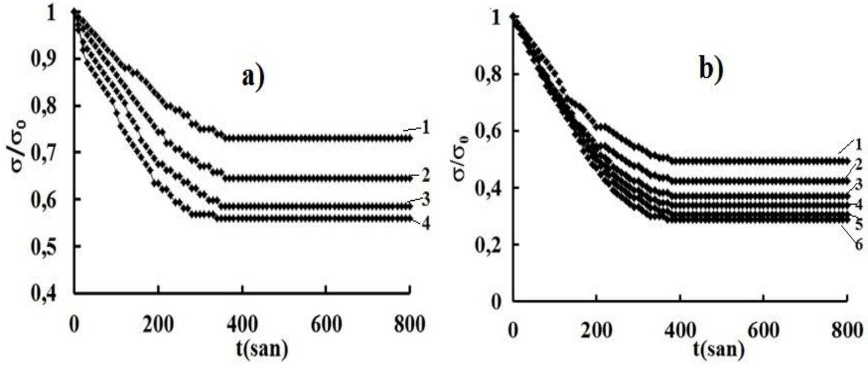

Figure 3. a) $\sigma(t)/\sigma_0(t)$ dependences of $(TlInS_2)_{0.95}C_{0.05}$ compound
1-350K; 2-400K; 3-450K;4- 500 K.

b) $\sigma(t)/\sigma_0(t)$ dependences of $(TlInS_2)_{0.9}C_{0.1}$ compound
1-350K;2-400K;3-470K; 4-500K;5-550K;6-600K

That is, with increasing temperature, the concentration of ions participating in ionic conductivity increases. At 550K, in the time dependence of the electrical conductivity of the $(TlInS_2)_{0.95}C_{0.05}$ system solid solution, ion portion to the conductivity increases to 44%, while in the $(TlInS_2)_{0.9}C_{0.1}$ compound, ion portion increases to 70% at the same temperature.

In this chapter, the impedance spectra of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds at different temperatures were also studied. The impedance spectroscopy (IS) method is used to obtain information about charge transport, taking into account the microstructure of solids. It is shown that the impedance spectra of the $(TlInS_2)_{0.9}C_{0.1}$ solid solution in the complex plane ($Z''=f(Z')$) at 300K, 380K, 450K, 550K are given. Here, the frequency range corresponding to the accumulation of volume charges in the complex plane varies from 25 Hz to 2 kHz, and the frequency value corresponding to the charge transport at the maximum of the frequency at different constant temperatures varies from 500 Hz to 50 kHz.

The fourth chapter of the dissertation presents the temperature dependence of the complex $\varepsilon^*(T)$ dielectric permittivity of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ compounds irradiated with gamma quanta (0÷0.8MGy) above room temperature Fig 4.(a,b).

It was determined that at radiation doses of 0÷0.8MGy, the numerical value of the dielectric permittivity increases, the peaks shift in the direction of increasing temperature, and the temperature range related to ionic conductivity expands. The formation of a defect by heat, if it is close to another defect (for example, an impurity atom), can accept an electron or vacancy, which leads to a decrease in the free energy of the crystal. If the resulting defect brings its energy level into the band gap and carries current at the resulting level, the presence of nearby defects is not necessary. The formed defect is energetically favorable because it traps a charge carrier. It is known that, the degree of disorder of crystals can be changed under the influence of γ-radiation. Under the influence of γ-quanta, vacancies lead to the formation of radiation defects in the form of interstitial atoms, and various types of defect complexes.

Ionization-type defects (charged defects), which are generated by irradiation with gamma quanta, play a primary role in these processes.

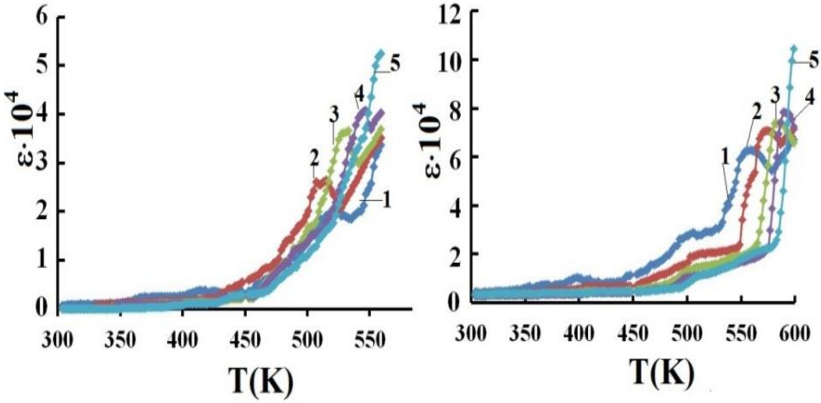

Figure 4. Temperature dependence of dielectric permittivity of (a) $(TlInS_2)_{0.95}C_{0.05}$ and (b) $(TlInS_2)_{0.9}C_{0.1}$ compounds:
1-0; 2-0.2 MGy; 3-0.4MGy; 4-0.6MGy; 5-0.8MGy.

That is, the reason for the increase in ionic conductivity of these compounds depending on the radiation dose (dose increase) is the increase in the concentration of ions. In the fourth chapter, the time dependence of the relative conductivity σ/σ_0 (t) of the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ at different constant temperatures in a constant electric field in the absorption dose range 0÷0.8MGy was studied. Figure 5.(a,b) shows the kinetics of the electrical conductivity of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ irradiated with γ-rays in the dose range 0÷0.8MGy at a temperature of T = 470K.

In Figure 5. (a,b), as the absorption dose of γ-rays increases, the electron portion to the total conductivity decreases and, consequently, the ion portion increases. The experimental results show that the ionic conductivity of $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ increases from 44% to 75% and from 63% to 78.5%, respectively, at radiation doses of 0÷0.8MGy.

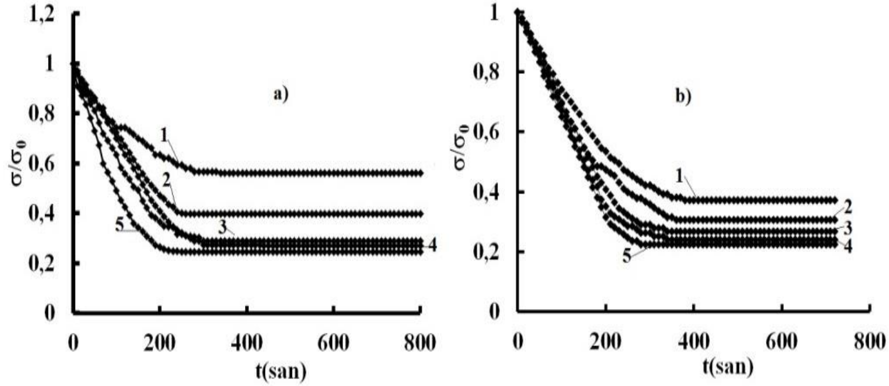

Figure 5. Time dependence of the ratio of electrical conductivities of (a) $(TlInS_2)_{0.95}C_{0.05}$ and (b) T=470K $(TlInS_2)_{0.9}C_{0.1}$ in a constant field T=550K:
1-0; 2-0.2 MGy; 3-0.4MGy;4-0.6MGy; 5-0.8MGy.

The dependence of the relaxation time and diffusion coefficient on the absorption dose is given in Table 1. In Table 1 with an increase in the absorption dose, the relaxation time decreases and the value of the diffusion coefficient increases accordingly.

Table 1

Dependence of relaxation time (τ) and diffusion coefficient (D) on absorbed dose for $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$.

$(TlInS_2)_{0.95}C_{0.05}$			$(TlInS_2)_{0.9}C_{0.1}$	
$D_{ab.dose}$ MGy	τ sec	D_{diffus} $\frac{m^2}{sec}$	τ sec	D_{diffus} $\frac{m^2}{sec}$
0	1728.9	$2.3\cdot10^{-10}$	1008.79	$5.7\cdot10^{-10}$
0.2	1083.05	$3.6\cdot10^{-10}$	842.61	$6.8\cdot10^{-10}$
0.4	807.83	$4.9\cdot10^{-10}$	761.158	$7.6\cdot10^{-10}$
0.6	742.34	$5.38\cdot10^{-10}$	704.402	$8.2\cdot10^{-10}$
0.8	700.71	$5\cdot10^{-10}$	669.797	$8.6\cdot10^{-10}$

For the $(TlInS_2)_{0.95}C_{0.05}$ compound, the relaxation time (τ) decreases from 1728 sec to 700 sec, the diffusion coefficient (D) increases from $2.3\cdot10^{-10}m^2/sec$ to $5\cdot10^{-10}m^2/sec$, and the ionic conductivities increase from 44% to 75%, corresponding to 0÷0.8MGy. For the $(TlInS_2)_{0.9}C_{01}$ compound, the relaxation time (τ) decreases from 1008 sec to 669 sec, the diffusion coefficient (D) increases from $5.7 \cdot 10^{-10}m^2/sec$ to $8.6 \cdot 10^{-10}m^2/sec$, and the ionic conductivities increase from 63% to 78.5%.

Figure 6. shows the dependences of the impedance spectra of the compounds $(TlInS_2)_{0.95}C_{0.05}$(a) and $(TlInS_2)_{0.9}C_{01}$(b) on the absorbed doses in the complex plane Z''= f(Z'). As can be seen from the figures, the hodograph dependences can be divided into two parts. The first part characterizes the sample and is in the form of a semicircle (high-frequency part). The other characterizes the sample-electrode boundary and is in the form of a straight line at an angle to the abscissa axis (low-frequency region).

As the absorbed dose increases, the frequencies corresponding to the maxima of the $(TlInS_2)_{0.95}C_{0.05}$(a) and $(TlInS_2)_{0.9}C_{01}$(b) hodographs shift towards the high-frequency region.The frequency maxima vary in the range of 5kHz÷50kHz for $(TlInS_2)_{0.95}C_{0.05}$ and 750Hs÷10kHs for $(TlInS_2)_{0.9}C_{01}$, respectively. The equivalent circuit corresponding to the impedance spectra in the complex plane Z''= f(Z') is given in Figure 6.(c).

This chapter presents the results of the study of the dielectric properties of the $(TlInS_2)_{0.95}C_{0.05}$ compound irradiated with H^+ ions. It is shown that the temperature dependence of the dielectric permittivity of the $(TlInS_2)_{0.95}C_{0.05}$ compound irradiated with protons with an energy of 150keV and a radiation dose of 10^{16} ions/cm^2 repeats the form of the dependence before irradiation, but the peaks shift towards the region of high temperatures. At the same time, the numerical value of the dielectric permittivity increases after irradiation with protons, and the increase is more pronounced at relatively high temperatures. As a result of irradiation with protons, an increase in the numerical value of the dielectric permittivity is observed due to an increase in the concentration of mobile ions in the $(TlInS_2)_{0.95}C_{0.05}$ compound.

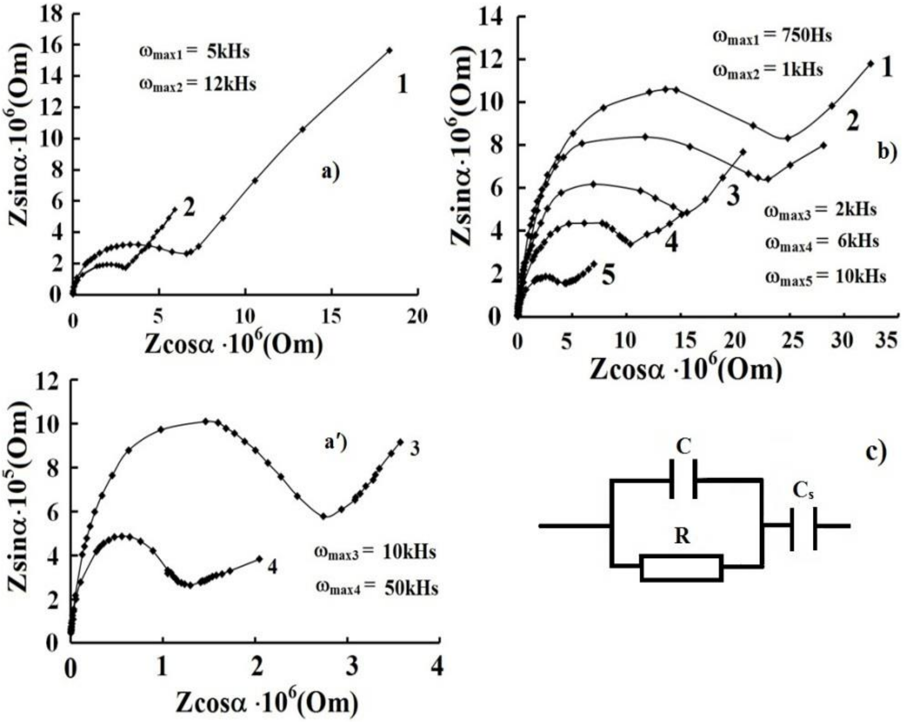

Figure 6. Dependence of the impedance spectra in the complex plane Z''=f(Z'), of the hodographs of the compounds (a),(a′) $(TlInS_2)_{0.95}C_{0.05}$ and (b) $(TlInS_2)_{0.9}C_{01}$. on the absorbed doses:

1-0MGy; 2-0.2MGy; 3-0.4MGy; 4-0.6MGy; 5-0.8MGy

c) Equivalent scheme corresponding to the hodographs

The electron and ion portions to the total conductivity of $(TlInS_2)_{0.95}C_{0.05}$ implanted with H^+ ions with an energy of 150keV and a radiation dose of 10^{16} ion/cm^2 were calculated. It was found that after irradiation with 150keV energy, the electron portions to the total conductivity decreases in the $(TlInS_2)_{0.95}C_{0.05}$ compound and, as a result, the ion portion increases. The portion to the electrical conductivity of $(TlInS_2)_{0.95}C_{0.05}$ at a temperature of 550K is 43.3% before irradiation, and 75.5% after irradiation with 150keV protons. The calculated values of the relaxation time and diffusion coefficient of the $(TlInS_2)_{0.95}C_{0.05}$ compound, unirradiated and irradiated with 150keV H^+ ions, are as follows. In the unirradiated case, the relaxation time is τ_{SC}=1728.939sec, the diffusion coefficient - $D=3.3\cdot10^{-10}$, and in the case of irradiated with H^+ ions, relaxation time is τ_{SC}=853.367 sec, the diffusion coefficient - $D=6.7\cdot10^{-10}$. The depth distribution of vacancies created by protons during both the penetration and recoil of the $(TlInS_2)_{0.95}C_{0.05}$ compound implanted with 150 keV H^+ ions was simulated.

It is shown that the distribution of vacancy-type defects created by proton ions reaches a peak at a depth of 980 nm during recoil and 860 nm during propulsion, then decreases with increasing depth and is distributed homogeneously at a depth of 1200-1500 nm. The concentration of defects reaches a maximum at this depth. On the other hand, the broadening of the depth distribution profile of vacancies created by recoiling proton ions is observed.

It can be assumed that as the implant ions slow down inside the material, their trajectories are relatively unstable due to their interaction with the atoms in the lattice. When protons are implanted into the sample, they lose more energy as they penetrate deeper into the material. A peak is observed in both spectra at a depth of approximately 160 nm. It should be noted that since the $(TlInS_2)_{0.95}C_{0.05}$ compound has a layered structure, there is a possibility of interstitial-type defects forming in this compound as a result of the action of low-energy protons.

Interstitial defects are atoms located between the normal positions of atoms in a crystal structure, that is, between the lattice sites. These atoms can be either the crystal's own atoms or a different type of atom. The causes of interstitial defects are varied. These causes can be due to high temperature, radiation, the introduction of external atoms, etc. At high temperatures, atoms gain more energy, which causes them to move out of their normal lattice sites and into interstitial regions. This process changes the dynamics of the crystal structure, leading to the formation of new defects. When high-energy particles (neutrons or charged ions) enter a solid, they can displace atoms. These atoms then settle in the interstitial regions, creating defects. Also, external atoms with smaller atomic radius can easily enter the voids inside the material and, as a result, cause the formation of interstitial defects. Interstitial atoms can cause the scattering of electrons, which changes the electrical conductivity of the material. Also, since interstitial atoms can move more easily in the material, they accelerate diffusion processes. Taking the above into account, we can again say that the carbon (C) atom with a small ionic radius enters the octahedral voids in the $TlInS_2$ crystal and creates interstitial defects. Figure 7.(b) shows the depth distribution of proton ions. It has been shown that in the $(TlInS_2)_{0.95}C_{0.05}$ compound, the depth distribution of H^+ ions reaches a maximum at a depth of 1060 nm and decreases with increasing depth, and after a depth of 1500 nm, the effect of protons with an energy of 150 keV disappears.

The spectra of H^+ ions in the $(TlInS_2)_{0.95}C_{0.05}$ compound before and after implantation were comparatively studied by the Combinational Light Scattering method. YAG: Nd laser with a wavelength of $\lambda = 532$ nm was used to record Raman spectra. The Raman spectra were recorded at room temperature in the frequency range of 20-500 cm^{-1} using the laser. In the combinational light scattering spectra of the $(TlInS_2)_{0.95}C_{0.05}$ compound before implantation, 7 peaks (20, 41, 70, 137, 308, 322, 391 cm^{-1}) corresponding to intralayer vibrations were observed. Also, 4 peaks (137, 193,287, 391) with weak and relatively strong intensity are observed in this compound after implantation. As can be seen from Figure 7.(a), there is a strong difference in the peaks of the photosensitivity of the CLS spectra of the $(TlInS_2)_{0.95}C_{0.05}$ compound before and after implantation. Taking this into account, it can be concluded that amorphization occurs in the near-surface regions of the $(TlInS_2)_{0.95}C_{0.05}$ compound.

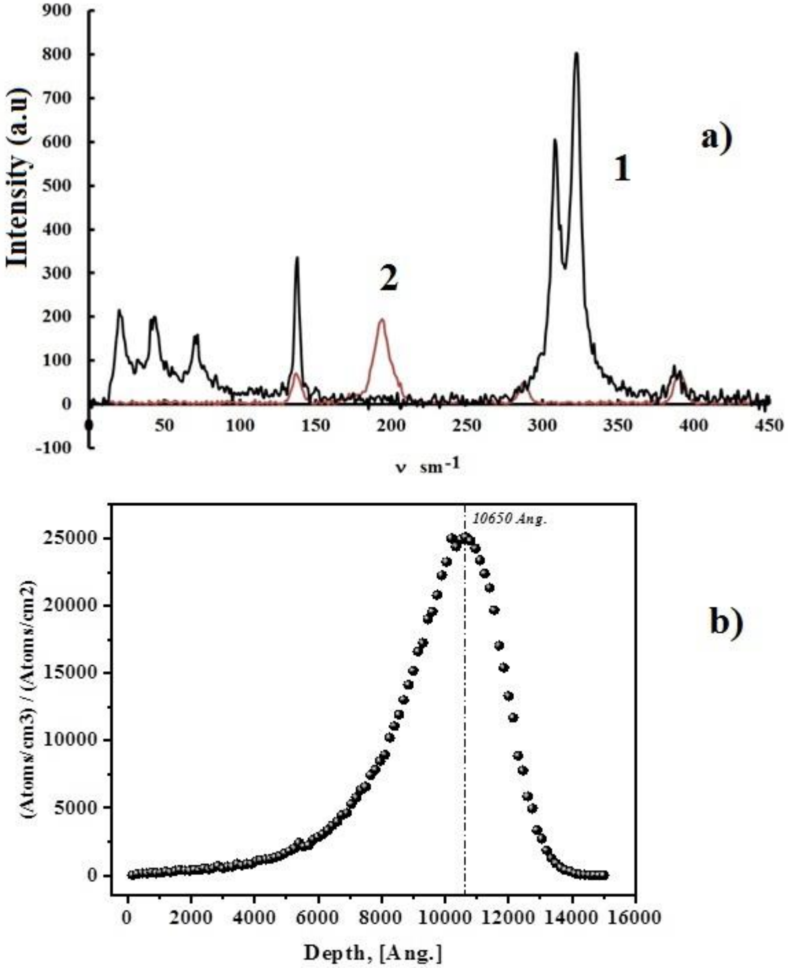

Figure 7. In the compound $(TlInS_2)_{0.95}C_{0.05}$:
a. CLS spectra (1-unirradiated, 2- implanted with H^+ ions).
b. Depth distribution of H^+ ions with 150 keV.

MAIN RESULTS

1. The results obtained from X-ray structural analysis and Combinational Light Scattering experiments show that the structures

of the compounds $TlInS_2$, $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ are identical. That is, the graphite (C) atoms with small ionic radius are located in the octahedral voids in the In_4S_{10} complex, creating interstitial-type defects and forming solid solutions with the introduction of the (C) atom.

2. For the first time, anomalies corresponding to ionic conductivity were observed in the ε(T) dependences: the anomalies observed in the $(TlInS_2)_{0.95}C_{0.05}$ compound at temperatures T_1 = 370K, T_2 = 415K and T_3 = 532K, and in $(TlInS_2)_{0.9}C_{0.1}$ at temperatures T_1 = 400K, T_2 = 509K and T_3 = 561 K are associated with the presence of a large number of relaxators in these compounds. The values of the activation energies of the relaxators were calculated from the experimentally obtained ln(ε)/1000/T dependences: they are ΔEa_1 = 0.6eV, ΔEa_2 = 0.38eV for $(TlInS_2)_{0.95}C_{0.05}$, and ΔEa_1 = 0.54eV, ΔEa_2 =0.32eV, ΔEa_3 =0.22eV for $(TlInS_2)_{0.9}C_{0.1}$. The increase in dielectric permittivity and ionic conductivity depending on the radiation doses (0÷0.80MGy) is due to the increase in the concentration of ions.

3. It is shown that as the frequency of change of the electric field increases, the peaks of tgδ(T) shift to a higher temperature region and the value of tgδ decreases. The frequencies of the charges leaving the potential hole for the compounds studied from the dispersion of tgδ(T) were determined from the 1/T dependence of lgf_{max}. For the compound $(TlInS_2)_{0.95}C_{0.05}$, the oscillation frequency was determined to be $\nu = 8\cdot10^{12}$ Hz, and for $(TlInS_2)_{0.9}C_{0.1}$, it was $\nu= 4\cdot10^{12}$. These oscillations correspond to the oscillations of thallium atoms.

4. The ion portion, electron portion, relaxation time (τ) and diffusion coefficient (D) of the $TlInS_2$(5%C) and $TlInS_2$(10%C) compounds were determined from the kinetics in a constant electric field (σ(t)). As a result of the increase in the concentration of ions in the region near the electrode with the increase in the absorption dose of γ-rays, their polarization increases, the electron portion, relaxation time decreases, and the ionic fraction and diffusion coefficient values increase. The ionic conductivities of the $(TlInS_2)_{0.95}C_{0.05}$ and

$(TlInS_2)_{0.9}C_{0.1}$ compounds vary from 44% to 75% and from 63% to 78.5%, respectively, at radiation doses of 0÷0.8 MGy.

5. The complex impedance spectra of the compounds $(TlInS_2)_{0.95}C_{0.05}$ and $(TlInS_2)_{0.9}C_{0.1}$ were studied in the complex plane $Z''=f(Z')$ at absorption doses (0÷0.8 MGy) and it was shown that, corresponding to the charge transport process, a semicircle is obtained in the diagram (high-frequency part of the hodograph), and the volume of the region near the electrode corresponds to a straight line inclined toward the low-frequency part of the hodograph due to the polarization of the charges. The frequency peaks vary in the range 5kHz÷50kHz for $(TlInS_2)_{0.95}C_{0.05}$, and 750Hz÷10kHz for $(TlInS_2)_{0.9}C_{01}$, respectively.

6. The electron-ion portion to the conductivity, relaxation time, diffusion coefficient, and complex impedance spectra of the $(TlInS_2)_{0.95}C_{0.05}$ compound irradiated with protons with an energy of 150keV and a radiation dose of 10^{16} ion/cm^{2}sec in a constant electric field was studied. Prior to implantation, the relaxation time was τ_{SC}=1728.939sec, diffusion coefficient was $D=3.3\cdot10^{-10}$m^2/sec, and the ion portion to the electrical conductivity was 43.3% before irradiation, while after irradiation with H^+ ions, the relaxation time was τ_{SC}=853.367sec, diffusion coefficient was $D=6{,}7\cdot10^{-10}$m^2/sec, and the ionic fraction of the conductivity varied up to 75.5%.

7. It has been demonstrated that amorphization occurs in the near-surface region due to proton irradiation. The depth distribution of H^+ ions reaches a maximum at a depth of 1060 nm and decreases with increasing depth, and after a depth of 1500 nm the effect of protons disappears. Additionally, the distribution of vacancy-type defects caused by proton ions reaches a maximum at a depth of 980 nm during recoil and at a depth of 860 nm during propulsion, then decreases with increasing depth and is distributed homogeneously at a depth of 1200-1500 nm. At these depths, the concentration of defects in depth reaches a maximum.

PUBLISHED SCIENTIFIC WORKS ON THE TOPIC OF THE DISSERTATION

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Personal participation of the author in printed works:

Works No. [1-12] – setting the problem, conducting experimental researches was performed by the author, solving the problem and processing of the results obtained from the researches were performed jointly with the co-authors

The dissertation defense will be held on 17 April, 2025, at 15:^{00}, at the meeting of the FD1.21 Dissertation Council operating under the Institute of Radiation Problems of the Ministry of Science and Education of the Republic of Azerbaijan.

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