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INFLUENCE OF ELECTRON IRRADIATION ON THE CRYSTAL STRUCTURE, SURFACE MICRORELIEF AND BANDGANP WIDTH OF THE TRIPLE CRYSTALS OF IRON DOPED MONOSELINIDE OF THALLIUM AND INDIUM

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In this work, the effect of electron irradiation on the structure, surface morphology and band gap of single- and polycrystals of iron-doped ternary crystals of thallium and indium monoselinides was investigated. It has been established that the synthesized polycrystalline samples, as well as the grown single crystals of thallium and indium monoselinides, are single-phase. The crystals have a tetragonal unit cell (space group I4/mcm) with the following lattice parameters: a ~ b = 8.12 Å, c = 6.88 Å, α=β=γ = 90°. Irradiation with electrons with an energy of 2 MeV and a beam current density of 0.085 μA/cm^2 leads to changes in the structure and properties of crystals in a complex manner depending on the electron fluence. Leads to a change in the parameters of the crystal lattice, an increase in the maximum value of the arithmetic mean deviation of the profile, as well as a decrease in the height of the average surface roughness of the irradiated crystals. Irradiation of a powder sample with electrons with a fluence of 5x10^{16} electron/cm^2 helps to increase the size of nanocrystallites from 32.50 nm to 43.33 nm.

Keywords: single crystal, crystal structure, unit cell, space group, electron irradiation, fluence, crystallite size, roughness.

1. Introduction

Ternary crystals of thallium and indium monoselinides $TlInSe_2$ belong to the group of ternary chalcogenide compounds of type $A^{III}B^{III}C_2^{VI}$ with a pronounced layered structure. A characteristic feature of crystals of this family is their manifestation of both semiconductor [1-3] and tensor-resistive [4,5] properties. Since the crystal structure of compounds of this type is layered, this, as a rule, leads to the formation of polytypic modifications, which significantly affects their physical properties. Crystals of these compounds are attractive from the point of view of their practical application - they are promising materials for the manufacture of photoelectric converters, spectrum analyzers and detectors of X-ray, gamma and neutron radiation on their basis [6,7] and it is important to develop methods for targeted control of the properties of these materials.

It is known that one of the powerful methods for controlling the properties of solids is the introduction of impurities into their structure [8], as well as irradiation of them with various types of nuclear and ionizing radiation [9], which requires a targeted study of physical processes in doped and irradiated materials. However, radiation effects in chalcogenide compounds of the $A^{III}B^{III}C_2^{V}$ type, especially in doped samples, have been studied very poorly and unsystematically. Available data are sometimes contradictory. For example, in [7] a study was carried out of the effect of electron irradiation on the dielectric constant ε and electrical conductivity σ of $TlInS_2$ and $TlGaS_2$ crystals in the low temperature region and it was shown that under the influence of electron beam irradiation, the dielectric constant ε of $TlInS_2$ and $TlGaS_2$ crystals decreases, and the conductivity σ significantly growing. In contrast, in [10] it was established that in $TlInS_2$ crystals of the hexagonal modification (HM), starting from a certain dose, radiation defects accumulate and electrical conductivity decreases. As the radiation dose increases, due to the interaction of radiation defects with the original inhomogeneities, complex defects are formed, as a result of which the electrical conductivity value increases exponentially. The authors of [10] suggested that the decrease in electrical conductivity at low doses is associated with partial compensation of the initial level of the donor type. Irradiation with large doses of n-$TlInS_2$ (above 200 krad) leads to the formation of accumulations of radiation defects and strong compensation of the material. According to the authors, the indicated nature of the change

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in the electrical conductivity of n-$TlInS_2$ HM crystals indicates the formation of a continuous series of deep acceptor levels during irradiation in the band gap of $TlInS_2$ [11], which accept part of the electrons. With further irradiation, the nature of the dependence $\sigma \sim f(T)$ changes.

An analysis of the results of experiments carried out in [10] showed that non-equilibrium point radiation defects formed in the layers, migrating, accumulate in the interlayer space and thereby reduce the anisotropy of the crystal at high irradiation doses.There is very little information about radiation effects in doped $TlInSe_2$ crystals. We are aware of the works [12-14] where the influence of γ-radiation on the thermal conductivity, electrical conductivity and dielectric properties of $TlInSe_2$ single crystals in the temperature range of 80-600 K was studied. It was found that in irradiated crystals above 400 K the thermal conductivity increases, which is associated with a decrease in concentration defects during γ-irradiation of the crystal.

Taking into account the above, the purpose of this work is to study the effect of electron irradiation on the crystal structure, surface morphology and band gap of crystals p-$TlIn_{0,98}Fe_{0,02}Se_2$ doped with an iron impurity, since experimental data on the effect of irradiation on the physicochemical characteristics of these crystals are not available in the literature.

2. Material and Methods

To achieve this goal, crystals were used that were synthesized by fusing the components in a stoichiometric ratio in evacuated (~10^{-3} Pa) and sealed quartz ampoules. Highly pure elements of thallium, indium, iron and selenium were used as starting components for the synthesis. Single crystals of the synthesized compounds were grown using the improved Bridgman method [15].

The possibility of replacing indium atoms in the $TlInSe_2$ crystal lattice with iron atoms is indicated by the fact that the ionic radius of the dopant Fe^{3+} (0.62 Å) is closer to the ionic radius of In^{3+} (0.81 Å) than to the ionic radius of Tl^{1+} (1.38 Å) [16], i.e., partial replacement of indium with iron in a layered $TlInSe_2$ crystal corresponds to the condition for the formation of a substitutional solid solution.

Irradiation of samples of the crystals under study with electrons with an energy of 2 MeV and a beam current density of 0.085 μA/cm^2 was carried out at the "Electronics U-003" accelerator of the Institute of Nuclear Physics of the Academy of Sciences of the Republic of Uzbekistan [17]. The electron energy was selected based on the density and thickness of the sample, and was determined using a standard measuring wedge (P4701) Riso 2 Piece Aluminum (Belgium) made of aluminum. The sample was installed perpendicular to the direction of the electron beam at a distance of 0.4 m from the accelerator sweep and irradiation was carried out to an electron fluence of 1.5×10^{17} electron/cm^2.

A three-dimensional image of the surface relief of the samples was obtained using an SPM 9700HT scanning probe microscope (Shimadzu). The study was carried out in the contact mode of operation of a scanning probe microscope. To do this, a section of the sample measuring 30x30 μm was selected and the number of "peaks-protrusions", their half-width and height, was determined before and after irradiation. In what follows in the text we will use the following notation: R_a is the arithmetic mean deviation of the profile from the center line drawn using the least squares method within the base length, R_z is the height of profile irregularities at 10 points, i.e. the average value of the absolute heights of the five largest protrusions of the profile and the depths of the five largest depressions of the profile within the base length. The base length is the length of the line used to highlight irregularities that characterize surface roughness and quantify its parameters.

X-ray diffraction studies of the structure of $TlIn_{0.98}Fe_{0.02}Se_2$ were carried out on a Malvern Panalytical Empyrean diffractometer. XRD data were recorded using a Malvern Panalytical Empyrean analytical diffractometer with CuKα radiation (λ=1.54 Å). In this experiment, the accelerating voltage of the radiation generator was set to 45 kV and the emission current was set to 40 mA. X-ray diffraction patterns were recorded in the Bragg–Brentano beam geometry at $2\theta_Б$ = 20° - 120° continuously with a scanning speed of 0.33 deg/min.

The X-ray diffraction data were processed by the Rietveld method using the FullProf program [18].Based on the obtained powder X-ray diffraction data, the crystallite size was determined using the Debye-Scherrer formula [19]:

$$D = K\lambda/(\beta\cos\theta) \quad (1)$$

where D is the average crystallite size, K is the geometric coefficient (0.9), λ is the X-ray wavelength (1.5406 Å), β is the diffraction reflection width at half maximum (FWHM), θ is the diffraction angle.The dislocation density was determined from the equation [20]:

$$\delta = 1/D^2 \quad (2)$$

The microstress value in the $TlIn_{0,98}Fe_{0,02}Se_2$ crystal was calculated using the Stokes-Wilson equation:

$$\varepsilon = \beta/(4\tan\theta) \quad (3)$$

The optical absorption of the $TlIn_{1-x}Fe_xSe_2$(x=0.02) single crystal was studied using a Lambda-35 UF-V spectrophotometer (Perkin Elmer) in the wavelength range 190–1100 nm. To determine the band gap of $TlIn_{0,98}Fe_{0,02}Se_2$ from absorption spectra in the UF-visible region, we used the Tauc relation[22]:

$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (4)$$

where $E_g = hc/\lambda$ – optical band gap energy, h – Planck's constant (6.626×10^{-34} J•s), c – speed of light (3×10^8 m/s), λ – absorbed wavelength, α – absorption coefficient, hν is the energy of the incident photon in eV, A is the band edge steepness constant.

3. Experimental results and discussion

The results of measuring and processing X-ray diffraction data of $TlIn_{0,98}Fe_{0,02}Se_2$ single crystals, measured under the same conditions, are shown in Fig. 1 and Table 1. It was revealed that the synthesized samples were single-phase. Interplanar distances calculated from X-ray patterns can be unambiguously identified on the basis of the tetragonal system, that the lattice parameters have the following values: $a \sim b$ = 8.067746 Å, c = 6.815503Å, (space group $I4/mcm$) α=β=γ = 90°.

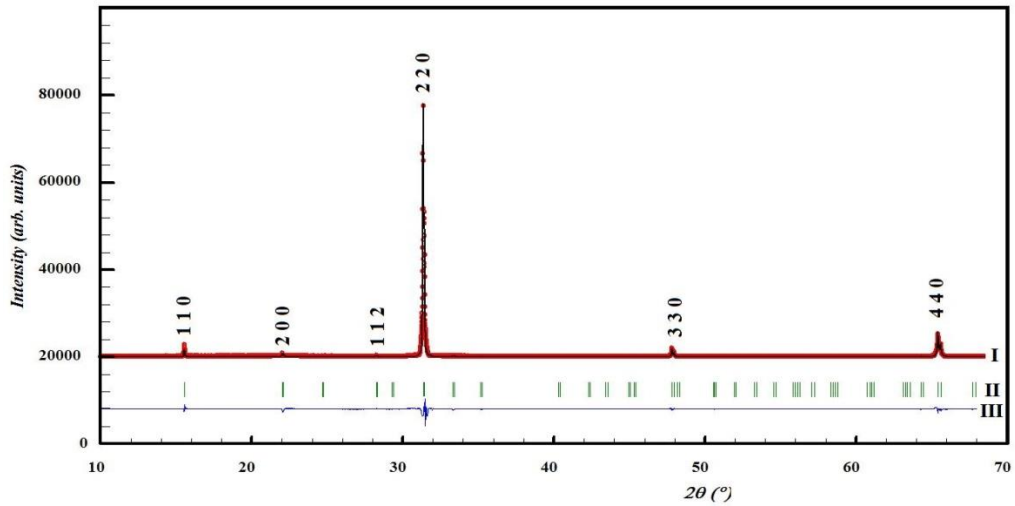

Fig.1. X-ray diffraction pattern of the initial $TlIn_{0,98}Fe_{0,02}Se_2$ single crystal: I – experimental and calculated data; II – Bragg reflections; III – difference curve between experimental and calculated data

To obtain relatively complete information about the structure and compare the structural data of a single crystal and a powdered sample of $TlIn_{0,98}Fe_{0,02}Se_2$, an X-ray diffraction study of a powdered sample of $TlIn_{0,98}Fe_{0,02}Se_2$was carried out, the results were compared with the X-ray diffraction characteristics of the single crystal (Fig. 2, Table 2). It can be seen that in the range of Bragg scattering angles $2\theta_Б=10^0$-60^0 (Fig. 2) there is a sufficient number of reflections that make it possible to obtain complete information about the structural characteristics of the sample. Processing by the full-profile method of X-ray diffraction data from a sample of $TlIn_{0,98}Fe_{0,02}Se_2$ powder showed that the sample, like the $TlIn_{0,98}Fe_{0,02}Se_2$ single crystal, has a tetragonal unit cell (space group $I4/mcm$) with the following lattice parameters: $a \sim b$ = 8.12 Å, c = 6.88 Å, α=β=γ = 90°.

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Table 1. Structural characteristics of a $TlIn_{0,98}Fe_{0,02}Se_2$ single crystal irradiated with accelerated electrons.

Electron radiation dose, e/cm^2	2θ(°)	β– (FWHM) (°)	D - Crystallite size (nm)	δ - Dislocation density (×10^{15} lines/m^2)	ε - Lattice strain (%)
0	31.34	0.099	76.56	0.17	0.15
5.10^{14}	31.28	0.0956	80.03	0.156	0.149
1.10^{15}	31.26	0.095	80.53	0.154	0.148
5.10^{15}	31.27	0.094	81.39	0.15	0.146
5.10^{16}	31.32	0.104	73.41	0.18	0.16

Based on calculations of X-ray diffraction data, the crystal structure was constructed (Fig. 3), the sizes of $TlIn_{0,98}Fe_{0,02}Se_2$ crystallites and some other characteristics were determined (Table 2), from which it can be seen that the crystallite size increases with increasing fluence of electron irradiation.

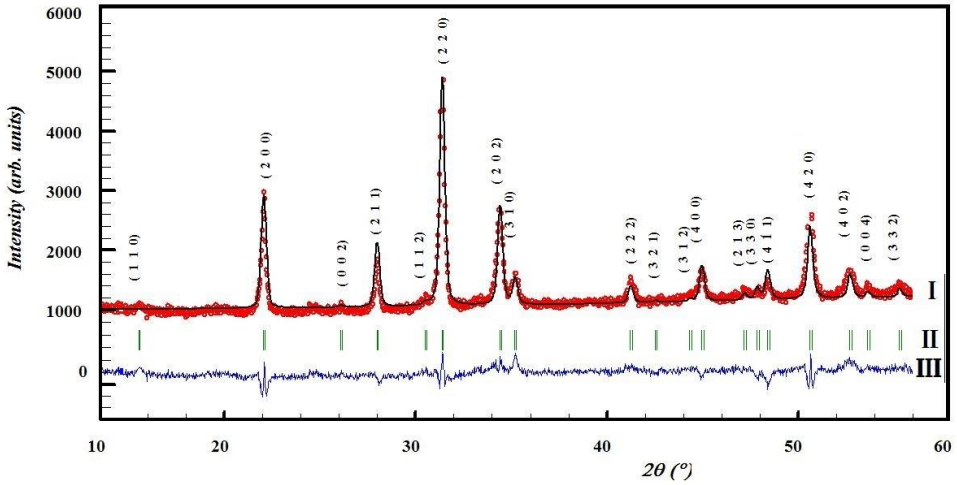

Fig.2. X-ray diffraction pattern of powdered $TlIn_{0,98}Fe_{0,02}Se_2$ irradiated with electrons with a fluence of 5×10^{16} el/cm^2: I – experimental and calculated data; II – Bragg reflections; III – difference curve between experimental and calculated data

Table 2. Structural characteristics of a $TlIn_{0,98}Fe_{0,02}Se_2$ powder sample irradiated with accelerated electrons

Electron radiation dose,e/cm^2	2θ(°)	β– (FWHM) (°)	D - Crystallite size, (nm)	δ– Dislocationdensity (×10^{15}lines/m^2)	ε–Latticestrain, (%)
0	31.2472	0.2387	32.05	0.97	0.37
5.10^{15}	31.2492	0.2241	34.14	0.86	0.35
5.10^{16}	31.2498	0.2156	35.49	0.79	0.34

A comparison of the results obtained in $TlIn_{0,98}Fe_{0,02}Se_2$ crystals irradiated with different doses of accelerated electrons reveals an interesting picture. With an increase in the electron fluence to 5×10^{15}, it leads to a decrease in the parameters of the unit cell of the crystal, and above this fluence value the parameters begin to increase (Table 3). In this case, the band gap of the crystal, determined using the Tauc[22] relation, changes in the opposite direction, i.e., it increases with an increase in electron fluence up to 5×10^{15} el/cm^2, and above this fluence it begins to decrease (Fig. 4 and Table 3). Previously, similar phenomena were discovered during the irradiation of $TlInSe_2$ crystals and other varieties of thallium chalcogenides [10] and [23,24].

For example, in [23,24] it was established that the photosensitivity of $TlInSe_2$ crystals depends in a complex way on the fluence of electrons and neutrons. With an increase in electron fluence to 1×10^{13} el/cm^2,

photosensitivity increases by 30-80%, and with a further increase in electron fluence it decreases and at a fluence of 10^{15} el/cm^2 it is 30-40% of the sensitivity of the original non-irradiated samples. The authors admitted the possibility of a relationship between this phenomenon and the effect of small doses, the essence of which is as follows.

It is known [25] that during irradiation in solids, unstable Frenkel pairs are created with a frequency 1-2 orders of magnitude higher than stable defects, which manage to have a significant impact on the processes of radiation-stimulated migration of point defects. During the irradiation of solids (metals, alloys, semiconductor and dielectric materials), metastable Frenkel pairs are created, which lead to changes in various physicochemical properties of the irradiated material [26.27]. One of the characteristic phenomena associated with unstable radiation defects is the so-called “radiation shaking” of crystals [26-29].

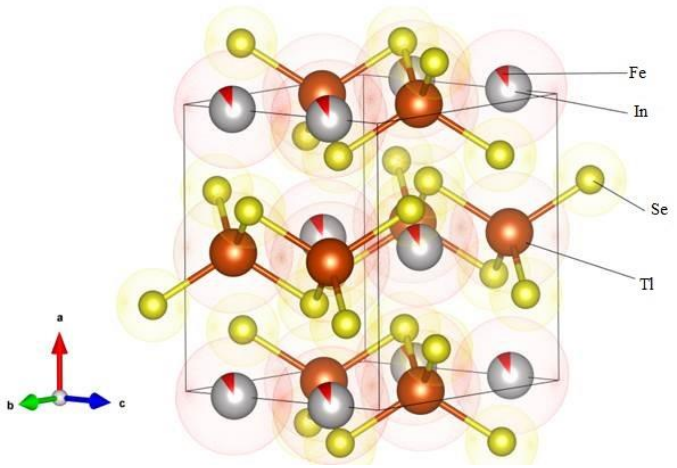

Fig.3. Crystal structure of $TlIn_{0,98}Fe_{0,02}Se_2$.

Table 3. Change in lattice parameters and band gap of $TlIn_{0,98}Fe_{0,02}Se_2$crystals under the influence of accelerated electrons

No.	Radiation dose, el/cm^2	Singonia	Latticeparameters	E_g,eV
1	unirradiated	I4/mcm tetragonal	a = 8.067746; b = 8.067746; c = 6.815503; α=β=ϒ = 90^0	1.652
2	5×10^{14}	I4/mcm tetragonal	a = 8.061345; b = 8.061345; c= 6.795211; α=β=ϒ = 90^0	1.657
3	5×10^{15}	I4/mcm tetragonal	a = 8.056042; b = 8.056042; c = 6.771103; α=β=ϒ = 90^0	1.666
4	5×10^{16}	I4/mcm tetragonal	a = 8.062459; b = 8.062459; c = 7.135920; α=β=ϒ = 90^0	1.663

The authors [25] of theoretically showed that during the irradiation of solids, unstable Frenkel pairs are created in them with a frequency 1-2 orders of magnitude higher than stable defects, therefore they manage to have a significant impact on the processes of radiation-stimulated migration of point defects. It has been

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established [30-35] that during irradiation, metastable Frenkel pairs are created in various solid materials: metals, alloys, materials of a semiconductor and dielectric nature and leads to a change in various physicochemical properties of the irradiated material. One of the characteristic phenomena associated with unstable radiation defects is the so-called "radiation shaking" of the crystal [26,28,29].

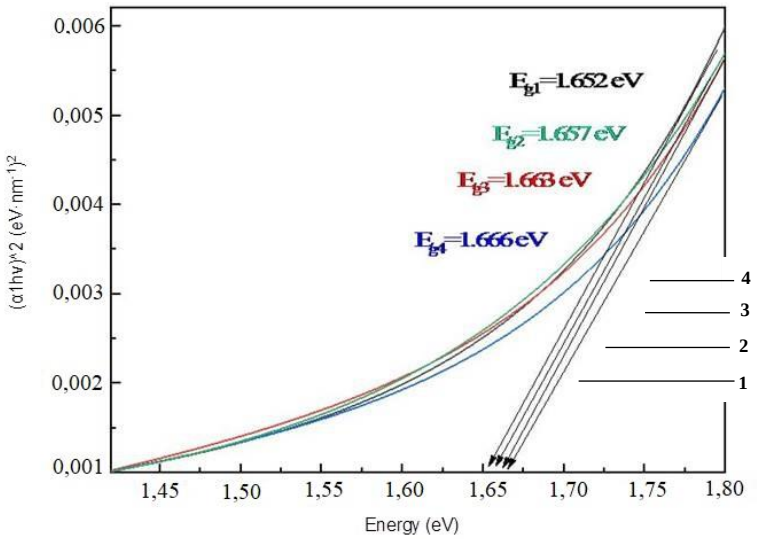


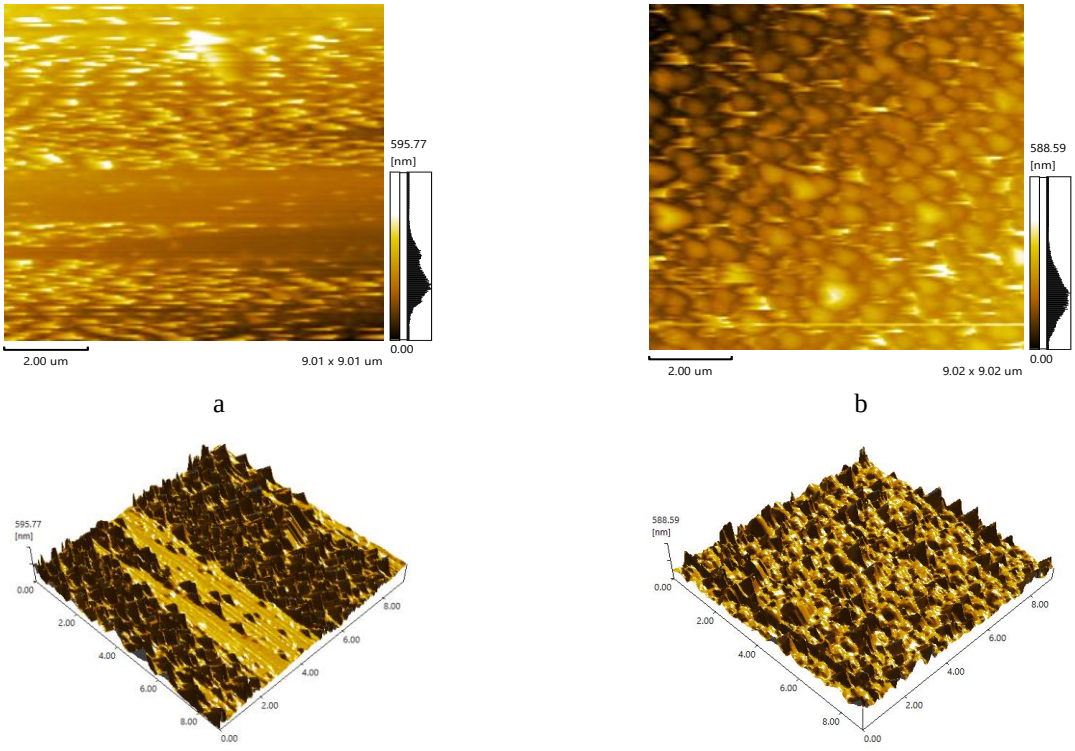
Fig.4. Absorption spectra of $TlIn_{0,98}Fe_{0,02}Se_2$ single crystals irradiated at different electron fluences: 1-initial; 2- $5\cdot10^{14}$ el/cm^2; 3- $5\cdot10^{15}$ el/cm^2; 4-$5\cdot10^{16}$ el/cm^2

Indenbom [26] proposed a mechanism for the phenomenon of "radiation shaking", which leads to radiation-accelerated diffusion, caused by the birth and death of unstable Frenkel pairs (NFPs). The essence of this phenomenon is that during the creation and annihilation of Frenkel pairs of radiation defects, elastic stress waves arise as a result of the resulting local volume change in the crystal.

The interaction of these waves with existing point defects can lead to the non-activation migration of interstitial atoms and their annihilation with the corresponding vacancies, which does not lead to the accumulation of structural defects, but, on the contrary, to their elimination and transfer of the material to a more equilibrium state compared to the initial one [26,28].

The proposed mechanism was confirmed by the results of numerous computer and real physical [28-35] experiments. It has been established that there is a so-called low-dose effect (D = 10^3–10^7 R), in which, in contrast to large doses, the crystal structure is rearranged and ordered due to the release of energy stored in the crystal as a result of chain reactions of defect annihilation initiated by ionization. The transition of a material to an equilibrium state is accompanied by an improvement in a number of its physical properties: a change in the concentration of traps, an increase in conductivity, an increase in the lifetime of minority charge carriers, etc.

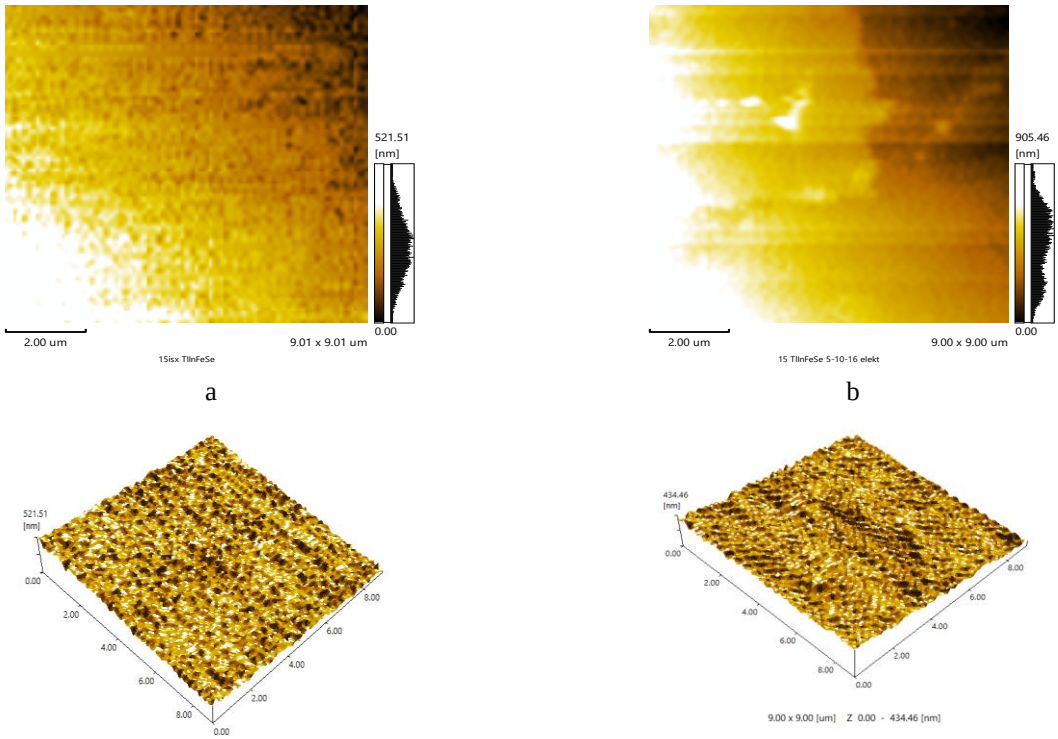
In our opinion, the observed decrease in the lattice parameter of the $TlIn_{0,98}Fe_{0,02}Se_2$crystal and the increase in the band gap is associated precisely with the ordering of growth defects - the effect of low doses. When the number of radiation-induced defects begins to exceed the number of biographical growth defects, an accumulation of radiation defects is observed in the crystal, which begins to deteriorate the characteristics of the crystals.The results of studying the ACM surface microrelief of undoped $TlInSe_2$ and iron-doped $TlIn_{0,98}Fe_{0,02}Se_2$ single crystals before and after irradiation with accelerated electrons are shown in Fig. 5 and 6, as well as in Table4. The study was carried out without preliminary treatments. According to the data obtained, the introduction of iron impurities into $TlInSe_2$ crystals significantly changes the surface morphology: it leads to a decrease in the maximum value of the arithmetic mean deviation of the profile from R_a = 40.869 to R_a = 17.7 nm, and also leads to a decrease in the height of the average roughness from R_z = 398.036 nm to R_z = 189.915 nm. (Fig. 5 and 6; Table 4). Thus, doping $TlInSe_2$ crystals with iron will significantly improve the condition of the crystal surface; it will become smoother during doping.



a b

c d

Fig.5. Two-dimensional (a and b) and volumetric (c and d) ACM images of the surface of a $TlInSe_2$ single crystal before (a and c) and after (b and d) irradiation with electrons with a fluence of 5×10^{16} electron/cm^2.



a b

c d

R_z=189/915 nm R_z=431/435 nm

Fig.6. Two-dimensional (*a* and b) and volumetric (c and d) AFM images of the surface of a doped $TlIn_{0,98}Fe_{0,02}Se_2$single crystal before (*a* and c) and after (b and d) irradiation with electrons with a fluence of 5×10^{16} electron/cm^2.

Table 4. Changes in the surface microrelief of $TlInSe_2$ single crystals upon doping with iron and irradiation with electrons with a fluence of 5×10^{16} electron/cm^2

Sample	Original		Electronirradiated,5×10^{16} electron/cm^2	
	R_a,nm	R_z,nm	R_a,nm	R_z, nm
$TlInSe_2$	40.869	398.036	40.998	444.579
$TlIn_{0,98}Fe_{0,02}Se_2$	17.693	189.915	33.716	431.435

In contrast to doping, irradiation with electrons with a fluence of 5×10^{16} el/cm^2 significantly worsens the surface condition of both undoped and, especially, doped crystals. If the morphological characteristics of an undoped sample change insignificantly, then in doped samples these changes are significant (see Fig. 5 and 6; Table 3): in iron-containing crystals, when irradiated with electrons with a fluence of 5×10^{16} electron/cm^2, the maximum value of the arithmetic mean deviation of the profile increases from R_a = 17.693 nm to R_a = 33.716 nm, and average roughness heights from R_z = 189.915 nm to R_z = 398.036 nm. (Table 4).

Thus, electron irradiation significantly worsens the surface condition of $TlIn_{0,98}Fe_{0,02}Se_2$crystals.

4. Conclusion

In this work, using X-ray diffraction research, it was established that when doping $TlInSe_2$ crystals with 2 mol. % $TlFeSe_2$, as well as their irradiation with accelerated electrons with an energy of 2 MeV and a beam current density of 0.085 μA/cm^2 to a fluence of 10^{17} electron/cm^2, the samples remain single-phase and tetragonal, with space group *I*4/mcm. With increasing exposure to electrons to a fluence of 5×10^{15} electron/cm^2, the unit cell parameter of the $TlIn_{0,98}Fe_{0,02}Se_2$crystal decreases, and the band gap Eg increases. With a further increase in the electron fluence, the value of the crystal lattice parameter increases, and E_g decreases. The observed phenomenon is explained from the point of view of the effect of low doses: during irradiation of solids with ionizing radiation, unstable Frenkel pairs are created in them with a frequency 1-2 orders of magnitude higher than stable structural defects. During the birth and annihilation of unstable Frenkel pairs of defects, short-term local volume changes in the crystal and its rapid recovery occur, resulting in elastic stress waves. The interaction of these waves with existing point defects can lead to the non-activation migration of interstitial atoms existing in the crystal volume and their annihilation with the corresponding vacancies, which leads to their healing and transfer of the material to a more equilibrium state compared to the initial defective one. Therefore, at low doses of irradiation, in our case up to an electron fluence of 5×10^{15} electron/cm^2, as a result of the recombination of biographical complementary pairs of defects, the irradiated crystal becomes more defect-free. At those irradiation doses when the number of radiation-induced defects becomes greater than the number of initial biographical defects (in our case, the fluence of electron irradiation is F>5×10^{15} electron/cm^2), structural defects begin to accumulate in the crystal, the parameters of the crystal lattice begin to increase, and accordingly the band gap of the crystal decreases.

Along with the above, doping, as well as electron irradiation of $TlInSe_2$ crystals, greatly changes the surface morphology of $TlFeSe_2$ and $TlIn_{0,98}Fe_{0,02}Se_2$ crystals. The introduction of iron impurities into $TlFeSe_2$ crystals leads to an improvement in the surface condition, a decrease in the maximum value of the arithmetic mean deviation of the profile (from R_a = 40.869 to R_a = 17.7 nm), as well as a decrease in the height of the average roughness (from R_z = 398.036 nm to R_z = 189.915 nm). In contrast, irradiation with electrons with a fluence of 5×10^{16} el/cm^2 significantly worsens the surface condition in both undoped and, especially, doped crystals. If the morphological characteristics of an undoped sample after irradiation change insignificantly, then in doped samples these changes are significant: in iron-containing crystals, when irradiated with electrons with a fluence of 5×10^{16} electron/cm^2, the maximum value of the arithmetic mean deviation of the profile increases (from R_a = 17.693 nm to R_a = 33.716 nm), and the height of the average roughness increases (from R_z=189.915 nm to R_z=398.036 nm).

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