Influence of accelerated electrons on the structure, crystallite size and surface of a $TlIn_{1-x}Cr_xS_2$ crystal with x = 0.01

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В работе исследовано влияние электронного облучения на структуру и морфологию поверхности монокристалла и поликристалла $TlIn_{1-x}Cr_xS_2$ (x=0.01). Облучение монокристалла $TlIn_{1-x}Cr_xS_2$ электронами с энергией 2 МэВ и флюенсом 2×10^{17} электрон/см^2 приводит к увеличению максимального значения среднего арифметического отклонения профиля от R_a=5.4 нм до R_a=25.2 нм, а также приводит к увеличению высоты средней шероховатости от R_z=72.0 нм до R_z=152.9 нм. Облучение порошкового $TlIn_{1-x}Cr_xS_2$ электронами флюенсом 2×10^{17} электрон/см^2 способствует увеличению размера кристаллитов от 36.87 nm до 41.76 nm, что, скорее всего, связано с уменьшением границ раздела.

Ключевые слова: монокристалл, структура кристаллов, элементарная ячейка, пространственная группа, электронное облучение, флюенс, размер кристаллитов, шероховатость.

In this work, we studied the effect of electron irradiation on the structure and morphology of the surface of a single crystal and polycrystal $TlIn_{1-x}Cr_xS_2$ (x=0.01). Irradiation of a $TlIn_{1-x}Cr_xS_2$ single crystal with electrons with an energy of 2 MeV and a fluence of 2×10^{17} electron/cm^2 leads to an increase in the maximum value of the mean arithmetic deviation of the profile from R_a=5.4 nm to R_a=25.2 nm, and also leads to an increase in the height of the average roughness from R_z=72.0 nm to R_z=152.9 nm. Irradiation of powder $TlIn_{1-x}Cr_xS_2$ with electrons with a fluence of 2×10^{17} electron/cm^2 promotes an increase in the crystallite size from 36.87 nm to 41.76 nm, which is most likely associated with a decrease in size of the interface.

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

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I. Introduction

At present, there is an increasing interest in the study of new semiconductor materials containing several elements, which makes it possible to control their properties by varying the content of elements. $TlInS_2$ single crystals are one of such multicomponent complex wide-band semiconductors of the $A^{III}B^{III}C^{VI}_2$ type, in which it is possible to replace part of the sulfur atoms, as well as part of the indium atoms. Changing the composition allows one to widely vary the electro-physical, photoelectrical, optical, and other properties. Wide-band semiconductors $TlInS_2$ are promising for the manufacture of photoelectric converters, spectrum analyzers, and radiation detectors [1].

The study of $TlInS_2$ is of interest, since many of their properties depend on the crystal structure, stability to external influences, defects, etc. Therefore, the

crystal structure and properties of the $TlInS_2$ crystal were studied in [1-2]. Among them, $TlInS_2$ [3] is the most promising semiconductor, which allows one to "select" the optimal properties by replacing some of the sulfur atoms with other atoms in a wide concentration range [3]. According to [4-9], $TlInS_2$ compounds have different crystal structures (monoclinic, hexagonal, tetragonal triclinic and orthorhombic). The effect of the substitution of a part of sulfur atoms by selenium atoms on the structure, phase state, optical characteristics, and Raman spectroscopy was studied in detail in [10-11].

The main parameters of semiconductors depend not only on the structure, but also on the defects present in them and their type. In many cases, to improve certain properties, defects are specially introduced by various methods. One of the ways to create (or anneal) defects is to irradiate the sample with gamma-rays, neutrons, protons, and electrons. The effect of gamma-rays on the structure of $TlInS_2$ has been studied in a few works, for example, in [12], where a phase transition from the initial monoclinic phase to the hexagonal phase in a thin surface layer was established at high doses of irradiation (1000–1500 Mrad) with gamma-rays. The influence of electrons on structures and structural parameters is not studied, although it is of interest, since the types of formed radiation defects in these cases can be different [3].

In [13-15], the interaction of $TlInS_2$ with $TlErS_2$ was studied, and was found that at a component ratio of 1:1, a compound Tl_2InErS_4 with congruent melting is formed, as well as solid solutions based on $TlInS_2$.

The $TIInS_2$-$TIYbS_2$ system was studied in [16]. The authors claim that the solubility of $TIYbS_2$ in $TIlnS_2$ at room temperature is 12 mol.%, and the $TlIn_{1-x}Cr_xS_2$ solid solutions have a tetragonal structure.

It should be noted that the structure of the ternary compound $TlCrS_2$ was studied in [17] and was found that $TlCrS_2$ has hexagonal symmetry with unit cell parameters a=3.538 Å and c=21.92 Å [18]. It should be noted that the substitution of a part of indium atoms in $TlInS_2$ with atoms of another group, chromium, is also of interest, since it can introduce a "change" in the initial crystal structure and its parameters, as well as on the surface, since the thermal characteristics of indium and chromium differ [18-19].

However, in the literature, there is almost no study of the $TlIn_{1-x}Cr_xS_2$ (x = 0.01) compound and the effect of radiation on its structures and surfaces, which is most likely due to the difficulty of synthesizing samples.

Therefore, the purpose of this work is to study the structure, surface morphology and the effect of accelerated electrons on the structure and surface of the $TlIn_{1-x}Cr_xS_2$ crystal (x = 0.01).

II. Experimental procedure

An assessment of the possibility of chromium solubility in the bulk of the crystal lattice of $TlInS_2$ indicates that the ionic radius of the dopant Cr^{3+} (0.65 Å) is closer to the ionic radius of In^{3+} (0.94 Å) than to the ionic radius of Tl^{1+} (1.49 Å), i.e. In a layered $TlInS_2$ crystal, partial replacement of indium with chromium corresponds to the condition for the formation of a substitutional solid solution. Highly pure chemical elements Tl (0%), In (0%), S, Sulfur elemental extra pure model (EP-16-5), and Cr (99.99%) were used as initial components. $TlIn_{1-x}Cr_xS_2$ (x = 0.01) was synthesized by direct fusion of chemical elements taken in stoichiometric ratios in a quartz ampoule with a vacuum of up to 10^{-5} Pa.

Electron irradiation of $TlIn_{1-x}Cr_xS_2$ (x = 0.01) was carried out at the «Electronika U-003» accelerator of the INP AN RUz. A three-dimensional image of the surface relief of a $TlIn_{1-x}Cr_xS_2$ sample (x = 0.01) was obtained with an SPM 9700 HT scanning probe microscope (Shimadzu). The study was carried out in a contact mode of operation of a scanning probe microscope. For this, a sample area of 30×30 μm was selected and the number of "peaks-protrusions", their half-width and height, before and after irradiation with accelerated electrons, were determined.

X-ray structural studies of $TlIn_{1-x}Cr_xS_2$ (x = 0.01) were carried out on an Empyrean diffractometer with CuK_α radiation in the angle range $2\theta_B$=(7–90)° at a scanning speed of 0.36 degrees/min. A full-profile analysis of the results obtained was carried out using the Rietveld method [20], and the center of the peak was determined using the OriginPro9 program [21].

To study the effect of accelerated electrons on the structure of a $TlIn_{1-x}Cr_xS_2$ single crystal (x = 0.01), it was irradiated with electrons with an energy of 2 MeV and a beam current density of 0.375 mkA/cm^2 [22]. The energy of the irradiated electrons was selected based on the density and thickness of the samples under study. Sample $TlIn_{1-x}Cr_xS_2$ (x = 0.01) was installed perpendicular to the direction of the electron beam at a distance of 0.4 m from the electron accele-

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rator sweep, and irradiation was performed to an electron fluence of 2×10^{17} el./cm^2.

To obtain more complete structural information on the effect of electron irradiation on the structure we also studied the initial and irradiated powdered $TlIn_{1-x}Cr_xS_2$ ($x = 0.01$).

III. Experimental results and discussion

The study of the effect of radiation on the surface roughness of $TlIn_{1-x}Cr_xS_2$ ($x = 0.01$) showed that it grows under the action of accelerated electrons. However, the width and height of the tubercle increases (Fig. 1, 2, 3).

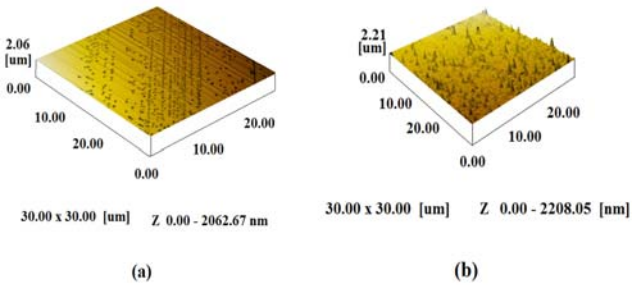


Figure 1. Topography of the surface of a $TlIn_{1-x}Cr_xS_2$ single crystal with $x = 0.01$: *a* – initial sample; *b* – irradiated with a dose of 2×10^{17} electron/cm^2.

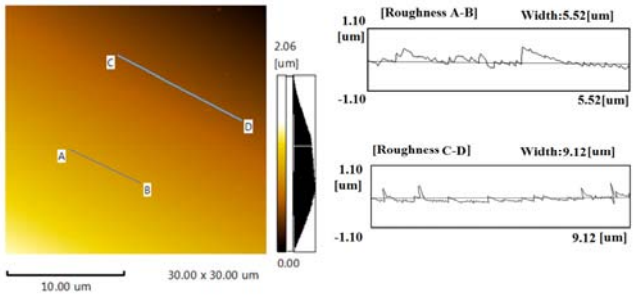


Figure 2. Roughness of the surface of a $TlIn_{1-x}Cr_xS_2$ single crystal with $x = 0.01$.

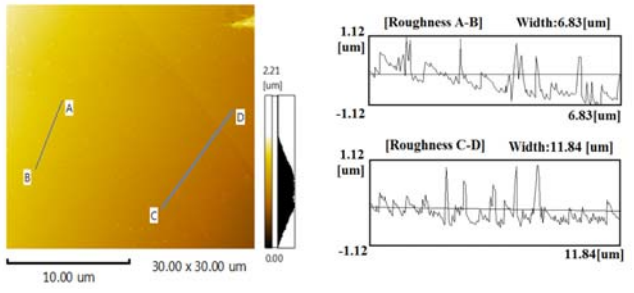


Figure 3. Roughness of the surface of a $TlIn_{1-x}Cr_xS_2$ single crystal with $x = 0.01$ after electron irradiation with a fluence of 2×10^{17} electron/cm^2 .

The measurement showed that the maximum value of the arithmetic mean deviation of the profile of the initial $TlIn_{1-x}Cr_xS_2$ crystal ($x = 0.01$) is R_a = 5.4 nm, the height of the mean roughness R_z = 72.0 nm, and after irradiation with electrons with a fluence of 2×10^{17} electron/cm^2 the maximum value of the mean arithmetic deviation of the profile is R_a = 25.2 nm and the average roughness height R_z = 152.9 nm.

The processing of the X-ray diffraction pattern (Fig. 4) showed that the initial single crystal $TlIn_{1-x}Cr_xS_2$ ($x = 0.01$) has a hexagonal (space group P63/mmc) structure. In this case, the unit cell parameters are a = 3.958 Å, b = 3.958 Å, c = 14.922 Å, and the atomic coordinates and thermal factors are given in Table 1.

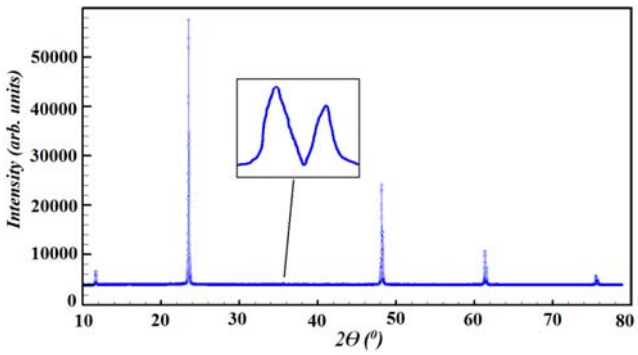


Figure 4. X-ray diffraction pattern of a $TlIn_{1-x}Cr_xS_2$ single crystal ($x = 0.01$).

Table 1. The positions and coordinates of the atoms of the $TlIn_{1-x}Cr_xS_2$ single crystal ($x = 0.01$) in the space group. $P6_3/mmc$.

№	Atom	Atomic coordinates			Heat factor
		x	*y*	*z*	*B*
1	Tl	0.3333	0.6667	0.2500	0.669
2	In	0.0000	0.0000	0.0000	0.878
3	S	0.3333	0.6666	0.9037	0.913
4	Cr	0.0000	0.0000	0.0000	0.132

After irradiation with 2 MeV electrons and a fluence of 2×10^{17} electron/cm^2, the unit cell parameters of the $TlIn_{1-x}Cr_xS_2$ single crystal ($x = 0.01$) are a = 3.852 Å, b = 3.852 Å, c = 14.911 Å and α = 90°, β = 90°, γ = 120°. The results obtained indicate that the unit cell volume of the $TlIn_{1-x}Cr_xS_2$ single crystal slightly decreased after irradiation with electrons; the introduced amount of Cr ($x = 0.01$) weakly affects the parameters of the crystal lattice of the $TlInS_2$ matrix. The obtained crystallographic data for the single crystal $TlIn_{1-x}Cr_xS_2$ ($x = 0.01$) agree with the $TlInS_2$ data

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(a = 10.90 Å, b = 10.94 Å, c = 15.18 Å, β = 100.21°) [6].

The X-ray diffraction pattern of the initial $TlIn_{1-x}Cr_xS_2$ powder sample (x = 0.01) was processed on the assumption of the formation of a hexagonal (space group $P6_3/mmc$) structure; however, the Bragg value R_B is 2.15 factor, indicating that the proposed model was consistent with the experimental results (R_B is 1.59). Calculations of the X-ray diffraction pattern assuming the formation of a monoclinic unit cell, as in $TlnS_2$, showed the formation of the same phase in powder $TlIn_{1-x}Cr_xS_2$, i.e. with space group C2/c (Fig. 5).

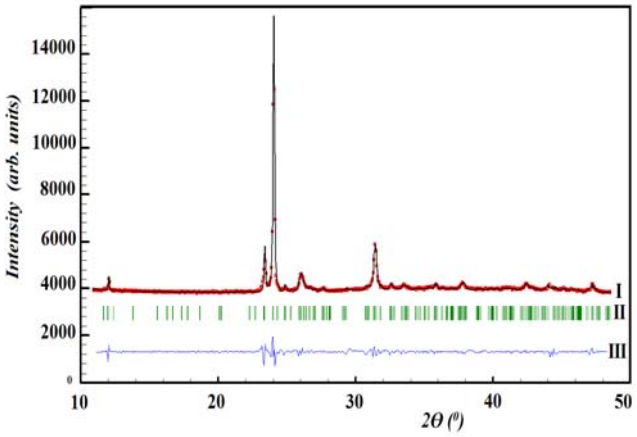


Figure 5. X-ray diffraction pattern of $TlIn_{1-x}Cr_xS_2$ powder (x = 0.01). I – comparison of the observed pattern with the fitted pattern, II – Bragg peaks, III – the difference between the observed pattern and the fitted pattern.

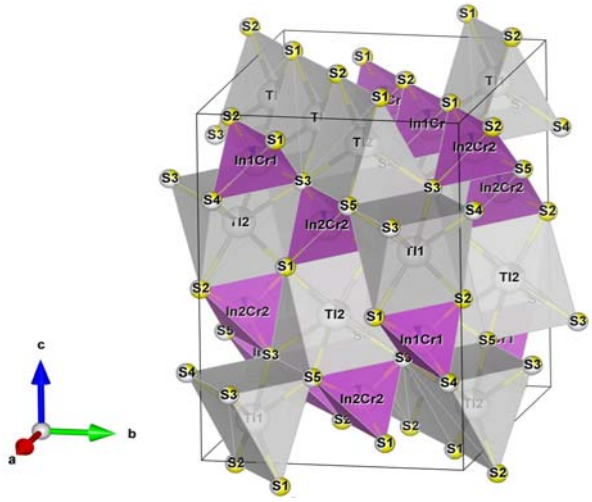


Figure 6. Crystal structure of $TlIn_{1-x}Cr_xS_2$ (x = 0.01) (monoclinic structure of space group C2/c)

The results of calculating the X-ray diffraction pattern of powdered $TlIn_{1-x}Cr_xS_2$ (x = 0.01) irradiated with electrons with a fluence of 2×10^{17} electron/cm^2 showed that the crystal structure was preserved; corresponds to a monoclinic structure (space group C2/c) (Fig. 6). However, the coordinates of the atoms, as well as the parameters of the unit cell, compared to the initial one, have changed (in Table 2.).

Table 2. Positions and coordinates of atoms of the irradiated $TlIn_{1-x}Cr_xS_2$ powder (x = 0.01) in space group C2/c.

№	Atom	Atomic coordinates			Heat factor
		x/a	*y/b*	*z/c*	*B*
1	Tl	0.0310	0.1874	0.3828	0.842
2	Tl	0.2142	0.4416	0.1195	0.910
3	In	0.0998	0.1886	0.6550	0.951
4	In	0.1497	0.4378	0.8451	0.852
5	Cr	0.0998	0.1886	0.6550	0.151
6	Cr	0.1497	0.4378	0.8451	0.132
7	S	0.0482	0.3150	0.9447	0.319
8	S	0.1990	0.0624	0.5554	0.027
9	S	0.2392	0.1870	0.2504	0.290
10	S	0.0000	0.0523	0.7500	0.676
11	S	0.0000	0.4267	0.2500	0.509

a, Å	*b*, Å	*c*, Å	β, °	*a*, Å	*b*, Å
10.873	10.956	15.175	100.63	10.873	10.956

The size of the coherent X-ray scattering region corresponding to the average crystallite size was determined from the width of the diffraction reflections of the powder sample. To calculate the size of the coherent scattering region (CSR), a reflection with $2\theta_B$=23.8064° was selected and the full width at half maximum was calculated. For this, the Scherrer formula was used [23, 24]:

$$D = \frac{K\lambda}{\beta_S \cos\theta}. \qquad (1)$$

Calculations showed that the average crystallite size of the initial powder sample $TlIn_{1-x}Cr_xS_2$ (x = 0.01) is d = 36.87 nm. After irradiation of the sample with accelerated electrons with a fluence of 2×10^{17} electron/cm^2, the average crystallite size increased to d = 41.76 nm.

Earlier, it was found that the initial $TlInS_2$ single crystal is two-phase with hexagonal (space group $P6_3/mmc$) and monoclinic (space group C2/c) phases [3]. The arithmetic mean deviation of the profile of the initial $TlInS_2$ crystal is R_a = 19.8 nm, the mean maximum peak height is R_z = 387.6 nm. Powdered $TlInS_2$ with a monoclinic (space group C2/c) structure has the following parameters: a = 10.926 Å, b =

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10.934 Å, c = 15.208 Å, β = 100.65°, and the average crystallite size is 54.7 nm.

After electron irradiation, the $TlInS_2$ single crystal passes from a two-phase state (space group $P6_3/mmc$ and space group C2/c) to a single-phase state with space group $P6_3/mmc$, the unit cell parameters are: $a = b$ = 10 Å, c = 14.923 Å [3]. It was found that the maximum value of the arithmetic mean deviation of the profile after irradiation with electrons 2×10^{17} electron/cm^2 was R_a = 48.1 nm, the average peak height R_z = 694.4 nm [3]. The results of the X-ray study of the $TlInS_2$ crystal in powder form after electron irradiation are as follows: a=10.881 Å, b= 10.955 Å, c=15.185 Å, and β=100.63° (space group C2/c) and the average crystallite size is 114.32 nm.

The initial $TlInS_xSe_{2-x}$ single crystal has a monoclinic structure (space group C2/c) (x = 1) [21] with a = 11.566 Å, b = 11.223 Å, c = 15.319 Å, and β=100.21°. Irradiation of a $TlInS_xSe_{2-x}$ single crystal (x = 1) with electrons with a fluence of 2×10^{17} electrons/cm^2 (energy 2 MeV) contributed to a change in the unit cell parameters with a = 10.969 Å, b = 11.06 Å, c = 15.327 Å, and β = 100.47°. And powder $TlInS_xSe_{2-x}$ (x = 1) consisted only of a monoclinic phase (space group C2/c) with a =11.109 Å, b=11.126 Å, c=15.35 Å and β=100.69° and with an average crystallite size of 56.5 nm. After electron irradiation with a fluence of 2×10^{17} electrons/cm^2, the structural state was preserved, the unit cell parameters underwent a slight change along the a axis, and the average crystallite size increased to 65 nm. Thus, it can be seen that the replacement of a part (50%) of sulfur atoms with selenium leads to a change not only in the lattice parameters, but also in the phase state and sizes of crystallites. And with a small substitution of chromium atoms for indium atoms (1%), the unit cell parameters and the crystallite size also change.

IV. Conclusions

It was found that the $TlIn_{1-x}Cr_xS_2$ single crystal (x = 0.01) has a hexagonal (space group $P6_3/mmc$) structure with the parameters a=3.958 Å, b=3.958 Å, c=14.922 Å and α = 90°, β = 90°, γ = 120°.

It was found that after irradiation of a $TlIn_{1-x}Cr_xS_2$ single crystal (x = 0.01) with electrons with a fluence of 2×10^{17} electron/cm^2, the original structure is retained, the maximum value of the mean arithmetic deviation of the profile increases by more than five times, and the height of the average roughness increases from R_z = 72.0 nm to R_z = 152.9 nm.

Powdered $TlIn_{1-x}Cr_xS_2$, as in $TlInS_2$ has a monoclinic structure (space group C2/c) (Fig. 5) with unit cell parameters a=10.864 Å, b=10.948 Å, c= 15.151 Å, and β = 100.61°.

Irradiation with 2 MeV electrons and a fluence of 2×10^{17} electron/cm^2 of powder $TlIn_{1-x}Cr_xS_2$ (x = 0.01) promotes an increase in the crystallite size from 36.87 nm to 41.76 nm, which is most likely associated with a decrease in the interfaces.

It was shown that the $TlInS_2$, $TlInS_xSe_{2-x}$ (x = 1), and $TlIn_{1-x}Cr_xS_2$ (x = 0.01) powder samples contain a monoclinic structure (space group C2/c) and no structural transitions were found under electron irradiation, but the average crystallite size increased.

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$TlIn_{1-x}Cr_xS_2$ (x=0.01) монокристалнинг структурасига, кристаллитлар ўлчамига ва юзасига тезлаштирилган электронларнинг таъсири

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Бу ишда $TlIn_{1-x}Cr_xS_2$ (x=0.01) монокристалининг структурасига, кристаллитлар ўлчамига ва юзасига тезлаштирилган электронларнинг таъсири тадқиқ қилинган. $TlIn_{1-x}Cr_xS_2$ (x=0.01) монокристалини энергияси 2 МэВ бўлган электронлар билан 2×10^{17} электрон/см^2 флюенсда нурлантириши профилни ўртача арифметик оғишининг максимал қийматини R_a=5.4 нм дан R_a=25.2 нм га ошишига олиб келади, шунингдек, ўртача ғадир-будурлик баландлигининг R_z = 72.0 нм dan R_z = 152.9 нм гача ошишига олиб келади. Энергияси 2 МэВ бўлган электронлар билан 2×10^{17} электрон/см^2 флюенсда $TlIn_{1-x}Cr_xS_2$ (x=0.01) кукунини нурлантириш кристаллитлар ўлчамини 36.87 нм дан 41.76 нм гача ўсишга олиб келади, бу эса кристалдаги соҳалар чегаралари билан боғлиқ бўлиши мумкин.

Калит сўзлар: монокристал, кристаллар структураси, элементар катак, фазовий гуруҳ, электрон билан нурлантириш, флюенс, кристаллитлар ўлчами, ғадир-будурлик.

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