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Structural and Optical Properties of $Cu_2ZnSn(S_xSe_{1-x})_4$ Thin Films

E.P. Zaretskaya[1], V.F. Gremenok[1], A.V. Stanchik[1], A.N. Pyatlitski[2], V.A. Solodukha[2], K.A. Urazov[3], M.B. Dergacheva[3], Süleyman Özçelik[4], N.A. Djuzhev[5], A.M. Alekseev[5]

[1]SSPA «Scientific-Practical Research Centre of the National Academy of Sciences of Belarus», Minsk, Belarus
[2]JSC «INTEGRAL» – «INTEGRAL» Holding Managing Company, Minsk, Belarus
[3]Sokolsky Institute of Fuel, Catalysis And Electrochemistry, Almaty, Kazakhstan
[4]Gazi University, Ankara, Turkey
[5]National Research University of Electronic Technology, Moscow, Russia

m_dergacheva@mail.ru

Abstract: Recently the films of solid solutions $Cu_2ZnSn(S_xSe_{1-x})_4$ (CZTSSe) are used as the light absorbing layers in the thin film solar elements. The CZTSSe films are the $Cu(In, Ga)Se_2$ analog, but do not contain rare and expensive In and Ga elements. In this study the CZTSSe absorbing films have been grown by a two-step process. At the first step the electrochemical deposition of the Cu, Zn, Sn and S components on the Mo coated glass has been performed. At the second step the annealing in the Se(S) atmosphere under the N flow has followed. It has been shown that the phase composition and structure of the synthesized CZTSSe thin films are strongly dependent on their elemental composition and processing regimes. The CZTSSe polycrystalline thin films of tetragonal structure, containing kesterite and stannite phases with the band gap energies 1.36 and 1.48 eV , have been obtained. The investigation of CZTSSe films by Raman spectroscopy confirms the formation of CZTSSe solid solutions with a two-mode character of vibrations for films, containing more than 30 at% sulfur. The preliminary results demonstrate the applicability of the proposed approach for producing the CZTSSe absorber for thin film solar cells.

Keywords: kesterite; CZTSSe; electrodeposition; Raman spectroscopy; thin film

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Структурные и оптические свойства тонких пленок $Cu_2ZnSn(S_xSe_{1-x})_4$

Е.П. Зарецкая[1], В.Ф. Гременок[1], А.В. Станчик[1], А.Н. Петлицкий[2], В.А. Солодуха[2], К.А. Уразов[3], М.Б. Дергачева[3], Сулейман Осцелик[4], Н.А. Дюжев[5], А.М. Алексеев[5]

[1]ГНПО «Научно-практический центр Национальной академии наук Беларуси по материаловедению», г. Минск, Беларусь
[2]ОАО «ИНТЕГРАЛ» – управляющая компания холдинга «ИНТЕГРАЛ», г. Минск, Беларусь
[3]Институт топлива, катализа и электрохимии им. Д. В. Сокольского, г. Алматы, Казахстан
[4]Гази Университет, г. Анкара, Турция
[5]Национальный исследовательский университет «МИЭТ», г. Москва, Россия

m_dergacheva@mail.ru

В последнее время пленки твердых растворов $Cu_2ZnSn(S_xSe_{1-x})_4$ (CZTSSe) используются в качестве светопоглощающих слоев в тонкопленочных солнечных элементах. Пленки CZTSSe являются аналогом $Cu(In,Ga)Se_2$, но не содержат редких и дорогостоящих элементов In и Ga. В настоящей работе пленки CZTSSe получены двухстадийным методом. На первой стадии проведено электрохимическое осаждение компонентов Cu, Zn, Sn и S (или Se) на стекло с подслоем Mo. На второй осуществлен последующий отжиг в атмосфере Se(S) в потоке N_2. Показано, что фазовый состав и структура синтезированных пленок CZTSSe сильно зависят от их элементного состава и режимов обработки. Получены поликристаллические пленки CZTSSe тетрагональной структуры, содержащие фазы кестерита и станнита, с энергией запрещенной зоны 1,36 и 1,48 эВ. Исследование комбинационного рассеяния пленок подтверждает формирование твердых растворов CZTSSe с двухмодовым характером колебаний для пленок, содержащих свыше 30 ат.% серы. Предварительные результаты демонстрируют применимость предлагаемого метода для создания пленок CZTSSe для тонкопленочных солнечных элементов.

Ключевые слова: кестерит; CZTSSe; электроосаждение; комбинационное рассеяние света; тонкие пленки

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Introduction. In spite of the great progress that has been achieved in the conversion efficiency $Cu(In,Ga)Se_2$ (CIGS) thin-film solar cells (over 22 %) [1], their mass production is limited by expensive and rare Ga and In materials. In last decade the researchers have focused on the kesterites like Cu_2ZnSnS_4 and $Cu_2ZnSn(S_xSe_{1-x})_4$ chalcogenide (CZTSSe) materials as a promising candidate for cost effective thin film solar cells [2]. CZTSSe a *p*-type semicon-

ductor with a high absorption coefficient above 10^4 cm^{-1} and a direct band-gap (E_g = 1.05–1.50 eV depending on the S/Se composition ratio) consists of inexpensive and abundant elements. It could therefore allow to reduce cost production of thin-film solar cells. The reported power conversion efficiency of 12.6 % had been achieved for the anion-substituted $Cu_2ZnSn(S_xSe_{1-x})_4$ solid solution [3]. Deposition of CZTS thin film absorber layer has been developed by using various vacuum and non-vacuum techniques. One step electrochemical deposition of Cu-Zn-Sn-Se(S) precursors and subsequent sulfurization (or selenization) is considered as a promising approach for production the CZTSSe based thin film solar cells.

This work is devoted to the structural properties analysis of CZTSSe thin films formed by one-step electrochemical deposition of Cu, Zn, Sn, and S (or Se) components followed by annealing in Se(S)-containing atmosphere under N_2 flow. The effect of precursor composition and anion S → Se substitution on the CZTSSe thin films growth was studied.

Experimental. ***Deposition of Coatings.*** Electrochemical deposition of Cu-Zn-Sn-S and Cu-Zn-Sn-Se precursor films was performed in a three-electrode electrochemical cell using potentiostat/galvanostat Gill AC (ACM Instruments, UK). Mo/glass substrate was used as a working electrode, platinum – as auxiliary electrode and silver chloride electrode – as a reference electrode.

Cu-Zn-Sn-S thin films were deposited onto the Mo/glass (soda lime glass - SLG) substrates from the electrolyte based on 0.2M tri-sodium citrate, which contains the following salts: 0.01M $CuSO_4 \cdot 5H_2O$, 0.01M $ZnSO_4 \cdot 7H_2O$, 0.005M $SnSO_4$ and 0.05M $Na_2S_2O_3$, all of analytically pure grade. To adjust pH to 4.6 of the electrolyte, 0.1M tartaric acid was used as described in [4]. The electrolysis was performed at the constant potential of –1.0 V for 30 min at room temperature.

The set of Cu-Zn-Sn-Se thin films were electrodeposited onto the Mo/glass substrates from the electrolyte with pH 1.5, containing 0.1M tartaric acid, 0.005M $CuSO_4 \cdot 5H_2O$, 0.01M $ZnSO_4 \cdot 7H_2O$, 0.01M $SnCl_4$ and 0.005M $NaHSeO_3$, all of analytically pure grade. The electrolysis was performed at the constant potential of –0.6 V for 30 min at room temperature.

The as-deposited precursors were annealed preliminary at 450°C for 30 min in Ar atmosphere for better crystallization. Then the precursors were reacted to Se(S)/N_2 in a tubular furnace under N_2 pressure of 0.8–1.0 mbar inside a partly closed graphite container. Sulfurization was conducted at 500ºC and selenization at 550ºC for the period 2 hours with subsequent natural quench down to room temperature.

Investigation Methods. The phase composition and crystalline structure of the deposited layers were studied by X-ray analysis (XRD) with a D8 Advance (Bruker AXS) diffractometer operating with CuK_α radiation (λ = 0.1542 nm) filtered by a multilayered Ni/graphite monochromator. The voltage supplied to the tube was 40 kV, and the current was 40 mA. X-ray diffraction (XRD) patterns were obtained at grazing incidence (GID) in the scanning mode of measurements with a step of 0.04° (on the 2θ scale) and a counting time of 5 s. The angle of incidence of the primary beam was ω = 0.5°. The phases were identified by comparing the experimentally determined interplanar spacing *d* with the data of the Joint Council for Power Diffraction Standards (JCPDS), 2013.

The chemical composition of the films was determined using an INCA 350 (Oxford Instruments, UK) energy-dispersive X-ray (EDX) spectral microanalyzer with a resolution of 1 μm^3 and a sensitivity of 0.1 at.%.

The surface topography of the precursors and $Cu_2ZnSn(S_xSe_{1-x})_4$ layers was studied using a SOLVER Pro 47 (NT-MDT) high resolution atomic-force microscope (AFM).

The spectral dependences of reflectance of the CZTSSe films were carried out using a spectrophotometer Proscan MC 122. Measurements were carried out for incident angles between 20º and 45º in the spectral range from 300 to 2000 nm with a resolution of 3 nm. The Raman (Raman scattering - RS) spectra were recorded with a Nanofinder High End spectrometer. For the excitation source, we used a solid laser emitting at a wave length of 473 nm. All of the measurements were conducted at a temperature of T = 293 K in the backscattering geometry of recording in the spectral range from 150 to 3000 cm^{-1}.

Results and discussion. ***As deposited precursors*****.** The morphological changes of the precursors before and after pre-heating in Ar atmosphere were analyzed by AFM. It revealed significant differences in compactness and flatness of their surface after the preheating. The as deposited precursors form a rough surface without pinholes and microcracks. However, the films adhered well to the SLG substrate and were relatively dense. AFM measurements showed that the voids and uneven surface morphology were reduced when the precursor were preheated at 450°C for 30 min. Root mean square (RMS) of the precursor films surface roughness decreased from 110 nm (for the as-deposited precursor film) to 60 nm for preheated.

It indicates that this stage plays essential role into transformation of the precursor into well mixed alloy and in controlling the morphology of thin film. On the other hand, XRD characterisation of pre-heated precursors showed mainly amorphous material with traces of CZTS (or CZTSe) phases and other phases without exact indicating (XRD pattern not shown). Precursors used for selenization experiments had the composition ratio Cu/(Zn+Sn) ≈ 0.6 and content of C_S = 44.5 at.% while precursors used for sulfurization experiments had the composition ratio Cu/(Zn+Sn) ≈ 0.87 and content of C_{Se} = 25.94 at.%.

Annealed films**.** It was found that the phases and the structure of the synthesized CZTSSe thin films to be strongly dependent on their composition as well as processing regimes.

The element composition of the precursors annealed in S/N_2 (or Se/N_2) atmosphere is shown in Table. During the selenization process sulfur in the precursors is substituted for selenium leading to S/Se ≈ 0.23 whereas during the sulfurisation process selenium is substituted for sulfur leading to S/(Se) ≈ 29.84. The values of ratio S/Se of the samples series *a* approach to the quaternary compound $Cu_2ZnSnSe_2$, while ratio S/Se of the samples series *b* approach to the quaternary compound Cu_2ZnSnS_2. It necessary to mention that the samples series *a* are Cu-rich and reveal Sn and Zn loss when compared with the initial precursor composition due to volatility of Sn and Sn-Se, Zn and Zn-Se phases during selenization process. The samples series *b* obtained by sulfurization are Cu-poor and Zn-rich. Both series have great deviation from stoichiometry in cation as well an anion sublattice.

CZTSSe thin films chemical composition determined by EDX after annealing in S/N_2 or Se/N_2 atmosphere

Series		Cu, at.%	Zn, at.%	Sn, at.%	S, at.%	Se, at. %	S/Se	Cu/(Zn+Sn)
a	1*a*	24.52	3.6	27.25	3.96	16.63	0.23	0.79
	2*a*	32.3	4.7	35.9	5.2	21.9	0.24	0.79
b	3*b*	11.91	26.27	5.99	41.18	1.38	29.84	0.36
	4*b*	13.7	25.98	7.30	47.5	1.6	29.68	0.41

The XRD pattern of the Cu-Zn-Sn-S precursor annealed in Se/N_2 atmosphere (series *a*) measured in GID mode are shown in Fig.1.

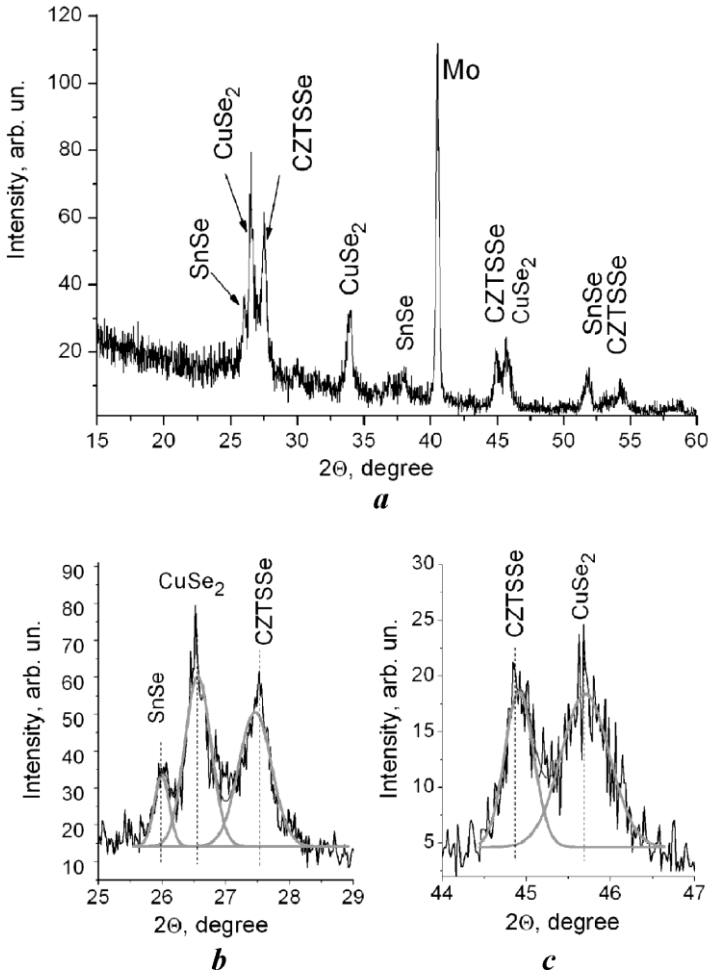

Fig.1. XRD pattern of CZTSSe films series *a* with ratio S/Se ≈ 0.23 (*a*). The inserts (*b*) and (*c*) presents fragments of the XRD pattern approximated by the Gaussian function in the 2θ-region of 14–34 and 44–47, respectively

The existence of the polycrystalline-kesterite CZTSSe phase close to CZTSe (JCDD data file # 96-153-1984) is fixed in the pattern by its main reflections at 27.49º, 45.25 º and 53.53 º and other peaks of very low intensity. In addition to the reflection of Mo at 40.27° minor peaks were registered, which coincide or lie very close to those of CZTSe and can be attributed to selenides. Detailed analysis of the samples series (*a*) (where Cu- and Sn are in excess) indicated on the possible presence of secondary SnSe and $CuSe_2$ phases (JCDD data file # 00-01-0984 and # 00-071-004, consequently) (Fig.1, *b*,*c*). Due to replacement S to Se and forming CZTSSe solid solutions the (112) peak of CZTS phase is shifted from 26.98º to 27.10°.

Fig. 2 shows XRD pattern of the Cu-Zn-Sn-Se precursor after sulfurization (series *b*). It was found that all dominant diffraction peaks of this film can be attributed to the peaks of tetragonal structure for CZTS: (112), (020/004), (132/116) at 28.4º, 33.05º, 47.39 º and 56.15 º (JCDD data file # 96-900-4751). As it can be seen from XRD fragments low intensity peaks can be attributed to minor sulfide phases CuS (JCDD data file # 00-085-39280) and ZnS (JCDD data file # 00-002-0564). The CZTS phase presents twin peaks 220/204 and 132/116 at 47.29 º and 56.03º, consequently which confirm the tetragonal structure existence.

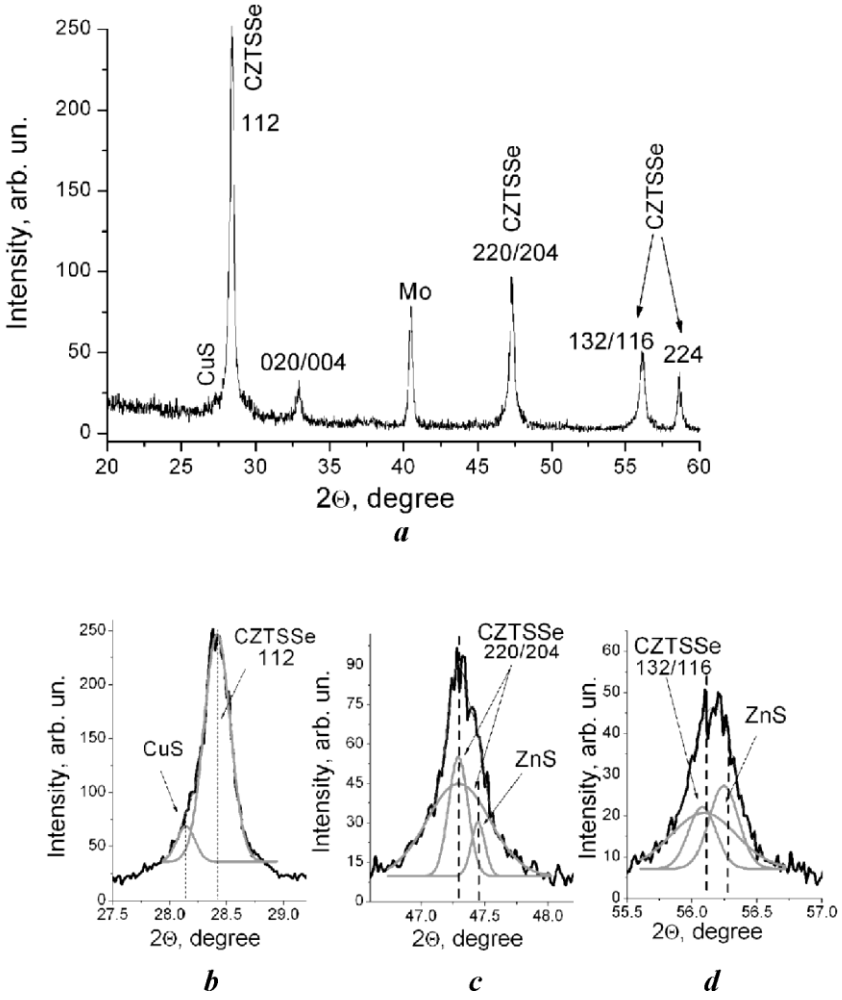

Fig.2. XRD patterns of CZTSSe films series *b* with ratio S/Se ≈ 29.84 (*a*). The inserts (*b–d*) presents fragments of the XRD pattern approximated by the Gaussian function in the 2θ-region of 27.5–29, 30–48.2 and 55.5–57, respectively

All peaks of CZTS phase shifted to larger angles indicating a decrease in the lattice parameters due to the replacement of Se with S. Taking into account these data it may be concluded that the latter film was nearly single-phase and crystallized in partially disordered kesterite structure. However, it is impossible to determine uniquely crystal structure of CZTSSe films based on XRD data only. To confirm this assumption Raman spectra of grown films were analyzed. All phases present in a solid mixture with CZTSe should be resolved by RS.

Typical RS spectrum of CZTSSe thin films series (*a*) is presented in Fig.3. In the spectral range of 180–280 cm^{-1} up to five Raman bands were detected at frequencies of about 130, 179, 198, 205 and 234 cm^{-1}. Three components 179, 198 and 234 cm^{-1} can be attributed to $Cu_2ZnSnSe_2$ phase. The major mode at 198 cm^{-1} can be attributed to either the A_1 mode of KS (kesterite) at 193.01 cm^{-1} or the B (TO LO) of KS at 193.22 cm^{-1} or A_1 mode of ST (stannite) 194.6 cm^{-1}. The peak at 179 cm^{-1} can be attributed to the mode A_2 of ST (184.50 cm^{-1}) composed of the vibration of Se anions [5,6]. The peak at 234 cm^{-1} correspond to the vibra-

tions of symmetry E (KS) (233.02 cm^{-1}) generated by the anion and cation vibrations of $Cu_2ZnSnSe_2$.

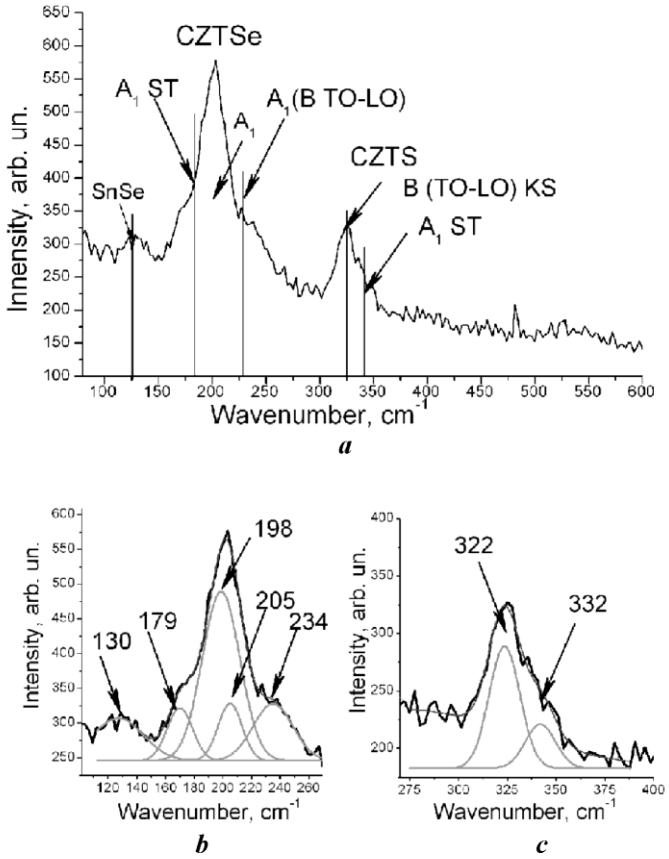

Fig.3. Raman spectrum of the CZTSSe thin film with S/Se ≈ 0.23 (series *a*) (*a*). The inserts (*b*) and (*c*) presents fragments of the Raman spectrum approximated by the Gaussian function in the wavenumber region of 100–270 and 275–400 cm^{-1}

The position of the dominant A_1 mode is shifted by 3 cm^{-1} to long-wave region from the value to theoretically predicted ordered kesterite $Cu_2ZnSnSe_2$ due to S→Se anion substitution and disordering of crystal structure [6]. Considerable broadening of the 198 cm^{-1} mode indicates on the possible presence of binary SnSe and CuSe phases. The low intensity peak visible at 130 cm^{-1} can be attributed to SnSe phase expected from the composition of the films series (*a*) [7]. However, there is no clear resolved peak in the region 260–270 cm^{-1} where vibration mode of CuSe phase should be detected [8]. It may be suggested that this phase statistically distributes in the film material and there is no it's local conglomerates in the mixture.

The appearance of intensive modes in the long-wave region 300–400 cm^{-1}, generated mainly by the S anion vibrations, confirms the forming of CZTSSe solid solutions with two-mode character of vibrations and justifies by other researches [9]. The phonon states at 322 cm^{-1} and 332 cm^{-1} are mainly a result of vibration of symmetry B (TO-LO) KS (309.56 and 313.19 cm^{-1}) and A_1 ST (334.08 cm^{-1}) of Cu_2ZnSnS_4 compound [5].

Fig. 4 presents RS spectrum of the CZTSSe thin film with S/Se ≈ 29.84 (series *b*). A critical effect of annealing in S/N_2 atmosphere is clearly observed. In this case the spectrum is similar to the ordered CZTS kesterite. Three phonon modes appear in the spectral range at 200–400 cm^{-1} with frequencies at 259, 284 and 332 cm^{-1}. Phonon states in the range

at 250–300 cm^{-1} are the result of vibration of the Zn cations and S anions with some contribution from the Cu cations.

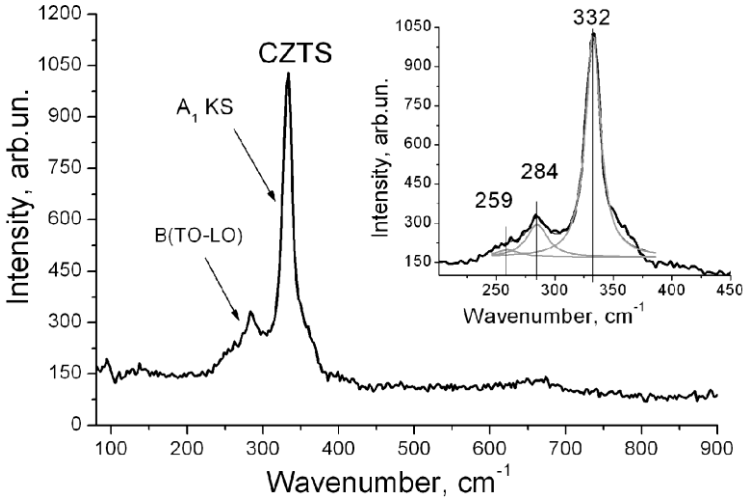

Fig.4. Raman spectrum of the CZTSSe thin film with ratio S/Se ≈ 29.84. The inset present fragments of the Raman spectrum approximated by the Gaussian function in the wavenumber region of 200–450 cm^{-1}

The main band at 332 cm^{-1} observed in Fig.4,*b* is supposed to be A-symmetry of KS CZTS or A_1-symmetry of ST [5]. Therefore, it is assumed that the films contain both KS and ST phases. A small shift of the A-peak with respect to more typical value of 338 cm^{-1} can be induced by disordering of crystal structure.

On the other hand, in the RS spectrum shown above (series *b*) there are no visible bands which might be associated with other phases. The analysis of the Raman data suggests that sulfurization of Sn-Cu-Zn-Se precursors leads to the formation of CZTS phase with quasi-ordered kesterite structure. The obtained result is explained by the high reactivity of elemental

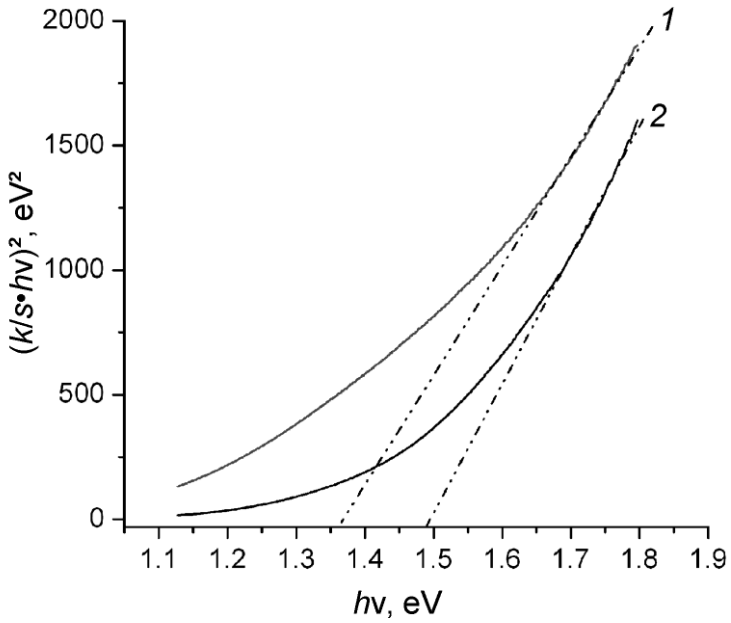

Fig.5. Dependency $[(k/s \cdot h\nu)]^2$ vs $h\nu$ of the sample 3*b* (curve *1*) and 4*b* (curve *2*)

sulfur at temperature of 500°C resulting in near complete S→Se substitution and formation of CZTS phase with very low Se content.

To approve this assumption band-gap value of grown films was evaluated from the measurements of reflectance spectra using the Kubelka-Munk (K-M) theory [10]. This theory provides the theoretical descriptions of diffuse reflectance spectroscopy. The K-M method is based on the following equation:

$$\frac{k}{s} = \frac{(1-R_\infty)^2}{2R_\infty} = f(R_1),$$

where R_∞ is the reflectance; k – absorption coefficient and s – scattering coefficient.

From the plot $[(k/s \cdot hv)]^2$ vs hv (Fig. 5), the band gap value of the CZTSSe films material was estimated as 1.36 and 1.48 eV for sample 3*b* (S/Se ≈ 29.68) and 4*b* (S/(Se) ≈ 29.84), consequently. It is seen that band gap energies are close to the band gap value of ternary CZTS compound and match well to the film compositions of series *b*. It should be noted that the correct estimation band gap value for the samples series *a* is not possible due to great deviation their composition from stoichiometry.

Conclusions. The dependence of the structural properties of CZTSSe thin films gown by selenization/(sulfurizatuion) of electrodeposited Cu-Zn-Sn-S(Se) precursors on the S→Se and Se→S substitution has been studied. It was detected that in the process of annealing there is a big loss of Sn and Zn when compared with the initial precursor composition. RS measurements revealed disordered kesterite structure containing both KS and ST phases of grown CZTSSe films. Depending on overall S and Se contents CZTSSe thin films exhibited a shift in band gap from 1.36 to 1.48 eV.

This work demonstrates that it is possible to grow the CZTSSe absorber layer by suggested approach which can be improved by using a more accurate composition control of the precursors and temperature of annealing.

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Information about the authors:

Ellen P. Zaretskaya – Cand. Sci. (Phys.-Math.), Leading Researcher, State Scientific and Production Association «Scientific-Practical Materials Research Centre of the National Academy of Sciences of Belarus» (Belarus, 220072, Minsk, 19 P. Brovki str., 19), ezaret@ifttp.bas-net.by

Valery F. Gremenok – Dr. Sci. (Phys.-Math.), Prof., Head of The Laboratory of Solid State Physics, State Scientific and Production Association «Scientific-Practical Materials Research Centre of the National Academy of Sciences of Belarus» (Belarus, 220072, Minsk, 19 P. Brovki str., 19), gremenok@ifttp.bas-net.by

Aliona V. Stanchik – PhD student, junior researcher, State Scientific and Production Association «Scientific-Practical Materials Research Centre of the National Academy of Sciences of Belarus» (Belarus, 220072, Minsk, 19 P. Brovki str., 19), alena.stanchik@bk.ru

Aleksandr N. Pyatlitski – Cand. Sci. (Phys.-Math.), Director, JSC «INTEGRAL» – «INTEGRAL» Holding Managing Company (Belarus, 220108, Minsk, Korzhenevskogo str., 12), office@bms.by

Vitaly A. Solodukha – Cand. Sci. (Phys.-Math.), Director, «INTEGRAL» Holding Managing Company (Belarus, 220108, Minsk, Korzhenevskogo str., 12), VSaladukha@integral.by

Kazhmukhan A. Urazov – master of science, Senior Researcher, Sokolsky Institute of Fuel, Catalysis and Electrochemistry (Kazakhstan, 050010, Almaty, Kunaev str., 142), u_kazhm@mail.ru

Margarita B. Dergacheva – Dr. Sci. (Chem.), Prof., Chief researcher, Sokolsky Institute of Fuel, Catalysis and Electrochemistry (Kazakhstan, 050010, Almaty, Kunaev str., 142), m_dergacheva@mail.ru

Süleyman Özçelik – Dr. Sci. (Phys.-Math.), Prof., Director, Gazi University, Photonics Application & Research Center, Central Campus D-Blok (Ankara, Turkey), sozcelik@gazi.edu.tr

Nikolay A. Dyuzhev – Cand. Sci. (Phys.-Math.), Head of Shared Facilities Center «MST&ECB», National Research University of Electronic Technology (Russia, 124498, Moscow, Zelenograd, Shokin sq., 1), dyuzhev@unicm.ru

Alexander M. Alekseev – Cand. Sci. (Phys.-Math.), Senior Engineer, Shared Facilities Center «MST&ECB», National Research University of Electronic Technology (Russia, 124498, Moscow, Zelenograd, Shokin sq., 1), alekseev@ntc-nmst.ru