FEATURES OF PHOTOVOLTAIC AND OPTICAL PROPERTIES OF CuInSe$_2$ FILMS PREPARED BY FLASH EVAPORATION[3]

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The morphology and the optical and photovoltaic properties are investigated for CuInSe$_2$ films prepared by flash evaporation. The change of a form of crystallites and the magnitude and spectral dependences of the photovoltage and additional absorption (as compared to the absorption spectra of CuInSe$_2$ single-crystal) on the substrate temperature are determined. These features are explained in fact by the formation of impurities of various selenides. The efficiency of the formation of these impurities depends on the substrate temperature. The increase of the photovoltage with the substrate temperature up to 720–770 K reaches three orders. This is important and can be used in the development of photosensitive structures based on CuInSe$_2$.

1. Introduction

The ternary compound CuInSe$_2$ (CISe) is one of the most widely used materials in the production of high-performance solar cells [1,2]. In 2005, the efficiency of 19.3 % [2] has been achieved for Cu(In,Ga)Se$_2$ thin-film solar cells. To prepare photosensitive CISe thin-film structures, a complex expensive technology is used. Therefore, the current topical research task is the development of cheaper and simpler techniques in the production of photosensitive CISe thin layers.

One of the perspective procedures in the preparation of CISe films is the method of flash evaporation [3—6]. The properties of the films obtained by this method depend mainly on the evaporator (T_e) and substrate (T_s) temperatures. Homogeneous and compact CISe films being adhesive to a substrate were prepared at $T_e = 1500 \div 1700$ K [7—10].

The data obtained by different authors on the influence of T_s on the properties of CISe films are very different, but they allow us to select two regularities:

CISe films are mainly amorphous at $T_s = 400 \div 600$ K [6,8—10], and they are polycrystalline at $T_s = 500 \div 800$ K [6—8,11].

CISe films reveal the excess of In at $T_s < 650$ K and that of Cu at $T_s > 700$ K [11].

Based on the literature sources, we failed to determine the optimal T_s for the preparation of photosensitive CISe films. Moreover, we found no data on the photovoltaic properties of flash-evaporated CISe films. Therefore, the aim of the present work is the research of the photovoltaic properties of CISe films and the influence of technology parameters on the photosensitivity of these films.

2. Technology of Films Preparation and Experimental Technique

CISe thin films were prepared by flash evaporation in vacuum (7×10^{-3} Pa) by the following procedure: a substrate holder and an evaporator were located in a quartz cartridge (18 cm^3 in volume) with the heatset purpose. A tantalum tape was used as the evaporator. We held T_e to be 1520 K, and T_s was varied in the range 525–769 K. T_e and T_s were maintained constant using a heat regulator during the preparation of films. The evaporator-substrate distance was 6 cm. To heat substrates, we used a graphite block with tungsten spiral. The pounded CISe crystals with 60–110$-\mu$m granule sizes were located in a bunker that was supplied by electromagnetic impulses (0.2–3.0 Hz). An impulse pulled the needle that opened the aperture, through which the material fell on the evaporator.

We studied the influence of the deposition growth rate on the properties of the films. To this end, CISe

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films were prepared at growth rates of 0.1, 0.2, and 0.4 μm/min and $T_s = 721$ K. Then their absorption spectra were measured. It was established that the maximal relative difference of the values of absorption at the energy of photons ($h\nu$) of 1.5 eV was about 0.1 at the variable growth rate. But this difference becomes about 0.55 (by 5 times greater) under the increase of T_s by 50 K (from 672 to 721 K). According to the above results, we considered that the growth rate is a less effective parameter than T_s. Consequently, the growth rate weakly affects the photovoltage of the films. Therefore, the influence of a growth rate on the properties of CISe films isn't discussed in this article.

The structure and morphology of the surface of films were studied by Atomic Force Microscopy (AFM). The AFM measurements were made by a silicon probe on a serial "Nanoscope IIIa" (DI, USA) microscope in the periodic contact mode (tapping mode^{TM}). The probe spike had the nominal 10-nm curvature radius. The optical properties of films were investigated by using a double-beam "HitachiM356" spectrophotometer.

The photovoltaic properties were investigated by the Bergman technique (the static capacitor method) modified and improved by Akimov [12,13]. According to this technique, the researched sample was deposited on a polished quartz substrate with a transparent and conductive indium tin oxide (ITO) layer. The sample was isolated by a transparent dielectric 20-μm spacer from the other electrode, a similar ITO layer on the other identical substrate. The difference of potentials (the capacitor photovoltage) arose between the surfaces of a sample under its illumination by light pulses during a time interval τ. As a result, the ac voltage appeared at a load resistance that was registered by the measuring equipment.

The measurements of photovoltage spectra ($\phi(h\nu)$) were carried out in the following way. The light from an iodinated tungsten lamp ("Hitachi") of 120 W in power fell through the focusing system of two quartz lenses, the modulator (80-Hz modulation frequency), and a corresponding optical filter into a monochromator MDR-4 (LOMO). The monochromatic light was focused on the window of a measuring cell, the sample being placed at its center.

The photovoltage was measured by a phase-sensitive selective "Unipan-232B" nanovoltmeter with a high-resistance "Unipan-233-7" preamplifier. For the synchronous detection, the reference signal was used from the photon-coupled pair consisting of a light-emitting diode (LED) and a photodiode placed at the modulator. The signal was supplied from a nanovoltmeter through an ET-1050 interface card to a computer. After each measurement, the spectral distribution of the light power at the monochromator output was measured by a pyroreceiver mounted at the sample place. The measured photovoltage was recalculated per the same number of incident and absorbed photons.

3. Results

3.1. Structure and morphology of the films

The prepared CISe films possessed a good adhesion and the smooth surface. The measurements of the surface morphology of films prepared at different T_s have shown that the films at $T_s = 528 \div 571$ K consist of great (≈ 100 nm) oval crystallites with clear edges against the background of a microcrystalline (< 30 nm) phase. With increase in T_s, the surface and height Δh of crystallites decrease by about 3 times, while the edges become fuzzy. The surface of films is most homogeneous at $T_s = 621 \div 667$ K. However, at $T_s > 721$ K, the great cone-shaped crystals with clear edges appear against the microcrystalline phase background.

These changes in the shape and size of crystallites can be conditioned by changes of both the structure and composition of CISe films with change in T_s. At that, the oval and cone-shaped crystallites are formed on the surface of films, respectively, at lower T_s and higher T_s against the microcrystalline phase background.

3.2. Absorption

The spectral dependences of the absorption coefficient ($\alpha(h\nu)$) of CISe films at different T_s are shown in Fig. 1. For comparison, α $(h\nu)$ of a CISe film at $T_s = 723$ K and $T_e = 1673$ K [7] and $\alpha(h\nu)_{\rm sc}$ of single crystals for the unpolarized illumination [14] are given in Fig. 1 (curves 5 and 6).

The absorbance of the films is greater than $\alpha(h\nu)_{\rm sc}$, especially in the range 1.2–2.8 eV. The maximal difference $\Delta\alpha(h\nu) = \alpha(h\nu) - \alpha(h\nu)_{\rm sc}$ is observed in the (1.5–1.6)-eV and 2.6-eV regions (Fig. 2).

The absorption coefficient $\Delta\alpha(h\nu)$ decreases with increase in T_s from 528 to 721 K at the practically constant maximum position of $\Delta\alpha(h\nu)$ (Fig. 3, curves *1–3*). At $T_s = 667 \div 721$ K, $\Delta\alpha$ are minimal (Fig. 2) like those for CISe films [7] at $T_s = 723$ K (Fig. 2, curve *5*). We should like to indicate the different dependences of $\Delta\alpha$ for different $h\nu$ on the temperature in the region

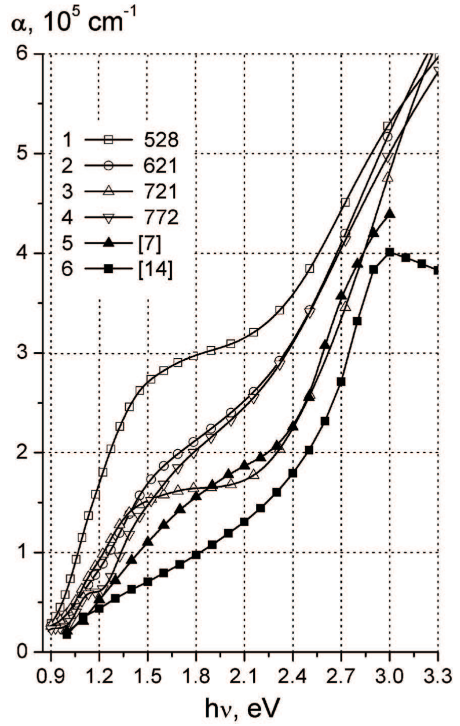


Fig. 1. Spectral dependences of the absorption coefficients $\alpha(h\nu)$ of CISe thin films at different T_s (curves 1–4) and the literature data for films (curve *5*) [7] and single crystals under unpolarized illumination (curve *6*) [14]

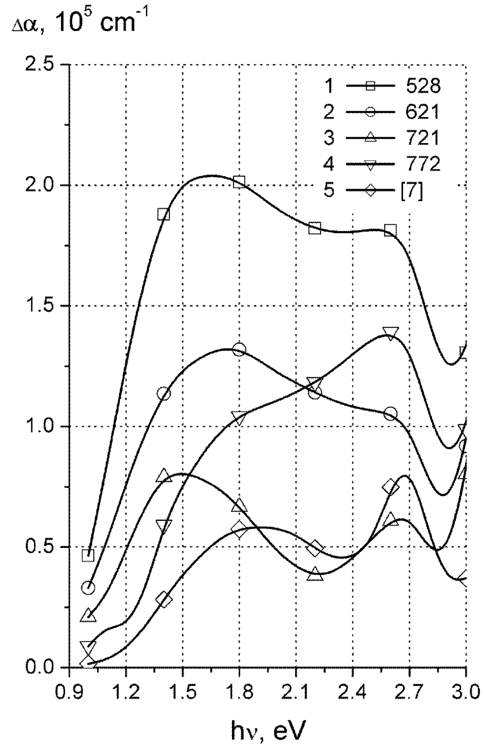


Fig. 2. Spectral dependences of the difference of the absorption coefficients $\Delta\alpha$ CISe thin films at different T_s (curves 1-4) and the data from [7] (curve 5) relative to that of a single crystal [14]

near T_s = 772 K: $\Delta\alpha$ increases in the range 2.0–2.8 eV and decreases in the range 1.0–1.5 eV.

3.3. Photovoltage

The photovoltage of ITO/CISe structures was very low at $T_s = 528\div571$ K, and we failed to measure its spectral dependence. Therefore, the measurements of the photovoltage were made under the LED's illumination, with the purpose to investigate the influence of T_s on the photovoltaic properties of CISe films in the wide range of T_s. For this reason, we applied the illumination from a collection of serial LEDs with different $h\nu$ (1.3, 1.9, 2.18, and 2.64 eV).

The photovoltage of the ITO/CISe structure upon the ITO contact illumination (ϕ_c) is greater than that upon the free surface illumination (ϕ_s). The ϕ_s/ϕ_c ratio increases with T_s independently on $h\nu$. In this case, the $\phi_c(T_s)$ and $\phi_s(T_s)$ dependences are close. The photovoltage rises with T_s for $h\nu = 2.18$ and 2.64 eV, but we observe the maximum of the photovoltage at 721 K for $h\nu = 1.34$ eV (Fig. 3). The peaks at 1.04, 1.26 and 2.4 eV are clearly observed on the spectral dependences $\phi_s(h\nu)$ at $T_s = 667\div721$ K and additional maxima in the range 1.4–1.6 and 2.0–2.2 eV are appeared at T_s = 772 K (Fig. 4). The appearance of these photovoltage maxima can be conditioned on the formation of impurities of a new photosensitive phase that does not present clearly in the absorption spectra. The similar spectra were obtained for $\phi_c(h\nu)$. We compared the relative changes of $\phi_s(h\nu)$ and $\phi_c(h\nu)$ ($\Delta\phi/\phi(h\nu)$) with increase in T_s from 667 to 772 K (Fig. 5). The content of additional compounds responsible for the appearance of the maxima at 1.26 and 1.8–2.2 eV at T_s = 772 K is shown to be higher at the free surface of the films. But the content related to the maximum at 1.04 eV is higher, on the contrary, at the ITO contact.

4. Discussion

The AFM topologies of CISe thin films at different T_s evidence for two types of the additional crystallization. The first type (the formation of oval crystallites) dominates at $T_s < 621$ K, and the second one (the

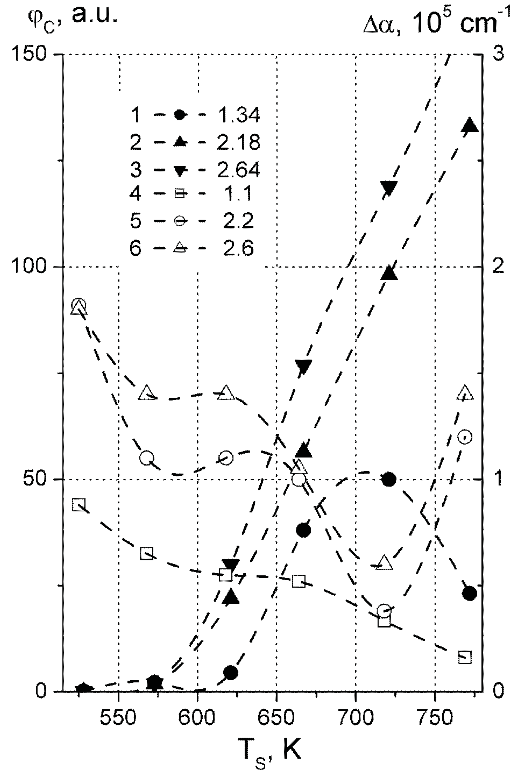


Fig. 3. Photovoltage of ITO/CISe structures upon the ITO contact illumination, ϕ_c, (*1–3*) and difference of the additional absorption, $\Delta\alpha$, of CISe thin films relative to that of a single crystal (*4–6*) vs T_s at different quantum energies: 1.32 (*1*), 2.18 (*2*), 2.64 (*3*), 1.1 (*4*), 2.2 (*5*) and 2.6 (*6*) eV

formation of cone-shaped crystallites) dominates at $T_s > 721$ K. This can indicate the formation of different additional phases of selenides in CISe films at $T_s < 621$ K and $T_s > 721$ K.

The additional absorption (Fig. 2) can be associated with both the appearance of optical effects, diffraction of light dispersed by crystallites [15,16], and the formation of additional phases of selenides that absorb in the range 1.2–2.7 eV. The optical effects can be seen in the spectral region of the essential selective difference of α under illumination at different polarizations (consequently, the difference of the refraction indices Δn) [15,16]. In accordance to [14], Δn is small for $h\nu < 2.7$ eV, and the relative difference of reflections $(R_\perp - -R_\parallel)/(R_\perp + R_\parallel)$ « 1 for a CISe single crystal. Therefore, we consider that the displaying of optical effects in the films seems to be unlikely. In addition, the optical effects cannot result in a strong change of the dependence of the photovoltage on T_s, what was experimentally observed (Fig. 3). According to the above-presented consideration, we will make the detailed analysis of the formation of additional phases of various selenides in the CISe films under study.

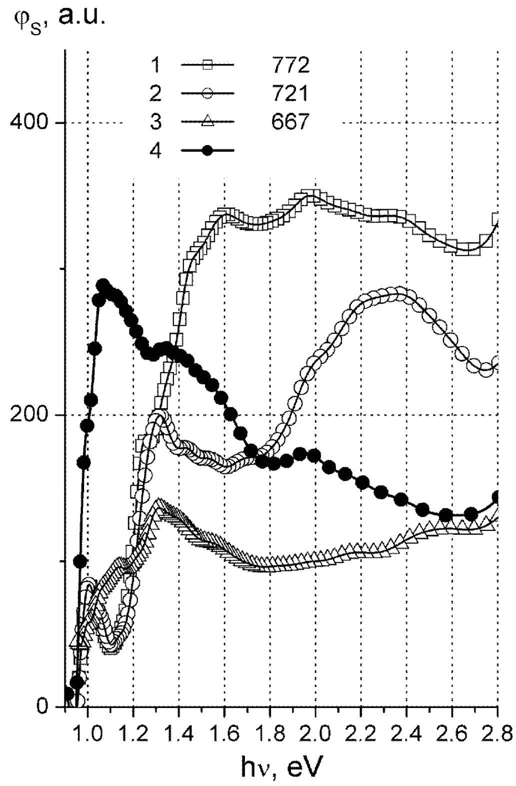


Fig. 4. Spectral dependences of the photovoltage of ITO/CISe structures, $\phi_s(h\nu)$, at $T_s = 772$ (*1*), 721 (*2*) 667 (*3*) K (normalized on the absorbed energy) and a CISe single crystal (*4*)

According to calculations [14], $\alpha(h\nu)$ of CISe single crystals in the range 0.9–3.4 eV consists of 5 electron transitions: $\Gamma_{4V} \rightarrow \Gamma_{1C}$ ($\Gamma_{5V} \rightarrow \Gamma_{1C}$) at $E_0(A,B)$=1.04 eV, $\Gamma_{3V} \rightarrow \Gamma_{1C}$ at $E_{01}(C)$=1.274 eV (from a subband of the valence band at the depth Δ_{SO}=0.235 eV – the spin-orbit interaction parameter), $\Gamma_{5V} \rightarrow \Gamma_{3C}$ at $E(\Gamma X)$=2.4÷2.5 eV, $N_{1V} \rightarrow N_{1C}$ at $E_1(A)$=2.82÷2.92 eV, and $\Gamma_{5V} \rightarrow \Gamma_{1C}$ at $E(X\Gamma)$=3.174÷3.24 eV. However, two maxima at 1.04 and 2.9 eV are displayed in $\alpha(h\nu)_{\mathrm{sc}}$ (Fig. 1, curve *6*) only, i.e. the predominant contribution to the absorption of single crystals is given by the transitions $\Gamma_{4V} \rightarrow \Gamma_{1C}$ ($\Gamma_{5V} \rightarrow \Gamma_{1C}$) and $N_{1V} \rightarrow N_{1C}$ [14]. The contribution of the other transitions (at 1.27, 2.4, and 3.2 eV) is small. These transitions are revealed only as weak shoulders (Fig. 1).

According to the previous evidence, the synthesis of ternary compounds of Cu, In, and Se with the stoichiometric composition can be accompanied by the formation of $CuInSe_2$ (the band gap energy $E_g = 1.04$ eV [1,6,7,14,17,18]), $CuIn_3Se_5$ (E_g = 1.24÷1.26 eV [18,19]), In selenide ($E_g(\mathrm{In_2Se_3}) = 1.15 \div 1.2$ eV [20,21]), and $\mathrm{Cu}_x\mathrm{Se}$ ($E_g = 1.4$ and 2.2 eV [6], and α at $h\nu = 2.2$ eV is several times greater than α at $h\nu = 1.4$ eV) compounds.

The formation of films at $T_s = 721 \div 772$ K can occur in the following way: $5\mathrm{Cu}+5\mathrm{In}+10\mathrm{Se} \rightarrow \mathrm{In_2Se_3}+\mathrm{CuIn_3Se_5}+2\mathrm{Cu_2Se}$ [22]. Naturally, the formation efficiency for each compound differently depends on T_s.

For the identification of additional phases in the films, we have estimated the value of E_g by using $\alpha^2(h\nu)$, because the majority of the literature data indicates that the direct optical transitions dominate in selenides [6,7,17] (Fig. 6 *a,b*). The linear interpolation of $\alpha^2(h\nu)$ showed that $\alpha(h\nu)$ consists of several components with the following E_{gi}: 1.02±0.02, 1.11±0.04, 1.22±0.02, 2.12±0.12, and 2.46±0.02 eV (see Table).

In CISe films at $T_s < 667$ K, E_{gi} are clearly determined in the range 1.11±0.04 eV, and oval crystallites are formed, which can be conditioned by the contribution of In selenide compound (most probably, $\mathrm{In_2Se_3}$ [20,21]). This suggestion agrees with the conclusion of [11] about the excess of In in CISe films at $T_s < 650$ K. The content of this phase decreases with increase in T_s from 528 to 667 K, which confirms the decrease of α and $\Delta\alpha$ in the range 1.2–1.55 eV (Figs. 1 and 2). The weak photosensitivity of CISe films at $T_s < 621$ K evidences for the low efficiency for the photogeneration of charge carriers upon the light absorption by the In selenide phase.

The increase of $\Delta\alpha$ in the range 2.2 eV (Fig. 3, curve *6*), the predominance of $E_g = 2.16$ eV (Fig. 6,*b*, curve *3*), the increase of $\phi(h\nu)$ in the range 1.4–2.4 eV with maxima at 1.4–1.6 and 2.1–2.2 eV (Fig. 4, curve *1*) are observed at the rise in T_s from 721 to 772 K. These data correlate with the value of E_g for $\mathrm{Cu}_x\mathrm{Se}$ films ($E_g = 1.4$ and 2.2 eV [6]). Also these facts agree with the evidence for the Cu and Se excess at $T_s > 700$ K [11] and the formation of cone-shaped crystallites at $T_s = 772$ K in the films.

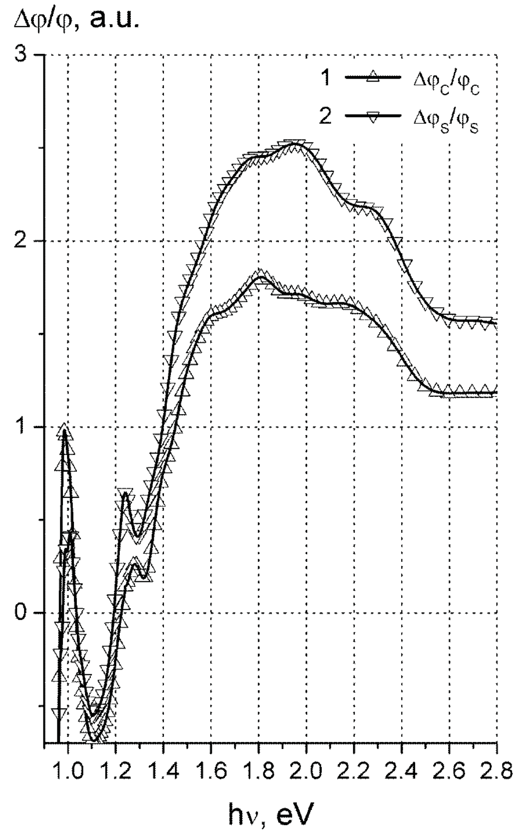
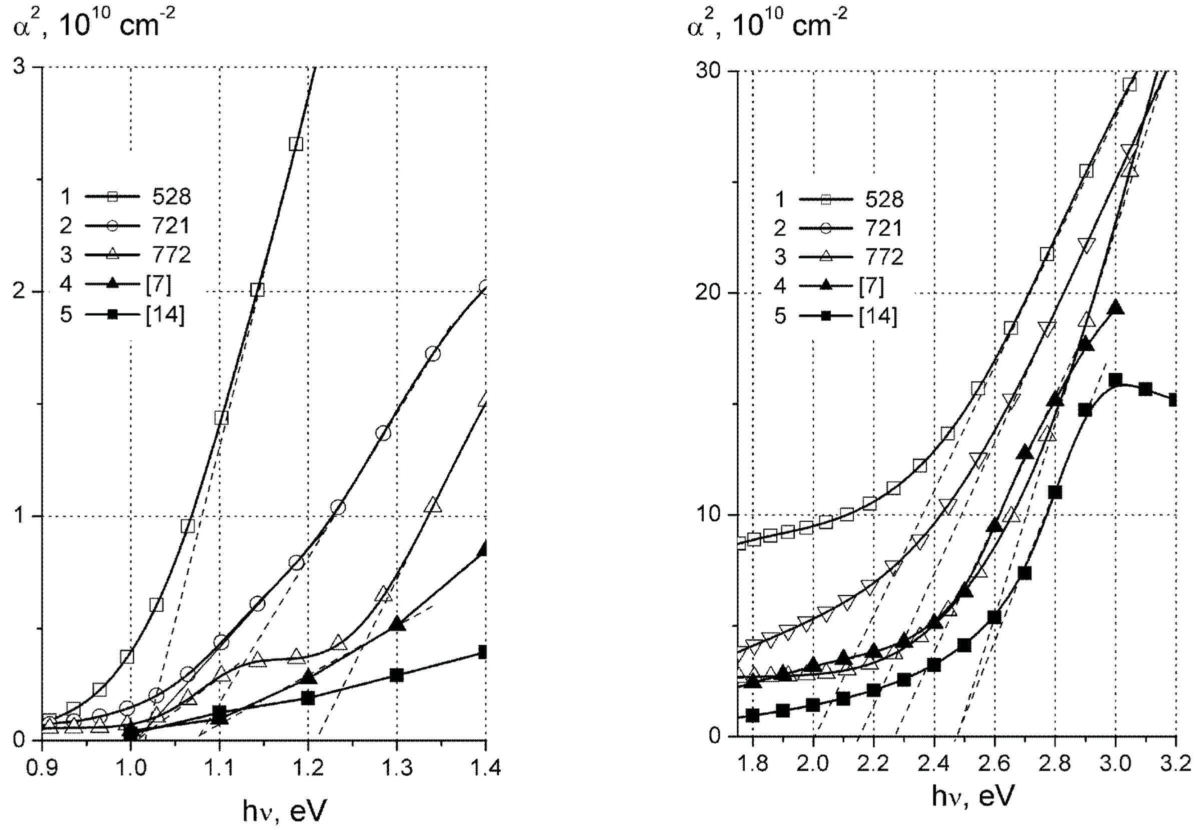


Fig. 5. Spectral dependences of the relative change of the photovoltage of ITO/CISe structures, $\Delta\phi/\phi(h\nu)$, when T_s increases from 667 to 772 K upon the illumination of the ITO contact (1) and the free surface (2)

The conclusions that the content of CISe films is strongly changed at temperatures more than 770 K and the $\mathrm{Cu_2Se}$ phase starts to form at this temperature have been made by using the method of TPD (Temperature Programmed Desorption) [23] and X-ray diffraction spectroscopy [24]. Hence, in CISe thin films at $T_s = 772$ K, $\mathrm{Cu}_x\mathrm{Se}$ crystallites can essentially contribute to the photogeneration of charge carriers and the photovoltage.

Band gap energies of CISe films that are estimated by using a linear interpolation of $\alpha^2(h\nu)$ at different T_s and the energy range of their manifestation ($\Delta h\nu$)

T_s, K	528	571	621	667	721	772	Single crystals [14]
E_{g1}, eV	1.02	1.02	1.02	1.02	1.04	1.02	1.00
$\Delta h\nu$, eV	1.1–1.4	1.05–1.2	1.05–1.2	1.15–1.45	1.15–1.4	1.05–1.1	1.05–1.45
E_{g2}, eV	–	1.08	1.15	–	–	1.24	–
$\Delta h\nu$, eV	–	1.2–1.45	1.25–1.55	–	–	1.3–1.5	–
E_{g3}, eV	2.00	2.19	2.24	–	–	2.16	–
$\Delta h\nu$, eV	2.65–3.1	2.65–3.1	2.65–3.2	–	–	2.7–3.5	–
E_{g4}, eV	–	–	–	2.45	2.47	–	2.47



a

b

Fig. 6. Spectral dependences of $\alpha^2(h\nu)$ of CISe thin films at different T_s (curves *1–3*) and the literature data for films [7] (curve *4*) and a single crystal [14] (curve *5*) in the ranges 0.9–1.4 eV (*a*) and 1.75–3.2 eV (*b*)

The unexpected intense peaks at 1.26 and 2.4 eV are observed in $\phi(h\nu)$ at T_s = 721 K (Fig. 4, curve *2*). It should be noted that the respective bands aren't displayed in the absorption spectra of both the researched films and a single crystal [14] (Fig. 1). The analysis of the literature data has shown that the peak at 1.26 eV can be associated with the formation of the additional $\mathrm{CuIn_3Se_5}$ phase in the films [18, 19]. The peak at 2.4 eV can be a result of the manifestation of the second electronic transition in $\mathrm{CuIn_3Se_5}$ at the same energy or the drastic increase of the quantum efficiency for the photogeneration of charge carriers under the excitation of the $\Gamma_{5V} \rightarrow \Gamma_{3C}$ $E(\Gamma X)$ transition at 2.4–2.5 eV in the CISe compound. To verify this assumption, we measured $\phi(h\nu)$ of the single crystals used for the preparation of the films to be studied. The comparison of $\phi(h\nu)$ of CISe films prepared at T_s = 721 K (Fig.4, curve *2*) and single crystals (Fig.4, curve *4*) shows that the peak of $\phi(h\nu)$ for single crystals in the range 2.4–2.5 eV isn't displayed. Consequently, the maxima of $\phi(h\nu)$ can be associated with the formation of the additional phase of selenide in the films at the energies of electronic transitions of (1.26±0.02) and (2.4±0.05) eV (most probably, $\mathrm{CuIn_3Se_5}$). Since the maximum at 1.26 eV isn't clearly seen in $\alpha(h\nu)$, the quantum efficiency of the photogeneration of charge carriers in this additional phase is significantly higher than that in the CISe phase. The concentration of this additional phase is lower than that of other additional phases in the films under study.

The comparison of $\Delta\phi_s/\phi_s(h\nu)$ and $\Delta\phi_c/\phi_c(h\nu)$ indicates that the content of CISe on the ITO contact decreases with increase in T_s from 667 to 772 K, while the contents of $\mathrm{CuIn_3Se_5}$ and $\mathrm{Cu_xSe}$ simultaneously increase on the free surface of the films. This agrees with the data on the predominant formation of $\mathrm{CuIn_3Se_5}$ on the surface of CISe films [19,25]. The decrease of the photovoltage difference upon the illumination of the different sides of the films with increase in T_s testifies that the films become more homogeneous in bulk with increase inT_s. So the researched structures at T_s = 720÷770 K were most homogeneous in bulk.

The peaks with maxima at 1.6 and 2.2 eV (they correspond to E_g of Cu selenides) are observed in the photoconductivity spectra of CISe films at greater T_e than the peaks with the maxima at 1.04 and 1.24 eV (they correspond to $CuInSe_2$ and $CuIn_3Se_5$ E_g, respectively) [26]. Moreover, the In_2Se_3 phase isn't displayed in these spectra. The efficiency of the photogeneration of charge carriers for the additional phases In_2Se_3, $CuIn_3Se_5$, and Cu_xSe in the films agrees with the data obtained in [26].

5. Summary

We have first shown that the spectra and magnitude of the photovoltage for the $CuInSe_2$ films prepared by flash evaporation are changed with increase in the substrate temperature (T_s), by reaching their maxima at $T_s =$ 720–770 K. At these T_s, the photovoltage difference upon the illumination of the different sides of the films is minimal; i.e. the films are most homogeneous in bulk.

Simultaneously, with a change in the photovoltage, the form and size of crystallites and the magnitude of the additional absorption (in comparison with that of a single crystal) are changed with increase in T_s. These features can be explained by the change in the content of impurities (phases) of various selenides with the energies of electronic transitions in the range 1.3-2.6 eV (most probably, In_2Se_3, $CuIn_3Se_5$, and Cu_xSe). Moreover, the efficiency of the formation of these impurities depends on T_s.

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ОСОБЛИВОСТІ ФОТОВОЛЬТАЇЧНИХ І ОПТИЧНИХ ВЛАСТИВОСТЕЙ ПЛІВОК $CuInSe_2$, ОТРИМАНИХ МЕТОДОМ ІМПУЛЬСНОГО НАПИЛЕННЯ

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Резюме

Досліджено морфологію, оптичні та фотовольтаїчні властивості полікристалічних плівок $CuInSe_2$, виготовлених методом імпульсного напилення. Встановлено зміни форми кристалітів, величини і спектральної залежності фото-ерс та додаткового поглинання (в порівнянні зі спектрами поглинання монокристалів $CuInSe_2$) в залежності від температури підкладок, на яких вирощено плівки. Ці особливості пояснено формуванням у плівках $CuInSe_2$ домішок інших селенідів, ефективність створення яких залежить від температури підкладок. Зростання фото-ерс з ростом температури підкладок до 720–770 K сягає трьох порядків. Отримані результати можуть бути використані при розробці фоточутливих структур на основі $CuInSe_2$.