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Structural and optical characteristics of ZnSe and CdSe films condensed on non-oriented substrates

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Abstract. In this work, the complex investigation of structural and optical properties of zinc and cadmium selenide semiconductor films deposited by close-spaced vacuum sublimation method using thermal evaporation on non-oriented substrates was carried out. The structural and phase analyses of the layers condensed at different substrate temperatures were performed. The transmission and reflection spectra of the investigated films have been measured and their main optical characteristics have been calculated.

Keywords: cadmium and zinc selenide, X-ray analysis, film structure, transmission and reflection spectra, wide band gap.

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1. Introduction

In recent time, A_2B_6 semiconductors attract attention due to their unique physical properties. Films of these materials can be used to make various device structures [1]. For example, cadmium selenide can be used as an absorption layer, while zinc selenide due to its considerably wide band gap ($E_g = 2.7$ eV) – as a “window” of high-effective tandem solar converters. Moreover, the above materials are already used in optoelectronics and acoustoelectronics as base layers of various irradiation detectors, vidicons, light-emitting diodes (LEDs) of visible and other spectra of irradiation, etc. [1, 2]. However, for practical usage of chalcogenide films in various branches of industry, it is necessary to optimize their optical and electro-physical characteristics that are determined by features of their structure and depend on physical and technological deposition regimes.

In recent years, to obtain zinc and cadmium selenide films, several methods are applied [3-6]. Among them, there are methods of condensation in vacuum owing to the possibility to synthesize layers of semiconductor purity [7-12]. Necessity to produce condensates with controlled characteristics makes an occasion to select of evaporates bi-component compounds by close-spaced vacuum sublimation

(CSVS). This method allows to obtain films in conditions close to thermodynamic equilibrium, when structural perfection of chalcogenide layers should increase [13]. An important feature is that the vapor phase is located in a small volume limited by hot walls. As a result, compound components that constituted at its dissociation are not removed outside this volume, and vapors molecules do not get to condensation volume. It allows to obtain high-purity films of approximately stoichiometric composition [13].

However, structural and optical characteristics of ZnSe and CdSe layers obtained by CSVS method and the influence of their physical and technological condensation regimes were insufficiently investigated. The only works [14-18] were devoted to study these films. But in [15], the authors studied only structural characteristics of ZnSe films obtained at room temperature. More fundamental research was carried out in [14] for films deposited at substrate temperatures 473 to 623 K. Some structural and optical characteristics of CdSe films were investigated in [16]. There these condensates were obtained in the narrow range of substrate temperatures 290 to 490 K. It should be noted that the measurement of lattice constants and sub-structural characteristics of ZnSe and CdSe films was done by the authors along (111) line at small angles that resulted in considerable mistakes in determining these

characteristics. In the works [14-16], a complex approach for studying the film properties is absent.

The main purpose of this work was to obtain polycrystalline ZnSe and CdSe films of semiconductor purity with controlled characteristics, the investigation of condensates structural and optical properties and their optimization to make heterosystems in future for instrumental usage on their base.

2. Experimental details

ZnSe and CdSe films were prepared on well-cleaned glass substrates in vacuum equipment VUP-5M. For their deposition, the CSVS method was used. The device scheme for films synthesis was shown in the work [19]. The evaporator temperature for deposition of ZnSe layers was $T_e = 1073$ K, CdSe – 973 K. Substrate temperatures varied within the range of: ZnSe – $T_s = 373...973$ K, CdSe – $T_s = 373...873$ K. For measuring the optical properties, the films with thickness $l = 0.2...1.1$ μm were used, for investigation of structural characteristics – thicker layers $l = 5...6$ μm . The films thickness was determined via their condensation time.

Structural characteristics of films were examined using the X-ray diffractometer DRON 4-07 in Ni-filtered K_α -radiation of copper anode. The survey was carried out within the range of angles 2θ from 20° up to 80° , where 2θ is the Bragg angle. The Bragg-Brentano method of X-ray radiation focusing was used. Corresponding diffractograms were normalized by the (111) peak intensity of cubic phase or (002) – of hexagonal phase. The phase analysis was carried out using comparison of interplanar spaces and relative intensities of X-ray peaks in the investigated patterns as well as references according to Joint Committee on Powder Diffraction Standards (JCPDS) [20].

Determination of lattice parameters inherent to sphalerite and wurtzite phases of cadmium and zinc selenide was carried out using the condition that K_{01} constituent of all the most intensive lines is presented on diffractograms. Then, to obtain the precise values of the material lattice crystallographic parameters the Nelson-Riley extrapolation method was used [21, 22]. To determine the lattice constant of hexagonal phase, the graphic convergence method was used [22]. Besides, the ratio c/a at the first iteration was accepted as a value typical for ideal lattice of wurtzite $c/a = 1.633$ [23]. Corresponding values c , a , c/a (a/c) obtained after the first iteration were used for new measurements of constants from equations used to determine a and c . This procedure was repeated several times (3 to 5), until the obtained values a , c and c/a become unchanged.

The pole density P_i and orientation factor f were estimated by the method described in [21, 22]. After that, plotted were the dependences $P_i - (hkl)$ and $P_i - \varphi_i$, where φ_i is the angle between the texture axis and normal to different crystallographic planes

corresponding to reflections in diffractograms. Texture axis has indexes that correspond to the higher value of P_i .

Measurements of optical characteristics of condensates were performed using the double-beam scheme of the spectrophotometer SF-26 within the range of wavelengths $\lambda = 300...600$ nm (ZnSe) and $600...900$ nm (CdSe). The spectral dependences of the reflection $R(\lambda)$ and transmission $T(\lambda)$ indexes were surveyed. Irradiation was carried out from the chalcogenide film side. For measuring the spectra $R(\lambda)$, the attachment PZO-2 was used, which provided at the survey a double reflection of light from the experimental pattern surface in comparison with reflection from the control pattern. Determination of the optical constants for these materials was carried out using the software environment Maple 7. The thickness of thin films was measured using the interference method with the MII-4 microscope, thicker films – by the fractography method (photography of the condensate cross-section).

3. Results and discussion

X-ray diffractograms for ZnSe and CdSe films obtained at various substrate temperatures are presented in Fig. 1. It was shown that zinc selenide films have a cubic (sphalerite) structure and cadmium selenide films have hexagonal (wurtzite) structure.

But in CdSe condensates obtained at $T_s = 373...473$ K at the equipment sensitivity boundary near the angle $2\theta \sim 60.90^\circ$, the additional line is observed, which may be interpreted as a reflection from the crystallographic plane (400) of cubic phase. The lattice constant of this phase that was found using this reflection is $a = 0.6075...0.6080$ nm, which well coincides with the value that was shown in the reference book $a = 0.6077$ nm [20]. It indicates the bi-phase condensates of low-temperature CdSe films. Evaluations were carried out by the method expounded in [21, 22], and they showed that the maximum mass fraction of meta-stable phase in films does not exceed 1–2 %.

In some cases, in diffractograms from cubic ZnSe layers the weak reflections were also observed from (101) plane of wurtzite at the angles $29.08^\circ...29.33^\circ$ ($a = 0.3976...0.4012$ nm, $c = 0.6503...0.6558$ nm, reference data are: $a = 0.4003$ nm, $c = 0.6540$ nm [20, 23]). The mass fraction of this phase in films was 1 to 4%. It was increased with the condensation temperature and decreased with the film thickness. Thus, ZnSe films deposited at high substrate temperatures were bi-phase with low wurtzite contribution. The dependence of peak intensity in the wurtzite phase on the film thickness specifies that this phase is located in the intermediate fine-crystalline condensate layer that is formed near the substrate [24]. This conclusion is also applied in full regards to CdSe films. Oxides and other impurity phases in chalcogenide films were not determined by X-ray structural method.

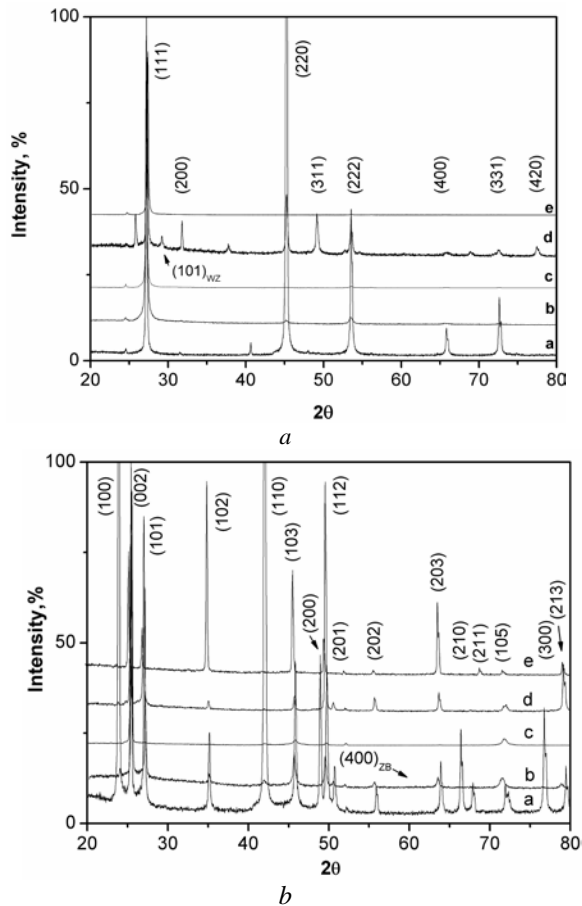


Fig. 1. X-ray diffractograms of ZnSe (a) and CdSe (b) obtained at various substrate temperatures: charge (a), 373 (b), 573 (c), 773 (d), and 873 K (e).

All investigated films of bi-component compounds were high-textured. In ZnSe films with cubic structure in all the range of the deposition temperatures, the texture [111] was observed (Fig. 2a). For low-temperature CdSe films with the hexagonal structure, the typical axial growth texture was observed [002] (Fig. 2b). With increasing the substrate temperature, the gradual transition from the [002] to [102] texture occurs. The dependence of orientation factor f on the condensation temperature T_s of chalcogenide films has a complex character (Fig. 3). So, the perfectness of CdSe film texture is slightly worsened at $T_s \sim 570$ K and in conditions close to thermodynamic equilibrium ($T_s \geq 780$ K). The conditions close to thermodynamic equilibrium were not achieved in our experiments because of insufficient temperature stability of substrates. Quality worsening the chalcogenide film texture at intermediate substrate temperatures is evidently related with the change of their growth mechanism from the layer-wise to columnar one. Similar tendencies in dependences $f - T_s$ were also observed in the works [19, 25] when investigating the CdTe films.

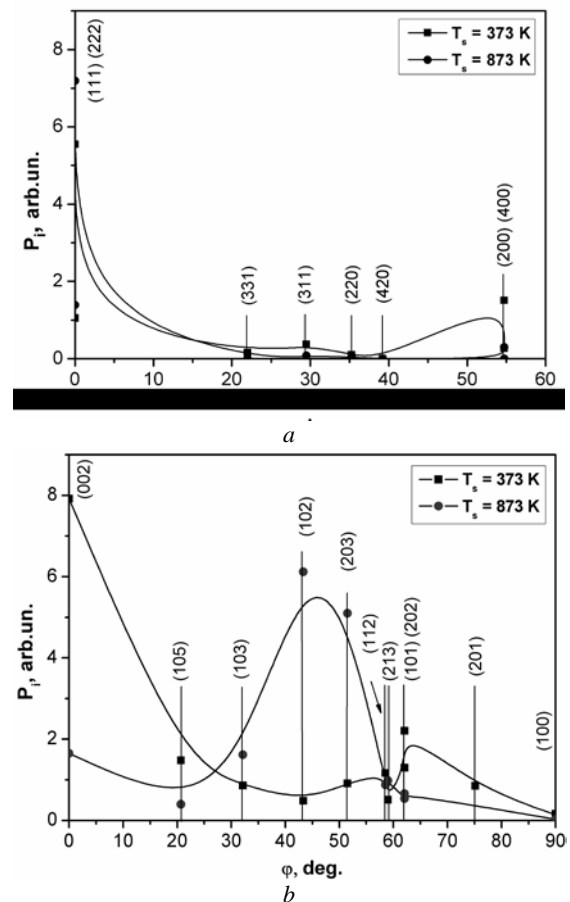


Fig. 2. Dependences of the pole density P_i on the angle φ between the texture axis and normal to different crystallographic planes, which are plotted for the films ZnSe (a) and CdSe (b).

Using X-ray diffraction, we made precise determination of the stock lattice period for evaporated stock as well as lattice periods of ZnSe and CdSe films that were obtained in various condensation regimes. These lattice parameters are very sensitive to the material stoichiometry changes, concentrations of an impurity, oxidation, etc., and allow studying these transformations.

When changing the condensation temperature, the lattice constant of ZnSe film sphalerite phase was varied within the range $a = 0.56557 \dots 0.56864$ nm (Fig. 4). It was observed a decrease of the period with increasing the condensation temperature of material up to $T_s \sim 770$ K, with the following increase of a to the values presented in JCPDS ($a = 0.56688$ nm). These results are well-correlated with the experimental data $a = 0.566 \dots 0.571$ nm obtained for (111) line of films deposited using the CSVS method by the authors of [14].

The results of measuring the lattice constants for cadmium selenide by using the method of consequent approximations after the first, third and fifth iterations have been presented in Table 1. As one can see, a , c and

Table 1. CdSe lattice constants determined by iteration method using the Nelson-Riley extrapolation graphs.

T_s, K	Third iteration			Fifth iteration		
	a, nm	c, nm	c/a	a, nm	c, nm	c/a
charge	0.42983	0.70116	1.631	0.42983	0.70116	1.631
373	0.43047	0.70268	1.632	0.43073	0.70240	1.631
473	0.43067	0.70182	1.630	0.43069	0.70246	1.631
573	0.43018	0.70227	1.633	0.43036	0.70149	1.630
673	0.43079	0.70241	1.630	0.43076	0.70167	1.629
773	0.43135	0.70021	1.623	0.43166	0.69987	1.621
873	0.43124	0.70237	1.629	0.43142	0.70214	1.628
$a = 0.42990 nm, c = 0.70100 nm, c/a = 1.631$ [20]						

their ratio c/a did not change after the third and, in some cases, after the fifth iteration. It allows to obtain their reliable values.

Dependences of CdSe lattice constants and their ratio c/a on the substrate temperature obtained as a result of the fifth iteration are depicted in Fig. 5.

It was estimated that the experimental values of CdSe stock lattice constants ($a = 0.42983 nm, c = 0.70116 nm$) are well-correlated with the data presented in JCPDS for bulk material ($a = 0.42990 nm, c = 0.70100 nm$) [20]. Simultaneously, the crystallographic constants of film material ($a = 0.43036...0.43166 nm, c = 0.69987...0.70246 nm$) are slightly higher than the data presented in the reference book (they were obtained without using the iteration method). But they are well-correlated with results obtained by Nelson-Riley method in some works, where films were deposited by condensation in vacuum ($a = 0.4281...0.4304 nm; c = 0.6981...0.7020 nm$) [10]. It should be noted that there is certain similarity of the dependences $c - T_s$ and $c/a - T_s$ obtained for CdSe films with the data described earlier for ZnSe films.

The complex dependence of ZnSe and CdSe lattice parameters on the condensation temperature may be explained by the stoichiometry change of compounds due to their non-congruent evaporation and by re-evaporation from the substrate.

In Fig. 6, we have shown the transmission spectra of zinc and cadmium chalcogenide films obtained under different condensation conditions and monitored near the energies corresponding to the wide band gap (BG) of materials.

In the dependences $T(\lambda)$ (Fig. 6), we can observe maximums and minimums of the intensities related with interference of light in thin layers of chalcogenides (thickness of investigated films was $l \leq 1.1 \mu m$). Interferential peaks presented by these spectra testify homogeneity of the investigated film areas.

To determine the optical width of BG E_g for these materials, extrapolation of the linear part in the dependence $(ahv)^2 - hv$ onto the energy axis was used [26, 27]. By monitoring the transmission and reflection spectra, one can obtain absorption spectra of chalcogenide films that allow to determine E_g using the following ratio:

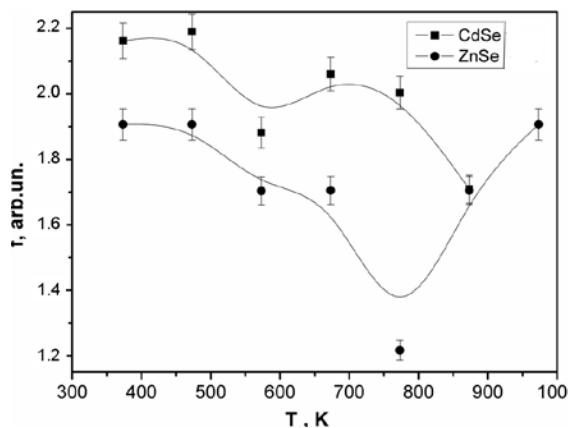


Fig. 3. Dependence of the orientation factor f on the film substrate temperature T_s .

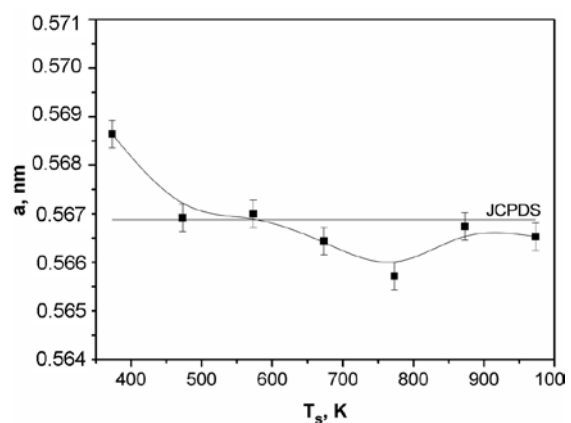


Fig. 4. Variation of the ZnSe lattice constant with the condensation temperature.

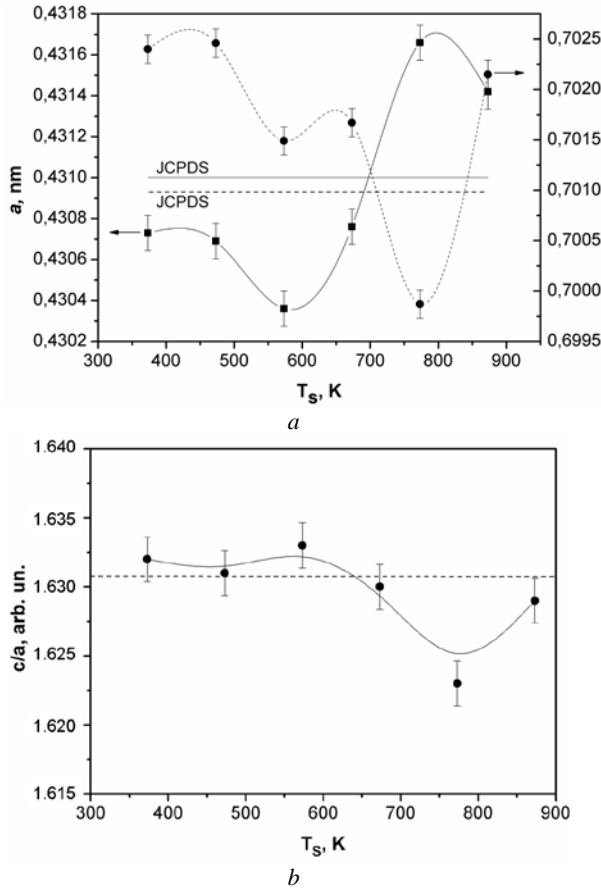


Fig. 5. Lattice parameters (a and c) of CdSe (a) and their c/a ratio (b) versus substrate temperature (fifth iteration).

$$\alpha = -\frac{1}{l} \ln \left(\frac{1}{R^2} \left(-\frac{(1-R)^2}{2T} + \left(\frac{(1-R)^4}{4T^2} + R^2 \right)^{1/2} \right) \right). \text{ This ratio}$$

was obtained taking into account repeated light reflexions inside the film, but it ignores its absorption in semiconductor transparency range [27-29]. $(\alpha h\nu)^2 - h\nu$ dependences that were used to determine the width of BG for both materials were presented in Fig. 7.

In the case of zinc selenide (Fig. 7a), the $(\alpha h\nu)^2 - h\nu$ dependences were approximated as a rule by a straight line. The energies corresponding to the intersection points of this line with the x -axis represent the width of BG for respective material ($E_{g1} = 2.579...2.67$ eV). The accuracy of E_g determination was ± 0.01 eV. These results coincide well with those presented in [11] for films obtained using the charge vacuum evaporation method (2.58...2.68 eV) and with the reference data (2.67 eV) [1].

In most of cases, two linear plots on dependences $(\alpha h\nu)^2 - h\nu$ of CdSe films (Fig. 7b) were observed, and their extrapolation may be obtained by two different values of energies. The lower BG values $E_{g1} = 1.67...1.74$ eV correspond to BG of material. These values are well-correlated with E_g of three-dimensional CdSe single-crystals. In compliance with the reference

data [23] at room temperature, their energy was $E_{g1} = 1.71$ eV when $E_0 \perp c$ and $E_{g1} = 1.73$ eV when $E_0 \parallel c$ (E_0 is the electric field vector of light wave). The obtained values of BG for film condensates were $E_{g1} = 1.73...1.74$ eV and gave an evidence of the presence of pronounced texture in these layers, and the axis c of the crystal lattice is perpendicular to substrate. It is proved by X-ray diffraction results for the films presented earlier with the growth texture [002]. The higher values of energies $E_{g2} = 1.87...2.25$ eV correspond to transitions of charge carriers excited by light between conduction band and segregated owing to spin-orbital interaction lower valence band of material (point Γ_7) [23].

In Table 2, some optical characteristics of cadmium selenide films calculated from reflection and transmission spectra at $\lambda = 850$ nm ($E = 1.46$ eV) are presented. Optical constant determination was carried out using the method presented in [30, 31].

It was estimated that values of the refraction index of material varied within the range $n = 2.23...2.74$, which is in a good correlation with values presented in the reference book for bulk semiconductor $n = 2.50...2.65$ ($\lambda = 700...1000$ nm) [32]. The real part

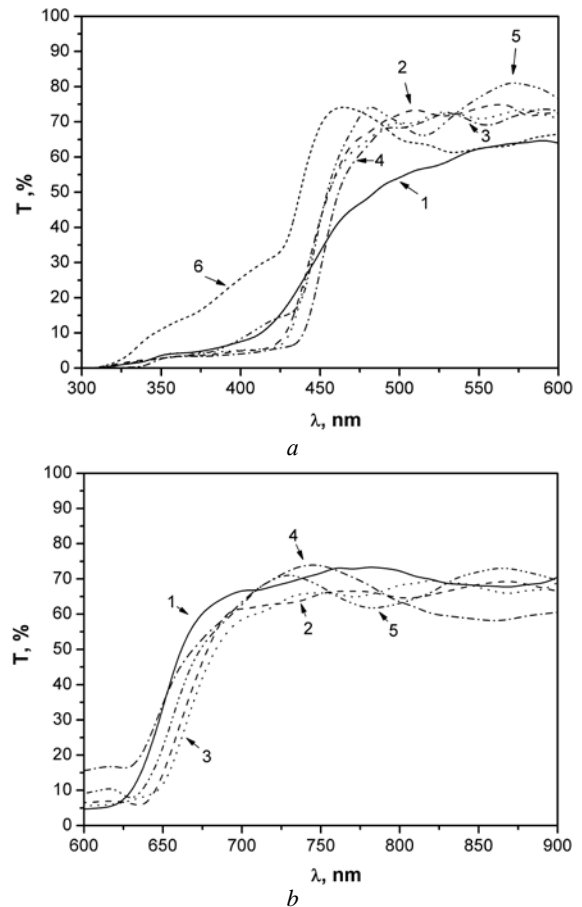


Fig. 6. Spectral distributions of transmission coefficients for ZnSe (a) and CdSe (b): $T_s = 373$ (1), 473 (2), 573 (3), 773 (4), 873 (5), and 973 K (6).

Table 2. Optical and dielectric characteristics of cadmium selenide films.

T_s , K	l , μm	E_{g1} , eV	E_{g2} , eV	n	k	ε_1	ε_2
373	0.70	1.73 ± 0.01	2.13 ± 0.01	2.74 ± 0.03	$10^{-2} \pm 0.001$	7.5 ± 0.4	0.05 ± 0.003
473	1.10	1.67 ± 0.01	–	2.68 ± 0.03	$4.6 \cdot 10^{-3} \pm 0.005$	7.2 ± 0.4	0.03 ± 0.002
673	0.55	1.70 ± 0.01	2.13 ± 0.01	2.23 ± 0.02	$4.4 \cdot 10^{-2} \pm 0.004$	7.5 ± 0.4	0.19 ± 0.01
773	0.40	1.68 ± 0.01	2.06 ± 0.01	2.64 ± 0.03	$4.2 \cdot 10^{-2} \pm 0.004$	7.1 ± 0.4	0.30 ± 0.015
873	0.60	1.71 ± 0.01	–	2.50 ± 0.03	$8.8 \cdot 10^{-3} \pm 0.001$	7.0 ± 0.4	0.04 ± 0.002

of the optical dielectric constant for this material is $\varepsilon_1 = 6.0 \dots 7.5$. These values are also in good accordance with those presented in references for single crystal three-dimensional cadmium selenide $\varepsilon_1 = 6.0 \dots 7.9$ [1, 28]. The imaginary part of the dielectric constant is 2- to 3-fold lower than the real part and equal $\varepsilon_2 = 0.002 \dots 0.300$, accordingly, the extinction coefficient is $k = 8.8 \cdot 10^{-3} \dots 4.4 \cdot 10^{-2}$.

For ZnSe at $\lambda = 410$ nm ($E = 3.0$ eV), these values of above parameters are as follows: $n = 2.48 \dots 2.74$, $\varepsilon_1 = 6.2 \dots 7.5$, $\varepsilon_2 = 0.005 \dots 0.010$, $k = 1.04 \cdot 10^{-3} \dots 1.91 \cdot 10^{-3}$.

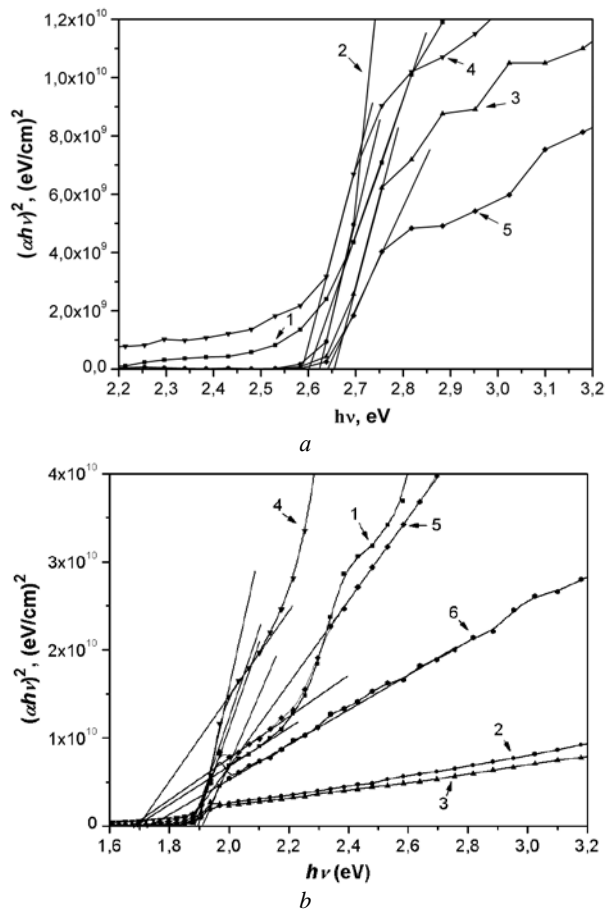


Fig. 7. Variation of $(\alpha h\nu)^2$ with photon energy $h\nu$ for ZnSe (a) and CdSe (b): $T_s = 373$ (1), 473 (2), 573 (3), 773 (4), 873 (5), and 973 K (6).

4. Conclusions

It has been found that ZnSe films deposited using the CSVS method possess a cubic structure, whereas CdSe layers are hexagonal. But in high-temperature condensates of zinc selenide and low-temperature cadmium selenide, by using XRD method it was fixed the tracks of a metastable phase that was hexagonal in ZnSe and cubic in CdSe. This phase is mainly located in the defect-containing fine-dispersed layer near the substrate.

For cubic ZnSe films in all the investigated range of condensation temperatures, the typical grown texture is [111]. In hexagonal CdSe layers deposited at $T_s \leq 773$ K, an axial grown texture [002] was observed and, with increasing the substrate temperature, changed to the texture [102]. It was estimated that perfection of the film texture for zinc and cadmium selenide is worsened to some extent at intermediate condensation temperatures under condensation conditions close to thermodynamic equilibrium.

It was discovered a complex dependence of crystallographic constants of materials on the condensation temperature, which can be caused by changes in their stoichiometry.

It was obtained spectral distributions of optical constants inherent to these materials. It was shown that $(\alpha h\nu)^2 - h\nu$ dependences of ZnSe films can be extrapolated by one line, while those of CdSe films had two linear parts. The lower values $E_{g1} = 1.67 \dots 1.74$ eV correspond to the BG width of CdSe and higher $E_{g2} = 1.87 \dots 2.25$ eV – to transitions of charge carriers excited by light between conduction band and segregated owing to spin-orbital interaction low valence band of material (point Γ_7). The BG width of ZnSe is $E_{g1} = 2.59 \dots 2.67$ eV.

The conditions for obtaining single phase, high-textured stoichiometric chalcogenide films with good optical characteristics have been determined. These may be used in device structures, first of all in thin-film solar cells.

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