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Study of structural and optical properties of CdS thin films depending on chemical deposition time

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CdS thin films have been prepared by chemical deposition onto glass substrates for potential use as buffer layers in thin film photoconverters. Using X-ray phase analysis and Raman spectroscopy, it was established that CdS films synthesized in optimal technological conditions are crystallized in the hexagonal wurtzite structure. It has been shown that deposition time affects the growth rate, morphological and microstructural characteristics of the synthesized material. With increasing deposition time at a given solution temperature, a significant decrease in surface roughness is observed, accompanied by a decrease in the size of crystallite clusters and microstructural defects. The optical band gap of CdS films is 2.53−2.57 eV. The presence of a pronounced green emission band in the photoluminescence spectra indicates that CdS films have a high degree of crystallinity with a minimum defect density.

Keywords: CdS films, chemical deposition, microstructure, optical properties, optical band gap, photoluminescence spectra.

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1. Introduction

One of the most promising materials for manufacturing of optoelectronic devices is Cadmium sulfide (CdS) — a direct-band-gap semiconductor of class II−VI. This material has the *n*-type of conductivity, the optical band gap 2.38−2.62 eV and high absorption coefficient ($> 10^4\,\mathrm{cm}^{-1}$), good thermal and chemical stability [1]. In thin-film solar cells based on CdTe and $\mathrm{Cu(In,Ga)(S,Se)_2}$ with efficiency more than 22%, CdS is used as buffer layer [2–4]. Apart from participation in formation of *p*-*n*-junction, the CdS film promotes passivation of the surface states of the absorber layer and mechanical protection upon deposition of the transparent conducting contact ZnO. Cadmium sulfide is a semiconductor with the band gap depending on its structural modification: $E_g = 2.58\,\mathrm{eV}$ for the stable structure of wurtzite and $E_g = 2.40\,\mathrm{eV}$ for the cubic (metastable) structure [4,5]. The specific feature of the hexagonal phase growth kinetics is formation of the columnar structure of grains along the axis C, perpendicular to the substrate, which creates less grain boundaries preventing the flow of photogenerated excessive carriers [6,7]. These properties determine the promising nature of using a highly stable hexagonal phase of CdS with a higher value of the band gap for the fabrication of different photoconverters. Formation of CdS films of the cubic and hexagonal phase depends on many factors, including on the method of deposition.

Thin films of this material may be easily prepared by several methods, such as RF sputtering, thermal evaporation, spray pyrolysis and chemical bath deposition [6]. Recently the technology of producing the thin films of metal sulfides is dominated by the methods on the basis of chemical processes [8]. One of the basic requirements for large-scale use in solar cells is the production of thin films over large areas and at temperatures below 100°C. The ideal choice for production of the thin semiconductor films is the technology of chemical surface deposition, which consists in application of a solution containing metal ions and the source of sulfur ions onto a substrate. The surface tension of the solution provides for minimization of the reaction mixture volume and its content on the substrate. CdS thin films from aqueous solutions are deposited as a result of a reaction between the cadmium salts and thiocarbamide (thiourea) in an alkaline medium. Besides, the CdS films grown in equilibrium conditions, which makes it possible to reduce the concentration of defects compared to other methods. The physical properties of the deposited films mainly depend on such parameters as the pH value and the temperature of the aqueous solution, the type of substrate and the relative concentration of the substances that create $\mathrm{Cd^{2+}}$ and $\mathrm{S^{2-}}$ ions in the solution for chemical reactions. The crystalline structure of CdS films formed by the method of chemical deposition, is determined by the composition of the bath, the temperature and pH of the solution. The authors of [9–11] report the preparation of CdS films with cubic modification from a bath containing cadmium chloride, potassium hydroxide, ammonium nitrate and thiourea at a temperature of 75°C. In the papers [12,13] the CdS films of hexagonal structure (wurtzite) were produced by chemical deposition from the solution containing the cadmium nitrate, sodium citrate, aqueous solution of ammonia and thiourea at the bath temperature 60°C. There are reports of the formation of two-phase CdS films with cubic and hexagonal modifications by chemical bath deposition (CBD) [14,15]. Considerable effect on the physical properties of CdS thin films made by the CBD method is provided by the duration of deposition. Despite the presence of quite many publications dedicated to the synthesis of CdS thin films, the structure and the phase composition of the synthesized layers remain understudied. The analysis of the references shows that the research of the effect of deposition time on the optical properties of CdS thin films is rare. The authors of the paper [16] during the research of the effect of deposition time (15−90 min) and temperature (55−75°C) on optical and structural properties of CdS thin films using as the main sources the atoms of Cd and S the cadmium sulfate ($CdSO_4$) and thiourea ($CS(NH_2)_2$) found that the structure of CdS films is transformed from the cubic structure into the mixture of the cubic and rhombic phases as the deposition duration increases to 90 min. They also indicated that crystallite sizes increase with increasing deposition time. However, the kinetics of the phenomenon observed by the authors is not clearly identified. Some studies propose the different mechanisms of film growth using the method of chemical deposition requiring additional study [17,18]. Besides, almost in all published papers to produce the CdS thin films by the CBD method used the complex agents, and the synthesized films were usually exposed to post growth treatment before research of their structural and optical properties.

This paper provides the results of studies of the physical characteristics of CdS thin films (optical absorption, band gap, Urbach energy, extinction and refraction indices), prepared on glass substrates by chemical bath deposition method without additional annealing.

2. Materials and research methods

2.1. Production of CdS films

CdS thin films were produced by the method of chemical deposition in the bath using a solution containing 90 ml deionized water, 30 ml 25% aqueous ammonia, 40 ml cadmium sulfate (0.0096M $CdSO_4$) as a source of cadmium and 40 ml thiourea (0.8M $CS(NH_2)_2$) as a source of sulfur. The pH value of the produced solution is 10.7. The CdS layer was applied on the glass substrates vertically immersed in the produced solution, at the temperature optimal for stable deposition of CdS (62 ± 1)°C (found experimentally). During the entire process of deposition, the solution was mixed using a magnetic mixer. The substrates were first cleaned by ultrasound in acetone and ethanol, washed in deionized water and dried with a jet of nitrogen. After deposition, the CdS films were washed for 10 min in an ultrasound bath to remove adhering CdS particles and dried under the jet of nitrogen. For three different cycles the deposition time was set to 5, 10 and 15 min. The thickness of CdS films measured by a profilometer Mitutoyo Surftest SJ-410 (Japan), was accordingly 102, 135 and 157 nm with the measurement error ±5 nm.

2.2. Experimental methods

The study of the elemental composition of films and surface morphology was carried out by the method of X-ray energy-dispersive microanalysis and scanning electron microscopy using a scanning election microscope of ZEISSEVO series. The structural characteristics of the deposited CdS films were analyzed using an X-ray diffractometer Ultima-IV with monochromatic radiation CuK_α ($\lambda = 0.15406$ nm) at sliding angles of incidence 1° (GIXD) by scanning in the range from 10° to 80°. Raman scattering spectra of CdS films were recorded at room temperature using a Nanofinder HE confocal Raman spectrometer (LOTIS TII, Belarus-Japan) with a spectral resolution of no worse than $3\,cm^{-1}$. Measurements were made in the geometry of back scattering without the analysis of polarization of scattered radiation. Raman scattering excitation was carried out by a solid-state laser in continuous mode with a wavelength of 473 nm. The diameter of the laser spot on the surface of the sample was $0.6-0.7\,\mu$m. The studies of the structure and roughness of the CdS film surface were performed using an atomic-force microscope NT 206 (Microtestmachines Co., Belarus) in a contact mode. To study the surface of the samples, at least 5 points were selected on each of them with a scanning area of $3 \times 3\,\mu$m. For each area, the values of the arithmetic mean and root mean square roughness, maximum height of the surface profile irregularity were found, and the results obtained were averaged for every sample. The transmission and reflection spectra of the films were recorded at room temperature on a Photon RT spectrophotometer (Essent Optics) in the wavelength range 400−2500 nm with a spectral resolution of at least 4 nm in unpolarized light. Photoluminescence (PL) spectra were recorded at room temperature using a PL-spectrometer of high resolution Nanofinder HE (LOTIS TII). A solid-state laser was used as source of excitation (355 nm). The diameter of the excitation area was $2\,\mu$m, the power of laser radiation incident on the sample was no more than 2.0 mW to avoid thermal damage, and the spectral resolution was no less than $2.5\,cm^{-1}$. A cooled silicon photodetecter was used as a light-sensitive detector, the signal accumulation time was 10 s.

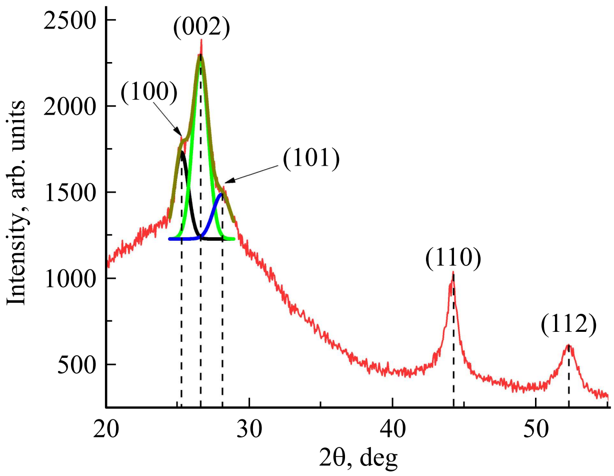

Figure 1. XRD pattern of CdS film deposited for 15 min.

3. Results and discussion

3.1. X-ray diffraction analysis and morphology

The CdS films deposited on the glass substrates were transparent, light yellow, had homogeneous surface without visible holes and had good adhesion to the substrate. When determining the elemental composition of CdS films, it was established that their composition was close to stoichiometric one. However, as deposition time is increased (from 5 to 15 min), the concentration of cadmium in CdS films slightly increases from 50.56 to 51.93 at.%, and the content of sulfur accordingly decreases from 49.44 to 48.07 at.%.

The study of the phase composition of the formed CdS films demonstrated that they are crystallized in a stable hexagonal structure of wurtzite with preferable orientation in direction [002]. Diffraction patterns of CdS films produced under the specified conditions had a similar nature with the wide reflex of high intensity in the area $2\theta = 26.50^\circ$ and reflexes of lower intensity at reflection angles 43.85° and 52.08°. Fig. 1 shows a diffraction pattern of CdS film deposited for 15 min.

Lorentz deconvolution of the dominating peak at $2\theta = 26.50^\circ$ identifies the imposition of characteristic reflexes from the planes (100), (002) and (101) at reflection angles 24.92°, 26.52° and 28.32° accordingly, which belong to the hexagonal phase (JCPDS № 80–0006) only. Low intensity peaks observed at $2\theta = 43.85^\circ$ and $2\theta = 52.08^\circ$, are related to planes (110) and (112) of the hexagonal structure of CdS (symmetry $C_{6\nu}$ ($P6_3mc$)) and are well matched with the literature data [19–21].

In order to confirm the phase composition of CdS films, their Raman spectra were studied, which were recorded in the frequency range of 20–1000 cm^{-1} when excited by light with wavelength 473 nm. The Raman spectra demonstrated asymmetric peaks in the region of ~ 300 and 600 cm^{-1}, specific for the hexagonal structure of CdS [9]. The dominating oscillation in these spectra was a longitudinal acoustic mode 1LO at 302 cm^{-1} with its first overtone 2LO at 601 cm^{-1}. The positions of 1LO and 2LO modes in the Raman spectra of the deposited CdS thin films are are shifted relative to the spectrum of bulk CdS due to a likely effect caused by small size of the microcrystals. No additional peaks corresponding to the oxide phase appeared in the spectra. Increased duration of synthesis to 15 min is accompanied with a minor increase in the intensity of the main reflexes, which is obviously caused by increased thickness of the synthesized CdS layer. The obtained data are well matched with the reported values of oscillating modes for CdS thin films with wurtzite structure [9,22] and confirm the results of the study of phase composition and structure of the CdS layers we synthesized.

To study the morphological characteristics of CdS films was used the method of atomic-force microscopy (AFM), the dependence was found between the topography of the surface, the size of crystallites and the ratio of their large and small fractions depending on the time of synthesis. Images of CdS sample surfaces prepared at different deposition time are given in Fig. 2. Samples deposited for 5 min are characterized by highest roughness with the arithmetic mean roughness $R_a = 7.8$ nm and root mean square roughness $R_q = 11.0$ nm.

Dimensions of crystallite clusters in CdS-layers deposited for the minimum time vary from 100 to 600 nm, besides, their largest part has the size from 200 to 400 nm. As the deposition duration increases, the surface roughness reduces to $R_a = 5.1$ nm, $R_q = 7.1$ nm (deposition for 10 min) and $R_a = 3.9$ nm, $R_q = 5.1$ nm (deposition for 15 min). Most grains in these samples have the size within 100–250 and 50–150 nm (for 10 and 15 min respectively).

It should be noted that the increased duration of synthesis results in a tighter stacking of crystallites accompanied with smoothening of the surface of the formed CdS film. This is quite an important factor in development of the technological process of buffer layer deposition onto absorbing layers of thin-film solar cells based on $Cu(In,Ga)(Se,Se)_2$ and $Cu_2ZnSnSe_4$.

3.2. Optical and luminescent characteristics

The resulting CdS films are characterized by high optical transmission in the wavelength region above 500 nm, where their transmittance reaches values of 80–90%. The highest transmission was observed for the films deposited for 5 min, which is insignificantly decreased as the deposition duration increased. Fig. 3, *a* shows the spectra of optical transmission and reflection of CdS-films depending on the synthesis time. Graphs of the extinction (attenuation) coefficient (k) depending on the wavelength for films (Fig. 3, *b*) deposited in all modes show that the values of k decrease to minimum values in the wavelength region of ≈ 500 nm, corresponding to the optical bandwidth of transmittance, and then increase slightly.

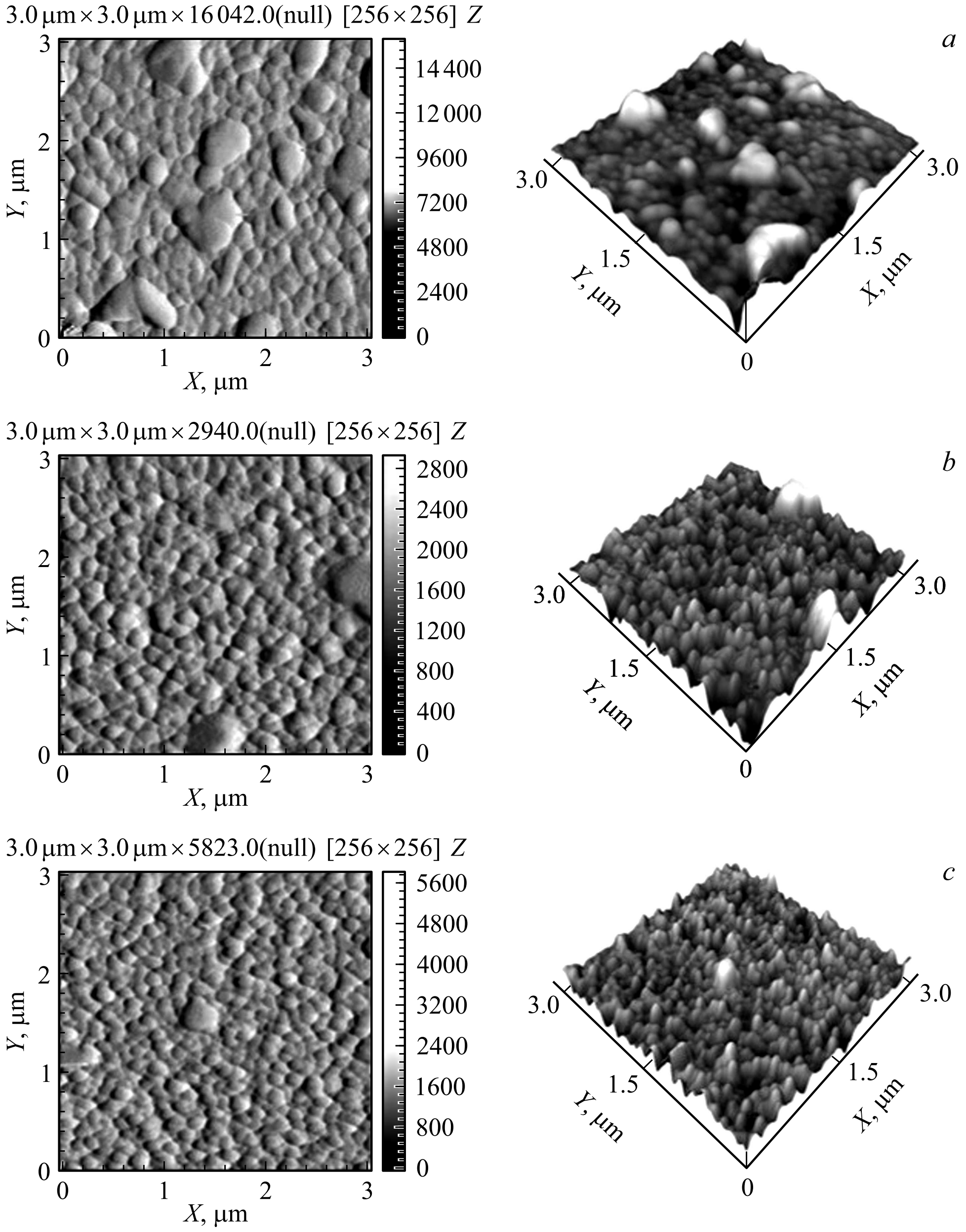

Figure 2. 3D AFM-image and topography of CdS film surface depending on the deposition time: 5 (a), 10 (b), 15 min (c).

The observed reduction of the value k shows that the maximum absorption of the incident radiation occurs in the region of energies that are larger than the optical band gap of CdS. As can be seen from Fig. 3, b, increasing the synthesis duration to 15 min results in a considerable reduction of absorption in the deposited layer.

Using the spectra of transmission and reflection, the absorption coefficient (α) was calculated taking into account of multiple internal reflections in a flat parallel sample from its bondaries [23] using the transmission and reflection spectra and the following formula:

$$\alpha = -\frac{1}{d}\ln\left(\frac{\sqrt{(1-R)^4+4T^2R^2}-(1-R)^2}{2TR^2}\right), \qquad (1)$$

where d — film thickness, T and R — transmission and reflection, coefficients respectively. Upon analysis of optical characteristics it was found that the films have a large coefficient of optical absorption ($\alpha > 10^4\,\mathrm{cm}^{-1}$) in the region of high photon energies ($h\nu > E_g$). The optical band gap (E_g) of the produced CdS thin film was determined by extrapolation of the linear spectral dependence $(\alpha h\nu)^2 = f(h\nu)$ towards the axis of photon energy in the assumption of the direct allowed interband transitions according to the Tauc equation [24]:

$$(\alpha h\nu)^2 = A(h\nu - E_g), \qquad (2)$$

where $h\nu$ — incident photon energy, A — constant.

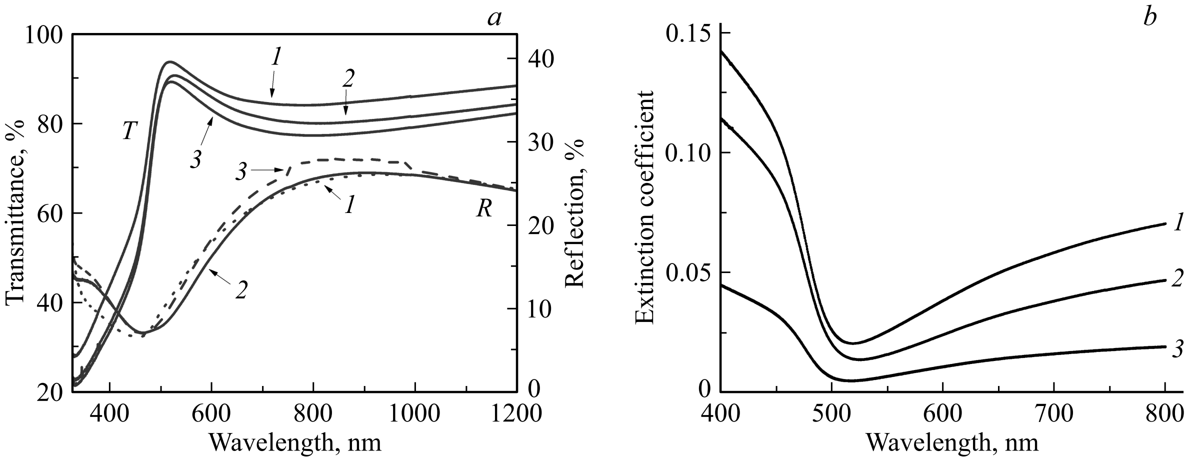

Figure 3. The transmittance and reflection spectra (a) and extinction coefficient (b) of CdS thin films depending on the deposition time: 5 (*1*), 10 (*2*), 15 min (*3*).

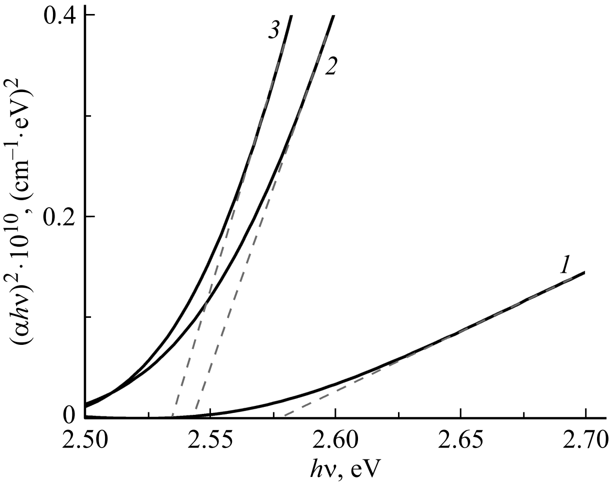

Figure 4. Tauc curves of CdS thin films depending on the deposition time: 5 (*1*), 10 (*2*), 15 min (*3*).

The band gap (E_g), Urbach energy (E_U) and refraction index (n) of CdS films depending on deposition conditions

Film thickness (d), nm	Deposition time (t), min	E_g, eV	E_U, meV	n
102	5	2.57	190	2.502
135	10	2.54	170	2.513
157	15	2.53	160	2.520

The graphs of dependence $(\alpha h\nu)^2 = f(h\nu)$ for CdS films deposited for different time are presented in Fig. 4.

As the thickness of the deposited films increases from 102 up to 157 nm with increasing synthesis time (from 5 to 15 min) optical band gap decreased from 2.57 to 2.53 eV. The similar effect observed by other authors as well may be related to the impact of various factors, such as the grain size, structural ordering, presence of admixtures, deviation from the film stoichiometry and deformation of the lattice [25].

Besides, we observed the strong absorption for the studied samples below the fundamental absorption edge, which is explained by transitions involving band tails states [26]. The optical absorption coefficient (α) below the edge of the band edge in crystalline and in disordered semiconductors includes transitions from the localized states or towards these stages and usually has exponential dependence on the photon energy. Such dependence of the coefficient α, which was first found by Urbach, and then Martienssen, is known as the Urbach-Martienssen rule. Urbach energy (E_U) in the region of low photon energies ($h\nu < E_g$) is described [26] by the following expression:

$$\alpha = \alpha_0 \exp\left[\frac{\sigma(h\nu - E_g)}{kT}\right], \tag{3}$$

where $h\nu$ — radiation photon energy, α_0 — coefficient of absorption at energy value $E = E_g$, k — Boltzmann constant, T — temperature, $\sigma/kT = E_U$ — Urbach energy characterizing the degree of steepness in the dependence of the absorption coefficient and depends on the material parameters.

The value of E_U may be found from the reverse inclination of the linear dependence curve $\ln(\alpha) = f(h\nu)$. Established reduction in E_U values from 190 to 160 meV (see Table) with increasing thickness of the deposited CdS films indicates the important role of deposition time in reduction of structural defects and density of the localized states in the band gap of the CdS material.

The refractive indices (n) of as deposited CdS films were estimated based on optical measurements using the Herve-Vandamme formula, where n is inversely proportional to E_g, and A and B are constants equal to 13.6 and 3.4 eV,

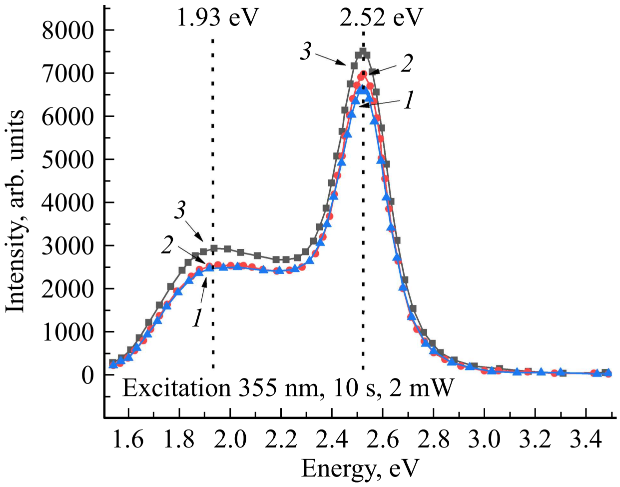

Figure 5. PL spectra of CdS films depending on the deposition time: 5 (*1*), 10 (*2*), 15 min (*3*).

respectively [27]:

$$n = \left[1 + \left(\frac{A}{E_g + B}\right)^2\right]^{1/2}. \quad (4)$$

The obtained values of the refractive index slightly rise from 2.502 to 2.520 with increasing deposition time (corresponding with increasing film thickness). This indicates that the layers were becoming more dense with the increase of the process time, which complies with the data of atomic-force microscopy. Changes in the values of n relative to the film thickness are also given in the table.

Fig. 5 presents the PL spectra of CdS thin films deposited over a period of 5 to 15 min at a constant solution temperature $(62 \pm 1)^\circ$C. Cadmium sulfide is a compensated semiconductor containing multiple defects of shallow and deep levels. The nature and type of defects strongly depend on the conditions of deposition and accordingly the PL spectrum varies. Two peaks in the range from 1.6 to 2.8 eV are seen in the given PL spectra of films. The red emission band located in the energy range 1.8–2.0 eV is due to election transitions involving deep levels of interstitial cadmium and sulfur vacancy [28–31], and therefore, this effect correlates with the accumulation of crystallographic defects in CdS layers grown at low deposition temperatures. In general the red emission band is related to involvement of sulfur vacancies into radiation recombination. The energetic band in the area from 2.40 to 2.60 eV in the PL spectra of CdS is known as the green emission band [30–33]. The emission peak at 2.52 eV, close to the obtained optical band gap values of CdS films can be attributed to the energy of bound excitons [32,34]. It is known that the valence band of CdS in the center of Brillouin band is split into two subbands — A, B and C [34]. In our case, the spectral position of this line corresponds to the ground state of A-type exciton of the hexagonal phase of CdS.

The presence of the green emission band in the PL spectra of CdS films indicates that the synthesized layers have crystalline structure with small quantity of grain boundaries and minimum density of intrinsic defects.

Conclusion

CdS thin films of various thickness were prepared by the method of chemical bath deposition. The effect of deposition time on the structural and optical properties of the formed films was studied. The films we obtained have a composition close to stoichiometry with a small deficit of sulfur and a hexagonal structure of wurtzite with the preferential orientation in the direction [002]. The dependence of the film transmission coefficient on their thickness was revealed. CdS films are characterized by direct allowed transitions with a band gap E_g in the range of 2.53–2.57 eV. It was found that the values of Urbach energy in CdS films decrease as the deposition time increases from 190 to 160 meV. In the PL spectra of all samples there is a relatively narrow intense peak observed at 2.52 eV (green emission band) and a wider peak of defect emission with the center at 1.93 eV (red emission band). The presence of the green emission band indicates that the deposited CdS films have high crystallinity and may be used as a buffer layer for thin-film solar cells.

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Conflict of interest

The authors declare that they have no conflict of interest.

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