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PHYSICAL METHODS OF INVESTIGATION

Deposition Conditions, Composition, and Structure of Chemically Deposited In_2Se_3 Films

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Abstract—In_2Se_3 films up to 300 nm thick have been obtained for the first time by hydrochemical deposition on glass, glass ceramic, and molybdenum substrates in the $In(NO_3)_3$–$C_4O_6H_6$–$CSeN_2H_4$ system with the use of selenourea as a chalcogenizing agent. The phase and element composition and morphological features of layers obtained at 353 and 363 K have been studied by X-ray photoelectron spectroscopy, energy-dispersive electron probe X-ray microanalysis, and scanning electron microscopy. The optical band gap width has been determined.

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Thin films of indium(III) selenide are widely used in micro- and optoelectronics. Owing to electrophysical properties, such as band gap width ($E_g^{\text{opt}} = 1.2$ eV) [1], high absorptivity ($\alpha \sim 10^5\ \text{cm}^{-1}$), and environmental safety, In_2Se_3 is used for production of nuclear radiation detectors, red and green semiconductor diodes, and microwave-safe coatings [2, 3]. However, the greatest interest in manufacturing In_2Se_3 films stems from the creation of solar cells based on copper indium diselenide $CuInSe_2$ with a solar conversion efficiency of ~20% [4] and from their potential use as environmentally safe buffer layer in active solar cells [5].

Indium(III) selenide is a representative of layered semiconductors and have several modifications with phase transitions at 473, 923, and 1023 K [1]. Currently, three major In_2Se_3 polymorphs are known: α, β and γ. The α-In_2Se_3 and β-In_2Se_3 modifications are metastable at room temperature and crystallize in the hexagonal crystal system [1]. The γ modification formed at $T > 650°C$ (cubic) is rather stable at room temperature, and the δ modification (monoclinic) and high-temperature α modification (hexagonal) are the most stable ones [1, 6]. Such a diversity of indium selenide polymorphs dictates the choice of a method for its synthesis.

At present, there are the following basic methods of fabrication of indium(III) selenide thin films: spray deposition onto a heated substrate [7], chemical vapor deposition [8, 9], atomic and molecular beam epitaxy [10, 11], vacuum deposition [12, 13], and chemical bath deposition from aqueous solutions [14].

Most of these methods require high temperatures, high vacuum, and high-purity precursors and are characterized by the difficulty of predicting the composition and properties. Taking into account low-temperature conditions of synthesis, its versatile control, and available technological equipment, we believe that chemical bath deposition from aqueous media is one of the most promising routes.

A relatively small number of studies [15–18] are known in which the hydrochemical deposition of indium(III) selenide films has been described. It should be specially noted that in [14, 16, 17], sodium selenosulfate has been used as a chalcogenizing agent in fabrication of indium selenide films. However, a Na_2SeSO_3 solution is difficult to prepare and not always gives a required result from the standpoint of reproducibility of film properties. In the cited works, there is no complex physicochemical approach to hydrochemical deposition of indium(III) selenide, and the choice of formulation and its composition have not been justified. At the same time, the use of selenourea as a chalcogenizing agent for deposition of In_2Se_3 films is of great practical interest; however, no reliable data on this issue are available.

This work deals with the development and justification of conditions for chemical bath deposition of indium(III) selenide films from aqueous solutions with the use of selenourea, the determination of their composition, and the study of their morphological features and semiconducting properties.

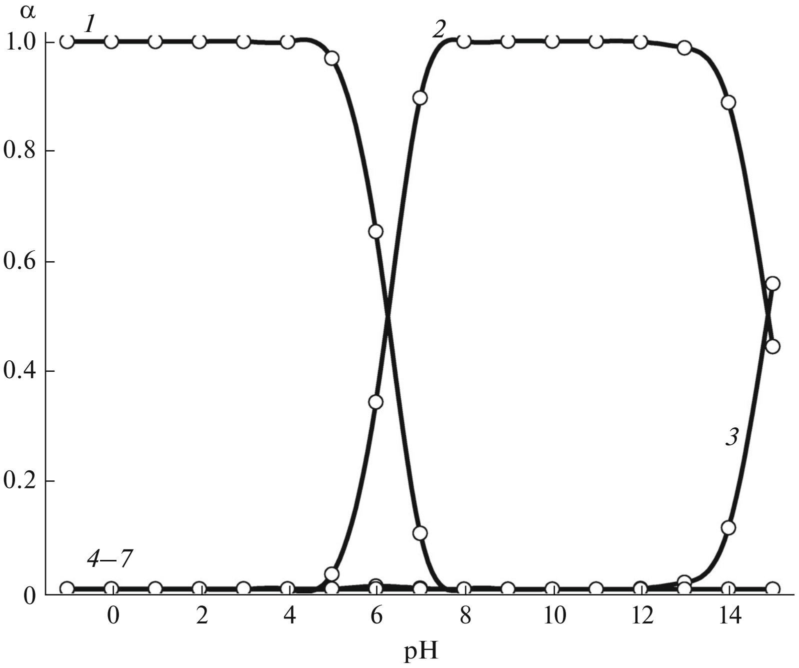

Fig. 1. Fractional concentration α of free and complex indium ions in a tartaric acid solution at 298 K with allowance for the formation of hydroxide and tartrate complexes: (*1*) $In(C_4O_6H_5)_2^+$, (*2*) $In(OH)_3$, (*3*) $In(OH)_4^-$, (*4*) $InOH^{2+}$, (*5*) In^{3+}, (*6*) $In(OH)_2^-$, and (*7*) $In(C_4O_6H_5)^{2+}$.

EXPERIMENTAL

Indium(III) selenide films were deposited on preliminarily degreased glass, molybdenum, and ST-50-1 glass ceramic substrates from an aqueous reaction mixture containing the following major components: indium nitrate $In(NO_3)_3$, selenourea $CSeN_2H_4$, sodium sulfite Na_2SO_3, and tartaric acid $C_4O_6H_6$. Deposition was carried out at 353–363 K in sealed molybdenum glass reactors in which substrates were fixed at angle of 15°–20° in specially constructed fluoroplastic holders. The reactors were placed into a TS-TB-10 thermostat. Temperature was maintained constant with an accuracy of ±0.1 K. The film deposition time was varied from 30 to 240 min.

The thickness of the synthesized films was estimated on an MII-4M interference microscope (Linnik imcrointerferometer).

The crystal structure and phase composition of the films were studied at room temperature by X-ray crystallography on a Shimadzu XRD-7000 diffractometer ($CuK_{\alpha1,2}$ radiation, λ = 1.54181 nm, 2θ range 20°–120°, step $\Delta(2\theta) = 0.03°$, counting time per point 15 s).

The elemental composition and speciation of elements in the compounds in films were studied by X-ray photoelectron spectroscopy(XPS) on a VG Scientific ESCALAB MK II electron spectrometer with a magnesium cathode as a source of non-monochromatic X-ray MgK_{α}-radiation (1253.6 eV). For the interpretation of the type of chemical bond of elements from the shift of XPS bands, the carbon C1*s* line with a binding energy of 284.5 eV was used for calibrating the binding-energy scale.

SEM images of deposited films were obtained on a MIRA 3 LMU scanning electron microscope coupled with a JED 2300 energy dispersive X-ray (EDX) analyzer.

The absorption and transmission spectra of indium(III) selenide films were recorded on a PE-5300VI spectrophotometer at 298 K.

RESULTS AND DISCUSSION

According to thermodynamic calculations of the boundary conditions of formation [19], the deposition of the In_2Se_3 solid phase at 298 K is possible in the pH range 3.0–10.0 [20, 21]. Taking into account that this process can be accompanied by vigorous formation of indium hydroxide $In(OH)_3$, we believe that the pH range 3.0–4.5 in which $In(OH)_3$ does not form is optimal for synthesis.

As a complexing agent for indium, tartaric acid $C_4O_6H_6$ was selected. Tartaric acid forms with indium relatively stable complex species $In(C_4O_6H_5)^{2+}$ and $In(C_4O_6H_5)_2^+$, for which the logarithmic instability constants (pk_i) are, respectively, 4.5 and 7.58 [22]. Figure 1 shows the distribution of fractional concen-

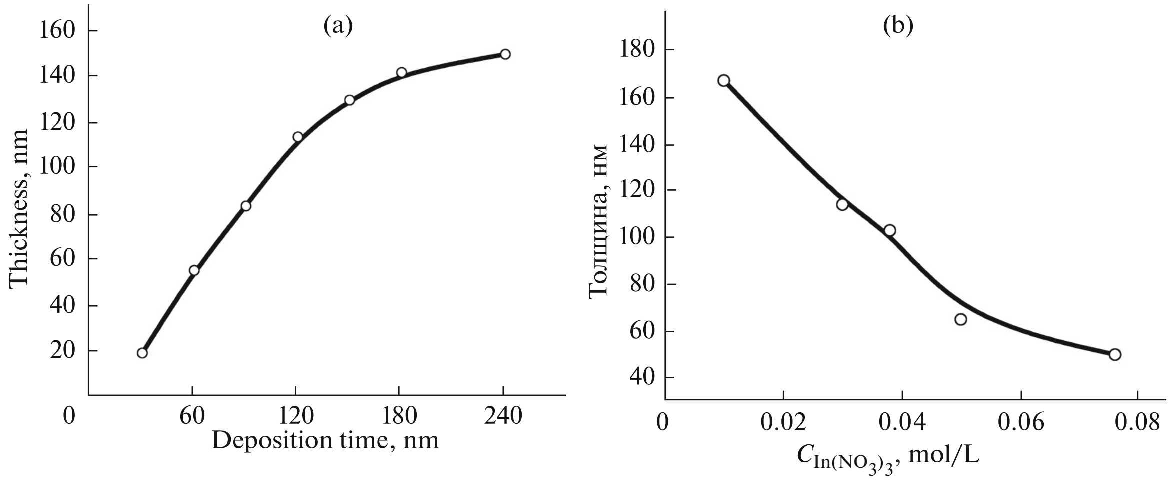

Fig. 2. Dynamics of indium selenide film growth as a function of (a) synthesis time and (b) concentration of the metal salt in solution. Synthesis temperature 353 K.

trations of metal complex species existing in a tartaric acid solution. It is seen that the presence of indium tartrate complexes makes it possible to control the concentration of free indium ions, thus decreasing the possibility of formation of polynuclear indium hydroxo complexes in the course of synthesis. An increase in deposition temperature will lead to a decrease of their stability and, hence, will promote the formation of the selenide phase.

The above range of In_2Se_3 formation was used for working out a procedure of film deposition with selection of concentrations of basic components of the reaction system. The addition of weak tartaric acid, taken as the basic complexing agent, simultaneously afforded smooth regulation of pH of the reaction mixture and its buffering. As an antioxidant for selenourea acting as the chalcogenizing agent, a small amount of sodium sulfite, which has a significant reduction potential, was introduced into the solution. The chemical reaction of formation of indium selenide with the use of selenourea and with allowance for the mechanism of decomposition of thio- and selenoamides [23] can be presented in the following form:

$$\begin{aligned} &2\mathrm{InL}_x^{3+} + 3\mathrm{CSeN_2H_4} + 12\mathrm{OH}^- \\ &= \mathrm{In_2Se_3} + 2x\mathrm{L} + 3\mathrm{CN}_2^{2-} + 12\mathrm{H_2O}, \end{aligned} \tag{1}$$

where L is a ligand, in particular, tartaric acid ions $C_4O_6H_5^+$.

If In_2Se_3 is synthesized with the use of sodium selenosulfate by reaction (2), the products contain an equivalent amount of sulfate ions, which hinder the formation of solid indium selenide and contaminate the resulting layers:

$$\begin{aligned} &2\mathrm{InL}_x^{3+} + 3\mathrm{Na_2SeSO_3} + 3\mathrm{H_2O} \\ &= \mathrm{In_2Se_3} + 2x\mathrm{L} + 3\mathrm{SO}_4^{2-} + 6\mathrm{H}^+ + 6\mathrm{Na}^+. \end{aligned} \tag{2}$$

In addition, the precipitation with sodium selenosulfate occurs at pH > 10 where it is stable, i.e., where the indium hydroxide phase is actively formed. Thus, the use of selenourea can considerably facilitate the deposition of In_2Se_3 films as compared with results in [15–18].

The hydrochemical synthesis with selenourea has led to the formation of layers up to 300 nm thick on glass and ceramic glass substrates. The layers have light red color and good adhesion strength. Figure 2 shows the film growth dynamics at 353 K as a function of deposition time. The longest deposition time was 240 min; however, in this case the layer growth continues. The increase in the initial concentration of the indium salt in a solution leads to a gradual decrease in the thickness of the layer (Fig. 2b); at the initial salt content 0.08 mol/L, the layer thickness was reduced to 50 nm. This is caused by the enhancement of oversaturation of the reaction mixture with indium selenide, with redistribution of formation of the In_2Se_3 solid phase into the bulk of the reaction mixture.

X-ray diffraction analysis of the synthesized films demonstrates that these films are made of hexagonal In_2Se_3 (JCPDS 12-117), with is consistent well with the literature data [24]. This is proved by the diffraction reflections from the (102), (103), (105), and (114) at 2θ angles of 27.187°, 29.086°, 34.771°, and 48.971° observed, according to JCPDS 4-809, on a typical X-ray powder diffraction pattern of a film 300 nm thick on the molybdenum substrate (Fig. 3). The unit cell parameters of synthesized In_2Se_3 are $a = 0.400$ nm and $c = 1.924$ nm. It is worth noting that the patterns also reveal the presence of molybdenum oxide MoO_3 (JCPDS 5-508), with is due to the response from the substrate material. No other impurity phases have been detected.

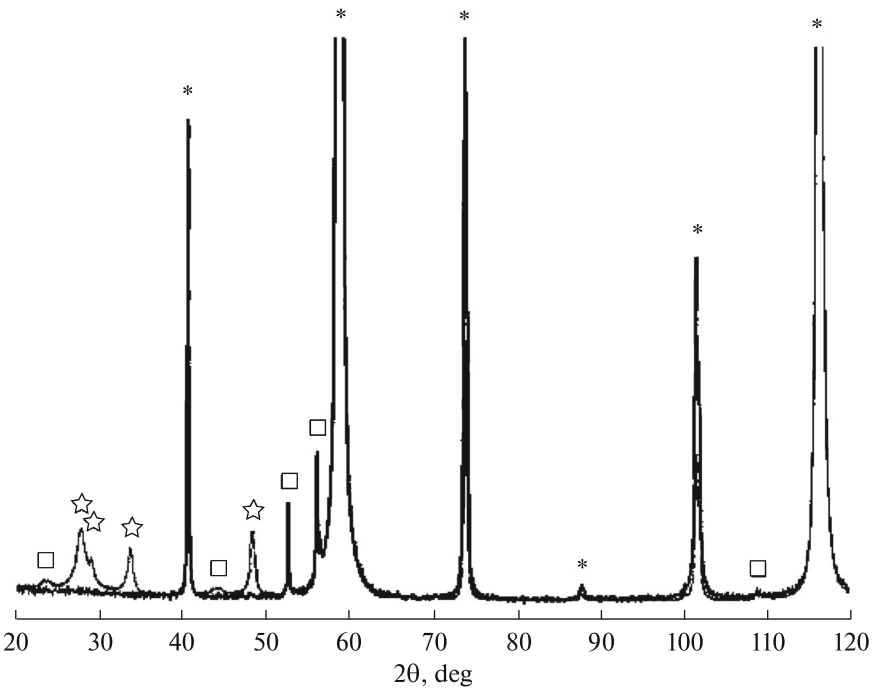

Fig. 3. X-ray powder diffraction pattern of the as-deposited nanocrystalline In_2Se_3 film obtains of molybdenum from a tartaric acid reaction mixture at 363 K. Reflections for molybdenum (*), molybdenum oxide MoO_3 (□), and indium selenide In_2Se_3 (☆) are shown.

XPS analysis of the deposited films has shown that, in addition to indium and selenium, basic elements of surface layers are oxygen and carbon (Table 1). Taking into account considerable content of the latter on the surface, we can state that they are incorporated, first of all, through the formation of oxide phases and surface adsorption processes. For more precise determination of the elemental composition of the film surface and speciation of the major elements, XPS survey spectra with expanded segments of the In4*d*, In3*d*, Se3*d*, and C1*s* lines, as well as of the indium Auger line, have been recorded (Fig. 4).

Comparison of the XPS spectra of the film before (Fig. 4, *a*) and after the 5-min Ar^+ beam etching to the depth of 30 nm (Fig. 4, *b*) shows a sharp decrease in the oxygen and carbon content in the bulk. At the same time, the stoichiometric In : Se ratio typical of indium(III) selenide is almost not disturbed.

All the films had a high electrical resistivity and a high charge (to 7.5 eV), which was taken into account when determining the binding energies of the elements (Table 2). The presented data indicate that all the elements, except carbon, have the same energy state both before and after etching, with a small deviation 0.1 eV. Figure 4, *d* shows well-defined ground-state indium In4*d* and selenium Se3*d* levels with binding energies of 17.6 and 53.8 eV, respectively. Taking into account the data obtained for InSe and α-In_2Se_3 synthesized by

Table 1. Content on basic elements (at %) and their ratio on the surface and in the bulk of as-deposited In_2Se_3 films

Sample	In	Se	O	C	O/In	In/Se	*T*, K
58_0	12.4	11.7	30.9	45.0	2.48	1.06	363
58_0 (at a depth of 30 nm)	50.0	41.8	2.4	5.8	0.05	1.20	363

Table 2. Binding energies of characteristic lines of chemical elements in as-deposited indium(III) selenide films with inclusion of the charge (in electron volts)

Sample	In4*d*	Se3*d*	In3*d*	O1*s*(I)	C1*s*(I)	C1*s*(II)	C1*s*(III)
58_0	17.8	53.9	444.8	532.0	284.6	285.9	288.6
58_0 (at a depth of 30 nm)	17.6	53.8	444.8	530.7	284.6	–	–

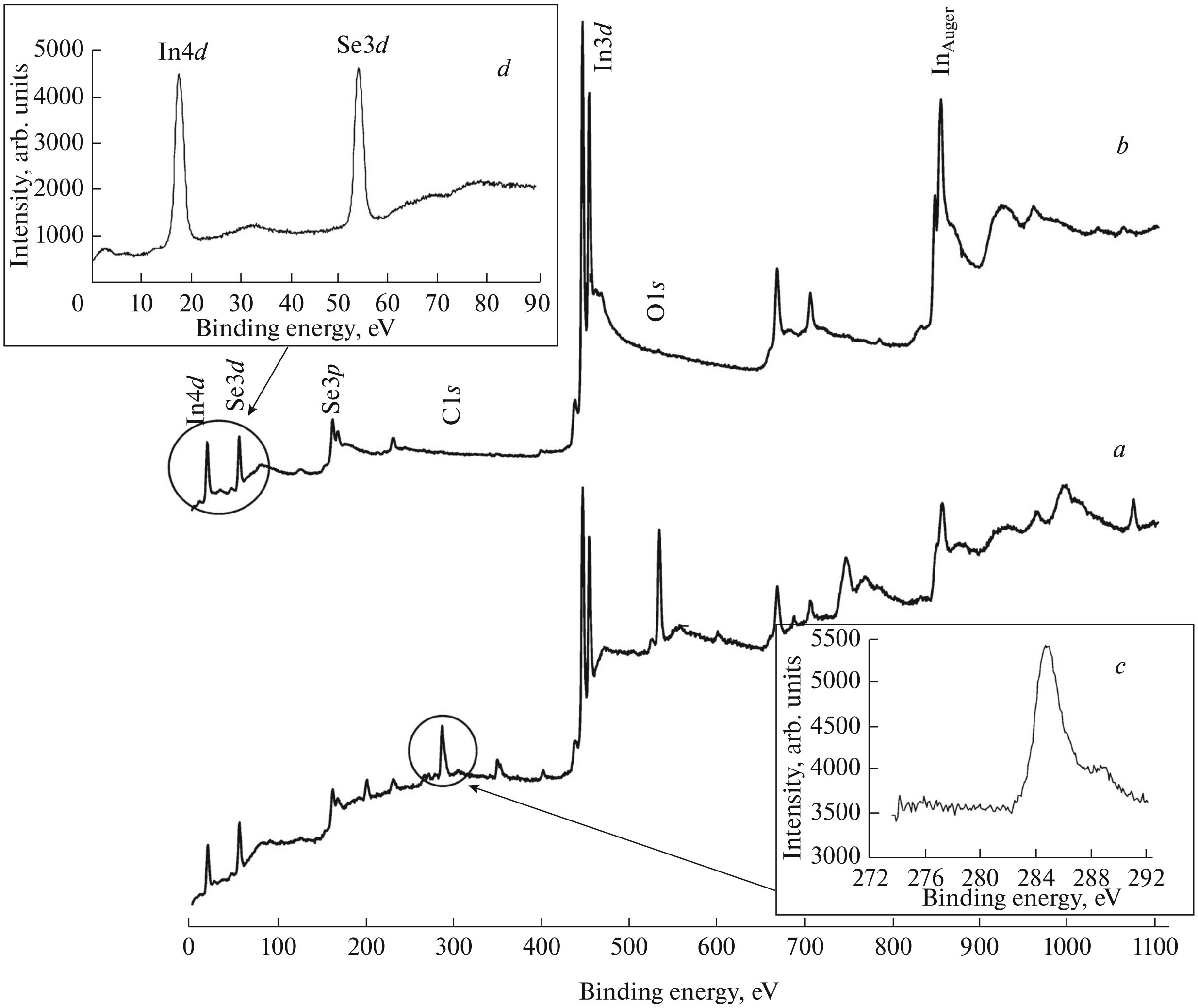

Рис. 4. XPS survey spectra of the surface of the thin indium selenide film (*a*) before and (*b*) after etching to the depth of 30 nm with expanded segments of (*c*) carbon C1*s* shell, and (*d*) indium In4*d* and selenium Se3*d* shells.

pulsed-laser deposition and elemental evaporation [8, 25], we can state that the Se3*d* level has an intermediate position (53.8 eV; 53.25 in [8] and 54.5 eV in [25]) close to the position for In_2Se_3. However, the position of the In3*d* line (444.8 eV) points to the formation of the In_4Se_3 phase in which the indium-to-selenium ratio is 1.33 [8].

The presence of oxygen in the layers follows from the existence of the O1*s* level. According to [26, 27], the O1*s* binding energy of 532.0 eV indicates the presence of C=O bonds, OH^- groups, and adsorbed water. The O1*s* binding energy of 530.7 eV after etching in evidence of the formation of the In–O. All these facts indicate that the film surface is contaminated with both organic compounds and indium oxide. After etching to the depth of 30 nm, the film contains a relatively small amount of oxide, whereas other contaminants are not detected.

For more reliable identification of the resulting compound, we recorded the positions of the metal Auger lines and calculated the α parameter, or Wagner's Auger parameter (Table 3):

$$E_\alpha(\mathrm{X}) = E_{\mathrm{Mg}K} + E_{\mathrm{X}} - E_{\mathrm{Auger}}, \qquad (3)$$

where $E_\alpha(\mathrm{X})$ is the α parameter (in electron volts), $E_{\mathrm{Mg}K}$ is the radiation energy of the magnesium cathode

Table 3. Energy of indium Auger line with inclusion of charging and α parameter for the indium(III) selenide film (in electron volts)

Sample	In Auger line	α Parameter
58_0	847.4	851.0
58_0 (at a depth of 30 nm)	845.9	852.5

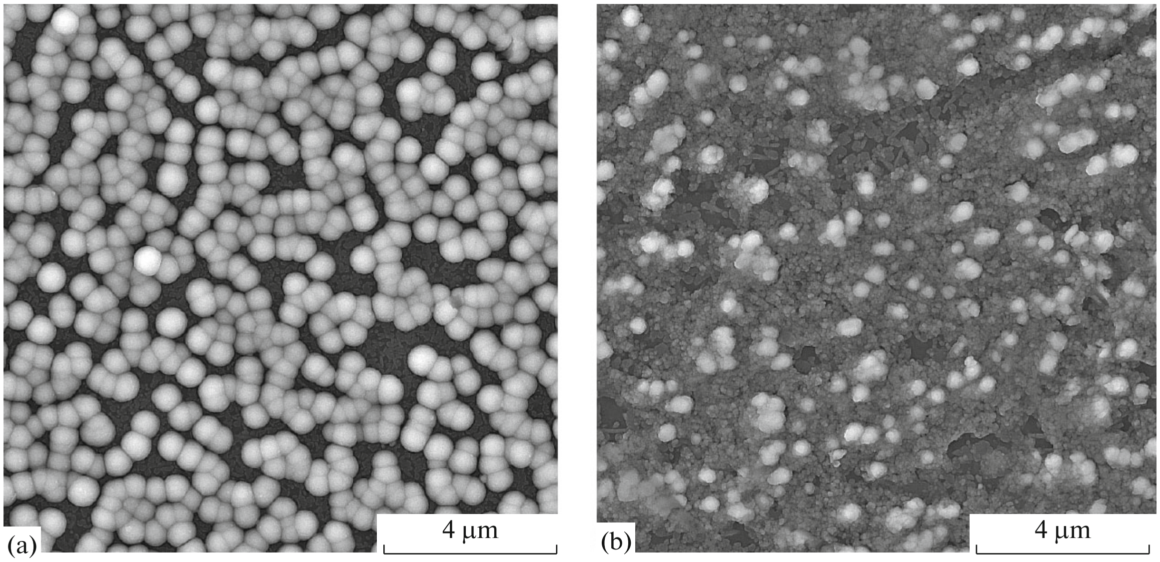

Fig. 5. SEM images of indium(III) as-deposited selenide films obtained at (a) 353 and (b) 363 K on glass ceramics.

(1253.6 eV), E_X is the characteristic line energy, and E_{Auger} is the Auger line energy (in electron volts).

According to [28], the phase of indium oxide In_2O_3 with a binding energy of 851.0 eV is well identified on the surface of the deposited In_2Se_3 film. The indium Auger line in this case is hardly observable. After etching of the surface layer, the α parameter increases to 852.5 eV, which corresponds to the In_2Se_3 phase.

Figure 5 shows SEM images of the surface of as-deposited In_2Se_3 films synthesized at two different temperatures. In both cases, the layers have a clearly pronounced nanocrystalline character. The synthesis at 353 K (Fig. 5a) leads to the formation of similar regular spherical particles about 500 nm in size uniformly distributed over the surface; on closer examination, these globules consist of spherical nanocrystallites up to 50 nm in size [29].

Increasing synthesis temperature to 363 K is accompanied by a considerable change in the microstructure and morphology of the In_2Se_3 film (Fig. 5b). As shown by interference microscopy, the film thickness decreased to 200 nm, which entailed a significant deterioration of its continuity. It is seen that partial destruction of the globular structure shown in Fig. 5a took place. On detailed examination, there are regions free of spherical particles but containing formations of less pronounced architectural forms. The size of nanocrystallites in globules increased to 70–80 nm.

Table 4. Elemental composition of the as-deposited indium(III) selenide film obtained at 353 K

Site	In, at %	Se, at %
1	38.5	61.5
2	38.7	61.3
3	40.1	59.9
4	39.6	60.4
5	39.4	60.6
Arithmetic mean	39.3	60.7

To determine the elemental composition of microcrystallites, the films deposited at 353 and 363 K were subjected to SEM/EDX analysis. The elemental analysis of the globules existing in the films (Fig. 5a) shows that the arithmetic mean indium and selenium concentrations (averaged over all points under consideration) are, respectively, 39.3 and 60.7 at % (Table 4). These values actually correspond to the ratio of these elements in indium triselenide In_2Se_3 and, thus, support the above XPS data.

The EDX analysis of the film deposited on glass ceramics at 363 K (Fig. 5b) is much more complicated. Intercrystallite regions of the film contain, in addition to indium, selenium, and substrate elements, up to 17 at % sulfur generated from sodium sulfite introduced into the reaction mixture. It can be assumed that there is a sublayer based on intermediate oxygen- and sulfur-containing indium compounds promoting nucleation of the In_2Se_3 film on the substrate. Formation of such a sublayer has been suggested in studies dealing with chemical bath deposition of indium sulfide [30, 31].

The optical properties of the deposited In_2Se_3 films were measured by recording an absorption spectrum in the range 300–1000 nm. Indium selenide layers about 200 nm thick on photo glass were studied. According to the data obtained, the synthesized films have a relatively high absorption coefficient of about 2.3×10^5 cm^{-1} (Fig. 6a).

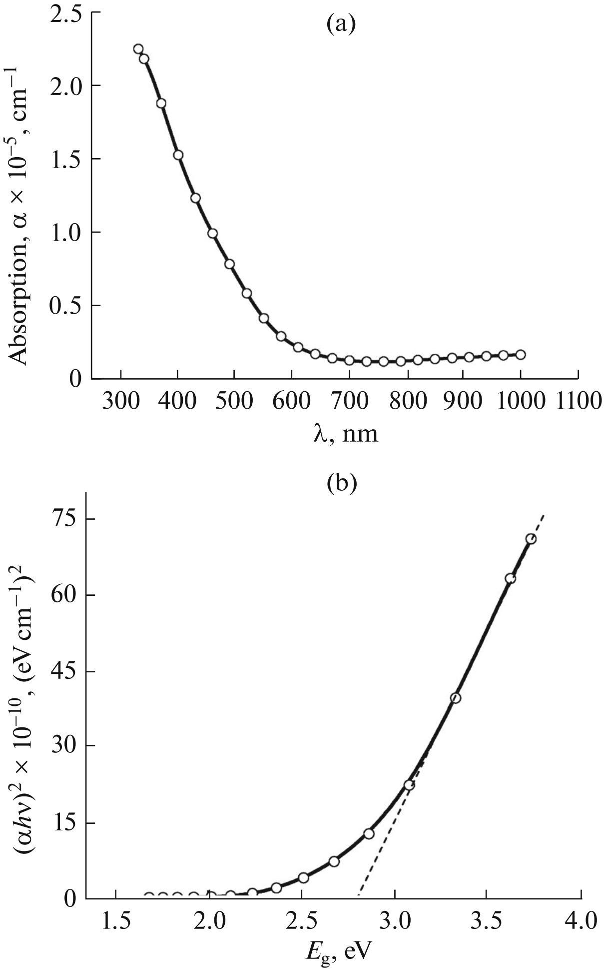

Fig. 6. (a) Absorption spectrum of the as-deposited indium(III) selenide film obtained at 353 K and (b) determination of its optical band gap.

Since indium selenide is a direct gap semiconductor, according to Bardin's relation [12], the absorption coefficient α of a material is related to the incident photon energy $h\nu$ by the following equation:

$$\alpha h\nu = A(h\upsilon - E_g)^n, \qquad (4)$$

where A is the coefficient depending on the transmissivity of the film and its thickness d, E_g is the optical band gap, n is the coefficient equal to 0.5 for direct transition in the case of In_2Se_3.

The graphical dependence of $(\alpha h\nu)^2$ on the incident photon energy $h\nu$ was found by Eq. (4). The projection of the tangent to this dependence onto the abscissa makes it possible to determine the optical band gap E_g of the film (Fig. 6b). For as-deposited films, E_g is 2.8 eV. This value is close to the E_g values obtained in [7, 14] for thin-film In_2Se_3: 1.96 and 2.5 eV, respectively. The larger optical band gap as compared with the bulk In_2Se_3 can be explained by the nanocrystalline character of the film with a typical crystallite size of about 50 nm, as well as by the presence in it of wide-band gap oxide phases (according to EDX and XPS), first of all In_2O_3 (E_g = 3.68 eV [32]) and SeO_2 (E_g = 3.77 eV [33]) shifting the absorption edge toward shorter wavelengths.

Thus, thin nanocrystalline films of In_2Se_3 up to 300 nm thick with good adhesion to glass ceramic, glass, and molybdenum substrates have been prepared for the first time by hydrochemical deposition in the $In(NO_3)_3$–$C_4O_6H_6$–$CSeN_2H_4$ system in the temperature range 353–363 K.

According to XPS data, surface layers of In_2Se_3 films contain up to 50.0 at % indium and 41.8 at % selenium. In addition, the films contain metal oxide phases with an oxygen content of up to 2.5 at % and organic contaminants.

The EDX analysis has confirmed the formation of the thin-film indium(III) selenide phase with the average indium and selenium content of 39.3 and 60.7 at %, respectively.

SEM studies have revealed the nanocrystalline nature of the chemically deposited indium selenide films. Increasing synthesis temperature from 353 to 363 K leads to an increase in the size of nanocrystallites composing globules from 50 to 70 nm.

According to optical absorption data, the optical band gap E_g of the deposited In_2Se_3 film is 2.8 eV.

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