
Regularities of electrochemical formation of thin CdSe films on Ti surface

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Electrochemical deposition of CdSe films on Ti electrode from acid CdSO₄ and H₂SeO₃ solutions has been investigated by means of linear sweep voltammetry as well as under potentiostatic conditions. It has been found that reduction of Se(IV) and Cd(II) ions yields nanometric layers of CdSe which have semiconductor properties. Successive deposition of Se and Cd monolayers allows obtaining CdSe films of a desirable thickness, which yield a stable photocurrent upon illumination. XPS analysis of the elements distribution in the coating surface has shown that with increasing the thickness a coating becomes inhomogeneous, consisting of separate CdSe, Cd(OH)₂, Cd and Se phases.

Key words: CdSe, thin films, electrochemical deposition, Ti electrode, semiconductor properties

INTRODUCTION

Materials synthesized from the elements of II and VI groups, such as CdSe, are characterized by their semiconductor properties which are of broad-based practical importance in modern technology. Various methods such as vacuum vapor deposition, chemical deposition, vapor condensation, etc., are employed to form thin films of such materials. All these methods are expensive and do not ensure the stability of the coating composition which is of great importance for the semiconductor properties of the coating. There is a simpler and rather cheap method of electrochemical formation of thin CdSe films. The coatings can be deposited from various aqueous and non-aqueous, boiling or room temperature solutions [1–10] on various substrates (Ti [8], Cd [2, 11], In [12], indium-tin oxide (ITO) [13], Au [4], Ag [14]). However, as experiments have shown, the electrochemical way of CdSe thin films formation also involves certain complications. The conditions of film formation strongly depend on the nature of the substrate. In the case of Au and Ag substrates underpotential deposition (UPD) of Cd and Se coatings takes place. Therefore thin Cd (or Se) films of 1–2 monolayer (ML) thickness can be deposited and subsequently coated under UPD conditions with another monolayer of Se (or Cd). Coatings up to 1000 MLs thick may be thus formed [4]. Such a method, called electrochemical atomic layer epitaxy (ECALE), allows obtaining coatings of stable compo-

sition characterized by good opti-electronic properties. However, it turned out that the surface of the substrate is not evenly coated and with increase in coating thickness its elemental composition changes. Besides, the equipment required for deposition of such coating is rather complex [4]. In [15] it has been shown that reduction of Se(IV) on graphite is facile, whereas on glassy carbon it is hindered. Various Se compounds formed during the electrode processes destabilize the solution, and the precipitation of Se⁰ occurs [16].

The object of the present study was to investigate the regularities of electrochemical formation of thin CdSe films on Ti substrate. The Ti electrode has been chosen as inert, conductive substrate, because it does not oxidize electrochemically in the potential (*E*) range of the coatings formation.

EXPERIMENTAL

The solutions were made from ultra-pure reagents and tridistilled water. A 10 cm³ cell connected with three closed vessels which allowed to change the solution in the cell without oxygen penetrating into the system, was used for voltammetric measurements. A Ti plate of 1 cm² geometric area served as the cathode and Ni plate of 2.8 cm² area coated with 5 μm thick gold coating was used as the anode. A reversible hydrogen electrode (RHE) in 0.1 M H₂SO₄ was used as the reference. All the potentials in the paper are referred to RHE. Prior to the measure-

ments the solutions were purged with pure Ar for *ca.* 30 min to remove oxygen. The measurements were carried out at 20 °C. A P-5848 potentiostat was used for voltammetric studies. The voltammograms were recorded with a H-307/1 recorder.

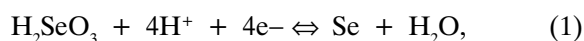
X-ray photoelectron spectroscopy (XPS) was employed to study the distribution of elements in the coating surface. The spectra were recorded with an ESCALAB MK II spectrometer (VS Scientific, UK). The spectrometer had a base pressure of 10^{-13} MPa in the analyzer chamber and pass energy of 20 eV. When investigating the samples the spectra of Cd 3d_{5/2} and 3d_{3/2}, Se 3d, O 1s and Ti 2p were recorded. The qualitative evaluation of the content of elements in the coating was based on the evaluation of peaks area, taking into account the sensitivity factors from [17]. The Auger peaks recorded were compared to standard spectra [18], which allowed identifying surface compounds according to binding energy (ϵ_b) values. No less than 3 spectra were recorded for each element. The accuracy of ϵ_b evaluation was ± 0.1 eV. The sensitivity of the method was 0.1 at. %. The samples for analysis were coated with 100 MLs of Se + 100 MLs of Cd.

Photoelectric investigations of the CdSe coating were performed in a quartz glass cell. The samples were illuminated with polychromatic light of 90 mW cm⁻² intensity. The method is described in [14].

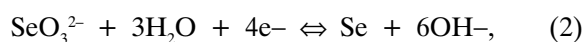
RESULTS AND DISCUSSION

On the basis of literature data [16, 19] acid CdSO₄ and H₂SeO₃ solutions were chosen for CdSe films formation on Ti and 0.1 M K₂SO₄ was used as a supporting electrolyte.

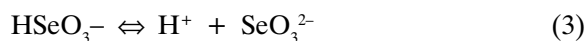
Figure. 1a shows cathodic voltammograms of the Ti electrode in 0.01 M H₂SeO₃ + + 0.1 M K₂SO₄, pH 2.4. One may see that there are two current peaks in the curves. The first one is observed at -0.3 V – -0.36 V depending on the potential scan rate ν . This peak may be ascribed to the following reactions:



or



Since $E^\circ \text{Se}/\text{H}_2\text{SeO}_3 = 0.744$ V and $E^\circ \text{Se}/\text{SeO}_3^{2-} = -0.366$ V [20]. One can see that reaction (1) proceeds at a large overpotential, therefore we have presumed that formation of Se⁰ takes place according to reaction (2) preceded by chemical step (3):



The latter reaction is fast and does not influence the rate of the overall process. The above presumption has been confirmed experimentally: if the potential scan is stopped for 1 min within the *E* range of the first current peak, a red Se coating is deposited on the electrode. When the potential is scanned further into the *E* range of the second current peak, *i.e.* -0.75 V – -0.9 V, the electrode surface turns lighter and a red precipitate begins to form at the electrode/solution interface. When the potential scan is stopped within the *E* range of the second current peak, the red precipitate continues to form and falls to the bottom of the cell. The latter peak has a rather broad maximum. There are two electrochemical reactions that may take place within the *E* range of interest:

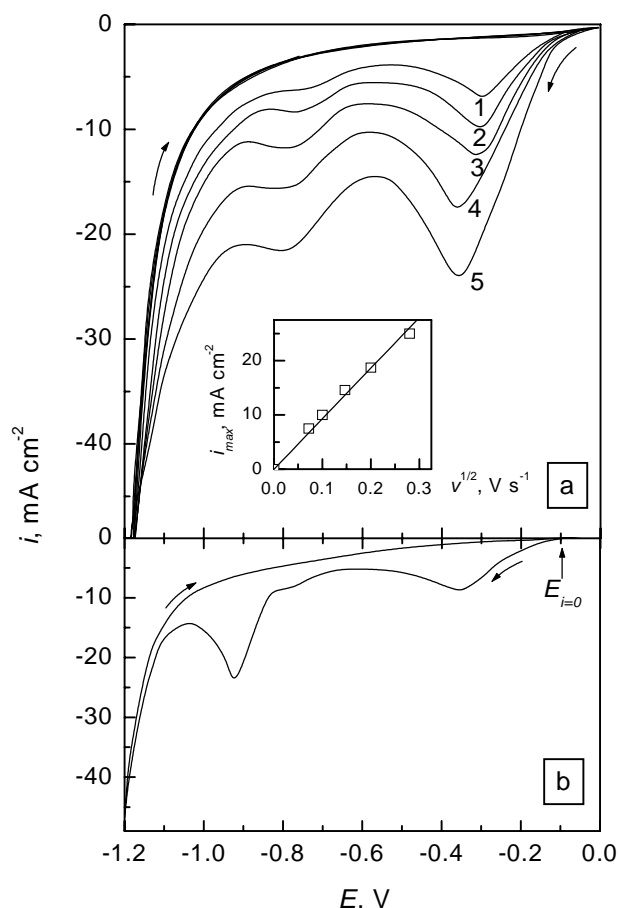


Fig. 1. Voltammograms of Ti electrode in 0.01 M H₂SeO₃ + + 0.1 M K₂SO₄, pH 2.4; 20 °C: a) dependence on potential scan rate, ν : 1–5 – 5; 10; 20; 40; 80 mV s⁻¹, respectively; inset – dependence $i_{max} - \sqrt{\nu}$ of the first current peak; b) the curve recorded after the electrode has been maintained at -0.8 V for 3 min, 10 mV s⁻¹

Standard potentials, E° , of the above reactions are -0.51 V and -0.92 V, respectively. In the course of reactions (1) or (2) the pH at the electrode surface increases and the rate of reaction (4) should decrease with an increase in the amount of Se^0 deposited, whereas the rate of reaction (5) should increase. Figure 1b shows that when the electrode is kept at -0.8 V for 3 min and then the potential scan is initiated promptly beginning at $E_{i=0}$, a distinct current peak appears in the voltammograms at -0.88 V. In our opinion, this may be attributed to reduction of Se^0 according to reaction (5). When Se^{2-} ions are formed, the following chemical reaction may occur in the solution:



The Se^0 formed during reaction (6) is seen as a red precipitate at the electrode/solution interface. Similar phenomena have been observed in the case of Au electrode [16, 19].

As ν increases, so does the current peak representing Se(IV) reduction (reaction (2)). As Fig. 1a shows, the $\sqrt{\nu}$ dependence of peak current i_{max} is linear and goes through the beginning of the coordinates. The diffusion coefficient D of the selenite ion calculated according to relation $i_{max} = f(\sqrt{\nu})$ given in [21] is $5.7 \cdot 10^{-6}$ cm²/s. The latter value is close, though slightly less than the diffusion coefficients of simple ions. This suggests the rate of Se(IV) reduction to be diffusion-limited. The rate of further Se^0 reduction is determined by the pH at the electrode/solution interface.

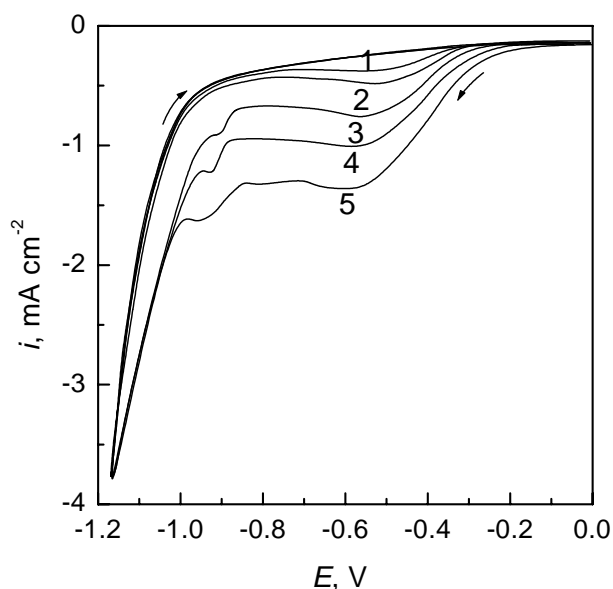


Fig. 2. Voltammograms of Ti electrode in 0.01 M H_2SeO_3 + 0.1 M K_2SO_4 , pH 4.1, 20 °C: dependence on potential scan rate, ν : 1–5 – 5; 10; 20; 40; 80 mV s⁻¹, respectively

As the pH of the solution increases up to 4.1, the shape of the voltammograms changes (Fig. 2). The rate of Se(IV) reduction decreases and so does the first current peak, a limiting current appears at more negative potentials, whereas the current peak representing reaction (5) remains at ca. -0.9 V. Such an influence of increase in solution pH confirms the above presumption about the route of cathodic process.

Figure 3 shows the anodic and cathodic voltammograms of Ti electrode in 0.01 M CdSO_4 + 0.1 M K_2SO_4 solution, pH 2.4. The current peak at -0.4 V – -0.45 V in cathodic curves may be attributed to the reaction



since $E^\circ_{\text{Cd}/\text{Cd}^{2+}} = -0.403$ V [20]. The anodic curves exhibit a current peak representing the process of Cd ionization. The rate of Cd^{2+} reduction increases with increase in ν . As Fig. 3 shows, the dependence $i_{max} - \sqrt{\nu}$ is linear and goes through the beginning of the coordinates. This suggests the rate of Cd^{2+} reduction to be diffusion-limited. The diffusion coefficient of Cd^{2+} ions was calculated to be $5.8 \cdot 10^{-6}$ cm²/s. The latter value is slightly less than D found for Cd^{2+} ions in acid solutions in [22].

When CdSe is deposited from 0.01 M H_2SeO_3 + 0.01 M CdSO_4 + 0.1 M K_2SO_4 solution, pH 2.4, there are three current peaks observed in voltammograms (Fig. 4). The peak potential E_{max} of the first one is close to E° of reaction (2) and therefore the peak may be ascribed to Se^0 formation. E_{max} of the second

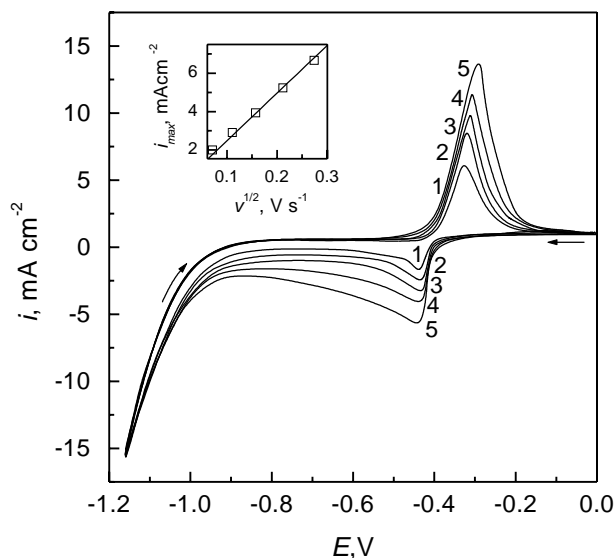


Fig 3. Voltammograms of Ti electrode in 0.01 M CdSO_4 + 0.1 M K_2SO_4 pH 2.4, 20 °C: dependence on potential scan rate, ν : 1–5 – 5; 10; 20; 40; 80 mV s⁻¹, respectively. Inset: dependence $i_{max} - \sqrt{\nu}$

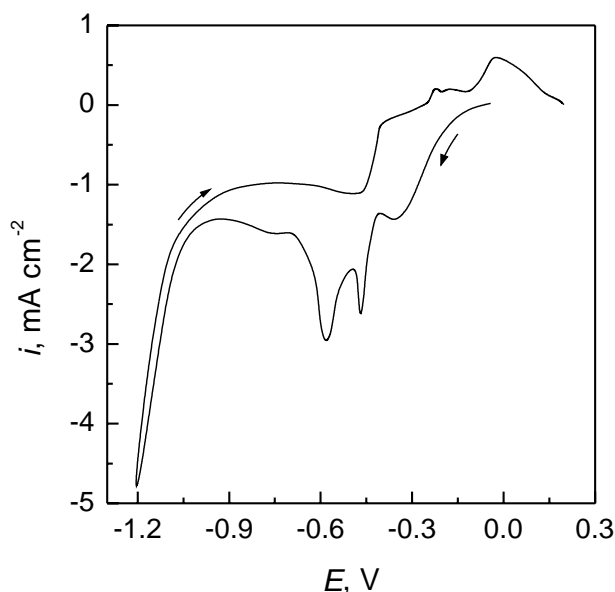


Fig. 4. Voltammogram of Ti electrode in 0.01 M CdSO₄ + 0.01 M H₂SeO₃ + 0.1 M K₂SO₄, pH 2.4, $\nu = 10 \text{ mV s}^{-1}$; 20 °C.

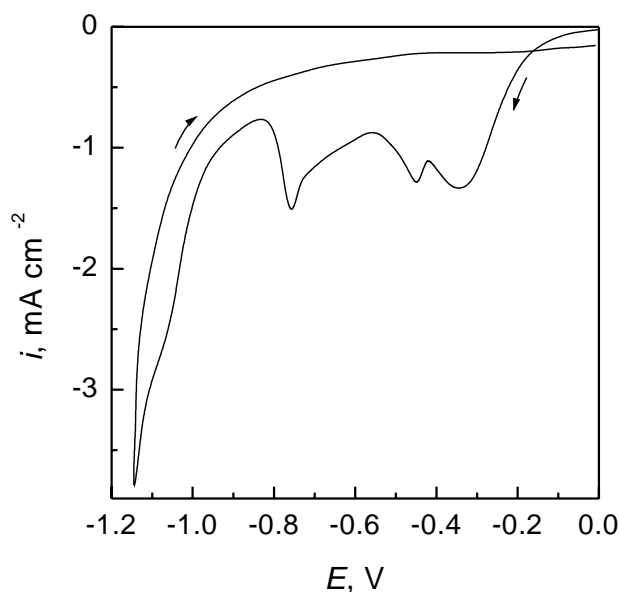


Fig. 6. Voltammogram of Ti electrode in 0.001 M CdSO₄ + 0.01 M H₂SeO₃ + 0.1 M K₂SO₄, pH 2.4, $\nu = 10 \text{ mV s}^{-1}$; 20 °C.

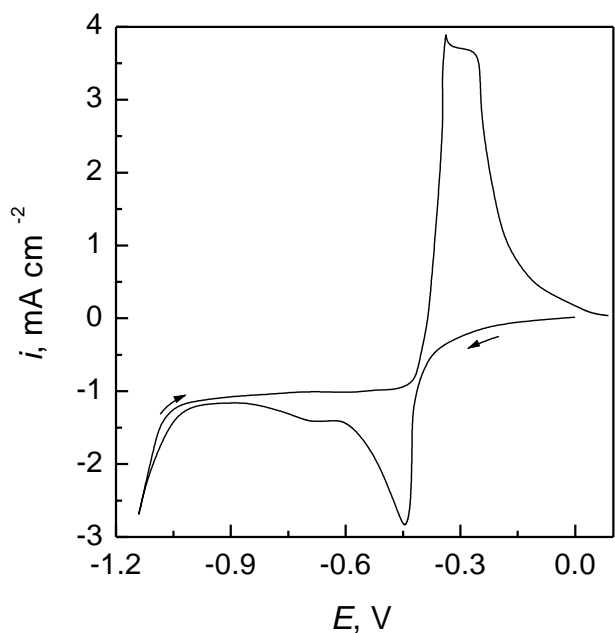
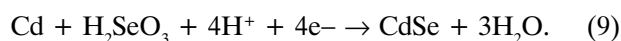
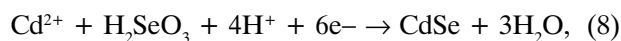


Fig. 5. Voltammogram of Ti electrode in 0.01 M CdSO₄ + 0.001 M H₂SeO₃ + 0.1 M K₂SO₄, pH 2.4, $\nu = 10 \text{ mV s}^{-1}$; 20 °C.

peak is close to the potential of Cd²⁺ electroreduction. The nature of the third current peak is unclear. When the concentration of H₂SeO₃ is reduced tenfold, the shape of voltammograms changes completely (Fig. 5). The first and the third current peaks disappear, whereas the second one increases. When the potential is scanned towards positive potentials, the process of Cd ionization becomes evident. These facts indicate that the second and third current peaks are related to electrochemical processes of selenium.

Figure 6 represents voltammograms of Ti electrode in 0.01 M H₂SeO₃ + 0.001 M CdSO₄ + 0.1 M K₂SO₄ solution, pH 2.4. As one can see, the first current peak related to Se(IV) reduction to Se⁰ reappears in the voltammograms. The current peak of Cd²⁺ reduction decreases and the current peak related to Se⁰ reduction to Se²⁻ appears. The diagram of the system Cd–Se/H₂O presented in [23] shows that in acid solutions CdSe is stable over a wide range of potentials (from –1.1 V to –0.7 V). This suggests that the first process taking place in solutions containing equivalent concentrations of Cd(II) and Se(IV) is reduction of Se(IV) to Se⁰. As the potential is scanned towards more negative values, the potential of Cd²⁺ reduction is reached and deposition of Cd on Se occurs to yield CdSe. Se⁰ is gradually consumed for CdSe formation and the peak of Se⁰ reduction disappears. As indicated in [24], when the potential grows more negative, a direct formation of cadmium selenide according to the following reactions becomes possible:



In the case of Au electrode reaction (8) may occur at –0.625 V. In our opinion, reaction (9) is more likely to occur, since data presented in [25] show that the composition of the coatings is potential-dependent. Initially the coatings with a high content of Se are deposited. The coatings deposited within the *E* range of the second current peak con-

tain about 80% of CdSe. The content of Cd in the coating increases at more negative potentials, because the contribution of reaction (2) to the overall rate of cathodic process decreases. Therefore the coatings of satisfactory composition may be deposited within a narrow range of potentials.

A rather promising way of CdSe coatings deposition has been proposed in [19]. Se coatings of a certain thickness were deposited on Au electrode and subsequently coated with a corresponding amount of Cd coating. Such a way of coating formation prevents side reaction (6) and allows obtaining coatings of definite composition and thickness.

Taking into account that the radius of Se atom is 0.16 nm [26] and 4 electrons are required to reduce one Se(IV) ion and assuming that the factor of Ti electrode surface roughness is 3 [27], it turns out that 1.87 mC cm⁻² are required to deposit one monolayer of Se. The radius of Cd atom is 0.156 [26] and 2 electrons are required to reduce one Cd²⁺ ion, consequently the amount of charge consumed for the formation of one Cd monolayer would be 1 mC cm⁻². Up to the thickness of 10 MLs of Se on Ti substrate the coating is reddish brown and its adhesion is good. As the thickness increases up to 50 MLs of Se, the coating turns bluish gray. With a further increase in coating thickness it turns lighter up to becoming yellow. In literature the formation of coatings of different color is ascribed to different activity of the coating in regard to reaction with Cd. In our opinion, the color of the coating is thickness-dependent.

Formation of CdSe was investigated in the following way: a Se coating 10 MLs thick was formed and then a certain amount of Cd was deposited on it. Curves presented in Fig. 7 show that when 5 or 10 MLs of Cd are deposited on 10 MLs of Se, there is no indication of free Cd phase dissolution when the potential is scanned into positive direction (Fig. 7 a, b). When the thickness of Cd coating is increased up to 15 MLs, the anodic current related to Cd dissolution appears (Fig. 7, c). From these results it follows that under study conditions CdSe coatings of stable composition are formed. However, when the thickness of Se coating is increased up to 100 MLs and 100 MLs of Cd are deposited on the top of it, a distinct wave of Cd dissolution appears in the voltammograms (Fig. 8). This suggests that in the case of thick Se and Cd layers the formation of CdSe takes place within a limited depth of the coating.

The results of XPS investigation of the coating composition are presented in Table. The data obtained indicate that the composition of the coatings consisting of 100 MLs of Se + 100 MLs of Cd (samples 1, 2) as well as of 200 MLs of Se + 200 MLs of Cd (samples 3, 4) depends on the conditions of their for-

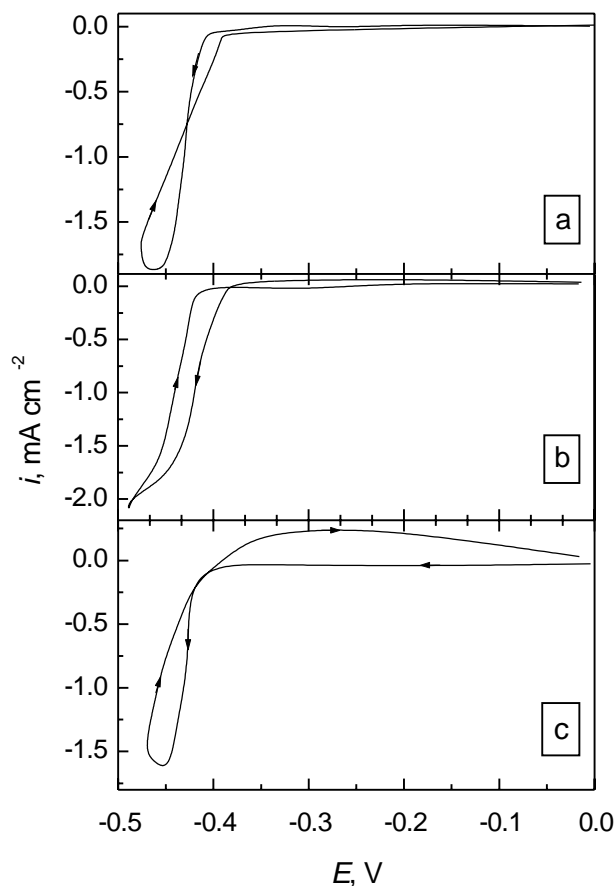


Fig. 7. Voltammograms of Ti electrode coated with 10 monolayers of Se in 0.01 M CdSO₄ + 0.01M K₂SO₄, pH 2.4. Dependence on the Cd coating thickness (in MLs): a - 5; b - 10; c - 15, $\nu = 10 \text{ mV s}^{-1}$; 20 °C

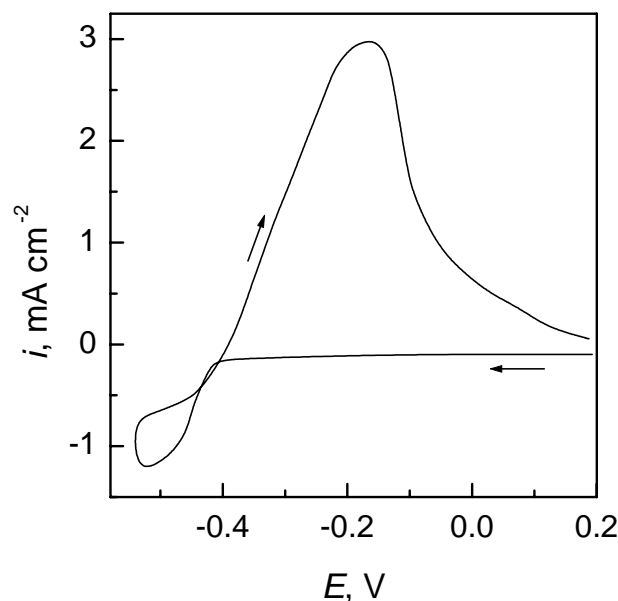


Fig. 8. Voltammogram of Ti electrode coated with 100 MLs Se + 100 MLs Cd in 0.01 M CdSO₄ + 0.1 M K₂SO₄, pH 2.4; $\nu = 10 \text{ mV s}^{-1}$; 20 °C

Table 1. Results of XPS investigation of coatings					
Elements	Content, at. %				Comments
	Sample 1	Sample 2	Sample 3	Sample 4	
Cd	32.9	9.8	24.0	18.0	Samples 1 and 2: 100 MLs Se + 100 MLs Cd Samples 3 and 4: 200 MLs Se + 200 MLs Cd Samples 1, 3 were taken out from the solution after the coating deposition. Samples 2, 4 were anodically polarized after the coating deposition.
Se	38.7	51.5	27.3	47.0	
O	24.4	35.6	43.3	37.9	
Ti	4.0	3.1	49	2.2	
K	–	–	0.5	0.9	

mation rather than on their thickness. Samples 1 and 3 were taken out from the solution immediately after coating formation. In the case of samples 2 and 4, after formation of the coatings they were anodically polarized to $E_{i=0}$. One can see that anodic polarization of the samples reduces relatively the amount of Cd, whereas the amount of Se increases. The oxygen present in the coating comes from $\text{Cd}(\text{OH})_2$ as well as from the adsorbed water. The amount of oxygen increases with an increase in coating thickness. The fact that Ti from the substrate is found in all of the samples shows that electrode coverage with CdSe coating is not even. Moreover, the fact that coating formation takes place not on the whole surface of Ti electrode explains why the calculated values of the diffusion coefficients of Cd^{2+} and SeO_3^{2-} ions are slightly lower

than those given in literature. Binding energy spectra of Cd and Se are shown in Fig. 9 and 10. The ϵ_b of Cd $3d_{5/2}$ electrons is 404.8 eV [18]. As Fig. 9 shows, in the case of samples 3 and 4 the latter value is 405.4 eV. Besides, the full-width-at-half-maximum (FWHM) of the peak is larger than in the case of pure Cd. This indicates that the coating consists of a mixture of pure Cd, CdSe and $\text{Cd}(\text{OH})_2$. The ϵ_b of Se 3d electrons is 55.3 eV. The results obtained show that the coating consists of CdSe and pure Se.

All of the data obtained indicate that in the case of thicker CdSe films, the coatings are inhomogeneous. To make a coating homogeneous, it should be heated at 300–500 °C.

Photoelectrochemical properties of the coatings have been investigated in the solution of 1.5 M Na_2SO_3 . The coatings consisting of 100 MLs Se + 100 MLs Cd

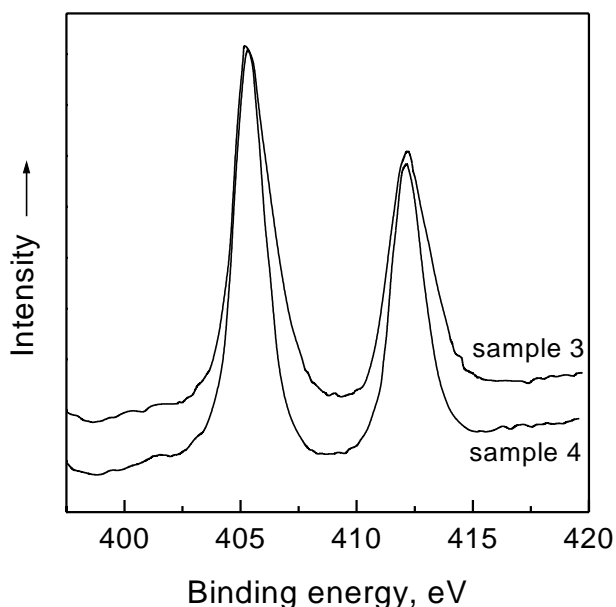


Fig. 9. XPS spectra of Cd $3d_{5/2}$ and $3d_{3/2}$ electrons. The samples were prepared by means of electrochemical deposition of 200 MLs Se + 200 MLs Cd on Ti substrate. Sample 3 was taken out from solution immediately after coating deposition; sample 4 was polarized anodically after coating deposition

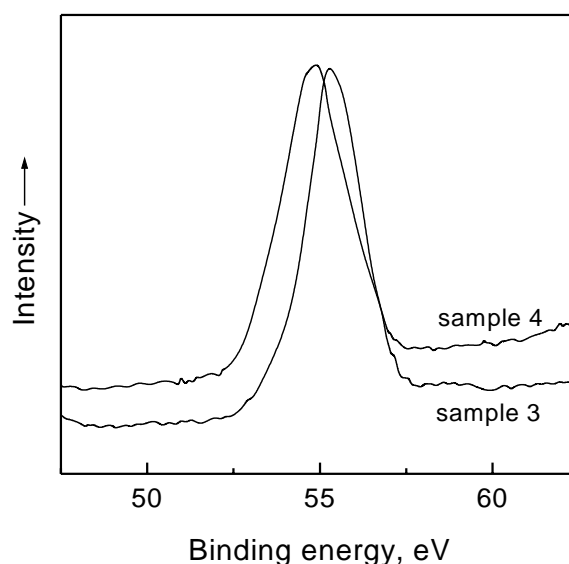


Fig. 10. XPS spectra of Se 3d electrons. The samples were prepared by means of electrochemical deposition of 200 MLs Se + 200 MLs Cd on Ti substrate. Sample 3 was taken out from solution immediately after coating deposition; sample 4 was polarized anodically after coating deposition

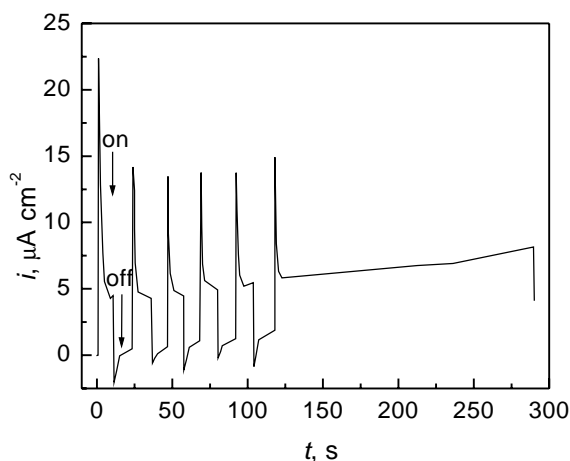


Fig. 11. Photocurrent dependence on illumination ($N = 90 \text{ mW cm}^{-2}$) under open circuit conditions ($E \approx -0.55 \text{ V}$). Sample – 100 MLs Se + 100 MLs Cd on Ti substrate; solution 1.5 M Na_2SO_3 ; 20°C

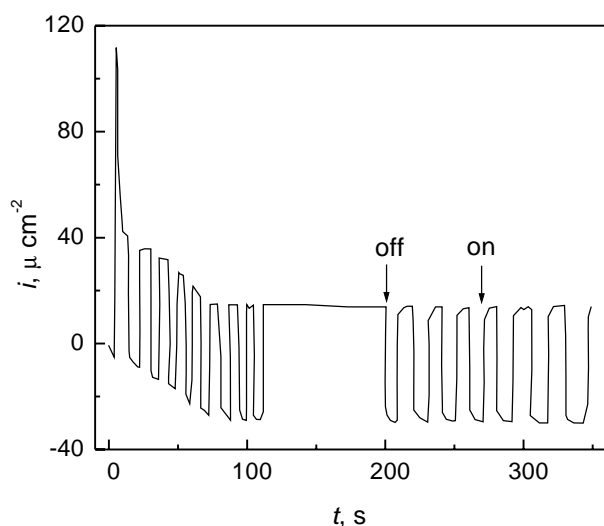


Fig. 12. Photocurrent dependence on illumination ($N = 90 \text{ mW cm}^{-2}$) at $E \approx 0 \text{ V}$. Sample – 100 MLs Se + 100 MLs Cd on Ti substrate; solution 1.5 M Na_2SO_3 ; 20°C

were illuminated with polychromatic light with intensity of 90 mW cm^{-2} . Figure 11 shows that when the electrode is illuminated under open circuit conditions at $E = -0.55 \text{ V}$, a current of $\sim 5 \mu\text{A cm}^{-2}$ is generated and remains constant for several minutes. At $E = -0.2 \text{ V}$ a current of $\sim 50 \mu\text{A cm}^{-2}$ is generated and remains constant for $> 5 \text{ min}$ (Fig. 12). When the potential is scanned into positive direction at 10 mV s^{-1} , photoelectric current of increasing strength is observed (Fig. 13). The fact that the anodic photocurrent is generated upon illumination indicates the coating to be a semiconductor of n-type. Such is CdSe. Thus photoelectrochemical investigations confirm the conclusion about the formation of a semiconductive layer of CdSe on the Ti electrode.

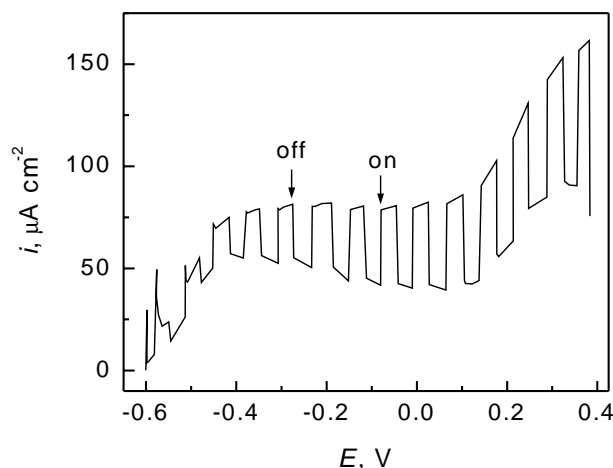


Fig. 13. Photocurrent dependence on illumination ($N = 90 \text{ mW cm}^{-2}$) when potential is scanned from -0.6 V to 0.4 V at $\nu = 10 \text{ mVs}^{-1}$. Sample – 100 MLs Se + 100 MLs Cd on Ti substrate; solution 1.5 M Na_2SO_3 ; 20°C

CONCLUSIONS

Qualitative electrochemical deposition of Se and Cd monolayers on Ti electrode allows obtaining n-type semiconductive CdSe films with stable photoelectric properties. With increase in film thickness its homogeneity decreases as a result of the formation of pure Cd and pure Se phases.

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PLONŲ CDSE SLUOKSNIŲ ELEKTROCHEMINIO NUSODINIMO ANT TI PAVIRŠIAUS DĖSNINGUMAI

S a n t r a u k a

Darbe tirtas plonų CdSe sluoksnių elektrocheminio nusodinimo procesas iš rūgščių CdSO₄ ir H₂SeO₃ tirpalų ant Ti elektrodo potenciodinaminėmis ir potenciostatinėmis sąlygomis. Nustatyta, kad redukuojantis Se(IV) ir Cd(II) jonams ant Ti paviršiaus formuojasi nanometriniai CdSe sluoksniai, pasižymintys puslaidininkinėmis savybėmis. Atskirai nusodinant Se ir Cd monosluoksnius galima gauti norimo storio CdSe dangas, kurias apšvietus generuojama pastovi fotosrovė. Ištyrus elementų pasiskirstymą dangoje rentgeno fotoelektroninės spektroskopijos metodu nustatyta, kad, dangai storėjant ji tampa nevienalytė, sudaryta iš atskirų CdSe, Cd(OH)₂, Cd ir Se fazių.