

## Electrochemical deposition of cadmium telluride films

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The mechanisms of electrochemical processes in the electrolyte for cadmium telluride semi-conducting film deposition and in corresponding partial solutions containing cadmium and tellurium ions have been studied by voltammetry with linear potential scanning and by cyclic voltammetry. The differences between cathodic processes on chemically inert titanium nitride substrates and catalytically active molybdenum surface have been revealed. Basing on these studies, the nature of impurities discovered in this work by X-ray photoelectron spectroscopy at the surface and inside the cadmium telluride films deposited on molybdenum and titanium nitride surfaces has been explained. The latter made it possible to select the electrodeposition parameters and the substrate material which provide the stoichiometric film composition.

Методами хроновольтамперометрии и циклической вольтамперометрии исследованы механизмы электрохимических процессов в электролите для получения полупроводниковых пленок теллурида кадмия и в соответствующих ему парциальных растворах, содержащих ионы кадмия и теллура. Обнаружены различия в катодных процессах на химически инертных подложках нитрида титана и на каталитически активной поверхности молибдена. На основании проведенных исследований объяснена природа примесей, обнаруженных в работе методом рентгеновской фотоэлектронной спектроскопии на поверхности и в объеме пленок теллурида кадмия, электроосажденных на подложки из нитрида титана и молибдена, что позволило осуществить выбор параметров процесса электроосаждения и материала подложек, которые обеспечивают изготовление пленок стехиометрического состава.

Electrochemical deposition technique provides an excellent control of the film properties by tuning such parameters as cathode potential, temperature, pH and composition of the reagents [1]. At the same time, no sufficient attention was paid yet to the scientific approach to selection of the electrochemical regimes for obtaining of stoichiometric cadmium telluride layers. As a consequence, the empirical selection of the potentials for the CdTe electrodeposition

onto various substrates has been proposed [2–7]. In our opinion, the scientific solution of this problem should consist in immediate investigation of mechanisms of processes taking place on the cathodes made from various materials and in near-cathode space during cadmium telluride electrodeposition, i.e. the studying of CdTe electrodeposition as depending on the substrate material. Our selection of the substrate materials is explained, at the one hand, by the most often

utilization of molybdenum (Mo) as a base for cadmium telluride absorbing layer in photovoltaic devices of substrate configuration [4–6]. At the other hand, titanium nitride (TiN) is chemically inert material and so can be used as control material in experiments to reveal some interactions between CdTe and Mo.

In this work, the CdTe electrodeposition kinetics on Mo and TiN surfaces has been studied using a potentiostat by means of voltammetry with linear potential scanning (VA) and by cyclic voltammetry (CVA) [8, 9]. During the studies, glass plates of 2 cm<sup>2</sup> area covered by reactively sputtered 500 nm thick titanium nitride or by magnetron sputtered 1 μm thick molybdenum layers having the layer resistance less than 1 Ohm were used as cathodes in three-electrode electrochemical cell. As the reference electrode, saturated Ag/AgCl electrode (SAE) ( $U_{SAE} = 0.22$  V respective to normal hydrogen electrode) was used while as the counter electrode, a platinum coil of 30 cm<sup>2</sup> area. The electrolyte temperature was supported at 50°C using a thermostat. The solutions were preserved from mixing (except for specially mentioned cases). To avoid the migration during the VA and CVA experiments, all solutions contained 0.1 M Na<sub>2</sub>SO<sub>4</sub>. Electrochemical processes were studied in the following media: (i) a solution corresponding to electrolyte for cadmium telluride deposition containing 0.5 M CdSO<sub>4</sub>, 0.0002 M TeO<sub>2</sub>, 0.1 M Na<sub>2</sub>SO<sub>4</sub>, 0.01 M H<sub>2</sub>SO<sub>4</sub>; (ii) partial cadmium solution: 0.5 M CdSO<sub>4</sub>, 0.1 M Na<sub>2</sub>SO<sub>4</sub>, 0.01 M H<sub>2</sub>SO<sub>4</sub>; (iii) partial tellurium solution: 0.0002 M TeO<sub>2</sub>, 0.1 M Na<sub>2</sub>SO<sub>4</sub>, 0.01 M H<sub>2</sub>SO<sub>4</sub>; (iv) background solution: 0.1 M Na<sub>2</sub>SO<sub>4</sub>, 0.01 M H<sub>2</sub>SO<sub>4</sub>. The polarization voltage scanning rates (V) were from 2 mV/s up to 500 mV/s, the range of scanning potentials  $E_c$  was from 0.2 V up to -1.4 V (here and below, all potentials are indicated with respect to SAE).

The cadmium telluride films onto TiN substrates (sample Nos. 1, 2, 3) and onto molybdenum substrates (sample Nos. 4, 5, 6) were electrodeposited in potentiostatic regime in the same three-electrode cell, but using stirring of the electrolyte (0.5 M CdSO<sub>4</sub>, 0.0002 M TeO<sub>2</sub>, 0.01 M H<sub>2</sub>SO<sub>4</sub>) by means of magnetic stirrer. The electrolyte temperature was 50°C, the electrodeposition duration was 1 hour. The cathode potentials during the film electrodeposition were supported at the following levels:  $E_c = -0.40$  V (sample No.1),  $E_c = -0.45$  V (sample Nos.2

and 4),  $E_c = -0.55$  V (sample No.5) or  $E_c = -0.65$  V (sample Nos.3 and 6), the current density  $j_c$  being regularly decreased due to increasing CdTe layer resistivity.

The sample surface composition was studied by X-ray photoelectron spectroscopy (XPS) using an XPS-800 Kratos spectrometer. The vacuum in the measuring chamber was 5·10<sup>-8</sup> Torr. Photoelectrons were excited by MgK<sub>α</sub> radiation ( $h\gamma = 1253.6$  eV). X-ray gun power was 15kV×20 mA. The hemispherical electrostatic analyzer was used to analyze the photoelectron kinetic energy. The instrument resolution was 1 eV, the binding energy was determined at an accuracy of ±0.3 eV. The spectra were processed using a computer: smoothing, subtraction of a constant and variable background (Shirley method), removal of the expanding effect of the X-ray line (iterative deconvolution) and expansion of complex line into its components were done. The sample surface composition was determined using ratio of areas under photoelectron lines of C1s, O1s, Cd3d, Te3d, Ti2p, Mo3d, Si2p core levels, taking into account their sensitivity factors [12]. A layer of about 5 nm thickness was analyzed. The compounds present in the subsurface layer of the samples were analyzed using expansion of the complex Te3d line into its components. The layer-by-layer etching of the sample surfaces was carried out using an ionic gun (Ar<sup>+</sup>,  $E = 2$  keV), the etching rate being 1 nm/min.

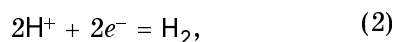
The studies have shown that in background solution two current peaks are revealed on cathodic branches of cyclic voltammograms (I-V curves) obtained when titanium nitride was used as the working electrode (Fig. 1). The small current peak shifts towards negative potentials as the potential scanning rate grows from 2 mV/s up to 500 mV/s, thus, the cathode half-peak potential  $E_{cp/2}$  changes from -0.4 V to -0.6 V. The differences between cathodic potentials of peaks and cathodic half-peak potentials are rather large. These differences change depending on  $V$ , but are about 0.1 V, thus exceeding more than thrice the value for reversible process. That peak is absent in the anodic branches of cyclic voltammograms in background solution.

All the above evidences that an irreversible electrochemical process complicated by following fast chemical dimerization reaction runs in background electrolyte at potentials near the cathodic current peak. For reversible electrochemical process, the half-

peak potential  $E_{cp/2}$  and standard potential  $E_c^0$  of the electrochemical cathodic reaction are connected by relation [8, 9]:

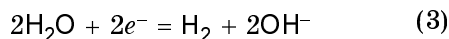
$$E_{cp/2} = E_c^0 + 1.09(RT/nF), \quad (1)$$

where  $R$  is the gas constant;  $T$ , the solution absolute temperature;  $n$ , number of electrons involved in electrochemical reaction;  $F$ , the Faraday number. Taking that fact into account, we can calculate that at 50°C half-peak potential for reversible cathodic process would be by 31/ $n$  mV less negative than the standard potential. Then, taking into account that the half-peak potential for the irreversible electrode process followed by chemical dimerization reaction is always shifted to the negative values as compared to the reversible process [8, 9] and in accordance with the standard potential data [10, 11], we can conclude that the first peak in the background solution corresponds to reaction:



which equilibrium cathode potential in this solution is  $-0.33$  V, as calculated using the Nernst equation [10]. The shift of this peak towards negative potentials is due to overvoltage which for irreversible processes followed by dimerization is at least 0.1 V [9]. The intensity of this peak is small, because the solution is slightly acidic (pH 2).

Current growing in the background solution at the potentials more negative than  $-0.85$  V is very intensive. According to our calculations, it corresponds to the water decomposition with the hydrogen creation according to reaction:



with standard potential of  $-1.05$  V [10].

When molybdenum working electrode is used in background solution, the current increase occurs at considerably more positive potentials ( $E_c = -0.5$  V), so the processes (2) and (3) are indistinguishable in the  $I$ - $V$ -curves. According to [8, 9], this electrodeposition process at more positive cathode potentials is associated with catalytic influence of the electrode surface. In this case, molybdenum catalyzes the electrochemical process of water decomposition.

In cadmium partial solution, voltammograms for titanium nitride substrates (Fig. 2) also include a weak hydrogen reduction peak corresponding to reaction (2).

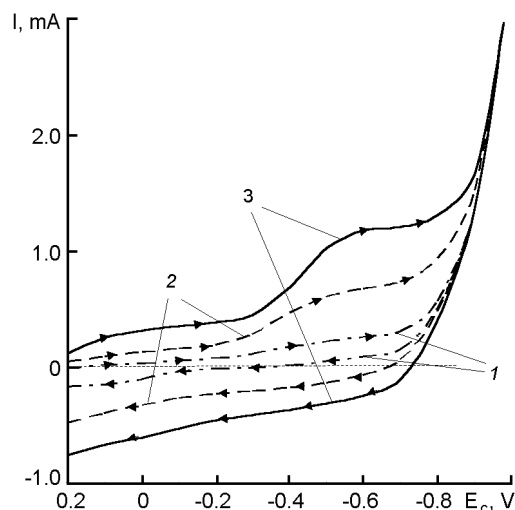
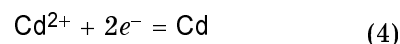


Fig. 1. Cyclic voltammograms on TiN substrates in background solution (0.1 M  $Na_2SO_4$ , 0.01 M  $H_2SO_4$ ) at potential scanning rates (mV/s): 10 (1); 50 (2); 100 (3).

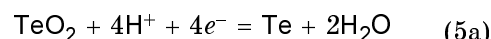
Then, independent from polarization voltage rate, a sharp current rise is seen at potentials more negative than  $-0.65$  V. According to [10, 11], equilibrium potential of the reaction:



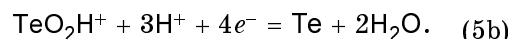
in this solution in accordance with Nernst equation is  $-0.63$  V. So, according to [8, 10], we can conclude that above-mentioned sharp current rise demonstrates the reversible cathodic process of cadmium electrochemical reduction.

The voltammograms in cadmium partial solution for molybdenum substrates differ from similar  $I$ - $V$ -curves for TiN ones only by the current rise at cathode potential  $-0.5$  V (as in the background solution for Mo substrates). That fact is also connected with the catalytic influence of molybdenum surface on the process (3).

The most considerable is difference between the tellurium electrochemical reduction mechanisms on TiN and Mo substrates. The cyclic voltammograms in partial tellurium solution for TiN substrates demonstrate (Fig. 3) electrochemical tellurium reduction according to reaction [10]:



or according to analogous reaction [11]:



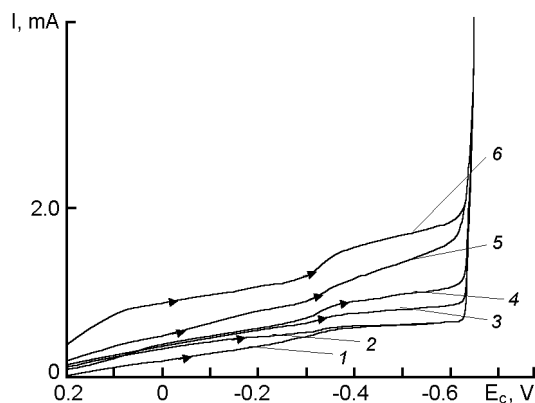


Fig. 2. Voltammograms on TiN substrates in cadmium partial solution (0.5 M  $\text{CdSO}_4$ , 0.1 M  $\text{Na}_2\text{SO}_4$ , 0.01 M  $\text{H}_2\text{SO}_4$ ) at potential scanning rates (mV/s): 5 (1); 10 (2); 20 (3); 50 (4); 100 (5); 200 (6).

For reaction (5a), standard potential  $E^0$  with respect to SAE at 50°C is 0.33 V [10], for reaction (5b),  $E^0 = 0.36$  V [11]. The equilibrium potentials in this solution  $E_{cp}$  are 0.27 V and 0.30 V, respectively. However, as our experiments revealed, the tellurium reduction occurs at more negative potentials than those corresponding to the reversible process. This is due to irreversibility of the electrochemical reduction of tellurium on titanium nitride substrate which is demonstrated by the following. First, when  $V$  rises, the cathodic peak potential ( $E_{cp}$ ) becomes more negative and the anodic peak potential ( $E_{ap}$ ), more positive. Second, the anodic peaks are far lower than cathodic ones. Third, the difference ( $E_{cp/2} - E_{cp}$ ) exceeds 0.1 V while ( $E_{ap} - E_{cp}$ )  $\approx$  0.4 V. So it is not surprising that in our experiments half-peak potentials in cathodic branches of  $I$ - $V$ -curves occurred at potentials changing from -0.12 V (if  $V = 2$  mV/s) to -0.24 V (if  $V = 500$  mV/s). The cathodic current rise in tellurium partial solution on TiN substrate occurs at the same potentials (more negative than -0.85 V) as in background solution. Therefore, it corresponds to water decomposition and hydrogen evolution process according to reaction (3).

Quite other are voltammograms obtained in tellurium partial solution on molybdenum substrate (Fig. 4). The  $I$ - $V$ -curves are characterized by two peaks, moreover, the first peak is at potential  $E_{cp} \approx -0.05$  V, and the second peak is similar to the tellurium reduction peak on cyclic voltammograms on titanium nitride substrates. The second peak is followed by an intense current rise at potentials of molybdenum cathode more negative than -0.5 V, which is

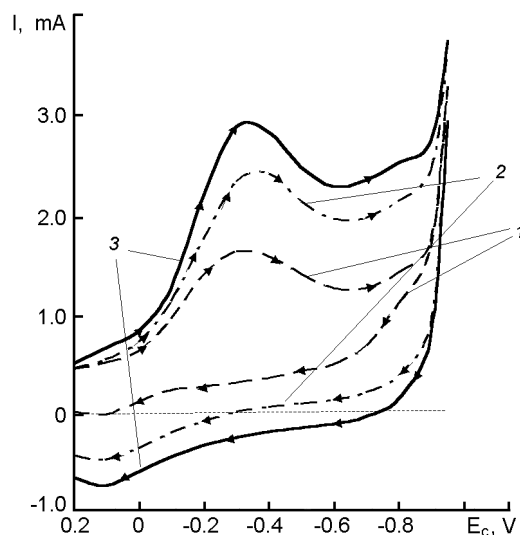


Fig. 3. Cyclic voltammograms on TiN substrates in tellurium partial solution ( $2 \cdot 10^{-4}$  M  $\text{TeO}_2$ , 0.1 M  $\text{Na}_2\text{SO}_4$ , 0.01 M  $\text{H}_2\text{SO}_4$ ) at potential scanning rates (mV/s): 50 (1); 100 (2); 200 (3).

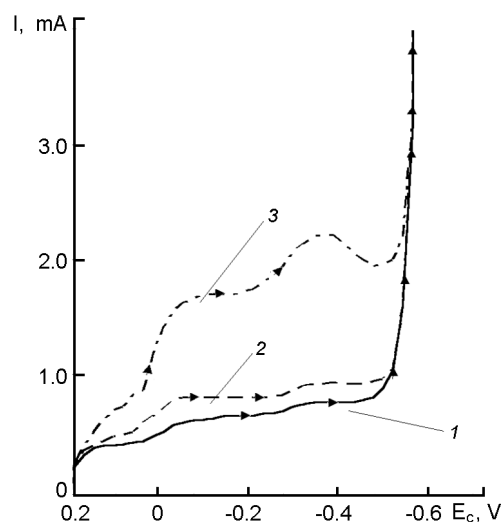


Fig. 4. Voltammograms on Mo substrates in tellurium partial solution ( $2 \cdot 10^{-4}$  M  $\text{TeO}_2$ , 0.1 M  $\text{Na}_2\text{SO}_4$ , 0.01 M  $\text{H}_2\text{SO}_4$ ) at potential scanning rates (mV/s): 1 (1); 10 (2); 100 (3).

related to water decomposition, free hydroxyl groups formation and hydrogen evolution according to reaction (3). The study of  $I$ - $V$ -curve character in partial tellurium solution as depending on the voltage scanning rate for the molybdenum cathode has shown that the additional peak at more positive potentials is connected with strong specific adsorption of the reaction (5) product (tellurium) on the molybdenum surface.

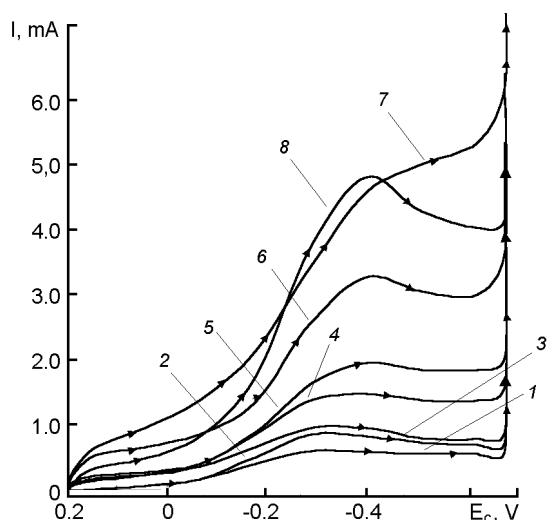
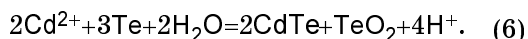


Fig. 5. Voltammograms on TiN substrates in solution corresponding to electrolyte for CdTe electrodeposition (0.5 M CdSO<sub>4</sub>, 2·10<sup>-4</sup> M TeO<sub>2</sub>, 0.1 M Na<sub>2</sub>SO<sub>4</sub>, 0.01 M H<sub>2</sub>SO<sub>4</sub>) at potential scanning rates (mV/s): 5 (1); 10 (2); 20 (3); 50 (4); 100 (5); 200 (6); 500 (7); 200 under electrolyte stirring (8).

As a confirmation for The above can be confirmed by a more sharp increase of the first (absorption) peak height during  $V$  growing as compared to the second peak, which corresponds to irreversible electrochemical process (5), and slight shift of both peaks towards negative potentials at increasing  $V$ .

The cathodic  $I$ - $V$ -curves in solution corresponding to the electrolyte for cadmium telluride electrodeposition on titanium nitride substrates (Fig. 5) correspond to well known [8, 9] regularities for electrode processes followed by chemical disproportioning reactions, namely, the current peak  $I_P$  is proportional to  $V^{1/2}$  and peak potential  $E_{cp}$  is proportional to  $\lg I/V$ . So in the electrolyte for cadmium telluride filmdeposition, the cathodic process of tellurium electrochemical reduction according to reaction (5) is followed by slow chemical reaction of elemental tellurium disproportioning. Moreover, since in the case of the cadmium ions absence  $I$ - $V$ -curves do not meet the regularities  $I_{cp} \sim V^{1/2}$  and  $E_{cp} \sim \lg I/V$ , we can conclude that disproportioning reaction involves Cd<sup>2+</sup>, so it can be described by equation:



As a result of reactions (5) and (6), the films electrodeposited on TiN substrate in cathode potentials interval  $E_c = -0.40$ — $-0.65$  V

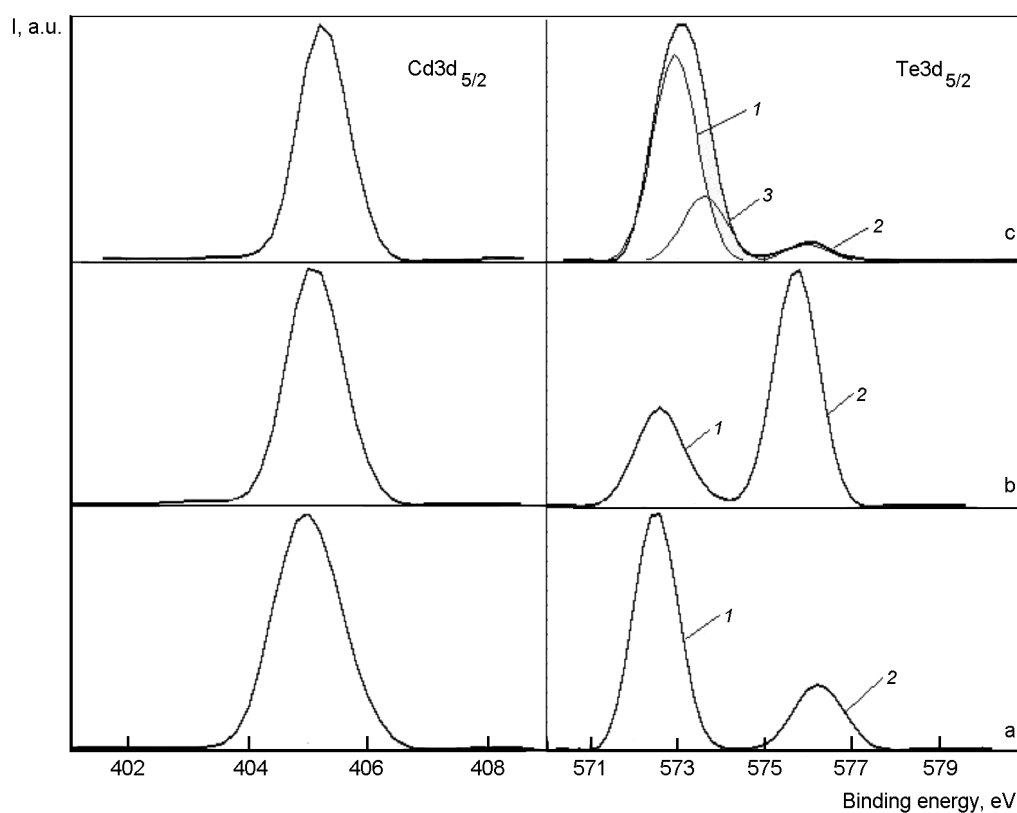
may comprise, along with cadmium telluride, significant amounts of tellurium dioxide. The presence of elemental tellurium in these films can be connected with slow chemical reaction (6). Note that mixing of the electrolyte by a magnetic stirrer results in the regular current increase due to delivery of greater amounts of reagents to the cathode surface, but does not change the number of reduction waves and the potentials at which the reduction processes take place (Fig. 5, curve 8).

As XPS investigations have shown, irrespective of substrate material, the carbon on the surface of cadmium telluride films presents as adsorbed hydrocarbon compounds. Oxygen is in form of TeO<sub>2</sub>, CdO (or Cd(OH)<sub>2</sub>) and O<sub>2</sub> and/or water adsorbed from air. Elements of substrates (Ti, Mo, Si) give very small signals or no signal, because the film completely covers the substrate and is nonporous. The CdTe and TeO<sub>2</sub> signals for the films are very well resolved for Te3d line ( $E_{cb} = 572.6$  eV for CdTe and  $E_{cb} = 576.2$  eV for TeO<sub>2</sub>, according to reference data for these compounds [12, 13]) while the signal for Cd(OH)<sub>2</sub> (or for CdO) is not resolved for Cd3d line but can be calculated from ratio of concentrations Cd/Te (Fig. 6 a,b). The CdTe amounts differ very slightly for different samples, but relative contents of oxides and hydroxides differ to a considerable extent.

According to XPS data, the surfaces of the films electrodeposited on titanium nitride substrates at potentials  $E_c = -0.40$  V (Sample 1) are enriched in tellurium. In the films obtained at  $E_c = -0.45$  V (Sample 2), the tellurium content is somewhat higher than that of cadmium, and the layers deposited at  $E_c = -0.65$  V (Sample 3) have equal amounts of these elements (Table 1). Analysis of X-ray photoelectron spectra of surfaces for the Samples 1, 2 and 3 revealed commensurable concentrations of CdTe, TeO<sub>2</sub> and CdO or Cd(OH)<sub>2</sub> (Table 1). Note that authors [5, 7] also observe large oxygen amounts at the surface of the electrodeposited cadmium telluride films, moreover, it was pointed out that TeO<sub>2</sub> was presented at the film surface. Besides, authors [7] have noted a positive influence of oxygen in the electrodeposited CdTe films on the change of their type of conductivity from n- to p- for their use as base layers of photovoltaic devices. The cadmium hydroxide and/or cadmium oxide impurities revealed by XPS are formed on the surface as a result of air oxidation of cadmium telluride.

Table 1. Electrodeposition regimes, atomic concentrations of elements and chemical compounds on the surface of cadmium telluride samples (XPS data)

Sample No.	Electrodeposition regimes			Atomic concentrations, %				Chemical compounds, %		
	Substrate	Cathode potential, $E_c$ , V (SAE)	Current density, $j_c$ , mA/cm <sup>2</sup>	C	O	Cd	Te	CdTe	TeO <sub>2</sub>	Cd(OH) <sub>2</sub> and/or CdO
1	TiN	-0.40	0.5-0.3	53.4	24.2	8.4	14.0	18	55	27
2	TiN	-0.45	0.5-0.3	58.8	20.1	9.7	11.4	22	42	36
3	TiN	-0.65	1.4-1.0	76.5	11.9	5.8	5.8	24	38	38
4	Mo	-0.45	0.4-0.3	65.5	17.9	7.4	7.2	18	41	41
5	Mo	-0.55	0.9-0.7	79.1	13.7	5.2	1.6	18	10	72
6	Mo	-0.65	1.5-1.2	72.8	17.8	7.4	2.0	19	7	74

Fig. 6. XPS spectra of Cd3d, Te3d-core levels of the surfaces of samples No.6 (a); 4 (b); Sample 1 after removing 200 nm thick layer by ion etching (c): CdTe (1); TeO<sub>2</sub> (2); Te (3).

Besides, the considerable amounts of cadmium hydroxide can be explained by interaction of cadmium ions and hydroxyl groups formed according to reaction (3).

Ion-etching of the surface decreases relative content of the oxides, the relation Cd/Te remaining essentially the same. In Te3d spectrum after ion-etching, it is possible to distinguish the line corresponding to the binding energy ( $E_b = 573.6$  eV) of TeO<sub>2</sub>

(Fig. 6,c). Table 2 demonstrates disappearing of cadmium hydroxide, decreasing of TeO<sub>2</sub> and increasing of cadmium telluride and of elemental tellurium contents in the layers as a result of ion-etching of Sample 1. So, the mechanism of cadmium telluride electrodeposition including the electrochemical stage of elemental tellurium formation followed by chemical stage resulting in formation of CdTe and TeO<sub>2</sub> revealed by

Table 2. Atomic concentrations of elements and chemical compounds revealed at the surface and in depth of Sample 1

Ionic etching depth	Atomic concentrations, %						Chemical compounds, %			
	C	O	Cd	Te	Te/Cd	Ti	CdTe	TeO <sub>2</sub>	Cd(OH) <sub>2</sub> and/or CdO	Te
Surface	53.4	24.2	8.4	14.0	1.7	0	18	55	27	0
30 nm	22.0	25.8	19.6	32.5	1.7	0	64	27	0	9
60 nm	14.2	21.1	23.2	41.5	1.8	0	60	20	0	20
90 nm	16.6	16.7	23.6	42.9	1.8	0	60	15	0	25
150 nm	13.8	13.3	26.5	46.4	1.8	0	60	13	0	27
200 nm	8.2	10.6	30.0	50.8	1.7	0.4	60	10	0	30

voltammetric analysis is well confirmed by XPS-data.

The voltammograms in solution corresponding to the electrolyte for cadmium telluride electrodeposition on molybdenum substrates (Fig. 7) comprise two peaks of tellurium reduction (the first is absorption and the second corresponds to irreversible electrochemical reduction of tellurium with subsequent slow chemical reaction of disproportioning according to equations (5) and (6), respectively). The current rise corresponding to the water reduction and formation of hydrogen and hydroxyl groups according to reaction (3) is observed at  $E_c = -0.5$  V, as in background and in partial solutions for those substrates. Such mechanism of electrochemical process explains why nearly equal amounts of cadmium and tellurium atoms are revealed on the molybdenum substrate only in the case of cathodic potential  $E_c = -0.45$  V (Table 1). The very small shift towards negative potentials ( $E_c = -0.55$  V) results in a considerable increase of cadmium content in the films on molybdenum substrates and to the large amounts of cadmium hydroxide in those films as a result of interaction of cadmium ions and hydroxyl groups according to reaction (3).

So, basing on the kinetic studies of separate and joint electrochemical deposition of cadmium and tellurium on molybdenum and titanium nitride substrates and consideration of XPS data for the electrodeposited films, it is possible to make following conclusions. On chemically neutral TiN substrates the irreversible electrochemical TeO<sub>2</sub> reduction to elemental tellurium is accompanied by chemical reaction between Te and cadmium ions resulting in formation of cadmium telluride and TeO<sub>2</sub> as side product. The TeO<sub>2</sub> and elemental tellurium impuri-

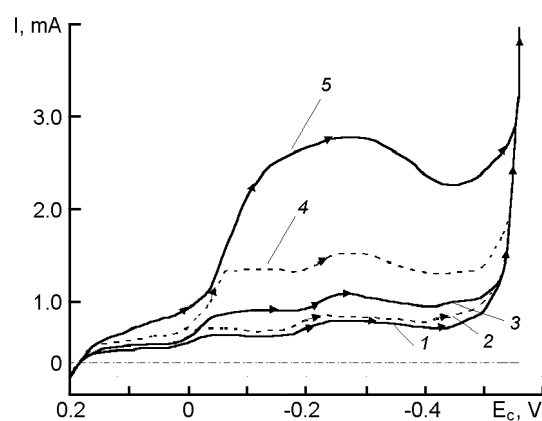


Fig. 7. Voltammograms on Mo substrates in solution for CdTe electrodeposition (0.5 M CdSO<sub>4</sub>, 2·10<sup>-4</sup> M TeO<sub>2</sub>, 0.1 M Na<sub>2</sub>SO<sub>4</sub>, 0.01 M H<sub>2</sub>SO<sub>4</sub>) at potential scanning rates (mV/s): 2 (1); 5 (2); 10 (3); 50 (4); 200 (5).

ties are found in deep layers of those films. Because of air oxidation, the surface of cadmium telluride films contains large amounts of tellurium oxide and cadmium oxide and/or cadmium hydroxide. The deposition of the films with equal amounts of cadmium and tellurium atoms on the TiN substrates occurs within the cathodic potential range from -0.45V to -0.65 V. Due to catalytic influence of molybdenum on electrochemical reaction of water reduction accompanied by increasing alkalinity of near-electrode space as well as a result of elemental tellurium absorption on molybdenum surface, a sharp rise of cadmium hydroxide content in the films on molybdenum substrates is observed at electrodeposition potentials more negative than -0.5 V. So, from the point of view of technical use of such electrolytes for cad-

mium telluride electrochemical deposition, molybdenum is an undesirable substrate.

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## Електрохімічне осадження плівок телуриду кадмію

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Методами хроновольтамперометрії та циклічної вольтамперометрії досліджено механізми електрохімічних процесів в електроліті для одержання напівпровідникових плівок телуриду кадмію та у відповідних йому парціальних розчинах, які вміщують іони кадмію і телуру. Виявлено відмінності у катодних процесах на хімічно інертних підкладках з нітриду титану і на каталітично активній поверхні молібдену. На підставі проведених досліджень пояснено природу домішок, які у роботі виявлено методом рентгенівської фотоелектронної спектроскопії на поверхні і в об'ємі плівок телуриду кадмію, електроосаджених на підкладки з нітриду титану і молібдену, що дозволило здійснити вибір параметрів процесу електроосадження і матеріалу підкладок, які забезпечують виготовлення плівок стехіометричного складу.