UDC 541.13.544.65

EFFECT OF VARIOUS FACTORS ON THE COMPOSITION OF ELECTROLYTIC THIN FILMS Sb-Se

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Received 30.06.2018

The effect to various factors such as temperature, concentration of components in the electrolyte, current density on the composition of thin electrodeposited semiconductive Sb-Se films were viewed in the work. Results obtained indicate that the stibium content in the deposited compounds increases as temperature and concentration of SbOCl rise. As the current density, H_2SeO_3 and $C_4H_6O_6$ concentration rises, the stibium content in the films obtained goes dpwn respectively. It revealed that black, uniform, crystalline and lustrous coatings of Sb_2Se_3 compound are generated in temperature interval 298-318К, current density 20-60 мА/cm^2 out of electrolyte 0,01-0,09 M SbOCl,0,01-0,09 MH_2SeO_3, and compounds of 0.001- 0.007 M tartaric acid.
Keywords: *electrodeposition, Sb-Se thin films, current density, semiconductors*

İNTRODUCTİON

Recently, the interest has been growing in thin film materials [1-5]. Note that thin films with varied physicochemical properties are applied widely in the capacity of various functional coatings for increasing stability, corrosion resistance, improving magnetic and electrical properties. The application of the thin film materials in aeronautics and space technology, mechanical engineering, medicine, solar energetic, accumulation of energy; as supercapacitors, digital microelectronics, etc. helps to decrease the bulk of materials in various devices [6-8]. Along with these, the nano-structural films have attracted much attention because of their unique properties in functional materials.

The Sb_2Se_3 films arouse interest from their application in microwave and thermo-electrical chilling devices, optic electronic mechanisms, etc. In this respect, Sb_2Se_3 compound is a laminated structural semiconductor with orthorhombic crystalline configuration where each Sb and Se atoms are bonded with three opposite atoms which, in turn, are connected through weak subordinate bonds in the crystal [9].

The thin Sb_2Se_3 semiconductive films are obtained by different methods, including chemical precipitation from solutions, electrodeposition, jet pyrolysis, vacuum thermal evaporation and so on.

In our work, the thin Sb_2Se_3 semiconductive films were synthesized by a chemical method from the tartaric electrolyte. The electroreduction of each initial component was tested individually before the study of Sb-Se electrodeposition [10, 11].

The goal of the work is a study an effect of some factors on the composition and quality of thin semiconductive Sb-Se films.

https://doi.org/10.32737/2221-8688-2018-3-331-336

EXPERIMENTAL PART

Experimental procedures based on the use of electrochemical method for production of Sb-Se thin films were carried out as follows.

At first, the tartaric acid was dissolved in bidistilled water for the preparation of electrolytes of initial components. Then SbOCl (analytical grade) and H_2SeO_3 (analytical grade) with required concentration were dissolved in the background solution of the tartaric acid, respectively.

The polarization curves were recorded by IVIUMSTATEl electrochemical Interface potentiometer. The electrochemical triode glass cell was used for this purpose. A Pt electrode with an area of 0.02 sm^2 was utilized as a working electrode. A silver chloride electrode served as a reference electrode, whereas a platinum disk with 4.0 sm^2 area was an auxiliary electrode. Also, a universal ultra thermostat UTU-4 was used to arrange temperature in the electrolyzer.

The electrochemical deposition processes proceeded through the use of potentio-dynamic and galvanostatic methods. In this case, Ni electrode with an area of 2.0 sm^2 was used.

The composition of Sb-Se cathodic deposits was determined by using JEOL JSX 3222 Element Analyzer with energy-dispersive X-ray fluorescence spectroscopy (EDXRF) system (JEOL, Japan). It is an analytical technique used for the elemental analysis or chemical characterization of a sample (metals and alloys).

RESULTS AND DISCUSSION

The potentiodynamic polarization investigations were carried out to determine a potential region where antimony and selenium co-deposited. The obtained results are given in Fig.1 which shows that the simultaneous electrodeposition process of stibium with selenium takes place within -0.42 – (-0.7) V.

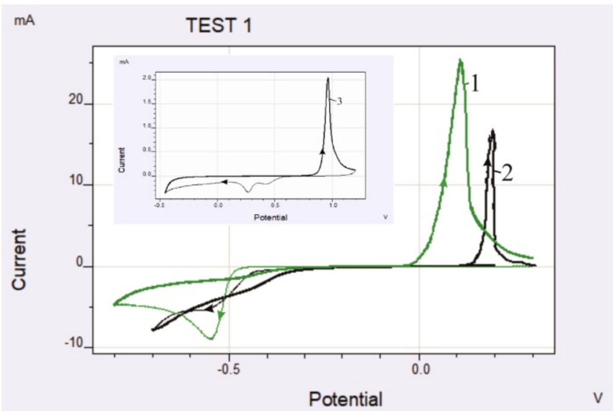

Fig. 1. The cyclic polarization curves of the electroreduction of stibium, selenium and Sb-Se. Electrolyte (M):
1- 0.05 SbOCl + 0.007 $C_4H_6O_6$
2- 0.05 SbOCl + 0.05H_2SeO_3 + 0.007 $C_4H_6O_6$
3- 0.05 H_2SeO_3 + 0.007 $C_4H_6O_6$
T=298K, E_V = 0.02 V/s.

Following the identification of potential region for electrodeposition of Sb-Se, the effect of some factors on the composition of the films has been studied.

The effect of temperature on the composition of the films has been studied within 298-358 K intervals (fig. 2.).

As it is seen from the figure, the stibium content in the composition of cathodic films increases as the electrolyte temperature rises. Moreover, the qualities of electrodeposited films are also changed. At 298-318 K temperature intervals a black, uniform, crystalline, shiny coating of Sb_2Se_3 compound is formed. But at high temperatures, the black, uneven, loose films are formed on the cathode with a weak clutch on the surface of the electrode. Therefore, all next experiments to obtain thin films of Sb_2Se_3 compound were accomplished at 298 K temperature.

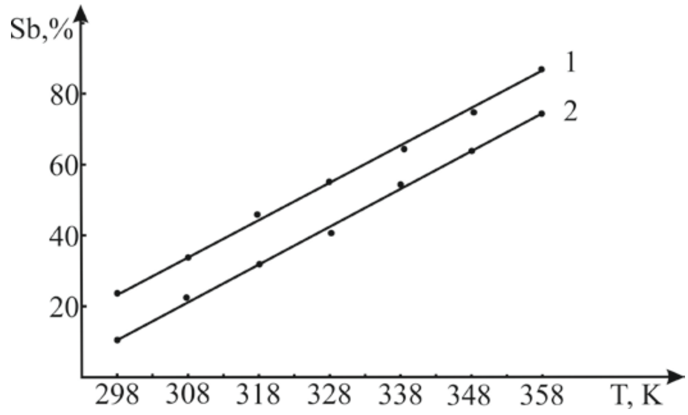

Fig. 2. The dependence between stibium content in the electrodeposited thin films and temperature. Electrolyte (M): 0.05 SbOCl + 0.05 H_2SeO_3 + 0.007 $C_4H_6O_6$. The current density (mA/sm^2): 1- 30;

Moreover, the concentration of the initial components effects on the composition of the electrodeposited thin films.

In Fig. 3, the effect of concentration of basic component SbOCl on deposited composition is shown. As follows from Fig. 3, the effect of SbOCl concentration was studied within 0.01-0.09 M intervals, and its increase in electrolyte affects compositions and qualities of the obtained deposits. Despite this, the rise in the current density reduces the stibium content in the composition of thin films.

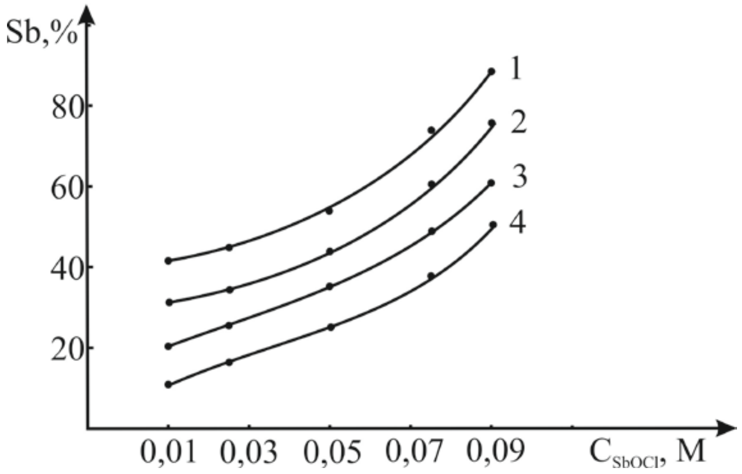

Fig. 3. The dependence between stibium content in the electrodeposited thin films and concentration of SbOCl in electrolyte. Electrolyte (M): 0.05 H_2SeO_3 + 0.007 $C_4H_6O_6$. The current density (mA/sm^2): 1- 20; 2- 30; 3- 40; 4- 60. T=298K.

The effect of current density on the stibium content in the obtained films was studied within 20-60 mA/sm^2 intervals. Note that the experiments were carried out with various concentrations of the initial components. According to Fig. 4, the increase of current density during the electrodeposition of Sb-Se has an effect on compositions and qualities of the obtained thin films. The results indicate that the thin films closer to stoichiometric composition were formed from an electrolyte with contents of 0.05 MSbOCl + 0.05M H_2SeO_3 + 0.007M $C_4H_6O_6$ and at 20-30 mA/sm^{2}current density. Thus, the stibium and selenium contents in the deposits are 51.4 % and 48.6 % respectively. However, as the current density increases up to 60 mA/sm^2, the stibium content in the deposits decreases sharply down to 18.8 %.

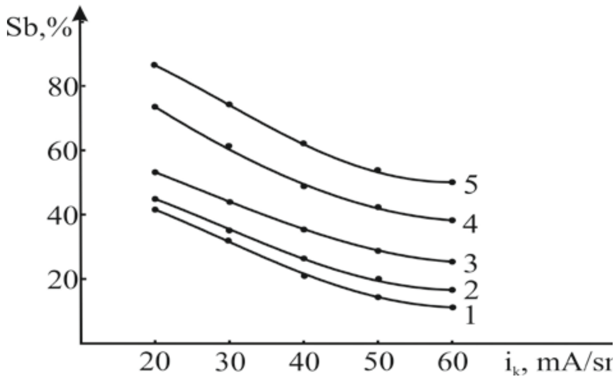

Fig. 4. The dependence between the stibium content in the electrodeposited thin films and current density.T=298K, Electrolyte (M):
1. 0.01SbOCl + 0.05 H_2SeO_3 + 0.007 C_4H6O_6
2. 0.25 SbOCl + 0.05 H_2SeO_3 + 0.007 $C_4H_6O_6$
3. 0.5 SbOCl + 0.05 H_2SeO_3 + 0.007 $C_4H_6O_6$
4. 0.75 SbOCl + 0.05 H_2SeO_3 + 0.007 $C_4H_6O_6$
5. 0.09 SbOCl + 0.05 H_2SeO_3 + 0.007 $C_4H_6O_6$

Changes in the concentration of H_2SeO_3 in the electrolyte also notably affect on the composition of the electrodeposited thin film. The dependence of the influence of this factor is shown in Fig. 5.

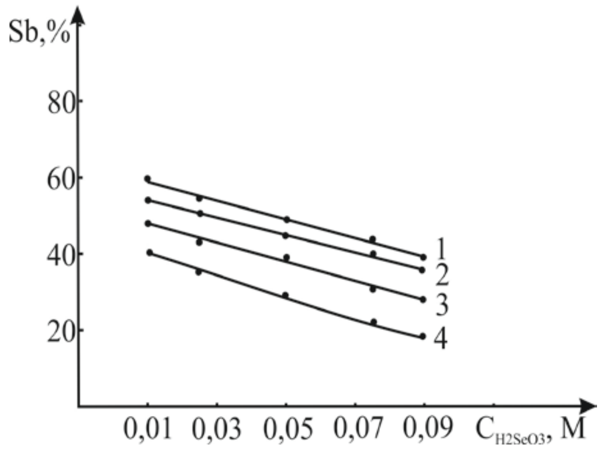

Fig. 5. The dependence between stibium content in the electrodeposited thin films and the concentration of H_2SeO_3in electrolyte. Electrolyte (M): 0.05 SbOCl + 0.007 $C_4H_6O_6$. The current density (mA/sm^2): 1 - 20; 2 - 30; 3 - 40; 4 - 60. T=298K.

Investigation of the effect of this factor shows that as H_2SeO_3 concentration in the electrolyte rises, the stibium content in the films decreases. Thus, at 20-60 mA/sm^2 current density in terms c H_2SeO_3 concentration rise in electrolyte from 0.01 to 0.09 M, the stibium content in the deposits decreases from 60.7 to 19.2 %.

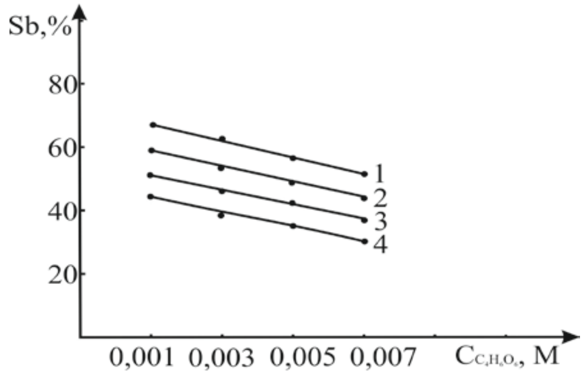

Fig. 6. The dependence between the stibium content in the electrodeposited thin films and the concentration of $C_4H_6O_6$ in electrolyte. Electrolyte (M): 0.05SbOCl + 0.05 H_2SeO_3. The current density (мA/cм^2): 1 - 20; 2 - 30; 3 - 40; 4 - 60. T=298K.

According to Fig. 6, as the concentration of tartaric acid in the electrolyte rises from 0.001 to 0.007 M, the stibium content in electrodeposited thin films decreases depending on current density from 67.2 to 30.1 %.

CONCLUSIONS

The simultaneous deposition process of stibium with selenium on the Pt electrodes from tartaric electrolytes has been studied by electrochemical method.

The effect of concentrations of the initial components, temperature and current density on electrodeposition process of the Sb-Se thin films was also investigated. The results of all experiments indicate that the increase in the temperature and concentration of SbOCl leads to the rise in the stibium content of the films. Also, when the current density, H_2SeO_3 and $C_4H_6O_6$ concentrations increase the stibium content in the obtained films decreases respectively. The optimal condition and electrolyte composition for electrodeposition process of Sb-Se compound were chosen due to the use of these results. Uniform, crystalline, shiny coatings of Sb_2Se_3 are reproduced within temperature intervals 298-318K, current density at 20-60 мA/cm^2 from electrolyte of 0.01-0.09 M SbOCl, 0.01-0.09 M H_2SeO_3 and 0.001- 0.007 M compounds of tartaric acid.

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ELEKTROKİMYƏVİ YOLLA ALINMIŞ Sb-Se NAZİK TƏBƏQƏLƏRİNİN TƏRKİBİNƏ MÜXTƏLİF AMİLLƏRİN TƏSİRİ

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Təqdim edilən iş elektrokimyəvi üsulla çökdürülmüş Sb-Se nazik təbəqələrinin tərkibinə müxtəlif amillərin – temperaturun, elektrolitdə komponentlərin qatılığının, cərəyan sıxlığının təsirinin tədqiqinə həsr edilmişdir. Nəticələr göstərir ki, temperaturun və SbOCl-in qatılığının artması ilə çökən nümunələrdə sürmənin miqdarı artır. Digər faktorların təsiri zamanı isə alınan nümunələrdə sürmənin miqdarı azalır. Müəyyən edilmişdir ki, Sb_2Se_3 kimyəvi birləşməsinin qara rəngli, eyni qalınlıqda, kristallik, parıltılı örtükləri 298-318 K temperaturda, 20-60 mA/sm^2 cərəyan sıxlığında, 0.01-0.09M SbOCl, 0.01-0.09M H_2SeO_3 və 0.001-0.007 M çaxır turşusu tərkibli elektrolitdən alınır.
Açar sözləri: *elektroçökmə, Sb-Se nazik təbəqələri, cərəyan sıxlığı, yarımkeçiricilər*

ВЛИЯНИЕ РАЗЛИЧНЫХ ФАКТОРОВ НА СОСТАВ ЭЛЕКТРОЛИТИЧЕСКИХ ТОНКИХ ПЛЕНОК Sb-Se

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В работе изучено влияние различных факторов – температуры, концентрации компонентов в электролите, плотности тока на состав электроосажденных тонких полупроводниковых пленок Sb-Se. Результаты показывают, что с повышением температуры и концентрации SbOCl содержание сурьмы в осадках увеличивается. А при увеличении плотности тока, концентрации H_2SeO_3 и $C_4H_6O_6$ содержание сурьмы в полученных пленках соответственно уменьшается. Установлено, что черные, равномерные, кристаллические, блестящие покрытия соединения Sb_2Se_3 получаются в интервале температур 298-318K, плотности тока 20-60 $мА/см^2$ из электролита состава 0.01-0.09 M SbOCl, 0.01-0.09 M H_2SeO_3 и 0.001-0.007 M винной кислоты.
Ключевые слова: *электроосаждение, тонкие пленки Sb-Se, плотность тока, полупроводники*