

Study of Formation and Phase Composition of Thallium Selenide Layers on Polycaproamide

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The polycaproamide PA 6 films were modified with layers of thallium selenide, Tl_xSe_y . A two stage process was used to deposit these thin layers. It involves: seleniumization in $0.05 \text{ mol}\cdot\text{dm}^{-3}$ $K_2SeS_2O_6$ solution of $0.1 \text{ mol}\cdot\text{dm}^{-3}$ HCl and 60°C followed by treatment with the solution of Tl_2SO_4 at 80°C for 10 min. The atomic absorption spectroscopy and XRD analysis for investigation the layers of Tl_xSe_y were used. The amount of selenium and thallium in the layers of Tl_xSe_y increases with increasing the duration of polymer seleniumization and varies: thallium from ~ 9.0 to $\sim 21.4 \text{ mg}\cdot\text{g}^{-1}$ and selenium from ~ 17.7 to $\sim 56.9 \text{ mg}\cdot\text{g}^{-1}$. The results of XRD analysis confirmed the formation of thallium selenide layers on the surface of polycaproamide 6 films. Three phases of tetragonal thallium selenide, Tl_2Se_2 (75–880), $Tl_2Se_{1.2}$ (75–1007), Tl_3Se_3 (41–1295) and two phases of monoclinic selenium Se_8 (71–528), Se (73–2121) were identified. The phase composition of layers of Tl_xSe changes depending on the duration of polymer seleniumization process.

Keywords: selenotrichionate, polycaproamide 6, sorption-diffusion, layers of thallium selenide.

1. INTRODUCTION

D-metal selenides because of the variety of their specific properties are widely used in various fields of modern technology. Composite materials composed of different compounds and having properties of both components are especially in use by modern technology. Thin layers of d-metal selenides on the surface of dielectrics belong to the composite materials too. Layers of binary d-metal chalcogenides formed on the surface of various dielectrics are used as semiconductors, photoconductors, sensors of heat, mineral pigments, optical filters and etc. [1]. Wide variety of d-metal selenides applications encourage studies of new methods of their formation and improvement of known methods as well looking for composite materials with the combination of wanted properties.

During the last decade binary compounds of IIIA group metals with the chalcogens because of their unique physical properties are intensively studied both by physicists and chemists in global scale [2, 3]. The thallium selenides can be used for the production the photocells [4] and photoconductors [5], which are sensitive to the infrared radiation as well as to applications in modern microelectronics [6]. The layers of thallium selenides are commonly produced by vacuum evaporation [7] or by the deposition method from solutions [8].

Sorption-diffusion method is a simple and promising for obtaining binary selenide layers. Hydrophilic and semi-hydrophilic polymers are capable of absorbing ions of various electrolytes from aqueous solutions [9]. Over the last decade, a sorption method for the formation of thin metal chalcogenides, layers on the surface of polycaproamide 6 (PA) based on the initial treatment of a polymer with the

solutions containing particles of chalcogen has been under extensive investigation [10–16]. It was shown that during treatment the anions of lower selenopolythionate – selenotrichionate, $SeS_2O_6^{2-}$, containing chains of divalent selenium atoms of low oxidation state, $-O_3S-Se-SO_3-$ [14–17], are sorbed by a polymer. Thin layers of copper or silver selenide were formed on a PA 6 surface if a polymer seleniumized in such conditions later is treated with a solution of copper(II/I) or silver(I) salt [14–16].

The aim of this study was to obtain the layers of thallium selenide of various compositions on the surface of PA film using as a precursor the solution of potassium selenotrichionate salt solution, and to investigate their composition by chemical and X-ray diffraction methods.

2. EXPERIMENTAL DETAILS

The films of polycaproamide 6 (specification TY 6-05-1775-76, grade PK-4) $15 \text{ mm} \times 70 \text{ mm}$ in size of $70 \mu\text{m}$ thickness were used. This PA 6 film is close to a non-porous material, because the pores of PA are much less than 1.5 nm [18]. The porosity was measured by a Brunauer Emmett Teller (BET) method using a Quantasorb (USA). Before the seleniumization, the samples of PA 6 were boiled in distilled water for 2 h to remove the remainder of the monomer. Then they were dried using filter paper and then over $CaCl_2$ for 24 h.

The PA 6 samples thus prepared were treated in a thermostatic vessel using a continually stirred $K_2SeS_2O_6$ solution. The $0.05 \text{ mol}\cdot\text{dm}^{-3}$ solutions of $K_2SeS_2O_6$ in $0.1 \text{ mol}\cdot\text{dm}^{-3}$ HCl at 60°C were used. At the certain time intervals, the samples were removed, rinsed with distilled water, dried over $CaCl_2$, and then used in the analysis and further experiments. The total duration of experiments was 4.5 h.

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The salt of potassium selenotriothionate, $K_2SeS_2O_6$, was prepared and chemically analyzed according to procedures published in [19].

The concentration of selenium and thallium in thallium selenide layers formed on PA 6 films surface were determined using the method of atomic absorption spectrophotometry [20]. The modified PA 6 films were dissolved in concentrated nitric acid. Selenium and thallium, present in the resulting solution, were determined by the atomic absorption spectrometer "Perkin-Elmer-503"; wavelength $\lambda = 196$ nm and $\lambda = 276.8$ nm, diffractive gap – 5 (2.0 nm) and 4 (0.7 nm), electrodeless discharge lamp, air-acetylene flame. For the standard conditions described above, the sensitivity of Se and Tl are about $0.5 \mu\text{g/ml}$ for 1% absorption.

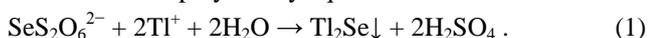
The solution of thallium(I) salt was made out of crystal thallium(I) sulphate. The concentration of composed solution of thallium(I) sulphate salt is $0.1 \text{ mol}\cdot\text{dm}^{-3}$ (pH ~ 2.8). KOH addition was put into this solution, till its concentration became $0.015 \text{ mol}\cdot\text{dm}^{-3}$ (pH ~ 11.4). Then the samples of seleniumized PA 6 were treated with the solution of Tl_2SO_4 at 80°C for 10 min, the thallium selenide layers on polyamide films were formed. After the treatment with the solution of Tl_2SO_4 salt, the samples of polymers were rinsed with distilled water, dried over anhydrous $CaCl_2$ and used in further experiments.

X-ray diffraction analysis the thallium selenide layers on the polymer surface was performed with a DRON-6 diffractometer provided with a special device for beam limitation at low and medium diffraction angles using graphite-monochromatized Cu-K α radiation source ($\lambda = 1.54178 \text{ \AA}$) under a voltage of 30 kV and a current of 30 mA. The XRD patterns were recorded with a step size of 0.05° from $2\theta = 25^\circ$ to 70° . X-ray diffractograms of modified PA 6 films were treated using the program "Search Match", "ConvX", "Xfit" and "Microsoft Office Excel".

3. ANALYSIS AND RESULTS

The main task of this work was to prove the suitability of seleniumized PA 6 films with solution of $K_2SeS_2O_6$ for formation the thallium selenide layers on polymer.

For this the seleniumized PA 6 films were treated with Tl_2SO_4 salt solution. Changes of a PA 6 films appearance after its treatment with the Tl(I) salt solution also indicate the formation of thallium selenide layers on the surface of a polymer by equation:



Colour of tapes from colourless, light yellow, orange or red transforms into brown-red or brown-grey.

In order to determine an influence of seleniumization duration of the initial solution of $K_2SeS_2O_6$ the kinetic studies of the sorption of selenium into PA 6 from prepared solution of potassium selenotriothionate were performed.

The investigation of sorption kinetics of selenium in thallium selenide layers when PA 6 films exposed during 4.5 h in the $0.05 \text{ mol}\cdot\text{dm}^{-3}$ concentration solution of $K_2SeS_2O_6$ shows that the exposure time increase leads to an significant increase of amount of selenium, expressed in mg per g of polymer, respectively, from $17.7 \text{ mg}\cdot\text{g}^{-1}$ to $57.0 \text{ mg}\cdot\text{g}^{-1}$ – a more than three – hold increase (Fig. 1).

The same dependence of the changing the amount of thallium in Tl_xSe layers on PA 6 films as in the case of the amount the selenium was observed. The PA 6 films exposure time increase leads to a significant increase of amount of thallium, expressed in mg per g of PA, respectively from $9.0 \text{ mg}\cdot\text{g}^{-1}$ to $21.4 \text{ mg}\cdot\text{g}^{-1}$ – a more than two – hold increase (Fig. 1).

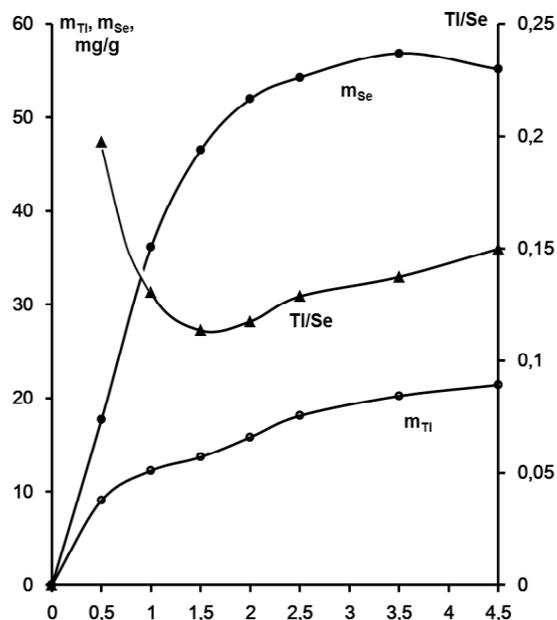


Fig. 1. Changes of the thallium (m_{Tl}) and the selenium (m_{Se}) amounts thallium selenide layers on PA 6 films with time during polymer treatment with the solution of $K_2SeS_2O_6$

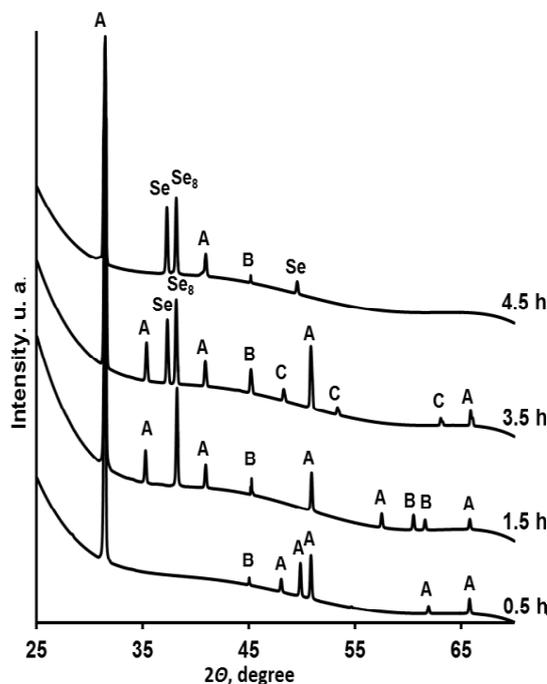


Fig. 2. X-ray diffraction patterns (A – Tl_2Se_2 (75-880), B – $Tl_2Se_{1.2}$ (75-1007), C – Tl_5Se_3 (41-1295), $Se_8 - Se_8$ (71-528), Se – Se (73-2121)) the layers of thallium selenide formed on the PA 6 films treated different duration with the $0.05 \text{ mol}\cdot\text{dm}^{-3}$ solutions of $K_2SeS_2O_6$ in $0.1 \text{ mol}\cdot\text{dm}^{-3}$ HCl at 60°C and with Tl_2SO_4 salt solution

Table 1. Comparison between the JCPDS data (File No. Tl_2Se_2 -75-880, $\text{Tl}_2\text{Se}_{1.2}$ -75-1007, Tl_5Se_3 -41-1295, Se_8 - Se_8 -71-528, Se - Se -73-2121) and experimentally observed values for the Tl_xSe_y layers in PA 6 films formed by sorption-diffusion method at different seleniumization time

Duration of seleniumization, h	2θ (°)	d -spacing (Å)		Compound	File No.
		Observed values	JCPDS data		
0.5	31.45	2.84	2.84	Tl_2Se_2	75-880
	45.08	2.01	2.01	$\text{Tl}_2\text{Se}_{1.2}$	75-1007
	48.12	1.89	1.89	Tl_2Se_2	75-880
	49.82	1.82	1.82	Tl_2Se_2	75-880
	50.86	1.79	1.79	Tl_2Se_2	75-880
	62.43	1.48	1.48	Tl_2Se_2	75-880
	65.91	1.42	1.42	Tl_2Se_2	75-880
1.5	31.45	2.84	2.84	Tl_2Se_2	75-880
	35.32	2.53	2.53	Tl_2Se_2	75-880
	38.2	2.35	2.35	Se_8	71-528
	40.9	2.20	2.20	Tl_2Se_2	75-880
	45.08	2.01	2.01	$\text{Tl}_2\text{Se}_{1.2}$	75-1007
	50.86	1.79	1.79	Tl_2Se_2	75-880
	57.56	1.59	1.59	Tl_2Se_2	75-880
	60.5	1.53	1.53	$\text{Tl}_2\text{Se}_{1.2}$	75-1007
	61.64	1.50	1.50	$\text{Tl}_2\text{Se}_{1.2}$	75-1007
65.91	1.42	1.42	Tl_2Se_2	75-880	
3.5	31.45	2.84	2.84	Tl_2Se_2	75-880
	35.32	2.53	2.53	Tl_2Se_2	75-880
	37.36	2.41	2.41	Se	73-2121
	38.2	2.35	2.35	Se_8	71-528
	40.9	2.20	2.20	Tl_2Se_2	75-880
	45.08	2.01	2.01	$\text{Tl}_2\text{Se}_{1.2}$	75-1007
	48.36	1.88	1.88	Tl_5Se_3	41-1295
	50.86	1.79	1.79	Tl_2Se_2	75-880
	53.46	1.71	1.71	Tl_5Se_3	41-1295
	63.3	1.47	1.47	Tl_5Se_3	41-1295
	65.91	1.42	1.42	Tl_2Se_2	75-880
4.5	31.45	2.84	2.84	Tl_2Se_2	75-880
	37.36	2.41	2.41	Se	73-2121
	38.2	2.35	2.35	Se_8	71-528
	40.9	2.20	2.20	Tl_2Se_2	75-880
	45.08	2.01	2.01	$\text{Tl}_2\text{Se}_{1.2}$	75-1007
	49.6	1.83	1.83	Se	73-2121

The molar Tl/Se ratio in thallium selenide layers formed on PA 6 provides same information about the composition of its layers. The molar Tl/Se ratio in the thallium selenide layers on the polymer surface was calculated from amounts of Tl and Se measured in the PA 6 samples. It was found that the Tl/Se ratio (Fig. 1) decreases to 1.5 h of initial polymer seleniumization and then begins to increase and the stoichiometric composition varying from $\text{Tl}_{0.2}\text{Se}$ to $\text{Tl}_{0.11}\text{Se}$. The compositions of thallium selenide with 1/5 or 1/10 of molar Tl/Se ratio are not known. Therefore we can expect the elemental Se is contained in the composition of the thallium selenide layers formed on PA 6 films.

X-ray diffraction analysis gives more accurate identification of thallium selenides formed on the surface

of PA 6 films. But structural studies of the thallium selenide layers deposited by sorption-diffusion method are limited by polycrystallinity of layers obtained, as well as by the existence of thallium selenide phases with various compositions and structures, and by the crystallinity of the polymer film itself. The intensities of its maximum at $2\theta < 25^\circ$ exceeds intensity of thallium selenide maximum few times. Therefore the area of $2\theta \geq 25.0^\circ$ was investigated more detail. Using the method described in [21], we also calculated the quantitative composition of these layers.

The fact, that the formation of thallium selenide layers takes place by keeping seleniumized PA 6 films in the solution of Tl_2SO_4 salts by the X-ray diffraction analysis was confirmed (Fig. 2 and Table 1). And the peaks of

elemental selenium in the XRD patterns were noticed. All peaks obtained are well matched with the Joint Committee on Powder Diffraction Standards (JCPDS) (Table 1). Three phases of tetragonal thallium selenide and two phases of monoclinic selenium were found, namely, Tl_2Se_2 (75-880) [21], $Tl_2Se_{1.2}$ (75-1007) [22], Tl_5Se_3 (41-1295) [23], Se_8 (71-528) [24] and Se (73-2121) [25]. The phase of Tl_2Se_2 (75-880) is predominant in the layers of thallium selenide on PA 6 films. But with the prolongation of the period of the polymer seleniumization this dominance decreases. For example, the six peaks of various intensity of tetragonal Tl_2Se_2 (75-880) are observed, when duration of polymer seleniumization was 0.5 h. When the duration of polymer seleniumization prolonged up to 4.5 h, only two peaks of Tl_2Se_2 (75-880) on X-ray pattern of thallium selenide layer were detected. When reducing the number of peaks of the tetragonal Tl_2Se_2 this phase in layers Tl_xSe_y also decreases: $99.2 \rightarrow 75.72 \rightarrow 68.61 \rightarrow 66.56$ (Table 2). In addition, with increasing duration of polymer seleniumization herewith of the dominant phase in the layers of Tl_xSe_y , the two new phases of tetragonal thallium selenide $Tl_2Se_{1.2}$ (75-1007) and Tl_5Se_3 (41-1295) (Fig. 2, Table 1) arise. These phases contain more thallium. That is in accordance with the results of chemical analysis, when molar ratio of Tl/Se in layers of thallium selenide formed on PA 6 films initial seleniumized 1.5 h and longer duration was increased from 0.11 to 0.15 (Fig. 1). The elemental selenium in the composition of these layers was contained as well. When further increasing seleniumization time, increase in the number of XRD peaks can be observed which can be assigned to a new monoclinic selenium phases (Se_8 (71-528) and Se (73-2121)) formation. Also the amount of total elemental selenium in the layers increases: $17.74 \rightarrow 23.2 \rightarrow 32.92$ (Table 2). This supports the assumption above that new, possibly elemental Se phase, is formed in increased seleniumization time, as inferred from bulk elemental composition. Elemental Se can form via decomposition [26] of adsorbed/diffused $SeS_2O_6^{2-}$ ions in PA 6 films according to reaction:



It is believable that elemental selenium was not interact with the Tl(I) ions and remained in PA 6 films, when the sulphur containing particles wash out from polymer.

From the data it was concluded, that the changing the conditions of PA 6 films seleniumization leads to the formation different composition of thallium selenide layers.

4. CONCLUSIONS

1. The layers of thallium selenide were formed on the surface of polycapromamide 6 films when the seleniumized polymer with solution of $K_2SeS_2O_6$ and then was treated with the solution of Tl_2SO_4 salt.

2. The amount of thallium in the layers of thallium selenide increases with the increasing of amount of selenium in polymer. The chemical composition of thallium selenide layers depends on the duration of polycapromamide 6 films seleniumization process and varies from $Tl_{0.1}Se$ and $Tl_{0.2}Se$.

3. Three phases of tetragonal thallium selenide (Tl_2Se_2 , $Tl_2Se_{1.2}$ and Tl_5Se_3) and two phases of monoclinic selenium (Se_8 and Se) have been identified in the layers of thallium selenide by XRD analysis. The phase of Tl_2Se_2 is predominant in the layers of thallium selenide. The phase composition of Tl_xSe_y layer depends on the period of treatment of polymer in solution of $K_2SeS_2O_6$.

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Table 2. The quantitative composition the layers of Tl_xSe_y in PA 6 films formed by sorption-diffusion method at different seleniumization time

Duration of seleniumization, h	Compound	Amount, %
0.5	Tl_2Se_2 -75-880	99.20
	$Tl_2Se_{1.2}$ -75-1007	0.80
1.5	Tl_2Se_2 -75-880	75.72
	$Tl_2Se_{1.2}$ -75-1007	6.54
	Se_8 -71-528	17.74
3.5	Tl_2Se_2 -75-880	68.61
	$Tl_2Se_{1.2}$ -75-1007	3.68
	Tl_5Se_3 -41-1295	4.51
	Se_8 -71-528	13.20
	Se -73-2121	10.00
4.5	Tl_2Se_2 -75-880	66.56
	$Tl_2Se_{1.2}$ -75-1007	0.52
	Se_8 -71-528	16.31
	Se -73-2121	16.61

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