

X-ray photoelectron spectroscopy investigation of $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$ layers on polyamide film formed by the use of potassium telluropentathionate

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The phase composition of polyamide 6 (PA) films modified by layers of copper sulfides – copper tellurides, $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$, was studied by the X-ray photoelectron spectroscopy method. Copper sulfide – copper telluride layers on the surface of PA film were formed by treating PA film samples in acidified 0.025, 0.05 and 0.1 mol · dm⁻³ solution of potassium telluropentathionate, $\text{K}_2\text{TeS}_4\text{O}_6$, at a temperature of 1, 10 and 20 °C and then with Cu(II / I) salt solution. In the surface of PA samples modified by copper sulfides – copper tellurides, the presence of various copper, sulfur, tellurium and oxygen compounds (Cu_2S , CuS , S_8 , Cu_xS , Cu_yTe , $\text{Cu}(\text{OH})_2$ and TeO_2) was determined. Copper sulfides and tellurides took the major part in the layer; the content of copper sulfides in the composition of layers was higher. The copper sulfur–tellurium ratio decreased with increasing the duration of chalcogenization, in agreement with the results of the samples' sheet resistance measurements since samples with the lower value of the copper : sulfur–tellurium ratio were more conductive.

Key words: copper sulfide-copper telluride layer, potassium telluropentathionate, polyamide film

INTRODUCTION

Numerous investigations over the past few decades have shown an increasing interest in semiconducting metal chalcogenide layers because of their wide range of applications in various fields of science and technology. Semiconductor transition metal chalcogenides have been of great interest due to their excellent properties and potential applications [1, 2]. Copper chalcogenide layers have a number of applications in various devices such as solar cells, super ionic conductors, photo-detectors, electroconductive electrodes, microwave shielding coatings, etc. [3–7].

Copper sulfide and copper telluride belong to chalcogenides (binary compounds of the elements of groups I–VI with chalcogens). Copper sulfides are of great interest owing to their variations in stoichiometric composition, nanocrystal morphology, complex structure, valence states, unique properties, and their potential applications in numerous fields [8–13]. Copper tellurides are semiconductors with thermoelectric properties and ionic conductivity. They have attracted considerable interest because of their potential applications in various devices such as solar cells, optical data storage, etc. [14].

Thin metal chalcogenide layer formation by a modified chemical method, for example, sorption–diffusion, has recently attracted interest. Factors such as control of layer thickness and formation rate by varying reagent concentration, temperature are allied with a potential to coat a large area at a low cost. The modified chemical method may be used at room temperature for deposition from aqueous solutions. The basic difference between chemical bath deposition and a modified chemical method is the growth mode. In chemical bath deposition, all the precursors are present at the same time in the reaction vessel, whereas in the modified chemical method, the substrate is treated separately with each precursor, and rinsing separates these treatments [3].

The formation of copper chalcogenides on the surface of semi-hydrophilic polymer – polyamide 6 – using telluropentathionic acid, $\text{H}_2\text{TeS}_4\text{O}_6$, as a precursor of chalcogens was discussed in [15].

Analysing the phase composition of polymers modified with copper chalcogenide layers, the method of X-ray photoelectron spectroscopy (XPS) is often used [16–24]. This method of the determination of surface atomic composition is based on the analysis of bonds formed in various compounds. In this case, the surface of a sample is studied first and in a deeper layer, when the coating surface is being etched by Ar^+ ions up to one or a few nanometers deep. This analysis is

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restricted only by a study of the chemical composition of a very thin surface layer when the thickness of the whole layer may reach a few tenths of a micron.

From the data gained by the XPS method, i.e. from the atomic percentage of elements, binding energies and the spectra of individual elements, it is possible to determine the layer surface composition.

The aim of the present work was to study by the X-ray photoelectron spectroscopy method the composition of PA film surface modified with copper sulfides – copper tellurides.

EXPERIMENTAL

Layers of copper sulfides – copper tellurides were deposited on polyamide 6 (PA) films produced in Russia (Yekaterinburg, “Uralplastik”) (manufacturer’s TU 6-05-1775-76, grade PK-4, 15 × 70 mm, 70 μm). PA films were boiled in distilled water for 2 h to remove the monomer residues. They were dried with filter paper and then over anhydrous CaCl₂ for 24 h.

PA films were sulfurized in a thermostatic vessel up to 24.5 h at a temperature of 1, 10 and 20 °C using a 0.025, 0.05 and 0.1 mol · dm⁻³ solution of K₂TeS₄O₆ in 0.2 mol · dm⁻³ HCl. Potassium telluropentathionate hemitrihydrate, K₂TeS₄O₆ · 3 / 2H₂O was prepared by the method presented in [25]. At certain time intervals, the PA film samples were removed from K₂TeS₄O₆ solution, rinsed with distilled water, dried with filter paper, left over CaCl₂ for 24 h and then used in further experiments and analysis. By the XPS method we studied Cu_xS–Cu_yTe layers formed by treating chalcogenized PA films for 10 min with Cu(II / I) salt solution at a temperature of 78 °C [26].

The conductivity at a constant current of copper sulfide and copper telluride layers was measured using an E7-8 numerical measuring instrument. A polymer film was placed under special electrode made from two nickel-plated copper plates with fixed 1 cm long dielectric material between them. We measured the sheet resistance of layers

obtained on the polyamide film. The concept of sheet resistance is used to characterize thin deposited layers. Sheet resistance is specified by the unit “ohms per square” (Ω/□, ohms / square).

The XPS spectra of Cu_xS and Cu_yTe layers were recorded with an ESCALAB MKII spectrometer (VG Scientific, radiation Mg K_α – 1253.6 eV, output 300 W). Vacuum in the analysing chamber was kept at a level of 1.33 · 10⁻⁸ Pa, and the distribution of elements in the depth was determined by sputtering with an Ar⁺ gun with the ion energy of about 1.0 keV. The samples were etched in a preparation chamber with the vacuum 9.3 · 10⁻³ Pa and current 20 μA; duration of etching was 15 s. The maximum accuracy of the method was ±0.1 at. %. To reveal the composition of the obtained copper chalcogenide layers, the photoelectron spectra of S 2p, Te 3d_{5/2}, Cu 2p_{3/2} and O 1s were recorded. Empirical sensitivity factors for these elements were taken from the literature [27], and the obtained spectra were compared with the standard ones [28, 29].

RESULTS AND DISCUSSION

Copper sulfide – copper telluride layers formed on the surface of polyamide 6 film were studied by the X-ray photoelectron spectroscopy (XPS) method. Analysing the samples, the composition of the copper chalcogenide coating in deeper layers (up to 1 nm) after its surface had been etched (bombardment with the Ar⁺ ions) was studied. From the data gained by the XPS method, i. e. from the elements’ atomic percentages, binding energies and the spectra of individual elements, it is possible to reveal the composition of the obtained layer. The analysis is restricted only by a study of the chemical composition of a very thin surface layer when the thickness of the whole layer may reach a few tenths of a micron.

The maxima in S 2p spectra, corresponding to the binding energy values of ~161–162 eV, show the presence of sulfide sulfur; the maxima corresponding to the binding energy values of ~163–164 eV indicate the presence of elemental sulfur [27, 28].

Table 1. XPS data on Cu_xS–Cu_yTe etched (15 s, 20 μA) layers on PA surface initially chalcogenized at 20 °C for 8.0 h in K₂TeS₄O₆ solution

K ₂ TeS ₄ O ₆ , mol · dm ⁻³	Element	Content, at. %	Binding energy, eV	Possible composition	Cu : (S + Te)	Sheet resistance, kΩ/□
0.025	Cu	62.87	932.3	Cu ₂ S, CuS		
	S	25.96; 1.69	161.54; 163.84	S ²⁻ (Cu _x S), S ₈		
	Te	9.48	572.4	Te ²⁻ (Cu _y Te)	1.69	7.7
	O	–	–	–		
0.05	Cu	60.13	932.3	Cu ₂ S, CuS		
	S	30.13	161.5	S ²⁻ (Cu _x S)		
	Te	9.74	572.5	Te ²⁻ (Cu _y Te)	1.51	2.08
	O	–	–	–		
0.1	Cu	58.54	932.2	Cu ₂ S, CuS		
	S	26.01; 3.79	161.5; 163.7	S ²⁻ (Cu _x S), S ₈		
	Te	11.65	572.6	Te ²⁻ (Cu _y Te)	1.41	1.63
	O	–	–	–		

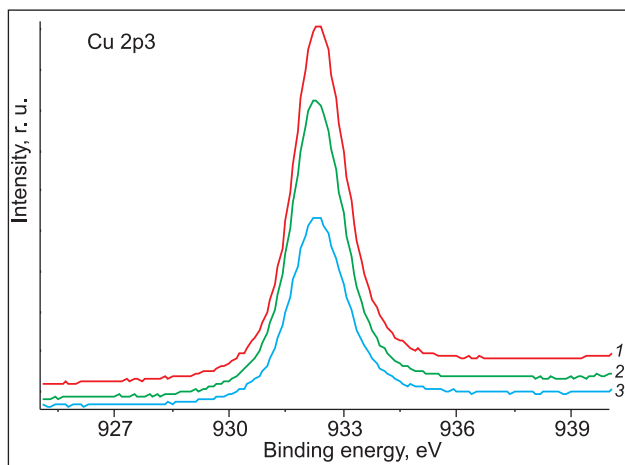


Fig. 1. XPS Cu 2p3 spectra of $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$ layers on PA surface obtained during PA treatment for 8 h at a temperature of 20 °C in $\text{K}_2\text{TeS}_4\text{O}_6$ solution. Concentration of $\text{K}_2\text{TeS}_4\text{O}_6$ solution, $\text{mol} \cdot \text{dm}^{-3}$: 1 – 0.025, 2 – 0.05, 3 – 0.1

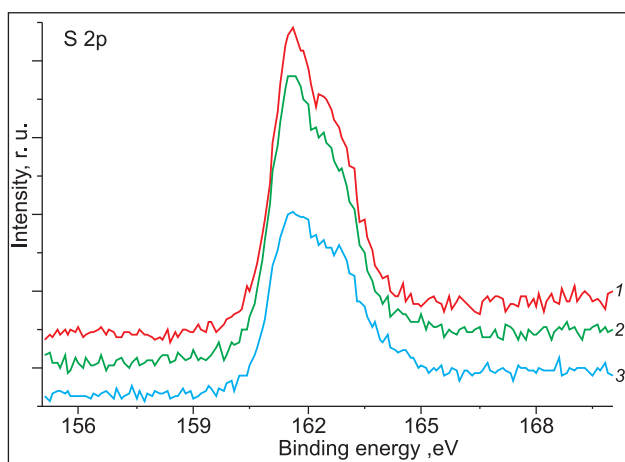


Fig. 2. XPS S 2p spectra of $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$ layers on PA surface obtained during PA treatment for 8 h at a temperature of 20 °C in $\text{K}_2\text{TeS}_4\text{O}_6$ solution. Concentration of $\text{K}_2\text{TeS}_4\text{O}_6$ solution, $\text{mol} \cdot \text{dm}^{-3}$: 1 – 0.025, 2 – 0.05, 3 – 0.1

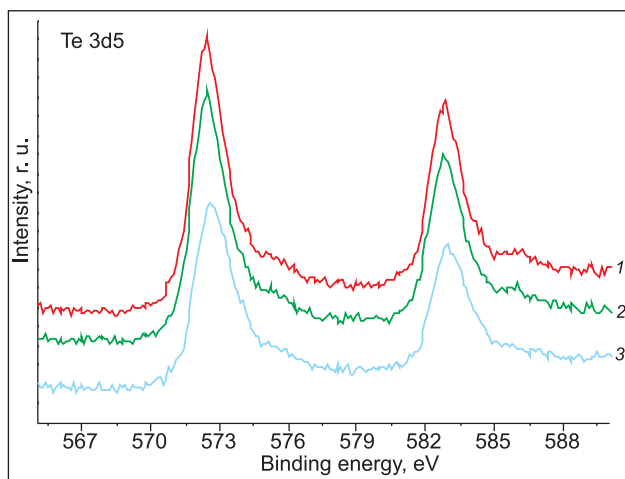


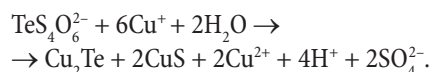
Fig. 3. XPS Te 3d5 spectra of $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$ layers on PA surface obtained during PA treatment for 8 h at a temperature of 20 °C in $\text{K}_2\text{TeS}_4\text{O}_6$ solution. Concentration of $\text{K}_2\text{TeS}_4\text{O}_6$ solution, $\text{mol} \cdot \text{dm}^{-3}$: 1 – 0.025, 2 – 0.05, 3 – 0.1

The maxima in Te 3d5 spectra, corresponding to the binding energy values of ~572–573 eV, show the presence of telluride ion, and the maxima corresponding to the binding energy value of ~575.8 eV indicate the presence of TeO_2 or Te(IV) [27–29].

In the present work, the dependence of the layer composition on the concentration of chalcogenization agent solution, temperature and treating duration in this solution was studied. The results are summarized in three tables and nine figures.

Data in Table 1 and Figs. 1–3 show the dependence the composition of the obtained layers on the concentration of chalcogenization agent solution. One can see that the composition of layers obtained in different conditions is similar (Table 1); this is confirmed by the similarity of spectra (Figs. 1–3).

Data in Table 1 show that in the etched surface various compounds of copper, sulfur and tellurium are present. The following compounds were identified: Cu_2S , CuS , S_8 , Cu_xS , Cu_yTe . In our work, only the layer's surface etched by Ar^+ ions was analysed, and data in Table 1 show that no oxygen in a deeper layer was detected. The atomic percentage of the elements shows that copper sulfides and tellurides take the major part in the layers. We may state also that the content of copper sulfides in the composition of a layer is higher since the content of sulfide sulfur in the layers studied was about three times higher than that of the telluride tellurium. These data are in agreement with the equation according to which telluropentathionate ions react with copper(I) ions [30]:



From the above equation it follows that more copper sulfide compared with copper telluride is formed.

Data in Table 1 and Figs. 1–3 show that the atomic percentage of the elements (Cu, S, Te) with increasing the concentration of $\text{K}_2\text{TeS}_4\text{O}_6$ solution changes insignificantly. Therefore, we may state that an increase in chalcogenization agent solution concentration almost does not change the composition of the layer.

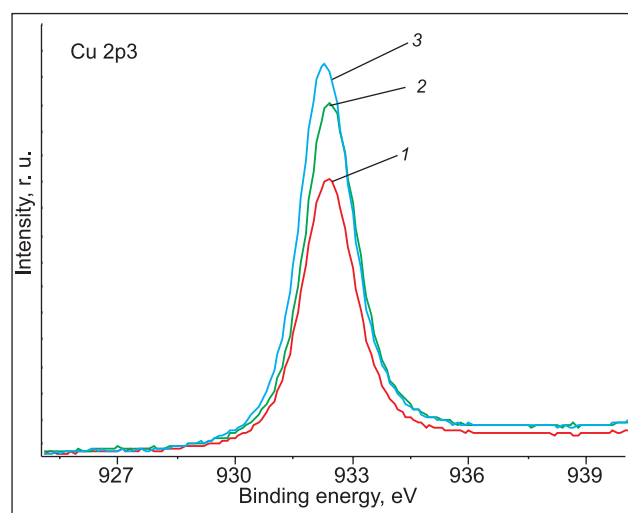
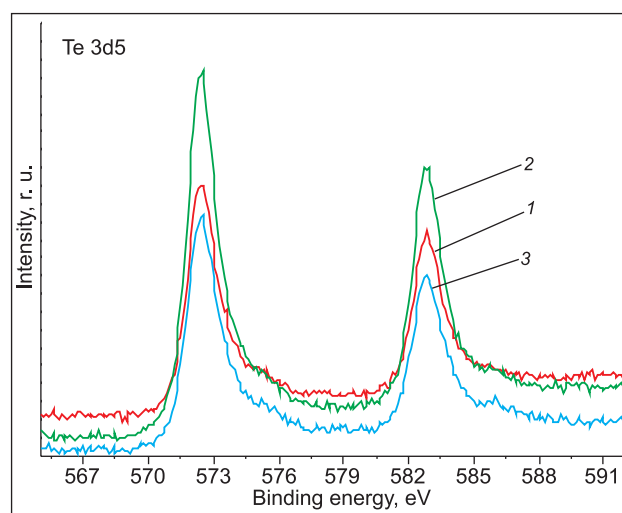
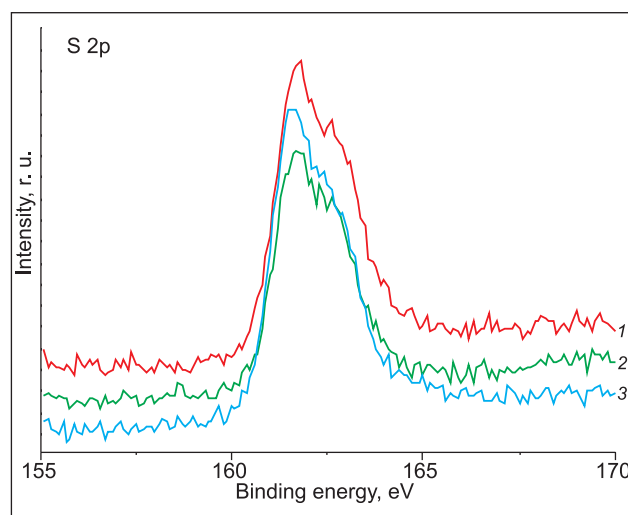
Upon calculating from atomic percentages the copper : sulfur–tellurium ratios we found that their values decreased with increasing the $\text{K}_2\text{TeS}_4\text{O}_6$ solution concentration (Table 1). These data are in good agreement with the results of the samples' electrical sheet resistance measurements, i. e. the samples were more conductive at a lower value of the copper : sulfur–tellurium ratio. When the Cu : (S + Te) ratio was equal to 1.69, the value of sheet resistance was 7.7 $\text{k}\Omega/\square$; when the ratio mentioned was 1.51–2.08 $\text{k}\Omega/\square$, and at a ratio value of 1.41–1.63 $\text{k}\Omega/\square$.

Data in Table 2 and Figs. 4–6 illustrate the dependence of the composition of the obtained layers on the temperature of chalcogenization agent solution.

A comparison of element atomic percentages shows that more copper is present in samples chalcogenized at a higher temperature: at 1 °C – 54.1 at. %, at 10 °C – 57.29 at. %, at

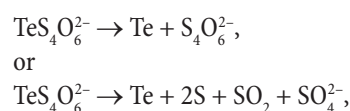
Table 2. XPS data on $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$ etched layers on PA surface initially chalcogenized for 8 h in $0.05 \text{ mol} \cdot \text{dm}^{-3} \text{K}_2\text{TeS}_4\text{O}_6$ solution

Temperature, °C	Element	Content, at. %	Binding energy, eV	Possible composition	Cu : (S + Te)	Sheet resistance, $\text{k}\Omega/\square$
1	Cu	54.1	932.4	Cu_2S , CuS	1.18	17.25
	S	34.44	161.8	$\text{S}^{2-}(\text{Cu}_x\text{S})$		
	Te	11.46	572.4	$\text{Te}^{2-}(\text{Cu}_y\text{Te})$		
	O	–	–			
10	Cu	57.29	932.4	Cu_2S , CuS	1.34	10.08
	S	27.34	161.7	$\text{S}^{2-}(\text{Cu}_x\text{S})$		
	Te	15.36	572.5	$\text{Te}^{2-}(\text{Cu}_y\text{Te})$		
	O	–	–			
20	Cu	60.13	932.3	Cu_2S , CuS	1.51	2.08
	S	30.13	161.5	$\text{S}^{2-}(\text{Cu}_x\text{S})$		
	Te	9.74	572.5	$\text{Te}^{2-}(\text{Cu}_y\text{Te})$		
	O	–	–			

Fig. 4. XPS Cu 2p3 spectra of $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$ layers on PA surface obtained during PA treatment for 8 h in a $0.05 \text{ mol} \cdot \text{dm}^{-3}$ solution of $\text{K}_2\text{TeS}_4\text{O}_6$. Temperature of $\text{K}_2\text{TeS}_4\text{O}_6$ solution, °C: 1 – 1, 2 – 10, 3 – 20Fig. 6. XPS Te 3d5 spectra of $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$ layers on PA surface obtained during PA treatment for 8 h in a $0.05 \text{ mol} \cdot \text{dm}^{-3}$ solution of $\text{K}_2\text{TeS}_4\text{O}_6$. Temperature of $\text{K}_2\text{TeS}_4\text{O}_6$ solution, °C: 1 – 1, 2 – 10, 3 – 20Fig. 5. XPS S 2p spectra of $\text{Cu}_x\text{S}-\text{Cu}_y\text{Te}$ layers on PA surface obtained during PA treatment for 8 h in a $0.05 \text{ mol} \cdot \text{dm}^{-3}$ solution of $\text{K}_2\text{TeS}_4\text{O}_6$. Temperature of $\text{K}_2\text{TeS}_4\text{O}_6$ solution, °C: 1 – 1, 2 – 10, 3 – 20

20 °C – 60.13 at. %. But the highest content of sulfide sulfur was found in a sample chalcogenized at the lowest (1 °C) temperature; the highest content of tellurium in the telluride form was found in a sample chalcogenized in $\text{K}_2\text{TeS}_4\text{O}_6$ solution at a temperature of 10 °C.

Upon calculating from the elements' atomic percentages the copper : sulfur–tellurium ratios, we found that the value of this ratio increased (Table 2) with increasing the temperature of $\text{K}_2\text{TeS}_4\text{O}_6$ solution. The increase of this ratio may be determined by a few reasons, for example, by the fact that a telluropentathionate ion at a higher temperature decomposes [30, 31]:



therefore, copper ions may react not only with $\text{TeS}_4\text{O}_6^{2-}$ ions according to the above mentioned equation, but also

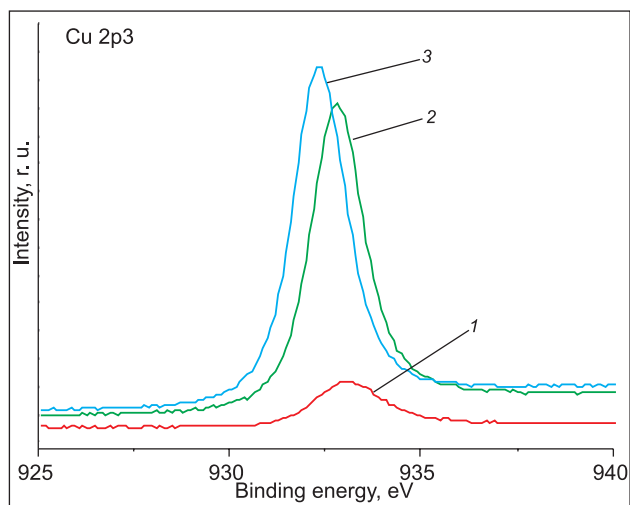


Fig. 7. XPS Cu 2p₃ spectra of Cu_xS–Cu_yTe layers on PA surface obtained during PA treatment in a 0.025 mol · dm⁻³ solution of K₂TeS₄O₆ at a temperature of 10 °C. Duration of treatment, h: 1 – 0.25, 2 – 2.0, 3 – 24

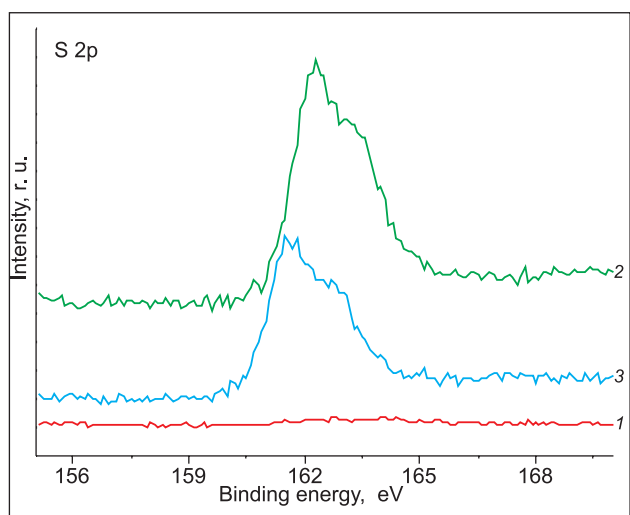
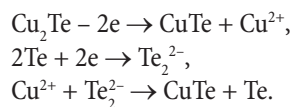


Fig. 8. XPS S 2p spectra of Cu_xS–Cu_yTe layers on PA surface obtained during PA treatment in a 0.025 mol · dm⁻³ solution of K₂TeS₄O₆ at a temperature of 10 °C. Duration of treatment, h: 1 – 0.25, 2 – 2.0, 3 – 24

with the products of this ion decomposition. The following processes are possible [32, 33]:



The decrease in the content of sulfur may be explained by formation and liberation of SO₂.

Data in Table 3 and Figs. 7–9 illustrate that the composition of the obtained layers depends on the duration of chalcogenization.

Data in Table 3 show that in an etched layer not only copper sulfide and copper telluride but also copper hydroxide are present. The metal sulfides forming layers in the polymer surface matrix by the sorption–diffusion method take the form of dendrites [34]; therefore, among the dendrites there may remain adsorbed compounds. In our case, insoluble copper hydroxide formed during washing the coating's surface with

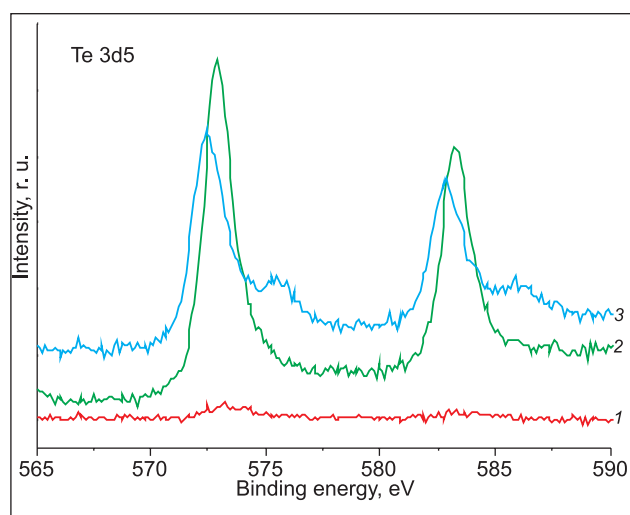


Fig. 9. XPS Te 3d₅ spectra of Cu_xS–Cu_yTe layers on PA surface obtained during PA treatment in a 0.025 mol · dm⁻³ solution of K₂TeS₄O₆ at a temperature of 10 °C. Duration of treatment, h: 1 – 0.25, 2 – 2.0, 3 – 24

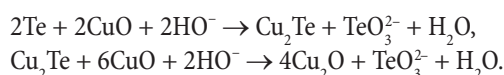
Table 3. XPS data on Cu_xS–Cu_yTe etched layers on PA surface initially chalcogenized in 0.025 mol · dm⁻³ K₂TeS₄O₆ solution at 10 °C

Duration, h	Element	Content, at. %	Binding energy, eV	Possible composition	Cu : (S + Te)	Sheet resistance, kΩ/□
0.25	Cu	47.6	930.6	Cu ₂ S, CuS	2.61	1.04 · 10 ³
	S	12.79	162.7	S ²⁻ (Cu _x S)		
	Te	5.42	572.4	Te ²⁻ (Cu _y Te)		
	O	34.19	531.5	Cu (OH) ₂		
2.0	Cu	64.97	933.1	Cu ₂ S, CuS	1.85	40.47
	S	25.14	162.3	S ²⁻ (Cu _x S)		
	Te	9.89	572.9	Te ²⁻ (Cu _y Te)		
	O	–	–			
24	Cu	55.77	932.3	Cu ₂ S, CuS	1.70	1.59
	S	24.93	161.5	S ²⁻ (Cu _x S)		
	Te	5.76; 2.05	572.45; 575.03	Te ²⁻ (Cu _y Te); TeO ₂		
	O	11.49	530.8	Cu (OH) ₂		

water remained among the dendrites. Data in Table 3 show that the largest content of oxygen (34.19 at. %) was detected in samples chalcogenized for a very short time (only 0.25 h). The reason may be the significantly smaller content of sulfur and tellurium in the sample; therefore, the chalcogenide layer is formed as separate islands, and more copper hydroxide is adsorbed in-between the islands or dendrites. Presence of separate islands predetermines a low electrical conductivity of a sample (the value of sheet resistance is $1.04 \cdot 10^3 \text{ k}\Omega/\square$); this is confirmed by the value of copper : sulfur–tellurium ratio calculated from the percentages of these elements, which was equal to 2.61.

Upon analysing data in Table 3 we have concluded that the copper : sulfur–tellurium ratio decreases with increasing the duration of chalcogenization; this is in agreement with the results of the sample sheet resistance measurements since samples with a lower value of the copper : sulfur–tellurium ratio were more conductive.

After a very long chalcogenization (24 h), in PA film a small amount of Te(IV) (2.05 at. %) was detected. It could be TeO₂ [29, 35] or TeO₃²⁻ [33]:



Summarizing XPS data, we may state that copper sulfides and copper tellurides take the major part in the layers on PA film samples.

CONCLUSIONS

1. By X-ray photoelectron spectroscopy, various copper, sulfur, tellurium and oxygen compounds (Cu₂S, CuS, S₈, Cu_xS, Cu_yTe, Cu(OH)₂, TeO₂) on the surface of polyamide samples modified with copper sulfides – copper tellurides were identified.

2. The atomic percentage of the elements has shown that copper sulfides and tellurides take the major part in the layer; the content of copper sulfides in the composition of layers is higher. The content of sulfide sulfur is highest in PA samples chalcogenized at a lowest (1 °C) temperature; the content of tellurium in the form of tellurides is highest in the sample chalcogenized in K₂TeS₄O₆ solution at 10 °C.

3. The copper : sulfur–tellurium ratio decreases with increasing the duration of chalcogenization; this is in agreement with the results samples' sheet resistance measurements since samples with a lower value of this ratio were more conductive.

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**Cu_xS–Cu_yTe SLUOKSNIŲ POLIAMIDO PLĖVELIŲ
PAVIRŠIUJE, SUDARYTŲ NAUDOJANT KALIO
TELŪROPENTATIONATĄ, TYRIMAS RENTGENO
FOTOELEKTRONINE SPEKTROSKOPIJA**

Santrauka

Fazinė poliamido 6 (PA) plėvelių, modifikuotų vario sulfidų–vario telūridų, Cu_xS–Cu_yTe, sluoksniais, sudėtis tirta Rentgeno fotoelektroninės spektroskopijos metodu. Vario sulfidų–vario telūridų sluoksniai PA plėvelių paviršiuje sudaryti, apdorojant PA plėveles 0,025, 0,05 ir 0,1 mol · dm⁻³ koncentracijos parūgštintu kalio telūropentationato, K₂TeS₄O₆, tirpalu 1, 10 ir 20 °C temperatūroje ir po to Cu(II/I) druskų tirpalu. Nustatyta, kad PA bandinių, modifikuotų vario sulfidais–vario telūridais, paviršiuje yra įvairių vario, sieros, telūro ir deguonies junginių – Cu₂S, CuS, S₈, Cu_xS, Cu_yTe, Cu(OH)₂ ir TeO₂, tačiau vario sulfidai ir telūridai sudaro didžiausią sluoksnių dalį. Vario santykio su siera–telūru vertė sluoksniuose mažėja ilginant PA chalkogeninimo trukmę. Tai sutampa su sluoksnių kvadrato varžos matavimų rezultatais, kadangi sluoksniai su mažesne vario santykio su siera ir telūru verte yra laidesni.