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The study of silver sulfide layers formed on the polyamide film surface

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Layers of silver sulfide, Ag_2S, on the PA film surface were formed by the chemical bath deposition method (CBD). Film samples were exposed in acidified solutions of silver nitrate, $AgNO_3$, and sodium thiosulfate, $Na_2S_2O_3$. Zeta potential (ζ) measurement was applied for estimating the stability of colloidal particles obtained by the reaction from $AgNO_3$ and excess of $Na_2S_2O_3$ solutions at pH 2.3 and 20 °C, respectively. Samples prepared as Ag_2S gave multiple reflections, all corresponding to monoclinic Ag_2S (acanthite). The morphology of polyamide PA films modified by silver sulfide layers was studied by the atomic force microscopy method. The formation of silver sulfide layers proceeded unevenly, in the form of islands, which later grew into agglomerates. The surface of the formed layers was relatively uneven and rather rough. The average height of Ag_2S layer increased (~145 – ~1 093 nm) with the increasing exposure time of PA treatment in solutions. The layer roughness varied from ~32 to ~244 nm and with the increasing exposure time in precursor solutions regularly increased and caused changes of the shape distribution function of the surface elements.

Key words: silver sulfide layer, polyamide, zeta potential, XRD, morphology, AFM

INTRODUCTION

Composite materials consisting of polymers covered by thin layers of inorganic compounds possess characteristic optical and semiconducting properties. Ag_2S has recently been recognized as a semiconductor with unique properties such as enhanced and rapid optical nonlinearity and high luminescence. Ag_2S can be both a direct and an indirect band semiconductor, with band gaps $E_{g,\,\mathrm{dir}}$ of ~1 and $E_{g,\,\mathrm{ind}}$ of ~2.3 eV [1]. The band gap varies depending on the particle size. The semiconductor silver sulfide thin films have a number of applications in various devices, such as solar cells, photoconductors, IR detectors, where adhesion, optical and semiconductor properties are needed [2–10].

A number of physical and chemical deposition techniques such as flash evaporation [11], radio frequency sputtering [12], thermal evaporation [13–15], solution growth [16, 17] and hot pressing [18] have been used for the preparation of metal chalcogenide layers under various experimental conditions. All these techniques have advantages and disadvantages, depending on the type of application intended for the films. It is important to obtain high quality Ag_2S films for improving solar cell performance. In addition, the CBD method was chosen because of its simple technique, low cost and low temperature atmospheric processing. Furthermore, preparation of Ag_2S is complicated by the strong tendency of Ag_2S clusters to aggregate to form bulk precipitates [19, 20].

Zeta potential ζ, which is a consequence of the existence of surface charge, can give information about electrical interaction forces between the dispersed particles. Zeta potential is defined as the electrokinetic potential at the boundary of the hydrodynamic shear plane of a charged particle adjacent to a solid surface exposed to a liquid. The concept of ζ has been applied very successfully in the field of colloid science seeking to understand the colloid interactions and control the stability of colloid system. For colloidal stability, the repulsive

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forces must be dominant [21]. The magnitude of ζ gives an indication of the potential stability of the colloidal system. Generally, when all the particles have a large positive or negative ζ (where the positivity and negativity are greater or lower than –30 mV and +30 mV), they will repel each other and the dispersion is stable. On the other hand, when the particles have low ζ values, there will be no force to prevent the particles from aggregating [22].

In this paper, an attempt has been made to grow Ag_2S layers on polyamide 6 (PA) substrates by the chemical bath deposition method, which has also been studied. It is a report on the synthesis of Ag_2S thin films using the CBD technique in which Ag_2S formation speed can be slowed.

The aim of the present work was to form Ag_2S layers on the surface of the hydrophilic polymeric material – polyamide 6 film (PA/Ag_2S) in order to obtain flexible thin films. The analysis of this material included studies on the structure and morphology of its surface. Several techniques such as zeta potential measurements, XRD and AFM were used to characterize the PA/Ag_2S film.

EXPERIMENTAL

Layers of silver sulfide, Ag_2S, were deposited on PA films (grade PK-4, 15 mm × 70 mm, 70 µm). PA films were boiled in distilled water for 2 h seeking to remove the monomer residues. They were dried with filter paper and then over anhydrous $CaCl_2$ for 24 h.

The porosity was measured by the BET method using a Quantasorb sorption system (USA). The pores of PA 6 are much less than 1.5 nm [23]. Film density was checked by the flotation method. It was found that the density of PA is equal to 1.13 g/cm^{-3} [23].

Distilled water, reagents of "especially pure" and "analytically pure" grades were used to produce solutions. Samples of PA were treated with aqueous $Na_2S_2O_3 \cdot 5H_2O$ (0.2 mol/dm^3) and $AgNO_3$ (0.06–0.08 mol/dm^3) solutions at 20 °C with pH 2.3 (by adding nitric acid). All operations of PA treatment were carried out in a glass reactor. PA films were immersed for 40 min up to 6 times in a precursor solution. At certain time intervals, samples were withdrawn, rinsed with distilled water, dried with filter paper, left over anhydrous $CaCl_2$ for 24 h and then used in analysis and in further experiments.

Solutions' pH was measured by using a pH-meter WTW330, with a combinative glass and Ag/AgCl electrode and a temperature meter WTW SenTix 41 (Germany).

The zeta potential (ζ) of particles was determined using an electroacoustic spectrometer DT-300 (Dispersion Technology Inc., USA). Measurements were carried out at 20 °C, with pH 2.3. 30 millilitres of solutions were used. The suspension was well mixed and allowed to sediment for 40 min.

X-ray diffractometry was carried out under a Brag Brendan circuit on a diffractometer (Dron-6, Russia) using Cu Kα (λ = 0.154178 nm) radiation, 30 kV voltage and 30 µA current. The scanning range was 2θ = 25–60°. The scanning speed was 1° · min^{-1}. Results were registered in in situ mode with a computer, and X-ray diffractograms of Ag_2S scrapings (powder) from PA samples surface were treated using the Search Match, Xfit, ConvX, Dplot95, and Photo Styler programs.

The morphology of Ag_2S layers surface and the quantitative microscopy of their roughness were studied with a NT-206 atomic force microscope (Belarus), in the contact regime with high resolution probes with the force constant k = 3 N/m. The characteristics of the atomic force microscope: the maximum scan field area from 12 × 12 up to 30 × 30 microns, the measurement matrix up to 512 × 512 points and more, the maximum range of measured heights 4 microns, lateral resolution 2 nm, and vertical resolution 0.1–0.2 nm. Lateral force microscopy studies are useful for imaging variations in surface friction that can arise from inhomogeneity in surface material, and also for obtaining edge-enhanced images. Data of measurements were analysed using the Surface View 2.0 program.

RESULTS AND DISCUSSION

Measurements of the zeta potential of particles in solutions

In order to obtain the silver sulfide layers on the PA film it was necessary to find the optimal conditions of the chemical bath deposition process. Silver sulfide may be incorporated in the films immersed in aqueous solutions of silver nitrate ($AgNO_3$) and sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$). When soluble thiosulfate reacts with silver nitrate, insoluble silver thiosulfate, $Ag_2S_2O_3$, (the solubility product for $Ag_2S_2O_3$ is $K_{sp} = 1 \times 10^{-13}$ [24]) is formed. $Ag_2S_2O_3$ was comparatively unstable and hydrolyzed to release Ag_2S into the reaction solution. In previous experiments trying to use $AgNO_3$ and $Na_2S_2O_3$ solutions for Ag_2S layers depositing on PA, Ag_2S layers could not be formed. This indicated that the slow release of silver sulfide particles was essential for the formation of the layers.

In order to slow down the speed of the formation process of silver sulfide, the silver ions can be complexed by the thiosulfate solution. When the sodium thiosulpfate solution was added into the silver nitrate aqueous solution, the silver ions were coordinating with some thiosulpfate groups, resulting in the relatively high silver ions concentration around these groups. In the presence of excess thiosulfate, the soluble $[Ag_2(S_2O_3)_2]^{2-}$, $[Ag_2(S_2O_3)_3]^{4-}$ and $[Ag_2(S_2O_3)_4]^{6-}$ complexes formed. In the nitric acid solution these complexes were decomposed and the slow-released S^{2-} ions combined with the Ag^+ ions to form very insoluble Ag_2S (the solubility product for Ag_2S is K_{sp} = 7.2 × × 10–50 [24]) nuclei on some special sites of PA.

It was found that the most suitable concentrations for the formation of silver sulfide layers on the polyamide surface were 0.08 mol/dm^3 $AgNO_3$ and 0.20 mol/dm^3 $Na_2S_2O_3$ solutions at pH 2.3 and 20 °C temperature.

The silver sulfide precipitate, which settled on the reactor bottom in the first series of experiments, was collected, filtered, washed, crystallized and dissolved in nitric acid.

Titration with the standard NaCl solution showed that the concentration of the produced $AgNO_3$ solution was 0.06 mol/dm³. This solution was used for the second series of polymer samples treatment for Ag_2S deposition.

Seeking to better understand the interaction of Ag_2S with the polymers, changes in the zeta potential were determined. From a theoretical viewpoint, the zeta potential is the potential difference between the dispersion medium and the stationary layer of fluid attached to the dispersed particle. Zeta potential measurements of aqueous solutions of 0.06 mol/dm³ and 0.08 mol/dm³ concentrations $AgNO_3$ and excess of $Na_2S_2O_3$ 0.20 mol/dm³ concentration dispersions at pH 2.3 gave the negative particle charge. The negative particle charge was found to be approximately –47 – –49 mV. In agreement with the literature data [21, 22, 25], the zeta potential has shown a good stability of particles. With increasing reaction time the zeta potential changed less and had a significantly negative charge with a value of approximately –52 – –55 mV. Changes of the zeta potential depending on the reaction time are shown in Fig. 1. This negative charge was reflected in the stability of these suspensions which showed little aggregation upon standing for 40 min.

X-ray analysis

Structural studies of the Ag_2S deposits on polyamide are limited by the crystallinity of the PA film itself. The intensity of its peaks exceeds the intensity of silver sulfide peaks several times. To characterize the deposited films, deposits from PA samples, immersed in the precursor solutions with 0.08 mol/dm³ $AgNO_3$ and 0.2 mol/dm³ $Na_2S_2O_3$ at 20 °C temperature for 240 min (6 immersions in 40 min), were scraped with a non-metallic scraper, and then these powders (scrapings) were subjected to X-ray diffractometry to investigate the crystallographic structure of a thin film.

Fig. 2 presents the X-ray diffraction pattern of the as-prepared Ag_2S scrapings (powders). The phase composition was determined by comparing its X-ray images with those of known minerals. The diffraction pattern gave dominant peaks 2θ at 26.189° (crystallographic planes (111), 29.063°(012), 31.820°(120), 33.666°(121), 34.743°(112), 36.806°(022), 37.6°(200), 40.798°(031), 43.47°(130), 46.28°(202) and 53.21°(004)) showing the polycrystalline nature of the prepared powder. In Fig. 2, all peaks can be indexed to the monoclinic Ag_2S phase, in good agreement with the reported data for α-Ag_2S (acanthite) (JCPDS Card File: 00-014-0072).

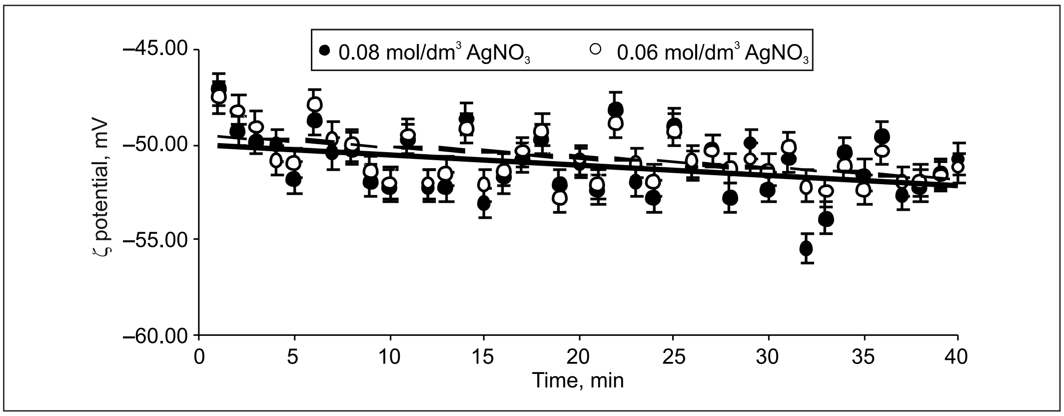


Fig. 1. Changes of zeta potential depending on the reaction time in 0.06–0.08 mol/dm³ $AgNO_3$ and 0.20 mol/dm³ $Na_2S_2O_3$ solutions at 20 °C

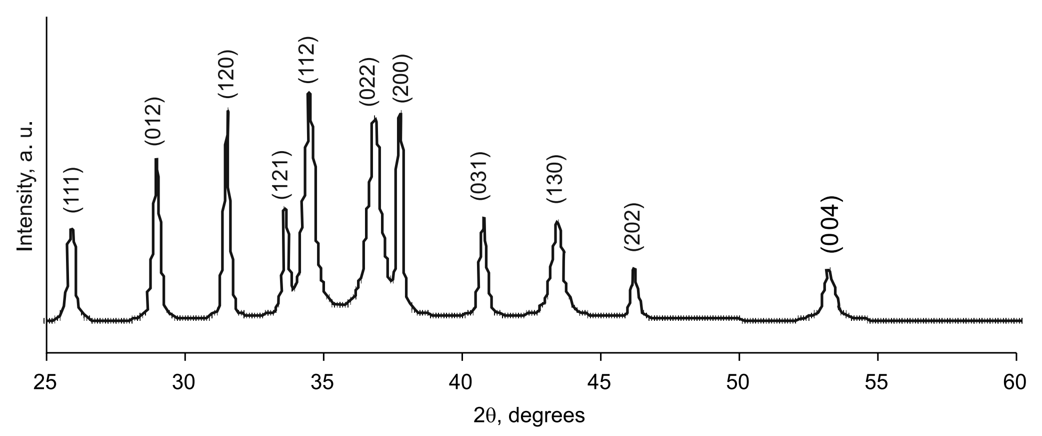


Fig. 2. X-ray diffractograms of the Ag_2S layers formed on PA surface scrapings (powder). PA was immersed 6 times for 240 min (6 × 40 min) at 20 ° in 0.06 mol/dm³ $AgNO_3$ and 0.20 mol/dm³ $Na_2S_2O_3$ solutions

AFM study

The samples of PA were immersed in the precursor solution with 0.06 mol/dm^3 $AgNO_3$ and 0.2 mol/dm^3 $Na_2S_2O_3$ at 20 °C temperature several times in order to study the morphology characteristics of the surface according to the incorporated Ag_2S amount. The immersion steps were accompanied by atomic force microscopy. Fields of 12 × 12 microns were investigated; for the quantitative estimation of the surface, the standard programs "Surface Scan" and "Surface Explorer" of the view treatment were used.

A view of the virgin PA surface is presented in Fig. 3 and shows a continuous roughness distribution. The maximum height reaches ~67 nm, the average height (H_{mean}) ~28 nm; the root means square roughness R_q 4.6 nm; the skewness is ~0.8.

The views of the PA/Ag_2S films with one and six immersions in the precursor solution are shown in Figs. 4–7. Fig. 4 and 5 indicate the creation of an irregular but continuous base of islands in the first step. The next immersion of PA in the precursor solution induced the Ag_2S island growth. When the number of immersions increased, the growth of the silver sulfide layer proceeded more intensively and the islands aggregation process was observed. After six immersions (Fig. 6), some growing regions of agglomerates over the previous layer can be seen. Then an increase of the agglomerates size and the maximum length of the grains covering the agglomerates was observed. Fig. 7 displays the modified PA surface after six immersions and it is possible to see regions with good agglomeration, higher density, making the surface more uniform and the fusion of the small crystallites. Figs. 4 and 6 illustrate the topographic profiles of the PA/Ag_2S films prepared with one and six immersions in the metallic sulfide precursor solutions.

Otherwise, regarding the number of immersions, different roughness profiles are observed. The regular growth of particles is shown in Fig. 4.

Profile sections of Ag_2S layers formed on the PA surface when the polymer was immersed for one and six immersions, allowed estimating quantitatively the height and diameter of separate grains (Fig. 4 and 6).

The dependence of the surface morphology of PA/Ag_2S layers and the number of immersions in precursor solutions were observed. It was also found that the uniformity of the layer depended on the number of immersions.

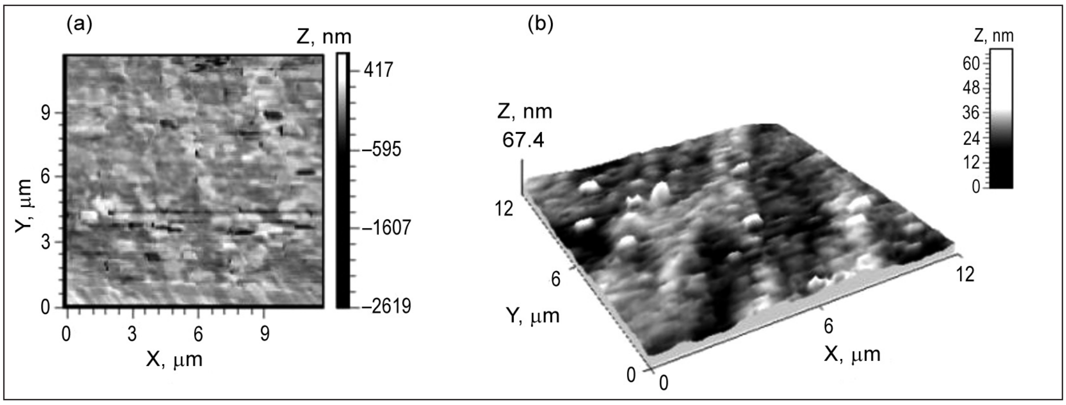
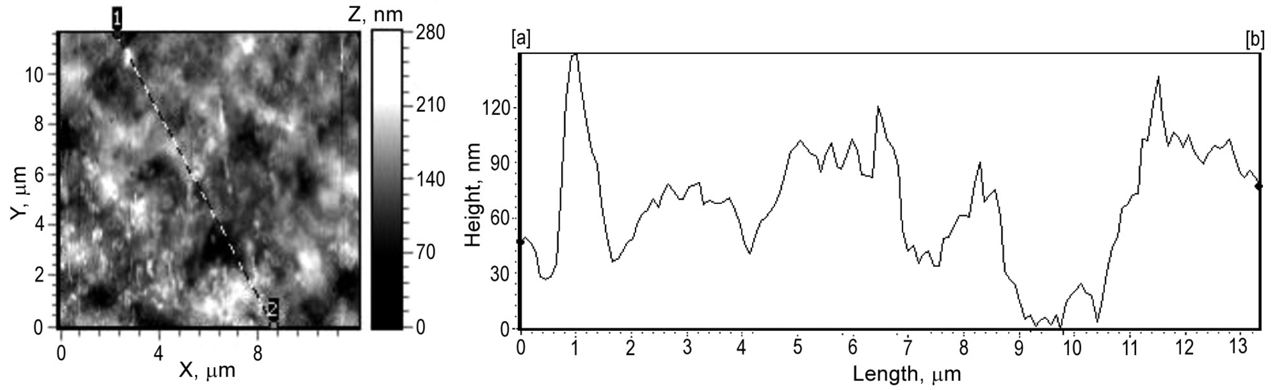


Fig. 3. Atomic force microscopy view of PA surface: 2D view of lateral forces microscopy (a) and 3D topography view (b). Scan field area 12 × 12 microns



Absolute values: [a] → $x = 0.0$ µm; $z(1) = 46.9$ nm; [b] → $x = 13.3$ µm; $z(1) = 77.3$ nm

Difference between markers: $dx = 13.3$ µm; $dz(1) = 30.4$ nm

$H_{mean} = 145.49$ nm; $R_a = 24.52$; $R_q = 31.75$; $R_{sk} = -0.12$

Fig. 4. View of the topographic image and the line profile of the silver sulfide layer formed on PA surface. PA was immersed 1 time for 40 min at 20 °C in 0.06 mol/dm^3 $AgNO_3$ and 0.20 mol/dm^3 $Na_2S_2O_3$ solutions

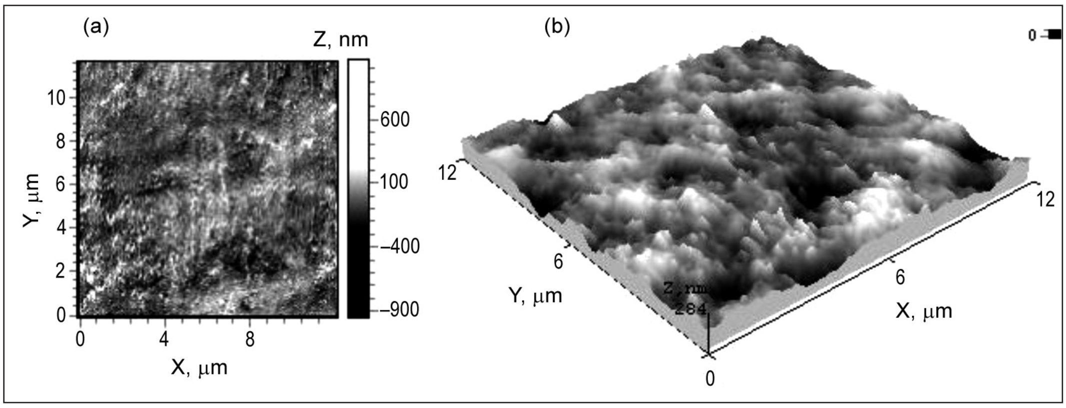
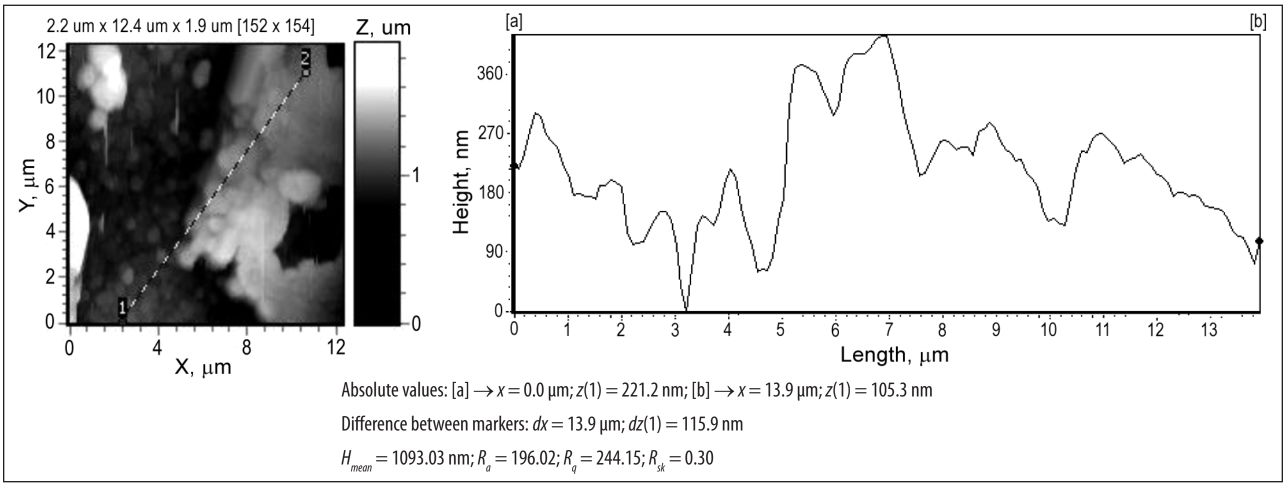


Fig. 5. Atomic force microscopy view of Ag_2S layers formed on PA surface: 2D view of lateral forces microscopy (a) and 3D topography view (b). PA was immersed 1 time for 40 min at 20 °C in 0.06 mol/dm³ $AgNO_3$ and 0.20 mol/dm³ $Na_2S_2O_3$ solutions

The growth of the silver sulfide layer begins from the formation of separate small islands (Fig. 4). Prolonging the immersion time to 6 cycles, the growth of the sulfide layer proceeds more intensively (Fig. 6) and clusterization begins; as a result, the grains of various diameter and height begin gradually joining into agglomerates (Fig. 7). The size of grains, like the roughness of the layer, increases with the increasing immersion time in precursor solutions. Roughness varies from ~32 nm after the first immersion to ~244 nm after six immersions (Fig. 8). This means that grains join into agglomerates



Absolute values: [a] ⟶ $x = 0.0$ µm; $z(1) = 221.2$ nm; [b] ⟶ $x = 13.9$ µm; $z(1) = 105.3$ nm

Difference between markers: $dx = 13.9$ µm; $dz(1) = 115.9$ nm

$H_{mean} = 1093.03$ nm; $R_a = 196.02$; $R_q = 244.15$; $R_{sk} = 0.30$

Fig. 6. View of the topographic image and the line profile of the silver sulfide layer formed on PA surface. PA was immersed 6 times for 240 min (6 × 40 min) at 20 °C in 0.06 mol/dm³ $AgNO_3$ and 0.20 mol/dm³ $Na_2S_2O_3$ solutions

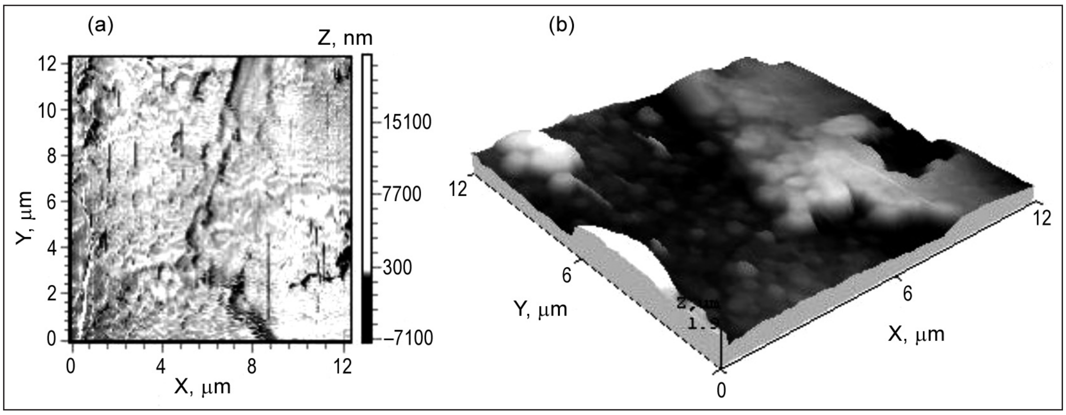


Fig. 7. Atomic force microscopy view of Ag_2S layers formed on the PA surface: 2D view of lateral forces microscopy (a) and 3D topography view (b). PA was immersed 6 times for 240 min (6 × 40 min) at 20 °C in 0.06 mol/dm³ $AgNO_3$ and 0.20 mol/dm³ $Na_2S_2O_3$ solutions

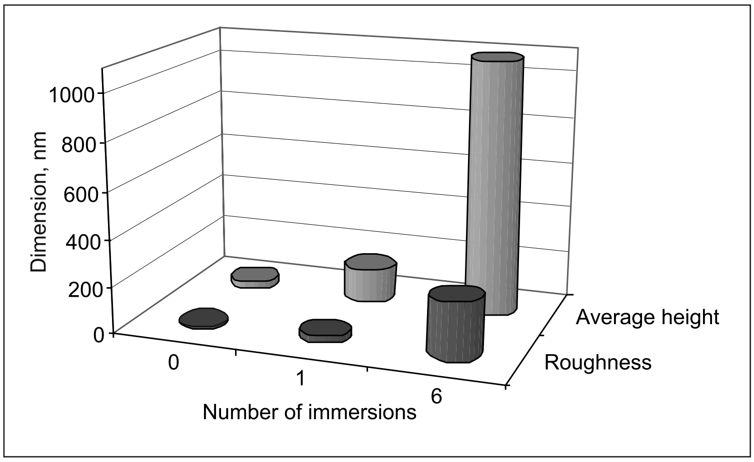


Fig. 8. Dependence of the layer mean square roughness (R_q) and the mean height (H_{mean}) on the immersion time of PA treatment in 0.06 mol/dm^3 $AgNO_3$ and 0.20 mol/dm^3 $Na_2S_2O_3$ solutions at temperature of 20 °C

covering the surface more evenly, and the layer becomes more homogeneous. The average height of the Ag_2S layer increases from ~145 nm after the first immersion to ~1 093 nm after six immersions (Fig. 8) in precursor solutions.

Thus, the AFM results indicate that depending on the immersion time in precursor solutions, the formation of Ag_2S layers proceeds regularly in the form of islands which later grow into larger agglomerates. The surface of the layer is uneven, rather rough.

The virgin PA skewness (R_{sk}) is equal to 0.8 and indicates that raised elements dominate on the surface. After the first PA samples immersion, skewness is equal to –0.12 and indicates that the surface becomes porous, deep elements dominate. With the increasing immersion time skewness becomes equal to 0.3, which indicates that raised elements again dominate in the surface. The height is a bit higher than the surface average height. The asymmetry parameter R_{sk} displays the domination of islands, grains and agglomerates on the surface.

Zeta potential measurements and AFM study let understand and explain the formation process of silver sulfide layers on the polyamide surface.

Zeta potential measurement shows that the reaction between $AgNO_3$ and $Na_2S_2O_3$, under low pH and temperature produces negatively charged colloidal particles of silver sulfide. In colloidal chemistry, particle size increasing usually occurs by the Ostwald Ripening Mechanism [26, 27]. This theory is based on the mechanism in which the particles of colloidal suspension grow up by the consumption of small particles, due to the different chemical potential between them. In polydisperse suspension, small particles tend to solubilize and re-precipitate closer to bigger ones and an increase of the average grain size occurs with time [28–30]. When a silver sulfide particle is formed in situ (i.e. in contact with the PA surface), electrostatic interactions between the charged sites of the polymer and the silver sulfide particles take place, providing its adhesion to the polymeric surface. Thus, the sulfide-polymer surface adhesion is the result of an interfacial reaction. Once the nuclei were formed, the growth of Ag_2S particles started on them. Then the colloidal Ag_2S particles gradually formed deposits, followed by the dissolution-precipitation equilibrium in the Ostwald Ripening process. As a result, the as-prepared Ag_2S layer was obtained. According to the literature [31, 32] and our experimental data [23], it is suggested that there might be the electrostatic interaction between metal sulfides and charged sites of the ionised polyamide functional groups –CO–NH– and $–NH_2$, which played an important role in the formation of metal sulfides layers. In addition, the Ag_2S particles can also be attracted to polyamide for surface binding via sulfur linkages.

According to the discussion above, it is proposed that the Ag_2S deposits could be formed through the Ostwald Ripening process, sulfide-polymer surface adhesion and electrostatic interaction.

CONCLUSIONS

1. Zeta potential of Ag_2S particles was found to be negatively charged by approximately –47 – –55 mV. It was reflected in the stability of suspensions which showed little aggregation upon standing for 40 min.

2. The X-ray analysis of deposited film scrapings from PA samples confirmed the presence of polycrystalline Ag_2S.

3. The Ag_2S film growth starts only on some spots of the substrate surface. Later, it is achieved on the total coverage of the substrate; the film gets smoother, more homogeneous and rather flat.

4. The surface morphology, contexture and uniformity of the silver sulfide layers on the PA surface depend on the immersion time of the polymer in precursor solutions.

5. The layer average height (~145 – ~1 093 nm) increased regularly increasing the immersion time. The roughness of modified PA films varies from ~32 to ~244 nm.

6. Ag_2S deposits on PA could be formed through the Ostwald Ripening process, sulfide-polymer surface adhesion and electrostatic interaction.

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SIDABRO SULFIDO SLUOKSNIŲ, SUDARYTŲ POLIAMIDO PLĖVELĖS PAVIRŠIUJE, TYRIMAS

Santrauka

Sidabro sulfido (Ag$_2$S) sluoksniai PA paviršiuje suformuoti cheminio nusodinimo metodu apdorojant plėveles 0,06–0,08 mol/dm^3 koncentracijos AgNO$_3$ ir 0,20 mol/dm^3 koncentracijos Na$_2$S$_2$O$_3$ tirpalais. Reakcijos metu esant pH 2,3 ir 20 °C temperatūrai, susidariusių koloidinių dalelių stabilumas įvertintas išmatavus dzeta potencialą (ξ). Rentgeno struktūrinė fazinė analizė parodė, kad susiformavo monoklininis Ag$_2$S (akantitas). Atominių jėgų mikroskopijos (AJM) metodu ištirta sidabro sulfido sluoksnių, sudarytų poliamido plėvelių paviršiuje, morfologija. Nustatyta, kad sidabro sulfido sluoksniai formuojasi netolygiai, salelėmis, kurios suauga į didesnius aglomeratus. Susidariusios dangos paviršius nelygus, gana šiurkštus. Vidutinis Ag$_2$S dangos aukštis kinta nuo ~145 nm iki ~1093 nm keičiantis polimero apdorojimo AgNO$_3$ tirpalo koncentracijai bei ciklų skaičiui. Sidabro sulfido sluoksnių paviršiaus šiurkštumas kinta ~32–~244 nm intervale.