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THE INFLUENCE OF THE TEMPERATURE OF A MIXTURE OF LEAD NITRATE AND SODIUM HYDROXIDE SOLUTIONS ON THE GROWTH PECULIARITIES OF LEAD SULFIDE FILMS

The present work is devoted to studying the effect of temperature on the formation of a submicron-sized particles of lead sulfide (PbS). Lead sulfide samples were obtained by chemical bath deposition method. 25 ml of an aqueous solution of lead nitrate $Pb(NO_3)_2$, 75 ml of sodium hydroxide NaOH and 50 ml of thiourea CH_4N_2S with molar concentrations of 0.18 M (1.52 g), 0.38 M (1.151 g) and 0.11 M (0.397 g), respectively, were used as reagents. In the process of obtaining samples, solutions of lead nitrate and sodium hydroxide were initially mixed at temperatures of 70°C and 100°C, then a thiourea solution was added to this mixture. It was found that temperature has an effect on the formation of PbS structures. It is shown that at a temperature of 100°C isolated particles are formed, whereas at a temperature of 70°C a film is formed.

The morphology and elemental composition of the obtained lead sulfide (PbS) samples were studied by scanning electron microscopy (SEM) and the surface structure was studied using an atomic force microscope (AFM). As a result of the study of the morphology and texture of the surface of lead sulfide samples, the average size of isolated particles was determined, which is ~144 nm, and these particles also had a cubic shape with a smooth surface.

Keywords: lead sulfide, film, isolated particles, temperature, optimal parameters.

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Қорғасын сульфиді пленкаларының өсу ерекшеліктеріне қорғасын нитраты мен натрий гидроксиді ерітінділер қоспасы температурасының әсері

Бұл зерттеу жұмысы температураның қорғасын сульфиді (PbS) субмикрондық өлшемді бөлшектерінің түзілуіне әсерін зерттеуге арналған. Қорғасын сульфидінің үлгілері сулы ерітіндіден химиялық тұндыру әдісімен алынды. Реагенттер ретінде 25 мл қорғасын нитраты Pb $(NO_3)_2$, 75 мл натрий гидроксиді NaOH және 50 мл тиомочевина CH_4N_2S тиісінше молярлық концентрациялары бойынша 0,11 М (0,397 г), 0,18 М (1,52 г), 0,38 М (1.151 г) сулы ерітінділері пайдаланылды. Үлгілерді алу процесінде бастапқы кезеңде қорғасын нитраты мен натрий гидроксиді ерітінділері 70°С және 100°С температурада араластырылды, содан кейін бұл қоспаға тиомочевина ерітіндісі қосылды. Температура PbS құрылымдарының қалыптасуына әсер ететіні анықталды. 100°С температурада оқшауланған бөлшектер пайда болды, ал 70°С температурада пленка пайда болды.

Алынған қорғасын сульфидінің (PbS) үлгілерінің морфологиясы мен элементтік құрамы сканерлеуші электронды микроскопия (СЭМ) әдісімен зерттелді және беттік құрылымы атомдық күштік микроскопы (АСМ) арқылы зерттелді. Қорғасын сульфидінің үлгілерінің морфологиясы мен беткі қабатын зерттеу нәтижесінде оқшауланған бөлшектердің орташа мөлшері анықталды, ол ~144 нм құрайды, сонымен қатар бұл бөлшектер беті тегіс текше түрінде болды.

Түйін сөздер: қорғасын сульфиді, пленка, оқшауланған бөлшектер, температура, оңтайлы параметрлер.

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Влияние температуры смеси растворов нитрата свинца и гидроксида натрия на особенности роста пленок сульфида свинца

Настоящее исследование посвящено изучению влияния температуры на образование частиц сульфида свинца (PbS) субмикронного размера. Образцы сульфида свинца были получены методом химического осаждения из водного раствора. В качестве реагентов использовались 25 мл водного раствора нитрата свинца $Pb(NO_3)_2$, 75 мл гидроксида натрия NaOH и 50 мл тиомочевины CH_4N_2S с молярными концентрациями 0,18 М (1,52 г), 0,38 М (1.151 г) и 0,11 М (0,397 г), соответственно. В процессе получения образцов на начальном этапе производилось перемешивание растворов нитрата свинца и гидроксида натрия при температурах 70°C и 100°C, затем в данную смесь добавлялся раствор тиомочевины. Было установлено, что температура оказывает влияние на формировние структур PbS. Показано, что при температуре 100°C образуются изолированные частицы, тогда как при температуре 70°C формируется пленка.

Морфология и элементный состав полученных образцов сульфида свинца (PbS) изучались методом сканирующей электронной микроскопии (СЭМ) и поверхностная структура была изучена с помощью атомно-силового микроскопа (АСМ). В результате исследования морфологии и струткутуры поверхности образцов сульфида свинца был определен средний размер изолированных частиц, который составляет ~144 нм, кроме того данные частицы имеют кубическую форму с гладкой поверхностью.

Ключевые слова: сульфид свинца, пленка, изолированные частицы, температура, оптимальные параметры.

Introduction

Lead sulfide (PbS) is an inorganic chemical compound of lead and sulfur. The appearance of crystalline substance ranges from blue-gray to silver-gray color. In nature it is found in the form of the mineral galena [1-3]. In the laboratory it can be obtained by reaction between solutions of lead salt and sodium sulfide. Lead sulfide (PbS) is recognized as an excellent material for infrared luminescence with several advantages including a direct narrow band gap of 0.41 eV, broad spectral absorption in the visible to near infrared region, and remarkable quantum confinement effects of charge carriers [4]. Lead sulfide is a semiconductor material. The lattice parameter is 0.593 nm. There are different methods of lead sulfide production. One method is chemical vapor deposition (CVD), where lead sulfidization occurs. This process uses lead (Pb) vapor, hydrogen sulfide (H_2S). This process uses atmospheric pressure, inert gas (Ar, N_2), reaction temperature is from 400-800°C. This process has disadvantages such as limited particle shape and size and the need to control temperature and pressure [5-9]. There is also pyrolysis of aerosol, in this method lead salt solution ($Pb(NO_3)_2$) and thiourea ($CS(NH_2)_2$) are used as starting substances. To make the process run well, the reaction temperature should be 400-600°C and the pressure should be atmospheric, inert gases such as (Ar, N_2) are used as an admixture. The advantages of this method are that the shape and size of the particles can be controlled, but the disadvantage is that the sedimentation rate is very low and the process is very complicated [10-14]. The third method is physical vapor deposition (PVD), which is a vacuum deposition method, in this method a target of lead (Pb) and sulfur (S) is used as a starting material. In this process the lead is evaporated in vacuum, the reaction takes place with sulfur vapor and sedimentation on the substrate. The advantages of this reaction are that the product has high purity, the film thickness can be controlled, but also this process has its disadvantages such as the cost of this process is very high and has its own difficulties in the reaction [15]. The fourth method is ion beam deposition. In ion beam deposition, lead ions (Pb^+) and hydrogen sulfide (H_2S) are used as precursors. This method is carried out through bombarding the substrate with lead ions and reacting with hydrogen sulfide. As a result lead sulfide is formed. The advantage of this process is that the process is carried out at low temperature and the shape and size of the particles can be controlled, besides it has the disadvantage that the deposition takes place at a low rate [16]. The fifth method is molecular beam epitaxy. In molecular beam epitaxy, molecular beams of lead (Pb) and sulfur (S) are used as a starting material and lead and sulfur molecules are deposited on a substrate. In this process, high quality crystals grow and the doping process can be controlled. The disadvantage of this process is that it is very expensive and has its own difficulties [17-20]. The sixth method is laser deposition. This process requires laser light and lead sulfide (PbS) target as the starting material. The process takes place by laser vaporization of the target and deposition on the substrate. The advantages of this method are that the process is very fast and the shape and size of the particle can be controlled. The disadvantage of this method is that the purity of the product is very low and the process has its own difficulties [21-23]. Seven is the most commonly used method of chemical bath deposition (CBD) because it is a very simple, cost-effective and economically reproducible method that can be used for large area deposition at low temperature. Chemical bath deposition is used to deposit thin films from a wide range of materials. The deposition mechanism is essentially the same for all such materials. A soluble salt of the desired metal is dissolved in an aqueous solution to release the cations. A non-metallic element is provided by a suitable starting compound, which is decomposed in the presence of hydroxide ions to release anions. The anions and cations then react to form a compound [24].

In this work, we report the effect of reaction temperature on the structure of lead sulfide obtained by chemical method from aqueous solution.

Experiment

Lead sulfide samples were obtained by chemical precipitation on silicon substrate from aqueous solution. A mixture of aqueous solutions of 25 mL of lead nitrate $Pb(NO_3)_2$ 0.18 M (1.52 g) and 75 mL of sodium hydroxide NaOH 0.38 M (1.151 g) were mixed using a magnetic stirrer for 60 minutes at temperatures of 70°C and 100°C. At the initial moment of the process, the mixture of the mixed solutions was transparent, further during the mixing process it started to change color from transparent to yellow and small golden particles were formed in the mixture (Figure 1*a*). 50 mL aqueous thiourea CH_4N_2S solution with a molar concentration of 0.11 M (0.397 g) was prepared for 30 minutes at room temperature (Figure 1*b*). Aqueous thiourea solution was added to the mixture of lead nitrate and sodium hydroxide solutions (Figure 1*c*).

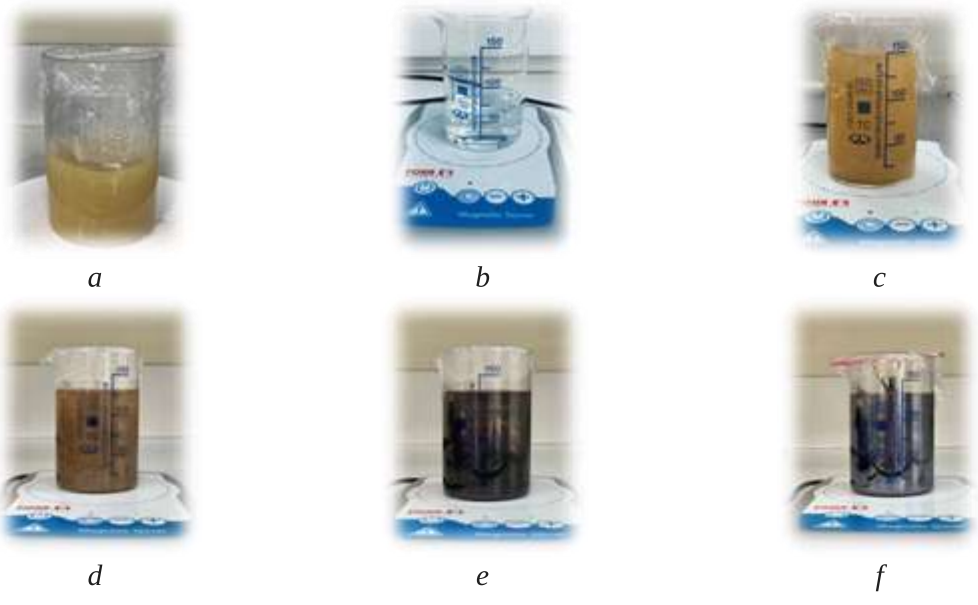

Figure 1-Solutions of lead nitrate, sodium hydroxide, and thiourea to produce isolated particles and films of lead sulfide (PbS) as a function of temperature

After adding a thiourea solution to a mixture of lead nitrate and sodium hydroxide, the process of lead sulfide synthesis began. During the process, during the first four minutes of the reaction, the resulting solution changed its color from brown to dark gray. At the first minute the color of the solution was light brown, and in the second minute it changed to a more brown color (Figure 1*d*), and in the third minute the solution had a darker shade (Figure 1*e*), and in the fourth minute a thin film of lead sulfide was formed on the surface of the beaker (Figure 1*f*). And after that, the silicon substrate was immersed in the solution. The synthesis was carried out for 35 minutes and the obtained samples were cleaned in an ultrasonic bath for 15 minutes at room temperature. The chemical reaction of the formation of inorganic lead sulfide compound took place according to the following formula:

$$Pb(NO_3)_2 + 2NaOH + CH_4N_2S \longrightarrow H_2O + PbS + CH_4N_2O + 2NaNO_3 \quad (1)$$

The obtained samples were examined by scanning electron microscope (SEM), and the elemental composition was also determined by energy dispersive anlysis on Quanta3D 200i. The surface structure of the obtained lead sulfide samples was investigated by atomic force microscopy (AFM), which allowed to study their morphological characteristics.

Results and discussion

Figure 2 shows the surface morphology of the samples obtained by scanning electron microscope. Figure 2*a* shows the morphology of lead sulfide sample obtained at a temperature of 70°C mixture of lead nitrate and sodium hydroxide solutions and its elemental composition is shown in Figure 2*b*. This figure shows that at this temperature, a continuous film with a rough surface formed from cubic shaped crystallites is formed. From the elemental analysis, the atomic of lead was determined to be 49.76% and sulfur 50.24%, which shows that the elemental composition corresponds to the stoichiometric percentage ratio of lead sulfide (PbS).

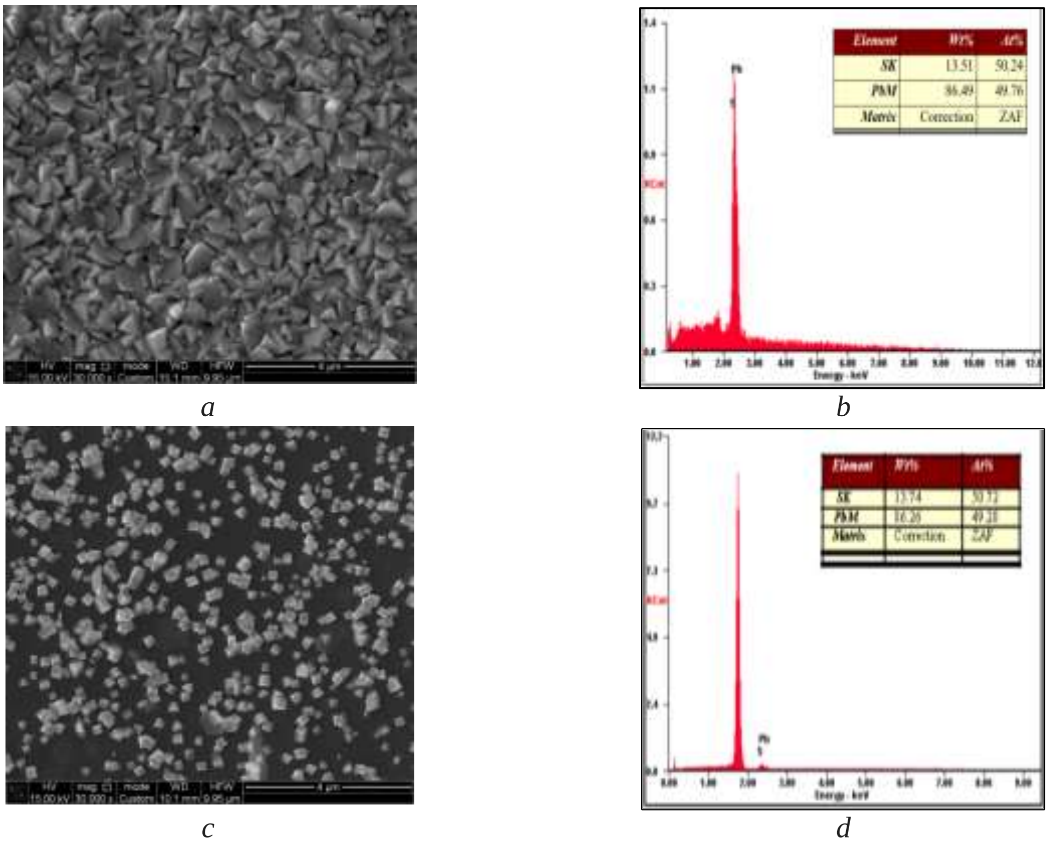

Figure 2-(*a*), (*b*), SEM image and energy dispersive analysis of PbS lead sulfide film and (*c*), (*d*) SEM image and energy dispersive analysis of isolated PbS lead sulfide particles

Figure 2 (*c*, *d*) shows the SEM image and elemental composition of lead sulfide obtained at the temperature of 100°C mixture of lead nitrate and sodium hydroxide solutions. As can be seen from the figure, the obtained sample of lead sulfide has separate isolated particles of cubic shape with an average size of ~144 nm, which repeats the crystal structure of lead sulfide itself. From the data of EDS analysis it follows that the particles have 49.28 at.% and sulfur 50.72 at.%, in which as well as in lead sulfide films the stoichiometric ratio is observed. The insignificant deviation of atomic percentages does not exceed 1%, which can be related to the accuracy of measurement.

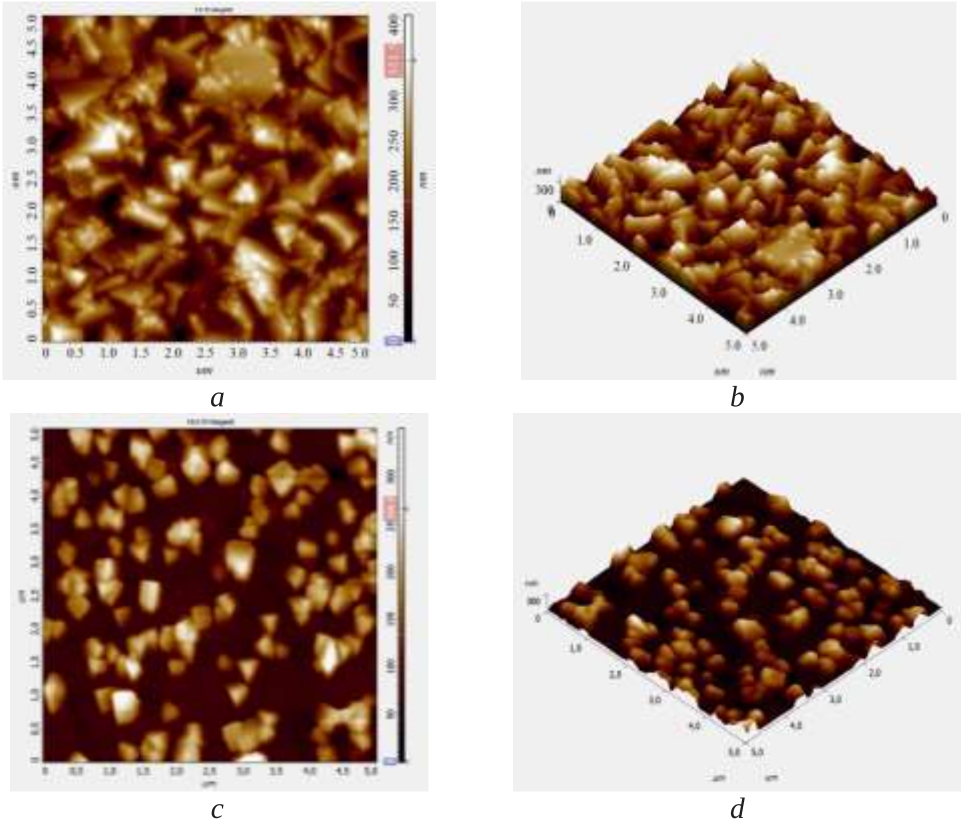

Figure 3 - AFM image of the surface of the film (*a*, *b*) and isolated particles (*c*, *d*) of lead sulfide (PbS)

Figure-3 shows the AFM images of PbS samples of 5x5 μm^2 scanning area. It can be seen from the figure that the results of the study are in good agreement with the results obtained by scanning electron microscope (SEM). Figure 3 (*a*, *b*) shows 2D and 3D images of the surface of lead sulfide film obtained at a temperature of 70°C mixture of lead nitrate and sodium hydroxide solutions. From the figure it can be seen that the film was formed and it has a rough surface of cubic crystallite shape with peak-to-peak value of ~ 300 nm.

AFM images of lead sulfide samples obtained at the temperature of the mixture of lead nitrate and sodium hydroxide solutions 100°C are presented in Figure 3 (*c*, *d*). The figure shows that lead sulfide is formed into separate isolated particles homogeneous in size and with a height similar to the thickness of the films, i.e. ~ 300 nm.

Conclusion

In this work, isolated submicron lead sulfide (PbS) particles were prepared using the chemical bath deposition method and it was determined that at a lower temperature (70°C) the growth of PbS film occurs and at a higher temperature (100°C) the formation of individual submicron particles.

Scanning electron microscopy (SEM) was used to study the morphology and elemental composition of lead sulfide samples. Atomic force microscope (AFM) allowed a detailed study of the surface structure of these samples. The analysis showed that the average size of isolated lead sulfide particles is about 144 nm. At the same time, all the particles have cubic shape and smooth surface.

Thus, isolated particles of PbS were obtained using temperature effects. On the next stage of the research it is planned to reduce the size of these particles, our research work will be devoted to the study of the influence of time on the synthesis of lead sulfide structures.

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