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CONTROL OF THE SIZES OF LEAD SULFIDE (PBS) NANOPARTICLES BY PLASMA TREATMENT METHOD

Abstract

In this work, the optimal parameters for the production of lead sulfide nanoparticles (PbS) were determined. Lead sulfide nanoparticles were obtained by chemical precipitation in an aqueous solution of lead nitrate ($Pb(NO_3)_2$) 25 ml 0.18 M (1.524 g), sodium hydroxide (NaOH) 75 ml 0.38 M (1.172 g), thiourea (CH_4N_2S) 50 ml 0.11 M (0.399 g), at a reaction temperature of 100 °C, the duration of the synthesis reaction was 120 minutes. The particles were deposited on a pre-purified silicon (Si) substrate. After synthesis, the particles were processed in a glow discharge plasma in an argon atmosphere at a pressure of the order of $Đ = 1$ Pa, for $t_1 = 1$ min and $U = 2$ min, at a voltage of $U = 2$ kV and a current strength of $I = 1,5$ mA. The morphology of the obtained structures was studied using a scanning electron microscope (SEM), the elemental composition of the particles was determined by energy dispersion analysis (EDX). Plasma treatment reduces the average particle size from the submicron to the nanometer range.

Key words: PbS particles, CBD method, plasma treatment, optimal parameters, Si substrate.

Introduction

Lead sulfide (PbS) is a semiconductor material that is one of the main materials in solar cells, photo detectors [1–5] and for selective coating for photo thermal conversion [6–9]. PbS is also used in devices such as LEDs, photoluminescence and infrared photo detectors [10] and in gas sensors [11]. This material is of particular interest, since its band gap (0.41 eV at 300 K) can increase with a decrease in its crystallite size and particle size become comparable to the exciton radius of Boron, this affects its physical properties and arouses the interest of scientific researchers, therefore they are intensively studied today [12].

PbS nanoparticles can be produced by various methods such as vacuum evaporation, pyrolysis by spraying, electro deposition, pulsed laser deposition, chemical vapor deposition (CVD), ultrasonic deposition and chemical bath deposition (CBD) [13].

In this work, the chemical bath deposition (CBD) method was used. This method is financially budgetary and very convenient, since it does not require a vacuum and the reaction takes place in an open space [14–18]. With the help of parameters (reaction time, temperatures and concentrations of reagents), the particle size can be varied and their properties can be influenced. If we compare, the physical method is energy-intensive and has a high cost of production [19]. Therefore, the (CBD) method is the most optimal method for the future, because it is more efficient, highly manageable and environmentally friendly [20].

Materials and methods

Lead sulfide (PbS) nanoparticles were obtained by chemical bath deposition (CBD). At the beginning of the experiment, the Si (100) substrate was washed with distilled water and the surface was cleaned with alcohol. The masses of the reagents were Pb $(NO_3)_2$ (lead nitrate) 25 ml 0.18 M (1.524 g), NaOH (sodium hydroxide) 75 ml 0.38 M (1.172 g), CH_4N_2S (thiourea) 50 ml 0.11 M (0.399 g), reaction temperature 100°C. Lead nitrate and sodium hydroxide were diluted in 75 ml of distilled water, with a turnover of 1200 rpm, using a magnetic stirrer at a temperature of 100 °C. And thiourea was diluted separately in 50 ml of distilled water with a turnover of 500 rpm, using an agitator at room temperature. Both reactions took place within 120 minutes. After that, the two solutions were mixed and after two minutes, when the color of the solution turned blue-black, at that moment the Si (100) silicon substrate was immersed in the solution for exactly 10 minutes, at room temperature. To maintain the uniformity of the solution, a magnetic stirrer was used. After receiving the PbS, the substrate surface was cleaned in an ultrasonic bath for 10 minutes.

Further, the resulting film was subjected to plasma treatment of a glow discharge in argon. The surface treatment was performed under the following parameters: pressure $Ð = 1$ Pa, voltage $I = 1{,}5$ kV, discharge current $I = 1{,}5$ mA and processing time $t_2 = 5$ min and $t_2 = 5$ min. The main parameters of the glow discharge (voltage, current, brightness) remain unchanged under constant external conditions. Figure 1 shows that the smoldering discharge is accompanied by a visible glow, the color of which depends on the type of gas.

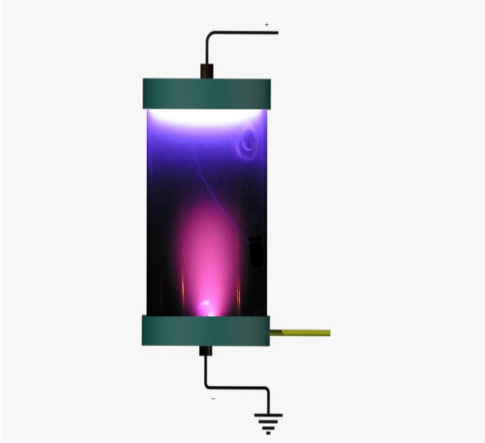


Figure 1 – Glow discharge

In the process of plasma generation, argon gas and direct current were used in the glow discharge, which is released from the power supply of the Aktakom APS-1915 High voltage DC Power supply. A constant current source provides stable voltage and current. A low pressure is created in the

vacuum chamber and the vacuum pump regulates the pressure in the chamber and the control system monitors and corrects the pressure using sensors and feedback. The vacuuming process takes place - the chamber is pumped out to a set pressure of 10^{-4} Pa, the pressure is not absolutely constant, but is usually maintained with high accuracy (for example, ± 5%). Argon is injected into the chamber until an operating pressure of 1 Pa is reached. A voltage of 2 kV is applied between the electrodes. During the collision of electrons with argon atoms, their ionization occurs and an avalanche of ionization leads to the formation of a quasi-neutral electron–ion gas - plasma. The camera material was made of stainless steel.

The choice of specific parameters for plasma surface treatment of lead sulfide on a silicon substrate is determined by a combination of factors aimed at achieving the desired effect. The pressure (1Pa) ensures a high degree of ionization of the working gas, which increases the number of active particles bombarding the surface. It helps to remove impurities and adsorbed molecules from the surface more effectively. Modifications to the surface of lead sulfide reduce the formation of byproducts associated with collisions of neutral particles. High voltage (2kV) prevents the destruction of lead sulfide by high-energy particles. Provides a controlled change in the chemical composition of the surface. Promotes the functionalization of the surface. A low current (1.5 mA) limits the thermal load on the sample, which reduces the risk of degradation. It promotes uniform surface treatment and increases the reproducibility of the process.

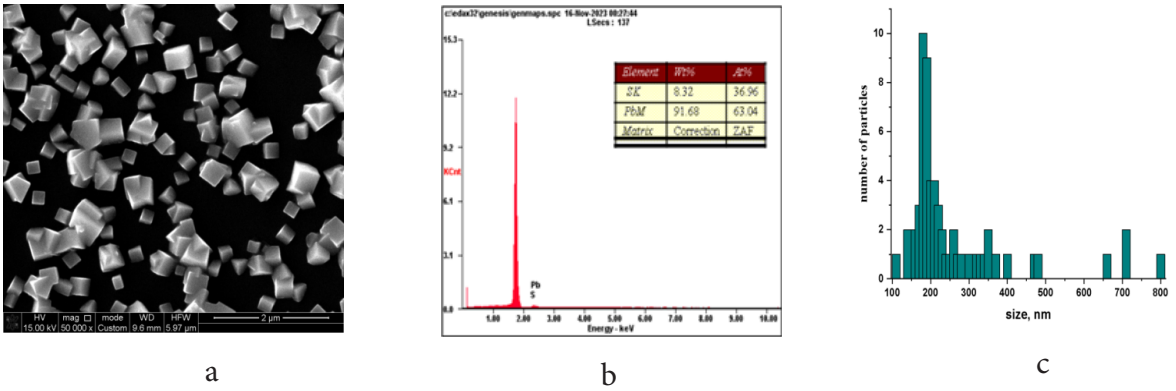
Quanta 200i 3D (FEI Company, USA) was used to measure the size of nanoparticles and study structural properties, and the energy dispersion spectroscopy (EDX) method was used to verify the stoichiometry of the obtained films.

Main provision

The treatment of cubic particles of lead sulfide with glow discharge plasma reduces the average particle size from the submicron to the nanometer range.

Results and discussion

The morphology and elemental composition of the particles were studied using a scanning electron microscope and energy dispersion analysis, respectively. The images were obtained at 50,000 times magnification. Figure 2 (a) shows that as a result of chemical deposition, individual cubic particles with an average size of 320 ± 70.1 nm are formed. It is worth noting that the reaction temperature and synthesis time affect their uniformity. Figure 2(b) is an energy dispersion analysis (EDS) of these particles. Figure 2 (c) shows the particle size distribution.



a b c

Figure 2 – SEM image, energy dispersion analysis and distribution of PbS particles obtained by the CBD method.

Further, these cubic particles were subjected to plasma treatment. Figure 3 (a) and 3 (d) show images treated with plasma for $t_1 = 1$ minutes and $t_2 = 5$ minutes. The average particle size during treatment with duration of $t_2 = 5$ min is 178 ± 44.7 nm. The reduction of the average size occurs linearly, which is very important for controlling the size of cubic particles.

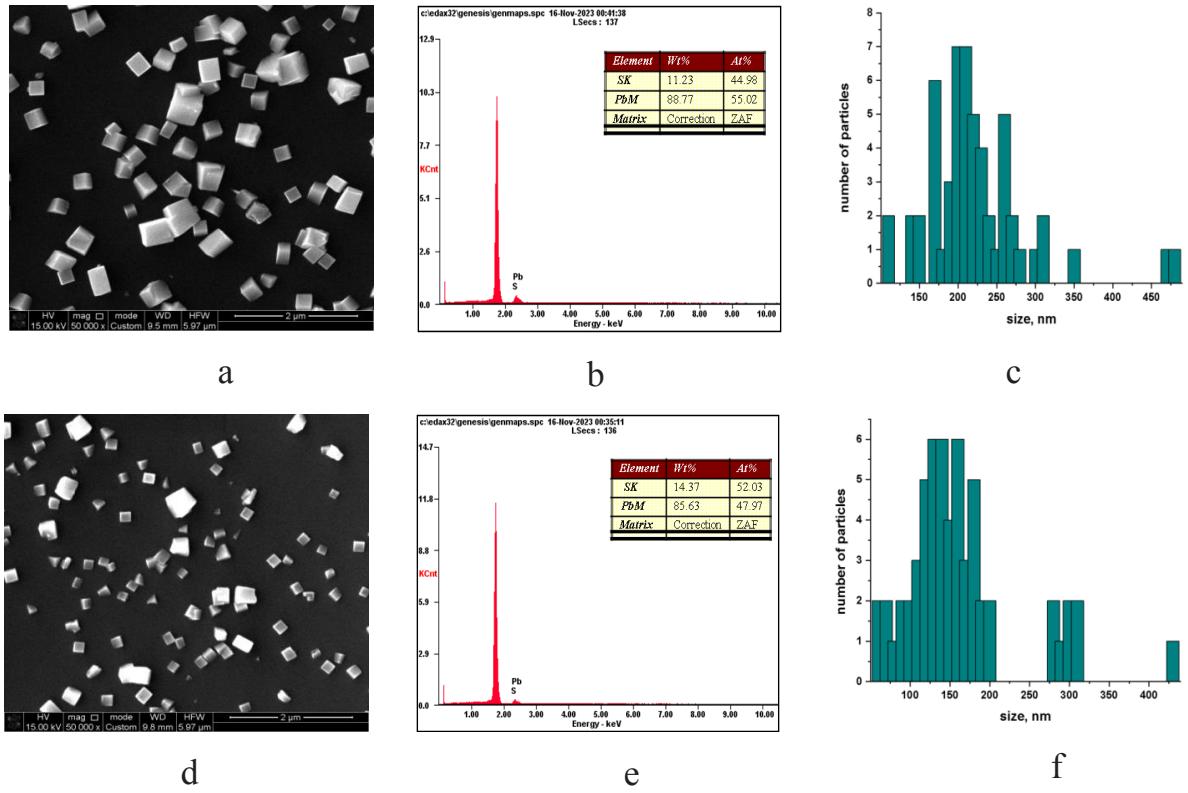


Figure 3 – SEM images, energy dispersion analysis and distribution of PbS particles obtained using CBD and plasma processing

Figure 3 (a) demonstrates PbS particles obtained by chemical method for 10 minutes and subjected to plasma treatment for 1 minute. Figure 3 (b) is an energy dispersion analysis (EDS) of these particles. Figure 3 (d) shows PbS particles obtained by chemical method for 10 minutes and subjected to 5-minute plasma treatment. Figure 3 (e) – EDS analysis of these particles. The elemental composition of PbS nanostructures was investigated using EDS. Energy dispersive spectroscopy of the samples revealed peaks of Si, Pb and S, which confirms the presence of these elements in the nanostructure. Figure 3 (c, f) shows the separation of particles by size, treated with plasma for one and five minutes, respectively. As can be seen from these figures, the smallest cubic particle size in 1 minute treatment was 110 nm, and in 5 minute treatment, the smallest cubic particle size decreased by 60 nm.

Lead sulfide crystallites have a cubic shape due to the peculiarities of the crystal structure of this substance. Lead sulfide crystallizes in the cubic symmetry that its unit cell has the shape of a cube. In this cell, lead and sulfur atoms are arranged in strictly defined positions, creating a repeating three-dimensional structure. Defects in the crystal lattice: may lead to the formation of imperfect cubic shapes.

Conclusion

In this paper, the effect of plasma treatment on the size of cubic particles of lead sulfide (PbS) obtained by chemical method was studied. It was found that plasma treatment for five minutes leads to a significant reduction in the particle size of PbS. As a result of plasma exposure to cubic particles of lead sulfide, a change is observed.

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ПЛАЗМАЛЫҚ ӨҢДЕУ ӘДІСІ БОЙЫНША ҚОРҒАСЫН СУЛЬФИДІ (PBS) НАНОБӨЛШЕКТЕРІНІҢ ӨЛШЕМДЕРІН БАСҚАРУ

Аңдатпа

Бұл жұмыста қорғасын сульфидінің (PbS) нанобөлшектерін қалыптастырудың оңтайлы параметрлері анықталды. Қорғасын сульфидінің нанобөлшектері қорғасын нитратының (Pb (NO3)2) 25 мл 0,18 М (1,520 г), натрий гидроксиді (NaOH) 75 мл 0,38 М (1,150 г), тиомочевина (CH4N2S) 50 мл 0,11 М (0,396 г) сулы ерітіндісінде, реакция температурасы 100°С, синтез реакциясының ұзақтығы 120 минут шамасында химиялық тұндыру арқылы алынды. Бөлшектер алдын ала тазартылған кремний (Si) төсенішіне қойылды. Синтезден кейін бөлшектер $I = 1,5$ кВ кернеуде және $I = 1,5$ мА тоқта, $t_2 = 5$ мин және $t_2 = 5$ мин уақыт шамасында, $t_1 = 1$ Па қысымда аргон атмосферасында солғын разряд плазмасында өңделді. Алынған құрылымдардың морфологиясы сканерлеуші электронды микроскоптың (SEM) көмегімен зерттелді, ал бөлшектердің элементтік құрамы энергодисперсиялық талдау (EDX) арқылы анықталды. Плазмамен өңдеу бөлшектердің орташа өлшемін субмикроннан нанометрлік диапазонға дейін кішірейтеді.

Тірек сөздер: PbS бөлшектері, CBD әдісі, плазмалық өңдеу, оңтайлы параметрлер, Si төсеніші.

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КОНТРОЛЬ РАЗМЕРОВ НАНОЧАСТИЦ СУЛЬФИДА СВИНЦА (PBS) МЕТОДОМ ПЛАЗМЕННОЙ ОБРАБОТКИ

Аннотация

В этой работе были определены оптимальные параметры получения наночастиц сульфида свинца (PbS). Наночастицы сульфида свинца были получены методом химического осаждения в водном растворе нитрата свинца (Pb $(NO_3)_2$) 25 мл 0,18 М (1,524 г), гидроксида натрия (NaOH) 75 мл 0,38 М (1,172 г), тиомочевины (CH_4N_2S) 50 мл 0,11 М (0,399 г), при температуре реакции 100 °С, длительность реакции синтеза составляла 120 минут. Частицы осаждались на предварительно очищенной кремниевой (Si) подложке. После синтеза частицы были обработаны в плазме тлеющего разряда в атмосфере аргона при давлении порядка $t_1 = 1$ Па, в течение $t_1 = 1$ мин и $I = 1{,}5$ мин., при напряжении $I = 1{,}5$ кВ и силе тока $I = 1{,}5$ мА. Морфология полученных структур была исследована с помощью сканирующего электронного микроскопа (СЭМ), элементный состав частиц определялся методом энергодисперсионного анализа (EDX). Обработка плазмой приводит к уменьшению среднего размера частиц от субмикронного до нанометрового диапазона.

Ключевые слова: PbS частицы. CBD метод, плазменная обработка, оптимальные параметры, подложка Si.