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The effects of the precursors $K_2Cr_2O_7$ and NH_4I on the composition, morphology and dark resistance of photosensitive elements based on PbS

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Abstract. The results of structural, morphological, and chemical compositional studies of photosensitive elements based on lead sulfide (PbS) thin films are presented. The films were obtained by chemical bath deposition in the presence of one (potassium dichromate — $K_2Cr_2O_7$) or two (ammonium iodide — NH_4I together with $K_2Cr_2O_7$) precursors. The data obtained from scanning electron and atomic force microscopy show that the surface of samples deposited in the presence of only $K_2Cr_2O_7$ consists of crystallites ranging in size from 300 to 900 nm, with a cubic facet. Adding NH_4I to the reaction bath results in a reduction in grain size (more than 75% of the surface consists of crystallites between 100 and 325 nm) and in the appearance of two percent of nanoscale structures (up to 100 nm). At the same time, the surface morphology becomes smoother. It has been established that iodine and its compounds lead to a significant increase in dark resistance (by 10...250 times), which in turn ensures an increase in film sensitivity.

Keywords: lead sulfide, thin films, morphology, chemical deposition method, photosensitive elements

Introduction

Lead sulfide (PbS) is a direct-gap semiconductor with a band gap of 0.4 eV at 300 K and an absorption coefficient of $\alpha \sim 10^5$ cm^{-1} (Ravich et al. 1968). The lifetime of charge carriers in bulk crystals was measured by Moss (Moss 1953), who obtained values from $6 \cdot 10^{-4}$ to 6 μs and attributed these differences to the influence of Auger recombination. According to the Pb–S phase diagram, lead sulfide has an excess of lead atoms relative to sulfur (Naşcu et al. 1996) and is therefore an *n*-type semiconductor.

In order to increase the lifetime of at least one type of charge carriers that determines the main parameters of photosensitive elements (PSEs), technologies for producing thin polycrystalline PbS films have been developing since the 1960s. These films are now used to manufacture PSEs for various optoelectronic systems operating in the IR spectral region from 1 to 3 μm (Butkevich et al. 1999; Baryshev et al. 2000; Pentia et al. 2004; Sadovnikov et al. 2013), sensors for various gases and highly selective sensors for detecting toxic compounds in the air (Burungale et al. 2016; Beatriceveena et al. 2018; Chen et al. 2021), solar cells (Huo et al. 2019, Mengting et al. 2019) and obtain quantum dots (Al-Ahmed et al. 2022; Babaev 2023; Popov et al. 2023; Ren et al. 2017; Shuklov et al. 2020).

Photosensitive polycrystalline films are deposited or sputtered using various methods, which can be divided into 'physical' (thermal evaporation) (Kumar et al. 2003, Patel et al. 2017; Rosario et al. 2019; Singh et al. 2015)) and 'chemical' (spray pyrolysis (Rajashree et al. 2014), SILAR (Successive Ionic Layer Adsorption and Reaction) method (Gülen 2014) and chemical deposition (Ahmed et al. 2020; Markov et al. 2006; Maskaeva et al. 2020; Maskaeva et al. 2019; Palomino-Merino et al. 2013; Touati et al. 2017)).

The chemical bath deposition (CBD) method has a number of advantages: simple equipment is used; films can be deposited on various substrates (Makaruk et al. 2025); and the introduction of precursors during the synthesis process allows for variation in the photosensitive properties of the elements (sensitivity and spectral characteristics). However, none of the numerous theories explaining the behavior of impurities in $A^{IV}B^{VI}$ compounds can predict which element or precursor added to the reaction bath will improve the basic parameters or lead to the development of unusual properties (Maskaeva et al. 2021).

The key technological step in producing highly sensitive structures is sensitization, which involves introducing acceptor-type traps that localize electrons, thereby increasing the hole lifetime. Oxygen, an isoelectronic impurity, is most often used, causing deformation of the PbS crystal lattice and the creation of localized electron trapping centers. The introduction of oxygen, in addition to structural disorder, is accompanied by a violation of the stoichiometric composition, the appearance of a large number of components and phases containing oxygen: PbO, $PbSO_4$, $PbO \cdot PbSO_4$, etc. (Jiang et al. 2021; Quanjiang et al. 2023).

It should be noted that introducing oxygen from a solution during hydrochemical precipitation does not allow for control over the concentration of the introduced impurity. For this reason, other methods for sensitizing the material are currently explored and developed. Common methods for increasing the lifetime of charge carriers include annealing samples in the air (Miroshnikov et al. 2014; Miroshnikov et al. 2015; Mohamed et al. 2014) or two-step annealing: first in an oxygen-containing environment and then in nitrogen (Fan et al. 2023).

To obtain elements with high sensitivity after their synthesis, oxidizing agents are added to the reaction bath beside the main components, which promote inclusion of oxygen atoms in the crystal lattice: H_2O_2, $K_2S_2O_8$ (Blount et al. 1973; Ghamsari et al. 2006; Kul 2019; Naşcu et al. 1996; Wolten 1975), or activators in the form of salts of various metals (silver, mercury, copper, calcium, cadmium, iron (II), gallium, magnesium) (Maskaeva et al. 2017; Simic et al. 1968; Venkoba Rao et al. 1963).

Halogen-containing compounds (for example, with iodine) are able to control the penetration of oxygen into the microcrystallites of the thin-film structure or promote the inclusion of oxygen into the lattice during chemical deposition. In addition, iodine-containing compounds can act as a catalyst, accelerating the formation of oxide phases in the film and ensuring high sensitivity of the PSE due to the penetration of oxygen deep into the grain, which leads to a change in the type of conductivity (Markov et al. 2000; Suh et al. 2016; Maskaeva et al. 2025). As follows from the above studies, doping PbS with iodine changes the photovoltaic parameters due to the formation of point defects in the form of PbI_2. This leads to an inversion of the conductivity type ($n \rightarrow p$) with optimization of the charge carrier concentration in the semiconductor layer.

Sensitization of PbS films with both oxygen and iodine is accompanied by a change in the conductivity type (from *n*-type to *p*-type) and an increase in the concentration of quasi-free holes (Espevik et al.

1971), while the hole lifetime, dark conductivity, and the sensitivity of the photosensitive element also increase.

The chromium (Cr) contained in the precursor (potassium dichromate — $K_2\,Cr_2\,O_7$) is a transition metal that can also influence the parameters of PbS-based photosensitive elements. It was found (Maskaeva et al. 2021) that chromium can use not only the electrons of the outer shell to form chemical bonds, but also d-electrons, which allow changing the concentration of electrons in d-states and having a variable valence (II, III, IV). Information on the photoconductivity of thin-film PbS:Cr cell is presented in the work (Huo et al. 2019), which found that the introduction of chromium leads to an increase in its photoconductivity.

Another study (Maskaeva et al. 2021) showed that the introduction of NH_4I and $CrCl_3$ precursors into the reaction solution preserves the cubic B1 structure of lead sulfide and leads to an increase in the gap width (E_g) by 0.16–0.20 eV, a decrease in the dark resistance R_T and an increase in the voltage sensitivity (U_s). The dependences of E_g and U_s on the chromium salt concentration in the reaction bath exhibit an extreme character with a maximum at 0.016 mol/l, which is due to the non-monotonic incorporation of chromium into the PbS lattice. The results of studies of the current-voltage characteristics of PbS(I) and PbS(I, Cr) thin-film layers are in good agreement with the results of structural, optical, and photosensitive properties.

This work seeks to investigate and compare the morphology, composition, and dark resistance of chemically deposited lead sulfide films obtained in a solution with precursors: potassium dichromate — $K_2\,Cr_2\,O_7$ and ammonium iodide — NH_4I.

Materials and methods

Thin lead sulfide films were synthesized by chemical precipitation from an ammonia-citrate reaction bath containing lead acetate $Pb(CH_3COO)_2$, sodium citrate $Na_3C_6H_5O_7$, ammonium hydroxide NH_4OH, and thiourea N_2H_4CS (source of sulfide ions), with varying concentrations of the oxidizing agent potassium dichromate ($K_2Cr_2O_7$) in a molar content from 10^{-5} mol/l to 10^{-4} mol/l and ammonium iodide (NH_4I) in a molar content of 0.2 mol/l. Film deposition was carried out for 90 min at a temperature of 353 K in sealed molybdenum glass reactors into which degreased glass slide substrates (72.2% SiO_2, 14.3% Na_2O; 1.2% K_2O, 6.4% CaO, 4.3% MgO, 1.2% Al_2O_3, 0.03% Fe_2O_3, 0.3% SO_3) fixed in fluoroplastic fixtures were immersed. The reactors were installed in a TS-TB-10 thermostat with a temperature control accuracy of ±0.1 K. All films were deposited on pre-degreased glass substrates. After synthesis, the samples were rinsed with distilled water and air-dried. The film thickness did not exceed 400 nm. The contacts were formed by electron beam evaporation of a nickel target with a preliminary stage of ion cleaning of the surface from the oxide layer and contaminants.

Film composition was analyzed at five different points on the surface (with subsequent averaging) using X-ray microanalysis on a Vega II SBU scanning electron microscope (Tescan, Czech Republic) equipped with an Inca X-act energy-dispersive spectrometer (Oxford Instruments, UK). The accelerating voltage was 10 kV; the detector acceptance angle, 15°; the working distance, 15 mm; and the data acquisition duration, 60 s.

The morphology of the film samples was studied using scanning electron microscopy (Vega II SBU, Tescan) and atomic force microscopy (AFM) using an INTEGRA Prima NanoLaboratory probe (NT-MDT, Zelenograd). The images were taken using a semicontact scanning technique with an NSG01 cantilever (rigidity 1.45–15.10 N/m). Crystallite size analysis was performed using Gwyddion v. 2.67 software.

The electrophysical parameters of the elements, their current-voltage characteristics, and their contact quality were studied using the ASEC-03E automated measurement system (SKB IRE RAS, Fryazino). The dark resistance of the elements was calculated from the experimental current-voltage characteristics.

Results and discussion

The study of the composition of lead sulfide film samples obtained by chemical deposition from a reaction bath containing the oxidizer $K_2Cr_2O_7$ at concentrations of 10^{-5} mol/l and 10^{-4} mol/l and NH_4I at a concentration of 0.2 mol/l for the main elements (lead, sulfur, and iodine) was carried out using the method of X-ray spectral microanalysis. The atomic oxygen content of the samples was not included in the analysis because the method under consideration is unable to separate the oxygen incorporated into the lead sulfide lattice from the oxygen contained in the glass substrate.

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The results of measurements averaged over five points for samples obtained only in the presence of potassium dichromate (samples 2 and 4) and for those obtained in the presence of potassium dichromate at concentrations of 10^{-5} mol/l and 10^{-4} mol/l and ammonium iodide (5 and 7) are given in Table 1.

The table demonstrates that all the film samples obtained with an oxidizer have the sulfur content of 51.77–52.22 at.% and the lead content of 47.78–48.23 at.%, at a rough ratio of 1.08. When ammonium iodide is added to the reaction bath, the sulfur content ranges from 51.04 to 51.71 at.% and lead, from 46.77 to 47.42 at.%. The atomic content of iodine changes insignificantly with variations in the oxidizer concentration in the reaction bath. The sulfur to lead ratio is 1.09. According to data available in literature (Suh et al. 2016), lead iodide (PbI_2) and oxide phases PbI_2O_2, Pb_2O_4 are formed in samples deposited with the NH_4I precursor. A slight decrease in the atomic sulfur content in the samples prepared with the additional ammonium iodide precursor, relative to samples obtained using only the oxidizer (potassium dichromate), may indicate the replacement of atoms of the main substance with iodine and/or oxygen.

The stoichiometry between the metal and the chalcogen is not observed in all cases, which indicates the *p*-type conductivity of the considered groups of samples as a result of a lack of lead.

Table 1. Elemental composition and dark resistance of lead sulfide films obtained using different concentrations and combinations of precursors

No.	Composition of the reaction bath, mol/l	[Pb], at. %	[S], at. %	[I], at. %	Dark resistance (R_T, kOhm
1	$K_2Cr_2O_7 = 10^{-5}$	48.23 ± 0.53	51.77 ± 0.53	–	3.3
2	$K_2Cr_2O_7 = 10^{-4}$	47.78 ± 0.47	52.22 ± 0.47	–	15
3	$K_2Cr_2O_7 = 10^{-5}$ $NH_4I = 0.2$	47.42 ± 0.46	51.04 ± 0.21	1.54 ± 0.27	820
4	$K_2Cr_2O_7 = 10^{-4}$ $NH_4I = 0.2$	46.77 ± 0.52	51.71 ± 0.85	1.52 ± 0.43	216

The efficiency of photosensitive elements depends largely on the reflectivity of the incident radiation. Reflectivity, in turn, is determined by the surface morphology of the samples (grain size and shape). Scanning electron microscopy was used to study the effect of the reaction bath composition used for PSE deposition on their surface morphology. Typical images of the PSE surface for the studied sample groups at various magnifications are shown in Figs. 1 and 2. As suggested by the figures, the surface of the samples obtained in the presence of potassium dichromate (Fig. 1) consists of crystallites with a pronounced cubic faceting, characteristic of lead sulfide, which has a face-centered cubic structure of the NaCl (rock salt) type with the space group $Fm\bar{3}m$ (Seghaier et al. 2006). Adding ammonium iodide to the reaction bath smooths the crystallite facets, but individual globules up to one micrometer in size form (Fig. 2). Maskaeva et al. (2021) noted that a similar surface appearance was also observed for chromium-containing samples.

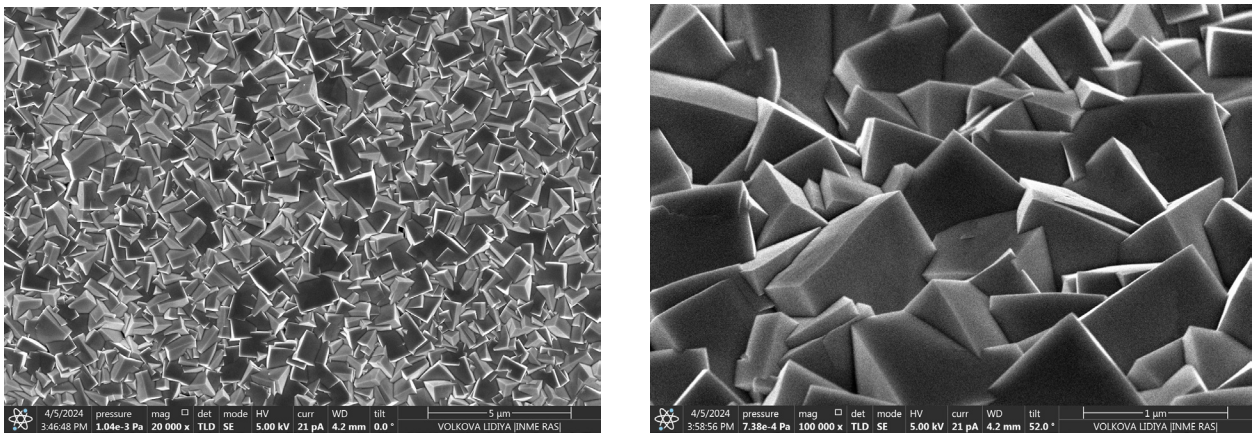

Fig. 1. Electron microscopic images of the surface of a typical sample obtained in a solution with $K_2Cr_2O_7$

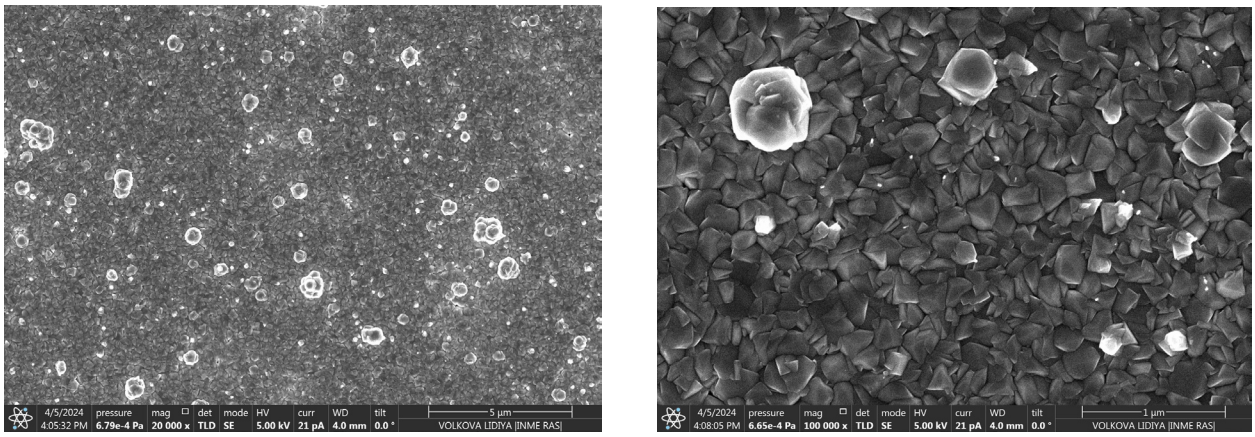

Fig. 2. Electron microscopic images of the surface of a typical sample obtained in a solution with $K_2Cr_2O_7$ and NH_4I

Electron microscopy studies were supplemented by atomic force microscopy (AFM) measurements. The resulting AFM images confirmed the surface morphology data obtained using scanning electron microscopy for two groups of samples (Fig. 3).

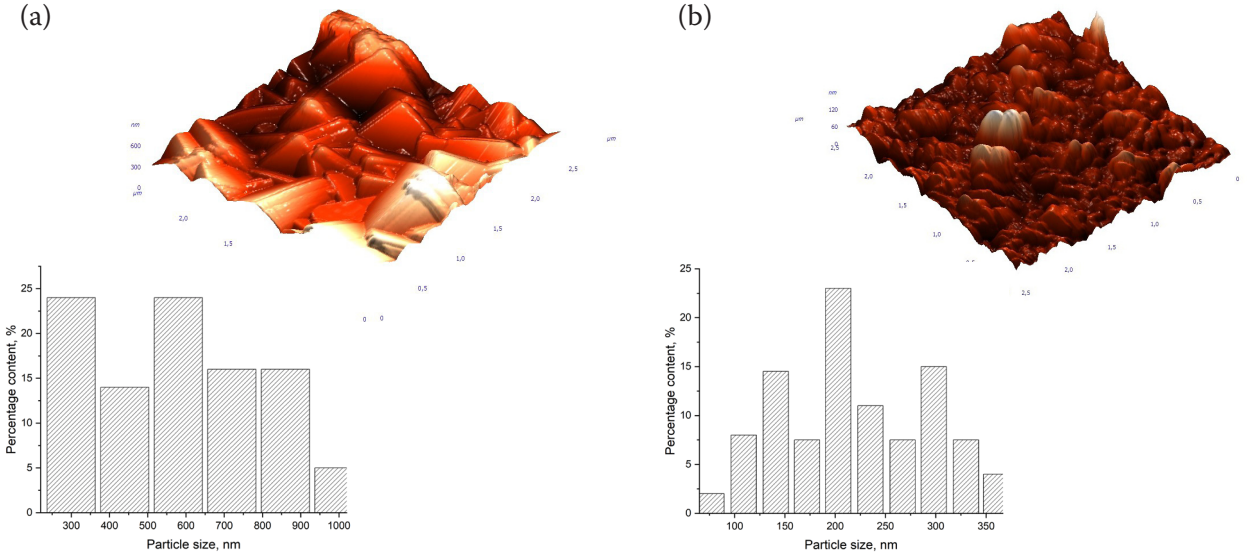

Fig. 3. AFM images of the surface of photosensitive elements based on PbS films chemically deposited in the presence of $K_2Cr_2O_7$ (a) and $K_2Cr_2O_7$ with NH_4I (b). The insets show histograms of the grain size distribution on scans of 5×5 µm^2

The histogram in Fig. 3a was calculated based on the faces of cubic grains. The grain size distribution shows that the samples synthesized in the presence of only the oxidizer are quite homogeneous, as evidenced by the uniform distribution of the percentage of grains ranging in size from 300 to 900 nm. Adding NH_4I to the reaction bath results in a decrease in particle size (histogram in Fig. 3b). Over most of the surface, crystallites range in size from 100 to 325 nm, with approximately two percent of particles smaller than 100 nm present. The presence of nanoscale crystallites can influence the band gap width of the material and, consequently, the spectral dependence of photosensitivity. The obtained data are consistent with the results presented in a study (Markov et al. 2022) in which PbS film samples were deposited in a reaction bath in the presence of ammonium iodide alone. The authors demonstrated that the PSE exhibited a broadening of the band gap and a shift in sensitivity toward higher energies (shorter wavelengths).

Table 2 shows the results of calculating the microrelief parameters of the studied groups of samples, obtained as a result of processing their AFM images.

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Table 2. Microrelief parameters of the studied groups of samples

Microrelief parameter	Sample number			
	1	2	3	4
Roughness average (R_a), nm	35.63	40.33	12.81	12.17
Root mean square roughness, (R_q), nm	45.51	53.02	18.76	17.65
Maximum average profile height (P_z), nm	228.20	248.50	97.02	116.10

A comparison of the microrelief parameters (roughness (R_a and R_q) and maximum average profile height (P_Z)) can indicate that samples 1 and 2, deposited in the presence of only an oxidizer, have a height difference 2.8–3.3 times greater than samples 3 and 4 with ammonium iodide, which suggests a smoother surface of the latter despite the presence of globules.

Changes in the type and concentration of precursors affect not only the chemical composition and morphology of the resulting samples, but also their electrophysical properties. Table 1 thus shows that elevating the oxidizer (potassium dichromate) concentration in the reaction bath from 10^{-5} mol/L to 10^{-4} mol/L increases the dark resistance of the samples by a factor of 4.5 (from 3.3 to 15 kOhm). The addition of ammonium iodide increases dark resistance by orders of magnitude. Augmenting the oxidizer concentration in the presence of ammonium iodide, however, decreases dark resistance from 820 to 216 kOhm.

The rise in resistance with increasing oxidizer concentration can be explained by the incorporation of more oxygen atoms into the lead sulfide crystal lattice, as well as the possible formation of the oxygen-containing thin layers observed before (Mohamed et al. 2014). Our previous works showed that the presence of ammonium iodide during the deposition of lead sulfide films leads to a decrease in the dark resistance (R_T) compared to samples obtained with the participation of sodium dithionite — $Na_2S_2O_4$ or sodium sulfite — Na_2SO_3 oxidizers. However, this combination demonstrates an increase in dark resistance by 10...250 times at the same concentrations of $K_2Cr_2O_7$. It should be noted that high R_T (low dark conductivity σ_0) contributes to an increase in the sensitivity of the PSE; the sensitivity criterion is not the increase in conductivity (photoconductivity — $\Delta\sigma$), but $\Delta\sigma/(\sigma_0\,\Phi)$, where Φ is the effective value of the radiant power. A decrease in dark conductivity leads to an increase in the sensitivity of the PSE.

Previous results of a study of lead sulfide films deposited in the presence of ammonium iodide using Auger spectroscopy and high-resolution transmission microscopy showed that iodine and oxygen atoms are concentrated to a greater extent near the PSE substrate. Consequently, iodine-containing compounds are formed at the initial stage of film deposition simultaneously with the formation of lead hydroxide crystallization centers — $Pb(OH)_2$. Due to this, the introduction of the ammonium iodide precursor into the reaction bath leads to the formation of a *p*-type conductivity layer near the substrate. As a result, a *p-n* junction is formed, the depth of which depends on the thickness of the iodine-enriched lower layer. This leads to a decrease in the effective cross-section of the *n*-type material and increases the dark resistance of the elements, which explains the significant change in the resistance of the photosensitive element described above.

Conclusion

Polycrystalline films of lead sulfide obtained in solutions with one (potassium dichromate) or two (potassium dichromate and ammonium iodide) precursors were studied.

We showed that the introduction of chromium salt into the reaction mixture neither changes the cubic crystalline structure of lead sulfide nor has a significant effect on the morphology of the films: the crystallite sizes are from 300 to 900 nm and are uniformly distributed over the entire surface of the PSE.

The simultaneous use of potassium dichromate and ammonium iodide precursors leads to a change in the crystallite faceting to a smoother one, a decrease in the particle size (sizes from 100 to 325 nm in the presence of about two percent of particles smaller than 100 nm) and the formation of segregate globules up to one μm in size.

It was established that iodine and its compounds formed at the early stage of thin film deposition lead to a significant increase in the dark resistance of photosensitive elements, which in turn contributes to an increase in their photosensitivity.

The obtained results indicate that the combined use of the precursors $K_2Cr_2O_7$ and NH_4I during the deposition of lead sulfide layers is an effective method for improving the technology of producing photosensitive elements for photoelectronic devices. Further studies of lead sulfide films obtained in a solution containing potassium dichromate will focus on investigating their magnetic properties, as chromium is a d-element, important for the development of spintronics.

Conflict of Interest

The authors declare that there is no conflict of interest, either existing or potential.

Author Contributions

The authors have made an equal contribution to the preparation of the paper.

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