Chalcogenide Letters Vol. 22, No. 10, October 2025, p. 883 - 888

Thermogravimetric analysis of the PbS, PbSe and $PbS_{0.5}Se_{0.5}$ powders obtained by chemical precipitation

S. N. Yasinova [a,b,*], A. B. Rzayeva [a,b], M. H. Huseynaliyev [b], B. T. Hagieva [a],
G. M. Huseynov [a,b]
[a] *Nakhchivan State University Ministry of Science and Education of the Republic of Azerbaijan, AZ7012, Nakhchivan, Azerbaijan*
[b] *The Ministry of Science and Education of Azerbaijan, Institute of Natural Resources, Nakhchivan State University, H. Aliyev ave. 35, AZ7000, Nakhchivan, Baku, Azerbaijan*

The thin films and powders of PbS, PbSe, and $PbS_{0.5}Se_{0.5}$ have been obtained by chemical precipitation method. The X-ray spectra of the obtained thin layers were obtained through the"Miniflex 600" diffractometer, and elemental analyses were performed. Thermogravimetric analyses of the powders of all three compounds were conducted in the temperature range of 20-800°C using the "NETZSCH STA 449 F3" apparatus. It has been determined from the X-ray spectra that the angular positions of all the peaks of PbS, PbSe, and $PbS_{0.5}Se_{0.5}$ thin layers coincide with the positions of the corresponding diffraction peaks obtained under normal conditions. The results of the thermogravimetric analysis measurements and calculations have shown that the compounds are consistent with the formulas of PbS, PbSe, and $PbS_{0.5}Se_{0.5}$ powders.

(Received June 23, 2025; Accepted October 8, 2025)

Keywords: Thin film, Chemical bath, X-Ray, Thermogravimetric analysis (TGA), Lead chalcogenides

1. Introduction

Lead chalcogenides (PbX, where X denotes S, Se, or Te) are materials of increasing research interest due to their fascinating physical characteristics and diverse application spectrum. Thin films of these compounds are particularly valuable for manufacturing a range of devices, including infrared detectors, gas sensors, photodiodes, solar cells, and thermoelectric modules [1]. A notable feature is their relatively large exciton Bohr radius, approximately 8 nm, which enables the observation of significant quantum confinement effects in their nanoscale structures [2].

Semiconductors belonging to the A^4B^6 group within the IV–VI family serve as crucial components in infrared optical systems. Their importance stems from the multifunctional capabilities they impart to various device applications. Specifically, these materials demonstrate two-photon absorption phenomena in both the near- and mid-infrared spectral regions, enhancing their utility in optoelectronic devices [3].

The methods for obtaining thin films are diverse: electroplating [7, 4], chemical deposition [5], electrochemical atomic layer epitaxy [8], molecular beam epitaxy [6] and so on. One of these methods is the chemical deposition method from various solutions. Among the listed methods chemical deposition stands out for its efficiency.

For instance, the preparation of PbS thin films can be achieved by blending equal volumes of several solutions: 0.07 M lead acetate, 0.3 M sodium hydroxide, 0.06 M triethanolamine, and 0.17 M thiourea, with the process conducted at 40°C [15]. Also, by taking the same amount of these solutions at room temperature, PbS thin film and powder were obtained at room temperature [9]. Similarly, PbSe films are fabricated using a bath maintained at 60°C, combining lead acetate (0.07 M), sodium hydroxide (0.3 M), triethanolamine (0.06 M), and sodium selenosulfate (0.17 M) [15]. The sodium selenosulfate solution itself is prepared by boiling 0.425 g of selenium and 1.245 g of sodium sulfide in 100 ml of distilled water for seven hours at 90°C [10].

---

[*] Corresponding author: sara.novruzova@yahoo.com

https://doi.org/10.15251/CL.2025.2210.883

2. Experimental details

The deposition procedure involved performing the process in a 60 ml beaker at 60°C, with a glass slide positioned vertically within the solution. Continuous agitation of the solution was maintained using a magnetic stirrer. After a duration of 20 minutes, the glass slide was removed, subsequently rinsed with distilled water, and allowed to air-dry.

For $PbS_{0.5}Se_{0.5}$, the deposition was carried out at 50°C using lead acetate (0.07 M), sodium hydroxide (0.3 M), triethanolamine (0.06 M), sodium selenosulfate (0.17 M), and thiourea (0.17 M). Equal volumes of the first three reagents (12 ml each) were combined, and 6 ml each of the latter two were added [15]. The experiment is performed by mixing equal volumes (12 ml each) of lead acetate, sodium hydroxide, and triethanolamine solutions. Meanwhile, sodium selenosulfate and thiourea are introduced in equal proportions—6 ml each. The test tube is positioned upright inside a 60 ml lab beaker. Following stirring for 20 minutes using a magnetic stirrer, a $PbS_{0.5}Se_{0.5}$ thin film with good adhesivity is obtained on the side of the beaker and on the microscope slide [15]. At the bottom of the beaker, $PbS_{0.5}Se_{0.5}$ powder is obtained as a precipitate, and a $PbS_{0.5}Se_{0.5}$ thin film forms on the glass [15].

As a result of all three reactions, thin films of PbS, PbSe and $PbS_{0.5}Se_{0.5}$ with approximate thicknesses in the range of 200-250 nm were obtained, along with PbS, PbSe and $PbS_{0.5}Se_{0.5}$ powders as precipitates at the bottom of the laboratory beaker.

3. Results and discussions

The X-ray spectra of the PbS, PbSe and $PbS_{0.5}Se_{0.5}$ thin films were recorded using the Miniflex 600 diffractometer, and elemental analyses were performed. Thermogravimetric analyses of the powders of all three compounds were conducted in the temperature range of 20-800°C using the "NETZSCH STA 449 F3" apparatus.

The chemical composition of PbS, PbSe and $PbS_{0.5}Se_{0.5}$ powders

Powder	Pb	S	Se
PbS	40.81%	59.19 %	-
PbSe	68.7%	-	31.3%
$PbS_{0.5}Se_{0,5}$	44.13%	33.14%	22.73%

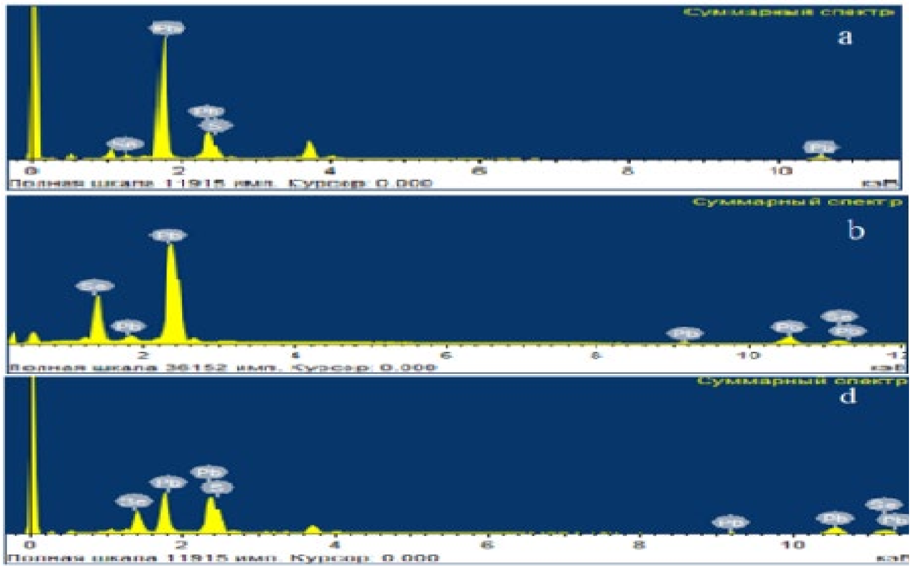

Fig. 1. Energy-dispersive spectra of thin films of PbS (a), PbSe (b) and PbSSe (d) obtained by chemical precipitation method.

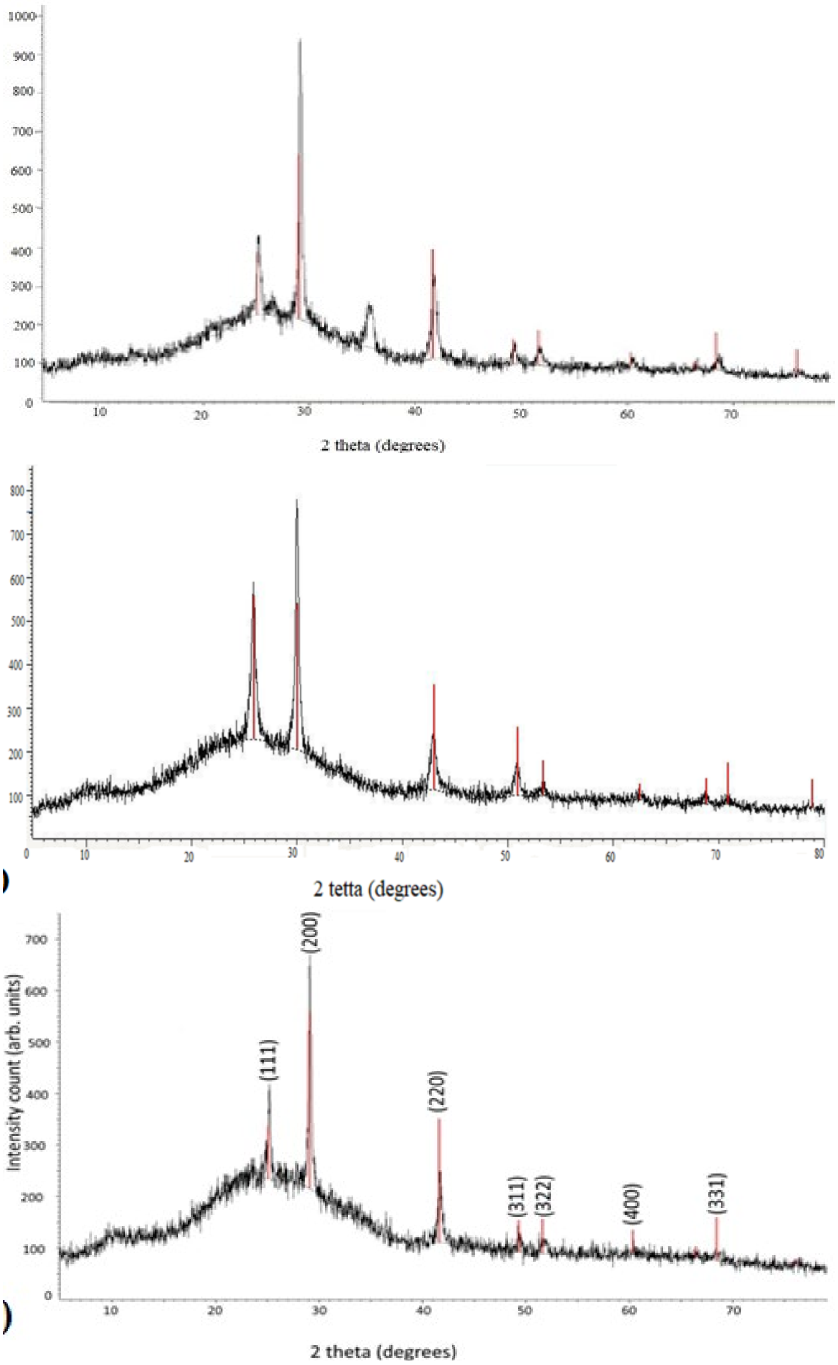

Fig. 2. X-ray diffraction scattering curves of thin films of PbSe (a), PbS (b) and $PbS_{0.5}Se_{0.5}$ (c) obtained by chemical precipitation method.

As seen from the spectra all the peak positions of the thin films of PbS, PbSe, and $PbS_{0.5}Se_{0.5}$ obtained by the chemical precipitation method coincide with the positions of the corresponding diffraction peaks in the standard X-ray diffraction patterns.

Thermogravimetric analyses of PbS, PbSe and $PbS_{0.5}Se_{0.5}$ powders obtained by the chemical precipitation method were performed and the results are presented.

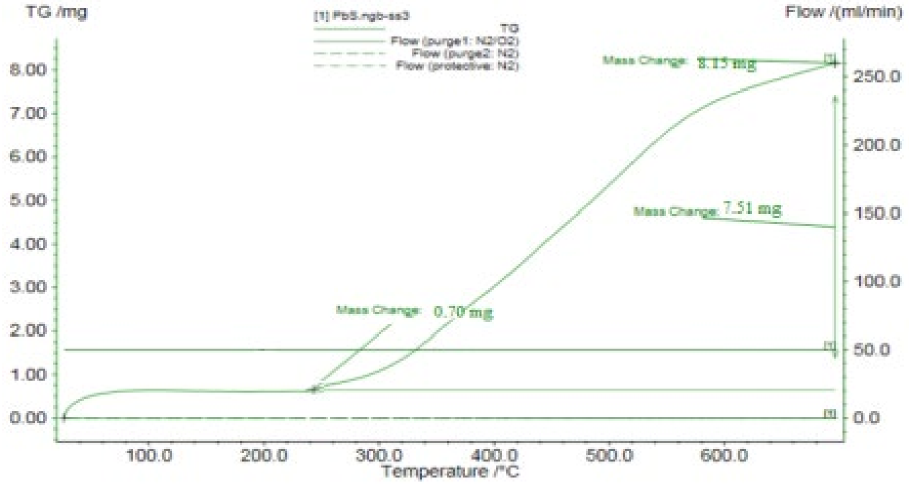

Fig. 3. The thermogram of PbS powder obtained by the chemical precipitation method.

During the heating of PbS powder in the temperature range of 20-700°C, a mass increase of 8.15 mg was observed. It is calculated that this increase occurred due to the oxidation of the sulfide ion to the sulfate ion.

$$PbS + 2O_2 \rightarrow PbSO_4$$

As stated in the literature [11, 13] during the oxidation of PbS, $PbSO_4$ is formed. The calculations show that there are 2.87 mg of sulfur in 21.5 mg of PbS powder. Theoretical calculations indicate that the oxidation of 2.87 mg of sulfur should produce 8.24 mg of SO_4.As seen from *Figure 3* 8.15 mg of SO_4 was produced from the oxidation of 2.87 mg of sulfur in the sample. Since the experimental values align with the theoretical calculations this confirms that the obtained compound corresponds to the PbS formula.

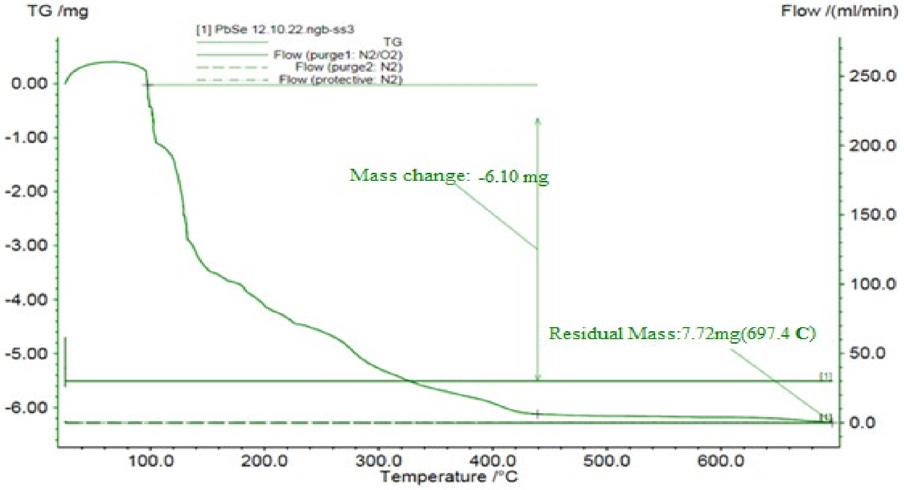

Fig. 4. The thermogram of PbSe powder obtained y the chemical precipitation method.

As seen from *Figure 4* the total mass of the 10.5 mg PbSe powder taken for analysis decreased by 6.10 mg at a temperature of 440°C. This loss is related to the evaporation of selenium in the form of SeO_2 oxide. It is known that the evaporation temperature of SeO_2 is 315°C [12]. Considering this information the mass loss at 440°C can be associated with the separation of

SeO_2. The 6.10 mg mass loss at 440°C indicates the volatilization of selenium in the form of SeO_2 as a result of the decomposition of PbSe. This helps in understanding the thermal properties of PbSe and the oxidation process of selenium. The residue remaining in the crucible corresponds to the amount of lead present in the PbSe composition. This demonstrates that lead is present in its residual form in the thermal analysis of PbSe.

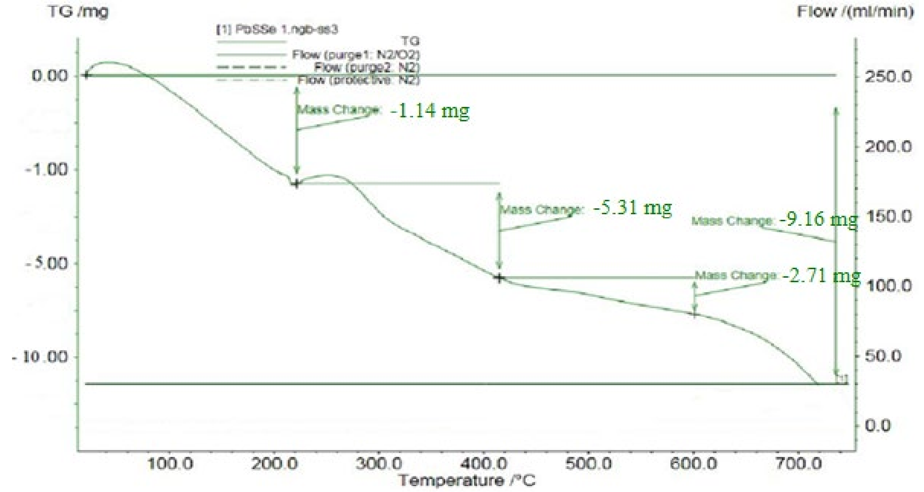

Fig. 5. Thermogravimetric analysis of $PbS_{0.5}Se_{0.5}$ *powder obtained by chemical precipitation.*

The total mass of 25 mg of $PbS_{0.5}Se_{0.5}$ powder taken for thermogravimetric analysis decreased by 3.56 mg at a temperature of 700 °C. According to the analysis results, the loss at 200 °C was 1.14 mg, which is related to sulfur. According to theoretical calculations the amount of sulfur in the sample should be 2.5 mg. The remaining residue (2.5 mg - 1.14 mg = 1.36 mg) was released as SO_4 at a temperature of 600 °C. The 1.14 mg loss at 200 °C indicates the sulfur separation due to heating at this temperature. Since the theoretical value is 2.5 mg, the remaining amount of sulfur is 1.36 mg. The 5.31 mg loss measured at 400 °C is related to the separation of selenium. This reflects the thermal decomposition of the $PbS_{0.5}Se_{0.5}$ compound. The remaining residue after the analysis is 15.84 mg, which indicates the presence of lead oxide (PbO). The obtained results reflect the thermal analysis of the $PbS_{0.5}Se_{0.5}$ compound. The loss values indicate the separation of sulfur and selenium, as well as the formation of lead oxide as the residue. Based on the research findings, the $PbS_{0.5}Se_{0.5}$ compound is confirmed. The thermogravimetric analysis of $PbS_{0.5}Se_{0.5}$ powder obtained by chemical precipitation shows that selenium is lost at 400°C, sulfur is released as a free element at 200°C, and SO_4 is released at 600°C. The remaining amount in the crucible corresponds to lead oxide.

4. Conclusion

X-ray diffraction (XRD) results confirmed that the peak positions of the PbS, PbSe, and PbS0.5Se0.5 thin films matched those in standard diffraction patterns, indicating phase purity and good crystallinity. Energy-dispersive X-ray spectroscopy (EDS) verified the elemental composition and confirmed the intended stoichiometry of each sample. Thermogravimetric analysis (TGA) revealed the thermal stability and oxidation behavior of each compound. For PbS, a mass gain was observed up to 700°C, attributed to the oxidation of sulfide to sulfate (PbSO4). PbSe showed a significant mass loss at 440°C due to the evaporation of selenium as SeO2. In the case of PbS0.5Se0.5, both sulfur and selenium components were released at different temperatures, with sulfur released at 200°C and sulfate formation at 600°C, while selenium evaporated at 400°C. The final residue corresponded to lead oxide (PbO). These findings demonstrate that chemical bath deposition is an effective method for producing high-quality PbS, PbSe, and PbS0.5Se0.5 thin films. Particularly, the PbS0.5Se0.5 compound shows promising structural and thermal characteristics, making it a strong candidate for applications in optoelectronics and infrared technologies.

References

[1] Maskaeva L.N. et al. Hydrochemical deposition of thin films of metal chalcogenides - practical course / Ural Federal University, -2017, -284 p. http://elar.urfu.ru/handle/10995/52376

[2] Maraeva E.V. Production and study of nanostructured polycrystalline layers and systems with quantum dots based on lead chalcogenides / Fundamental problems of radioelectronic instrument making, -2014. -V. 14, No. 3, -pp. 47-50. https://etu.ru/assets/files/nauka/dissertacii/2014/Maraeva_avt_1-(1).pdf

[3] Gamarts, A.E. Photoluminescence in polycrystalline layers of Pb1− xCdxSe activated in the presence of iodine vapor / Physics and technology of semiconductors, -2006. -V. 40, No. 6, pp. 683-685. https://journals.ioffe.ru/articles/viewPDF/6080

[4] Anwar, Materials Science in Semiconductor Processing, -2015. -T.40, -p.910-916; https://doi.org/10.1016/j.mssp.2015.07.077

[5] Grozdanov, I., Materials letters, -1999. -T.38, № 1, -p. 28-32; https://doi.org/10.1016/S0167-577X(98)00127-X

[6] Gautier C., Thin solid films, -1998. № 1-2, -p.118-122; https://doi.org/10.1016/S0040-6090(97)00785-2

[7] Rusu, G.I., Applied Surface Science, -2003. -T. 218, №1-4, -p.223-231; https://doi.org/10.1016/S0169-4332(03)00618-4

[8] Zhu, J., Materials research bulletin, -2001. -T.36, №7-8, - p.1169-1176; https://doi.org/10.1016/S0025-5408(01)00592-X

[9] Nasirov E.F. Synthesis of ZnSe nano and micro particles by chemical precipitation method / AJP PHYSICS, -2019. volume XXV, №3, -p.50-54. https://aak.gov.az/upload/dissertasion/fizika/Sara_Yasinova_Avtoreferat_Az.pdf

[10] Luther, J.M., Nano letters., -2008. T.8, № 10, -p.3488-3492; https://doi.org/10.1021/nl802476m

[11] Nasirov E.F. Synthesis of ZnSe nano and micro particles by chemical precipitation method / AJP PHYSICS, -2019. volume XXV, No. 3, p. 50-54.

[12] I.R.B.Ribeiro, J.Suela, Oliveira et. al.. Journal of Physics D: Applied Physics, -2007. -T.40, № 15, -p.4610-4614; https://doi.org/10.1088/0022-3727/40/15/037

[13] Sevda H.Aliyeva, Aliya B.Rzayeva, Journal of Chemistry and Technologies 2024, 32(2), 304-311; http://chemistry.dnu.dp.ua/article/view/299093

[14] P.G. Etchegoin, M. Cardona, R. Lauck, R.J.H. Clark, J. Serrano, A.H. Romero, Phys. Stat. Sol. B, 245 (6), 1125 (2008); https://doi.org/10.1002/pssb.200743364

[15] S.N. Yasinova, S.I. Mekhtiyeva, M.H.Huseynaliyev, R.I. Alekberov, Chalcogenide Letters Vol.21, No 5, May 2024, p. 377-383; https://doi.org/10.15251/CL.2024.215.377