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Deposition of zinc sulphide films from thiourea complexes and a study of their optical properties

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Abstract

This work presents the results of a study of zinc sulphide films deposited by aerosol pyrolysis from aqueous solutions of thiourea complex compounds $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ in the temperature range of 350–500 °C.

The IR and Raman spectra of zinc complexes were studied. It was determined that in the studied complexes, the thiourea molecule was coordinated to the metal cation through the sulphur atom. In the low-frequency Raman region ($\nu < 400 \text{ cm}^{-1}$), we recorded the bands characterising the vibrations of the zinc-sulphur and zinc-chlorine (bromine) bonds of the studied complex compounds in the Raman scattering spectra. The optical properties of zinc sulphide films were studied using optical spectrophotometry. Based on the absorption spectra, the optical band gap of ZnS films was determined. It was 3.67–3.74 eV and 3.63–3.70 eV for the samples deposited from $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ complexes, respectively. We recorded a decrease in the band gap of the synthesised layers upon an increase in the deposition temperature. It is due to changes in their defect structure.

One of the main types of defects in the ZnS films deposited from $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ complexes is a halogen atom in the anion sublattice of the sulphide (Cl_s^- , Br_s^-). As the deposition temperature increases, the content of these defects in the films decreases due to the complete destruction of Zn–Cl and Zn–Br bonds and volatilisation of halogen during the thermolysis of the complexes. Oxygen (O_s^x) occupies the vacated places of Cl_s^- , Br_s^- . The films contained oxygen as they were synthesised in an oxidising atmosphere and due to partial hydrolysis of the initial zinc salt. An increase of oxygen content in the samples upon an increase of the deposition temperature results in a decrease of the optical band gap of the ZnS films.

Keywords: Thiourea complex compounds, Aerosol pyrolysis, Zinc sulphide, Films, Optical band gap

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1. Introduction

Zinc sulphide films are of great interest because of their promising application in modern microelectronics. Various optoelectronic and recording devices, information display systems, electroluminescent light sources, etc., are based on zinc sulphide [1, 2]. Therefore, the synthesis of ZnS films with variable optical properties by a convenient and affordable method of aerosol pyrolysis of thiourea complex compounds (TCC) is a topical issue.

Aerosol pyrolysis makes it possible to synthesise targeted metal chalcogenide films with required semiconductor characteristics by producing complex compounds of different compositions and structures in a solution [3–5]. The sulphide phase is formed as a result of thermal destruction of the complex on a heated substrate. The crystal and defect structures of the sulphide films deposited by this method, as well as their properties, depend on the nature of the initial complex.

The aim of this study was to deposit zinc sulphide films from $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ complex solutions at different temperatures and to study the optical properties of the deposited films.

2. Experimental

Zinc sulphide films were synthesised by aerosol pyrolysis using aqueous solutions of thiourea complex compounds formed by the interaction of zinc salt and thiourea. To prepare TCC in an aqueous solution, we used chemically pure $\text{ZnCl}_2 \cdot 2.5\text{H}_2\text{O}$ and ZnBr_2 salts, as well as extra pure $\text{N}_2\text{H}_4\text{CS}$ thiourea. The concentration of the metal salt in the sprayed solution was 0.05 mol/l, the concentration of thiourea was 0.2 mol/l. Previous studies showed that at certain molar ratios of the used components, the formation of $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ complex compounds occurred in the solution at room temperature [6].

The TCC solutions were sprayed onto a heated substrate by a pneumatic nozzle. As a result of the thermal destruction of the complexes, metal sulphides were obtained. The substrates were silica plates, which were prewashed in nitric acid and chromic mixture, then washed repeatedly in distilled water. The deposition temperature

was varied from 350 to 500 °C. Each sample was sprayed for 1 to 2 minutes.

To study the optical properties of the films, we recorded absorption spectra using a Shimadzu UV-2550 spectrophotometer in the range of 190–900 nm versus a pure substrate (SiO_2 glass). The optical band gap E_g was determined based on the self-absorption edge from the spectral dependence $D = f(h\nu)$ (D is the optical density), assuming direct allowed transitions [7]. The measurements were carried out at room temperature.

The infrared (IR) and Raman spectra of the complexes were recorded to understand the mechanism of interaction between zinc salt and thiourea. IR spectroscopy was carried out by the method of attenuated total reflectance. The transmission spectra were recorded using a Vertex 70 IR Fourier spectrometer in the range of 400–4000 cm^{-1} . The zinc complexes were separated from the working solutions by the slow evaporation of the solvent. Initially, the samples were prepared in powder form.

The Raman spectra of the TCC were recorded using a RamMix M532 Raman microscope in the spectral range of 90–4000 cm^{-1} . The study was conducted using a laser with a radiation wavelength of 532 nm, a radiation region of 4 μm , and a spectral resolution of 2 cm^{-1} .

3. Results and discussion

The IR spectroscopy of the complex compounds obtained from aqueous solutions of zinc salts and thiourea (Fig. 1, 2) showed a shift of the frequency of stretching vibrations ν_{NH} and ν_{CN} to the high frequency region compared with the vibration of free thiourea, and ν_{CS} shifted to the low-frequency region of the spectrum (Tables 1, 2). It confirmed that the coordination of thiourea with zinc salt occurred through the sulphur atom $\text{N}_2\text{H}_4\text{CS}$ [8]. This is attributed to the redistribution of electron density in the thiourea molecule upon S-coordination, which causes a decrease in the order of the C–S bond and an increase in the order of the C–N and N–H bonds [8, 9].

In the Raman spectra of the studied complex compounds, there were intense bands in the low-frequency region ($\nu < 400 \text{ cm}^{-1}$) characterising the vibrations of the metal-ligand bonds (Fig. 3, 4). According to the results of studies [10–13], the bands with wave numbers of 250–280 cm^{-1}

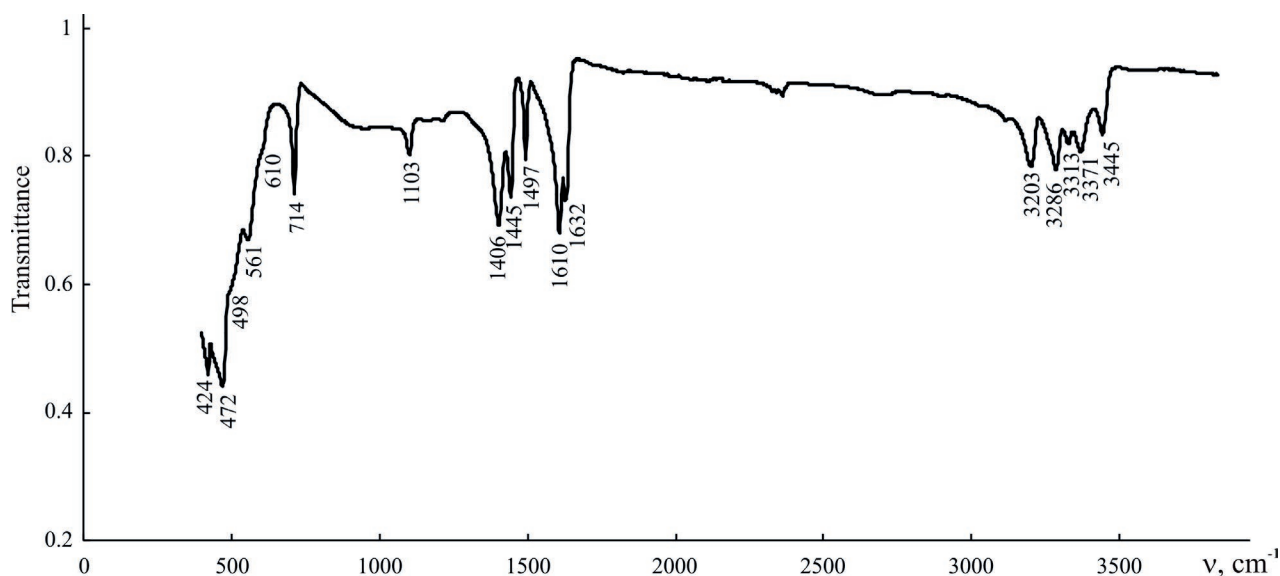


Fig. 1. The IR spectrum of the complex compound obtained from an aqueous solution of zinc chloride and thiourea

Table 1. Maximum of absorption bands (cm^{-1}) of IR spectra and Raman spectra of complex compounds, prepared from an aqueous solution of zinc chloride and thiourea

Assignment	$\text{N}_2\text{H}_4\text{CS}$ [8, 9]	$[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ (ИК, [8])	$\text{ZnCl}_2 + \text{N}_2\text{H}_4\text{CS}$ (IR, experiment)	$\text{ZnCl}_2 + \text{N}_2\text{H}_4\text{CS}$ (Raman, experiment)
$\nu(\text{NH})$			3445	3449
	3375	3370	3371	3379
	3273	3330	3313	3339
	3160	3210	3286	3297
			3203	3212
$\delta(\text{HNH})$		1630	1632	1630
$\delta(\text{HNC})$	1606	1612	1610	1612
$\nu(\text{CN})$	1464	1494	1497	1491
$\nu(\text{HNC})$	1408	1448	1445	1444
$\nu(\text{CS})$		1415	1406	1402
$\nu(\text{CN})$	1082	1106	1103	1107
$\nu(\text{CS})$	729	718	714	721
$\delta(\text{NCN})$	629	600	610	619
			561	
$\delta(\text{NCN})$	484	478	498	479
			472	
$\nu(\text{NCS})$	420	422	424	430
$\delta(\text{ZnCl})$				238
$\delta(\text{ZnS})$				275
				162

correspond to vibrations of the zinc-sulphur bond (Tables 1, 2). In the Raman spectrum of the $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ compound, we recorded a low intensity peak at 162 cm^{-1} , which can also be attributed to the vibrations of the Zn–S bond [14].

The band recorded in the Raman spectrum of the $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ compound at 238 cm^{-1} is

attributed to the vibrations of the Zn–Cl bond [15, 16]. For heavier elements, the vibration frequencies shifted to the low-frequency region. Thus, the Zn–Br bond vibration in the $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ complex was observed at 184 cm^{-1} [17, 18].

In the Raman spectra of the studied complexes, there are two bands in the range of $50\text{--}140 \text{ cm}^{-1}$.

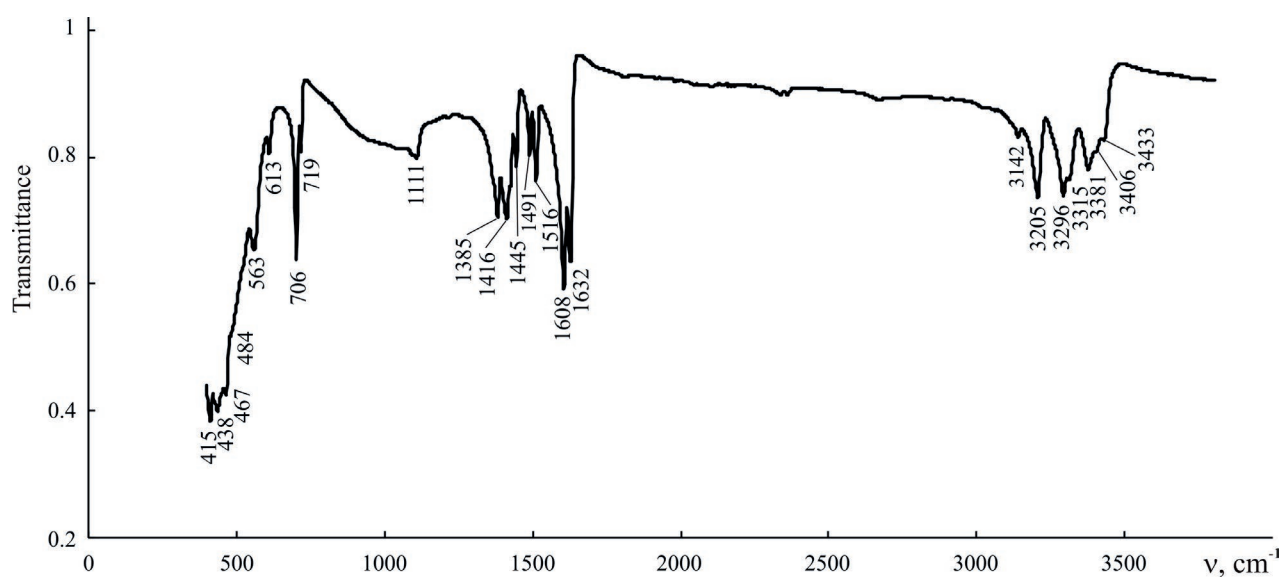


Fig. 2. The IR spectrum of the complex compound obtained from an aqueous solution of zinc bromide and thiourea

Table 2. Maximum of absorption bands (cm^{-1}) of IR spectra and Raman spectra of complex compounds, prepared from an aqueous solution of zinc bromide and thiourea

Assignment	$\text{N}_2\text{H}_4\text{CS}$ [8, 9]	$\text{ZnBr}_2 + \text{N}_2\text{H}_4\text{CS}$ (IR, experiment)	$\text{ZnBr}_2 + \text{N}_2\text{H}_4\text{CS}$ (Raman, experiment)
$\nu(\text{NH})$		3433	3431
		3375	3398
		3273	3380
		3160	3327
			3217
			3205
			3142
$\delta(\text{HNH})$		1632	1631
$\delta(\text{HNC})$	1606	1608	1615
$\nu(\text{CN})$	1464	1516	1518
		1491	1491
$\nu(\text{HNC})$	1408	1445	1431
$\nu(\text{CS})$		1416	1385
		1385	
$\nu(\text{CN})$	1082	1111	1112
			1098
$\nu(\text{CS})$	729	719	721
		706	705
$\delta(\text{NCN})$	629	613	605
		563	528
$\delta(\text{NCN})$	484	484	473
		467	
$\delta(\text{NCS})$	420	438	430
		409	
$\nu(\text{ZnBr})$			184
$\nu(\text{ZnS})$			258

The more intense band is in the frequency range of 100–115 cm^{-1} , and the less intense one is in the range of 140–145 cm^{-1} (Fig. 3, 4). According to the authors of [12, 19], the bands observed in the above region characterise the translational and rotational motion of the thiourea molecule, which is part of the complex compound. Apparently, the first band recorded at 113 cm^{-1} for the chloride complex and at 100 cm^{-1} for the bromide TCC characterises the translational motion of the coordinated $\text{N}_2\text{H}_4\text{CS}$ molecule. The low intensity band with maxima at 141 and 145 cm^{-1} for the chloride and bromide complexes, respectively, characterises the rotational motion of the

thiourea molecule. The deformation vibrations $\delta(\text{ClZnCl})$ and $\delta(\text{BrZnBr})$ in zinc complexes, as a rule, appear in the lower frequency region, at 95–100 cm^{-1} and 70–75 cm^{-1} , respectively [20, 21].

Thus, the results of the IR and Raman spectroscopy prove the formation of metal-sulphur bonds in the inner sphere of the complex compound. The bonds are the fragments of the crystal structure of the prospective sulphide, which is formed during the thermolysis of the respective complex. The defect structure of the obtained sulphide depends on the immediate environment of the complexing substance in the first coordination sphere. Therefore, it is possible

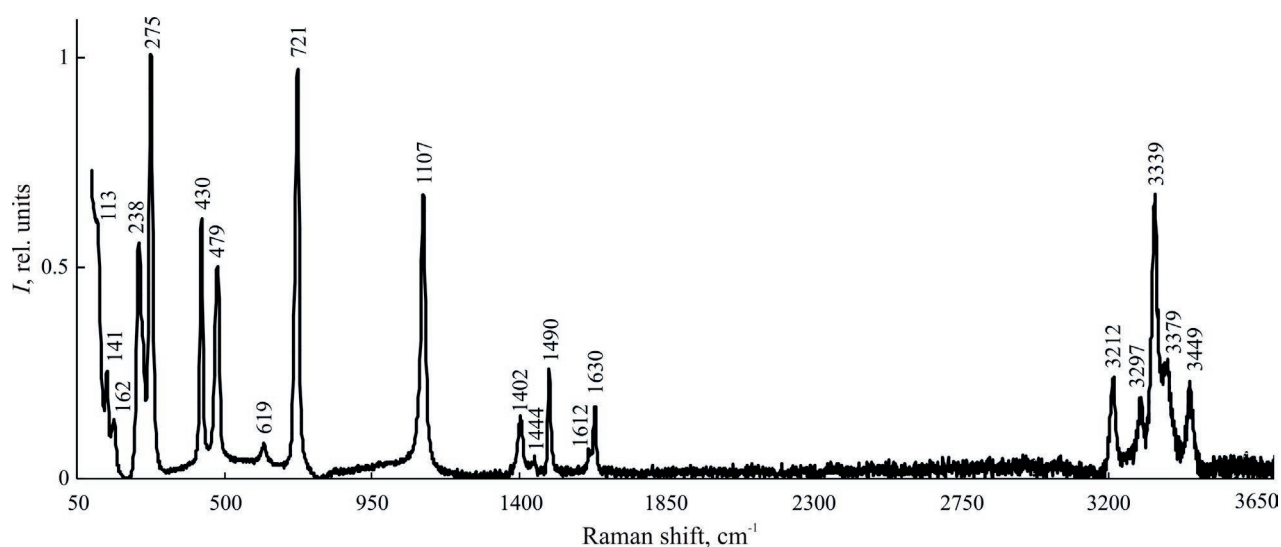


Fig. 3. The Raman spectrum of the complex compound obtained from an aqueous solution of zinc chloride and thiourea

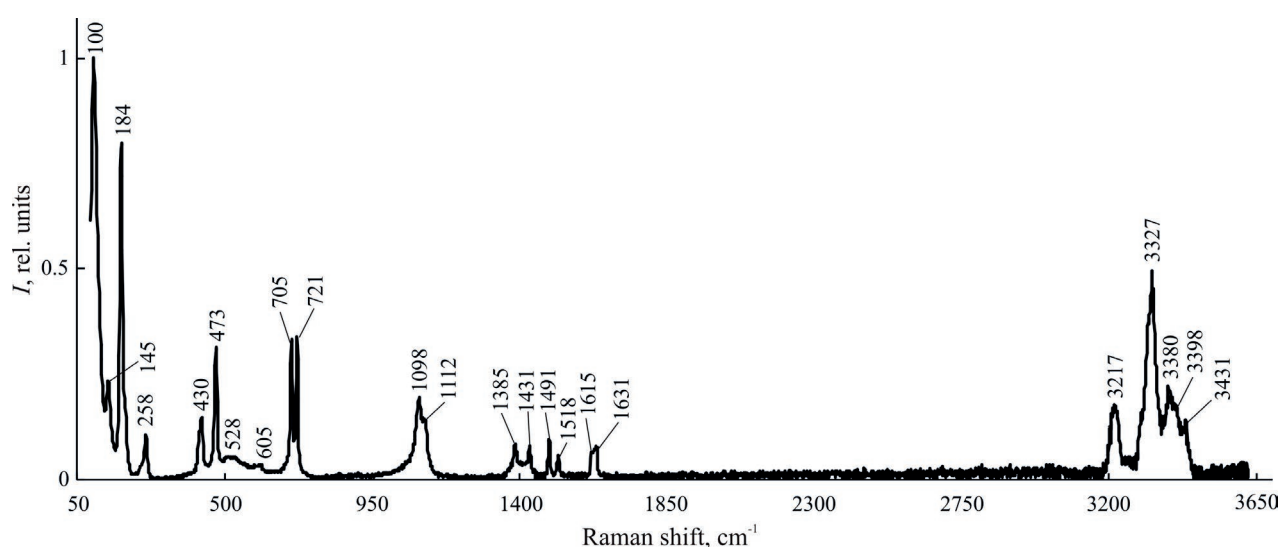


Fig. 4. The Raman spectrum of the complex compound obtained from an aqueous solution of zinc bromide and thiourea

to model the structure by introducing different acidic ligands [3–5]. The relationship between the composition and structure of the environment of the central ion in the TCC and the environment of the metal cation in the sulphide lattice is the basis for the directed synthesis of metal sulphide films with a particular set of semiconducting properties.

When studying the optical properties of the synthesized ZnS films, we obtained the absorption spectra in the region of the fundamental absorption edge. By extrapolating the linear section of the power dependence of the optical density on the photon energy $(hvD)^2 = f(hv)$ (Fig. 5) to the abscissa axis, we determined the optical band gap of the zinc sulphide layers. The ZnS films deposited from the $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ complex solutions are characterised by the optical band gap of 3.67–3.74 eV. For the samples obtained using the $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ TCC, the E_g values are 3.63–3.70 eV (Table 3). It is clear from the obtained data that the optical band gap of ZnS films generally decreases when the deposition temperature

increases from 350 to 500 °C. A similar trend was observed in studies of other authors [22, 23].

The specifics of the change in E_g depending on the spraying temperature of the samples can be associated with a change in the defect structure of the films. Thus, the samples deposited from the $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ TCC contain chlorine (bromine) that replaces sulphur during the decomposition of the initial complexes. They also contain oxygen due to the synthesis in the air and partial hydrolysis of the initial zinc salt. With an increase in the deposition temperature, the Zn–Cl and Zn–Br bonds decompose more completely, and the content of halogen atoms in the films decreases. The reason is that they are removed as volatile products during the thermolysis of the $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ compounds. In this case, there are less Br_s^{\cdot} and Cl_s^{\cdot} defects in ZnS films, and the vacated places of sulphur are occupied by oxygen, forming the O_s^x defects. In addition, due to the partial hydrolysis of the initial zinc

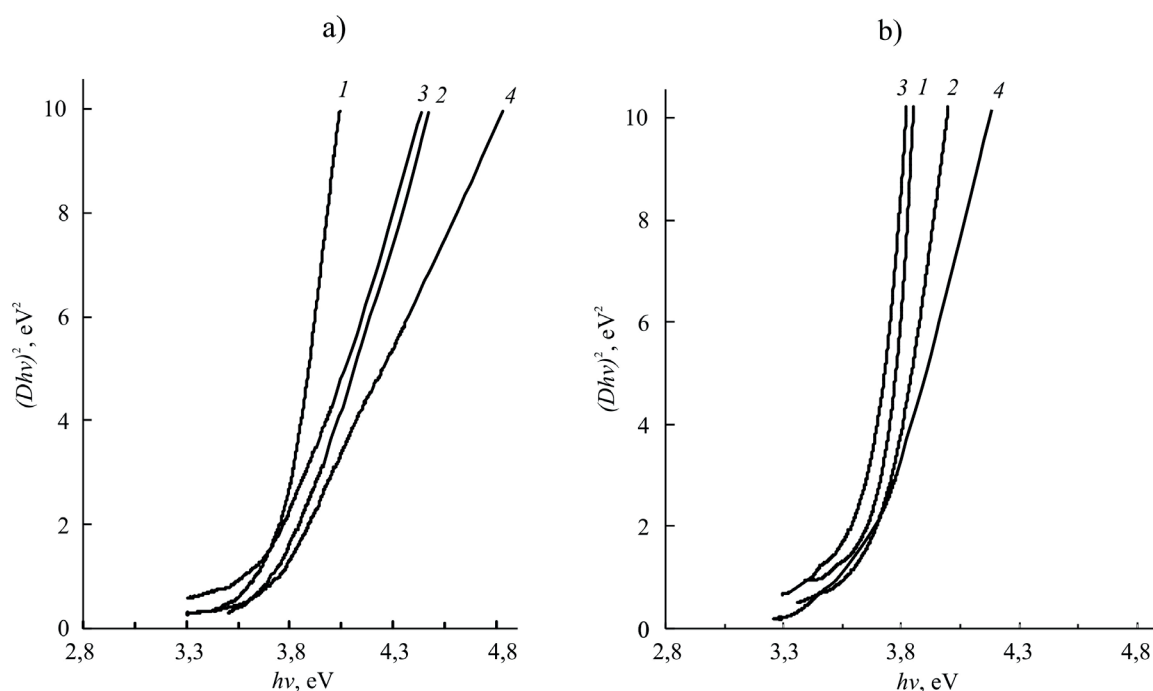


Fig. 5. The absorption spectra of the ZnS films obtained from the $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ (a) and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ (b) TCC solutions at different temperatures: 1 – 350 °C; 2 – 400 °C; 3 – 450 °C; 4 – 500 °C

Table 3. Optical band gap (eV) of ZnS films deposited at different temperatures

Complex compound	350 °C	400 °C	450 °C	500 °C
$[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$	3.74	3.73	3.70	3.67
$[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$	3.70	3.70	3.65	3.63

salt in the samples synthesised by this method, ZnS may contain an impurity of ZnO oxide, which has a lower band gap than that of zinc sulphide. Its values are 3.35–3.43 eV [24]. Therefore, the presence of oxygen in the samples leads to a decrease in the optical band gap of ZnS with an increase in the deposition temperature.

It should be noted that pyrolytic deposition of zinc oxide films from an aqueous solution of zinc nitrate also showed a decrease in the optical band gap of the samples with an increase in the synthesis temperature [25]. Thus, for ZnO films obtained at 250 °C, the value of E_g was 3.45 eV, and when the deposition temperature reached 295 °C, the band gap decreased to 3.25 eV.

The zinc sulphide films obtained by the spraying of $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_4](\text{NO}_3)_2$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2(\text{CH}_3\text{COO})_2]$ complex solutions in the temperature range of 350–500 °C are characterised by the optical band gap of 3.39–3.41 eV and 3.38–3.50 eV, respectively. The lower E_g values of the ZnS layers obtained from the above compounds compared with those of the samples from the halide complexes are also due to the oxygen present in them.

4. Conclusions

We synthesised zinc sulphide films from $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Cl}_2]$ and $[\text{Zn}(\text{N}_2\text{H}_4\text{CS})_2\text{Br}_2]$ thiourea complex compounds by aerosol pyrolysis at temperatures from 350 to 500 °C. The coordination of thiourea to metal cation in the complex compound occurs through the sulphur atom. Using the absorption spectra of the ZnS films, we determined the values of their optical band gap (3.63–3.74 eV). The optical band gap of the ZnS films decreases as the deposition temperature increases from 350 to 500 °C. It is due to a change in the defect structure of the sulphide.

Author contributions

All authors made an equivalent contribution to the preparation of the publication.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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