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Physical Properties of Zinc Compounds Obtained by Electrolytic Method

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The influence of the chemical composition of the electrolyte and its temperature on the process of synthesis of nanosized zinc compounds by electrolytic method using zinc electrodes was investigated. X-ray studies have been conducted and its results were used to determine the composition of the obtained nanocrystal samples and its dimensioning using the Debye and the Williamson Hall methods. Comparisons of the results of dimensioning of nanocrystals by both methods were made. Also discussed the possibilities of synthesis of nanoparticles of zinc oxide, zinc sulfide and hydrozincite by electrolytic method. It is shown, that depending on the electrolyte composition, nanocrystals of zinc oxide, zinc sulfide, hydrozincite or their mixture are obtained. The effect of thermal annealing on the samples composition and dimensioning was investigated.

Keywords: zinc oxide, zinc sulfide, hydrozincite, XRD analysis, nanoparticles dimensioning, Scherrer method, Williamson Hall method, thermal annealing.

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Introduction

The creation of high-quality electronic devices is possible only in the presence of appropriate materials with the necessary physical properties, which depend on their chemical composition, purity, structural perfection, synthesis technologies and post processing. A^{II}B^{VI} solid solutions and compounds are promising semiconductor materials for use in modern science and technology [1].

Nowadays we see a growing interest to A^{II}B^{VI} semiconductor compounds in nanostructured form. Zinc oxide (ZnO) is a multifunctional material that is widely used in semiconductor devices, optoelectronics, gas sensors, production of dielectric and magnetic materials. Nanosized zinc oxide has a significant potential for use in spintronics and photonics [2].

Renewable energy sources provide the production of environmentally friendly energy, which is an alternative to traditional energy, and solar cells are a common form of renewable energy. Solar thin-film elements based on Cu (In, Ga) Se₂ are considered to be one of the most promising for solar generation. Cadmium sulfide (CdS)

is used as a buffer layer in such solar cells, but with use of zinc sulfide (ZnS), the efficiency of a solar cell will increase [3].

In addition, ZnS is widely used in photoluminescent and electroluminescent devices, optical sensors. Both, ZnS and ZnO, are used for the manufacture a conductive optical windows for optoelectronics and solar cells [4, 5].

A^{II}B^{VI} semiconductor nanoparticles are obtained by various methods such as: laser sputtering, electrochemical deposition, thermal sputtering, magnetron sputtering, mechanochemical milling, chemical deposition, molecular beam epitaxy, synthesis in colloidal solutions, electric discharge in water, hydrothermal synthesis. The development of methods of study and control dimensional and structural characteristics has great importance in the synthesis of nanomaterials. The main methods of studying particle dispersion are electron transmission and scanning microscopy (TEM and SEM), optical and X-ray studies [6-8].

This work is a continuation of our work [9] where the influence of type of the electrolyte on the synthesis of zinc compounds nanocrystals at room temperature was

studied. The aim of this work is to study the influence of temperature and electrolyte composition on the processes of synthesis of zinc compounds nanocrystals by the electrolytic method.

I. Experiment and materials

Nanocrystals of zinc compounds were synthesized by the electrolytic method in an open glass electrolytic cell with a volume of 1 l with zinc electrodes. A stabilized DC source was used as cell power supply. Distilled water and the following reagents were used to prepare the electrolytes: Sodium Sulfite (Na_2SO_3), Sodium Sulfide 9 Hydrate ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$), Sodium Carbonate (Na_2CO_3). The weights of the reagents (except of sodium carbonate) were calculated so, that the volume of 0.8 l of electrolyte contained the same amount of sulfur, namely 2.581 g. In the case of sodium carbonate, 5 g of Na_2CO_3 per 0.8 l of distilled water was used. The electrolyte temperature through the process of synthesis maintained constant, but varies from room temperature to 100 °C in different experiments.

The duration of the nanocrystal synthesis was 3 hours; the density of current was $1.3 \cdot 10^{-2} \text{ A/cm}^2$. For uniform use of the material of the electrodes, the direction of the current was reversed every 30 minutes. After synthesis, the electrolyte has been decanted and filtered using a paper filter. Obtained powder-like product was washed five times its volume with distilled water. The samples were dried in air at room temperature.

After drying, some of the samples were annealed in air at a temperature of 550 °C for one hour. The annealing process was carried out in an electric

resistance furnace. The annealing temperature was controlled by a differential chromel-alumel thermocouple.

XRD studies were performed at room temperature on a DRON-4 Bragg–Brentano focusing geometry (θ - 2θ) diffractometer using $\text{CuK}\alpha$ radiation. The X-ray tube anode voltage and current were 41 kV and 21 mA, respectively. The XRD scanning step was 0.05° , the exposure time – 5 sec.

For detailed analysis of gathered XRD data, it was processed as follows: each experimental reflex was described by a Gaussian function and gathered the following information: angular position 2θ , half-width (width at half height) β , integral intensity I . These data were used to interpret experimental diffraction patterns, determine dimensioning of nanocrystals and its mechanical stresses.

II. Research results

The experimental XRD patterns (Fig. 1) of samples obtained by the electrolytic method using an $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ solution as electrolyte are given. The XRD pattern of the sample obtained at electrolyte temperature of 19 °C (Fig. 1a) was analyzed in detail in our work [9], this XRD pattern contains reflexes belonging to zinc sulfide and hydrozincite ($\text{Zn}_5(\text{CO}_3)_2(\text{OH})_6$). The XRD pattern of the sample obtained at an electrolyte temperature of 98 °C (Fig. 1b) differs significantly from the previous one. Those four intense reflexes with an angular position of 2θ : 28.7°; 47.7°; 56.6°; 77.1°, which are indexed as (111), (220), (311), (331) of cubic syngony ZnS.

Besides, there are reflexes of lower intensity with an angular position of 2θ : 31.8°; 34.5°; 36.3°; 62.9°, which

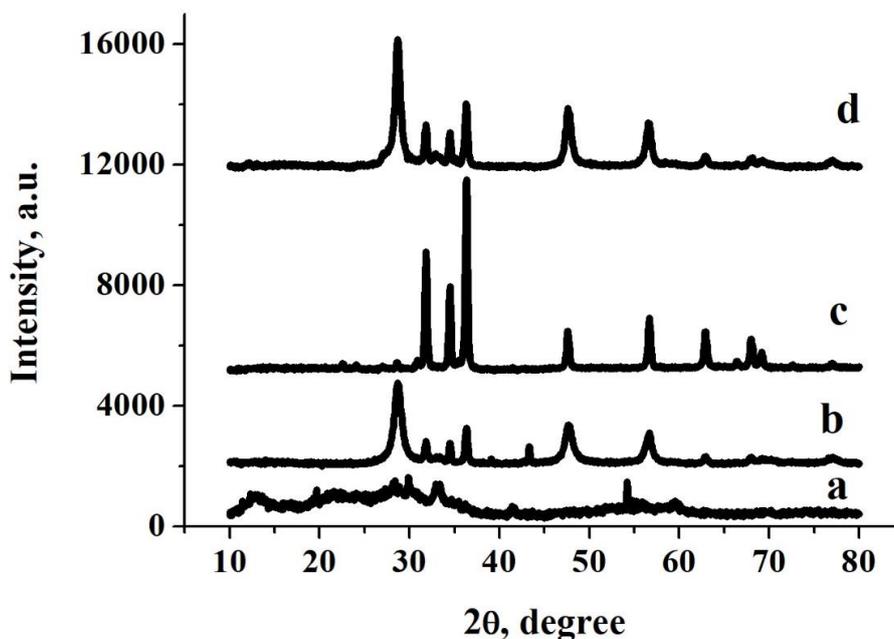


Fig. 1. X-ray diffraction patterns of samples obtained by the electrolytic method using $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ solution as electrolyte: a – electrolyte temperature 19°C; b – electrolyte temperature 98°C; c – obtained at an electrolyte temperature of 19°C and annealed at a temperature of 550°C; d – obtained at an electrolyte temperature of 98°C and annealed at a temperature of 550°C.

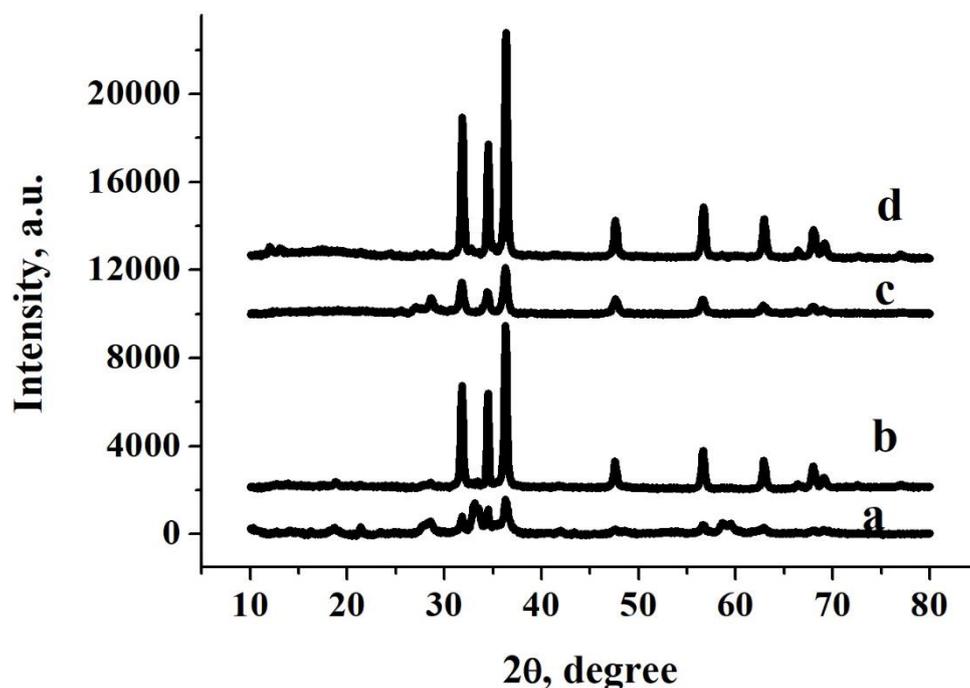


Fig.2. XRD patterns of samples using Na_2SO_3 solution as electrolyte: a – electrolyte temperature 19°C; b – electrolyte temperature 98 °C; c – obtained at an electrolyte temperature of 19 °C and annealed at a temperature of 550 °C; d – obtained at an electrolyte temperature of 98 °C and annealed at a temperature of 550 °C.

belongs to zinc oxide in the hexagonal wurtzite structure with Miller indices (100), (002), (101), (103). It is known [10] that the diffraction pattern of zinc oxide nanocrystals obtained by the electrolytic method is characterized by reflexes (100), (002), (101), (102), (110), (103), (200), (112), (201), with angular position of 2θ : 31.8°; 34.5°; 36.3°; 47.6°; 56.7°; 62.9°; 66.5°; 68.0°; 69.2°. In our case, we do not observe (102) and (110) reflexes because their angular position is quite close to the angular position of the (220) and (311) reflexes of zinc sulfide, and their intensity is less compared to intensity of ZnS reflexes. (200), (112), (201) reflexes have the lowest intensity among other ZnO reflexes in this 2θ range; therefore they are practically not registered on the XRD pattern.

In Fig. 2 shown experimental XRD of samples obtained by electrolytic method using an electrolyte based on Na_2SO_3 . The diffraction pattern of the sample synthesized at an electrolyte temperature of 19 °C (Fig. 2a) was analyzed in detail in our work [9] and it contains reflexes both of zinc oxide and hydrozincite.

When the synthesis temperature increases to 98 °C (Fig. 2b), there is a significant increase of reflexes intensity of zinc oxide and a decrease intensity of hydrozincite reflexes on the diffraction pattern. To experimentally confirm the formation of hydrozincite, we obtained samples by electrolytic method using an electrolyte prepared from sodium carbonate (Na_2CO_3). In Fig. 3 shown XRD patterns of the obtained samples at an electrolyte temperature of 19 °C and annealed at a temperature of 550 °C in an electric resistance furnace in air. Analysis of the XRD data and comparisons with the data from [11-12] shows that we obtained hydrozincite by the electrolytic method.

On XRD pattern (Fig. 3b) of the sample after heat treatment at a temperature of 550°C a weak reflexes of zinc oxide are observed in addition to the hydrozincite reflexes. This means that heat treatment process led to a decomposition of hydrozincite by the reaction:



We observed this process during the differential thermal analysis (DTA) of our samples synthesized at 19 °C. Carbon dioxide, needed for the formation of hydrozincite, it is a gas, dissolved in distilled water, which is used to prepare the electrolyte, also it may income from the atmosphere during the synthesis, because an open cell is used for. Besides, the lower is a temperature of synthesis, the higher is content of hydrozincite in the samples is observed, it evidenced by the intensity of reflexes $\text{Zn}_5(\text{CO}_3)_2(\text{OH})_6$. The authors of works [13-14] observed the production of cadmium carbonate when trying to obtain nanocrystals of cadmium compounds $\text{Cd}(\text{OH})_2$ and CdS by the electrolytic method at room temperature. This confirms the presence of carbonate ions CO_3^{2-} in the electrolyte and the possibility of its income into the cell during the synthesis of nanoparticles.

Annealing of the sample obtained at 19 °C using solution $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ as electrolyte, leads to significant changes in the XRD pattern (Fig. 1c). On the diffraction pattern there are intense reflexes of zinc oxide and almost no reflexes of hydrozincite and zinc sulfide. Annealed sample obtained at 98 °C shows on XRD (Fig. 1d), only increased reflexes of ZnO and ZnS. Annealing of samples obtained from the Na_2CO_3 electrolyte leads to an intensity increase in the of ZnO reflexes and a significant

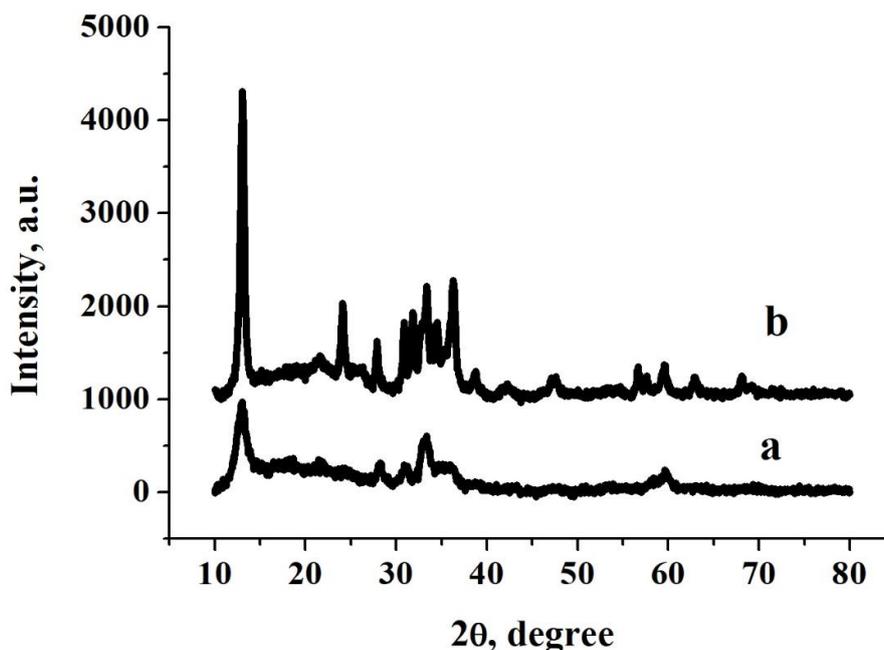


Fig. 3. XRD patterns of samples obtained by the electrolytic method using Na_2CO_3 solution as electrolyte: a – electrolyte temperature 19°C ; b – obtained at an electrolyte temperature of 19°C , annealed at a temperature of 550°C .

decrease in the intensity of $\text{Zn}_5(\text{CO}_3)_2(\text{OH})_6$ reflexes (XRD patterns on Fig. 2c, Fig. 2d).

The results of XRD analysis were used to determinate the size of nanocrystals using the Scherrer formula [15]:

$$D = \frac{0.89\lambda}{\beta \cdot \cos \theta}, \quad (2)$$

where λ is X-rays wavelength; β is the half-width of the reflex; θ is the diffraction angle. The physical broadening of the half-width β is calculated by the formula:

$$\beta = \left(\beta_1^2 - \beta_2^2 \right)^{1/2}, \quad (3)$$

where β_1 is the experimental value of the half-width of the XRD reflex; β_2 is the instrumental broadening of the half-width of the XRD reflex.

The instrumental broadening β_2 was determined by analysis and comparison our XRD data of powders of silicon and Al_2O_3 (obtained under the same experimental conditions as samples) with well known reference XRD patterns.

The application of the Scherrer formula is based on the dependence of the half-width β of the reflex on the crystal size, and the half-width increases with decreasing size. Besides, is known that the half-width is affected by mechanical stresses that occur due to defects in the crystal structure. In the case of nanoparticles, defects can occur due to the fact that nanosized crystals have a significant part of the atoms on the surface, and the contribution of surface atoms will increase with decreasing of crystalline size.

Therefore, to determine the size and mechanical stresses acting in ZnO nanocrystals, we used the Williamson-Hall method [15-16]. In this method, the half-width of the reflex is described by the formula:

$$\beta = \frac{0.89\lambda}{D \cos \theta} + 4\varepsilon \tan \theta, \quad (4)$$

where λ is the wavelength of X-rays, ε is the relative elongation. The first term of last formula shows the contribution of the dimensional effect to the half-width, and the second – the contribution of mechanical stresses. Rewrite last equation in the form:

$$\beta \cos \theta = \frac{0.89\lambda}{D} + 4\varepsilon \sin \theta. \quad (5)$$

According to Hooke's law, mechanical stress at elastic deformations is equal to:

$$\sigma = E \cdot \varepsilon, \quad (6)$$

where E is the Young's modulus. Determining the relative elongation from Hooke's law, we obtain the dependence:

$$\beta \cos \theta = \frac{0.89\lambda}{D} + \frac{4\sigma \sin \theta}{E}. \quad (7)$$

If to plot this dependence using the $4\sin\theta/E$, $\beta\cos\theta$ coordinate system, then the graph will be a straight line, using it parameters we can determine the size of nanocrystals D and the mechanical stress σ . For monocrystals, the Young's modulus will depend on the direction inside the single-crystal, ie, on the values of the

Miller indices (h, k, l) and the type of crystal system. The dependence for zinc oxide (belongs to the hexagonal

system) is described by the formula [17]:

$$E^{-1} = \frac{\left(h^2 + k^2 - hk\right)^2 a^4 s_{11} + l^4 c^4 s_{33} + \left(h^2 + k^2 - hk\right) l^2 a^2 c^2 \left(s_{44} + 2s_{13}\right)}{\left(\left(h^2 + k^2 - hk\right) a^2 + l^2 c^2\right)^2}, \quad (8)$$

where $S_{11}, S_{13}, S_{33}, S_{44}$ are the elastic coefficients, a and c are the parameters of the elementary cell ZnO. Zinc sulfide crystallizes in cubic syngony, the equation has the form [17]:

$$E^{-1} = s_{11} - \frac{\left(2s_{11} - 2s_{12} - s_{44}\right)\left(h^2 k^2 + k^2 l^2 + l^2 h^2\right)}{\left(h^2 + k^2 + l^2\right)^2}, \quad (9)$$

where S_{11}, S_{12}, S_{44} are the elastic coefficients of ZnS.

Using the known elastic coefficients, the parameters of the elementary cells [17] of zinc oxide and zinc sulfide, as well as the Miller indices of the reflexes experimental diffractograms, we calculated the values of the Young's modulus for our reflexes. In Fig. 4 shown examples of using the Williamson-Hall method for ZnO of a sample obtained from an electrolyte (Na_2SO_3) at a temperature of 98 °C and zinc sulfide of a sample obtained from an electrolyte ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$) at 98 °C and annealed in air at a temperature of 550 °C (Fig. 5). It is seen that there is a deviation of the experimental points from the straight line. Therefore, the least squares method was used to obtain reliable values of 0.89 λ/D and mechanical stress.

The results of dimensioning of the nanocrystals by the Scherrer method for samples obtained using different electrolytes after annealing are given in Table 1. To determine the size of nanocrystals of a particular substance used several reflexes, therefore in Table 1 the mean values of the sizes received from various reflexes are resulted.

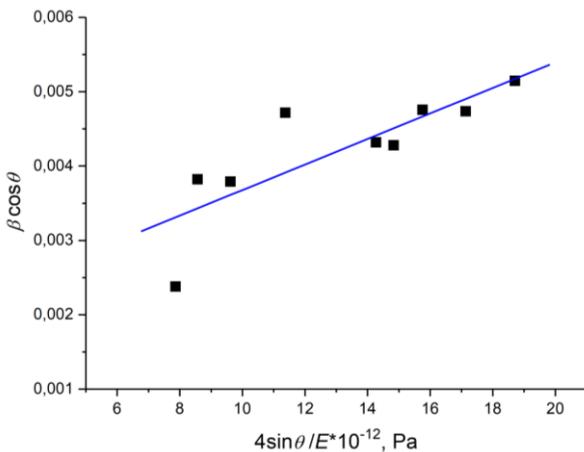


Fig. 4. The results of using the Williamson-Hall method for ZnO nanocrystals obtained from Na_2SO_3 solution as electrolyte at temperature of 98 °C.

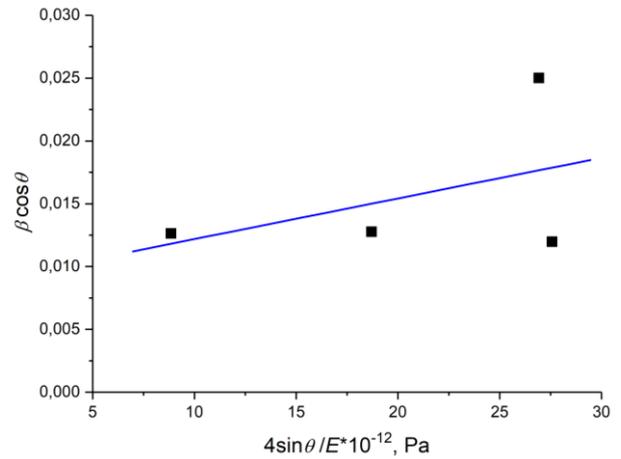


Fig. 5. The results of using the Williamson-Hall method for ZnS nanocrystals synthesized from $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ solution as electrolyte at temperature 98 °C and annealed at a temperature of 550 °C.

Information from Table 1 shows that if used sodium sulfite solution as electrolyte, then at room temperature a mixture of zinc oxide and hydrozincite is obtained. Increasing the temperature of the electrolyte to 98°C, as well as annealing leads to the formation of only ZnO and change its crystalline size.

If $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ solution is used for electrolyte preparation, synthesis at temperature of 19 °C leads to obtaining zinc sulfide and hydrozincite. Increasing the temperature of the synthesis to 98 °C leads to the production of ZnS and ZnO (hydrozincite reflexes on the XRD are absent). Annealing of the sample leads to the appearance of ZnO nanocrystals with a size of 33.2 nm. This fact can be explained by the oxidation of zinc sulfide and the decomposition of hydrozincite, as it mentioned above.

On Table 2 shown the results of applying the Williamson-Hall method to determine the size of nanocrystals and mechanical stresses for substances

Table 1

The results of dimensioning of nanocrystals (by the Scherrer method)

Electrolyte	Temperature		Substance		
	Synthesis	Annealing	ZnS	ZnO	Zn ₅ (CO ₃) ₂ (OH) ₆
			<i>D</i> , nm	<i>D</i> , nm	<i>D</i> , nm
Na ₂ SO ₃	19°C	-	-	25.1	5.6
	19°C	550°C	-	17.0	-
	98°C	-	-	34.1	-
	98°C	550°C	-	26.8	-
Na ₂ S·9H ₂ O	19°C	-	1.6	-	2.3
	19°C	550°C	-	33.2	-
	98°C	-	8.9	24.1	-
	98°C	550°C	9.6	18.2	-
Na ₂ CO ₃	19°C	-	-	-	1.6
	19°C	550°C	-	15.0	2.3

Table 2

The results of study of the samples using the Williamson-Hall method

Electrolyte	Temperature		Substance			
	Synthesis	Annealing	ZnS		ZnO	
			<i>D</i> , nm	$\sigma \cdot 10^8$, Pa	<i>D</i> , nm	$\sigma \cdot 10^8$, Pa
Na ₂ SO ₃	19°C	-	-	-	52.1	3.4
	19°C	550°C	-	-	13.7	1.2
	98°C	-	-	-	69.0	1.7
	98°C	550°C	-	-	66.0	2.5
Na ₂ S·9H ₂ O	19°C	-	-	-	-	-
	19°C	550°C	-	-	33.1	0.18
	98°C	-	7.2	1,6	25.5	0.98
	98°C	550°C	20.9	0.53	15.3	3.2

obtained by the electrolytic method.

To the samples obtained using an electrolyte based on Na₂S·9H₂O at a temperature of 19 °C, the Williamson-Hall method was not applied because the intensity of the cubic ZnS XRD reflexes is quite small. Also, this method was not used for hydrozincite.

In the works [19-20] authors investigated the physical properties of nanocrystals obtained by the electrolytic method using an electrolyte based on sodium thiosulfate. It was found that when the electrolyte temperature changes from room to 85 °C, nanocrystals of zinc sulfide and hydrozincite are obtained, and in the temperature range of 85 - 100°C, nanocrystals of zinc oxide and sulfide are synthesized. The sizes of ZnS nanocrystals obtained at room temperature were 1.6 nm, at 98 °C – 1.8 nm (for ZnS) and 98 nm (for ZnO). In our case, the sizes of zinc sulfide nanocrystals using the

Scherrer formula are also 1.6 nm (electrolyte based on Na₂S·9H₂O, temperature is 19 °C), and the sizes of ZnO nanoparticles are smaller. Increasing of the synthesis temperature to 98°C leads to rise (up to 8.9 nm) of the of ZnS nanocrystals size.

Authors of the work [21] used a mixture of solutions of zinc acetate and sodium sulfide to obtain ZnS in a magnetic stirrer at room temperature. As a result, they obtained a nanoparticle of zinc sulfide with a size of 1.8 nm. In the work [22] authors investigated mixtures of nanoparticles of zinc sulfide and zinc oxide. Nanocrystals of ZnS were obtained in a magnetic stirrer at room temperature using solutions of zinc chloride and sodium sulfide; using a solution of zinc acetate and sodium hydroxide leads to ZnO obtaining. The dimensioning of nanoparticles were made by the Scherrer formula, the results were: 5.34 nm for ZnO and 2.1 nm

for ZnS. The mixtures obtained by mixing suspensions of nanoparticles of ZnO to ZnS in ethanol with a molar ratio: 1:5 and 5:1 were investigated. When the ratio of ZnO to ZnS was 1:5, on the XRD pattern has reflexes of both zinc oxide and zinc sulfide. When the ratio of ZnO to ZnS was 5:1, the XRD pattern has reflexes only of zinc oxide. We obtained similar results with the use of an electrolyte based on Na₂S·9H₂O.

Na₂SO₃ and Na₂S·9H₂O at room temperature, nanoparticles of hydrozincite must be formed.

3. If an electrolyte is based on Na₂SO₃ is used, then zinc oxide nanoparticles are synthesized.

4. Nanocrystals of zinc sulfide and zinc oxide are synthesized using Na₂S·9H₂O electrolyte at a temperature of 98 °C.

Conclusions

1. The composition of the electrolyte and its temperature significantly affects to the formation of nanocrystals of zinc compounds using the electrolytic method.

2. Using an electrolyte composition prepared from

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Фізичні властивості нанокристалів сполук цинку отриманих електролітичним методом

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Досліджено вплив хімічного складу електроліту і його температури на процес отримання нанокристалів сполук цинку електролітичним методом з використанням цинкових електродів. Проведені рентгеноструктурні дослідження результати яких були використані для визначення складу отриманих зразків та розмірів нанокристалів методами Шеррера та Вільямсона-Холла. Проведено порівняння результатів визначення розмірів нанокристалів обома методами. Обговорюються можливості утворення оксиду цинку, сульфїду цинку і гідроцинкїту при використанні електролітичного методу отримання наночастинок. Показано, що в залежності від виду електроліту отримуються нанокристали оксиду цинку, сульфїду цинку, гідроцинкїту або їх суміші. Досліджено вплив термічного відпалу на отримані зразки.

Ключові слова: оксид цинку, гідроцинкїт, рентгеноструктурні дослідження, розміри наночастинок, метод Шеррера, метод Вільямсона-Холла, термічний відпал.