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Optics

Thermostimulated luminescence and photoluminescence of microcrystalline zinc sulphide ZnS:Cu

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Abstract. In the work, photoluminescence (PL) and thermoluminescence (TL) spectra of a zinc sulfide phosphor series, namely microcrystalline ZnS:Cu obtained by self-propagating high-temperature synthesis (SHS), were obtained and analyzed. Based on the spectral analysis, the character of the influence of annealing parameters on the intensity of the "blue" (B) and "green" (G) PL bands is established. The behaviors of the PL and TL bands are compared and the TL bands in the blue and green spectral regions are identified. The TL mechanism was assumed to be the specific nature of the centers that cause the B- and G-bands of PL.

Keywords: thermoluminescence, photoluminescence, zinc sulfide ZnS:Cu, annealing, SHS method.

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1. Introduction

$A^{II}B^{VI}$ compounds are some of the most studied semiconductor materials. A large number of studies are devoted to $A^{II}B^{VI}$ micro- and nanocrystals. The reasons for this are their manufacturability, *i.e*. relatively easy fabrication by chemical methods (in many cases even from aqueous solutions) as well as high resistance to oxidation, which is especially important for the particles with large specific surface areas. Despite significant amount of research done so far, $A^{II}B^{VI}$ materials still have great potential for further investigations [1].

Phosphors based on a wide-bandgap (~3.7 eV) ZnS semiconductor doped with various impurities are known to exhibit broadband emission from the near ultraviolet to the near infrared region. In particular, copper-activated zinc sulphide (ZnS:Cu) is one of the most effective phosphors in the blue-green spectral region [2–4]. The blue B-Cu band has a maximum at 440…465 nm and the green G-Cu band at 500…530 nm, respectively. These bands manifest themselves depending on the conditions of material synthesis and subsequent technological processing, which impact on the formation of the structure of luminescence centers in ZnS:Cu. It is believed that the G-Cu band is caused by isolated Cu^{2+} ions in substitutional positions of Zn^{2+} ions in the lattice. The nature of the B-Cu band is not definitively established. It is assumed to be due to present complex defects–associates, such as donor-acceptor pairs Cu_i-Cu_{Zn}, which form during Cu_2S phase nucleation [5].

It should be noted that other bands are also present in the PL spectra in addition to the main B- and G-bands. In [6], five PL bands of bulk ZnS:Cu crystals in the UV, blue, green, red, and IR spectral regions were observed. In [7], the PL spectra of ZnS:Cu nanoparticles consisting of four radiation bands were obtained. In [8], three PL peaks were observed in Mn, Cu, and (Mn, Cu) co-doped ZnS, which were attributed to different centers. In many cases, the structure of the glow centers is not definitively elucidated. Hence, the distribution of impurities and the nature of the luminescence centers in ZnS:Cu powders obtained by self-propagating high-temperature synthesis (SHS) and doped during the cultivation process remain poorly studied. Consequently, a question about the existence and influence of defects in such structures on their properties arises.

One of the main methods of indirect examination of defects is thermoluminescence (TL). A number of $A^{II}B^{VI}$ bulk micro- and nanostructures were already studied by TL methods. Among them there were ZnS structures, both pure and doped by various impurities including Cu (see, *e.g*., [9–11]). However, ZnS:Cu nanostructures were insufficiently studied by TL methods so far.

This paper presents the results of the thermo- and photoluminescence study of microcrystalline ZnS:Cu obtained under different heat treatment conditions. Based on the analysis of the spectra, the character of the influence of the heat treatment parameters on the intensity of the "blue" (B) and "green" (G) PL bands is established. The behaviours of the PL and TL bands are

compared and the TL bands corresponding to the photoluminescence in the blue and green spectral regions are identified. Some assumptions are made about the TL mechanisms, *i.e.* about the nature of the centers that determine the PL B- and G-band. The energy spectrum of adhesion centers is determined.

2. Experimental technique and research methods

Thermoluminescence. Thermoluminescence from microcrystalline ZnS:Cu was studied in the temperature range of 80…400 K, the heating rate was 0.2±5% K/s. The samples were excited at the temperature of 77 K by ultra-violet radiation of a deuterium lamp through a UFS-1 filter (excitation band 240…400 nm). The linear increase of the temperature and the data registration were controlled by a computer program. The excitation duration was varied in the range from 5 to 20 min. The integral TL intensity in the photomultiplier sensitivity region (300…800 nm) was measured. Strong phosphorescence was observed after excitation of the sample at 77 K. Therefore, the sample was maintained at the liquid nitrogen temperature for 30…40 min before registering thermoluminescence emission.

Photoluminescence. PL spectra were recorded under the excitation by a 406 nm semiconductor laser on a spectral setup with DFS-24 and MDR-2 monochromators.

Samples. ZnS, a zinc salt of hydrochloric acid, occurs in the form of sphalerite α-ZnS (cubic structure of zinc blende type), and wurtzite β-ZnS (hexagonal structure). The structure of α-ZnS is stable. The materials in this phase is a semiconductor with a band gap of 3.54…3.91 eV[12].

ZnS:Cu samples were grown by the SHS method and doped during the growth [5, 13]. The ratio of the starting materials was as follows: Zn – 0.45 mol, S – 0.56 mol, and CuCl – 0.006 mol. Next, the samples were annealed at $T = 800$ °C for 120 min. The samples Nos 1–6 differed in the time of heating up the furnace to the annealing temperature (800 °C), namely: No 1 – 0 min (*i.e.* without annealing), No. 2 – 15 min, No 3 – 30 min, No 4 – 60 min, No 5 – 120 min, and No 6 – 240 min.

X-ray diffraction studies of the powdered ZnS:Cu showed [5] that the starting powder mainly consisted of an equilibrium cubic phase and to a lesser extent of a non-equilibrium hexagonal phase of ZnS. Annealing further reduced the proportion of the hexagonal phase. At the same time, due to the non-equilibrium of the SHS process, it was possible to introduce impurities at the concentrations significantly exceeding their equilibrium values.

If the impurity concentration exceeds a certain amount, the impurity can act as a quencher due to non-radiative transitions, which leads to a decrease in the luminescence intensity. In other words, a decrease in theluminescence intensity may be associated with quenching due to excessive dopant concentration.

ZnS:Cu samples for measuring TL and PL spectra were prepared in the form of a thick mass using ethyl alcohol, and then applied as thin layers on a substrate and dried. The layer thickness was chosen so to obtain a sufficient TL signal and ensure the minimum temperature gradient over the sample thickness. The substrate was a 15×15 mm 0.2 mm thick. All the plates were polished on both sides to improve the thermal contact, on the one hand, and to ensure smoothness of the surface, on the other hand.

3. Experimental results and discussion

3.1. Thermoluminescence

Analysis of the TL intensity versus temperature curves enables obtaining the trap energies. The partial glowing technique [14, 15] as well as decomposition of the TL curves into elementary peaks were used [16, 17]. The activation energy was one of the decomposition parameters.

At the initial stage of the thermal glowing process, the TL intensity exponentially increases with increasing temperature according to the following equation:

$$I = \text{const} \cdot \exp\left(-\frac{E}{kT}\right), \qquad (1)$$

where E is the trap activation energy.

Eq. (1) describes well the low-temperature part of the TL curve corresponding to a small portion of charge carriers released from the traps. An important condition for applicability of the method is approximately constant concentration of charge carriers on the traps n (lightsum). The change in the lightsum during the registration of the growth curve Δn was defined as the area under this curve. The total lightsum n is proportional to the total area under the peak. We used the condition $\Delta n/n < 0.05$.

Normalized TL curves for the samples Nos 1 to 6 are shown in Fig. 1. As can be seen from this figure, the TL shows wide, intense and complex bands.

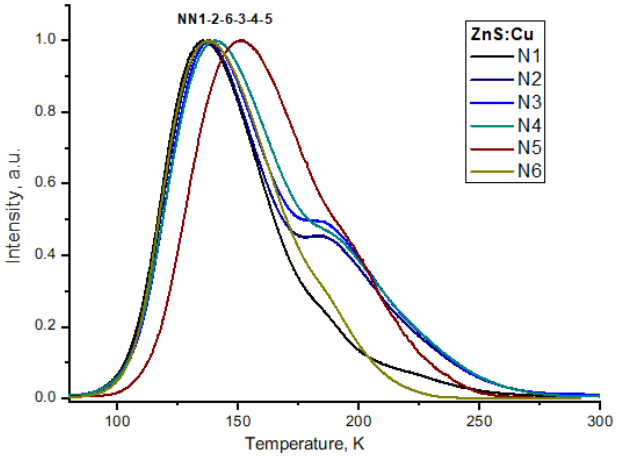


Fig. 1. Normalized curves of the integrated intensity of thermo-luminescence of the Nos 1–6 ZnS:Cu samples measured under UV-excitation (200…240 nm) at 80 K. (Color online)

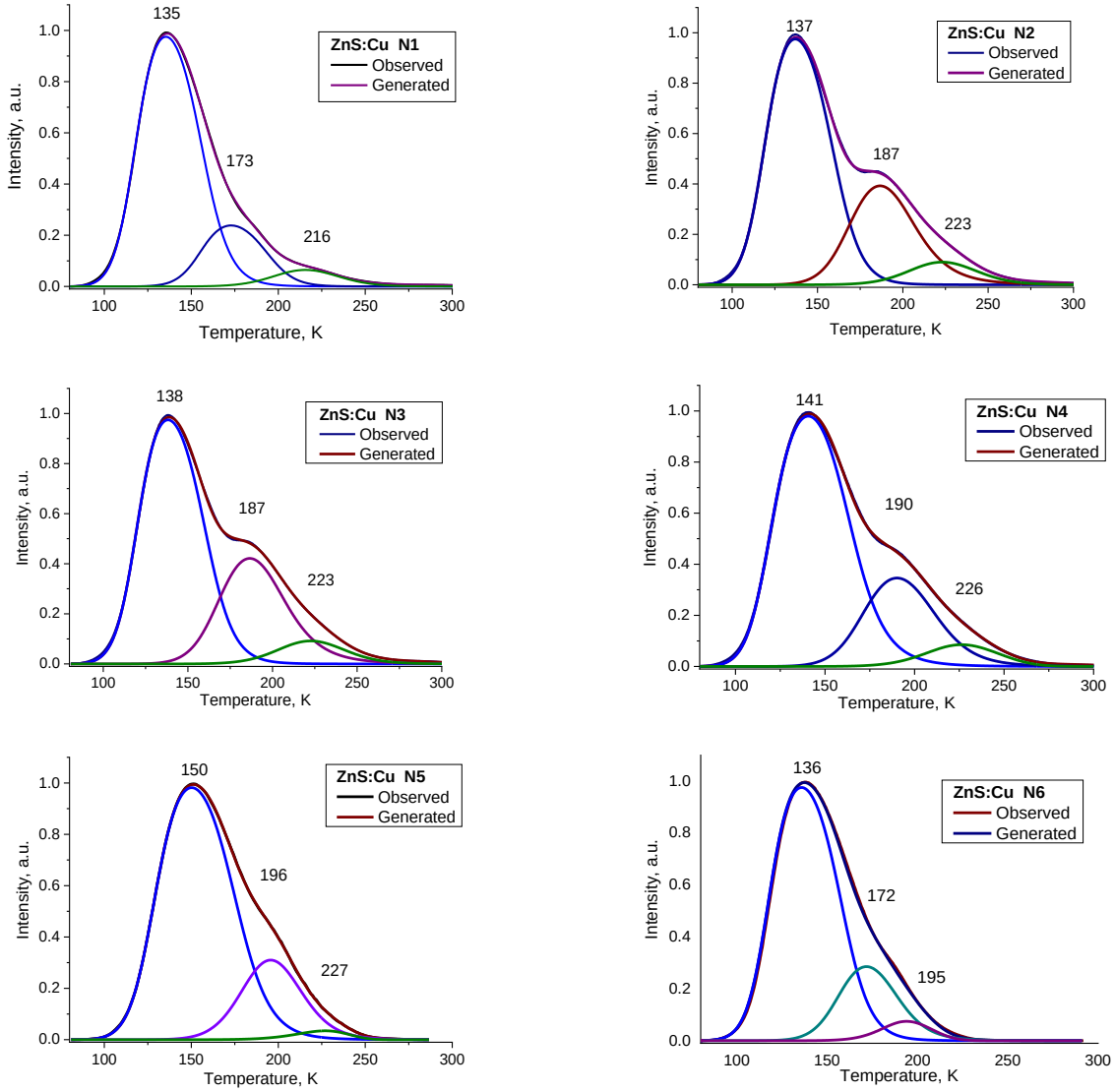


Fig. 2. Decomposition of the TL curves of the Nos 1–6 ZnS:Cu samples into elementary peaks by expressions (2) and (3). The observed and generated by decomposition curves are practically coincident. (Color online)

To determine the activation energies of the single glow TL peaks, as well as for comparative analysis with the PL curves of the respective samples, the TL curves should be decomposed into elementary peaks according to formulas (2), (3) for the first and second order kinetics, respectively [18]:

$$I(T) = -\frac{dn}{dt} = $$

$$= n_0 p_0 \exp\left(-\frac{E}{kT}\right)\exp\left[-\frac{p_0}{\beta}\left(\int_{T_0}^{T}\exp\left(-\frac{E}{kT}\right)dT\right)\right], \qquad (2)$$

$$I(T) = -\frac{dn}{dt} = n_0 p_0 \exp\left(-\frac{E}{kT}\right)\left[1+\frac{p_0}{\beta}\left(\int_{T_0}^{T}\exp\left(-\frac{E}{kT}\right)dT\right)\right]^{-2}. \qquad (3)$$

Here, n_0 is the initial concentration of carriers on the traps (lightsum), p_0 is the frequency factor, β is the heating rate, E is the energy of thermal activation of the traps, and n_0, p_0 and E are the decomposition parameters corresponding to the minimum standard deviation (SD) of the analytical curve from the experimental one, respectively.

Using the decomposition into elementary peaks according to (2) and (3) (see Fig. 2), the trap activation energy values for the Nos 1–6 ZnS:Cu samples were determined (see Table).

Three peaks with the maximum temperatures of ~140, ~180 and ~220 K are observed on the TL curves for all the Nos 1–6 ZnS:Cu samples. Depending on the sample number, the positions of the TL peaks and their intensities slightly vary. The intensities of the high-temperature peaks gradually increase for the sample Nos 1 to 5, while for the sample No 6, the intensity of the

Sample (No) T_m, K	No 1 135, 173, 216	No 2 137, 187, 223	No 3 138, 187, 223	No 4 141, 190, 226	No 5 150, 196, 227	No 6 136, 176, 215
peak	E_a, eV p_0	E_a, eV p_0	E_a, eV p_0	E_a, eV p_0	E_a, eV p_0	E_a, eV p_0
1 (~140 K) frac	0.21	0.18	0.22	0.20	0.21	0.23
calc	0.18 $1.1\cdot10^5$	0.18 $6.2\cdot10^4$	0.18 $6.5\cdot10^4$	0.18 $4.7\cdot10^4$	0.19 $3.2\cdot10^4$	0.19 $1.2\cdot10^5$
2 (~180 K) frac	0.23	0.23	0.27	0.24	0.27	0.26
calc	0.29 $4.1\cdot10^6$	0.26 $2.3\cdot10^5$	0.26 $1.6\cdot10^5$	0.26 $1.2\cdot10^5$	0.26 $7.5\cdot10^4$	0.26 $7.6\cdot10^5$
3 (~220 K) frac	–	–	–	–	–	–
calc	0.29 $6.3\cdot10^4$	0.29 $3.5\cdot10^4$	0.29 $3.6\cdot10^4$	0.28 $2.1\cdot10^4$	0.28 $2.5\cdot10^4$	0.31 $1.5\cdot10^6$

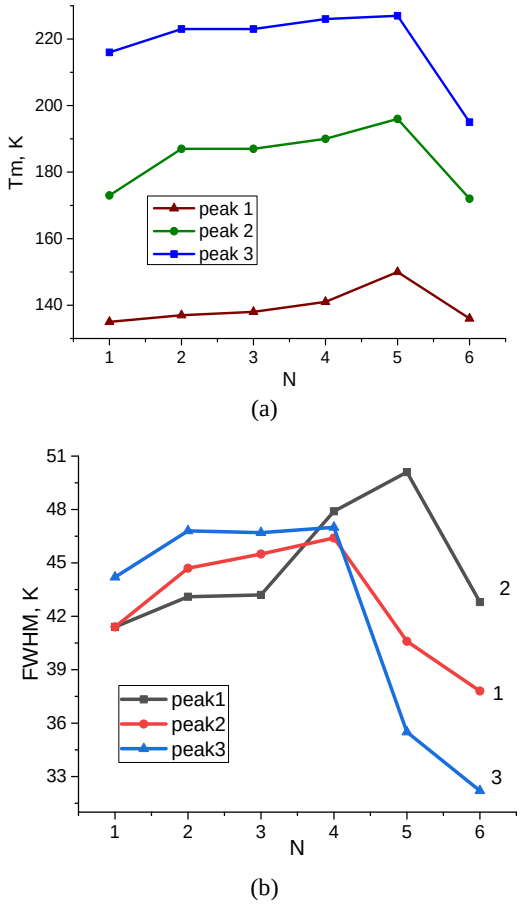


Fig. 3. Change of the maximum positions (a) and the half-widths (b) of the TL peaks (~140, 180 and 220 K) with the sample number.

peak corresponding to ~180 K decreases, and the peak corresponding to ~220 K is practically absent. It should be noted that the best distribution of the TL curves is formed by kinetic peaks of the order of ~1.5…1.6, but some curves show quadratic kinetics (peak No 2 for the sample Nos 2 and 3) or close to it. The latter kinetics is explained by the high probability of charge recapture, which in turn results from the significant concentration of impurities in the original samples. Close to linear kinetics was observed only for the weak No 3 peak in some samples.

The change in the maximum positions and the half-widths of the peaks with the sample number (actually, the furnace heating time to the annealing temperature) is presented in Fig. 3. It can be seen from this figure that the positions of the maxima shift fairly evenly towards higher temperatures for the sample Nos 1–5 (*i.e.* at increasing the heating time to the annealing temperature), and decrease relatively sharply for the No. 6 sample (maximum heating time of 240 min), see Fig. 3a. In their turn, the half-widths of all three peaks decrease rather sharply for the sample No 6, but their monotonic growth is not observed for the sample Nos 1–5 (see Fig. 3b).

The values of the trap activation energies were also found from the analysis of the TL intensity growth curves (initial slope or fractional glowing method) for the TL peaks corresponding to ~140 and ~180 K. The high-temperature TL peak has low intensity. As an example, the data for the sample Nos. 2 and 6 are shown in Fig. 4. The statistics of the calculated values of the trap activation energy for the sample No. 3 are presented in Fig. 5. The maximum position of the peak on the statistical diagram corresponds to the calculated value of the trap energy, and the half-width reflects the experimental error. The frequency factor p_0 for the traps in ZnS:Cu was also found. Its values are in the range of 10^4…10^6. Summary data on the activation energies and frequency factors are presented in Table.

3.2. Photoluminescence

Fig. 6 shows the normalized PL spectra of the studied samples. As can be seen from this figure, there is a certain non-monotonous shift of the maximum position in the short-wave direction for different samples (corresponding to the increase of the heating time of the furnace). The half-width of the bands change little at this.

To analyze the changes in the elementary peaks of the PL spectra, these peaks should be extracted by decomposition of the complex PL bands. It can be seen from the decomposition results (see Fig. 6, sample No 6) that the PL blue band in the studied samples does not appear. There is only a “blue”-shift of the short-wave component of the wide “green” PL band. The spectrum of the “green” band is quite wide. It depends on the sample, *i.e.* the heating time, varying from “blue-green” to “green-yellow”. The third, longer-wavelength band, may be also caused by uncontrolled Mn impurity, in addition to Cu ions [5]. Hence, the TL spectra of the studied samples are determined by the defects that form the wide “green” band of PL.

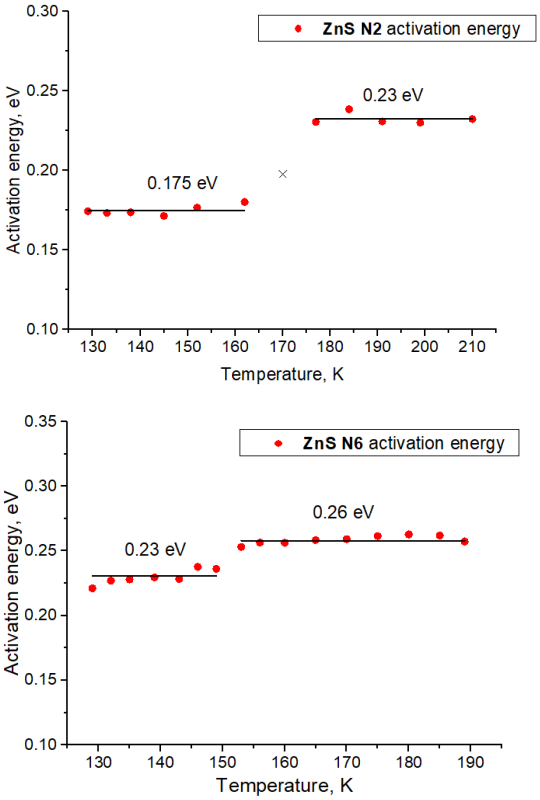


Fig. 4. Values of activation energy for samples Nos 2 and 6 determined by the fractional glowing method.

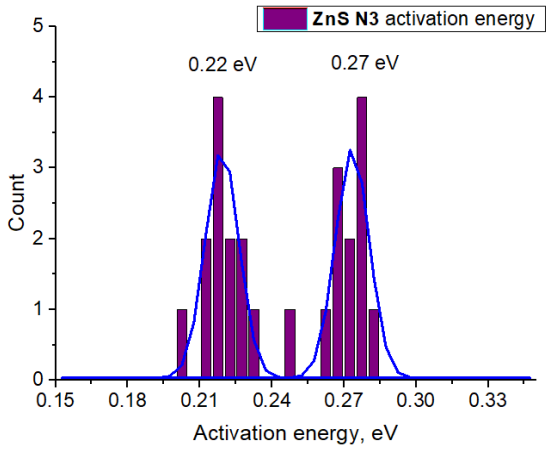


Fig. 5. Statistics of the calculated values of activation energy, sample No 3.

Despite some inaccuracy of the decomposition of the PL curve into elementary Gaussian components, certain conclusions may be drawn. To achieve the highest intensity of the "green" PL band, the heating time to the annealing temperature should be approximately 120 min. At this, the discussed band becomes slightly "blue"-shifted because of the increase of intensity of the short-wave peak. For maximum glow in the green-yellow region, the heating time should be relatively short – about 30 min (see inset in Fig. 6).

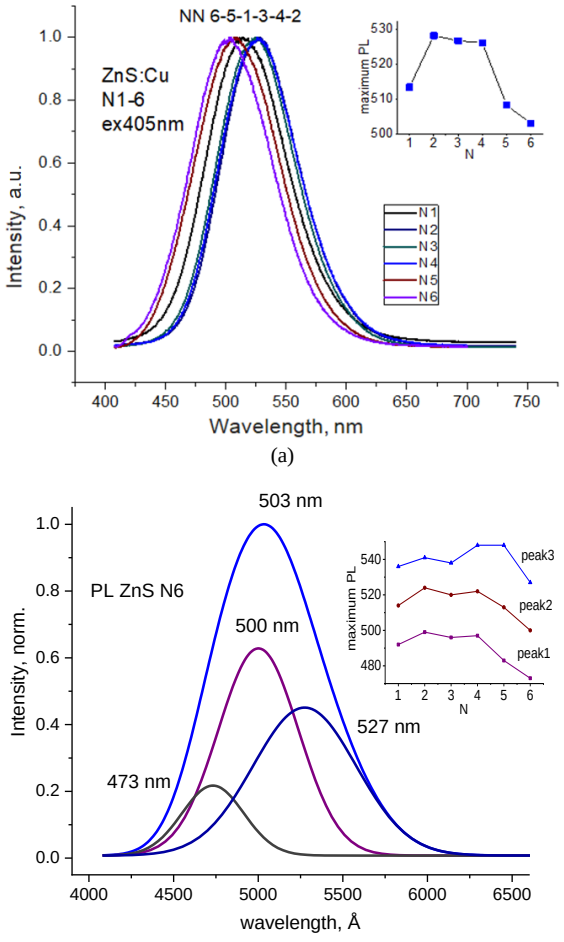
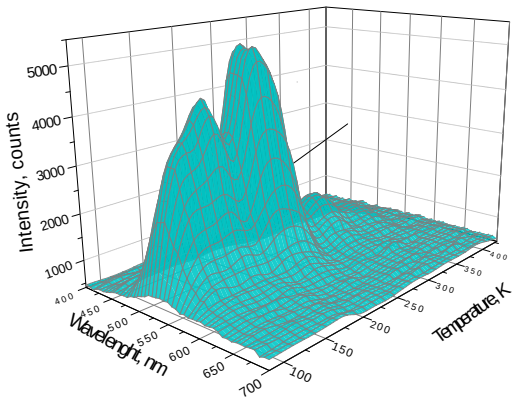


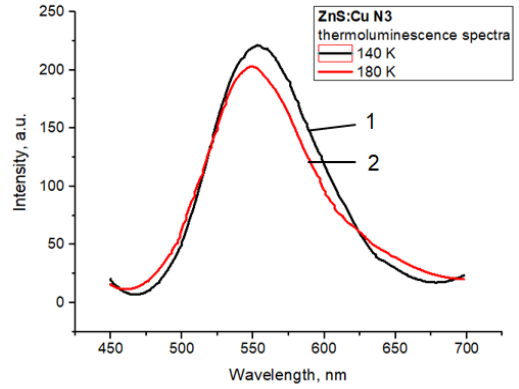
Fig. 6. (a) normalized PL spectra of ZnS:Cu measured under 405 nm excitation (the inset shows the change of the maximum position of the integrated PL band with the sample number), and (b) an example of the decomposition (sample No 6) of the PL band into Gaussian components (the inset shows the change of the maximum positions of the PL peaks with the sample number).

It is difficult to make unambiguous conclusions about the nature of TL and the correspondence of the TL and PL elementary peaks by comparing the changes in the TL and PL peak parameters with the sample number. Therefore, additional experiments were performed to elucidate the spectral composition of the thermo-luminescence curves. Namely, the evolution of the TL spectra in the temperature range of 100…450 K was studied, *i.e.* a number of luminescence spectra excited at 77 K were obtained at different temperatures (see Fig. 7a).

Particular attention was paid to the registration of the spectra at the temperatures corresponding to the maxima of the TL curves, *i.e.* at ~140 and ~180 K (see Fig. 7b). Only one band with a maximum close to 550 nm was observed in the TL spectra at both 140 K and 180 K. Therefore, the TL peaks at ~140 and ~180 K correspond to the wide "green" PL band caused by isolated Cu ions in the positions of Zn ions in the lattice, as previously reported in [3]. The PL spectrum also contained two elementary short-wave bands.



(a)



(b)

Fig. 7. (a) ZnS:Cu PL-TL spectra measured under 405 nm excitation and (b) PL spectra measured at the temperatures of 140 (*1*) and 180 K (*2*).

Thermoluminescence in semiconductor nanoparticles under UV excitation is known to be largely associated with surface defects, because UV radiation cannot penetrate deeply in the material and generate a significant number of bulk lattice defects. Moreover, with decreasing nanoparticle size, the surface/volume ratio, the number of surface ions and surface states, and the relative number of captured charge carriers by surface traps (lightsum) increase. Furthermore, when the particle size decreases, the wave functions of electrons and holes overlap more, which makes recombination more efficient. All these factors lead to a relative increase in the TL intensity [19]. However, the question of the extent to which surface or bulk defects are involved in the TL and PL processes is rather complicated [20], thus requiring a separate study to get the answer.

4. Conclusions

Thermo- and photoluminescence spectra of microcrystalline copper-doped zinc sulphide samples, ZnS:Cu, undergoneheat treatments in the temperature range of 80…450 K were obtained.

The spectrum of the trap activation energies in the studied structures is calculated. The thermal activation energies of the adhesion centers in all the samples are in the range of 0.18…0.30 eV.

Influence of heat treatment on the glow region of the ZnS:Cu phosphors is analyzed. Analysis of the TL and PL spectra revealed that heat treatment under the conditions used here leads to the formation of the centers responsible for the glow in the "green" spectral region with a slight extension outside it (Cu ions in the positions of Zn ions). The centers responsible for the "blue" luminescence band are practically not formed as they do not manifest themselves in the TL spectra. Preliminary conclusions are made regarding the annealing conditions to achieve more efficient glow of the elementary components (*i.e.* colour variants) of the wide green PL band.

5. Acknowledgements

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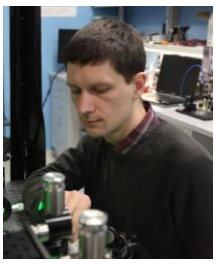

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Authors' contributions

S.Yu. Kutovyy: data handling, writing, review and editing.
O.P. Stanovyi: experiment, data handling, review.

Термостимулювана люмінесценція та фотолюмінесценція мікрокристалічного сульфіду цинку ZnS:Cu

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Анотація. У роботі отримано та проаналізовано спектри фотолюмінесценції (ФЛ) та термолюмінесценції (ТЛ) ряду люмінофорів на основі сульфіду цинку, а саме мікрокристалічного ZnS:Cu, отриманого методом самопоширюваного високотемпературного синтезу (СВС). На основі аналізу спектрів було встановлено, як параметри відпалу впливають на інтенсивність «зелених» (G) та «блакитних» (B) смуг фотолюмінесценції. Проведено порівняння поведінки смуг ФЛ та ТЛ, встановлено, які смуги ТЛ зумовлюють свічення в синій та зеленій області спектра. Зроблено припущення про механізми ТЛ, тобто про природу центрів, що зумовлюють свічення G- та B-смуг ФЛ.

Ключові слова: термолюмінесценція, фотолюмінесценція, нанопорошки ZnS:Cu, відпал, метод СВС.