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PHOTOLUMINESCENCE IN ZINC OXIDE AND ZINC SELENIDE MICROPOWDERS UNDER PULSED-PERIODIC LASER EXCITATION

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Abstract. This study analysed the luminescence spectra of zinc oxide (ZnO) and zinc selenide (ZnSe) micropowders obtained by single- and two-photon excitation with pulsed laser radiation on copper vapour. The mechanisms of photoluminescence spectrum formation in the studied samples were established. It was found that in zinc oxide and selenide micropowders, a superluminescence effect is observed at a laser radiation intensity of 10^6-10^7 W/cm^2. It was found that with an increase in the intensity of the exciting radiation in the studied samples, the maximum of the luminescence spectrum band shifts to 5 nm.

Keywords: zinc oxide, zinc selenide, micropowder, laser, luminescence, secondary radiation, spectrum, pulse.

Impuils-davriyli lazer nuri ta’sirida rux oksidi va rux selenid mikrokukunlarida uyg‘otilgan fotolyuminesensiya

Annotatsiya. Ishda mis bug‘larida impulsli-davriy lazer nurlanishida rux oksidi (ZnO) va rux selenid (ZnSe) mikrokukunlarining bir- va ikki fotonli uyg‘otilgan lyuminesensiya spektrlari o‘rganilgan. O‘rganilgan namunalarning fotolyuminesensiya spektrlarining shakllanish mexanizmlari aniqlandi. Rux oksidi va selenidi mikrokukunlarida 10^6-10^7 Vt/sm^2 lazer nurlanishi intensivligida superlyuminesensiya effekti kuzatilishi aniqlandi. Uyg‘otuvchi nurlanish intensivligining ortishi bilan lyuminesensiya intensivligining ortishi kuzatildi. O‘rganilgan namunalarda uyg‘otuvchi nurlanish intensivligi oshirilganda lyuminesensiya spektri polosasi maksimumining 5 nm gacha siljishi aniqlandi.

Kalit so‘zlar: rux oksidi, rux selenid, mikrokukun, lazer, lyuminesensiya, ikkilamchi nurlanish, spektr, impuls.

Фотолюминесценция в микропорошках оксида цинка и селенида цинка при импульсно-периодическом лазерном возбуждении

Аннотация. В данном исследовании анализировались спектры люминесценции микропорошков оксида цинка (ZnO) и селенида цинка (ZnSe), полученные при одно- и двухфотонном возбуждении импульсным лазерным излучением на парах меди. Установлены механизмы формирования спектров фотолюминесценции изученных образцов. Установлено, что в микропорошках оксида цинка и селенида наблюдается эффект суперлюминесценции при интенсивности лазерного излучения 10^6-10^7 Вт/см^2. Обнаружено, что при увеличении интенсивности возбуждающего излучения в исследуемых образцах максимум

полосы спектра люминесценции смещается до 5 нм.

Ключевые слова: оксид цинка, селенид цинка, микропорошок, лазер, люминесценция, вторичное излучение, спектр, импульс.

Introduction. Zinc oxide (ZnO) is a material that is attracting interest due to its excellent electrical, optical, piezoelectric and other properties. ZnO semiconductors are widely used in ultraviolet detectors, emission devices, thin-film transistors and surface acoustic wave systems. The large binding energy of the exciton (60 meV) allows for the emission of intense ultraviolet luminescence in ZnO due to the radiative recombination of excitons at room temperature and above (up to 550 K) [1].

Zinc selenide (ZnSe) crystals are wide-bandgap semiconductors with an $A^{II}B^{VI}$ crystal structure. These crystals are distinguished by their relatively high mobility of up to 700 $cm^2/(V \cdot s)$ at room temperature [2]. The intense luminescence in the short-wavelength region of the visible spectrum makes ZnSe a promising material for the manufacture of blue-light-emitting devices [3,4].

The optical and luminescent properties of these objects have been studied in numerous works. However, the mechanisms responsible for the generation of secondary radiation in fine and ultra-dispersed media have not yet been sufficiently investigated. Furthermore, when powdery materials are irradiated with high-intensity pulses from pulsed lasers, their initial characteristics change significantly: the medium heats up intensely and even photodestruction of the material may occur [5–8].

Therefore, the aim of this work is to investigate the formation mechanisms of the photoluminescence spectra of ZnO and ZnSe microstructures during pulsed-dye laser excitation.

Experimental. For the study, 99% pure ZnO and ZnSe ready-made white powders from Sigma-Aldrich were used. The optical method, detailed in [5–8], was employed to excite and record the secondary radiation (SR) spectra. A copper-vapour laser line at λ = 510.6 nm was used as the excitation source, and the laser's yellow line at λ = 578.2 nm was filtered out. The average power of the laser radiation is 10 W. The radiation is generated in a pulsed mode with a high repetition rate (10^4 Hz) and short (20 ns) generation pulses, with a peak power of 10^5 W. Radiation is generated in a pulsed mode at a high repetition rate (10^4 Hz) with short (20 ns) generation pulses, producing a peak power of 10^5 W. Calibrated neutral density filters (NF) were used to analyse the intensity of the two-photon-excited luminescence (TPEL) spectra. Microparticle samples were densely packed between the flat, parallel quartz windows of the cuvette. Quartz cuvettes for spectrophotometry were made from “KU” grade quartz glass. The layer thickness was 1 mm. The resolving power for recording spectra in the range of the measured wavelengths was 0.1 nm.

Measurements were carried out at room temperature. For studying single-photon-excited luminescence (SPEL) spectra, the second optical harmonic (SOG) of the green line (510.6 nm) of laser generation in copper vapour corresponding to a wavelength of λ = 255.3 nm. The high power of the laser radiation allows for the effective doubling and summing of the laser generation line frequencies using nonlinear optical crystals (BaB_2O_4). The radiation from the active element of the laser, operating in a unstable resonator mode, was focused in BaB_2O_4 using a long-focal-length lens. The visible-range laser radiation was eliminated by an absorption filter placed after the BaB_2O_4.

Results and Discussion. Figure 1a shows the SPEL spectra of ZnO microcrystals obtained at various intensities of the excitation radiation (λ_{exc} = 255.3 nm). At low excitation intensity, the SPEL spectrum exhibits a maximum at around 386 nm (curves 1 and 2). In semiconductor materials with low concentrations of impurities and defects, excitation at high intensities leads to radiative recombination of the free excitons. saturation of the excitation intensity occurs, and in this context, the following aspect of the large-scale annihilation of free excitons is of particular interest: the possibility of obtaining stimulated emission arises [9]. It is well known that free excitons do not undergo an inversion distribution, whereas in photon and phonon emission by excitons inversion arises automatically. When the excitation intensity exceeds 1.5×10^6 W/cm^2 (curves 3–5), a narrowing of the exciton luminescence line is observed, and its intensity increases non-linearly with further increases in

the excitation intensity. Furthermore, the SPEL band narrows and the peak of this spectral intensity shifts into the longer-wavelength region by up to 4 nm.

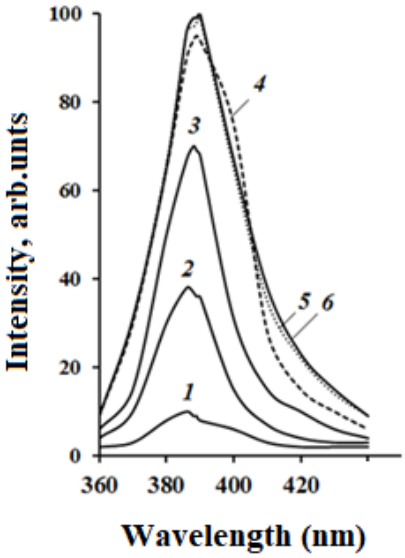

Figure 1a. SPEL spectra of ZnO micropowder (d_{mean} = 2 μm) obtained at different intensities of stimulating radiation: (1) - I_{exc} = 5; (2) - I_{exc} = 11; (3) - I_{exc} = 15; (4) - I_{exc} = 18; (5) - I_{exc} = 21; (6) - I_{exc} = 24. (I_{exc} ~ 10^6 W/cm^2).

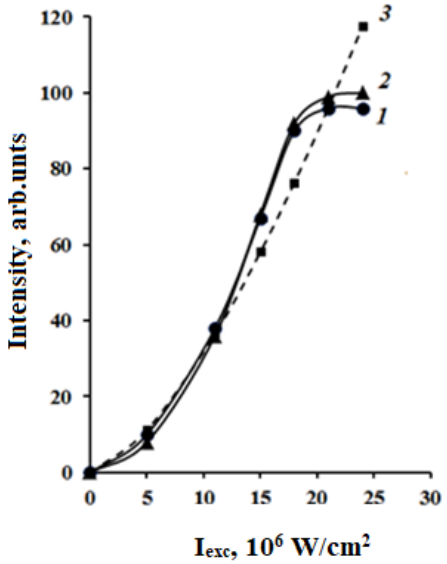

Figure 1b. The dependence of the SPEL intensity (I) on the excitation radiation intensity (I_{exc}) for ZnO microcrystals (d_{mean} = 2 μm). The line (1) corresponds to a wavelength of 386 nm; (2) – λ= 390 nm. (3) – the theoretical dependence in accordance with the law $I \approx I_{exc}^{1.5}$.

Under our experimental conditions (I_{exc} ≈ 10^6 W/cm^2), a power-law dependence with an exponent α≈1.5 is observed for the ZnO microstructures (Figure 1a, 3-line), which corresponds to the α = 1.5 ± 0.2 value found for bulk ZnO in [10]. At high intensities, the growth of SPEL intensity slows down, which may be caused both by heat effects and by the formation of defects through photolysis and thermodestruction. A similar trend was observed for monocrystalline ZnO and quantum dots in [11]. The absence of visible SPEL in our ZnO samples indicates a low concentration or absence of oxygen defects [12,13] and the high purity of the ZnO powders studied.

Figure 2 shows the SPEL spectra of ZnSe microcrystals (d = 2 μm) obtained at various excitation-ray power densities. The SPEL band in the 460–490 nm range under investigation exhibits the following characteristics. Firstly, we note that the 476–480 nm emission maximum is located closer to the E_g forbidden-band width; secondly, the position of the maximum depends on the excitation level, shifting towards lower energies as I_{exc} increases. Thirdly, the dependence of B-band radiation intensity is of $I \approx I_{exc}^{1.4}$ degree-law character. All the above-mentioned features are characteristic of the annihilation of excitons in inelastic scattering to free charge carriers in the 460–480 nm region [14,15]. In this range lies the intrinsic absorption edge of ZnSe, where the absorption coefficient can reach up to 10^4 cm^{-1}, and it varies sharply with even slight changes in wavelength within this spectral region. [16] shows that the spectrum of the main ZnSe band at 77 K (2.789 eV) is of a complex character: the band corresponds to 2.793 (E_x), into three main components, which are separated into the additional low-energy region of the spectrum and have maxima at 2.705 (A), 2.676 (A^{LO}), 2.645 (A^{2LO}) and 2.775 eV (I_1), and at 2.745 eV (I^{LO}). In the additional low-energy region of the spectrum, a series of maxima is observed at 2.705 (A), 2.676 (A^{LO}), 2.645 (A^{2LO}) and 2.611 eV (A^{3LO}).

The energies of the free excitons in ZnSe are E_x = 2.812 eV and A = 2.690 eV, corresponding to wavelengths of 440.9 and 460.9 nm. In these spectra, maxima arise from processes in which excitons split into a photon and one or more longitudinal optical phonons. The intensity maximum (λ_{max} = 476 nm) in the SPEL spectrum obtained in our work corresponds to a three-phonon transition (A^{3LO}).

A similar effect was also observed in the study of 0.4 μm-thick ZnSe epitaxial films [17]. Thus, the obtained results can be explained by the transition from spontaneous luminescence to stimulated luminescence, i.e. superluminescence, at high annealing intensities.

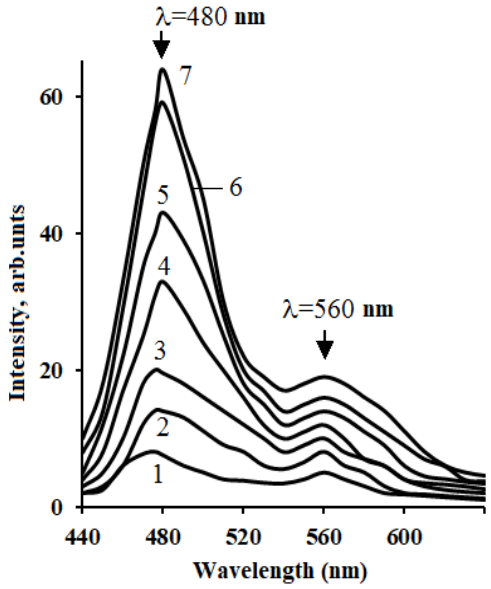

Figure 2. SPEL spectra of ZnSe micropowder (d_{mean} = 2 μm) obtained at various intensities of excitation radiation: (1) – I_{exc} = 5; (2) – I_{exc} = 7; (3) – I_{exc} = 9; (4) – I_{exc} = 11; (5) – I_{exc} = 13; (6) – I_{exc} = 15; (7) – I_{exc} = 18; (I_{exc} ~ 10^6 W/cm^2).

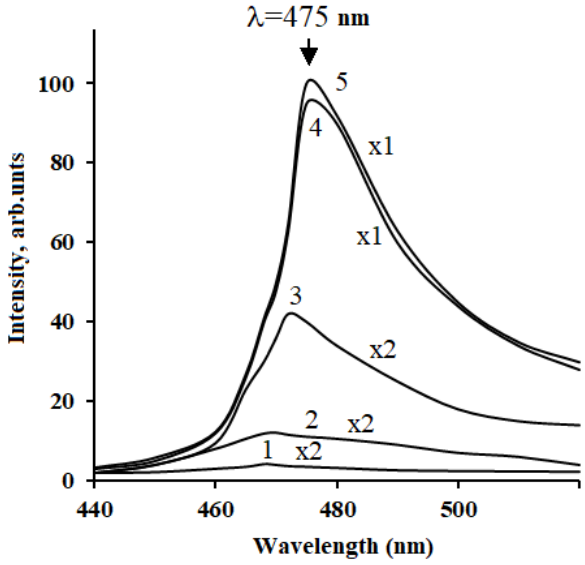

Figure 3. TPEL spectra of ZnSe micropowder (d_{mean} = 5 μm) obtained at different intensities of excitation radiation: (1) – I_{exc} =0.8; (2) – I_{exc} = 1.4; (3) - I_{exc} = 2; (4) - I_{exc} = 2.6; (5) - I_{exc} = 3.2 (I_{exc} ~ 10^6 W/cm^2).

Figure 3 shows the TPEL spectra of ZnSe microcrystals under copper vapour at various intensities of the green laser light (λ_{exc} = 510.6 nm). As can be seen from this figure, the TPEL spectrum we observe has a maximum in the 440–460 nm region. The emission maximum at 470–480 nm lies closer to the E_g forbidden-band width. The results clearly show that as the excitation light intensity increases, the peak shifts towards lower energies. The position of the maximum depends on the excitation intensity level. The 460–480 nm region is characteristic of the annihilation of excitons in non-elastic scattering with free charge carriers [14,15]. In this case, the TPEL maximum has shifted towards shorter wavelengths by 5 nm relative to the SPEL maximum (see Figure 2). Furthermore, the SPEL and TPEL spectra differ from each other in intensity as well.

Conclusion. Thus, in our experiments, the superluminescence effect is observed in ZnO and ZnSe micro-powders at laser excted intensities of 10^6-10^7 W/cm^2. With increasing excitation radiation intensity, a shift of the band maximum by 4–5 nm was observed in both samples. The experiments showed that laser effects in ZnO and ZnSe micro-powders are clearly observed at room temperature. The high radiation intensity of the studied objects opens up new possibilities for their widespread application as effective frequency converters in the visible and near-ultraviolet regions.

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