

Study of the photoluminescence spectra observed from different points on the surface of a polycrystalline CVD ZnSe sample with an excess of selenium and oxygen with a change in the wavelength of the exciting light

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We studied photoluminescence (PL) spectra in the wavelength range of 458–565 nm, as well as excitation spectra with maxima at $\lambda = 505$, 529, 542, and 565 nm, obtained using a xenon lamp (Cary Eclipse spectrofluorimeter) from polished and unpolished surfaces of the same previously undoped polycrystalline CVD (chemical vapor deposition) ZnSe sample with excess selenium and oxygen (ZnSe(O)Se). It has been shown that the spectral positions of all the maxima observed from the polished and unpolished surface of the sample are the same, and the intensity and structure of the excitation spectrum for the maxima at $\lambda = 529$ nm and 542 nm differ slightly, but are different for $\lambda = 505$ nm and 565 nm. We believe that the differences may be related to the uneven distribution of oxygen and background impurities in the sample under study, where oxygen complexes with their intrinsic point defects play an important role in the formation of emission centers of polycrystalline CVD ZnSe.

Keywords: polycrystalline CVD (chemical vapor deposition) ZnSe, photoluminescence spectra, additional absorption edge, point defects, band anti-crossing theory.

Дослідження спектрів фотолюмінесценції, що спостерігаються з різних точок поверхні полікристалічного зразка CVD ZnSe з надлишком селену та кисню при зміні довжини хвилі збуджуючого світла. *І.І.Аббасов, М.А.Мусаєв, Дж.І.Гусейнов, Л.А.Алієва, С.К.Нурієва, А.В.Шаріфова, Р.Ш.Рахімов, Дж.І.Ісмаїлов, Н.Н.Хашімова*

Досліджувалися спектри фотолюмінесценції (ФЛ) в діапазоні довжин хвиль 458–565 нм, а також спектри збудження з максимумами при $\lambda = 505$, 529, 542 і 565 нм, отримані за допомогою ксенонової лампи (спектрофлуориметр Cary Eclipse) з полірованої та неполірованої поверхні того самого раніше нелегованого полікристалічного CVD (хімічного осадження з газової фази) зразка ZnSe з надлишком селену та кисню (ZnSe(O)Se). Показано, що спектральні положення всіх максимумів, які спостерігаються на полірованій і неполірованій поверхні зразка, однакові, а інтенсивність і структура спектра збудження для максимумів при $\lambda = 529$ нм і 542 нм незначно відрізняються, але відрізняються для $\lambda = 505$ нм і 565 нм. На нашу думку, відмінності можуть бути пов'язані з нерівномірним розподілом кисню та фонових домішок у досліджуваному зразку, де комплекси кисню з властивими їм точковими дефектами відіграють важливу роль у формуванні емісійних центрів полікристалічного CVD ZnSe.

Introduction

In the photoluminescence (PL) spectrum of polycrystalline CVD (chemical vapor deposition) ZnSe, along with edge luminescence, long-wavelength luminescence bands have been observed, the study of the features of which is necessary for modern optoelectronics as promising materials operating in the blue-green and red spectral regions [1–4]. However, the data obtained on the nature of the green luminescence of zinc selenide are sometimes contradictory and different, and this may be due to the fact that the intensity and wavelength of the excitation light, temperature, crystal purity, and synthesis conditions affect the green luminescence spectrum. Furthermore, the role of oxygen, as well as the effect of surface treatment of the studied samples in luminescent processes on green luminescence, has recently been intensively studied [5–12]. The authors of [2, 5–10] have shown that the intensity of the green band depends on the oxygen content; and based on the band anticrossing (BAC) model, it was assumed that in crystals with sp^3 under the action of an isoelectronic impurity (including in ZnSe under the action of oxygen) the band gap is bent. In [11–14], PL was studied from the polished and unpolished sample surfaces (the same sample and the same excitation), and some difference was observed in the spectra, which was considered to be a consequence of the uneven distribution of background impurities (they can be called uncontrolled impurities) over the depth of the sample. Luminescence processes in samples with a slight excess of selenium with an uneven distribution of oxygen and background impurities (including copper), as well as their complexes with IPD, have been studied very little [2, 8, 10] and require more extensive study. Therefore, the aim of

this paper is a more detailed study of the PL spectra in the wavelength range of 458–565 nm, as well as the excitation spectra with maxima at $\lambda = 505, 529, 542$ and 565 nm, observed from a polished and unpolished surface of the same previously undoped polycrystalline CVD ZnSe sample upon excitation with a xenon lamp.

2. Experimental

Polycrystalline ZnSe samples with a thickness of 3 mm (Fig. 1a) have been obtained by chemical vapor deposition (CVD) at the Institute of Chemistry of High-Purity Substances of the Russian Academy of Sciences (Nizhny Novgorod) [15]. Crystal growth from the vapor phase occurs at a lower temperature compared to the melt technology. This contributes to a decrease in the concentration of bulk defects, and also makes it possible to reduce the growing crystal contamination by the material of the ampoule. The concentration of background impurities for the polycrystalline CVD ZnSe sample under study was determined by two methods: atomic emission spectroscopy and laser mass spectrometry. The total impurity content is $< 10^{16}–10^{17} \text{ cm}^{-3}$ ($\text{Cu} \sim 10^{16} \text{ cm}^{-3}$). Oxygen concentration has been controlled by chemical gas chromatographic analysis: $\text{O} \sim 10^{18}–10^{20} \text{ cm}^{-3}$ [8]. The phase composition and distribution of chemical elements on the surface of the sample was determined by the method of quantitative X-ray microanalysis and the results showed the homogeneity of the surface and a change in stoichiometry within the homogeneity region towards a slight excess of selenium [16]. The authors of [2, 8, 10] showed that with an excess of selenium, the background impurity of copper contributes to the dissolution of oxygen, since the isoelectronic oxygen impurity O_{Se}^* , which carries

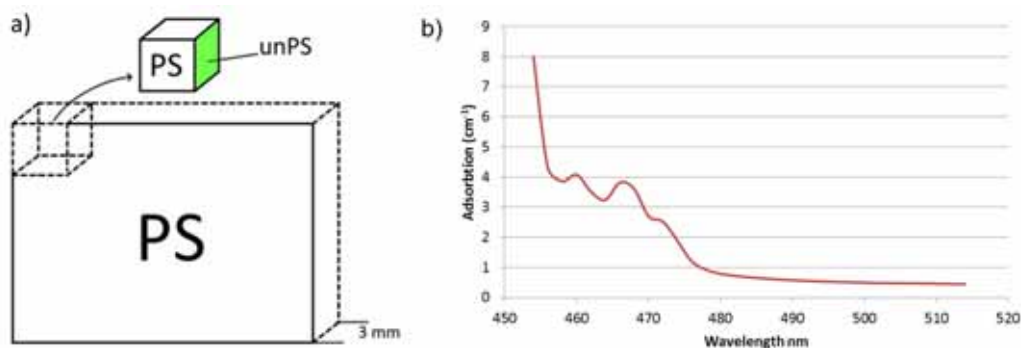


Fig. 1. a) Polished (PS) and unpolished (unPS) surface of the polycrystalline CVD ZnSe sample, b) UV-visible absorption spectra of polycrystalline CVD ZnSe at $T = 300 \text{ K}$ [17].

an effective negative charge, enters the ZnSe lattice, trapping copper in the complexes $\{O_{Se}^* - Cu^+\}$.

The entire surface of the sample was polished at the same Institute (we abbreviate it as polished surface (PS)). We divided this sample into small pieces (parts) (Fig. 1a) without changing the thickness (4 mm×4 mm×3 mm). The cut out part is shown in green; this part of the sample surface is abbreviated unPS (unpolished surface).

The luminescent properties of polycrystalline CVD ZnSe were studied using a Cary Eclipse spectrofluorometer manufactured by Varian, and all measurements were carried out at $T = 300$ K. The radiation source was a xenon lamp with an extended service life, operating in the pulsed mode with a pulse width of 2 μ s and power of 75 W. The monochromators of the spectrofluorimeter have diffraction gratings with parameters of 1200 lines/mm and software that makes it possible to determine the wavelength down to 0.01 nm. Both monochromators have high-speed scanning capabilities. The software of the device allows you to select various measurement modes and simultaneously control the working elements. The emission spectra were obtained at a spectral slit width of 2.5 nm in the wavelength range from 400 to 900 nm with a spectral resolution of ~ 0.024 nm. The polished and unpolished surfaces of the polycrystalline CVD ZnSe sample were excited by light with a wavelength of $\lambda_{ex} = 330\text{--}414$ nm ($h\nu_{ex} > E_g$) and $\lambda_{ex} = 478$ nm ($h\nu_{ex} < E_g$), and the PL spectra of the sample were observed in a wide spectral range of 440–700 nm. The green bands in the PL spectrum with maxima at wavelengths $\lambda = 505$ nm, 529 nm, 542 nm, and 565 nm, observed from the polished and unpolished surfaces of the polycrystalline sample, have mainly been investigated. Along with PL, the luminescence excitation spectra (LES) of these maxima ($T = 300$ K) were studied.

UV and visible absorption spectra of polycrystalline CVD ZnSe were recorded on a Specord-250 setup at 300 K (Fig. 1b). The spectra were processed, including the selection of linear sections, using the Excel program. Extrapolation of the linear parts to the intersection with the abscissa axis also makes it possible to reveal the absorption bands. $E_g = 2.714$ eV was determined by linear extrapolation to the region of strong absorption, which corresponds to the band

gap for polycrystalline CVD ZnSe [1, 2]. The optical transition energies 2.696 eV (460 nm), 2.661 eV (466 nm) and 2.638 eV (470 nm) are associated with the absorption of light during the interaction of free excitons (18 meV) and excitons (53 meV, 76 meV) with lattice phonons. In the range of 450–478 nm, a high absorption of CVD ZnSe is observed, and it is known that there is the intrinsic absorption edge of ZnSe in this wavelength range [1].

Results and discussion

The luminescence spectra shown in Fig. 2–7 were recorded when excited with different wavelengths ($\lambda_{ex} = 330, 370, 387, 397, 414$ and 478 nm) from polished (Fig. 2, 3a–7a) and unpolished (Fig. 3b–6b) sample surfaces; along with the PL spectrum, there are excitation spectra with maxima at $\lambda = 505$ nm, 529 nm, 542 nm and 565 nm. Initially, the PL was excited by light with $\lambda_{ex} = 330$ nm and in the resulting PL spectrum (Fig. 2, red line), maxima are observed at wavelengths: 450 nm, 486 nm, 505 nm. In the same figure, the green line shows the LES maximum at $\lambda = 504$ nm. It is seen that the LES maxima for a narrow peak at $\lambda = 504$ nm lie in the wavelength range of 330–414 nm and are most efficiently excited by luminescence photons with $\lambda_{ex} = 370$ nm (Fig. 3a, b), i.e. green luminescence at $\lambda = 504$ nm is effectively excited by light from the intrinsic absorption region for ZnSe (Fig. 1b).

This is typical for ZnSe crystals with a low copper content [9]. The maximum in the spectrum (Fig. 2, red line) at $\lambda = 458$ nm corresponds to the edge luminescence of undoped ZnSe crystals [1, 2], and the maximum at $\lambda = 486$ nm is associated [2, 8, 10] with neutral oxygen centers (O_{Se}). With a change in the wavelength of the exciting light in the range of 330–414 nm, a change in these maxima recorded from the polished and unpolished sample surfaces was practically not observed. When the sample is excited with light $\lambda_{ex} = 330$ nm, a narrow band with a maximum at a wavelength of 450 nm ($h\nu_{max} > E_g$) is observed; and if the excitation is carried out with $\lambda_{ex} = 370$ nm, then the maximum at $\lambda = 450$ nm disappears, and instead peaks are observed at $\lambda = 458$ nm ($h\nu_{max} = E_g$) and $\lambda = 441, 448$ nm ($h\nu_{max} > E_g$). Similar results have been obtained even earlier [11], and we propose to consider these results in terms of polaritons [17, 18].

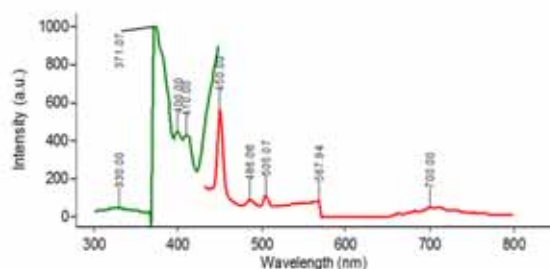


Fig. 2. PL(PS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 330$ nm — red line; green line — excitation spectrum of the maximum at 505 nm.

When the sample is excited by light with a wavelength $\lambda_{ex} = 370$ nm, in addition to the maximum at 504 nm, another maximum is observed in the spectrum at $\lambda = 529$ nm (Fig. 3a, green line; Fig. 3b, blue line). The excitation spectra of the maximum at $\lambda = 529$ nm, which are shown in Fig. 4a by red and Fig. 4b by blue lines have maxima in the 370–478 nm wavelength range and are more intensely excited by light from the intrinsic absorption region ($\lambda_{ex} = 387$ nm). At the same time, in this case, in contrast to the excitation spectrum for the maximum $\lambda = 504$ nm (Fig. 3a, b), the excitation spectra exhibit a less intense maximum at $\lambda = 478$ nm, which closes the intrinsic absorption edge of ZnSe.

The maxima in the excitation spectrum (shown by the red line in Fig. 5a, green line in Fig. 7a1 and blue line in Fig. 5b), which correspond to the maximum in the luminescence spectrum at $\lambda = 542$ nm (green line in Fig. 5a, red line in Fig. 7a1 and the red line in Fig. 5b) lie in the range of 370–478 nm, but are most effectively excited by luminescence photons with a wavelength of $\lambda = 398$ nm. In this case, a less intense maximum is also observed in the LES spectrum at $\lambda_{ex} = 478$ nm.

A narrow band in the PL spectrum at $\lambda_{max} = 565$ nm (Fig. 6a,b, red lines) is observed only when the sample is excited with light $\lambda_{ex} = 414$ nm from the polished and unpolished sample surface, and the excitation spectrum at $\lambda_{max} = 565$ nm covers the range 370–414 nm (purple line in Fig. 6a, orange line in Fig. 6b). Narrow bands at $\lambda = 504$ nm (Fig. 3a,b) correspond the zero-phonon component of the edge SAL radiation of ZnSe, and the peak at $\lambda = 519$ nm is its phonon component.

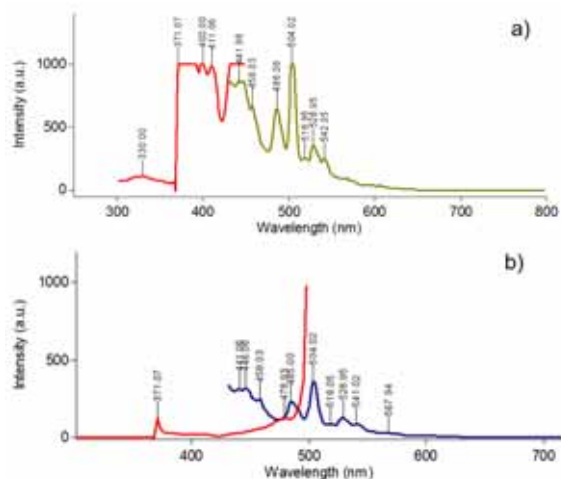


Fig. 3. a) PL (PS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 370$ nm — green line; red line — excitation spectrum of the maximum at 505 nm, b) PL (unPS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 370$ nm — blue line; red line — excitation spectrum of the maximum at 505 nm.

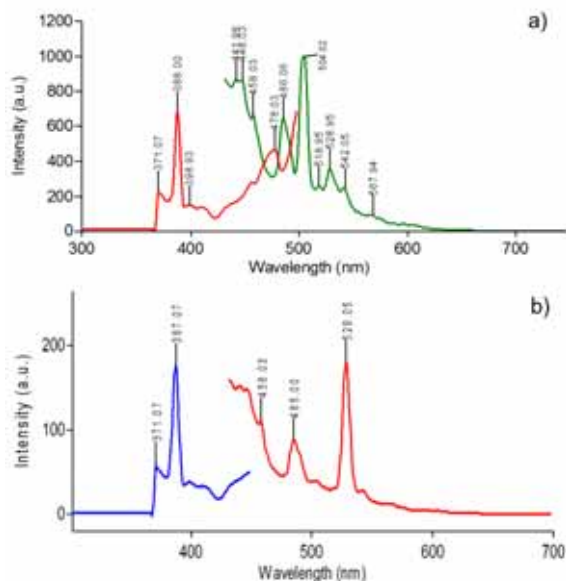


Fig. 4. a) PL (PS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 370$ nm — green line; red line — excitation spectrum of the maximum at 528.95 nm, b) PL (unPS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 387$ nm — red line; blue line — excitation spectrum of the maximum at 528.95 nm.

We also believe that the observation of long-wavelength "anomalous" edge luminescence of ZnSe at room temperature is asso-

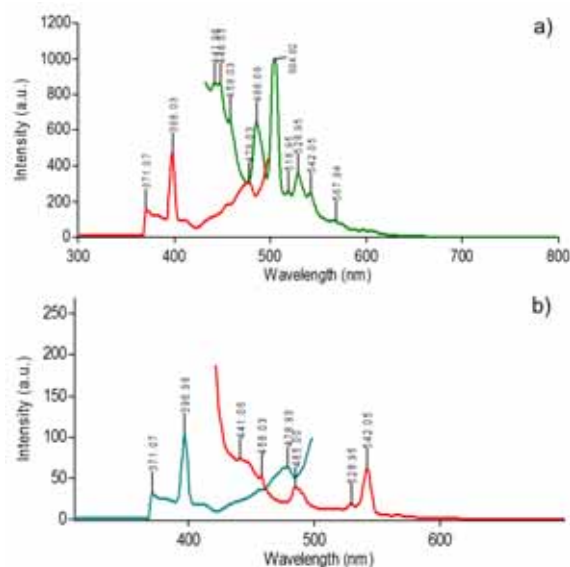


Fig. 5. a) PL (PS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 370$ nm — green line; red line — excitation spectrum of the maximum at 542 nm, b) PL (unPS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 397$ nm — red line; purple line — excitation spectrum of the maximum at 542 nm.

ciated not only with an excess of oxygen and selenium, but also with the presence of copper [8, 10]. The excitation spectra of narrow bands at $\lambda = 504$ nm, observed upon excitation with light $\lambda_{ex} = 370$ nm of polished and unpolished surfaces of polycrystalline CVD ZnSe, have a different structure (Fig. 3a, b, red lines). The LES bands at $\lambda = 504$ nm observed from the polished surface (Fig. 3a, red line) have several maxima ($\lambda = 370$ nm, 400 nm, 410 nm), and the LES bands at $\lambda = 504$ nm observed from the unpolished sample surface (Fig. 3b, red line), have only one maximum ($\lambda = 370$ nm) and it is very weak compared to Fig. 3a (red line). Also, the intensities of the bands at $\lambda = 504$ nm observed from the polished and unpolished sample surface are quite different: the intensity of the band in Fig. 3a (red line) is almost twice as high as the intensity of the $\lambda = 504$ nm band in Fig. 3b (red line).

When the sample is excited with light $\lambda_{ex} = 330$ nm, the maxima at 529 and 542 nm are not observed. The excitation spectrum of the maximum at $\lambda = 529$ nm observed from the polished and unpolished surfaces of polycrystalline CVD ZnSe at various excitation modes, has narrow bands

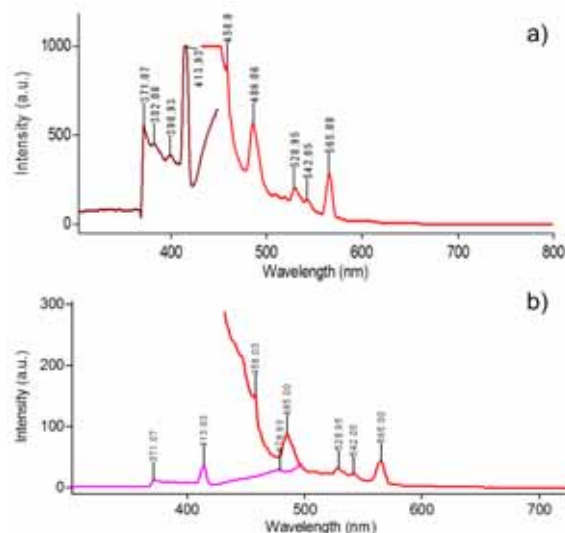


Fig. 6. a) PL (PS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 414$ nm — red line; purple line — excitation spectrum of the maximum at 565 nm, b) PL (unPS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 397$ nm — red line; orange line — excitation spectrum of the maximum at 565 nm.

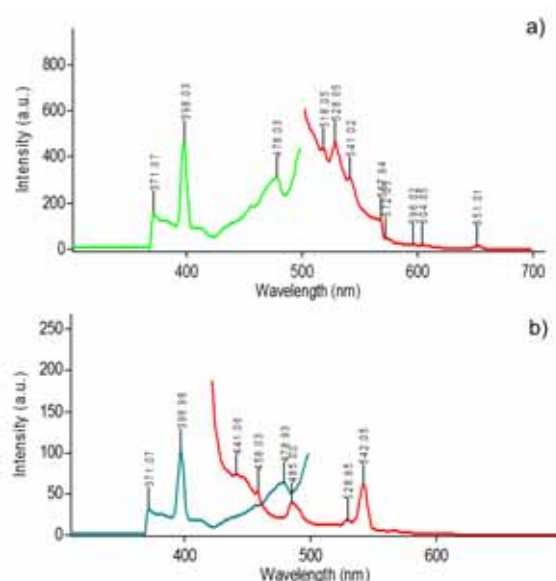


Fig. 7. a1) PL (PS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 478$ nm — red line; green line — excitation spectrum of the maximum at 542 nm, a2) PL (PS) spectrum of polycrystalline CVD ZnSe upon excitation with a xenon lamp, $\lambda_{ex} = 397$ nm — red line; blue line — excitation spectrum of the maximum at 542 nm.

at $\lambda = 387$ nm (3.2 eV) (red line in Fig. 4a, blue line in Fig. 4b); also, there are narrow bands in the excitation spectrum of the maximum at $\lambda = 542$ nm (blue line in Fig. 7a; red line in Fig. 4a), but they are observed at $\lambda = 399$ nm (3.1 eV). The maximum in the PL spectrum at $\lambda = 529$ nm is due to the electronic transitions from a shallow donor to an associative acceptor center $\{\text{Cu}_{\text{Zn}}^{++} - \text{Cu}_i^+, \text{O}_{\text{Se}}\}$ [9]. Selenium vacancies V_{Se} ($E_D \sim 10\text{--}20$ meV) or other point defects formed on the basis of uncontrolled background impurities can be such shallow donors in the sample under study. The maximum at $\lambda = 542$ nm in the spectra can be considered the result of the presence of a phonon component at $\lambda_{\text{max}} = 529$ nm.

The maximum (Fig. 6a, b, red lines) at $\lambda = 565$ nm in the PL spectrum is observed only when polished and unpolished surfaces are excited with light $\lambda_{\text{ex}} = 414$ nm, which is confirmed by the presence of a narrow maximum at $\lambda = 414$ nm in the excitation spectra (purple line in Fig. 6a, orange line in Fig. 6b). It is noteworthy that the intensity of the 565 nm band (Fig. 6a,b, red lines) and the maximum at $\lambda = 414$ nm in the excitation spectrum (purple line in Fig. 6a, orange line in Fig. 6b) from the unpolished sample surface, several times less than from the polished sample surface. The band observed at 565 nm is most likely due to the luminescence of impurity-defect complexes, which consist of intrinsic point defects of oxygen and other background impurities [2, 8, 10].

In addition, a maximum is observed in all spectra at a wavelength of $\lambda = 486$ nm (except for excitation with light $\lambda = 478$ nm); in cathodoluminescence (CL) spectra [10], it is observed at $\lambda = 490$ nm, i.e., at a wavelength longer than that of PL, and is considered as the edge luminescence of SAL during the growth of crystals with an excess of Se and an increased oxygen concentration. In ZnSe:Se(O) samples with an excess of selenium and oxygen close to *p*-type conductivity, segregation of oxygen-containing complexes was observed at stacking faults (SFs) [2, 10, 17], where the oxygen concentration $[\text{O}_{\text{Se}}] \sim 10^{20} \text{ cm}^{-3}$; while, in "pure" layers, it can be $\sim 10^{19} \text{ cm}^{-3}$. The authors of [2, 10, 17] showed that, depending on the oxygen content, the change in the band structure is different, which determines the formation of a complex multiband structure. Volume and charge compensa-

tions of the isoelectronic acceptors $[\text{O}_{\text{Se}}]$ with a concentration of $\sim 10^{19}\text{--}10^{20} \text{ cm}^{-3}$ in ZnSe:Se(O) samples are possible with the introduction of copper, the average concentration of which $\sim 10^{16} \text{ cm}^{-3}$ ensures the formation of a stable $\{\text{O}_{\text{Se}}\text{--Cu}\}$ complex. The authors of [2, 10, 17] believe that copper in the complexes can only be located in interstices as Cu_i^+ donor defects, and these complexes are concentrated in SF layers. Narrow-line cathodoluminescence (CL) spectra of these complexes with a leading line of 508 nm (with a phonon repetition of a wide band at 520 nm) were observed ($T = 80$ K) even at a background copper concentration of 10^{16} cm^{-3} for relatively oxygen-pure CVD ZnSe:O(Se) [2, 10]. In our PL spectra, a maximum was observed at $\lambda = 504$ nm (with phonon repetition at $\lambda = 518$ nm), i.e. PL bands [17] were observed as somewhat shorter wavelengths than the CL bands (at $T = 300$ K). The advantage of photoexcitation is the deep penetration of the exciting light into the bulk of the crystal. This fact distinguishes photoluminescence spectra from CL ones and makes it possible to excite spatially separated luminescence centers.

The analysis of the PL spectra agrees with the measurements of the absorption spectrum as follows: three bands were detected in the area accessible to our measurements (Fig. 1b). The highest SW absorption occurs in the exciton band (2.696 eV) of the pure layers, and the main maximum (2.661 eV) – on the SF. Moreover, the LW band (2.638 eV) can be associated with absorption by the SA(L) center. The presence of several bands in the absorption spectrum and a long-wavelength shift of the additional absorption edge by hundreds of meV also indicate the existence of spatially separated luminescence centers in ZnSe(O)Se crystals. A comprehensive study of the photoluminescence and excitation spectra of polycrystalline CVD ZnSe, as well as the absorption spectrum, makes it possible to better understand the problems associated with the spatial distribution of individual luminescence centers in ZnSe(O)Se crystals; this, in turn, contributes to the improvement of the method of optical diagnostics of single-crystal systems with defects.

4. Conclusions

Spectral positions of all maxima at wavelengths (energy): 486 nm (2.55 eV), 505 nm (2.46 eV), 529 nm (2.34 eV), 542 nm (2.29 eV) and 565 nm (2.19 eV) are the

same for the polished and unpolished surfaces of the sample; this indicates that all recombination centers are also the same on the surface and in the depth of the sample. However, the differences in the intensity and structure of the excitation spectrum for the maxima at $\lambda = 505$ and 565 nm may be associated with the inhomogeneous distribution of oxygen and background impurities over the volume of the sample under study. In the wavelength range of 370 to 414 nm (3.35 to 3 eV), several maxima of the excitation spectrum namely, 505 nm (2.46 eV), 529 nm (2.34 eV), 542 nm (2.29 eV) and 565 nm (2.19 eV) show the presence of non-elementary isoelectronic oxygen luminescence centers in ZnSe(O)Se crystals, the study of which opens up new possibilities for the method of optical diagnostics of single-crystal systems with defects.

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