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REGULAR ARTICLE

Light Emitters Based on ZnO:Mg Heterolayers

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The possibility of obtaining light emitters on ZnO:Mg heterostructures has been established. In these structures, high radiation intensity is created as a result of preliminary doping of ZnSe substrates with an isovalent Mg impurity. ZnO:Mg was grown by isovalent substitution of Zn anions with oxygen at temperatures of 950-1050 °C. This results in the formation of heterostructures with intense radiation in the short-wave region $\Delta\lambda \approx 0.36$-$0.42$ μm with a maximum at 0.38 μm. Preliminary doping of the base material with an isovalent Mg impurity causes a significant increase in the quantum efficiency of ZnO radiation to $\eta \approx 20$-25 %. It has been established that it is formed by the annihilation of bound excitons, recombination through local Mg energy states, and intense interband recombination.

Keywords: Light emitters, Zinc oxide, Isovalent magnesium impurity, Recombination.

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1. INTRODUCTION

Zinc oxide is a wide-bandgap semiconductor material that plays an important role in modern electronics [1]. A number of properties are important for its practical use, in particular, high radiation resistance and significant piezoelectric and pyroelectric effects. The technological processes used to manufacture ZnO layers, such as magnetron and cathode sputtering and various types of epitaxies, provide intense radiation with a quantum yield of $\eta = 0.5$-2.5 %. It should be noted that such technological processes require expensive equipment and, under certain conditions, highly toxic organometallic compounds. In addition, the parameters and characteristics of the layers obtained by cathodic sputtering and chemical transfer are difficult to reproduce. At the same time, the direct bandgap and large width of the ZnO bandgap correspond to the solution of an important task of obtaining effective light emitters and photosensitive structures in the poorly mastered short-wave region [2, 3]. Therefore, it is relevant and important to further improve the methods of obtaining the material, as well as to develop a new technology for obtaining layers. In this regard, the selection of the type of dopants and doping methods for the creation of highly efficient radiation sources is an important task in the manufacture of devices.

2. EXPERIMENT

2.1 Tested Samples

ZnO heterostructures (HSs) were obtained by isovalent substitution (IVS) on specially prepared single-crystal substrates of zinc II-VI compounds. Their dimensions are 4×4 mm and thickness are 1 mm. The HSs were fabricated on substrates previously prepared using a classical physicochemical method [4]. For this purpose, grinding and polishing operations were performed sequentially using standard powders and diamond paste. The surface condition and possible damage were checked visually under an MBS-9 microscope at a magnification of at least 40^{X}. Accordingly, the surface layer with defects after processing (microcracks, increased dislocation density) was removed by chemical etching in appropriate etching solutions, followed by washing of the samples in deionised water on an UZDN-2T ultrasonic unit. Complete removal of the damaged surface layer was monitored by the appearance of intense photoluminescence (FL) at 300 K, which was not observed after mechanical polishing.

To obtain ZnO layers, prepared II-VI compound wafers were annealed in purified air at $T = 1000$-1100 K. In the surface layer of the compounds, the anions of the substances were replaced by an isovalent oxygen impurity, and a ZnO heterolayer was obtained. Its formation is due to the processes of oxygen diffusion with the subsequent replacement of anions in semiconductor compounds.

2.2 Research Methods

The main subject of the research was their optical characteristics, which made it possible to determine the main properties of the obtained HSs and the possibilities for their practical use. Comprehensive studies of optical transmittance T_ω, reflectance R_ω and photoluminescence N_ω were carried out [4, 5]. The measurements were carried out using an extended methodology on a universal optical setup with an MDR-23 spectral device, an FEP-79 photodetector and a synchronous detection system [6]. This made it

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possible to determine the possibility of creating light-emitting devices based on the samples under study. The measurements were carried out both using classical methods and using the λ-modulation method [7]. This yielded the differential spectra T'_ω, R'_ω, N'_ω, which made it possible to determine the values of the main parameters and characteristics of the material obtained, establish the properties of the samples under study, and determine the possibilities for varying their properties in the manufacture of emitters.

The source of optical radiation in the study of FL N_ω was an LGN-21 nitrogen laser with an excitation radiation wavelength $\lambda = 0.337$ μm ($\hbar\omega = 3.68$ eV), and a special ELC/C halogen lamp with a monotonic smooth spectrum was used to study T_ω and R_ω. It should be noted that a specially designed complex optical system in front of the MDR-23 entrance slit was used for irradiation in the studies of absorption, reflection and luminescence. It should be noted that a specially designed complex optical system in front of the MDR-23 entrance slit was used for conducting absorption, reflection and luminescence studies for irradiation. It ensured the combined use of light flux from the ELC/C lamp in the study of optical absorption and reflection. An LGN-21 laser was used to study luminescence, the intensity of which was varied by a system of calibrated light filters. The corresponding light signal from the sample in the specified studies was focused on the entrance slit of the monochromator, after which it was registered by a photodetector and a synchronous detection system.

3. RESULTS AND DISCUSSION

The possibility of obtaining ZnO:Mg HSs by isovalent substitution (IVS) on base substrates of II-VI semiconductor compounds ZnTe, ZnSe, and ZnS has been established. They are characterised by high quantum efficiency emission in the poorly explored short-wave region. For this purpose, the base substrate, particularly ZnSe, was pre-doped by known classical diffusion with Mg impurity in a quartz ampoule at a vacuum of 10^{-4} Torr [8]. Such special preparation of the substrates significantly increased the FL efficiency of the obtained ZnO layers, whose emission spectrum falls in the edge region of quantum energies $\hbar\omega = 2.95$-3.45 eV ($\lambda \approx 0.36$-0.42 μm).

It should be noted that doped ZnO:Mg layers were obtained after preliminary doping of Mg ZnSe with an isovalent impurity (IVI) to form a ZnSe:Mg layer on their surface with high quantum efficiency of luminescence $\eta = 12$ % in the edge region of radiation [9]. Such doping ensured the subsequent formation of ZnO:Mg HSs at appropriate temperatures. Emitters on Mg-doped ZnO HSs are characterised by a quantum luminescence efficiency of $\eta \approx 20$-25 %. It should be noted that existing methods for obtaining ZnO HSs allow ZnO films with weak emission ($\eta \approx 0.5 - 0.7\%$) in the green-blue region to be obtained.

A typical spectrum of radiation from the obtained layers is shown in Fig. 1. Based on the nature and characteristics of the spectral distribution of radiation in the short-wave range and its dependence on the conditions of use, its components were established [9].

It is important for practical applications to obtain intense light emitters with effective photon yield in the short-wave range under the energy condition $\hbar\omega \geq Eg$, component *A*. This is due to recombination during interband transitions of free charge carriers. Its spectrum is well described by the well-known theoretical expression [10]

$$N_\omega = (\hbar\omega)^2\left(\hbar\omega - E_g\right)^{1/2} exp\left[-\frac{\hbar\omega - E_g}{kT}\right] \qquad (1)$$

where k is Boltzmann's constant, T is temperature. The interzone nature of recombination processes is also confirmed by the following properties of component *A*: intensity I changes with variation in excitation level L according to the known dependence $I \sim L^2$; the position of the maximum does not depend on L. In addition, when the temperature T changes, the position $\hbar\omega_m$ repeats the temperature dependence of the forbidden zone width, and the half-width is

$$\hbar\omega_{1/2} \approx 1.5\kappa T.$$

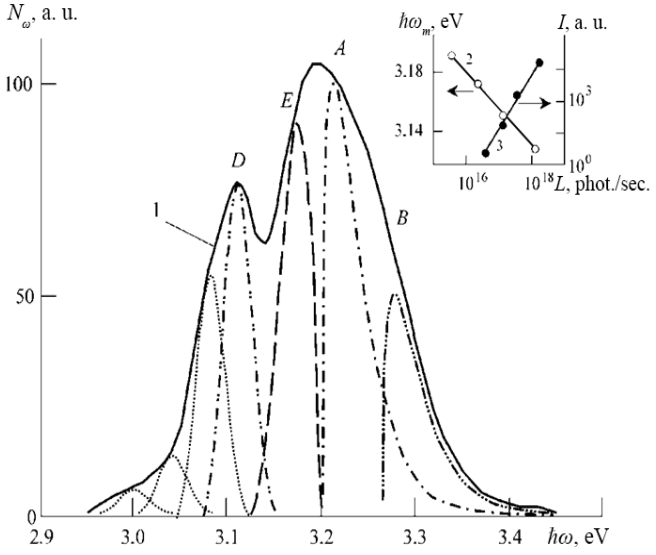


Fig. 1. – Photoluminescence spectrum of ZnO:Mg (1) layers: bands *A*, *B* – interband radiative recombination; *E* – exciton; *D* – recombination involving simple Mg_{Zn} centres. The inset shows the dependence of the position of the maximum $\hbar\omega_m$ of band *E* (2) and intensity (3) on the excitation level *L*

At the same time, in the energy range of quanta $\hbar\omega \geq Eg$, the spectral distribution of the intense interband radiation of component *A* is characterised by a slight difference in intensity from the distribution calculated using the theoretical expression for interband transitions. This slight discrepancy in the spectral curves is completely eliminated when optical transitions involving a subzone split by spin-orbit interaction $\Delta_{SO} \approx 0.06$ eV are taken into account [11]. Its spectrum can be calculated using (1), and the calculated band *B* in terms of intensity complements band *A*. Overall, the resulting spectrum correlates with the measured experimental curve.

The established nature of generational-recombinational processes is confirmed by singularities in the differential spectra R'_ω λ-modulated optical reflection, Fig. 2.

Singularities are observed in the differential spectra λ–modulated optical reflection R'_ω of ZnO:Mg HSs, which are determined by the above-mentioned optical processes during luminescence. In particular, the main feature A at quantum energies $\hbar\omega \approx 3.208$ eV is formed

by charge carrier transitions through the forbidden zone and agrees with the value of E_g ZnO. Feature B at $\hbar\omega$ = 3.268 eV is determined by transitions involving the split spin-orbital interaction Δ_{SO} of the valence subband. The difference in quantum energies $\hbar\omega - Eg$ is Δ_{SO} = 3.268 – 3.208 = 0.06 eV, which correlates with the spin-orbit interaction energy [12]. The recombination processes in bands A and B complement each other and, in general, form zone-zone transitions of free charge carriers during the formation of radiation in the region $\hbar\omega \geq Eg$.

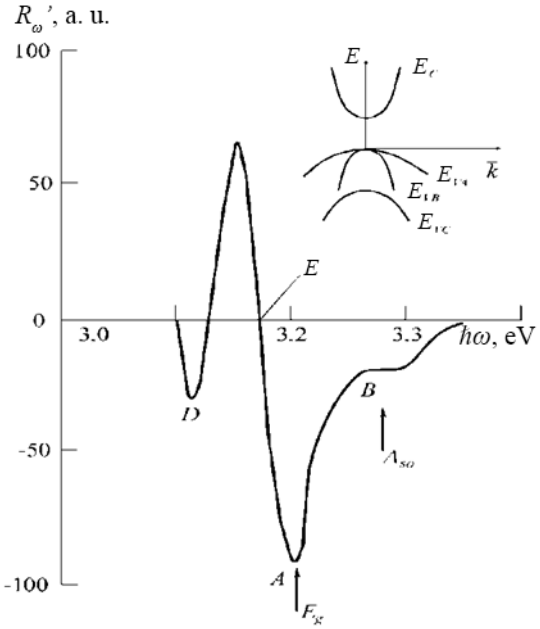


Fig. 2. – Optical reflection spectrum of *ZnO:Mg* layers under λ-modulation. Insert: energy band structure (for cubic lattice)

The high intensity of interzone radiation in light emitters based on ZnSe – ZnO:Mg HSs indicates the high perfection of the crystal structure obtained precisely by the IVS method of ZnO:Mg layers. In addition, a similarly high intensity of edge radiation is also observed in the case of HSs of other II-VI compounds obtained by IVS [13, 14]. At energies $\hbar\omega \geq Eg$, a band with a maximum of $\hbar\omega \approx 3.17$ eV is formed, Fig. 1. On the optical reflection spectrum R'_ω HS ZnO:Mg, it corresponds to the intersection point E, Fig. 2. The energy difference 3.208 – 3.17 is 0.038 eV and, due to its small value, indicates the formation of bound excitons.

Radiation of a similar nature is also observed for Mg-doped II-VI compounds CdTe, ZnS, CdSe [15, 16]. This type of excitons is formed precisely by the impurity IVI Mg. It has been shown [16] that they cause the formation of local short-range forces in the crystal lattice. One of the confirmations of this is the changes in the X-ray diffraction reflection curves. In particular, studies conducted on ZnSe:Mg substrate layers revealed a slight change in the half-width of the curves from 45 arcseconds to 57 arcseconds when Mg was doped at 1000 °C on the *i*-ZnSe substrates used, Fig. 3 [17]. This results in minor local disturbances in the periodicity of the tetrahedral configuration of atoms due to the action of an IVI [13, 18]. These are caused by the short-range potential of the IVI, which causes so-called 'short-range disorder' [11]. It is this disorder that causes the binding of excitons in accordance with the Hopfield phenomenological model [19]. It should be noted that the exciton nature of the E band is confirmed by the corresponding properties of radiation, namely, the maximum $\hbar\omega_m$ shifts with an increase in the excitation level L to the region of lower photon energies, insert in Fig. 2. For the spectrum R'_ω HS ZnO:Mg, it corresponds to the point E of intersection of the curve with the abscissa axis. The intensity I of the radiation of this E band with the excitation level changes according to the law $I \sim L^{1.5}$. Note that compared to classical bound excitons, the half-width of the exciton band increases to $\Delta\hbar\omega_{½} \sim kT$ [20]. However, due to the short-range potential, the efficiency of exciton radiation increases significantly, in particular to $\eta \sim$ 20-25 % for the studied ZnO:Mg layers.

Also, the radiation of ZnO:Mg HSs has a maximum at a photon energy of $\hbar\omega_m \approx 3.12$ eV. This component B is caused by the formation of Mg_{Zn} centres. No such band was observed in unalloyed layers. Their formation is caused by the substitution of point defects (zinc vacancies) in ZnO by Mg atoms due to their identical valences. Moreover, this process is possible due to the close values of the covalent radii of Mg and Zn. Therefore, deep Mg_{Zn} substitution centres are formed, on which excitons with their properties discussed above are localised.

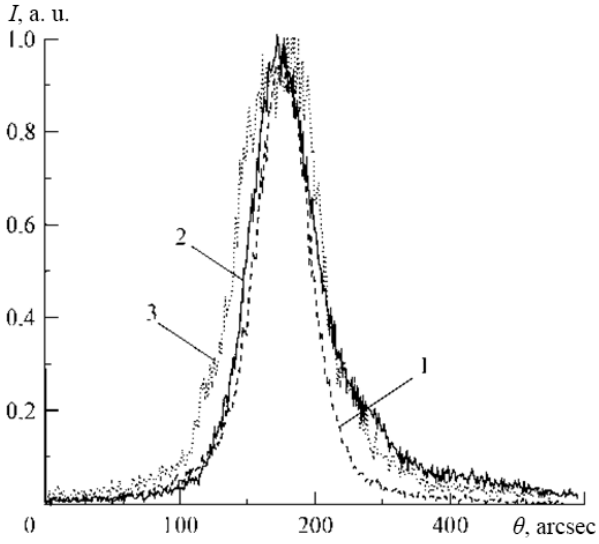


Fig. 3. – Diffraction reflection curves of initial (1) and *i*-ZnSe crystals doped with an isovalent magnesium impurity at $T_{diff.}$ 1000 °C (2)

Band B is characterised by a large half-width, independence of the position of the maximum $\hbar\omega_m$ from the excitation level L, but a significant dependence on L of its intensity. In the low-energy region relative to $\hbar\omega_m$, a gradual decrease in intensity is observed. These properties are characteristic of the recombination of charge carriers through local centres during their interaction with crystal lattice vibrations [2]. Such processes are well explained by the Yuls-Kreger model, according to which PL spectra, assuming a Gaussian distribution in component bands, are described by the expression [21]

$$N_\omega = \sum_{n=0}^{\infty} N_{\omega_n} exp\left[-\frac{(\hbar\omega - \hbar\omega_m \pm n\cdot\hbar\omega_{LO})^2}{\sigma^2}\right] \quad (2)$$

where $\hbar\omega_{LO} \approx 0.059$ eV is the energy of the *LO* phonon in β-ZnO, σ is a parameter related to the width of the $\hbar\omega_{1/2}$ band by the well-known relation [4]

$$\sigma = \hbar\omega_{1/2}/8 \cdot ln\,2 \qquad (3)$$

The experimental spectrum of the band is well described by the sum of the calculated phonon repetitions of the main band [22].

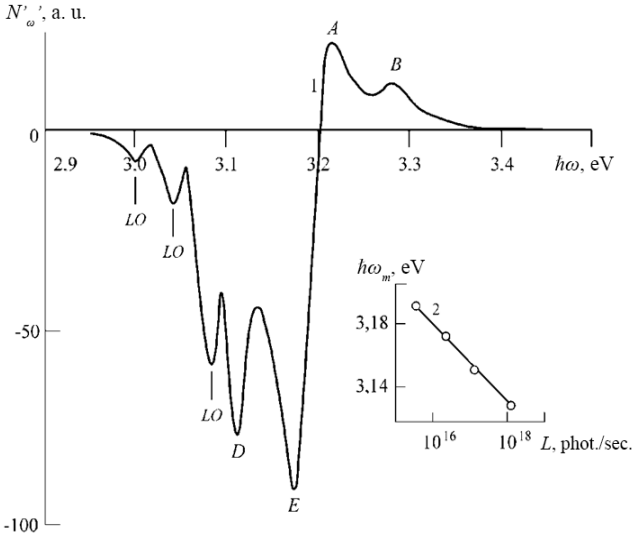


Fig. 4. – Photoluminescence spectrum of ZnO:Mg layers under λ-modulation. The inset shows the dependence of the position of the maximum $\hbar\omega_m$ of the *E* band on the excitation level *L*

The legitimacy of these recombination processes in the emission of ZnSe – ZnO:Mg HSs is confirmed by studies of spectra using λ-modulation, Fig. 4. The selection of the appropriate modulation frequency allowed us to determine the components *A*, *B*, *E*, and *D* of the emission spectrum. When measuring the second derivative, the established components with their defined properties are clearly manifested. The inset also shows the nature of the dependence of the intensity of the excitons dominating the spectra.

The formation of Mg simple energy centres causes optical transitions involving them. They are characterised by a feature at 3.11 eV, marked with the letter *D*. The difference in quantum energy values $E_g - \hbar\omega_m = 3.208$ eV – 3.11 eV = 0.088 eV corresponds to the depth of occurrence of the simple centres formed, caused by the isovalent impurity of magnesium. It was the use of λ-modulation with its high sensitivity and resolution that made it possible to establish the characteristics and energy parameters of the components obtained. In general, the detected radiation properties correlate well with the features on the differential curve R'_ω, which confirms the nature of generation-recombination processes in bands *A*, *B*, *E*, and *D* in light emitters based on ZnO:Mg HSs.

Thus, the use of the IVS method ensures the production of ZnO:Mg HSs on pre-doped Mg substrates of ZnSe. HSs are characterised by high quantum efficiency of radiation η = 20-25 %, which is important for obtaining high-intensity radiation sources. Their emission spectrum covers the short-wave region $\lambda \approx 0.36$-0.42 μm and is formed by recombination processes of different nature. They are determined by the corresponding processes during the formation of the crystal lattice by the IVS method. The isovalent nature of the Mg dopant causes the annihilation of excitons bound to isovalent Mg dopants, which determines the nature of the dominant radiation with a maximum at a photon energy of $\hbar\omega \approx 3.17$ eV ($\lambda_{max} \approx 0391$ μm). The local energy centres of Mg_{Zn} formed by IVI Mg determine the recombination of charge carriers with their participation in the interaction with crystal lattice vibrations. The high perfection of the ZnSe-ZnO:Mg crystal structure determines a significant contribution of interband transitions of free charge carriers in the formation of radiation in the photon energy range $\hbar\omega \geq Eg$. They determine their significant contribution to the formation of the total radiation spectrum of ZnO:Mg layers obtained on pre-doped IVI Mg substrates ZnSe:Mg. The quantum efficiency of the latter is η = 12 % in the edge region of radiation.

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Світловипромінювачі на основі гетерошарів ZnO:Mg

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Встановлено можливість отримання світловипромінювачів на гетерошарах ZnO:Mg. У них висока інтенсивність випромінювання створюється внаслідок попереднього легування підкладок ZnSe ізовалентною домішкою Mg. Вирощування ZnO:Mg проводилося методом ізовалентного заміщення аніонів Zn киснем при температурах 950-1050 °C. За ним формуються гетероструктури з інтенсивним випромінюванням у короткохвильовій області $\Delta\lambda \approx 0{,}36$-$0{,}42$ мкм із максимумом на 0,38 мкм. Попереднє легування базового матеріалу ізовалентнтою домішкою Mg обумовлює істотне збільшення квантової ефективності випромінювання ZnO до $\eta \approx 20$-25 %. Встановлено, що воно формується анігіляцією зв'язаних екситонів, рекомбінацією через локальні енергетичні стани Mg та інтенсивною міжзонною рекомбінацією.

Ключові слова: Світловипромінювачі, Оксид цинку, Ізовалентна домішка магнію, Рекомбінація.