EFFECT OF THE DEPOSITION TECHNOLOGY AND STRUCTURE OF ZnO FILMS ON THEIR PHOTO- AND CATHODOLUMINESCENCE

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The photoluminescence (PL) of ZnO films deposited on Si and SiC substrates using the PEMOCVD method is investigated under various technological conditions. The most intense ultraviolet (UV) PL is observed for the zinc oxide films grown on Si with the use of a SiN$_x$:H buffer layer at a temperature of 350 °C. It is assumed that the intensity of "green" luminescence decreases due to the diffusion of hydrogen into the ZnO film. The thickness uniformity of the optical properties of the films is estimated with the help of the cathodoluminescence (CL) method which is important for the creation of UV light-emitting devices on the basis of ZnO. An ultralinear behavior of the integral CL intensity as a function of the excitation power was observed.

1. Introduction

In the recent years, a much attention is attracted by zinc oxide ZnO as a material for the creation of devices of spintronics, nanoelectronics, optoelectronics, etc. [1]. ZnO represents a wide-gap, direct-band semiconductor. As compared to GaN, zinc oxide is especially attractive for the creation of ultraviolet and blue light-emitting devices due to the high exciton binding energy ($\approx$60 meV at room temperature) [2, 3]. The emission spectrum of ZnO usually consists of two luminescence bands: the narrow UV exciton luminescence (EL) and the "green" deep-level emission (DLE) [4, 5]. The nature of the DLE is most probably related to the presence of such defects as Zn atoms in interstitial sites (Zn$_i$) and/or oxygen vacancies (V$_\mathrm{O}$) [6]. The attainment of intense UV luminescence at the background of the weak "defect" DLE in ZnO films favors the creation of high-quality devices of UV optoelectronics.

However, there exist problems that need to be solved in order to develop ZnO-based devices. The first of them is the absence of stable reproducible methods allowing one to obtain ZnO with conductivity of the p-type. But some scientific groups recently reported the encouraging results [7–9]. The difficulties also lie in obtaining the intense luminescence of ZnO in the short-wave spectral region. Having the band-gap energy of 3.37 eV, zinc oxide must emit in the range 366–380 nm (A-UV range).

The crystal perfection of ZnO films influences the optical and electric properties; that's why the choice of the backing material for their deposition must be well-grounded. Unfortunately, the substrates most widely used for today (ScAlMgO$_4$ [10], SiC, Al$_2$O$_3$, ZnO) are expensive or have small dimensions. In contrast, silicon substrates are cheap, technological, and have sufficient dimensions, but they are characterized by a substantial mismatch of the lattice spacing with the ZnO film, which complicates the growth of films with high crystal perfection [11, 12]. An increase in the deposition temperature results in the appearance of a SiO$_x$ layer at the Si substrate [13]. The SiO$_x$ layer is formed between the ZnO film and the Si substrate due to the diffusion of oxygen from the ZnO layer, which influences the stoichiometry of ZnO and, correspondingly, the optical characteristics. It is desirable to grow grain-oriented ZnO films at lower temperatures. A possible way lies in the use of buffer layers: GaN, MgO, and ZnS [14–16]. In addition, it is promising to apply such technological methods as the use of low- and high-temperature buffer layers (LTBL or HTBL) [13, 17].

Recently, it was shown that the SiN_x layer can be used as an interlayer protecting the silicon surface against oxidation during the growth of ZnO films [18,19]. Hydrogenated SiN_x layers were used as a passivating material and as antidiffusion layers in the production of solar cells [20].

The given work was aimed at the achievement of the effective UV emission of the material on the basis of thin ZnO films. ZnO films were deposited on silicon substrates with the help of the technology of plasma-enhanced metallorganic chemical vapor deposition (PEMOCVD) with the use of a SiN_x:H buffer layer or without it at three different temperatures. The post-growth annealing was applied under various conditions.

2. Experimental Part

2.1. Production of samples

Thin ZnO films ($\sim$ 200 nm) were grown by means of the PEMOCVD method with the use of zinc acetylacetonate and oxygen as precursors. It was earlier reported that the PEMOCVD technology allows one to decrease the substrate temperature due to the use of a high-frequency plasma discharge [21]. In this work, three series of ZnO samples were obtained: at the low (150 °C), medium (350 °C), and high (550 °C) temperatures of the substrate. In each series of the samples, various kinds of the substrates were used: Si(100), Si(111), and SiN_x:H/Si(110). The SiN_x:H layer (150 nm in thickness) was grown on Si (110) by means of the PECVD technology [18] at the substrate temperature of 300 °C. For the sake of comparison, we investigated ZnO/SiC samples ($T_s = 350$ °C) as well as ZnO films deposited on SiN_x:H/Si(110) with the use of a low-temperature buffer layer [13]. The more detailed technological conditions of deposition can be found in [22]. After that, a part of the ZnO/SiN_x:H/Si(110) samples ($T_s = 350$ °C) was annealed in the air at 850 °C during 90 min using two methods: isochronal thermal annealing (ITA) and face-to-face annealing (FTFA) [23].

2.2. Investigation methods

For the purpose of a further practical use, the luminescence was measured at room temperature. The PL spectrum was investigated with the help of a monochromator Acton SP500i, the PL was excited by the third harmonic of the radiation of a femtosecond sapphire-Ti laser Mira 900F (37 mW, 170 fs, 76 MHz) [18]. The cathodoluminescence (CL) spectrum was investigated using a scanning electron microscope Leo 1550 Gemini and a MonoCL system (Oxford Instruments, the grating of 1800 groove/mm) at the standard value of the voltage of 10 kV and the aperture of the electron beam of 30×30 μm. The latter has a form of a square orifice and represents a limiter of the anode current. After the limiter, the beam is focused at the sample having a form of a spot of $\sim$50 nm in diameter. The focused beam scanned a square section of the sample surface with the side of 30 μm. The signal was accumulated in order to average the properties of the sample. The magnitude of excitation of the samples was changed by means of the variation of the aperture (20–120 μm), which resulted in the change of the electron beam current in the range 9×10^{-11}–5×10^{-9} A. Structural properties were investigated with the help of a diffractometer Siemens D5000 (Cu$K\alpha$, $\lambda = 0.1542$ nm). In order to investigate the uniformity of the film and the quality of the film/substrate interface, a scanning electron microscope Leo 1550 Gemini SEM was used. The morphology of the film surface was studied with the help of atomic force microscopy (AFM) in the tapping mode. For the most promising samples, we investigated the CL depending on the electron energy in the beam in the range 2.5 – 30 keV that determines the depth of their penetration into the film [24, 25].

3. Results and Their Discussion

We investigated the PL spectra of thin ZnO films deposited on silicon substrates (Si(100), Si(111), SiN_x:H/Si(110)) at three different temperatures (T_s = 150, 350, and 550 °C). In the ZnO films deposited at the low temperature ($T_s = 150$ °C), luminescence was not observed, which is related to the low crystal perfection of the films and a high concentration of nonradiative relaxation centers in them.

All the films deposited at the temperature T_s = 550 °C demonstrated two radiation bands: the EL and the DLE ones. In the case of direct deposition of ZnO on Si, such a result can be explained by the chemical interaction of the substrate with ZnO. The formation of the SiO_x layer due to the diffusion of oxygen from the ZnO film results in the oxygen depletion of the interface and influences the PL spectrum in two ways: a) formation of additional vacancies of oxygen that represent radiation (DLE) centers; b) appearance of

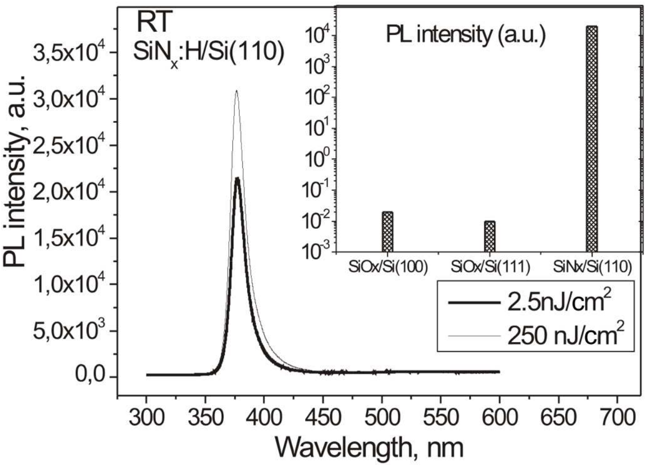

Fig. 1. PL spectrum of the ZnO film deposited on the SiN$_x$:H/Si(110) substrate at 350 °C. In the inset – the diagram comparing the maxima of the intensities of the exciton PL for ZnO/Si(100), ZnO/Si(111), and ZnO/SiN$_x$:H/Si(110)

nonradiative centers, which results in a decrease of the total intensity of radiation. In the case of deposition of ZnO on SiN$_x$:H/Si(110), the DLE intensity becomes weaker, which can be explained by a decrease of the oxygen diffusion from the ZnO film to the surface of the silicon substrate due to the SiN$_x$:H protective antidiffusion buffer layer.

The ZnO films deposited at $T_\mathrm{s} = 350$ °C demonstrated the best PL properties. We observed a significant difference between the PL spectral intensities for the ZnO films deposited on silicon substrates and the films with the use of the SiN$_x$:H buffer layer. The ZnO/Si(100) and ZnO/Si(111) samples showed the EL emission peaks at 387 and 388 nm, respectively. A wide DLE was also observed in the range 450–650 nm. The as-deposited ZnO/SiN$_x$:H/Si(110) films ($T_\mathrm{s} = 350$ °C) have a narrow peak of UV luminescence (379 nm) with an approximate half-width of 9 nm and the absence of DLE at room temperature (Fig. 1).

The structure of the ZnO/SiN$_x$:H/Si films ($T_\mathrm{s} = 350$ °C) was investigated with the help of X-ray structural analysis and a scanning electron microscope (SEM). The films were polycrystalline and consisted of grains with the (002) preferred orientation. For a part of the grains, the (100) and (101) preferred orientations were observed (Fig. 2). We suppose that this stricture was formed as a result of the "self-textural" growth [26]. Initial small grains originated with an arbitrary random crystallographic orientation. In the process of growth, there takes place a competitive survival of the most energy efficient orientations with the absorption of neighbor grains for which the free surface energy is higher. The most energy efficient orientation of the grains ($\langle 002\rangle$) becomes dominant due to the least free surface energy of the (0001) crystallographic plane.

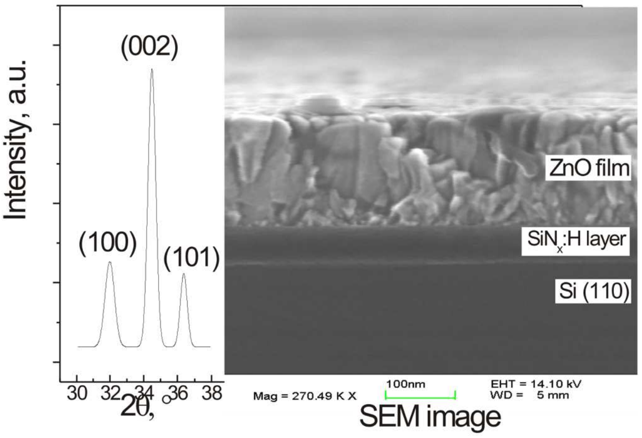

Fig. 2. X-ray diffraction pattern and SEM image of the ZnO/SiN$_x$:H/Si film deposited at T_s= 350 °C

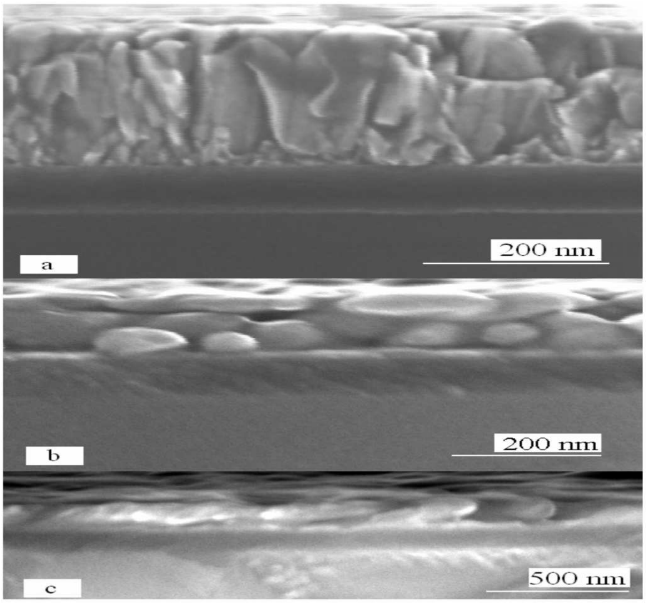

Fig. 3. SEM image of the as-deposited ZnO/SiN$_x$:H/Si film (*a*) and the films obtained by the FTFA (*b*) and ITA (*c*) methods

The SEM image in Fig. 2 confirms the proposed evolution of the structure of the film during its growth. It is worth noting that the SiN$_x$:H layer does not decay in the process of growth of the ZnO film, and there exists a distinct substrate/film interface in such heterostructures (Fig. 3). These results testify to the

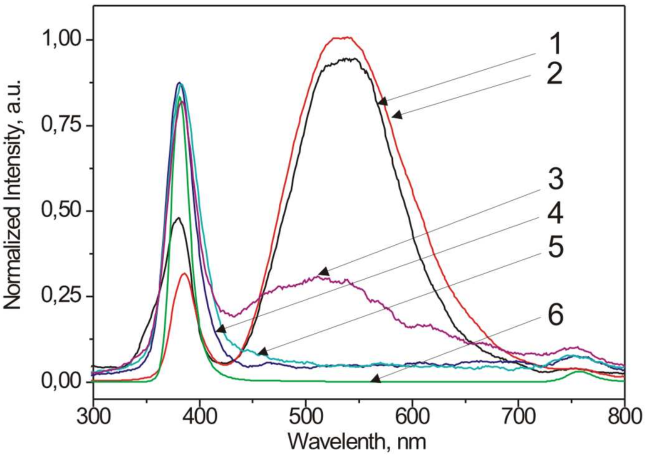

Fig. 4. Normalized CL spectra of the ZnO films: *1* – ZnO/SiN$_x$:H/Si annealed by the ITA method; *2* – ZnO/SiN$_x$:H/Si annealed by the FTFA method; *3* – ZnO/SiN$_x$:H/Si deposited at 350 °C; *4* – ZnO/SiN$_x$:H/Si deposited with the use of LTBL; *5* – ZnO/SiN$_x$:H/Si deposited at 300 °C; *6* – ZnO/SiC

fact that SiN$_x$:H layers can be used as a barrier for the diffusion of oxygen from the ZnO film to the silicon substrate.

As the ZnO films deposited on the SiN$_x$:H/Si substrate demonstrated the best PL properties, we investigated their CL properties. For these investigations, we chose the samples deposited at T_s = 300 and 350 °C as well as the sample with a low-temperature buffer layer. For the sake of comparison, the ZnO film deposited directly on SiC was also studied. SiC has the least divergence in the lattice spacing with ZnO (≈ 5 %) and is considered to be one of the best backing materials used for the deposition of zinc oxide films.

The normalized CL spectra of the above described samples are shown in Fig. 4. The spectra were normalized in order to present them in one figure as the CL intensities for the films significantly differed. The CL spectrum of each film was normalized to the maximal value of the intensity in it.

In order to estimate the relation of the EL and DLE contributions, we introduced the quality coefficient S that was determined as $S = I_{\lambda(\mathrm{EL})}/I_{\lambda(\mathrm{DLE})}$, where $I_{\lambda(\mathrm{EL})}$ is the integral intensity of the EL emission peak and $I_{\lambda(\mathrm{DLE})}$ is the integral intensity of the DLE peak. For the samples that had no visible luminescence, $I_{\lambda(\mathrm{DLE})}$ was calculated as the area under the curve of the background signal in the range 450–620 nm. The numerical data for the analysis of the nonnormalized CL spectra are given in the Table. Based on the comparison of such data, one can prove that the most promising UV emissive material for the creation of optoelectronic devices is ZnO/SiN$_x$:H/Si ($S \approx 42$). ZnO/SiN$_x$:H/Si annealed by the FTFA method can be considered as the best material with "green" luminescence ($S \approx 0.07$). As one can see from Table, the ZnO/SiC sample demonstrates the presence of DLE close to 512 nm and EL with the maximum at 380 nm in spite of the fact that SiC is one of the most appropriate substrates for ZnO with a similar hexagonal structure, low mismatch of the lattice spacings and thermal expansion coefficients. In contrast, the ZnO/SiN$_x$:H/Si samples deposited at the temperatures of the substrate of 300 and 350 °C and having a low-temperature buffer layer (LTBL) demonstrated only EL with the maximum at 380–381 nm in the absence of luminescence in the visible-light region. These results give reasons to suppose that the SiN$_x$:H interlayer is the determinant in the behavior of luminescence. We consider that hydrogen qualitatively influences the luminescence intensity of ZnO. As was reported in [20], SiN$_x$:H layers were used in the production of solar cells for the hydrogen passivation of defects. In our case, we consider that H diffuses into the ZnO film during its growth and passivates defects responsible for the visible luminescence. The role of hydrogen in the suppression of visible luminescence was discussed earlier for the cases of deuterium implantation [27] and hydrogen plasma treatment [28, 29].

In many works dealing with the investigation of "green" luminescence, it was reported that the DLE

CL properties of the ZnO films

Substrate	Deposition temperature T_s, °C (annealing)	EL			DLE			$S_{\mathrm{EL/DLE}}$
		$I_{\max}$, rel.un.	$\lambda_{\max}$, nm	FWHM, nm	$I_{\max}$, rel.un.	$\lambda_{\max}$, nm	FWHM, nm	
SiC	350	70	380	29	23	512	173	0.73
SiN$_x$:H/Si	350	14300	380	14	–	–	–	42
SiN$_x$:H/Si	300	90	381	33	–	–	–	2.96
SiN$_x$:H/Si	300–500 (LTBL)	140	380	22	–	–	–	2.53
SiN$_x$:H/Si	350 (FTFA at 850 C°)	230	382	21	600	533	127	0.07
SiN$_x$:H/Si	350 (ITA at 850 C°)	97	381	25	150	536	116	0.16

Notations: $I_{\max}$ – maximum peak intensity; $\lambda_{\max}$ – maximum peak wavelength; FWHM – half-width; $S_{\mathrm{EL/DL}}$ – quality coefficient.

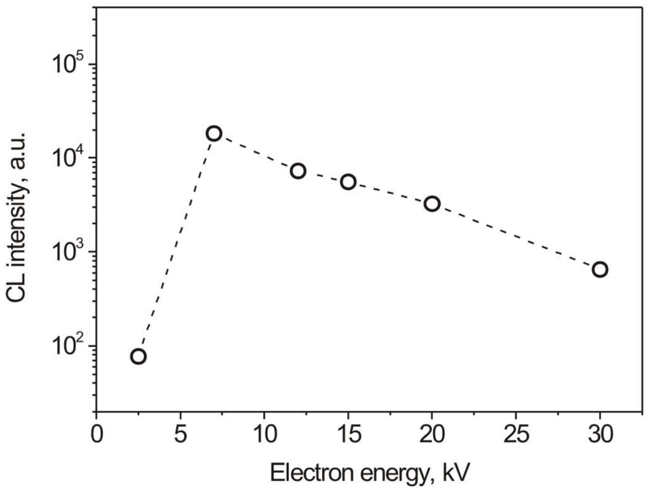

Fig. 5. Dependence of the intensity of the exciton CL of the ZnO/SiN$_x$:H/Si(110) films on the electron energy

arises due to the presence of Zn$_i$ and/or V_O, which still remains a debatable question [4, 30–32]. The authors of [28] assumed that the hydrogen doping results in the charge compensation of acceptors and ionized donors. Possibly, a shift of the Fermi level in the case of hydrogen doping results in a change of the charge states of the ions of proper defects, V_O. According to [33], hydrogen represents a shallow donor in ZnO. This theory agrees with the results of the electric investigations: we observed a low specific resistance in the samples with the best optical properties [18]. In order to inspect the influence of hydrogen and the reversibility of the given effect, the ZnO/SiN$_x$:H/Si samples were annealed during 90 min at 850 °C using the ITA and FTFA methods (T_s = 350 °C). Under the given conditions, there can take place the liberation of hydrogen, which must influence the optical properties. Indeed, the diffusion of hydrogen from SiN$_x$:H at a temperature higher than 500 °C was reported in [20, 34]. After the annealing, the luminescence spectrum of the ZnO films is absolutely changed: in the ZnO/SiN$_x$:H/Si films annealed by the ITA method, there appeared an intense wide DLE band with the center at 536 nm, and the same intense DLE band with the center at 533 nm was observed in the luminescence spectrum of the sample annealed with the help of the FTFA method. We explain it in the following way: the annealing temperature and time are sufficient for the active release of hydrogen from the ZnO films. Loosing hydrogen, the ZnO films again radiate in DLE even in spite of the possible improvement of the structure due to the thermal annealing.

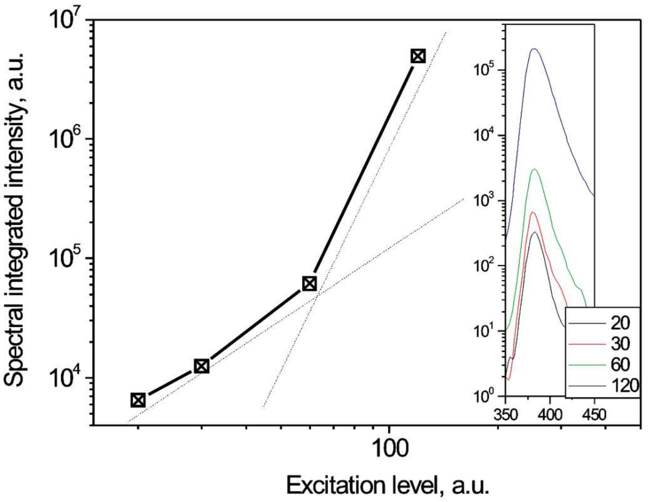

Fig. 6. Dependences of the integral intensity of the exciton CL on the excitation power of the ZnO/SiN$_x$:H/Si film

In order to obtain additional information on the luminescence of the most interesting samples, we investigated the intensity of the exciton CL depending on the electron energy (Fig. 5). We obtained the data on the thickness uniformity of the optical properties of the material, as the depth of the electron penetration is proportional to the electron energy E. This calculated depth amounted to ∼ 100 nm at 10 keV [25]. At a low electron energy (2.5 keV), the CL intensity is significantly lower, which can be explained by the essential influence of nonradiative surface recombination. With an increase in the penetration depth of the electron beam, the luminescence intensity increases and reaches a maximum at $E \sim 7$ keV. As one can see in the SEM image (Fig. 3), the structure of the ZnO film becomes worse with the approximation to the film/substrate interface, which is accompanied with the further decrease of the CL intensity. This result can be explained by the influence of structural defects and the DLE absorption. In addition, a part of the radiation is spent in the material due to the self-absorption of the exciton line [25].

The possibility of the stimulated radiation of the ZnO films was studied by investigating the dependence of the integral CL intensity on the excitation power in the range 0.25–12.5 MW/cm^2 (Fig. 6). As one can see from the figure, the curve has two regions with different slopes depending on the level of excitation. At low excitation levels, this dependence is linear, whereas at the density of the excitation power of ∼2.5 MW/cm^2, it becomes nonlinear. If the edge emission linearly depends on the excitation level, this means that it is conditioned by the exciton recombination [35,36]. The nonlinear increase of the emission intensity can take place in the case of the contraction of the peak with increase in the excitation intensity, which represents an indication of stimulated luminescence and laser generation [37]. The laser effect is mainly determined by two factors: the optical gain coefficient of the active medium and the geometry of laser resonators [35]. In order to make conclusions on the laser properties of our samples, further detailed investigations should be performed.

4. Conclusions

The most important results of this work can be formulated in the following way:

The best UV photoluminescence was obtained in the ZnO films deposited on SiN_x:H/ Si at 350 °C. It is suggested that the intensity of "green" luminescence decreases due to the diffusion of hydrogen into the ZnO film.

The dependence of CL on the penetration depth of the electron beam allowed us to estimate the thickness uniformity of the optical properties of the films, which is important for the production of UV light-emitting devices on the basis of ZnO. The integral intensity of cathodoluminescence has a nonlinear character at the density of the excitation power higher than 2.5 $\mathrm{MW/cm}^2$, that has attributes of stimulated emission in the ZnO films deposited on the Si substrate by the PEMOCVD method with the use of a SiN_x:H buffer layer.

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ВПЛИВ ТЕХНОЛОГIЇ ОСАДЖЕННЯ ТА СТРУКТУРИ ПЛIВОК ZnO НА ЇХ ФОТО- ТА КАТОДОЛЮМIНЕСЦЕНЦIЮ

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Резюме

Дослiджено фотолюмiнесценцiю (ФЛ) плiвок ZnO, осаджених методом PEMOCVD у рiзних технологiчних умовах на пiдкладки з кремнiю та SiC. Найiнтенсивнiша ультрафiолетова (УФ) ФЛ спостерiгалася на плiвках оксиду цинку, вирощених на Si при температурi 350 °C з використанням буферного шару SiN_x:H. Висловлено припущення, що iнтенсивнiсть "зеленої" люмiнесценцiї зменшується внаслiдок дифузiї водню у плiвку ZnO. Методом катодолюмiнесценцiї (КЛ) оцiнено однорiднiсть оптичних властивостей по товщинi плiвок, що є важливим для створення УФ-свiтловипромiнюючих приладiв на основi ZnO. Спостерiгалася надлiнiйна поведiнка iнтегральної iнтенсивностi КЛ як функцiї потужностi збудження.