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ФОТОЛЮМИНЕСЦЕНЦИЯ ЭПИТАКСИАЛЬНЫХ СЛОЕВ $Al_xGa_{1-x}P$, ВЫРАЩЕННЫХ МЕТОДОМ ЖИДКОФАЗНОЙ ЭПИТАКСИИ

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Аннотация. Исследованы эпитаксиальные слои $Al_xGa_{1-x}P$ (x = 0,06–0,61), выращенные на подложках GaP путем кристаллизации из расплавов-растворов на основе индия в интервале температур 975–950 °С. Толщина слоев варьировалась в диапазоне 3–19 мкм. Элементный анализ проводился с помощью локального рентгеновского зондового микроанализа. Измерение спектров фотолюминесценции при температуре 4,2 К дало следующие результаты. В диапазоне 2,0–2,4 эВ во всех спектрах исследованных образцов $Al_xGa_{1-x}P$ наблюдался ряд полос. В случае увеличения концентрации алюминия они смещались в область высоких энергий. При содержании в расплаве-растворе алюминия в количестве 0,16 мас. % наиболее интенсивная полоса имела максимум в области 549 нм, что соответствует зеленому цвету излучения. Вероятно, эти полосы были вызваны рекомбинацией донорно-акцепторных пар. Легирование эпитаксиальных слоев цинком и магнием осуществлялось диффузионным путем. В эпитаксиальных слоях AlGaP были обнаружены микрочастицы GaP размером до 4 мкм. Показана возможность легирования эпитаксиальных слоев AlGaP азотом путем добавления в расплав P_3N_5. Сделан вывод о том, что легирование эпитаксиальных слоев $Al_xGa_{1-x}P$ азотом и серой происходит путем автодиффузии этих примесей

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с подложки ввиду их присутствия в частицах GaP. Легирование эпитаксиальных слоев AlGaP редкоземельным элементом гадолинием путем его введения в расплав-раствор, а также легирование этих эпитаксиальных слоев цинком посредством диффузии не привели к какому-либо изменению спектров фотолюминесценции в диапазоне 2,0–2,4 эВ. В эпитаксиальных слоях AlGaP, легированных магнием путем диффузии, наблюдалась широкая полоса с максимумом вблизи 1,99 эВ. Установлено, что заметное загрязнение эпитаксиальных слоев $Al_xGa_{1-x}P$ кислородом при жидкофазной эпитаксии отсутствует. Проанализированы изменения фотолюминесценции, которые вызваны излучательной рекомбинацией на глубоких дефектах и примесях, внесенных в слои при различных обработках.

Ключевые слова: соединения AlGaP; эпитаксиальные слои; фотолюминесценция; жидкофазная эпитаксия; редкоземельные элементы; акцепторные примеси.

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PHOTOLUMINESCENCE OF $Al_xGa_{1-x}P$ EPITAXIAL LAYERS GROWN BY LIQUID-PHASE EPITAXY METHOD

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Abstract. $Al_xGa_{1-x}P$ epitaxial layers ($x = 0.06–0.61$) grown on GaP substrates by crystallisation from indium-based melt-solutions in the temperature interval of 975–950 °C were investigated. The thickness of layers varied in the range of 3–19 μm. Elemental analysis was made using local X-ray probe microanalysis. Measurement of the photoluminescence spectra at temperature of 4.2 K gave the following results. A number of bands were observed in the range of 2.0–2.4 eV in all spectra of the studied $Al_xGa_{1-x}P$ samples. With an increase in aluminium concentration, they shifted to the high-energy region. With an aluminium content in the melt-solution in the amount of 0.16 wt. %, the most intense band had a maximum at 549 nm, which corresponds to the green colour of the radiation. These bands were probably caused by the donor – acceptor pairs recombination. Doping of the epitaxial layers with zinc and magnesium was carried out by diffusion. GaP microparticles up to 4 μm in size were detected in the AlGaP epitaxial layers. The possibility of doping the AlGaP epitaxial layers with nitrogen by adding P_3N_5 to the melt was demonstrated. It was concluded that doping of $Al_xGa_{1-x}P$ epitaxial layers with nitrogen and sulfur occurs through autodiffusion of these impurities from the substrate due to their presence in GaP particles. Doping of AlGaP epitaxial layers with the rare-earth element gadolinium by introducing it into the melt-solution as well as doping these epitaxial layers with zinc by diffusion did not result in any changes in the photoluminescence spectra in the range of 2.0–2.4 eV. The broad intensive band with maximum near 1.99 eV was observed in AlGaP epitaxial layers doped with magnesium by diffusion. It was established that noticeable contamination of $Al_xGa_{1-x}P$ epitaxial layers with oxygen during liquid-phase epitaxy is absent. Changes in photoluminescence caused by radiative recombination on deep defects and impurities introduced into the layers during various processing stages were analysed.

Keywords: AlGaP compounds; epitaxial layers; photoluminescence; liquid-phase epitaxy; rare-earth elements; acceptor impurities.

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Introduction

The human eye sees light spanning roughly the wavelength range of 400–700 nm with most sensitivity appearing at the centre of this interval. Green colour is where human eyes' sensitivity peaks. This has important implications when it comes to developing artificial light sources for illuminating human living and working environments. However, whereas emitters in the red and blue-violet regions have been relatively easy to develop, green emitters have presented considerable challenges. This resulted in a conspicuous shortage of affordable and efficient solid-state green light emitters [1].

An AlGaP alloy is one of the wide-gap semiconductors with zinc blende structure. An addition of Al to GaP enlarges the band gap and shifts the emission luminescence peaks to shorter wavelengths. The band gap in this material can be varied in the range of 2.26–2.45 eV (in wavelength of 550–506 nm). This makes it possible to produce optoelectronic green emitters devices. In addition, the use of AlGaP – GaP heterojunctions makes it possible to increase the efficiency of radiative recombination and improve the conditions for light output due to the change in absorption in the near-surface layer [2; 3]. However, since the covalent radius of the aluminium atom significantly differs from the size of the main atoms of the GaP crystal lattice, therefore, there are difficulties in obtaining AlGaP ternary compounds with a uniform distribution of aluminium impurity [4; 5]. There are only a few reports on the production of AlGaP ternary compounds with an even distribution of aluminium impurity. Despite extensive studies of A_3B_5 ternary alloys, there is little information about the properties of the AlGaP – GaP heterojunctions. It should be noted that properties of AlGaP epitaxial layers grown from indium-based melt-solutions on the practically have not been investigated. Although it is well known that the compounds obtained by liquid-phase epitaxy have the higher quantum output of photoluminescence compared with the compounds obtained by other methods of epitaxial growth. Keeping this in mind, it is of great interest to study the structural and optical properties of the AlGaP epitaxial layers on the GaP substrates grown by liquid-phase epitaxy method in order to create green light emitting diodes.

Experimental part

The purpose of the present work have been to study the $Al_xGa_{1-x}P$ epitaxial layers ($x = 0.06-0.61$) grown on the (111)B-oriented GaP substrates doped with sulfur (GaP : S) by the crystallisation from indium-based melt-solutions in the temperature interval of 975–950 °C. The cooling rate of the solutions was varied in the range of 0.5–4.0 °C/min. The thickness of the epitaxial layers varied in the range of 3.5–10.0 μm. It was determined by the spherical grinding method. In all the epitaxial layers, the percentage of indium (an isovalent background impurity) was approached its solubility limit in GaP (~1 %) [6]. Rare-earth element gadolinium was introduced into melt-solutions for gettering the technological impurities [7]. P_3N_5 has been used for doping by nitrogen. Doping by zinc and magnesium acceptor impurities have been realised by diffusion at temperature of 600–650 °C during 1–4 h.

The photoluminescence (PL) measurements were carried out at temperature of 4.2–300.0 K [7]. Optical excitation was carried out by a DKsEl-1000 xenon arc lamp or Ar^+-laser (wavelength was 514 nm, power was 100 W). InGaAs PIN photodetectors were used as a receiver of recombination radiation. The lock-in nanovoltmeter type 232B (*Unipan*, Poland) was used for impedance matching of the latter signal and narrowband low-frequency signal from amplifier. Amplification was performed to the signal modulation frequency (~16 Hz) of light beam that was determined by the rotational speed of the mechanical chopper. The PL spectra were detected from the illuminated side of samples. The aluminium content was determined by local electron probe microanalysis on the plant NanoLab 7 (*Opton*, Germany) (diameter of electron probe was 4 μm) along the corresponding X-ray lines.

Results and discussions

Parameters of the investigated samples of $Al_xGa_{1-x}P$ epitaxial layers are presented in the table. The series of Z, Z_1 and Z_2 bands was observed in all PL spectra of $Al_xGa_{1-x}P$ epitaxial layers at irradiating by ultraviolet part of xenon lamp emission (fig. 1). Two last bands were photon replicas of Z band. It was established that the impurities content and the regime of epitaxial growth were exerted influence on their energy position (see table) and wide ($\Delta E = 50-100$ meV). These bands were displaced to high energy region at the increase of aluminium content (samples No. 2 and 3). With an aluminium content in the melt-solution in the amount of 0.16 wt. %, Z band has a maximum at 549 nm (sample No. 2), which corresponds to the green colour of the radiation. The zinc diffusion (sample No. 4), magnesium diffusion (sample No. 6) (fig. 2) and doping of nitrogen (sample No. 7) intensified this effect. The considerable broadening of Z band was observed later. The intensity of this band strongly decreased at the increase of measurement temperature and it did not observe in PL spectra at $T_{reg} > 100$ K. Probably, Z band is caused by the donor – acceptor pair (DAP) recombination. Its shift into high energy region concerning DAP band in GaP (see fig. 1) is associated with the increase of the band gap at aluminium addition into melt-solutions. The absence of DAP recombination narrow lines on its background is caused by the deformation broadening of this lines.

At the irradiating by laser with wavelength of 488 nm the PL intensity in the energy range of 2.1–2.4 eV increased in all investigated samples (see fig. 1). However, in this case typical for GaP : S lines dominate in the spectra [8]: bound exciton recombination of sulphur (NP_S) and nitrogen (NP_N) as well as a series of narrow lines against on the background of the broad band caused by DAP recombination. It is indicated that in this case the GaP substrate gave the main contribution into the luminescence.

Parameters of the investigated samples of $Al_xGa_{1-x}P$ epitaxial layers

Number of sample	Aluminium content in the melt-solution, wt. %	Method of doping epitaxial layers		Energy position of Z band	
		Introducing into melt-solution	Diffusion	*E*, eV	λ, nm
1	0.07	–	–	2.237	554
2	0.16	–	–	2.258	549
3	0.61	–	–	2.250	542
4	0.41	–	Zn	2.290	541
5	0.31	Gd	–	2.269	546
6	0.46	Gd	Mg	2.280	543
7	0.30	P_3N_5	–	2.295	540
8	0.06	P_3N_5	–	2.260	548

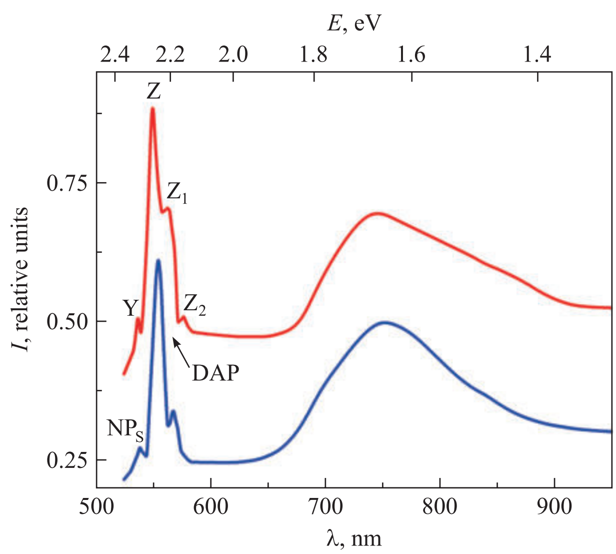

Fig. 1. PL spectra of $T = 4.2$ K of the sample No. 1 of AlGaP epitaxial layers at the irradiating by xenon lamp (red curve; for clarity, its spectral intensity has been doubled) or laser (blue curve) with the epitaxial layer side

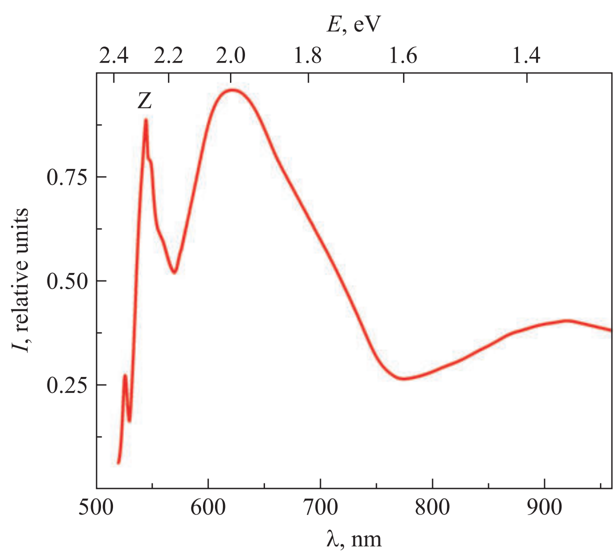

Fig. 2. PL spectra of $T = 4.2$ K of the sample No. 6 of AlGaP epitaxial layers at the irradiating by xenon lamp with the epitaxial layer side

It should be noted that in some spectra the weak Y band was observed (see fig. 1). It is situated 70 meV upward in energy from Z band. Its maximum intensity was observed at an aluminium content of 0.16 wt. % (sample No. 2). With a further increase in the aluminium content, the width of the Y band increases, while its intensity decreases. When the aluminium content in the melt-solution is 0.61 wt. % (sample No. 3), it was not observed in the PL spectrum. Most likely, this is a band of NP_S or NP_N centres in the AlGaP epitaxial layers.

In the $Al_xGa_{1-x}P$ epitaxial layers, the broad unstructured band was registered in spectral range of 700–900 nm (energy range of 1.75–1.35 eV). The same band was observed in the GaP spectra [8]. The spectral form and intensity of this band depended on the regime of epitaxial growth and the doping. This band is described by a superposition of the bands with maxima near 1.53; 1.69 and 1.85 eV and weak band in the region of 1.35 eV. At T_{reg} = 4.2 K maximum of this band was near 1.70 eV (prevalence 1.69 eV band). At the temperature increase the intensity of 1.69 eV band was decreased as well as in GaP [9]. With an increase in temperature, maximum of broad unstructured band displaced to longer wavelengths and only 1.35 and 1.53 eV bands were observed the PL spectra at 300 K.

The absorption lines were registered on the background of the broad Z band in the $Al_xGa_{1-x}P$ epitaxial layers (fig. 3). Their energy position were corresponded to free (2.327 eV) exciton recombination as well as bound exciton recombination on nitrogen (2.318 eV) and sulfur (2.310 eV) centres in GaP spectra. The wide of these bands was varied in the range of 2.0–3.5 meV. Probably, those lines are caused by absorption of radiation on GaP particles in the $Al_xGa_{1-x}P$ epitaxial layers. Earlier it has been reported on forming of GaP clusters in the $Al_xGa_{1-x}P$ epitaxial layers grown by the crystallisation from gallium melt [10]. Electron probe microanalysis shown that aluminium was placed relatively homogeneously. Thus, the GaP particles size at least should be less than the diameter of the electron probe of 4 μm. Free or bound exciton luminescence from GaP particles is not observed due to intensive non-radiative recombination at defects localised within GaP particles and their boundaries.

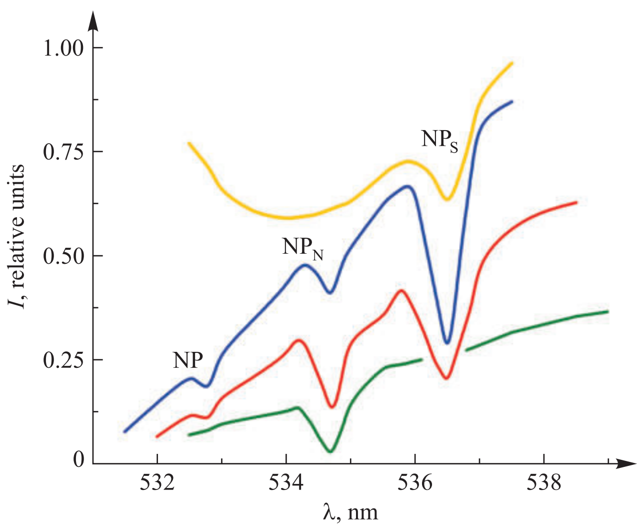

Fig. 3. Lines caused by absorption on GaP particles in the sample No. 6 (yellow curve), sample No. 7 (blue curve), sample No. 4 (red curve) and sample No. 2 (green curve) of AlGaP epitaxial layers

The comparative analysis of PL spectra of substrate, the absorption lines on GaP particles and the condition of the epitaxial growth showed that the selfdoping of epilayers by the nitrogen and sulfur from the substrate takes place at the liquid phage epitaxy. A correlation has been established between the concentration of impurities in the substrates and the intensity of the corresponding absorption lines on the GaP particles.

In the research [11], it was shown that aluminium introduction into the gallium melt-solution leads to effective gettering of the nitrogen impurity. The opportunity of the doping of AlGaP by the nitrogen at the P_3N_5 addition into the indium-based melt-solutions were founded by us. It was revealed that even at $x = 0.47$ in the PL spectra of the $Al_xGa_{1-x}P$ epitaxial layers a nitrogen absorption line was observed (see fig. 3). It indicates on availability of nitrogen impurity into epitaxial layers (at least in GaP particles).

The broad intensive band with maximum about 1.99 eV was observed in AlGaP epitaxial layers doped with magnesium diffusion (see fig. 2). The authors of the study [12] found that the 2.06 eV band was observed in specially undoped *n*-type GaP monocrystal and it was assigned to $Si_{Ga} - Si_P$ DAP recombination. Probably, in this case, contamination of AlGaP epitaxial layers with technological dopant of silicon by self-diffusion from the GaP substrate occurred during diffusion heat treatment.

The introduction of rare-earth element gadolinium into the melt-solution as well as the doping by zinc did not result in the qualitative essential change of PL spectra of $Al_xGa_{1-x}P$ epitaxial layers (1.8–2.4 eV). It is known that Zn – O and Mg – O complexes are caused the PL bands with maxima at 1.76 and 2.10 eV respectively [11; 12]. The absence of these bands in the PL spectra of AlGaP epitaxial layers indicates the absence of noticeable oxygen contamination during liquid-phase epitaxy as well as at diffusion heat treatment.

The availability of AlGaP epitaxial layers was essentially changed the conditions of exit of luminescence from the GaP substrate. The distinction in spectra of substrate at excitation by laser with substrate or AlGaP epitaxial layer side is testified about it. The effect was consisted in the change of ratio of intensity of A and B lines (fig. 4) stipulated by sovalent impurity of nitrogen as well as in reduction of intensity of broad unstructured band with maximum of about 1.7 eV in a GaP substrate. This indicates various options for the location of the impurities in the crystal lattice of the GaP substrate and at the AlGaP and GaP interface.

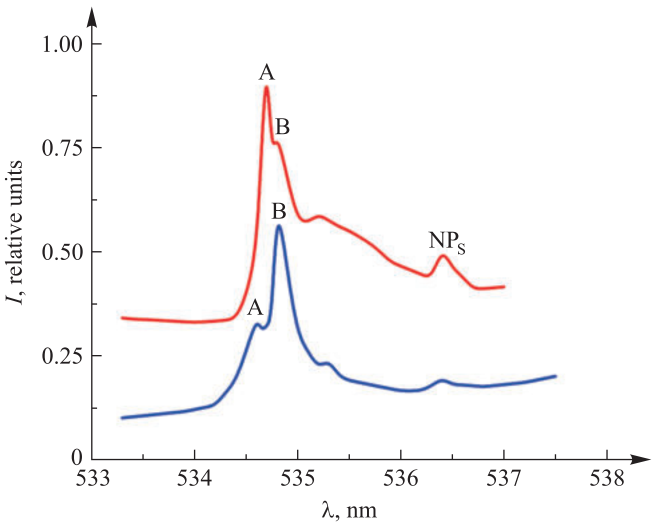

Fig. 4. Spectra of the GaP substrate of the sample No. 6 at the irradiating by the Ar^+-laser with the substrate side (red curve) or epitaxial layer side (blue curve)

Conclusions

It was established that the spectral position of the PL bands responsible for the DAP recombination in the AlGaP epitaxial layers depends on the aluminium content in the indium-based melt-solution. These bands shifted to the region of higher energies as the aluminium content increased. With an aluminium content in the melt-solution in the amount of 0.16 wt. %, the most intense band had a maximum at 549 nm, which corresponds to the green colour of the radiation. Diffusion doping epitaxial layers by zinc and magnesium impurities were enhanced this effect. GaP microparticles were detected in the AlGaP epitaxial layers. Due to intense non-radiative recombination at defects within the GaP microparticles and (or) at their boundaries no free or bound exciton luminescence from the microparticles was no observed. It was revealed that during the growth of AlGaP epitaxial layers they are doped with nitrogen and sulfur due to the autodiffusion of these impurities from the substrate. These impurities are presented in GaP particles. Existence of nitrogen and sulfur impurities in AlGaP epitaxial layers was manifested by bound exciton absorption lines of bound exciton seen on the background of the broad luminescence bands of AlGaP. The possibility of AlGaP doping with nitrogen by adding P_3N_5 to the indium-based melt-solutions was shown. The doping of AlGaP epitaxial layers with rare-earth element gadolinium by the introducing into melt-solution as well as zinc diffusion did not lead to any change of the PL spectra in the range of 2.0–2.4 eV.

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