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ФОТОЛЮМИНЕСЦЕНЦИЯ ЭПИТАКСИАЛЬНЫХ СЛОЕВ GaP, ПОЛУЧЕННЫХ ИЗ РАСПЛАВОВ НА ОСНОВЕ ИНДИЯ

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Аннотация. Исследованы эпитаксиальные слои фосфида галлия, выращенные на подложках GaP и легированные редкоземельными элементами (РЗЭ) Gd и Dy в процессе кристаллизации из расплавов-растворов на основе In в интервале температур 975–670 °C. Концентрация РЗЭ в эпитаксиальных слоях была ниже предела обнаружения рентгеноспектральным анализом (0,01 ат. %). Измерение спектров фотолюминесценции проводилось в диапазоне температур 4,2–300,0 К. В спектрах исследованных образцов наблюдались типичные для монокристаллического GaP линии: линия экситона, связанного на сере и фосфоре, а также серия узких линий на фоне широкой полосы, обусловленной донорно-акцепторной парой с участием примесей углерода и серы. В ближнем инфракрасном диапазоне (1,4–1,8 эВ) присутствовала широкая полоса, обусловленная суперпозицией четырех полос с максимумами вблизи 1,53; 1,69; 1,85 и 1,35–1,40 эВ. Спектральная форма и интенсивность этой полосы зависели от условий роста и легирования. Введение Gd и Dy в расплав-раствор приводило к появлению узкой X-линии ($\lambda = 541$ нм). Ее интенсивность возрастала с увеличением концентрации РЗЭ в расплаве. Интегральная интенсивность фотолюминесценции во всей исследуемой области длин волн также возрастала при добавлении РЗЭ в расплав-раствор. Скорее всего, это связано с увеличением времени жизни неравновесных носителей заряда в GaP : РЗЭ. Упомянутая X-линия наблюдалась и в особо чистых слоях GaP. Однако ее интенсивность была значительно ниже, чем в эпитаксиальных слоях GaP : РЗЭ. Экспериментальные данные объясняются геттерированием в расплаве донорных примесей (S, Se, Te) и образованием дефектов акцепторного типа (предположительно, V_P или Ga_P) в эпитаксиальных слоях фосфида галлия при введении в расплав РЗЭ.

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PHOTOLUMINESCENCE OF GaP EPITAXIAL LAYERS OBTAINED FROM INDIUM-BASED MELTS

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Abstract. In the present work, we investigate epitaxial layers of gallium phosphide grown on GaP substrates and doped by rare-earth elements (REE) Gd and Dy in the process of crystallisation from the melt-solutions on the base of In in the temperature interval of 975–670 °C. REE concentration in the epitaxial layers was below the detection limit by X-ray spectral analysis (0.01 at. %). Photoluminescence spectra were measured in the temperature range of 4.2–300.0 K. In the spectra of the studied samples, typical for single-crystal GaP lines were observed: line of exciton bound on sulphur and phosphorus, as well as a series of narrow lines on the background of a broad band due to the donor – acceptor pair involving carbon and sulphur impurities. In the near-infrared range (1.4–1.8 eV), a broad band due to the superposition of four bands with maxima near 1.53; 1.69; 1.85 and 1.35–1.40 eV was observed. The spectral shape and intensity of this band depended on the growth and doping conditions. The introduction Gd and Dy in melt resulted in occurrence of narrow X line ($\lambda = 541$ nm). Its intensity increased with the concentration increase of the REE in the melt. The photoluminescence intensity in all investigated region of waves lengths increased also with the addition of the REE in the melt-solution. This is most likely due to the increase in the lifetime of non-equilibrium charge carriers in GaP : REE. The mentioned X line was also observed in especially pure GaP layers. Then its intensity was considerably lower than in GaP : REE. The experimental data are explained by gettering in the melt of donor impurities (S, Se, Te) and formation of acceptor-type defects (presumably V_P or Ga_P) in epitaxial layers of gallium phosphide when REE are introduced into the melt.

Keywords: gallium phosphide; epitaxial layers; rare-earth elements; photoluminescence; crystallisation from the melt-solutions.

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Introduction

The interest to the GaP is caused by wide application of this semiconductor in the production of diodes for the visible region and multi-junction solar cells [1–5]. Rare-earth doped semiconductors exhibit sharp intra-*f* optical transitions and it has become apparent that in wide-band materials such as GaN and SiC, or in Si nanocrystals, the luminescence persists to room temperature. This has naturally led to an increased interest in the dopants in these materials [5; 6]. III–V semiconductors doping by rare-earth element (REE) Er have sharp and temperature-independent 4*f*-intrashell emission near 1.55 μm. It determines the prospects for their use in silica-based optical fibres with the lowest attenuation wavelength [2; 4].

The mechanism of the processes which occur when III–V monocrystal doped with the REE impurities is still open. For example, the authors of works [6; 7] assert, that in monocrystal GaP : REE the introduction of additional acceptor defects takes place. Another viewpoint is that the gettering of donor impurities by REE have been discussed [8; 9]. It should be noted that properties of GaP epitaxial layers grown from the melt with the doping of REE have not been investigated. Keeping this in mind, it is of great interest to study the electrical and recombination activity of the REE in GaP epitaxial layers.

Experimental part

The experiments were carried out on epitaxial layers of gallium phosphide (up to 15 μm thick) grown on GaP substrates. The epitaxial layers have been doped by REE Gd and Dy (GaP : REE) in the process of crystallisation from the melt-solutions on the base of In in the temperature interval of 975–670 °C. The cooling rate of the solution was varied in the range of 0.5–4.0 °C/min. The concentration of the REE in the melt did not exceed 0.2 wt. %. A part of samples has been grown from the melt treated by a high temperature (900 °C, 33 h) vacuum treatment. The photoluminescence (PL) measurements were carried out at 4.2–300.0 K. Optical excitation was carried out by a DKsEl-1000 xenon arc lamp. InGaAs PIN photodetectors were used as a receiver of recombination radiation. Then 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.

Results and discussion

The concentration of the REE in the epitaxial layers was lower than the limit of detection by X-ray spectral analysis. Analysis of the capacitance – voltage measurements shows [10] that the doping of the epitaxial layers with shallow impurities is non-uniform over the depth. No electrically active defects with deep levels were discovered in the epitaxial layers (in concentrations $>10^{11}$ cm^3) by capacitance spectroscopy. Doping with REE resulted in a sharp decrease of the concentration of free charge carriers, up to the point of inversion of the conductivity to the hole-type conductivity. It intensified the non-uniformity of the charge-carrier distribution in the epitaxial layer.

The typical GaP lines were observed in the PL spectra of all investigated samples (fig. 1): bound exciton recombination of sulphur (NP_S) and phosphorus (NP_P), as well as a series of narrow lines in the spectral region of 535–565 nm on the background of the broad band caused by the donor – acceptor pair (DAP) (fig. 2) involving the carbon and sulphur impurities [11].

In the near-infrared range (1.4–1.8 eV) the typical wide I band was observed in all PL spectra (see fig. 1). The spectral form and intensity of this band depended on the growth conditions and doping. It is a superposition of bands with maxima near 1.53 eV (I_2), 1.69 eV (I_3) and 1.85 eV (I_4) and weak band in the energy region of 1.35–1.40 eV (I_1), which were observed in GaP : REE (fig. 3). I_2, I_3, I_4 bands were previously observed by the authors of work [12] in GaP single crystals. However, their nature was not established.

The introduction of Dy and Gd into the melt resulted in increased the intensity of all PL lines and the appearance a narrow X line localised at 541 nm (fig. 4). Its intensity increased with increasing of the REE concentration in the melt. In addition, a reduction of the ratio of the intensities NP_S/DAP was also observed (by a factor of ~1.5). On the other hand, a decrease in the intensity of excitation (by a factor of ~4) also resulted in a similar decrease in the intensity ratio NP_S/DAP. Therefore, the introduction of a REE impurity into the melt is identical to increasing the excitation intensity. The addition of REE into the melt would also increase the PL intensity in the entire experimental range of wavelengths (table 1). This is most likely due to an increase in the lifetime of the non-equilibrium charge carriers in GaP : REE (according to our estimates, by almost an order of magnitude).

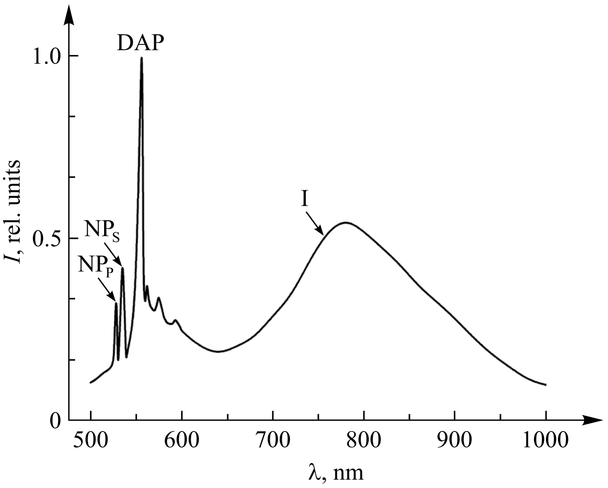

Fig. 1. PL spectra of standard
GaP epitaxial layers at $T = 4.2$ K

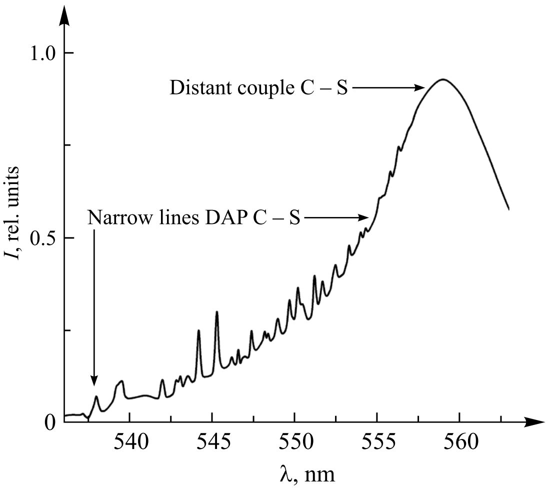

Fig. 2. PL spectra of DAP C – S
in GaP epitaxial layers at $T = 4.2$ K

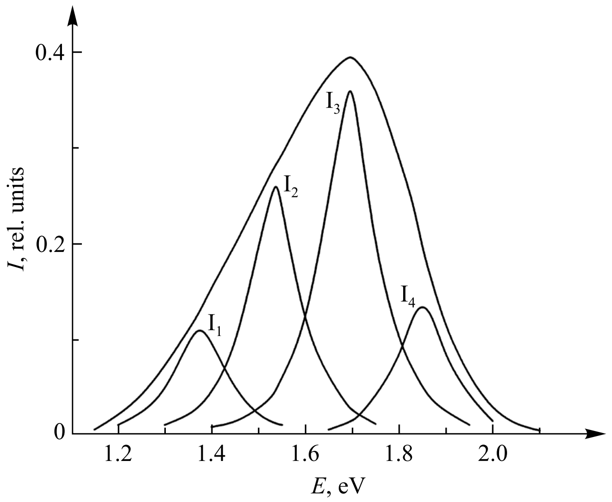

Fig. 3. Decomposition into I band components of GaP : REE epitaxial layers obtained from melt-solutions containing REE in the amount of 0.1 wt. %

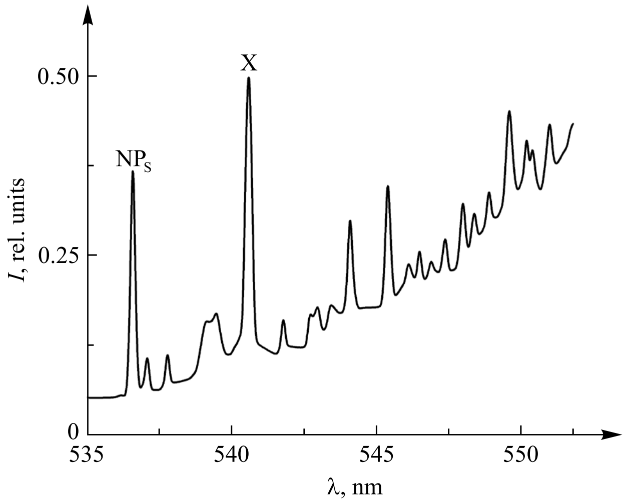

Fig. 4. PL spectra at $T = 4.2$ K of GaP : REE epitaxial layers obtained from melt-solutions with REE content of 0.2 wt. %, as well as ultrapure films obtained from the melt subjected to high-temperature heat treatment

Table 1

PL band intensities of GaP : REE epitaxial layers normalised to the DAP band intensity in control samples

REE content, wt. %	Band intensity, rel. units			NP_S/DAP
	X	DAP	I	
–	–	1.00	0.60	0.41
0.05	0.25	1.10	0.68	0.33
0.10	0.30	1.25	0.70	0.29
0.20	0.33	1.30	0.70	0.27

As the excitation intensity decreased, the intensity of the X band decreased synchronously with NP_S. Their intensity dropped sharply as the measurement temperature increased, and at $T > 100$ K they were not observed (table 2). This suggests that the above band is due to an exciton associated with a structural defect. However, the obtained experimental data do not allow to definitely interpret unambiguously the nature of the above mentioned X band. It can only be asserted reliably that it does not involve the REE, since it was also observed in ultrapure samples prepared from vacuum-annealed melt.

Table 2

Temperature dependences of the normalised intensities of the NP_S and X bands in GaP : REE grown from a melt-solution containing 0.2 wt. % REE

Temperature, K	Band intensity, rel. units	
	NP_S	X
4.2	0.41	0.62
78.0	0.06	0.09
110.0	–	–

In analysing the experimental data it is helpful, in our opinion, to take into account the results of investigations of Si : REE, in which the effect of the lanthanides on the impurity composition of the crystal can be traced unequivocally. In previous studies [13; 14] we determined that the introduction of a REE in concentrations of ~0.1 wt. % into the melt results in the removal carbon and other technological impurities (Au, Cu, etc.), which are effective recombination centres in silicon, from silicon single crystals and epitaxial layers. In addition, the REE in a melt effectively interact with carbon, forming compounds of the type REE_xC_y, which precipitate in the form of slag. It was also found that the REE interact with phosphorus, which is the main shallow impurity in silicon; this results in a lower concentration of the majority charge carriers in *n*-Si.

It is known [9; 11] that REE actively interact with impurities belonging to the group VI of the periodic system (specifically, with sulphur) in the melt. The interactions with the elements of groups II and III are less effective. It leads to decreasing the content of technological impurities in III–V compounds. On the other hand, when the apparatus with the melt is annealed, the volatile impurities evaporate first (in our case sulphur and phosphorus). To take the above in to consideration, we can resume that the decrease of the relation NP_S/DAP in pure samples and GaP : REE is stimulated by the melt purification from the technological impurity S. The increasing of the PL bands intensity determined by the increasing of the lifetime of the non-equilibrium charge carriers due to the REE gettering effect in the melt.

The X band is most likely due to a structural defect. The research of the static magnetic susceptibility of GaP : Dy [15] has shown that microinclusions of Dy interacted with elements of the group V (DyN and DyP). This fact testifies the ability of REE to shifting the stoichiometric equilibrium of the melt in the direction of Ga. It is should lead to generate the defect structure in GaP. The analysis of the experimental results does not exclude that the presence of the X line in the PL spectra is due to defects in V_P and Ga_P.

It is unlikely that the defect responsible for the X line contains carbon, since otherwise the X line in the epitaxial layers of ultrapure GaP would be more strongly expressed than in GaP : REE, which is not in agreement with the experimental data. Long-time heating of the melt should result in dissolution of the graphite apparatus, carbon enrichment of the melt, and an increase in the carbon impurity concentration (DAP band intensity) in the epitaxial layer. However, introduction of the REE lead to the reduction of carbon concentration in the epitaxial layer due to the formation of insolubility slags (such as REE_xC_y) in the melt. We observed a similar effect of carbon gettering by REE in melt during the growth of single-crystal silicon, as well as in obtaining Si epitaxial layers by liquid-phase epitaxy method [9; 14]. Therefore, from what we have said above, the assumption that the increase in the acceptor concentration accompanying doping of III–V compounds with lanthanides is connected with an increase in the carbon solubility for GaP is, in our opinion, not entirely correct. Thus, the participation of carbon impurities in the formation the defect responsible for the X line is improbable.

Conclusions

In the spectra of GaP epitaxial layers grown on gallium phosphide substrates during crystallisation from In-based melt-solutions in the temperature range of 975–670 °C, typical for single-crystal GaP lines were observed: line of exciton bound on sulphur and phosphorus, as well as a series of narrow lines on the background of a broad band caused by the DAP involving carbon and sulphur impurities. In the near-infrared range (1.4–1.8 eV), a broad band due to the superposition of four bands with maxima near 1.53; 1.69; 1.85 and 1.35–1.40 eV. The spectral shape and intensity of this band depended on the growth and doping conditions. The introduction of Gd and Dy into the melt-solution resulted in the appearance of a narrow X line ($\lambda = 541$ nm). Its intensity increased with increasing concentration of REE in the melt-solution. The integral intensity of PL in the whole investigated wavelength region also increased with the addition of REE in the melt-solution. The mentioned X line was also observed in particularly pure GaP layers. However, its intensity was much lower than in GaP : REE epitaxial layers. Content of REE in the epitaxial layers was below 0.01 at. % (limit of detection by X-ray spectral analysis). The totality of the experimental data obtained suggests that the X band is most likely due to an exciton bound on a structural defect (presumably V_P or Ga_P). Based on the experimental results and analysis of literature data, we can conclude that two mechanisms occur simultaneously when REE is introduced into the melt-solution: technological impurities in the melt are removed from the material and additional centres unrelated to REE are introduced.

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