

Features of Formation of $\text{In}_x\text{Ga}_{1-x}\text{N}$ Bulk Layers in the Immiscibility Gap of Solid Solutions ($x \sim 0.6$) by Molecular Beam Epitaxy with Plasma Nitrogen Activation

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In this paper, the features of the formation of bulk InGaN layers with an indium content of $\sim 60\%$ in the immiscibility gap of InGaN ternary solid solutions by the method of molecular-beam epitaxy with plasma nitrogen activation are studied. The structures under study were grown on sapphire substrates, while the epitaxy temperature and the ratio of metal (In + Ga) and activated (atomic) nitrogen fluxes were varied. It has been demonstrated that the rates of thermal decomposition and phase separation for $\text{In}_{0.6}\text{Ga}_{0.4}\text{N}$ ternary solutions depend nonmonotonically on the growth temperature in the range $T_{\text{gr}} = 430\text{--}470^\circ\text{C}$. It is shown that InGaN thermal decomposition processes occur on the growth surface and lead to the appearance of surface phases of metallic In and binary InN, while phase separation leads to the appearance of InGaN phases of various compositions throughout the volume of the deposited InGaN layer. It is shown that, in the temperature range under study, phase separation is determined by surface diffusion, which can be suppressed by growth under highly nitrogen-enriched conditions, which made it possible to obtain homogeneous InGaN layers with an In content of $\text{In} \sim 60\%$ during high-temperature ($T_{\text{gr}} = 470^\circ\text{C}$) growth. It is shown that the suppression of InGaN thermal decomposition processes is decisive in achieving effective interband luminescence of the obtained structures, while the presence of phase separation affects the radiative properties of InGaN layers to a lesser extent, at least in the region of low ($T = 77\text{ K}$) temperatures.

Keywords: indium and gallium nitride, molecular beam epitaxy, photoluminescence, thermal decomposition, spinodal decomposition.

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1. Introduction

Indium-gallium nitride (InGaN) solid solution is one of the most promising materials for wide application in modern telecommunications and information display systems. Due to the possibility of varying the band gap width by changing the InGaN composition, it is possible to create optoelectronic devices operating in the spectral range from ultraviolet to near infrared (IR) region. Currently, the InGaN epitaxial layer growth technology has been successfully developed only for compositions with a high Ga content, on the basis of which optoelectronic devices operating in the blue-green region of the spectrum have been created [1,2]. At the same time, in the IR region of the spectrum, communication systems are interested in devices operating in one of the optical fiber transparency windows (0.85, 1.3 and $1.55\ \mu\text{m}$), and modern information display systems require efficient LEDs operating in the entire visible spectrum.

The advancement of the working spectral range of InGaN-based optoelectronic devices into the red and near-IR spectral regions is difficult due to problems with obtaining high-quality InGaN layers with an $\text{In} > 30\%$

content. The main problems here are: high dislocation density associated with a large mismatch of the lattice parameters InN and GaN ($\sim 11\%$) [3], low decomposition temperature ($\sim 470^\circ\text{C}$ for InN), as well as phase separation (PS) InGaN „of medium“ compositions associated with the thermodynamic instability of this solution at typical epitaxy temperatures [4–9]. There is only one solution to combat thermal decomposition and PD — physical limitation of the kinetics of the processes. It is possible to limit the kinetics of the decomposition and PD process due to a significant decrease in the growth temperature, which makes it possible to obtain homogeneous InGaN solutions, but this leads to a significant degradation of the crystal quality and optical properties of [10–12]. It is possible to suppress the decomposition of group III nitrides at elevated growth temperatures due to growth in highly nitrogen-rich conditions ($V/\text{III} \gg 1$). In particular, it is the ratio $V/\text{III} > 1000$ when growing by gas-phase epitaxy from organometallic compounds that makes it possible to suppress decomposition and grow structures of III-nitrides at elevated temperature and higher crystal quality than by molecular beam epitaxy (MBE) [13]. The phenomenon of PD is directly related to the diffusion and redistribution of

atoms of the growing layer. Both bulk and surface diffusion increase with an increase in temperature. Nevertheless, in the growth method of molecular beam epitaxy with plasma activation of nitrogen, surface diffusion significantly depends on the growth conditions, in particular on the ratio of III/V fluxes. Various authors obtained values for the activation energy of surface diffusion from 1.15 eV (metal-enriched conditions III/V > 1) [14] to 3.7 eV (nitrogen-enriched conditions III/V < 1) [15]. Thus, surface diffusion can be significantly slowed down by growth in highly nitrogen-rich conditions.

Recently, the authors of this work have successfully demonstrated the possibility of forming homogeneous epitaxial InGaN layers with the content of InN \sim 80–50% at temperatures close to the beginning of active decomposition of InN \sim 470°C [16]. Despite the fact that many works have been devoted to the InGaN PD phenomenon (for example, [5,8,9]), there are very few detailed studies of the effect of the growth conditions of MBE with plasma activation of nitrogen on phase separation in InGaN solutions with high and medium In content, as well as on their optical properties.

The purpose of this work is to study the effect of growth parameters on the formation of the InGaN layer (content In \sim 60%) formed away from thermodynamic equilibrium, as well as on its crystal quality, electrical and optical characteristics.

2. Experiment procedure

InGaN layers were grown using the MBE method with plasma activation of nitrogen in the STE 3N3 facility (CJSC „NTO“). Sapphire (Al_2O_3) substrates with a diameter of 2" with orientation [0001] were used, on which high-temperature buffer layers AlN and GaN ($T_{\text{gr}}^{\text{AlN}} = 820^\circ\text{C}$ and $T_{\text{gr}}^{\text{GaN}} = 710^\circ\text{C}$) were sequentially grown with thicknesses of $d_{\text{AlN}} \sim 200$ nm and $d_{\text{GaN}} \sim 700$ nm. Next, the layer $\text{In}_x\text{Ga}_{1-x}\text{N}$ was grown with a thickness of ~ 700 nm with an In content within $60 \pm 3\%$ at significantly lower growth temperatures. The indium and gallium fluxes were $F_{\text{In}} \sim 0.2 \mu\text{m/h}$ and $F_{\text{Ga}} \sim 0.13 \mu\text{m/h}$, respectively with the growth of all structures. The InGaN growth temperature varied ($T_{\text{gr}} = 430 \rightarrow 470^\circ\text{C}$) in the first series of samples with an unchanged ratio of fluxes (III/V \sim 0.85), and the ratio of III/V gradually decreased to 0.65 in the second series of samples with an unchanged $T_{\text{gr}} = 470^\circ\text{C}$. RF Atom Source HD 25 plasma source manufactured by Oxford Applied Research was used to create a stream of activated nitrogen; the nitrogen flow remained unchanged during growth at the level of 2 sccm (standard cubic centimeters per minute), the discharge power of the plasma nitrogen source varied, providing the necessary ratio of the flows of group III atoms to active nitrogen III/V \sim 0.65–0.85. Studies were carried out using X-ray diffraction (XRD) analysis with BRUKER D8 DISCOVER diffractometer, using a SUPRA 50VP scanning electron microscope (SEM), Hall

effect measurements and photoluminescence spectroscopy to characterize the grown structures. The spontaneous PL spectra were measured by pumping structures with a continuous laser with a wavelength of 450 nm and a pumping power density of $\sim 20 \text{ W/cm}^2$ at a temperature of $T = 77 \text{ K}$. Spontaneous radiation was collected from the sample surface and analyzed by a lattice monochromator equipped with a multi-element InGaAs photodetector with a cut-off wavelength of $2.1 \mu\text{m}$. Ion etching Ar^+ was used to study the properties of InGaN layers in thickness, the etching depth was controlled using an optical interferometer TALYSURF CCI 2000 and SEM.

3. Results and discussion

It was found that partial decomposition of InGaN during growth is observed in the study of the first series of samples in which the growth temperature of InGaN $T_{\text{gr}} = 430\text{--}470^\circ\text{C}$ varied with an unchanged ratio of III/V \sim 0.85 even in samples grown at the lowest temperature $T_{\text{gr}} = 430^\circ\text{C}$. This is reflected in the presence in the spectra of X-ray diffraction analysis at $\omega - 2\theta$ scanning, along with a symmetric signal from the InGaN layer, a weak (several orders of magnitude smaller) signal from InN (Figure 1, *a*). It is assumed that, despite nitrogen-enriched growth conditions, part of In is not embedded in the growing InGaN layer and metal In accumulates on the surface. Since in this work T_{gr} is significantly lower than the re-evaporation temperatures of metallic In, all accumulated In remains on the growth surface [14]. The fact that the embedding coefficient In into the growing film ≤ 1 and decreases with increasing growth temperature (decomposition increases) has been reported in various papers [17,18]. Apparently, for $\text{In}_{0.6}\text{Ga}_{0.4}\text{N}$, the growth temperature of $T_{\text{gr}} = 430^\circ\text{C}$ is quite high, the embedding coefficient is significantly < 1 , and nitrogen-enriched conditions with a flow ratio of III/V \sim 0.85 is not enough to suppress decomposition. At the end of growth, during the cooling of the sample, its surface is kept for some time under the flow of active nitrogen, which leads to the formation of the InN phase on the surface of the InGaN base layer. The fact that the InN phase is formed on the surface, and not in the volume of the InGaN layer, will be shown later. The peak from InGaN (Figure 1, *a* — spectrum *I*) at $T_{\text{gr}} = 430^\circ\text{C}$ is symmetrical and homogeneous, and there are no signs of PD in InGaN at this growth temperature.

The signal from InN increases noticeably with an increase in the growth temperature to 450°C , which indicates an increase in the decomposition of InGaN, and at the same time, the main peak from the InGaN layer has a shoulder that indicates PD and the appearance of InGaN phases of different composition (Figure 1). The increase in diffusion with temperature, which leads to the redistribution of In and Ga, PD atoms and the appearance of InGaN phases of different composition, is the expected effect. However, a further increase in the growth temperature to 470°C ,

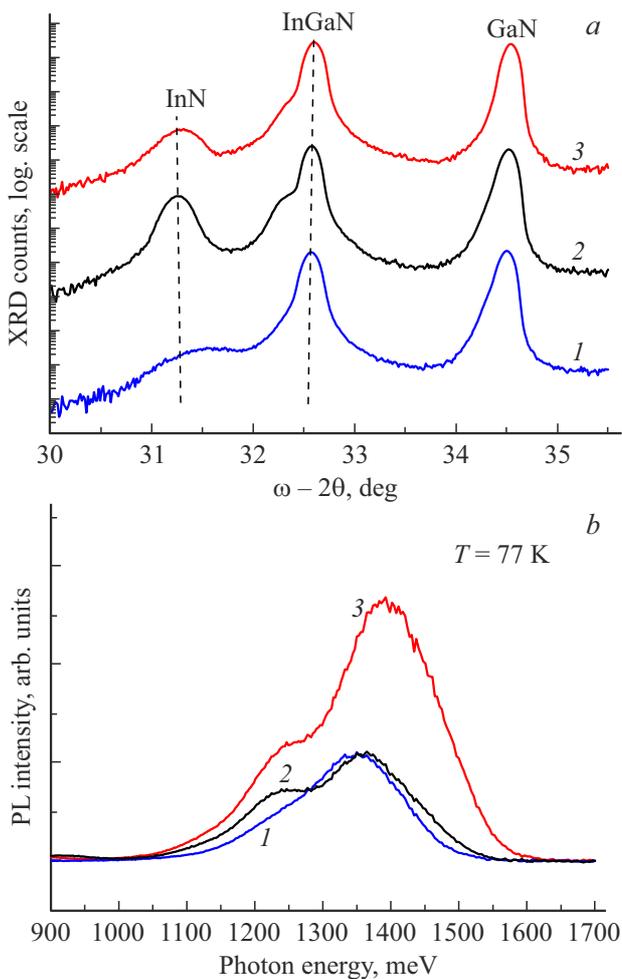


Figure 1. *a* — (0004) $\omega - 2\theta$ X-ray diffraction spectra of samples with InGaN layers grown at the ratio III/V ~ 0.85 and $T_{gr} = 430$ (1), 450 (2) and 470°C (3); the peak from GaN comes from the buffer layer. *b* — spontaneous photoluminescence spectra measured at 77 K from samples 1 (1), 2 (2) and 3 (3) with InGaN layers.

corresponding to the beginning of the active decomposition of the binary InN [4], despite the expected further increase in diffusion, leads to a weakening of the effects of both decomposition and phase separation. A decrease in the intensity of the shoulder of the main InGaN peak indicates a decrease of the effects of PD (Figure 1, *a*). One of the reasons reducing the effects of PD with an increase of T_{gr} may be an approximation to the solidus line on the phase diagram of the InGaN solid solution state, i.e. to the state of thermodynamic equilibrium [5,8,9]. The decrease of decomposition rates can be judged by the decrease in the intensity of the signal from InN with an increase of T_{gr} from 450 to 470°C. Also, the decrease of the amount of accumulated metallic In is confirmed by the time of change of the pattern of reflected high-energy electron diffraction (RHEED). Immediately after the end of InGaN growth, the RHEED shows a dim dot pattern, which indicates a

rough InGaN surface and the presence of metal on the growth surface. The metallic In transforms into InN after exposure to a stream of active nitrogen, and the diffraction becomes bright with the reconstruction characteristic of two-dimensional InN [0001] ($\sqrt{3} \times \sqrt{3}$). The time of change of the diffraction pattern is directly proportional to the amount of accumulated In and decreases from 7 min in the case of growth at $T_{gr} = 450^\circ\text{C}$ to 4 min for $T_{gr} = 470^\circ\text{C}$. Knowing the deposition rate of the nitrogen component ($0.38 \mu\text{m/h}$ for III/V = 0.85), multiplying it by the time of the change of the diffraction pattern (the time of binding of the accumulated metallic In), it is possible to estimate the amount of formed InN ~ 44 nm for $T_{gr} = 450^\circ\text{C}$ and ~ 25 nm for $T_{gr} = 470^\circ\text{C}$. These estimates are estimates from above, since at such high T_{gr} and strong elastic stresses (InN is formed at $\text{In}_{0.6}\text{Ga}_{0.4}\text{N}$), InN is actively decomposed and the rate of InN formation is significantly lower than the deposition rate of the nitrogen component. The decrease in the decomposition rate with an increase of T_{gr} from 450 to 470°C is not obvious, since various papers report a monotonous decrease of the coefficient of In embedding in InGaN with a growth temperature of [17,18], which should lead to an increase of the amount of accumulated In on the growth surfaces with an increase of T_{gr} . It is possible that the decrease in the decomposition rate, as well as the intensity of FR, with an increase of T_{gr} from 450 to 470°C is associated with an approach to the solidus line, i.e. to the state of thermodynamic equilibrium on the phase diagram of the solid solution InGaN [5,8,9].

The decomposition and PD processes occurring in InGaN solid solution are also reflected in the photoluminescence spectra (Figure 1, *b*). In the InGaN sample grown at $T_{gr} = 430^\circ\text{C}$, in which no PD signs are observed in the XRD spectrum (Figure 1, *a*), the photoluminescence signal from InGaN is a single peak (Figure 1, *b*). A low-energy shoulder is observed in InGaN samples obtained at higher temperatures along with the main PL peak. This fully corresponds to the results of the XRD analysis, according to the results of which in these samples, in addition to the main reflection peak (0004) from InGaN with InN content, $\sim 58\%$, a shoulder corresponding to the InGaN phase with InN content, $\sim 65\%$ is observed. It is assumed that a more „narrow-band“ phase with a high InN content is associated with the appearance of a low-energy arm in the PL spectrum (Figure 1, *b*). The integral intensity of PL increases with T_{gr} , which reflects an improvement in the crystal quality of InGaN, a decrease in the density of germinating dislocations and the background electron concentration in InGaN (see Table). The same monotonous increase in the intensity of PL with the growth temperature, even despite the beginning of decomposition, was observed for InGaN layers with a content of InN $\sim 80\%$ up to the beginning of their decomposition [19].

As shown above, an increase in the growth temperature reduces the density of germinating dislocations, the background electron concentration (see Table) and has a positive effect on increasing the photoluminescence intensity

The main parameters of the studied structures with InGaN layers*

Number sample	x_{In} , %	T_{gr} , °C	III/V	N_{D} , 10^{11} cm^{-2}	n_e , 10^{19} cm^{-3}
1	58	430	0.85	2.4	2
2	58	450	0.85	1.8	1.3
3	58	470	0.85	1.3	0.8
4	61	470	0.75	0.6	1.1
5	62	470	0.65	0.6	1.8
6	60	450	0.85	1.2	2

Note*: x_{In} — InN content in InGaN determined by XRD analysis; T_{gr} — InGaN growth temperature; III/V — ratio of flows during InGaN growth; N_{D} — density of germinating dislocations in InGaN, determined from RD analysis; n_e — background electron concentration in InGaN, obtained from Hall effect measurements at 300 K.

of heterostructures with InGaN layers. However, growth at high temperatures can also cause decomposition of the InGaN solid solution PD. A series of structures with $\text{In}_{0.6}\text{Ga}_{0.4}\text{N}$ layers at $T_{\text{gr}} = 470^\circ\text{C}$ with different flow ratios III/V ~ 0.65 – 0.85 were grown to investigate the possibility of suppressing these effects and their effect on the photoluminescence of the samples.

X-ray diffraction analysis revealed that a decrease in the III/V ratio from 0.85 to 0.75 leads to a disappearance in the diffraction spectrum at $\omega - 2\theta$ shoulder scanning at the main peak of reflection from the InGaN layer (Figure 2, a). This indicates a significant suppression of PD in solution $\text{In}_{0.6}\text{Ga}_{0.4}\text{N}$ under these growth conditions. At the same time, a weak signal from InN is still observed, indicating incomplete suppression of decomposition. Only lowering the III/V ratio to 0.65 leads to the complete disappearance of the signal from the binary InN, and at $\omega - 2\theta$ scanning, only a symmetrical peak of reflection from InGaN is observed. Since $T_{\text{gr}} = 470^\circ\text{C}$ is the same in samples 3, 4 and 5, the volume diffusion in them should also not differ. The change in the III/V ratios, as noted in the Introduction, affects only the surface diffusion. With these T_{gr} this diffusion is the main mechanism responsible for the redistribution of In and Ga atoms and leading to PD. The limitation of surface diffusion due to the transition to highly nitrogen-rich growth conditions leads to the suppression of PD and the possibility of obtaining homogeneous layers of InGaN.

Analysis of the PL spectra of samples with InGaN layers grown at $T_{\text{gr}} = 470^\circ\text{C}$ and different III/V ratios showed that suppression of PD with a decrease in III/V from 0.85 to 0.75 leads to a significant (~ 6 times) increase in the intensity of PL (Figure 2, b). This may be partly due to an improvement in the crystal quality of the InGaN layers obtained and a decrease in the density of germinating dislocations (see Table), however, it can be assumed that the presence of a near-surface InN layer plays a major role in the „quenching“ PL of sample 3 relative to samples 4 and 5. This layer introduces both strong absorption of pump radiation during PL measurements and reabsorption of spontaneous radiation (PL) by InGaN, reducing the

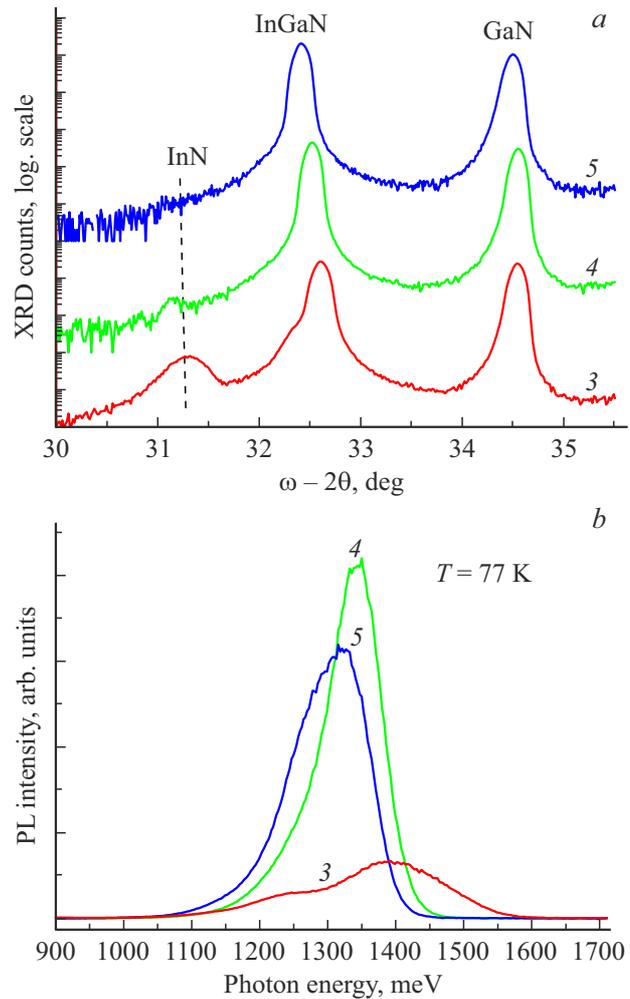


Figure 2. *a* — (0004) $\omega - 2\theta$ X-ray diffraction spectra of samples with InGaN layers grown at $T_{\text{gr}} = 470^\circ\text{C}$ and III/V ratio ~ 0.85 (3), 0.75 (4) and 0.65 (5); the peak from GaN comes from the buffer layer. *b* — spontaneous photoluminescence spectra measured at 77 K from samples 3 (3), 4 (4) and 5 (5) with InGaN layers. (A color version of the figure is provided in the online version of the paper).

efficiency of radiation output from the sample. At the same time, with a further decrease of III/V to 0.65 and complete suppression of both PD and InGaN decomposition, the intensity of PL even decreases slightly, despite a decrease in the density of germinating dislocations (see Table). It is possible that a decrease in the intensity of PL from homogeneous InGaN (III/V ~ 0.65) compared with partially decomposing InGaN (III/V ~ 0.75) is associated with an increase in the background electron concentration and an increase in nonradiative auger recombination in this sample (see Table). As stated by some authors, the presence of excess metallic In on the growth surface prevents the incorporation of various impurities into the growing InN layer, which, being small donors, make the main contribution to the background electron concentration [20].

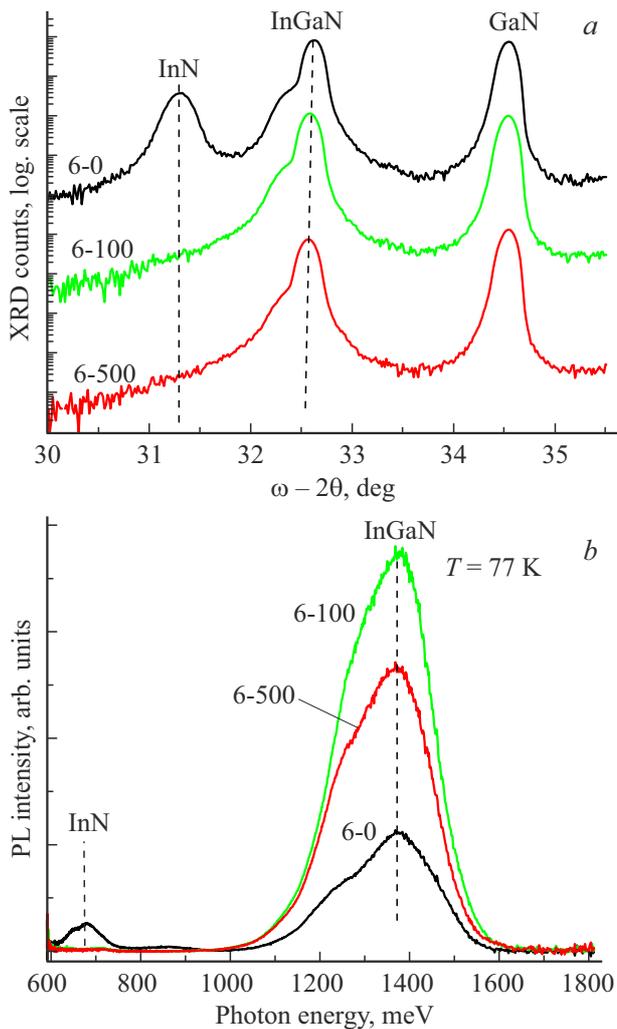


Figure 3. *a* — (0004) $\omega - 2\theta$ X-ray diffraction spectra of sample 6 with an InGaN layer grown at $T_{gr} = 450^\circ\text{C}$ and ratio III/V ~ 0.85 after growth (6-0), after ion etching to a depth of 100 nm (6-100), after ion etching to a depth of 500 nm (6-500). *b* — spontaneous photoluminescence spectra measured at 77 K from samples 6-0, 6-100 and 6-500 from Figure 3, *a*.

Perhaps this leads to a reduced n_e in partially decomposing compared to homogeneous InGaN.

As stated above, the decomposition of InGaN during growth leads to the accumulation of metallic In on the InGaN surface, which forms InN during the exposure of the surface under the nitrogen stream at the end of growth. At the same time, PD leads to the formation of phases of different composition throughout the thickness of the InGaN layer. A sample with a layer $\text{In}_{0.6}\text{Ga}_{0.4}\text{N}$ grown at $T_{gr} = 450^\circ\text{C}$ and a flux ratio of III/V ~ 0.85 was examined to verify these statements, X-ray diffraction analysis was used to observe both decomposition (phase InN), and phase separation (the shoulder of the main InGaN peak, indicating the presence of an InGaN phase of a composition other than nominal) in this sample (Figure 3, *a*). A layer with a thickness of ~ 100 nm on one part of the sample

was removed using ion etching from above the grown heterostructure, and on the other ~ 500 nm at the initial thickness of the InGaN layer ~ 700 nm. As can be seen in Figure 3, after removing the near-surface layer with a thickness of ~ 100 nm in the X-ray diffraction spectrum at $\omega - 2\theta$ scanning, the signal associated with InN disappeared. This suggests that the InN phase is present only in the near-surface region. It is important to note that the shape, position and mutual intensities of both the main peak from the $\text{In}_{0.6}\text{Ga}_{0.4}\text{N}$ -layer and the shoulder almost do not change when etched at ~ 100 and 500 nm. This suggests that the quantitative ratio of phases of different InGaN compositions also does not change, and confirms the idea that phase separation occurs throughout the entire volume of the InGaN layer.

Figure 3, *b* shows the PL spectra of the considered series of samples. So, there is also a PL in the long-wave region characteristic of InN in the initial structure („6-0“), in addition to the main intense response of the InGaN layer. Etched samples do not have such a long-wave PL which also confirms the near-surface formation of InN in case of decomposition of the InGaN layer during growth. At the same time, the intensity of InGaN PL in the sample „6-100“ turns out to be significantly higher than in the sample „6-0“ which can be explained (as before for samples 4 and 5 in comparison with the sample 3) the absence of a highly absorbing near-surface layer InN. With a greater etching depth (~ 500 nm in the sample „6-500“), the intensity of InGaN PL decreases slightly. This may be due both to the fact that the emitting layer itself becomes quite thin (~ 200 nm) and to the increasing defectiveness of InGaN as it approaches the interface with the GaN buffer layer, which enhances the processes of non-radiative recombination.

4. Conclusion

Thus, the features of the formation of bulk layers $\text{In}_x\text{Ga}_{1-x}\text{N}$ using the molecular beam epitaxy method with plasma activation in the region of compositions corresponding to the immiscibility zone of triple solid solutions of InGaN ($x_{\text{In}} \sim 60\%$) are investigated. The manifestations of the effects of decomposition of the deposited InGaN and its phase separation depending on the growth conditions — epitaxy temperature T_{gr} and the ratio of III/V fluxes were studied. It is shown that during the formation of InGaN with an In $\sim 60\%$ content, the occurrence of the binary InN phase is associated with decomposition processes that occur in the near-surface region, while phase separation occurs throughout the entire volume of the InGaN layer; at the same time, the intensities of decomposition and phase separation processes depend nonmonotonically on temperature epitaxies in the range $T_{gr} = 430\text{--}470^\circ\text{C}$. It is demonstrated that in the studied temperature range, phase separation in InGaN layers is determined by surface diffusion, which can be suppressed due to growth in highly nitrogen-rich conditions, which made it possible to obtain homogeneous

InGaN layers. It is confirmed that the suppression of these processes due to a decrease in growth temperatures also allows the formation of homogeneous InGaN layers, while the structural quality of such layers turns out to be low, which is expressed in the low luminescence efficiency of the obtained samples. High-temperature ($T_{gr} = 470^{\circ}\text{C}$) growth in highly nitrogen-enriched conditions is much more effective in this case ($\text{III/V} \sim 0.65$). It is shown that under such conditions homogeneous InGaN layers of significantly better quality are formed, demonstrating intense PL in the near-IR region of the spectrum.

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Conflict of interest

The authors declare that they have no conflict of interest.

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