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Article

Effect of nitrogen pressure on the composition and structure of thin films $GaAs_{1-x-y}N_xBi_y$

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Abstract. Thin films of $GaAs_{1-x-y}N_xBi_y$ were deposited on a GaAs (100) substrate by pulsed laser deposition using an argon-nitrogen gas mixture at a pressure ranging from 1 to 60 Pa. The film thickness is found to decrease from 527 to 127 nm as the pressure of the argon-nitrogen gas mixture increased from 20 Pa to 60 Pa due to reflection and scattering of the plasma torch flow on nitrogen and argon atoms. The increase in pressure results in a significant decrease in the size and density of droplets on the film surface. All samples exhibit a polycrystalline structure, and the film obtained at a pressure of 60 Pa exhibits the highest crystalline perfection. The VASP software package was used to calculate theoretically the diffractogram for a $(2 \times 2 \times 2)$ GaAs_{0.889}N_{0.037}Bi_{0.074} supercell, and it has been observed that the width at half maximum intensity for the GaAsNBi (004) reflection decreases with increasing pressure of the argon-nitrogen gas mixture. The nitrogen concentration in the thin film is found to increase linearly with the increase in the pressure of the argon-nitrogen gas mixture, which was established using X-ray diffraction and photoluminescence methods. The composition of the film obtained at a pressure of 60 Pa is determined to be GaAs_{0.957}N_{0.012}Bi_{0.021}. **Keywords:** thin films, pulsed laser deposition, III–V-N-Bi, GaAs_{1-x-y}N_xBi_y, X-ray diffraction, scanning electron microscopy

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Влияние давления азота на состав и структуру тонких пленок GaAs_{1-x-y}N_xBi_y

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Аннотация. Методом импульсного лазерного напыления в атмосфере аргоно-азотной газовой смеси при давлении от 1 до 60 Па были получены тонкие пленки GaAs_{1-x-y}N_xBi_y на подложке GaAs (100). Установлено, что с увеличением давления аргоно-азотной газовой смеси от 20 до 60 Па толщина пленок снижалась с 527 до 127 нм в следствии отражения и рассеяния потока плазменного факела на атомах азота и аргона. Показано, что увеличение давления способствовало значительному снижению размеров и плотности капель на поверхности пленок. Все полученные пленки имеют поликристаллическую структуру, а наибольшим кристаллическим совершенством обладает тонкая пленка, полученная при давлении 60 Па. Был проведен теоретический расчет дифрактограммы для суперячейки размером $2 \times 2 \times 2$ (64 атома) GaAs_{0.889}N_{0.037}Bi_{0.074} при помощи программного пакета VASP. Величина ширины на половине максимума интенсивности для рефлекса GaAsNBi (004) снижается с ростом давления аргоно-азотной газовой смеси. Установлено, что при повышении давлении азота в тонкой пленке линейно возрастает. Методами рентгеновской дифракции и фотолюминисценции определен состав пленки, полученной при давления аргоно-азотной газовой смеси 60 Па – GaAs_{0.957}N_{0.012}Bi_{0.021}. Ключевые слова: тонкие пленки, импульсное лазерное напыление, III–V-N-Bi, GaAs_{1-x-y}N_xBi_y, рентгеновская дифракция, сканирующая электронная микроскопия

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The development of modern optoelectronics is directly related to the creation of new materials based on III-V, which have optimal optical and electrical properties, in which Auger recombination and intraband absorption would be minimized [1]. It was shown in [2] that in order to minimize Auger recombination, it is necessary to implement the conditions when the energy of spin–orbit splitting (Δ_{SO}) becomes greater than the band gap energy (E_{g}) . This condition can be realized for compounds with III-V-Bi [3, 4], which have a high Δ_{SO} value. To improve the optical and electrophysical properties of III-V, engineering of the band structure of isovalent III-V-N compounds, in particular $GaAs_{1-\nu}N_{\nu}$ [5, 6], is used. Taking into account these facts, it is logical to assume that the joint integration of Bi and N atoms into the III–V lattice will provide the prospect of creating thermally stable optoelectronic devices with a spectral range extended to the mid-IR region. One of the promising III-V-N-Bi compounds for optoelectronic devices is $GaAs_{1-x-y}N_xBi_y$ [7], whose lattice constant can be matched to the lattice constant of the GaAs substrate. Currently, the main methods for obtaining thin $GaAs_{1-x-y}N_xBi_y$ films are molecular-beam epitaxy [8] and APCVD [9]. An alternative to these methods for obtaining thin $GaAs_{1-x-y}N_xBi_y$ films can rightfully be considered the method of pulsed laser deposition (PLD) [10]. In [11], the production of multicomponent InGaAsN films from an InGaAs target in an argon-nitrogen gas mixture by this method is described. It was shown that at a pressure as

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low as 10 Pa, the nitrogen concentration in InGaAsN films on GaAs (100) substrates was 1.9%. By increasing the pressure and using nitrogen activation methods, it is possible to obtain thin films with a controlled nitrogen concentration in the composition. In turn, the concentration of bismuth in thin films during PLD can be controlled by changing the energy density in the laser pulse. Thus, in the case of LSI of $GaAs_{1-x-y}N_xBi_y$ thin films, it is possible to independently control the concentration of nitrogen and bismuth and obtain films that are lattice-matched to GaAs with a ratio between y/x equal to 1.718 [12]. The purpose of this work is to study the dependence of the composition and structure of $GaAs_{1-x-y}N_xBi_y$ thin films on GaAs substrates on the pressure of the argon-nitrogen gas mixture during pulsed laser deposition.

Pulsed laser deposition of thin $GaAs_{1-x-y}N_xBi_y$ films onto a GaAs (100) substrate took place for 120 minutes at a laser pulse energy density of 2.9 J/cm², pulse repetition rate 15 Hz and duration pulse 10 ns from a target with the composition $GaAs_{0.95}Bi_{0.05}$. The target surface was scanned with a laser beam of an area of 5×5 mm². We used laser radiation of the second harmonic of an AYG: Nd³⁺ laser with a wavelength of 532 nm (LOTIS TII LS-2134Y, Belarus). The distance between the target and the substrate was 50 mm, and the substrate temperature was 360°C. The volume of the vacuum chamber was pumped out to 10^{-4} Pa, after which it was isolated and a mixture of highly pure argon and nitrogen was injected to the required pressure using an electronic gas flow regulator RRG-12 (Eltochpribor, Russia). The volume fraction of nitrogen in the argon-nitrogen gas mixture was 80%. The pressure of the argon-nitrogen gas mixture varied from 10^{-3} to 50 Pa. GaAs_{0.95}Bi_{0.05} target. was formed by uniaxial pressing from GaAs powder (99.99%) and chemically pure crystalline bismuth. GaAs_{1-x-y}N_xBi_y thin films were obtained on n-GaAs substrates with a misorientation of 1° between the (100) and (111) planes. A negative electrostatic bias of -300 V was applied to the substrate. The structural properties were studied by X-ray diffraction using an ARL X'TRA diffractometer (Thermo Fisher Scientific, Switzerland) with $CuK_{\alpha 1}$ radiation type (1.54056 Å), equipped with a Goebel parabolic mirror and a thin-film collimator. Studies of the

crystal structure and determination of the preferred orientation of thin films in the Bragg-Brentano geometry (XRD) ($\theta/2\theta$) and grazing small-angle beam incidence (GIXRD) ($\omega/2\theta$) at $\omega = 0.5^{\circ}$. The surface morphology of the films was studied using a MIRA3-LMH scanning electron microscope (SEM) (Tescan, Czech Republic) with an AZtecEnergy Standart/X-max20 elemental composition (EDX) system. Photoluminescence spectra were obtained using a spectrometric complex assembled on the basis of MDR-23 and MDR-41 monochromators (LOMO, Russia). The excitation wavelength was 450 nm, and the luminescence intensity was studied in the wave-

Figure 1 shows SEM images of the surfaces and cleavages of $GaAs_{1-x-v}N_xBi_v$ thin films obtained at

length range 800–1500 nm.

Fig. 1. SEM images of the surface and cleavages of $GaAs_{1-x-y}N_xBi_y$ thin films obtained at a pressure of 1 Pa (*a*, *c*) and 60 Pa (*b*, *d*)

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pressures of 1 and 60 Pa. From Fig. 1, it can be observed that the film obtained at 1 Pa has a textured surface developed with submicron bismuth droplets, and the film structure is formed by grains. The films obtained at a pressure of 60 Pa have a uniform structure and a much smoother surface with practically no droplets. A detailed study of the growth kinetics of $GaAs_{1-x-y}N_xBi_y$ thin films at pressures of 1–60 Pa (Fig. 2) showed that an increase in pressure results in a decrease in film thickness and drop density on their surface. This phenomenon can be attributed to the reflection effect of the plasma plume flow on nitrogen and argon atoms. A significant decrease in the droplet density occurs at 20 Pa, likely due to an increase in the angular diagram of expansion and hindrance to the atomic component of bismuth condensation at the plasma torch expansion stage.



Fig. 2. Dependence of the thickness of $GaAs_{1-x-y}N_xBi_y$ films and the surface density of drops on their surface on the pressure of the argon-nitrogen gas mixture during pulsed laser deposition

Considering that $GaAs_{1-x-y}N_xBi_y$ thin films contain substrate compounds and have a thickness of only 140–530 nm, a qualitative analysis of the concentration ratio of Bi and N - y/x was conducted using the EDX method to determine the chemical composition of the films. An elemental composition analysis of the thin films was carried out under pressure from 1 to 10 Pa. The results showed that the y/x ratio did not change significantly and remained at 3.56. This was likely due to the presence of bismuth droplets on the film surface.

At pressures of 20, 40, and 60 Pa, the ratio of *y* to *x* was 2.9, 2.1, and 1.75, respectively. The change in the ratio y/x occurred due to an increase in the fraction of nitrogen introduced into the thin film, while the concentration of bismuth in the target and the

energy density in the pulse remained constant. The increase in nitrogen concentration in thin film x was almost linear within the pressure range of 20–60 Pa, which is logical considering its activation due to the dissociation of nitrogen molecules during collisions with photoexcited argon atoms, resulting in a sharp increase in the content of atomic and activated molecular nitrogen [13].

Figure 3 shows the X-ray diffraction pattern of a $GaAs_{1-x-y}N_xBi_y$ thin film obtained at a pressure of 60 Pa for the (004) reflection (curve 1). To determine the crystal structure, a theoretical calculation of the diffraction pattern (curve 3) for a supercell of $2 \times 2 \times 2$ (64 atoms) with the composition $GaAs_{0.95315}N_{0.0156}Bi_{0.03125}$ was carried out using the VASP software package [14]. The calculations were performed on the Blokhin cluster of the Southern Federal University, and the visualization was done using the VESTA program.

Based on the available data [15], we calculated and visualized the most stable and probable variant of the incorporation of nitrogen atoms into the crystal lattice of GaAs vapor NN₄ (Fig. 3, right inset). Fig. 3 demonstrates a close coincidence between the angular position of the 64.52° GaAsNBi (004) reflection for the experimental curves 1 and 2, and the GaAs_{0.95315}N_{0.0156}Bi_{0.03125} model (curve 3), which proves the formation of a GaAs_{1-*x*-*y*}N_{*x*}Bi_{*y*} solid solution. The half-width at half-height of the rocking curve for GaAs_{1-*x*-*y*}N_{*x*}Bi_{*y*} is only 0.39°, which indicates good layer homogeneity and its crystalline perfection. Moreover, we observed a weak reflection at an angular position of 67.3°, which we attribute to GaAs_{1-*x*}N_{*x*} (004).

Reflections in the GIXRD diffraction patterns (Fig. 3, curve 2) arise from crystal planes deviating only slightly from the film surface. The GaAsBiN (004) reflection observed in curve 2 at an angle $2\theta = 64.58^{\circ}$ corresponds to the simulated reflection for GaAs_{0.95315}N_{0.0156}Bi_{0.03125} (curve 3). Comparison of the number of peaks and the ratio of their intensities with the data of ICDD PDF-2 pattern no. 00-032-0389 for the GaAs substrate indicates that during growth, crystalline films with different textures along the (400) plane were obtained.

An analysis of the diffraction patterns from the $GaAs_{1-x-y}N_xBi_y$ thin films obtained showed that all the films had a polycrystalline structure. Furthermore, the thin film produced under a pressure of 60 Pa had the highest level of crystalline perfection. The study revealed that as the pressure increased, the change in the Full Width at Half Maximum (FWHM) values



Fig. 3. Diffraction pattern in the Bragg–Brentano geometry (curve 1) and grazing small-angle beam incidence (curve 2) of a $GaAs_{1-x-y}N_xBi_y$ thin film obtained at a pressure of 60 Pa for the (004) reflection, calculated diffraction pattern of a $GaAs_{0.95315}N_{0.0156}Bi_{0.03125}$ thin film (curve 3) and ICDD PDF-2 pattern no. 00-032-0389 GaAs (curve 4) (coor online)

of the crystallographic reflection decreased, as illustrated in Fig. 3 (inset on the left).

In the literature, the work [16] presents a closely related study on the production of epitaxial thin films of $GaAs_{1-x-y}N_xBi_y$ on a GaAs (001) substrate using molecular beam epitaxy. It is noteworthy that the angular position of the GaAsNBi (004) reflection had almost complete coincidence, indicating a close composition of the grown films. However, the composition measured by other methods may differ slightly, as X-ray diffraction only considers N and Bi atoms built into the crystal lattice and does not account for atoms present in the interstitial position.

To determine the precise composition of pseudomorphically grown $GaAs_{1-y}Bi_y$ thin films that consist of three components, XRD measurements around symmetric (004) and asymmetric (115) reflections are typically sufficient. However, for four-component $GaAs_{1-x-y}N_xBi_y$ thin films, obtaining an accurate composition measurement requires an array of results for the two variables *x* and *y*, which determine the concentrations of N and Bi. Consequently, the XRD method provides only the relationship between N and Bi concentrations. To fully determine the composition of a thin film, another method that examines an independent material property, such as the band gap, is necessary. In this study, the band gap of the $GaAs_{1-x-y}N_xBi_y$ thin films obtained at pressures of 10 and 60 Pa was determined by measuring photoluminescence at room temperature (Fig. 4).



Fig. 4. Room temperature photoluminescence spectra for $GaAs_{1-x-y}N_xBi_y$ thin films obtained at a pressure 10 Pa and 60 Pa showing the optical band gap

The position of the photoluminescence maxima for the $GaAs_{1-x-y}N_xBi_y$ film obtained at a pressure of 10 Pa was 1.11 eV, which agrees with literature data [1, 17, 18], while that of the GaAs substrate was 1.417 eV. It was noted that the FWHM value of the photoluminescence maximum was 142 meV, which can be explained by the formation of nitrogen clusters with various configurations and composition fluctuations. This is because the main contribution to the photoluminescence intensity at low and moderate excitation densities is made by strongly localized states. For a $GaAs_{1-x-y}N_xBi_y$ film obtained at a pressure of 60 Pa, the maximum photoluminescence occurs at 1.03 eV, and its FWHM is 129 meV. Hence, the composition of $GaAs_{1-x-y}N_xBi_y$ thin films obtained at pressures of 10 and 60 Pa can be determined as GaAs_{0.968}N_{0.007}Bi_{0.025} and GaAs_{0.967}N_{0.012}Bi_{0.021}, respectively.

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