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STRUCTURAL FEATURES OF EPITAXIAL SOLID SOLUTION FILMS $(Si_2)_{1-X}(GaN)_X$ GROWN ON Si SUBSTRATES

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On Si (111) substrates with a diameter of 20 mm, continuous monocrystalline epitaxial layers of $(Si_2)_{1-x}(GaN)_x$ solid solutions were grown by liquid-phase epitaxy from a limited volume of a tin solution-melt. The grown films had *n*-type conductivity. An analysis of the X-ray diffraction pattern showed that the grown epitaxial film has (111) crystallographic orientation with evident single-crystal sings. The size of the film subcrystallites is~40 nm. Due to the replacement of paired Si atoms by a molecule consisting of Ga and N atoms, a slight bending of the film towards the perpendicular to the reflecting surface was observed. Coherently arranged nanocrystals of cubic (*c* - GaN) and hexagonal (*h* - GaN) modification of gallium nitride with a crystallite size of L_{GaN}≈47 nm are formed in the crystal lattice of the epitaxial film.
Keywords: *Liquid-phase epitaxy; Silicon; Gallium nitride; Solid solution; X-ray structural analysis*

1. INTRODUCTION

Gallium nitride (GaN) is a direct-gap semiconductor with a band gap of 3.4 eV, enabling the use of a wide range of electromagnetic wavelengths. The excellent optical and electrical properties of GaN make it an attractive material for optoelectronics and high-speed electronic devices, including LEDs, detectors, lasers, and high-power and high-frequency devices [1-7]. GaN-based devices are ideal for power amplifiers operating at frequencies that are unattainable with Si-based technology. GaN power electronics offer less power loss than Si devices and can operate at higher temperatures. The integration of GaN devices with Si integrated circuits provides a scalable CMOS (complementary metal-oxide-semiconductor) technology platform [8] for high-power and high-performance optoelectronics. Over the past 20 years, GaN has become the most researched semiconductor after silicon due to its excellent optical and electrical properties.

Currently, due to the high cost of native substrates, GaN is grown on foreign substrates such as sapphire (Al_2O_3), SiC, and Si [9–11]. However, there is a mismatch between the lattice constant and the thermal expansion coefficient of the GaN film and the foreign substrate. As a result, dense point defects, threading dislocations, and residual stresses appear in finished films, which reduce the service life and efficiency of optoelectronic devices fabricated on their basis [12–14]. When growing GaN on Si (GaN-on-Si), two main problems arise: (1) the nucleation of an AlN buffer layer grown to prevent the formation of a Ga-Si alloy and to compensate for thermal stresses, as well as the quality of the resulting AlN/Si interface; and (2) cracking of the epitaxial layer due to the higher thermal expansion coefficient of nitrides compared to Si. GaN layers crack when the layer thickness exceeds ~1 µm [15]. At present, GaN-on-Si technology has greatly improved. However, the mass adoption of GaN-on-Si for illumination remains controversial, mainly because epitaxial layers on Si are more difficult to grow than GaN on Al_2O_3. For electronic applications, GaN-on-Si is more favorable due to the poor electrical and thermal conductivity of Al_2O_3 (sapphire) and the high cost of SiC, which are commonly used as GaN substrates [16, 17]. High-quality GaN layers can be obtained on Si substrates through buffer layers from a continuous solid solution of molecular substitution $(Si_2)_{1-x}(GaN)_x$, with a smoothly changing composition from silicon to pure gallium nitride ($0 \le x \le 1$). This buffer layer, by leveling the mismatch between the lattice constant and the thermal expansion coefficient of the Si substrate and the GaN epitaxial film, prevents residual mechanical stresses in the transition region and, consequently, reduces point defects and threading dislocations in the epitaxial layer.

In this article, we report the possibility of growing epitaxial layers of solid solutions of molecular substitution $(Si_2)_{1-x}(GaN)_x$ on Si (111) substrates by liquid-phase epitaxy from a tin solution-melt and present the results of an experimental study of the structural features of epitaxial films using the X-ray diffraction method. Epitaxial layers of $(Si_2)_{1-x}(GaN)_x$ were grown using the technology described in [18].

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2. MATERIALS AND METHODS

Solid solutions $(Si_2)_{1-x}(GaN)_x$ were grown on Si (111) substrates by liquid-phase epitaxy from a limited volume of Sn solution-melt in an ambient H_2 atmosphere. Before entering the reactor, hydrogen was deeply purified using palladium diffusion filtration, removing oxygen, moisture, and other impurity gases. Palladium purification produced high-purity hydrogen, which was essential for stabilizing liquid-phase epitaxy conditions, preventing oxidation of the melt solution components and the substrate surface, and improving the structural quality and homogeneity of the grown epitaxial layers. The substrates were silicon washers of grade KDB-0.01, p-type, boron-doped, with a resistivity of 0.01 Ω·cm. The diameter and thickness of the substrates were 20 mm and 400 μm, respectively. The growth of epitaxial layers was carried out by varying the following parameters of the technological process: the composition of the solution-melt, the temperature of the beginning and end of solid solution crystallization, the rate of forced cooling and the thickness of the solution-melt. The composition of the Sn-Si-GaN solution melt, in the weight ratio of components, was determined based on the results of preliminary experiments. Epitaxial layers of solid solutions were grown at a temperature range of 950–850°C, a cooling rate of 1 °C/min, and a solution-melt thickness of 1 mm. At the selected process parameters, the entire substrate surface was covered with a continuous epitaxial film. The adhesion of the epitaxial films to the substrate surface was of high quality. Over the entire surface, the thickness of the epitaxial layer was uniform at ~10 μm. Epitaxial films had *n*-type conductivity with a concentration of ~ $3.4 \cdot 10^{16}$ cm^{-3}, a mobility of ~ 133 $cm^2 \cdot V^{-1} \cdot sec^{-1}$ of charge carriers and a resistivity of ~ 1.38 Ω·cm at 300 K.

Structural studies of the resulting epitaxial film were carried out in the Institute of Nuclear Physics of the Academy of Sciences of the Republic of Uzbekistan on a DRON-3M X-ray diffractometer (radiation CuK_α, λ = 0,15418 nm) with improved the Θ–2Θ optical scheme in step scan mode.

3. RESULTS AND DISCUSSION

Fig. 1 shows an X-ray diffraction pattern of the Si substrate. The diffraction pattern contains several selective structural reflections with different intensities and one diffuse reflection at small scattering angles. The analysis showed that the substrate surface corresponds to the (111) crystallographic plane.

This is evidenced by the presence of a series of selective reflections of the (HHH) type (where H = 1, 2, 3), intense lines $(111)_{Si}$ with d/n = 0.3142 (2θ = 28.4°), $(222)_{Si}$ with d/n = 0.1568 (2θ = 58.8°) and $(333)_{Si}$ with d/n = 0.1045 nm (2θ = 94.95°). The beta (β) component of the structural line of the first order $(111)_{Si}$ is visible at 2θ = 25.8°, and the third order is visible at 2θ = 83.5°. High intensity ($2 \cdot 10^5$ pulses·sec^{-1}) of the main reflection $(111)_{Si}$, narrow width (FWHM ≈ 0.25°), testifies to the perfection of the substrate crystal lattice. Furthermore, the X-ray diffraction pattern of the substrate contains only the reference reflection (111) and its high orders (222) and (333), while diffraction reflections with other indices are absent. These are signs of its single-crystal state.

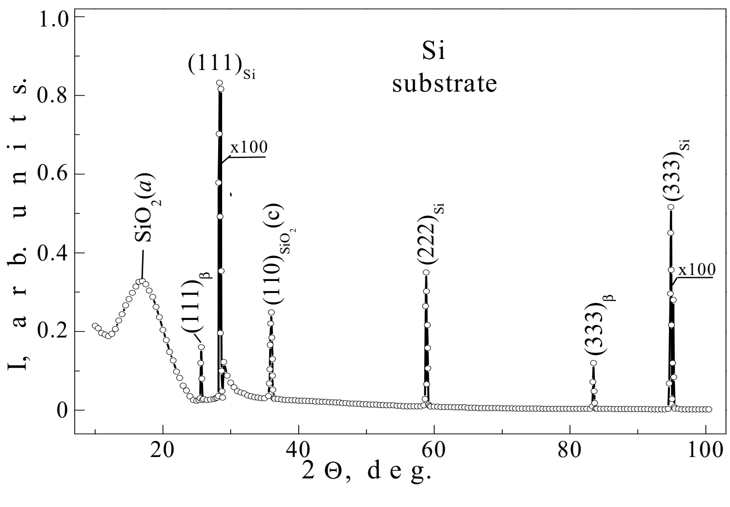

Figure 1. X-ray pattern of a silicon substrate

Good, close to the calculated splitting of the $(333)_{Si}$ reflection with characteristic intensity ratios for α_1 and α_2 radiation $[I_{333}(\alpha_1) \approx 2I_{333}(\alpha_2)]$, indicates that the elastic stress is predominantly concentrated in the near-surface layer of the volume, close to the surface of the substrate and at the boundaries between the blocks of the matrix lattice (Fig. 2).

The X-ray diffraction pattern shows a noticeable (110) reflection of SiO2 with d/n = 0.2501 nm (2θ = 35.9°), attributed to the SiO_2 impurity in the substrate. The narrow width (FWHM = $2.62 \cdot 10^{-3}$ rad) of this reflection indicates that the SiO_2 impurity phase in the bulk of the matrix is in the crystalline modification, SiO_2(c). The characteristic size of the structural fragments (subcrystallites) of this phase is ~ 58 nm. Due to the large ionic radius of oxygen compared to silicon, they are energetically preferred to be located in the interstices of boundary and near-boundary regions between grains and blocks (subcrystallites) of the substrate [19]. In these areas, the chemical bonds between the ions of the matrix are not saturated, and the nodes are somewhat displaced from their ideal positions.

These factors contribute to the formation of structural fragments with low symmetry, consisting of silicon and interstitial ions, in comparison with the structure with high symmetry in the bulk of the blocks of the substrate, consisting only of silicon ions. The experimental value of the substrate lattice parameter was a_{Si} = 0.54293 nm, which is comparable with the tabulated value a_{Si} = 0.54307 nm. This shows that the volume fraction of the distorted lattice region is negligible compared to the entire volume of the substrate.

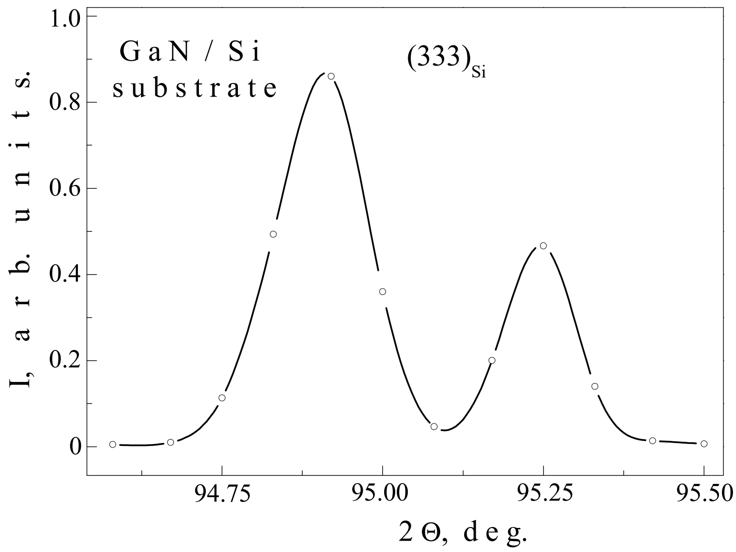

Figure 2. The shape of the reflections (333) of the silicon substrate

Figure 3 shows the X-ray diffraction pattern of an epitaxial layer of the $(Si_2)_{1-x}(GaN)_x$ solid solution grown on silicon substrates. The high-resolution (333) reflection shape is shown in Fig. 4. This result significantly differed from the X-ray diffraction pattern of the substrate. Here, the intensity of diffuse reflection at small scattering angles decreased by more than 3 times, and the intensity of the main reflection $(111)_{Si}$ (2θ = 28.33°) of the film increased by ~ 6% compared to the intensity of the same reflection of the substrate. The intensity of the forbidden reflection (222) (2θ = 58.65°) decreased by a factor of 2, its intensity of the third order (333) (2θ = 94.70°) decreased by almost an order of magnitude, and the intensity of the reflection (110) of SiO_2 (2θ = 35.8°) from the impurity phase of crystalline quartz decreased by 2 times. The average level of the inelastic background also decreased by 2 times.

The relatively narrow width (FWHM = $3.78 \cdot 10^{-3}$ rad) and high intensity ($2 \cdot 10^5$ pulses·sec-1) of the main reflection $(111)_{GaN/Si}$ indicate a high degree of perfection of the crystal lattice of the epitaxial layer of the solid solution $(Si_2)_{1-x}(GaN)_x$; that is, the grown film has the sign of a single crystal with a (111) orientation. The sizes of subcrystallites (blocks) of the film, estimated from the width of this peak by the Selyakov–Scherrer method [20], were ~ 40 nm.

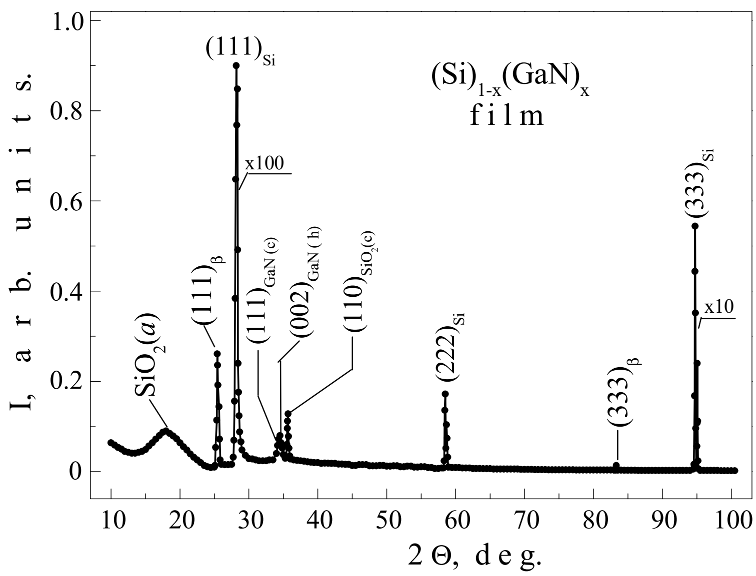

Figure 3. X-ray pattern of the epitaxial layer of the $(Si_2)_{1-x}(GaN)_x$ solid solution

However, the intensity of the main reflection of the $(111)_{GaN/Si}$ film is 6% higher than the intensity of the same line of the substrate, $(111)_{Si}$. This indicates the partial replacement of some pairs of silicon ions by gallium and nitrogen molecules in the film's silicon lattice. Since the X-ray scattering intensity is proportional to the square of atomic number (Z^2) of the elements (Z_{Si} = 14, Z_N = 7, and Z_{Ga} = 31), such a replacement of ions should lead to some increase in the intensity of this reflection, which is observed in the experiment.

Since the thermal expansion coefficients of GaN are 2.2 times greater than those of Si (α_{GaN} - 5.59·10^{-6}/ºC, α_{Si} - 2.6 ·10^{-6}/ºC), when cooled from the epitaxy temperature (950–850°C) to room temperature, the fabricated heterostructure bends in the direction perpendicular to the reflective surface of the film.

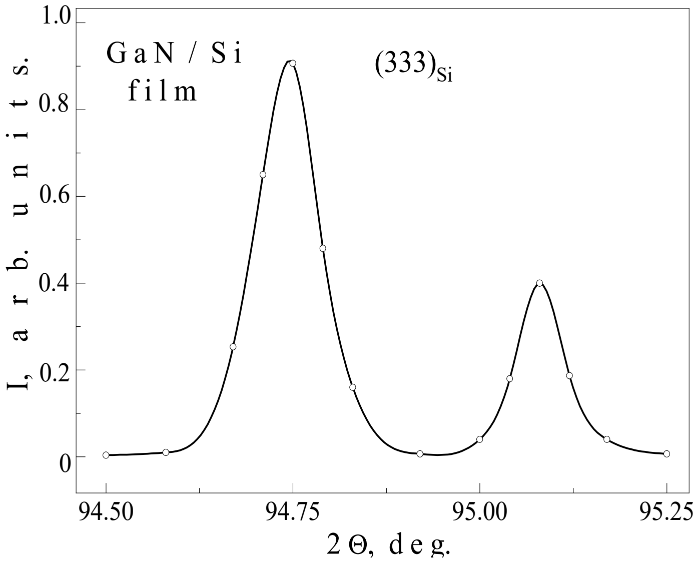

Figure 4. The shape of reflections (333) of the epitaxial layer of the solid solution $(Si_2)_{1-x}(GaN)_x$

In addition, this conclusion is confirmed by the appearance of a new selective reflection with a noticeable intensity and a width of 3.2·10^{-3} rad. near the main reflection (111) of the film. An analysis of the nature of this structural line showed that it consists of two selective lines very close in angular position and corresponding to two modifications of gallium nitride: cubic (c-GaN) - $(111)_{GaN}$ with d/n = 0.2599 nm (2θ = 34.4°) and hexagonal (h-GaN) - $(002)_{GaN}$ with d/n = 0.2592 nm (2θ = 34.6°). They are caused by gallium nitride nanocrystals $L_{GaN} \approx 47$ nm in size. These nanocrystallites are located in the near-surface layer of the film, as evidenced by the angular arrangement of reflections associated with them. The close angular position of these reflections to the main $(111)_{GaN/Si}$ reflection made it possible to determine the GaN content of c-GaN and h-GaN nanocrystals in the film from the ratio of their intensities. It was 9.1 and 8.9 mol. %, respectively. Therefore, it can be argued that during liquid-phase epitaxy on a Si(111) substrate, it is possible to form a GaN-Si heterostructure with GaN nanocrystallites, both cubic (c-GaN) and hexagonal (h-GaN) modification.

4. CONCLUSIONS

Continuous epitaxial layers of $(Si_2)_{1-x}(GaN)_x$ solid solutions were grown on Si(111) substrates 20 mm in diameter by liquid-phase epitaxy from a limited volume of a tin solution-melt.

Analysis of the X-ray diffraction pattern showed that the resulting film has the perfect crystalline structure with (111) orientation and evident single-crystal sings. Coherently located nanocrystallites of cubic (c-GaN) and hexagonal (h-GaN) modification of gallium nitride with a crystallite size of $L_{GaN} \approx 47$ nm are formed in the crystal lattice of the epitaxial film.

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Authors' Contribution A.S. Saidov: Project administration, Sh.N. Usmonov: Supervision M. Kalanov: Writing-original draft, D.V Saparov: Editing, Data curation, T.T. Ishniyazov: Carried out the experiment. M.B. Tagaev: Performed the measurements. A.M. Akhmedov: Assisted measurements. A.Sh. Razzokov: Validation, K.G. Gaymnazarov: Funding acquisition.

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СТРУКТУРНІ ОСОБЛИВОСТІ ЕПІТАКСІАЛЬНИХ ПЛІВОК ТВЕРДИХ РОЗЧИНІВ (Si2)1-X(GaN)X ВИРОЩЕНИХ НА Si-ПІДЛОЖКАХ

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На підкладках Si (111) діаметром 20 мм методом рідкофазної епітаксії з обмеженого об'єму розчину-розплаву олова вирощували суцільні монокристалічні епітаксіальні шари твердих розчинів $(Si_2)_{1-x}(GaN)_x$. Вирощені плівки мали n-тип провідності. Аналіз рентгенограми показав, що вирощена епітаксіальна плівка має кристалографічну орієнтацію (111) з чіткими монокристалічними ознаками. Розмір субкристалітів плівки становить ~ 40 нм. Через заміщення парних атомів Si молекулою, що складається з атомів Ga та N, спостерігався незначний вигин плівки відносно перпендикуляра до поверхні відбиття. У кристалічній решітці епітаксіальної плівки утворюються когерентно розташовані нанокристали кубічної (c-GaN) та гексагональної (h-GaN) модифікацій нітриду галію з розміром кристалітів $L_{GaN} \approx 47$ нм.

Ключові слова: *рідкофазна епітаксія; кремній; нітрид галію; твердий розчин; рентгеноструктурний аналіз*