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## Structural and spectroscopic studies of epitaxially overgrown GaN, n-GaN, and n<sup>+</sup>-GaN contact layers

P. V. Seredin<sup>1✉</sup>, D. L. Goloshchapov<sup>1</sup>, D. E. Kostomakha<sup>1</sup>, Y. A. Peshkov<sup>1</sup>, N. S. Buylov<sup>1</sup>,  
A. A. Gaivoronskaya<sup>1</sup>, A. M. Mizerov<sup>2</sup>, S. N. Timoshnev<sup>2</sup>, M. S. Sobolev<sup>2</sup>, E. V. Ubyivovk<sup>3</sup>,  
V. I. Zemlyakov<sup>4</sup>, P. P. Kutsko<sup>5</sup>, P. L. Parmon<sup>5</sup>

<sup>1</sup>Voronezh State University,  
Universitetskaya pl. 1, Voronezh 394018, Russian Federation

<sup>2</sup>Alferov University,  
8/3 Khlopina st., St. Petersburg 194021, Russian Federation

<sup>3</sup>Saint-Petersburg State University,  
7-9 Universitetskaya emb., St. Petersburg 199034, Russian Federation

<sup>4</sup>National Research University of Electronic Technology,  
1, Shokin Square, Zelenograd, Moscow 124498, Russian Federation

<sup>5</sup>Research Institute of Electronic Technology,  
5 Staryh Bolshevikov st., Voronezh 394033, Russian Federation

### Abstract

The paper demonstrates that the technology of plasma-assisted molecular beam epitaxy (PA MBE) can be used to form epitaxially overgrown GaN, n-GaN, and n<sup>+</sup>-GaN contact layers with a high structural quality on virtual GaN/c-Al<sub>2</sub>O<sub>3</sub> substrates under Ga-enriched conditions at relatively low growth temperatures of ~700 °C.

It was shown that the initial stage of growth of the contact layers was accompanied by effective filtration of dislocations threading from the buffer GaN layer of the virtual substrate formed by MOCVD.

The values of residual stresses calculated using the data of Raman microspectroscopy indicate a high structural quality of GaN, n-GaN, and n<sup>+</sup>-GaN contact layers regardless of the level of silicon doping.

The contact resistance reduced to the pad width determined using the transmission line method for the structure with n<sup>+</sup>-GaN contact layer was ~0.11 Ohm-mm and for the n-GaN contact layer it was ~0.5 Ohm-mm.

**Keywords:** Molecular beam epitaxy, GaN, n-GaN, and n<sup>+</sup>-GaN contact layers, Virtual substrate, Raman microspectroscopy

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✉ Pavel V. Seredin, e-mail: [paul@phys.vsu.ru](mailto:paul@phys.vsu.ru)

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## 1. Introduction

Many studies conducted in recent decades have been dedicated to group III nitrides (AIIIN) (third-generation semiconductors) and the extension of the range of their applications [1]. However, the key problem in creating viable AIIIN-based device technology solutions is the choice of a substrate for epitaxial synthesis and the method of integration with it [2–4]. In most cases, lattice-matched GaN substrates are not commercially viable despite the fact that they allow synthesizing GaN-based device heterostructures with better material characteristics and crystalline quality. An alternative solution to this problem is to form the active regions of AIIIN heterostructures by using virtual substrates (templates) of the GaN/substrate type, in which the buffer layer has already been synthesized on a commercially available substrate (Si, SiC, sapphire) by epitaxial technologies. Currently, two methods are mainly used to form GaN/substrate templates: metalorganic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE) [3].

The second important issue in the development of AIIIN-based devices is the formation of ohmic contacts, which not only provide the connection between the device structure and the signal processing circuitry, but also are the basis for further improving the performance of final devices. The ohmic contact between metal and semiconductor must not only be compatible, but also durable and temperature stable, [5] taking into account the type of conductivity of the contact layer and technological postoperations.

The formation of high-quality ohmic contact in devices based on wide-gap AIIIN compounds is still an unresolved problem [6]. Defects in AIIIN layers and the tendency to metal oxidation at high temperatures can also affect the contact

properties. Therefore, studies aimed at reducing the contact resistance during the formation of ohmic contacts in AIIIN heterostructures are becoming increasingly relevant today.

Currently, there are a number of technological solutions to reduce the contact resistance, including the use of Ti/Al/X/Au multilayers [7]. It was also shown that the formation of fully buried Ti-based ohmic contacts results in good values of contact resistivity [6]. However, along with the traditional technology of annealing ohmic contacts [5], a wide range of studies are currently being conducted to create ohmic contacts without using the procedure of high-temperature annealing, or so-called non-annealed ohmic contacts [8–13]. In particular, papers [10–13] show that it is possible to form such contacts with a relatively low contact resistance by the epitaxial overgrowth of highly silicon-doped n<sup>+</sup>-GaN contact layers with an electron concentration of more than  $1 \cdot 10^{19} \text{ cm}^{-3}$  using the PA MBE technique.

This work presents comparative studies of the structural, morphological, and electrical properties of undoped GaN and silicon-doped n-GaN and n<sup>+</sup>-GaN contact layers synthesized by the PA MBE technique on GaN/c-Al<sub>2</sub>O<sub>3</sub> virtual substrates grown using the method of pre-epitaxial cleaning of GaN surfaces of substrates previously developed by the authors and described in detail in [14]. The review of scientific literature has shown that there have been no studies of this kind, which emphasizes the relevance of this work.

## 2. Materials and methods

During the study, GaN, n-GaN, and n<sup>+</sup>-GaN contact layers were formed using the PA MBE technique on an industrial Veeco Gen 200 setup [14]. Synthesis was carried out on

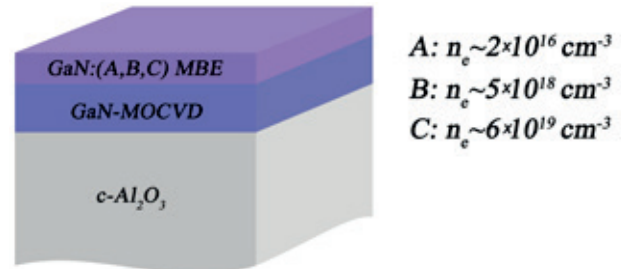
virtual substrates, i.e. undoped GaN layers with a thickness of about 2.5  $\mu\text{m}$  grown on sapphire substrates with (0001) orientation by metal organic chemical vapor deposition (MOCVD) [18].

After MOCVD synthesis, GaN/c-Al<sub>2</sub>O<sub>3</sub> virtual substrates were extracted to the atmosphere for standard preparation procedures for PA MBE layer synthesis, which are described in detail in [14].

Immediately before the start of the PA MBE growth of the contact layers, the surfaces of the virtual substrates were cleaned following the procedure in [14]. First, the pre-epitaxial cleaning of virtual substrates was carried out in the growth chamber of the PA MBE system in a flow of activated nitrogen particles with a gradual increase in the temperature of the substrate from  $T_s = 400$  °C to  $T_s = 600$  °C followed by exposure of the substrate surface to a flow of activated nitrogen at a fixed value of  $T_s = 600$  °C for 1 hour. This was followed by the final procedure of GaN surface cleaning based on the deposition of several Ga monolayers on the surface of the virtual substrate followed by thermal desorption of the deposited Ga from the surface. The study involved five periods of gallium precipitation/desorption on the GaN surface of the virtual substrate heated to  $T_s = 700$  °C, i.e. to a temperature at which there was still no intensive decomposition of GaN, however, the flow of the thermal desorption of Ga from the liquid phase was  $F_{\text{Ga}}^{\text{des}} \sim 0.3$   $\mu\text{m}/\text{h}$  [15]. During each cycle of the stage, the precipitation of gallium atoms was completed within a 10 second exposure of GaN on the surface of the virtual substrate in a gallium flow  $F_{\text{Ga}} \sim 0.4$   $\mu\text{m}/\text{h}$ , which corresponded to the precipitation of about one Ga monolayer. The Ga flow was then shut off with a valve for 10 seconds to enable thermal desorption of the precipitated gallium from the GaN surface. Throughout the procedure of gallium adsorption/desorption, there was a “linear” pattern of reflected high energy electron diffraction (RHEED) with bright and thin reflexes characteristic of a relatively smooth GaN surface.

Immediately after the pre-epitaxial cleaning of the surfaces of the virtual substrates, undoped (sample A) and silicon-doped (samples B and C) GaN layers with thicknesses of about 250 nm were grown on the virtual substrates by PA MBE

at constant values of  $T_s = 700$  °C,  $F_{\text{Ga}} \sim 0.25$   $\mu\text{m}/\text{h}$ ,  $F_{\text{N}} \sim 0.05$   $\mu\text{m}/\text{h}$  and different temperatures of the silicon effusion source  $T_{\text{Si}} = 1,020$ – $1,110$  °C. The general scheme of samples with GaN, n-GaN, and n<sup>+</sup>-GaN contact layers is shown in Fig. 1.



**Fig. 1.** Schematic representation of heterostructures with contact layers: sample A – GaN; sample B – n-GaN; sample C – n<sup>+</sup>-GaN

The GaN/c-Al<sub>2</sub>O<sub>3</sub> virtual substrate with a precisely oriented c-Al<sub>2</sub>O<sub>3</sub>(0001) substrate were used to synthesize samples A and C, while the c-Al<sub>2</sub>O<sub>3</sub> substrate with a deviation of 0.5° from [0001] direction was used to produce sample B.

The crystal structure of the samples was studied using high-resolution X-ray diffraction using a DRON-8T X-ray diffractometer with CuK $\alpha$  radiation and an angular reproducibility of  $\pm 0.0001$ °.

A RamMics 532 Raman microscope (EnSpectr, Moscow, Russia) with a radiation wavelength of 532 nm was used to obtain Raman spectra. The scanning was carried out using a 20 $\times$  lens, the focal spot size of  $\sim 8$   $\mu\text{m}$ , and power of 30 mW. Raman spectra were recorded in the (xy)<sup>-</sup> geometry in the range of 100–2,000  $\text{cm}^{-1}$  and a spectral resolution of 1  $\text{cm}^{-1}$ . The position of the lines and the values of the full width at half maximum (FWHM) for both the GaN and Al<sub>2</sub>O<sub>3</sub> layers was determined by approximating the maxima using a set of Voigt functions [16]. OriginPro software was used for processing. Spectral position of the maxima of the Raman lines was determined by measuring the position of the LO-mode of the Si calibration plate. Calibration was performed both before and after each sample measurement using a  $\times 20$  lens.

The quality of surface and hetero-interfaces was studied using scanning transmission electron microscopy (STEM) and high angular annular dark-field scanning transmission electron microscopy (HAADF-STEM) on a Zeiss Libra 200

FE transmission electron microscope with an accelerating voltage of 200 keV.

Ohmic contacts were metalized on a Kurt J. Lesker AXXIS unit for the electron-beam sputtering of metals.

Electrophysical measurements were performed at room temperature in a four-probe van der Pauw configuration on an Ecopia HMS-3000 measurement system.

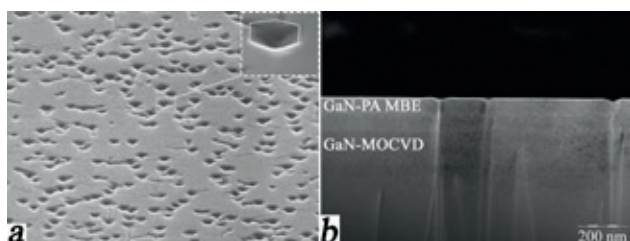
Contact resistance was measured using the transmission line method (TLM).

### 3. Results and discussion

The characteristics of the epitaxial layers of GaN, such as surface structure and morphology, crystalline perfection, and density of the threading dislocations, play an important role in determining the characteristics of the heterostructure. Therefore, the surface and hetero-interface were first studied.

Images of the surface and profile of the GaN contact layers obtained at different levels of silicon doping are shown in the STEM and HAADF-STEM images (Fig. 2). For all samples, regardless of the size of the introduced impurity, there was a good smooth surface morphology with pits or V-shaped defects evenly distributed on the surface. These defects had a characteristic shape of inverted hexagonal pyramids (see the insert to Fig. 2a). These pits formed by low-index crystallographic planes can be observed during the deposition of nitride films on various substrates [17,18].

Since GaN/sapphire templates were used to form the GaN, n-GaN, and n<sup>+</sup>-GaN contact layers, it is obvious that the heteropairs should have a significant discrepancy in both the parameters



**Fig. 2.** Image of sample C, in which the contact n<sup>+</sup>-GaN layer was synthesized by the MIIЭ ПIA method on a virtual GaN/c-Al<sub>2</sub>O<sub>3</sub> substrate under Ga-enriched growth conditions: STEM image of the surface (a); HAADF-STEM image of the heterointerface (b)

of the crystal lattices and the thermal expansion coefficients (16 and 34%, respectively). Both factors led to deformations and stress distribution gradients in the thick GaN buffer layer [19]. They were also the driving force for the formation of V-shaped pits in the upper contact epitaxial layers.

Previously, it was shown that the bottom of each pit is always associated with a threading dislocation (TD), and the density of the defects is almost always equivalent to the density of TDs [20]. Thus, the formation of V-shaped defects in the upper GaN, n-GaN, and n<sup>+</sup>-GaN contact layers can be explained both by the threading dislocations from the virtual substrate and the emergence of new dislocations on the interface between the surface of the virtual substrate and the GaN contact layer due to insufficiently effective pre-epitaxial cleaning of the template surface.

To study the evolution of threading dislocations during the PA MBE synthesis of GaN layers on GaN/c-Al<sub>2</sub>O<sub>3</sub> templates, cross-section of one of the samples with an upper n<sup>+</sup>-GaN contact layer was studied by dark-field scanning transmission electron microscopy. Figure 2b shows a typical HAADF-STEM image of the interface between the GaN layer of the virtual substrate and the upper n<sup>+</sup>-GaN contact layer obtained by PA MBE.

The analysis of the HAADF-STEM data allows concluding that threading dislocations in the GaN, n-GaN, and n<sup>+</sup>-GaN contact layers were filtered. It was seen that the number of threading dislocations in the upper PA MBE layer was smaller than in the lower buffer GaN layer of the GaN/c-Al<sub>2</sub>O<sub>3</sub> virtual substrate grown by MOCVD. Some of the TDs were filtered near the interface with the upper GaN contact layer, though some dislocations were still present.

The observed effect of filtration of threading dislocations may be due to the evolution of the surface morphology of the GaN contact layer at the initial stage of PA MBE synthesis on the GaN surface of the virtual substrate. In particular, the relatively low substrate temperatures used in the PA MBE synthesis of GaN could lead to the formation of three-dimensional GaN nucleation islands, which quickly grew and coalesced into a continuous GaN layer with a smooth surface

morphology, as shown in [21]. However, during the initial stage of the island PA MBE growth of GaN, it became energetically more advantageous for the TDs to deviate from the initial vertical direction of their propagation [22]. The energy gain was due to a reduction in the TD energy since the line of the threading dislocation became shorter when it deviated to the edge of the island as compared to the energy of dislocations propagating in the direction of the GaN layer growth.

In addition, TEM studies of the synthesized samples also showed that there were no additional defects in the GaN, n-GaN, and n<sup>+</sup>-GaN contact layers, which could appear as a result of insufficiently effective cleaning of the surface of the GaN MOCVD layer of the virtual substrate from foreign atoms or due to surface degradation that could occur during the process of pre-epitaxial cleaning. This indicates the effectiveness of the used method of pre-epitaxial cleaning of the GaN surface of the virtual substrate.

The crystalline structure and the quality of epitaxial heterostructures were studied using high-resolution X-ray diffraction (XRD), which can provide direct information on the effect of silicon impurity on the crystalline properties of the AIII<sub>2</sub>BV and AIII<sub>2</sub>IN semiconductors [23, 24]. Figure 3 shows the results of XRD measurements over a wide range of Bragg angles.

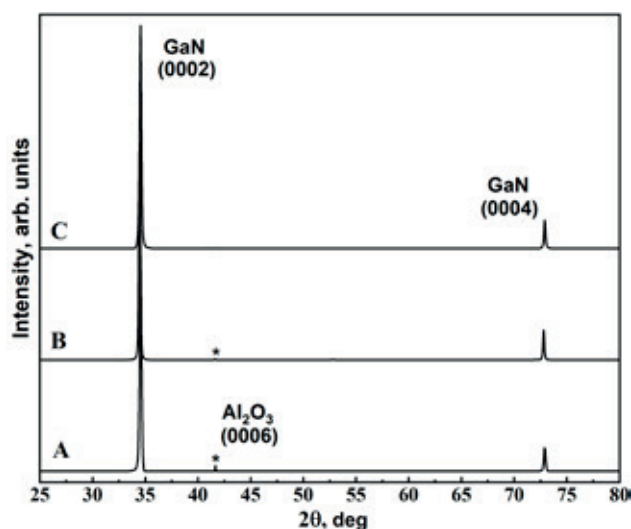


Fig. 3. XRD 2 $\theta$ -scans for samples with GaN, n-GaN and n<sup>+</sup>-GaN contact layers. Reflexes from the substrate are indicated by the index\*

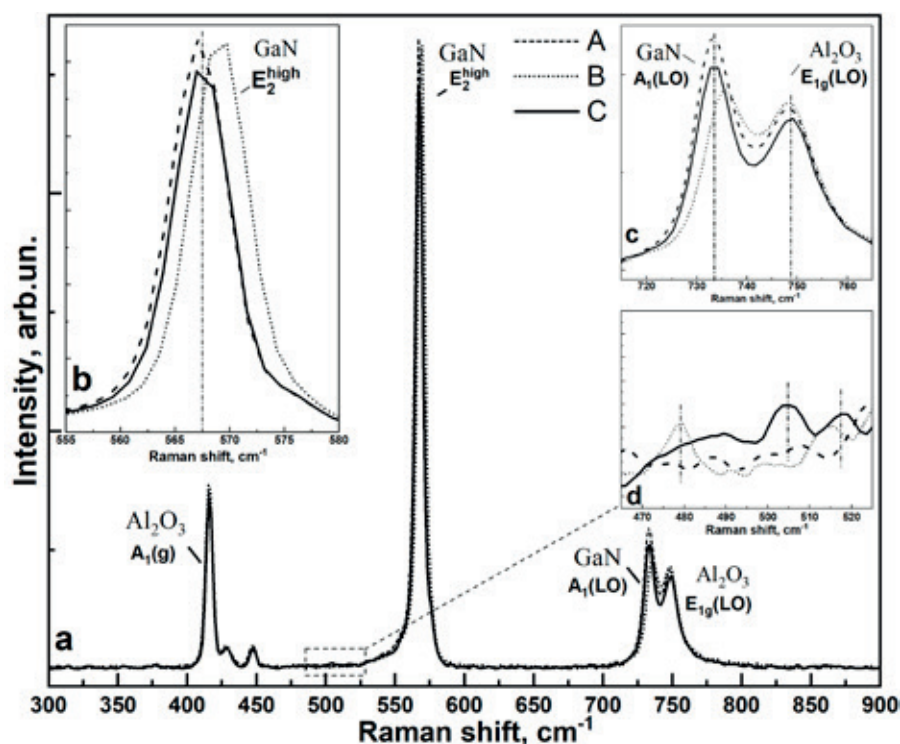
XRD 2 $\theta$ -scans of all samples had maxima that also belonged to 0002 and 0004 GaN reflections with a wurtzite lattice. However, the scans also showed low-intensity reflexes belonging to the sapphire substrate. The presence of diffraction maxima of the same basal series on XRD scans suggests the monocrystalline state of the epitaxial GaN layers.

Additional structural information on the properties of the GaN, n-GaN, and n<sup>+</sup>-GaN contact layers was obtained by Raman spectroscopy, which allows studying the features of the crystal structure of thin epitaxial films and their perfection on the scale of crystal lattice parameters [25]. The Raman spectra of the samples are shown in Fig. 4. The Raman spectra shown in Figure 4 are typical of the *c*-plane (0001) of the GaN single crystal. They have maxima of about ~567 and 734 cm<sup>-1</sup> (Fig. 4a,b), which, in accordance with the group theory for GaN with a wurtzite crystal lattice, are transverse A<sub>1</sub>(TO) and E<sub>2</sub><sup>high</sup> phonon modes [26, 27]. However, the peaks present in the spectra in the region of 415 cm<sup>-1</sup> and 750 cm<sup>-1</sup> are typical characteristic longitudinal optical (LO) modes with A<sub>1g</sub> and E<sub>1g</sub>(LO) symmetry, which belong to the sapphire substrate [28].

Attention should be paid to another spectral feature observed in Raman scattering for the n-GaN and n<sup>+</sup>-GaN contact layers (samples B and C, see insert to Fig. 4d). In the region of 465–500 cm<sup>-1</sup>, there was a low-intensity shoulder, the appearance of which was associated with a high level of silicon impurity in the n-GaN and n<sup>+</sup>-GaN contact layers. It was previously shown that the appearance of this phonon mode in the Raman spectra is associated with the formation of Si clusters in various materials [29, 30]. It should also be noted that n<sup>+</sup>-GaN had low-intensity Raman bands in the region of 505 and 517 cm<sup>-1</sup>. According to [29, 30], the appearance of these oscillations in the Raman spectra suggests the formation of both silicon nanocrystals of various sizes and amorphous silicon.

The frequency position of active oscillations from GaN and c-Al<sub>2</sub>O<sub>3</sub> in Raman spectra recorded using a x20 lens is shown in Table 1.

According to the results of our study, the value of the full width at half maximum (FWHM) of the Raman mode E<sub>2</sub><sup>high</sup> GaN in the spectra of the GaN, n-GaN, and n<sup>+</sup>-GaN contact layers was ~5.0–



**Fig. 4.** Raman spectra for epitaxial heterostructures with contact layers of GaN, n-GaN and n<sup>+</sup>-GaN (a) – in the full range; (b) – GaN  $E_2^{\text{high}}$  and (c) –  $A_1(\text{LO})$  modes; (d) – spectral region 465–525  $\text{cm}^{-1}$ , in which modes associated with the formation of silicon clusters of various sizes in the contact n<sup>+</sup>-GaN layer are recorded

5.2  $\text{cm}^{-1}$ , which indicates their high structural quality. However, this parameter was at a level characteristic of the epitaxial layers of GaN grown on a sapphire substrate [26].

The exact position of the Raman phonon mode  $E_2^{\text{high}}$  GaN compared to the undeformed GaN single crystal was used to estimate changes and to control residual stresses in the epitaxial layers due to the high sensitivity of  $E_2^{\text{high}}$  to crystal lattice deformations [31]. The following typical formula was used to estimate the value of residual biaxial stresses in the epitaxial contact layers:

$$\Delta\omega = k\sigma_{xx}. \quad (1)$$

Here,  $\Delta\omega$  is the Raman shift of the mode frequency  $E_2^{\text{high}}$  GaN,  $\sigma_{xx}$  is the value of the residual biaxial stress in the GaN epitaxial film,  $k$  is the conversion ratio of biaxial stress to the Raman shift. The value of the conversion ratio for  $E_2^{\text{high}}$  GaN mode was 4.3 ( $\text{cm}^{-1} \cdot \text{GPa}^{-1}$ ) [32].

According to the results, the value of residual stresses in the GaN contact layer (sample A) was at the level of  $\sigma_{xx} \sim 0.1$  GPa. A similar small value of  $\sigma_{xx} \sim 0.11$  GPa was observed for the n<sup>+</sup>-GaN layer (sample C), which indicated a high degree of stress relaxation. What is more, the obtained results correlate with the known data on residual deformation in Si-doped GaN layers when grown

**Table 1.** The frequencies and full width at half maximum (FWHM) of the main oscillations observed in the Raman spectra of the GaN, n-GaN, and, n<sup>+</sup>-GaN contact layers

Sample	Modes of Raman spectra				Biaxial stress $\sigma_{xx}$ , GPa
	$\text{Al}_2\text{O}_3$ $A_1(\text{g})$ $\omega$ , $\text{cm}^{-1}$	GaN $E_2^{\text{high}}$ $\omega/\text{FWHM}$ , $\text{cm}^{-1}$	GaN $A_1(\text{LO})$ $\omega/\text{FWHM}$ , $\text{cm}^{-1}$	$\text{Al}_2\text{O}_3$ $E_{1g}(\text{LO})$ $\omega$ , $\text{cm}^{-1}$	
A (GaN)	415.6	567.0/5.1	732.6/7,9	747.5	0.11
B (n-GaN)	415.6	569.3/5.0	735.2/9,3	748	0.42
C (n <sup>+</sup> -GaN)	415.6	567.2/5.2	732.6/7,9	747.5	0.07

on sapphire substrates [33]. However, the residual biaxial stresses in the n-GaN layer (sample B) were at a higher level of  $\sigma_{xx} \sim 0.4$  GPa, which was probably due to the use of a misoriented sapphire substrate when creating a GaN/c-Al<sub>2</sub>O<sub>3</sub> virtual substrate (see Methods).

During the last stage of the studies, ohmic Ti/Al contacts (20/100 nm) were formed on the surface of the samples using magnetron sputtering. After that, the mobility and concentration of carriers and resistivity were determined according to Hall measurements using the van der Pauw method. The results are given in Table 2.

**Table 2.** Hall measurement data by the van der Pauw method

Sample	Mobility of carriers cm <sup>2</sup> /Vs	Carrier concentration, cm <sup>-3</sup>	Resistivity Ohm·cm
A (GaN)	–	2·10 <sup>16</sup>	–
B (n-GaN)	265	–5·10 <sup>18</sup>	4.7·10 <sup>-2</sup>
C (n <sup>+</sup> -GaN)	105	–6·10 <sup>19</sup>	1·10 <sup>-2</sup>

The contact pads of the test structure of ohmic contacts had an annular geometry with a length of the outer circumference of 400 μm. Using such topology allowed measuring contact resistance without isolating the active elements [34]. As a result, using the transmission line method (TLM) in accordance with [35] allowed determining the contact resistance reduced to the pad width, which for the structure with the n<sup>+</sup>-GaN contact layer (sample C) was ~ 0.11 Ohm·mm, and for the n-GaN contact layer (sample B) ~ 0.5 Ohm·mm. Both values are in agreement with the results of the van der Pauw method (see Table 2).

It should be noted that it appeared challenging to measure the Hall characteristics for the structure with the GaN contact layer (sample A) due to the high values of resistivity (~ 1·10<sup>4</sup> Ohm·cm), which may indicate a sufficiently low intrinsic conductivity of this layer.

#### 4. Conclusions

The study showed that plasma-assisted molecular beam epitaxy can be used to create overgrown GaN, n-GaN, and n<sup>+</sup>-GaN contact layers on GaN/c-Al<sub>2</sub>O<sub>3</sub> virtual substrates under Ga-enriched conditions. This involves effective filtration of dislocations threading from the

buffer GaN layer of the virtual substrate formed by MOCVD.

Residual stresses calculated using the data of Raman microspectroscopy indicate a high structural quality of GaN, n-GaN, and n<sup>+</sup>-GaN contact layers regardless of the level of silicon doping.

Contact resistance reduced to the pad width determined by the transmission line method for the structure with the n<sup>+</sup>-GaN contact layer was ~ 0.11 Ohm·mm, and for the n-GaN contact layer it was ~ 0.5 Ohm·mm.

#### Contribution of the authors

The authors contributed equally to this article.

#### Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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### Information about the authors

*Pavel V. Seregin*, Dr. Sci. (Phys.–Math.), Full Professor, Head of the Department, Department of Solid State Physics and Nanostructures, Voronezh State University (Voronezh, Russian Federation).

<https://orcid.org/0000-0002-6724-0063>  
paul@phys.vsu.ru

*Dmitry L. Goloshchapov*, Cand. Sci. (Phys.–Math.), Assistant Professor, Department of Solid State Physics and Nanostructures, Voronezh State University (Voronezh, Russian Federation).

<https://orcid.org/0000-0002-1400-2870>  
goloshchapov@phys.vsu.ru

*Danila E. Kostomakha*, graduate student, Department of Solid State Physics and Nanostructures, Voronezh State University (Voronezh, Russian Federation).

danilkostomaha@gmail.com

*Yaroslav A. Peshkov*, Cand. Sci. (Phys.–Math.), Research Assistant, Department of Solid State Physics and Nanostructures, Voronezh State University (Voronezh, Russian Federation).

<https://orcid.org/0000-0003-0939-0466>  
tangar77@mail.ru

*Nikita S. Buylov*, Cand. Sci. (Phys.–Math.), Educator, Department of Solid State Physics and Nanostructures, Voronezh State University (Voronezh, Russian Federation).

<https://orcid.org/0000-0003-1793-4400>  
buylov@phys.vsu.ru

*Alisa A. Gaivoronskaya*, student, Department of Solid State Physics and Nanostructures, Voronezh State University (Voronezh, Russian Federation).

alisa.a.gaivoronskaya@gmail.com

*Andrey M. Mizerov*, Cand. Sci. (Phys.–Math.), Leading Researcher, Alferov University (St. Petersburg, Russian Federation).

<https://orcid.org/0000-0002-9125-6452>  
andrey@mizerov@rambler.ru

*Sergey N. Timoshnev*, Cand. Sci. (Phys.–Math.), Leading Researcher, Alferov University (St. Petersburg, Russian Federation).

<https://orcid.org/0000-0002-9294-3342>  
timoshnev@mail.ru

*Maksim S. Sobolev*, Cand. Sci. (Phys.–Math.), Acting Head of the Laboratory of Nanoelectronics, Alferov University (St. Petersburg, Russian Federation).

<https://orcid.org/0000-0001-8629-2064>  
sobolevsm@gmail.com

*Evgeniy V. Ubyivovk*, Cand. Sci. (Phys.–Math.), Senior Researcher, Research Laboratory of Mechanics of Advanced Bulk Nanomaterials for Innovative Engineering Applications, Saint Petersburg State University (St. Petersburg, Russian Federation).

<https://orcid.org/0000-0001-5828-4243>  
ubyivovk@gmail.com

*Valeriy E. Zemlyakov*, Cand. Sci. (Eng.), Leading Researcher, Laboratory of Nanoelectronics Element Base, National Research University of Electronic Technology (Moscow, Russian Federation).

<https://orcid.org/0000-0001-5681-9603>  
vzml@rambler.ru

*Pavel P. Kutsko*, Cand. Sci. (Eng.), General Director, Research Institute of Electronic Technology (Voronezh, Russian Federation).

kutsko@niiet.ru

*Pavel L. Parmon*, Director of Quality, Research Institute of Electronic Technology (Voronezh, Russian Federation).

p.parmon@niiet.ru

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