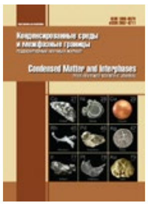

ISSN 1606-867X (Print)
ISSN 2687-0711 (Online)

Condensed Matter and Interphases

Kondensirovannye Sredy i Mezhfaznye Granitsy
https://journals.vsu.ru/kcmf/

Original articles

Research article
https://doi.org/10.17308/kcmf.2023.25/11474

Confirmation of spontaneous doping of GaN nanowires grown on vicinal SiC/Si substrate by electron beam induced current mapping

R. R. Reznik[1–3]✉, V. O. Gridchin[1–3], K. P. Kotlyar[1–3], V. V. Neplokh[2], A. V. Osipov[4], S. A. Kukushkin[4], O. Saket[5], M. Tchernycheva[5], G. E. Cirlin[1–3]

*[1]St. Petersburg State University,
7-9 Universitetskaya Embankment, St. Petersburg 199034, Russian Federation*

*[2]Alferov University,
8/3 Khlopina st., St. Petersburg 194021, Russian Federation*

*[3]Institute for Analytical Instrumentation of the Russian Academy of Sciences,
26 Rizhsky st., St. Petersburg 190103, Russian Federation*

*[4]Institute for Problems in Mechanical Engineering of the Russian Academy of Science,
61 Boljshoy prospekt V.O., St. Petersburg 199178, Russian Federation*

*[5]Centre de Nanosciences et de Nanotechnologies (C2N), University Paris-Saclay,
10 Boulevard Thomas, Gobert, Palaiseau 91120, France*

Abstract

This study is devoted to the confirmation of spontaneous doping of GaN nanowires grown on vicinal SiC/Si hybrid substrates by electron beam induced current mapping.

GaN nanowires (NWs) were grown on singular and vicinal SiC/Si substrates by molecular beam epitaxy with nitrogen plasma activation. The morphological properties of the NWs were studied by scanning electron microscopy. The electrophysical properties of the obtained nanostructures were studied by electron beam induced current mapping.

By electron beam induced current mapping, we confirmed the spontaneous doping of the GaN NWs grown on vicinal SiC/Si wafers. It was also shown that the GaN NWs grown on singular SiC/Si substrates did not exhibit an induced current signal, indicating that they were not doped.

Keywords: Semiconductors, GaN, Nanowires, Molecular beam epitaxy, Spontaneous doping, Silicon, Silicon carbide, Electron beam induced current method

Funding: The synthesis of the experimental samples was financially supported by the Russian Science Foundation, Grant No. 23-79-00012. The morphological studies of the obtained samples were financially supported by the Ministry of Science and Higher Education (FSRM 2023-0007). The electrophysical studies of the obtained samples were supported by PHC KOLMOGOROV project No. 43784UJ (2019).

For citation: Reznik R. R., Gridchin V. O., Kotlyar K. P., Neploh V. V., Osipov A. V., Kukushkin S. A., Saket O., Tchernycheva M., Cirlin G. E. Confirmation of spontaneous doping of GaN nanowires grown on vicinal SiC/Si substrate by electron beam induced current mapping. *Condensed Matter and Interphases*. 20223;25(4): 526–531. https://doi.org/10.17308/kcmf.2023.25/11474

Для цитирования: Резник Р. Р., Гридчин В. О., Котляр К. П., Неплох В. В., Осипов А. В., Кукушкин С. А., Saket O., Tchernycheva M., Цырлин Г. Э. Подтверждение методом картирования тока, наведенного электронным пучком, самопроизвольного легирования GaN нитевидных нанокристаллов из вицинальной подложки SiC/Si. *Конденсированные среды и межфазные границы*. 2023;25(4): 526–531. https://doi.org/10.17308/kcmf.2023.25/11474

✉ Rodion R. Reznik, e-mail: moment92@mail.ru

1. Introduction

Nowadays, wide-gap semiconductor nanostructures based on nitride compounds are of great interest to researchers due to their unique optical, electrophysical, transport, and other properties [1-3]. Nitride nanostructures are already used in a number of applications, such as LEDs, solar cells, transistors, single photon sources, and others [4–7]. Modern methods for the formation of semiconductor nanostructures make it possible to grow two-dimensional, one-dimensional, and zero-dimensional structures based on nitride compounds, as well as their combinations [8–11]. One of the most common methods for growing such nanostructures is molecular beam epitaxy (MBE). The advantages of this method include the ultrahigh vacuum level in the growth chamber, low growth rate, precise control over the growth processes, and, as a consequence, high crystallographic and optical quality of the resulting nanostructures [12–14]. It is important to note that nonplanar nitride nanostructures, such as nanowires (NWs), in some cases make it possible to increase the efficiency of devices or design a new generation of devices [15, 16]. Moreover, producing nanostructures in the form of nanowires allows us to solve the issue of integrating nitride compounds with mismatched substrates [17, 18]. Nevertheless, in most cases, to develop applications based on nitride nanowires, it is necessary to obtain nanostructure sites of P- and/or N-type conductivity to enable contact with the nanowires or the formation of the P-N junctions. To obtain efficient devices, it is necessary to consider all doping mechanisms of semiconductor nanostructures. As we demonstrated earlier, the substrate on which the nanostructures grow can also influence the type and level of doping of the nanowires [19]. In this study, we proved the fundamental possibility of growing GaN nanowires on singular and vicinal Si hybrid substrates with a thin SiC layer on the surface. Due to the optical studies, we observed the effect of spontaneous doping of the GaN nanowires grown on vicinal substrates with Si atoms. We also described the mechanism of doping of GaN nanowires with Si atoms from the SiC layer. It should be noted that the doping level of nanowires turned out to be higher than the solubility level of Si in GaN, which provides prospects for new applications based on this material.

The aim of the study was to confirm the spontaneous doping of the GaN nanowires grown on vicinal SiC/Si hybrid substrates by electron beam induced current mapping (induced current method).

2. Experimental

The GaN nanowires were grown using a Riber Compact 12 MBE unit equipped with a Ga effusion source and a plasma nitrogen source. Singular and vicinal Si(111) wafers with a thin SiC buffer layer were used as substrates. The technology for growing GaN nanowires on SiC/Si substrates is described in detail in [19]. All samples were grown under identical conditions. During the first stage, the substrate was placed in the growth chamber and heated to 950 °C in order to thermally clean the surface. After holding the substrate at a high temperature for 20 min, the substrate temperature was lowered to the growth temperature of 870 °C. During the next stage, nitrogen plasma was initiated at the source power of 520 W, with the N^+ flux rate of 1.5 sccm. Then, the Ga source was opened to grow GaN nanowires on the substrate surface. The total GaN nanowire growth time was 16 hours. The Ga flux from the source had a pressure of $1.6 \cdot 10^{-7}$ according to preliminary calibrations using the Bayard–Alpert gauge.

Changes in the surface morphology were recorded *in situ* by reflected high energy electron diffraction (RHEED), which indicated the wurtzite crystallographic phase of the growing nanostructures. The morphological properties of the grown nanostructures and the induced current maps were analyzed at room temperature using a Hitachi SU8000 scanning electron microscope (SEM) equipped with a Gatan DigiScan system for electron beam induced current mapping. Micromanipulators (probes) connected to a SR570 low-noise current preamplifier were used to contact the grown nanostructures. The methodology of the induced current measurements is described in detail in [20].

3. Results and discussion

The GaN nanowires were formed in the (111) direction on the singular SiC/Si substrate and on the flat surface of the vicinal SiC/Si substrate. However, the nanowires grown on the slopes of the vicinal substrate also had another direction. The average height of the GaN NWs was 1.5 μm, and their average diameter was 300 nm.

The doping of the GaN NWs grown on vicinal SiC/Si substrates was confirmed by electron beam induced current mapping. In the induced current experiment, a tungsten probe contacted the top of the GaN nanowire, which generated a built-in electric field near the probe/nanowire contact due to the formation of a Schottky barrier at the tungsten/GaN interface. This built-in electric field effectively separated the electron-hole pairs generated by the electron beam. So, the induced current signal could be registered by an external measurement circuit connected to the probes. Moreover, the doping level of GaN nanowires could be estimated by the size of the space charge region (SCR), which is plotted as a plateau on the signal profile on the induced current maps [21]. The resistivity of undoped nanowires was high, the field magnitude of the Schottky barrier was low, and, consequently, the size of the SCR was large. So, electron-hole pairs could not be effectively separated by the built-in electric field. Together with the high resistivity of the NWs, it resulted in a very small measured value of the induced current signal.

A typical SEM image with a research probe and a characteristic induced current map for the GaN NWs grown on a vicinal SiC/Si substrate are shown in Figs. 1a and 1b, respectively. In Fig. 1a, it can be seen that the probe touches the apex of the GaN NWs. As can be seen from Fig. 1b, a GaN nanowire grown on vicinal SiC/Si substrate has a clearly distinguishable region of the induced current signal near the probe/nanowire interface, indicating an extremely high degree of NW doping.

The opposite pattern was observed for the GaN NWs grown on singular SiC/Si substrates. Figures 2a and 2b show a typical SEM image with a research probe and a characteristic map of the induced current for such a nanowire. As can be seen from the figures, a nanowire grown on singular GaN substrate does not manifest the induced current signal, which proves that it was not doped.

4. Conclusions

Thus, we confirmed the spontaneous doping of GaN nanowires grown on vicinal SiC/Si substrates by electron beam induced current mapping. It was also shown that the GaN NWs grown on singular SiC/Si substrates did not exhibit an induced current signal, indicating that they were not doped.

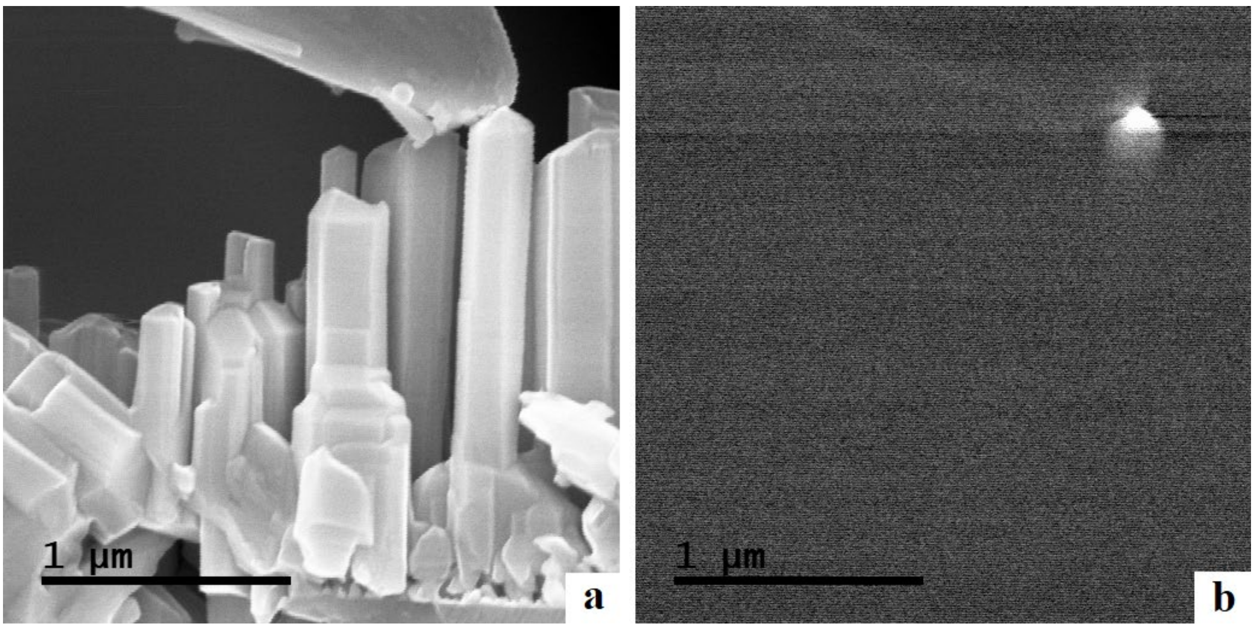

Fig. 1. Typical SEM image with a research probe (a) and a characteristic map of the induced current for a GaN NW grown on vicinal SiC/Si substrate (b)

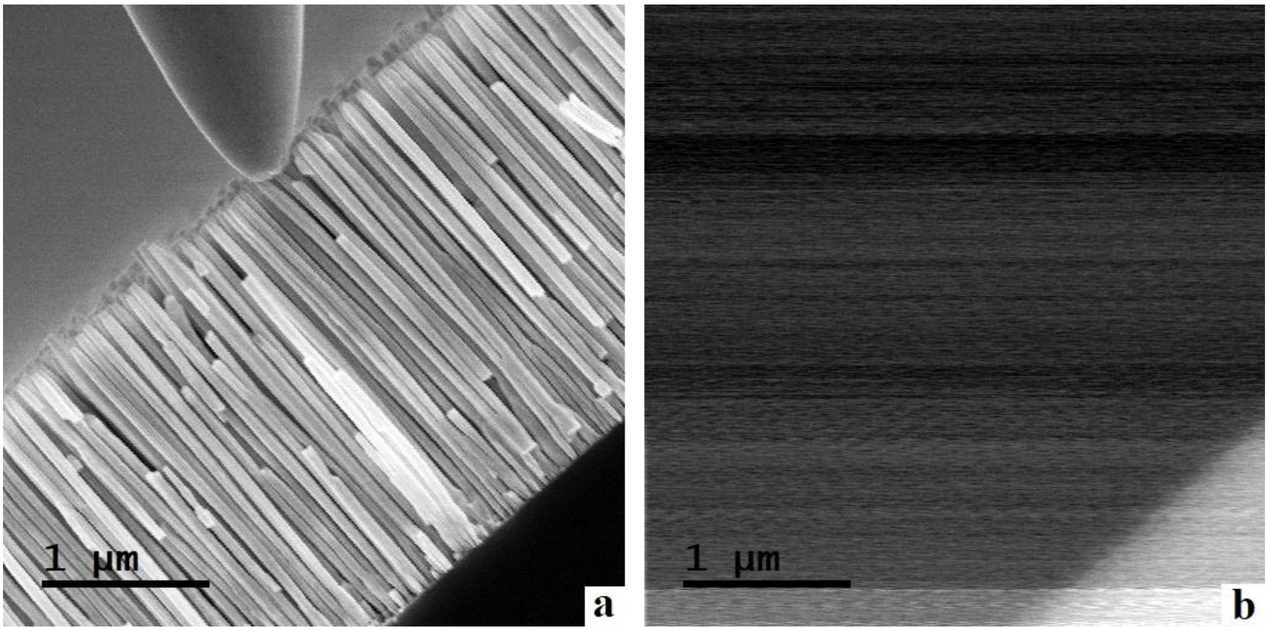

Fig. 2. Typical SEM image with a research probe (a) and a characteristic map of the induced current for a GaN NW grown on singular SiC/Si substrate (b)

Author contributions

R. R. Reznik – growing the samples, planning the experiment, text writing, and final conclusions. V. O. Gridchin – growing the samples. K. P. Kotlyar – SEM measurements of the samples. V. V. Neplokh – analysis of the results. A. V. Osipov – production of the substrates for growing the samples. S. A. Kukushkin – production of the substrates for growing the samples. O. Saket – study of the electrical properties of the samples. M. Tchernycheva – study of the electrophysical properties of the samples and analysis of the results. G. E. Cirlin – planning the experiments, analysis of the results.

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.

References

1. Kente T., Mhlanga S. D. Gallium nitride nanostructures: Synthesis, characterization and applications. *Journal of Crystal Growth*. 2016;444: 55–72. https://doi.org/10.1016/j.jcrysgro.2016.03.033

2. Patra S. K., Schulz S. Electrostatic built-in fields in wurtzite III-N nanostructures: Impact of growth plane on second-order piezoelectricity. *Physical Review B*. 2017;96(15): 155307. http://dx.doi.org/10.1103/PhysRevB.96.155307

3. Gridchin V. O., Kotlyar K. P., Reznik R. R., Borodin B. R., Kudryashov D. A., Alekseev P. A., Cirlin G. E. Electrical properties of InGaN nanostructures with branched morphology synthesized via MBE on *p*-type Si (111). *Journal of Physics: Conference Series*. 2020;1695(1): 012030. https://doi.org/10.1088/1742-6596/1695/1/012030

4. Pearton S. J., Ren F. GaN electronics. *Advanced Materials*. 2000;12(21): 1571–1580. https://doi.org/10.1002/1521-4095(200011)12:21<1571::AID-ADMA1571>3.0.CO;2-T

5. Chen F., Ji X., Lau S. P. Recent progress in group III-nitride nanostructures: From materials to applications. *Materials Science and Engineering: R: Reports*. 2020;142: 100578. https://doi.org/10.1016/j.mser.2020.100578

6. Gridchin V. O., Kotlyar K. P., Reznik R. R., … Cirlin G. G. Multi-colour light emission from InGaN nanowires monolithically grown on Si substrate by MBE. *Nanotechnology*. 2021;32(33): 335604. https://doi.org/10.1088/1361-6528/ac0027

7. Tijent F. Z., Voss P., Faqir M. Recent advances in InGaN nanowires for hydrogen production using photoelectrochemical water splitting. *Materials Today Energy*. 2023;33: 101275. https://doi.org/10.1016/j.mtener.2023.101275

8. Mäntynen H., Anttu N., Sun Z., Lipsanen H. Single-photon sources with quantum dots in III–V nanowires. *Nanophotonics*. 2019;8(5): 747–769. https://doi.org/10.1515/nanoph-2019-0007

9. Leandro L., Gunnarsson C. P., Reznik R., … Akopian, N. Nanowire quantum dots tuned to atomic resonances. *Nano Letters*. 2018;18(11): 7217–7221. https://doi.org/10.1021/acs.nanolett.8b03363

10. Heiss M., Fontana Y., Gustafsson A., … Fontcuberta i Morral A. Self-assembled quantum dots in a nanowire system for quantum photonics. *Nature Materials*. 2013;12(5): 439–444. https://doi.org/10.1038/NMAT3557

11. Deshpande S., Frost T., Yan L., … Bhattacharya P. Formation and nature of InGaN quantum dots in GaN nanowires. *Nano Letters*. 2015;15(3): 1647–1653. https://doi.org/10.1021/nl5041989

12. Consonni V. Self-induced growth of GaN nanowires by molecular beam epitaxy: A critical review of the formation mechanisms. *Physica Status Solidi (RRL)–Rapid Research Letters*. 2013;7(10): 699–712. https://doi.org/10.1002/pssr.201307237

13. Arthur J. R. Molecular beam epitaxy. *Surface Science*. 2002;500(1-3): 189–217.

14. Dubrovskii V. G. Theory of diffusion-induced selective area growth of III-V nanostructures. *Physical Review Materials*. 2023;7(2): 026001. https://doi.org/10.1103/PhysRevMaterials.7.026001

15. Tribu A., Sallen G., Aichele T., … Kheng K. A high-temperature single-photon source from nanowire quantum dots. *Nano Letters*. 2008;8(12): 4326–4329. https://doi.org/10.1021/nl802160z

16. Alekseev P. A., Sharov V. A., … Lähderanta E. Piezoelectric current generation in wurtzite GaAs nanowires. *Physica Status Solidi (RRL) – Rapid Research Letters*. 2018;12(1): 1700358. https://doi.org/10.1002/pssr.201700358

17. Cirlin G. E., Reznik R. R., Shtrom I. V., … Akopian N. AlGaAs and AlGaAs/GaAs/AlGaAs nanowires grown by molecular beam epitaxy on silicon substrates. *Journal of Physics D: Applied Physics*. 2017;50(48): 484003. https://doi.org/10.1088/1361-6463/aa9169

18. Cirlin G. E., Dubrovskii V. G., Soshnikov I. P., … Glas F. Critical diameters and temperature domains for MBE growth of III–V nanowires on lattice mismatched substrates. *Physica Status Solidi (RRL) – Rapid Research Letters*. 2009:3(4): 112-114. https://doi.org/10.1002/pssr.200903057

19. Talalaev V. G., Tomm J. W., Kukushkin S. A., … Cirlin G. E. Ascending Si diffusion into growing GaN nanowires from the SiC/Si substrate: up to the solubility limit and beyond. *Nanotechnology*. 2020;31(29): 294003. https://doi.org/10.1088/1361-6528/ab83b6

20. Lavenus P., Messanvi A., Rigutti L. … Tchernycheva M. Experimental and theoretical analysis of transport properties of core–shell wire light emitting diodes probed by electron beam induced current microscopy. *Nanotechnology*. 2014;25(25): 255201. https://doi.org/10.1088/0957-4484/25/25/255201

21. Yakimov E. B., Borisov S. S., Zaitsev S. I. EBIC measurements of small diffusion length in semiconductor structures. *Semiconductors*. 2007;41: 411–413. https://doi.org/10.1134/s1063782607040094

Information about the authors

Rodion R. Reznik, Cand. Sci. (Phys.–Math.), Head of the Laboratory, St. Petersburg University; Alferov University; Institute for Analytical Instrumentation of the Russian Academy of Sciences (St. Petersburg, Russian Federation).

https://orcid.org/0000-0003-1420-7515
moment92@mail.ru

Vladislav O. Gridchin, Cand. Sci. (Phys.–Math.), Junior Researcher, St. Petersburg University; Alferov University; Institute for Analytical Instrumentation of the Russian Academy of Sciences (St. Petersburg, Russian Federation).

https://orcid.org/0000-0002-6522-3673
gridchinvo@gmail.com

Konstantin P. Kotlyar, Cand. Sci. (Phys.–Math.), Junior Researcher, St. Petersburg University; Alferov University; Institute for Analytical Instrumentation of the Russian Academy of Sciences (St. Petersburg, Russian Federation).

https://orcid.org/0000-0002-0305-0156
konstantin21kt@gmail.com

Vladimir V. Neploh, Cand. Sci. (Phys.–Math.), Senior Researcher, Alferov University (St. Petersburg, Russian Federation).

https://orcid.org/0000-0001-8158-0681
vneplox@gmail.com

Andrei V. Osipov, Dr. Sci. (Phys.–Math.), Main Researcher, Institute for Problems in Mechanical Engineering of the Russian Academy of Sciences (St. Petersburg, Russian Federation).

https://orcid.org/0000-0002-2911-7806
andrey.v.osipov@gmail.com

Sergey A. Kukushkin, Dr. Sci. (Phys.–Math.), Head of Laboratory, Institute for Problems in Mechanical Engineering of the Russian Academy of Sciences (St. Petersburg, Russian Federation).

https://orcid.org/0000-0002-2973-8645
sergey.a.kukushkin@gmail.com

Omar Saket, PhD, Researcher, Centre de Nanosciences et de Nanotechnologies (C2N), de l'université Paris-Saclay (Palaiseau, France).

https://orcid.org/0000-0002-9002-5871
omar.saket@c2n.upsaclay.fr

Maria Tchernycheva, Dr. Sci. (Phys.–Math.), Head of Laboratory, Centre de Nanosciences et de Nanotechnologies (C2N), de l'université Paris-Saclay (Palaiseau, France).

https://orcid.org/0000-0003-4144-0793

maria.tchernycheva@c2n.upsaclay.fr

George E. Cirlin, Dr. Sci. (Phys.–Math.), Head of Laboratory, St Petersburg University; Alferov University; Institute for Analytical Instrumentation of the Russian Academy of Sciences (St. Petersburg, Russian Federation).

https://orcid.org/0000-0003-0476-3630

cirlin.beam@mail.ioffe.ru

Received 20.09.2023; approved after reviewing 28.09.2023; accepted for publication 16.10.2023; published online 26.12.2023.

Translated by Anastasiia Ananeva