

# SEMICONDUCTORS

Founded by Ioffe Institute

Published since January 1967

12 issues annually

Editor-in-Chief: Sergei V. Ivanov

## Editorial Board:

Vasilii V. Bel'kov (Deputy Editor-in-Chief), Vadim P. Evtikhiev (Deputy Editor-in-Chief),  
Mikhail O. Nestoklon (Managing Editor), Alexander A. Andronov, Alexander L. Aseev,  
Levon V. Asryan, Alexey A. Gippius, Yuri V. Gulyaev, Mikhail I. Dyakonov, Vadim V. Emtsev,  
Dmitry R. Khokhlov, Semen G. Konnikov, Petr S. Kop'ev, Zakhary F. Krasilnik,  
Alexander V. Latyshev, Nikolay N. Ledentsov, Sergei A. Nемов, Sergei G. Tikhodeev, Andrei G. Zabrodskii

*ISSN: 1063-7826 (print), 1090-6479 (online)*

SEMICONDUCTORS is the English translation  
of ФИЗИКА И ТЕХНИКА ПОЛУПРОВОДНИКОВ  
(FIZIKA I TEKHNIKA POLUPROVODNIKOV)

Published by Ioffe Institute

Saint Petersburg  
Ioffe Institute

© Ioffe Institute, 2025  
© Composed by the Editorial Board of the Journal  
FIZIKA I TEKHNIKA POLUPROVODNIKOV, 2025

UDC 621.315.592

# The investigation of the impact of nano-structured AlN/Si(100) templates for the growth of semipolar AlN(10 $\bar{1}$ 1) layers

© V.N. Bessolov<sup>1</sup>, E.V. Konenkova<sup>1</sup>, S.N. Rodin<sup>1</sup>, A.V. Solomnikova<sup>2</sup>, Sh.Sh. Sharofidinov<sup>1</sup><sup>1</sup> Ioffe Institute,  
194021 St. Petersburg, Russia<sup>2</sup> St. Petersburg State Electrotechnical University „LETI“,  
197022 St. Petersburg, Russia

E-mail: lena@triat.ioffe.ru

Received February 10, 2025

Revised April 16, 2025

Accepted April 23, 2025

Semipolar AlN(10 $\bar{1}$ 1) layers grown by hydride vapour-phase epitaxy (HVPE) on AlN/Si(100) templates were studied by X-ray diffraction, atomic force, and scanning electron microscopy. Two types of structures were prepared as AlN/Si(100) templates on a Si(100) substrate with a symmetrical V-shaped nano-relief with a depth of about  $\sim 40$  nm at a step of 40–60 nm, in which one of the templates had only a layer grown by metal organic chemical vapor phase deposition (MOCVD), and the second also had an additional intermediate layer a layer grown by reactive magnetron sputtering (RMS). It was shown that all structures formed the HVPE layer of AlN in the form of blocks, the sizes of which were larger for the template grown by MOCVD than on the template grown by two methods. It was found that the templates affect the structure of the AlN layer: on a template grown by two methods, with a thickness of 115 microns separated from the substrate, X-ray diffraction showed the presence of (0002) and (10 $\bar{1}$ 1) blocks with FWHM  $\omega_\theta = 5$  arcgrad, and on the MOCVD template, the structure of AlN(10 $\bar{1}$ 1) with  $\omega_\theta = 1.5$  arcgrad with cracks in the direction perpendicular to the V-groove at a thickness of 7 microns.

**Keywords:** aluminum nitride, nano-structured silicon substrate, hydride vapour-phase epitaxy.

DOI: 10.61011/SC.2025.01.61067.7599

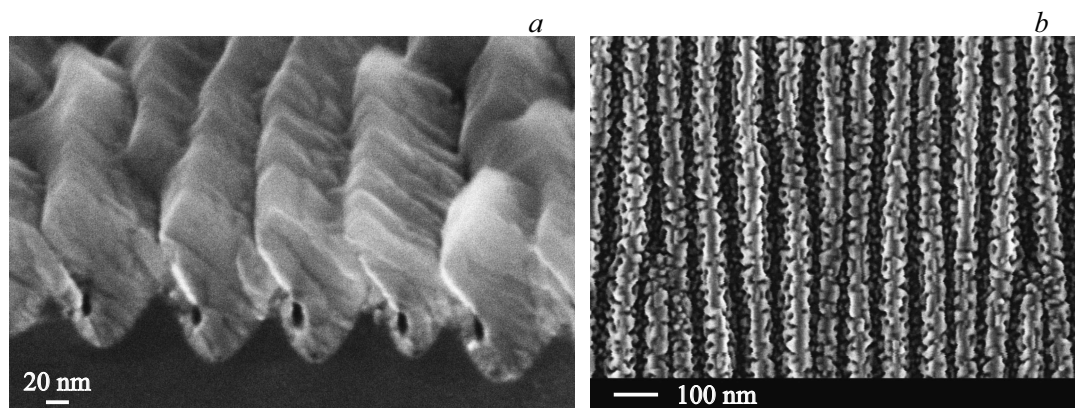
## 1. Introduction

Owing to their optical and electrical properties, wide-band III-N semiconductors have become attractive for manufacturing optoelectronic devices over recent years [1]. From this group, aluminum nitride (AlN) has the widest band gap, high ultraviolet transparency, outstanding piezoelectric properties and decent thermal stability [2]. Note that AlN as a buffer layer plays an important role in gallium nitride epitaxy on a silicon substrate because it solves a problem of reverse etching caused by reactions between Ga and Si atoms at high temperatures that destroy the epitaxial GaN layers [3].

Since bulk substrates are limited in size and very expensive, AlN layers are grown on foreign substrates, such as sapphire and silicon [4]. Moreover, silicon substrates are more promising for integration of silicon-based and III-nitride-based electronics because they have a set of advantages such as low cost, large sizes and high heat conductivity [5]. Silicon substrate can be easily removed by means of chemical etching. Large ( $\sim 19\%$ ) mismatch of lattices between AlN and Si(111) causes structural deformation and high dislocation density. Moreover, mismatch ( $\sim 43\%$ ) of thermal expansion coefficients between AlN and Si stimulates cracking during cooling to room temperature [6], which affects functioning of III-nitride-based devices [7]. Therefore, growth of high quality thick (of

several microns) AlN layers on silicon substrate is a difficult task. Attempts to synthesize hexagonal AlN on a Si(100) substrate showed that growth of layers was additionally hindered by the fact that layers grew in two crystal-lattice orientations during the synthesis of a hexagonal AlN structure on a cubic Si(100) structure [8].

Deformation in AlN heterostructures on silicon can be reduced by employing magnetron sputtering [9]. Synthesis temperature in this method is reduced considerably leading to reduction of layer deformation and reduces the mutual diffusion between Si and AlN [10]. In addition, the magnetron sputtering method is well proven for deposition of amorphous and textured thin films, but due to lower growth temperatures it can potentially simplify the integration of III-nitride layers with the silicon-based metal-oxide-semiconductor technology [11]. Currently optoelectronic devices based on III-nitride structures are mainly accommodated on polar GaN substrates that contain undesirable strong polarization field in devices. One of methods to avoid this is to grow nonpolar and semipolar bulk substrates because such structures have high potential of increasing internal quantum efficiency of LEDs [12]. Attempts are currently undertaken to grow semipolar gallium and aluminum nitrides on micro- [13] and nanostructured Si(100) [8] substrates, where it is proposed to use an inclined Si(111) face for synthesis. Hydride vapor phase epitaxy (HVPE) was successfully used to make



**Figure 1.** SEM images of 60 nm AlN layers after RMS (*a*) and of 30 nm AlN layers after MOCVD (*b*) processes.

thick ( $\sim 4.5\mu\text{m}$ ) AlN(0001) layers on a nanostructured *c*-oriented sapphire substrate [14], however, no information regarding fabrication of semipolar AlN layers separated from a substrate was found in the literature.

The objective of the study is to investigate the influence of nanostructured AlN/Si(100) templates on the fabrication of micron-sized semipolar AlN(10 $\bar{1}$ 1) layers.

## 2. Experiment

To meet the set goal, semipolar AlN(10 $\bar{1}$ 1) layers grown by the hydride vapor phase epitaxy (HVPE) on AlN/Si(100) templates were studied. Two types of structures, A and B, were prepared as AlN/Si(100) templates on a Si(100) substrate with symmetrical V-shaped nanorelief with a depth of  $\sim 40\text{ nm}$  at  $40\text{--}60\text{ nm}$  steps (NP-Si(100)). Structure A was made as follows: first, the reactive magnetron sputtering (RMS) method was used to grow 60 nm AlN layers (Figure 1, *a*), then the metalorganic vapour phase epitaxy (MOCVD) method was used to grow a 300 nm layer and finally the HVPE method was used to grow a semipolar AlN layer with a thickness up to  $115\mu\text{m}$  that was separated from the substrate. Structure B had 300 nm AlN layers grown by the MOCVD method (Figure 1, *b*) and then a layer up to  $7\mu\text{m}$  in thickness grown by the HVPE method. Before the epitaxy, NP-Si(100) were treated in a fluoridic acid water solution in standard conditions and then by the RMS or MOCVD methods at deposition temperatures of  $450^\circ\text{C}$  and  $1080^\circ\text{C}$ , respectively, then AlN layers were grown at a rate of  $\sim 50\mu\text{m/h}$  using the HVPE method at  $950^\circ\text{C}$  in argon. The AlN layer was formed on NP-Si(100) either using the RMS method in a similar way as in [15] or the MOCVD [15] and HVPE methods in a similar way as in [16]. The grown samples were examined by the X-ray diffraction, scanning electron microscopy (SEM) and atomic force microscopy (AFM). For structures grown on template A, the Si(100) substrate was removed by the chemical etching method in ( $\text{HNO}_3:\text{HF} = 2:1$ ).

## 3. Findings

Fabrication of structures A using the RMS method made it possible to form a solid AlN layer on the Si(111) and Si( $\bar{1}\bar{1}$ 1) faces at the initial stage with maintaining the nanostructured surface (Figure 1, *a*) to avoid silicon oxidation when the structures were moved from the RMS reactor to the MOCVD unit.

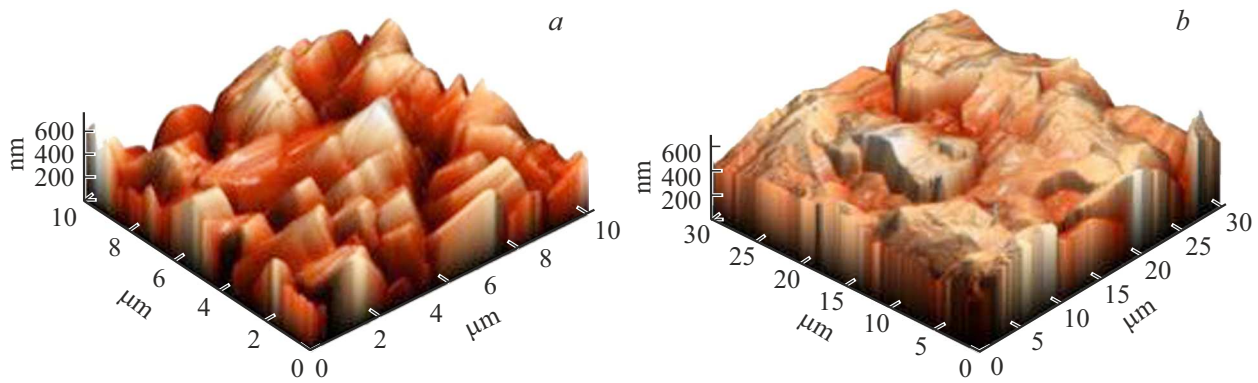
X-ray diffraction analysis showed that the layer synthesized by the RMS method in structure A had a textured block form consisting of cubic and hexagonal nuclei similar to [15]. SEM image of the layer surface showed that it consisted of periodic columnar crystallite blocks perpendicular to the Si(111) face, and the distance between hump peaks corresponded to the profile step on the NP-Si(100) substrate surface (Figure 1, *a*).

Fabrication of structure B by the MOCVD method showed (Figure 1, *b*) that at the initial growth stage the 30-nm layer, on the one hand, covers the substrate surface and, on the other hand, keeps the initial form of the substrate. Atomic-force microscopy image of the structure A surface with the  $\sim 1\mu\text{m}$  HVPE layer showed that there were both polar and semipolar oriented blocks (Figure 2, *a*).

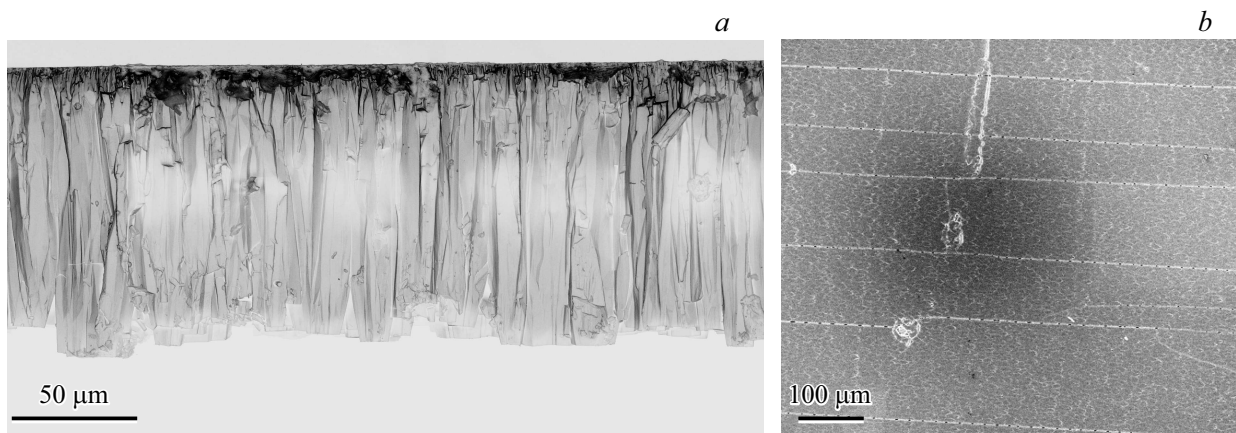
Atomic-force microscopy image of the  $\sim 1\mu\text{m}$  AlN layer grown by the HVPE method on structure B showed that there were semipolar blocks larger than those on structure A (Figure 2, *a*, *b*).

X-ray diffraction analysis of structure A showed a semipolar block structure of layers with the (10 $\bar{1}$ 1) and (0002) orientation, while the X-ray diffraction FWHM was  $\omega_\theta = 5\text{ arcgrad}$ . SEM image of structure A with a  $115\mu\text{m}$  layer thickness showed that there were no cracks, due to which the substrate could be removed and a bulk semipolar block layer could be made (Figure 3, *a*).

X-ray diffraction analysis of structure B showed that layers had a semipolar block structure (10 $\bar{1}$ 1) with the X-ray diffraction FWHM of  $\omega_\theta = 1.5\text{ arcgrad}$ . Surfaces of structure B  $7\mu\text{m}$  in thickness after HVPE examined by the electron microscopy demonstrated cracks perpendicular to



**Figure 2.** AFM image of the surface of A (*a*) and B (*b*) structures with a  $\sim 1\ \mu\text{m}$  HVPE layer.



**Figure 3.** SEM image of the AlN layer cross-section after removal of structure A substrate (*a*) and of structure B surface (*b*).

the groove at  $100\text{--}200\ \mu\text{m}$  steps in the AlN( $10\bar{1}1$ ) layer (Figure 3, *b*).

#### 4. Results and discussion

Data concerning the different sizes and form of AlN blocks for A and B obtained from the atomic-force microscopy of layers may be explained as follows.

Difference in sizes and form of AlN blocks for samples A and B grown on the Si substrates may be explained using a model describing AlN growth at different temperatures on the sapphire substrate. At high temperatures of AlN epitaxy on the sapphire substrate, a surface face has a form of the (0001) plane because its free energy is the lowest. When the temperature decreases below a certain value, then other crystal planes such as ( $10\bar{1}1$ ) will have a lower surface energy than the (0001) plane and the AlN( $10\bar{1}1$ ) plane becomes a face on the surface of the growing AlN layer on the sapphire substrate at the epitaxy temperatures of  $< 1390\ ^\circ\text{C}$  [10]. However, the face growth rate influences the face structure: at a higher growth rate, formation of the ( $10\bar{1}1$ ) face prevails with respect to the (0001) face [12].

Formation of larger blocks of structures B may be explained using a model of dependence of the Al adatom diffusion length on the AlN surface on the atomic structure of layer surface. Note that employment of the AlN buffer layer leads to a decrease in the bond energy and diffusion barrier for Al adatoms on the AlN layer surface compared with the Si surface leading to improved migration and enhanced coalescence processes.

The surface diffusion length for epitaxy on structure A is supposed to be shorter because the surface of the first layer consists of fine-grain blocks inherent in the low-temperature RMS layer (Figure 2, *a*) compared with the magnitude of diffusion for a structure that was grown at high temperature using the MOCVD method. This leads to larger sizes of blocks and in future to the growth of a semipolar layer with lower X-ray diffraction FWHM  $\omega_\theta = 1.5\ \text{arcgrad}$ . At thicknesses of  $7\ \mu\text{m}$ , such layers appeared to show crack lines spaced at  $100\text{--}200\ \mu\text{m}$  (Figure 3, *b*). This is caused by the appearance of deformation along the V-groove due to the difference in thermal expansion coefficients of AlN and Si. In the direction perpendicular to the groove, this deformation is removed by the presence of surface relief with a  $40\ \text{nm}$  step. In structure A, formation of fine-grain

blocks removes deformation in both directions and doesn't induce cracking even in the 115- $\mu\text{m}$  AlN layers.

## 5. Conclusion

Thus, the study of semipolar AlN(10 $\bar{1}$ 1) layers grown by the HVPE method on AlN templates made by the reactive magnetron sputtering and metalorganic vapour phase epitaxy on the Si(100) substrate with symmetric V-shaped nanorelief has shown that templates A and B demonstrate different results of HVPE growth of the thick AlN layer:

- layers were formed as blocks whose sizes were larger than those for templates B;
- 7  $\mu\text{m}$  semipolar AlN(10 $\bar{1}$ 1) layers grown on template B
- had the X-ray diffraction FWHM  $\omega_\theta = 1.5$  arcgrad and cracks in one of directions;
- 115  $\mu\text{m}$  AlN layer was separated from template A, had the X-ray diffraction FWHM  $\omega_\theta = 5$  arcgrad and consisted of the (0002) and (10 $\bar{1}$ 1) blocks.

## Conflict of interest

The authors declare no conflict of interest.

## References

- [1] R.S. Pengelly, S.M. Wood, J.W. Milligan, S.T. Sheppard, W.L. Pribble. IEEE Trans. Microw. Theory Techn., **60** (6), 1764 (2012). <https://doi.org/10.1109/TMTT.2012.2187535>
- [2] D. Zhu, D.J. Wallis, C.J. Humphreys. Rep. Progr. Phys., **76** (10), 106501 (2013). <https://doi.org/10.1088/0034-4885/76/10/106501>
- [3] J.J. Freedman, A. Watanabe, Y. Yamaoka, T. Kubo, T. Egawa. Phys. Status Solidi A, **213**, 424 (2016). <https://doi.org/10.1002/pssa.201532601>
- [4] L. Huang, Y. Li, W. Wang, X. Li, Y. Zheng, H. Wang, Z. Zhang, G. Li. Appl. Surf. Sci., **435**, 163 (2018). <https://doi.org/10.1016/j.apsusc.2017.11.002>
- [5] Y. Sun, K. Zhou, M. Feng, Z. Li, Y. Zhou, Q. Sun, J. Liu, L. Zhang, D. Li, X. Sun, D. Li, Sh. Zhang, M. Ikeda, H. Yang. Light: Sci. Appl., **7** (1), 13 (2018). <https://doi.org/10.1038/s41377-018-0008-y>
- [6] Z.-Z. Zhang, J. Yang, D.-G. Zhao, F. Liang, P. Chen, Z.-S. Liu. Chin. Phys. B, **32** (2), 028101 (2023). <https://doi.org/10.1088/1674-1056/ac6b2b>
- [7] A. Bardhan, S. Raghavan. J. Cryst. Growth, **578**, 126418 (2022). <https://doi.org/10.1016/j.jcrysgro.2021.126418>
- [8] V.N. Bessolov, E.V. Konenkova. Zh. Tekh. Fiz., **93** (9), 1235 (2023). (in Russian). <https://doi.org/10.21883/JTF.2023.09.56211.31-23>
- [9] H.Y. Liu, G.S. Tang, F. Zeng, F. Pan. J. Cryst. Growth, **363**, 80 (2013). <https://doi.org/10.1016/j.jcrysgro.2012.10.008>
- [10] T. Yamada, T. Tanikawa, Y. Honda, M. Yamaguchi, H. Amano. Jpn. J. Appl. Phys., **52** (8S), 08JB16 (2013). <https://doi.org/10.7567/JJAP.52.08JB16>
- [11] A. Dadgar, F. Hrich, R. Borgmann, J. Blsing, G. Schmidt, P. Veit, J. Christen, A. Strittmatter. Phys. Status Solidi A, **2200609** (2022). <https://doi.org/10.1002/pssa.202200609>
- [12] Y. Zhao, S.-H. Oh, F. Wu, Y. Kawaguchi, S. Tanaka, K. Fujito, J.S. Speck, S.P. DenBaars, S. Nakamura. Appl. Phys. Express, **6**, 062102 (2013). <https://doi.org/10.7567/APEX.6.062102>
- [13] Y. Honda, N. Kameshiro, M. Yamaguchi, N. Sawaki. J. Cryst. Growth, **242**, 82 (2002). [https://doi.org/10.1016/S0022-0248\(02\)01353-2](https://doi.org/10.1016/S0022-0248(02)01353-2)
- [14] Ch. Wang, X.-D. Gao, D.-D. Li, J.-J. Chen, J.-F. Chen, X.-M. Dong, X. Wang. Chin. Phys. B, **32**, 026802 (2023). <https://doi.org/10.1088/1674-1056/ac6865>
- [15] V.N. Bessolov, M.E. Kompan, E.V. Konenkova, T.A. Orlova, S.N. Rodin, A.V. Solomnikova. ZhTF, **94** (6), 944 (2024). (in Russian). <https://doi.org/10.61011/JTF.2024.06.58135.296-23>
- [16] V.N. Bessolov, M.E. Kompan, E.V. Konenkova, V.N. Panteleev, S.N. Rodin, M.P. Shcheglov. Pisma ZhTF, **45** (11), 3 (2019). (in Russian). <https://doi.org/10.21883/PJTF.2019.11.47813.17756>

*Translated by E.Ilinikaya*