Semiconductors, 2025, Vol. 59, No. 5

Dislocation structure of bulk AlN crystals under indentation

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Received May 5, 2025
Revised July 28, 2025
Accepted July 28, 2025

The results of cathodoluminescence (CL) studies of the defect structure created by indentation of the basic and prismatic surfaces of low-dislocation bulk crystals of aluminium nitride and gallium nitride are presented. It is found for the first time that the dislocation structure in the near-surface region at the indenter imprint in AlN is qualitatively different from that well known for other semiconductors with the wurtzite structure which is well explained by the Peierls model. It is concluded that this model is inapplicable for the characterisation of dislocations in AlN and that it is necessary to construct new theoretical approaches for this purpose.

Keywords: aluminium nitride, AlN, GaN, dislocations, cathodoluminescence.

DOI: 10.61011/SC.2025.05.61997.8095

Aluminum nitride (AlN) has recently attracted worldwide attention as an ultra-wide-gap semiconductor with excellent characteristics, which, combined with the availability of efficient techniques for growth of bulk AlN crystals, open wide prospects for application of AlN in the design of high-performance optoelectronic devices, high-power and high-frequency electronic devices, ultra-high-voltage power electronic devices, warning and guidance sensors, and deep UV disinfection systems [1,2].

The process of growth of crystals and heterostructures for electronic components is often accompanied by the introduction of dislocations, which may affect the functional electronic properties of the material. This necessitates a correct understanding of the key characteristics of dislocations, such as their types and dislocation slip systems; the structure of their cores; and mobility under the influence of various mechanical stresses, temperature, and injected carriers.

Among III-nitrides with a hexagonal wurtzite lattice that are related to AlN, gallium nitride is the one with the best studied electronic and mechanical properties of dislocations. Dozens of papers (see reviews on electronic properties [3] and microhardness [4]) focused on GaN have already been published. Dislocation slip systems in indented GaN samples were studied by cathodoluminescence (CL) and transmission electron microscopy (TEM), and they fit quite well into the concept of Peierls dislocation motion. As for AlN, such data are rather scarce and have not been interpreted unambiguously yet. The structure of cores of growth dislocations was investigated in just two TEM studies [5,6], and their authors did not reach any definite conclusion as to the prevalent core type (i.e., perfect or split into partial cores). The key information on slip systems was obtained via transmission electron microscopy in experiments on nanoindentation of AlN [7–9], where the range of the introduced dislocations did not exceed a few micrometers. With the exception of [9], these experiments were performed for thin films grown on a substrate with a high density of growth dislocations ($10^9-10^{10}\,\mathrm{cm}^{-2}$). It turned out that the dislocation structure detected in TEM studies of the cross section of nanoindented samples differed somewhat from that observed in GaN; however, as far as we know, no data on slip systems near the surface directly subjected to indentation have been obtained yet.

The present study is the first to report on the defect structure that arises during indentation under high loads of *a* and *m*-planes of bulk and low-dislocation AlN crystals and is recorded on the same planes using the cathodoluminescence method.

AlN crystals were grown using the sublimation sandwich method (SSM) by sublimation and recondensation from the vapor phase of high-purity AlN powder on a (0001) seed of single-crystal 6*H*-SiC 60 mm in diameter. The conditions and results of growth of bulk AlN crystals by SSM were presented in review [10]. The obtained AlN layer was as thick as 1.8 mm. According to TEM data, the dislocation density in the 1.8-mm-thick AlN crystal varied from $8\cdot10^7\,\mathrm{cm}^{-2}$ near the interface with the substrate to $10^5\,\mathrm{cm}^{-2}$ near the growth surface, which is 3–5 orders of magnitude lower than the values reported in earlier studies on AlN indentation. Samples with a flat surface of the growth basal and transverse prismatic $(10\bar{1}0)$ orientations were prepared for experiments and subjected to Vickers indentation under loads varying from 1 to 10 N on a Shimadzu HMV-2T microhardness tester. Mechanical grinding and chemical-mechanical polishing were performed for successive removal of layers in $1-3\,\mu$m steps from the indented basal surface. This made it possible to obtain a three-dimensional CL pattern of dislocation distribution. With such processing, the surface was mirror-smooth, and electron backscatter diffraction (EBSD) studies verified the lack of a near-surface damaged layer.

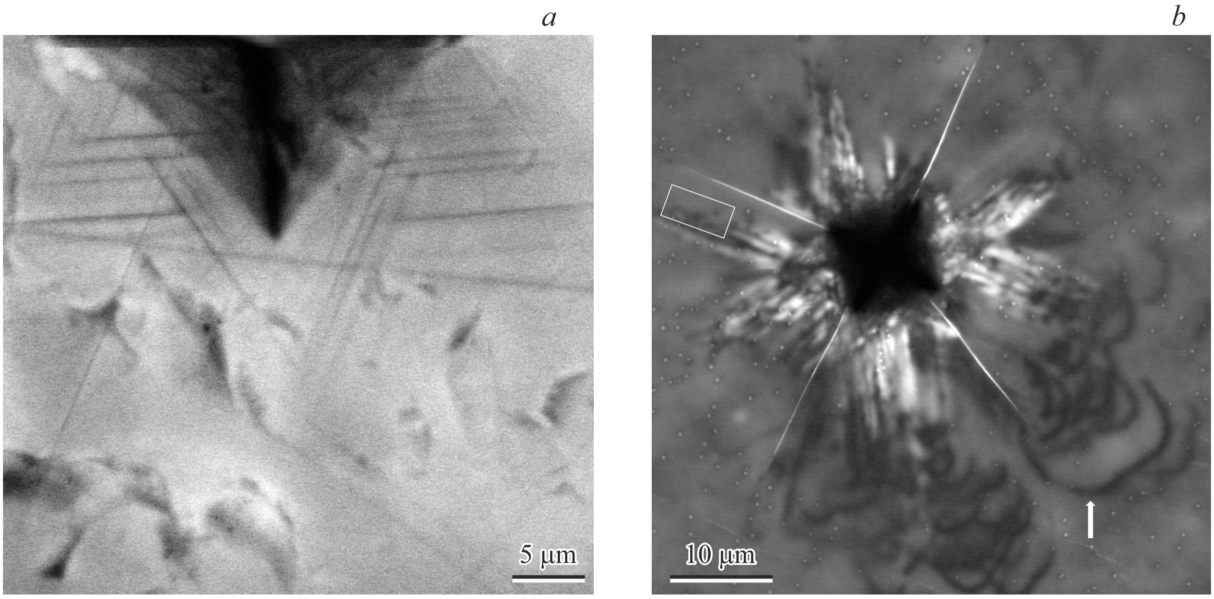

Figure 1. Panchromatic maps in the vicinity of the Vickers indentation point of the (0001) surfaces of AlN (*a*) and GaN (*b*) crystals with loads of 3 and 1 N, respectively. The accelerating voltage is 20 kV.

The dislocation structure was studied with scanning electron microscopes (SEM) provided by the Interdisciplinary Resource Center for Nanotechnology (St. Petersburg State University) by CL and EBSD. The latter method was used to determine the orientation of the detected CL contrast directions.

Figure 1 shows the panchromatic CL maps in the vicinity of the indentation point on the growth basal surface of aluminum and gallium nitride samples obtained under loads of 3 and 1 N, respectively. It can be seen from Figure 1, *a* that dislocations in AlN are visualized as dark strictly rectilinear contrasts, which form a pattern similar to a star of David around the indentation point.

The CL spectrum of AlN featured a wide band with a maximum of ~ 500 nm both in the undeformed part of the sample and near the indentation point. No additional luminescent spectral bands were detected near dislocations within the studied spectral range from 200 to 800 nm. Thus, the observation of dark contrasts from dislocations corresponds to a reduction in intensity of the luminescence band characteristic of the studied sample and to an enhancement of nonradiative recombination of excess carriers.

Analyzing the CL images in Figures 1, *a* and *b* together with the crystallographic orientation data, one may note the following differences in the dislocation structures of indented AlN and GaN.

Two slip systems are seen clearly in indented GaN. The first is represented by rectilinear contrasts spreading from the indentation point in six radial directions of the $\langle 1\bar{2}10\rangle$ type. Most of them have a bright contrast, which is attributable to intrinsic luminescence of screw dislocations (see [11] for an in-depth analysis, where it was also demonstrated that rectilinear luminescent contrasts often end in dark point contrasts; some of these are highlighted by a rectangle in Figure 1, *b*).

This combination of point and linear contrasts is a consequence of formation and slip of dislocation loops in prismatic slip planes of the $\{10\bar{1}0\}$ type, where dark point contrasts are emerging edge dislocations that are extended along hexagonal axis *C* of crystals with a wurtzite structure. This distribution of dislocations near the indentation point on the (0001) plane matches the one observed repeatedly in earlier studies [4,12] and may be explained by the model proposed there.

The second type of contrasts in GaN corresponds to irregular dislocations, which evidently form half-loops in the basal planes (one of them is marked with an arrow at the bottom of Figure 1, *b*). They consist of segments extended both along and across the radial direction of propagation of the loops. Certain longitudinal components originate from the indentation point and correspond primarily to screw dislocations. The half-loops more distant from the center in Figure 1, *b* are not connected by visible contrasts to the indenter, which is attributable either to a greater depth of positioning of longitudinal components or to the predominant emergence of their screw sections on the surface.

The line contrasts in AlN seen in Figure 1, *a* do not correspond to any type characteristic of GaN. Dark point contrasts were lacking at all applied indenter loads, and the CL map pattern with rare linear contrasts remained similar to that shown in Figure 1, *a* even after removal of successive surface layers down to the indentation depth. Moreover, according to EBSD data, the dark lines in Figure 1, *a* are elongated along $\langle 10\bar{1}0\rangle$, which may lie in either basal or prismatic planes $\{1\bar{2}10\}$. If they are *a* *b*

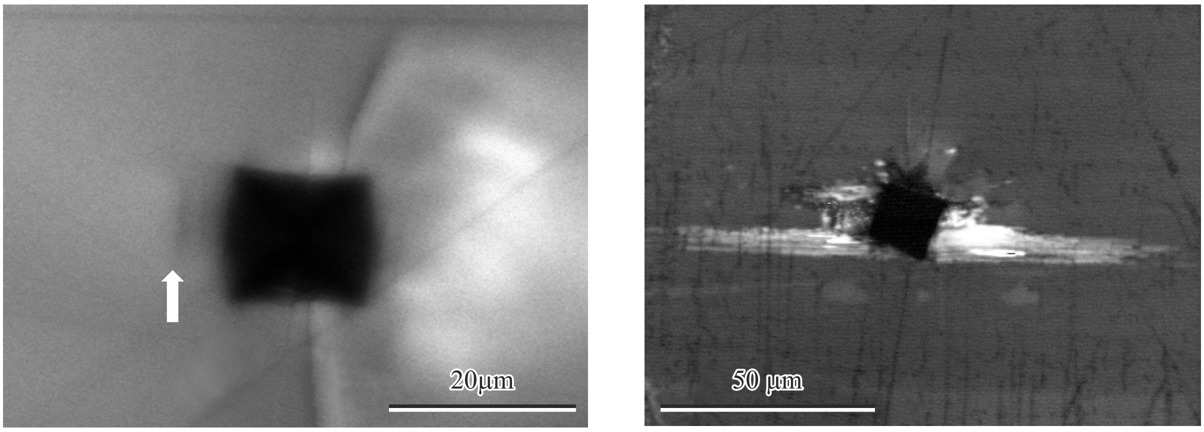

Figure 2. Panchromatic maps of the CL intensity distribution near the indentation point for the *m*-plane of AlN (*a*) and GaN (*b*). The vertical direction in the figures is [0001].

classified as dislocations in the basal plane with Burgers vector $b = 1/3$ $[1\bar{2}10]$, they could potentially be 30- or 90-degree ones [13], although, as far as we know, their observation has not been reported yet. At the same time, it turned out that the majority of such strictly linear contrasts are preserved and do not change their position after each of the seven successive steps of removal of a 1-μm-thick surface layer. This result demonstrates that these contrasts are related to two-dimensional defects in the $\{1\bar{2}10\}$ planes.

The conclusion regarding a significant qualitative difference in defect structure between GaN and AlN is also confirmed by the data on indentation of the prismatic *m*-surface $(10\bar{1}0)$ presented in Figs. 2, *a* and *b*. A two-ray rosette is seen in the GaN CL maps near the indentation point. Each ray consists of long and short components spreading from the edges of the indentation mark (i.e., in regions with the maximum lateral shear stresses). This rosette type was observed in many semiconductors with a wurtzite structure and reflects the difference in mobility of dislocations with different atomic cores [14].

It follows from the CL map in Figure 2, *a* that such a dislocation rosette does not form at all in AlN. Only the segments of dark linear contrasts elongated in the direction of hexagonal axis *C* are observed instead. Their length matches the size of the indentation mark. This fact indicates that dislocation slip in the basal plane is lacking and extended defects induced by the applied load propagate deeper into the sample, which agrees with the data obtained on the indented basal surface.

Thus, it was established experimentally that the defect structure arising in the process of indentation of AlN is qualitatively different from the one characterized by the minimum Burgers vector for this type of lattice, which was used to interpret the defect structure in nanoindented AlN in [8]. Although the detailed crystallography of defects underlying the observed contrasts remains unclear, it is fair to assume that they are stacking faults in prismatic planes $\{1\bar{2}10\}$, which, according to the analysis performed by Osipyan and Smirnova in [15], may be limited by mobile Shockley dislocations with Burgers vectors 1/2 $\langle 1\bar{1}01\rangle$ along axis *C* and 1/6 $\langle 02\bar{2}3\rangle$ along directions $\langle 10\bar{1}0\rangle$.

One possible explanation for such a structural feature of extended defects in AlN is that, at close values of the parameters included in the Peierls model (the shear modulus and lattice parameters; see [16]), the energy of interplane interaction [17] and the energy of formation of a stacking fault in the basal plane in AlN ($\sim 120\,\mathrm{meV/A^2}$) [18,19] are 4–5 times higher than in GaN, while the same energies for prismatic stacking faults are, according to calculated data [20], close in these compounds (79 and $72\,\mathrm{meV/A^2}$, respectively), which makes them energetically preferable for AlN.

The inapplicability of the Peierls model to AlN makes it necessary to develop a new theory for characterizing the dynamics of dislocations in AlN or, as was already noted by the authors of [9], perform complex molecular dynamics calculations.

Acknowledgments

Equipment provided by the Interdisciplinary Resource Center for Nanotechnology (St. Petersburg State University) was used in experiments.

Conflict of interest

The authors declare that they have no conflict of interest.

References

[1] R. Yu, G. Liu, G. Wang, C. Chen, M. Xu, H. Zhou, T. Wang, J. Yu, G. Zhao, L. Zhang. J. Mater. Chem. C, **9**, 1852 (2021).

[2] H. Amano, R. Colllazo, C. De Santi, S. Einfeldt, M. Funato, J. Glaab, S. Hagedorn, A. Hirano, H. Hirayama, R. Ishii, Y. Kashima, Y. Kawakami, R. Kirste, M. Kneissl, R. Marti et al. J. Phys. D: Appl. Phys., 53 (50), 503001 (2020).

[3] O. Vyvenko, A. Bondarenko. *Crystal Lattice Defects as Natural Light Emitting Nanostructures in Semiconductors*, in *Progress in Photon Science: Recent Advances*, ed. by K. Yamanouchi, S. Tunik, and V. Makarov (Springer International Publishing, Cham, 2019) p. 405.
[4] P. Kavouras, I. Ratschinski, G.P. Dimitrakopulos, H.S. Leipner, Ph. Komninou, G. Leibiger, F. Habel. Mater. Sci. Technol., **34**, 1531 (2018).
[5] P. Delavignette, H.B. Kirkpatrick, S. Amelinckx. J. Appl. Phys., 32, 1098 (1961).
[6] M. Azzaz, J.P. Michel, V. Feregotto, A. George. Mater. Sci. Eng. B, **71**, 30 (2000).
[7] I. Yonenaga, T. Shima, M.H.F. Sluiter. Jpn. J. Appl. Phys., **41**, 4620 (2002).
[8] Y. Tokumoto, K. Kutsukake, Y. Ohno, I. Yonenaga. J. Appl. Phys., **112**, 093526 (2012).
[9] J. Chen, K. Chen, X. Su, M. Niu, Q. Wang, K. Xu. Thin Sol. Films, **791**, 140240 (2024).
[10] E.N. Mokhov, A.A. Wol'fson, O.P. Kazarova. Phys. Solid State, **61**, 2286 (2019).
[11] O. Medvedev, O. Vyvenko, E. Ubyivovk, S. Shapenkov, A. Bondarenko, P. Saring, M. Seibt. J. Appl. Phys., **123**, 161427 (2018).
[12] I. Ratschinski, H.S. Leipner, F. Heyroth, W. Fränzel, R. Hammer, M. Jurisch. Phys. Status Solidi C, **8**, 1325 (2011).
[13] Yu.A. Osipiyan, I.S. Smirnova. Phys. Status Solidi B, **30**, 19 (1968).
[14] *Elektronnye svoistva dislokatsii v poluprovodnikakh*, Ed. by Yu.A. Osipyan (M., Editorial URSS, 2000). (in Russian).
[15] Yu.A. Osipyan, I.S. Smirnova. J. Phys. Chem. Solids, **32**, 1521 (1971).
[16] J. P. Hirth, J. Lothe. *Theory of Dislocations* (McGraw-Hill, 1968).
[17] A.F. Wright. J. Appl. Phys., **82**, 5259 (1997).
[18] K. Suzuki, M. Ichihara, S. Takeuchi. Jpn. J. Appl. Phys., **33**, 1114 (1994).
[19] C. Stampfl, C.G. Van de Walle. Phys. Rev. B, **57**, R15052 (1998).
[20] J.E. Northrup. Appl. Phys. Lett., **86** (2005).

Translated by D.Safin