Comparative Study of Optical and Electrical Properties of Grown-In and Freshly Introduced Dislocations in GaN by SEM Methods

P.S. VERGELES [iD],[1,3] E.B. YAKIMOV,[1] and V.I. ORLOV[1,2]

1.—Institute of Microelectronics Technology and High Purity Materials RAS, Chernogolovka, Russia. 2.—Institute of Solid State Physics RAS, Chernogolovka, Russia. 3.—e-mail: vergelesp@gmail.com

Electrical and optical properties of grown-in and freshly introduced dislocations in GaN have been studied by the electron beam induced current and cathodoluminescence methods. It is observed that the recombination properties of grown-in and freshly introduced basal plane and threading dislocations are comparable. That allows to assume the intrinsic nature of dislocation recombination activity in GaN. It is demonstrated that the recombination properties of basal plane dislocations weakly depend on their type. The behavior of dislocation-related luminescence at 3.1 eV is more complex. It can be observed not in all GaN crystals even when dislocations are introduced in the similar conditions. Besides, it is not observed on basal plane and threading grown-in dislocations. This luminescence is not produced by freshly introduced basal plane dislocations. These observations can be explained assuming that the dislocation-related luminescence is associated with point defects generated by dislocations gliding in pyramidal or prismatic slip planes.

Key words: GaN, Indentation, dislocation, cathodoluminescence, point defects

INTRODUCTION

Dislocation densities in GaN and GaN based structures usually exceed 10^6 cm^{-2}.[1–5] Moreover, at least some dislocations in GaN are mobile at room temperature under shear stress.[6–15] The excess carrier injection was also shown to stimulate their glide (so called recombination enhanced dislocation glide (REDG)[15–18]). Thus, dislocations can be unintentionally introduced under technology processes even if the dislocation-free crystals will be grown, therefore, a study of dislocation electrical and optical properties is important from both fundamental and practical points of view. It is well established that dislocations in GaN increase the local nonradiative recombination rate[1,19–21] although, as shown in[21–24] they are not the main lifetime killers in the state-of-the-art GaN. Numerous investigations by different methods indicated that the total line charge per unit distance along dislocations can reach 4×10^7 e/cm.[25–28] The dislocation-related luminescence was found on freshly introduced dislocations.[13,15,29–31] However, the nature of dislocation electrical activity is not totally clear up to now. Particularly, it is not clear if it is intrinsic or determined by a capture of point defects. Theoretical investigations showed[32–35] that threading and basal dislocations can introduce gap states, i.e., the activity at least of some types of dislocations can be intrinsic. However, while the recombination activity of edge threading dislocations was assumed in[34,36] to be low, the electron beam induced current (EBIC) investigations showed[37] that the density of revealed dislocations was close to the threading dislocation density estimated from x-ray diffractometry and atomic force microscopy. As shown in[32,38] the nonradiative recombination on threading dislocations can be determined by the piezoelectric field.

Point defect atmospheres, in particular oxygen segregation on dislocations and decoration of dislocations by vacancy-type defects, were also suggested as a reason for electrical activity of dislocations.[28,39,40] For dislocations gliding at room temperature formation of point defect atmospheres by point defects trapped by the strain field around dislocations can be excluded. Therefore, a comparison of electrical and optical properties of such dislocations with those of grown-in dislocations can shed some light on the nature of dislocation electrical and optical activity.

EXPERIMENTAL

As shown in,[21] the dislocation contrast in the EBIC method depends on impurity content. Therefore, a comparison of grown-in and "fresh" dislocations was carried out on the same crystals. Most of the studies were carried out on two structures with < 0001 > growth direction. The first ones is a free-standing n-GaN grown by hydride vapor phase epitaxy (HVPE) with a donor concentration of 5×10^{16} cm^{-3}, excess carrier diffusion length of 450 nm and dislocation density about 10^6 cm^{-2}. The second one is a 6 μm thick n-GaN film grown by epitaxial lateral overgrowth (ELOG) with the dislocation density reduced down to 10^6 cm^{-2} over the SiO$_2$ stripes (wing regions, 12 μm in width) and of 10^8 cm^{-2} for the material directly grown in the GaN windows of the SiO$_2$ mask (slit region, 4 μm in width). The diffusion length of 240 nm and the donor concentration of 5×10^{16} cm^{-3} and 1.5×10^{17} cm^{-3} for wing and slit regions, respectively, were obtained in this ELOG film. These values well correlate with results of,[41–43] where it has been shown that the donor concentrations in the wing and slit regions differ by two–three times. The stripes are aligned along the [1–100] direction, and as shown in,[44] dislocations in the ELOG wing regions have the form of half-loops with the bottom located in the basal plane. Besides, some structures with different densities of grown-in dislocations from different suppliers were used to clarify the conditions for the appearance of dislocation-related luminescence because in the structure studied in [14] such luminescence was not revealed.

The characterization of defect structure was carried out by EBIC and cathodoluminescence (CL) methods at room temperature. EBIC measurements were carried out in the scanning electron microscope JSM-840A and the CL measurements in JSM 6490 with the MonoCL-3 system. In most cases the beam current of about 0.1 nA was used. Beam energy of 10 keV and 35 keV was used for the CL and EBIC measurements, respectively, to achieve the appropriate lateral resolution. To stimulate REDG low energy, electron beam irradiation (LEEBI) was carried out in the same microscopes at room temperature at beam energies 10 keV or 35 keV and beam current 1 nA. Fresh dislocations were introduced at room temperature by indentation of {0001} plane with a Vickers type indenter. The load was varied in the range from 0.2 N to 0.8 N. The dislocation velocity V_d in REDG experiments was estimated to be of the order of 10 nm/s.[18]. However, it should be taken into account that this value was estimated for a beam current smaller than 1 nA. Besides, irradiation time was assumed to be equal to the frame scan duration. Real duration of beam interaction with a dislocation can be shorter by one-two orders of magnitude, i.e,. the dislocation velocity also can be by one-two orders of magnitude larger. As shown in Ref. 15 the velocity of dislocations in pyramidal planes under LEEBI is a few times higher. The velocity of dislocation moving from indentation can be about 1000 nm/s.[14] Thus, the dislocation velocity can be estimated in both cases as 100–1000 nm/s. Taking into account that an impurity can follow dislocation if its diffusivity $D > (V_d b^2)/a$, where b is the dislocation Burgers vector and a is some parameter (usually $a < 5$ nm),[45] D for this impurity should be larger than 10^{-14}–10^{-13} cm^2/s at room temperature and the migration enthalpy smaller than 0.6–0.77 eV. It should be also taken into account that point defects in dislocation atmospheres most probably formed complexes, the diffusivity of which should be rather low. Therefore, it is reasonable to assume that dislocations gliding at room temperature are indeed much cleaner than grown-in ones.

RESULTS AND DISCUSSION

Typical EBIC images of ELOG GaN before and after LEEBI are shown in Fig. 1. One can see that the EBIC contrast of dislocation segments, which
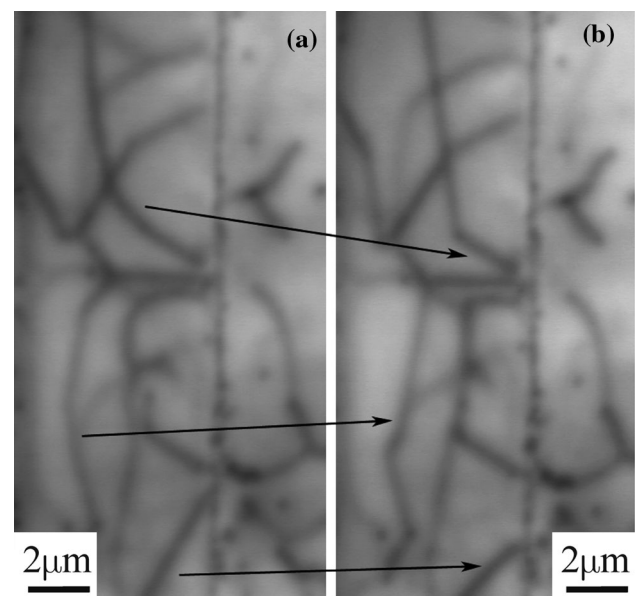

Fig. 1. EBIC images of basal dislocations in ELOG GaN before (a) and after e-beam irradiation at 35 keV up to a dose of 2×10^{-2} C/cm^2 (b). Shifted dislocation segments are shown with arrows.

are shifted in the basal plane due to REDG, does not change and is similar to that of neighboring immobile segments. As dislocations glide in the basal plane their depth does not change and from a similarity of EBIC contrasts it follows that the recombination properties of grown-in basal plane dislocations and those of dislocations gliding at room temperature are approximately the same. It should be also noted that the dislocation segments shifted under LEEBI change their orientation. Therefore, it can be assumed that the contrast dependence on the dislocation type is not so strong. In the case of threading dislocations the EBIC contrast of grown-in dislocations can be compared with that of fresh dislocations introduced under applied shear stress and dislocations gliding at room temperature due to REDG effect. The first case is illustrated in Fig. 2. It should be taken into account that a length of dislocation segments intersecting the surface can differ from that of threading dislocations due to a different depth of dislocation half-loops that could determine the difference in the EBIC contrast. Nevertheless, it can be stated that the EBIC contrast of grown-in threading dislocations is comparable with that of some "clean" dislocations. EBIC images of dislocation rosettes before and after LEEBI are shown in Fig. 3. It is clear seen that the EBIC contrast of dislocation generated by the LEEBI near indentation is close to that of grown-in threading dislocations. These results allow to conclude that the essential part of dislocation recombination activity is intrinsic in nature. If so, the recombination activity of dislocation should depend on its type. The methods used in this work do not allow to determine the dislocation
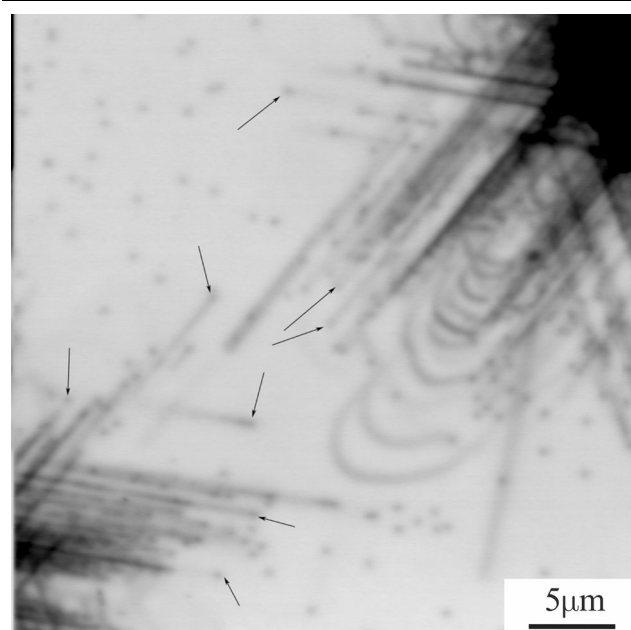

Fig. 2. EBIC image of imprint in HVPE GaN at 10 keV. Dislocation segments intersecting the surface are shown with arrows.

type and to conclude if they are dissociated or not. Nevertheless, for basal plane dislocations, some conclusions can be made. An indentation of GaN at room temperature introduces a lot of dislocation loops in the basal plane (see Fig. 2). In most cases their segments do not coincide with the crystallographic directions. However, a few loops have a pronounced hexagonal form (see, e.g., Figure 4). Perfect basal dislocations should be of screw or 60°-mixed types, therefore for this particular loop at least one segment should be of a screw type and others are 60° dislocations. The contrast is more or less homogeneous along the loop, which means that freshly introduced screw and 60° basal dislocations increase the nonradiative recombination rate and have close recombination strength values.

Thus, from the one side, as shown in Ref. 21, the recombination strength of threading dislocations pronouncedly depends on the impurity content. That seems to support the extrinsic nature of dislocation electrical properties. However, from the other side, a movement of dislocations at room temperature, which can be assumed to release them from contaminations, does not noticeably change their recombination activity. This contradiction may be explained by the assumption made in Ref. 32 that the giant local strain field around a threading dislocation in GaN leads to an appearance of the deep electronic states. The observed, in Ref. 21, decrease in the dislocation recombination strength with the dopant concentration increase could be explained by screening of the piezoelectric field.

Optical properties of dislocations behave quite differently. First of all, it should be noted that the dislocation-related luminescence at about 3.1 eV was observed on freshly introduced dislocations only[13,15,29–31] and was never observed on grown-in dislocations. Moreover, the investigation of a set of GaN layers from different suppliers has shown that it is observed not in all epilayers. The reason is unclear up to now but it should be noted that in the present work this luminescence is not observed on three epilayers, grown by metalorganic chemical vapour deposition (MOCVD) or molecular beam epitaxy (MBE) methods, and the only common feature of these structures is rather high dislocation density exceeding 10^9 cm^{-2}. At the same time the dislocation-related luminescence was observed on the structures grown by the MOCVD, HVPE and ELOG methods with the lower dislocation densities. Besides, as a comparison of EBIC and CL images of the same dislocation rosette has shown, dislocation-related luminescence was not observed on the basal plane dislocation loops (Fig. 5). As shown in Ref. 15, LEEBI leads to a disappearance of some luminescence lines, while new ones are generated. This is illustrated in Fig. 6. In both cases, after deformation and after LEEBI luminescent features have a form of lines elongated along the $< 11 \bar{2} 0 >$ directions. That means that they are associated with
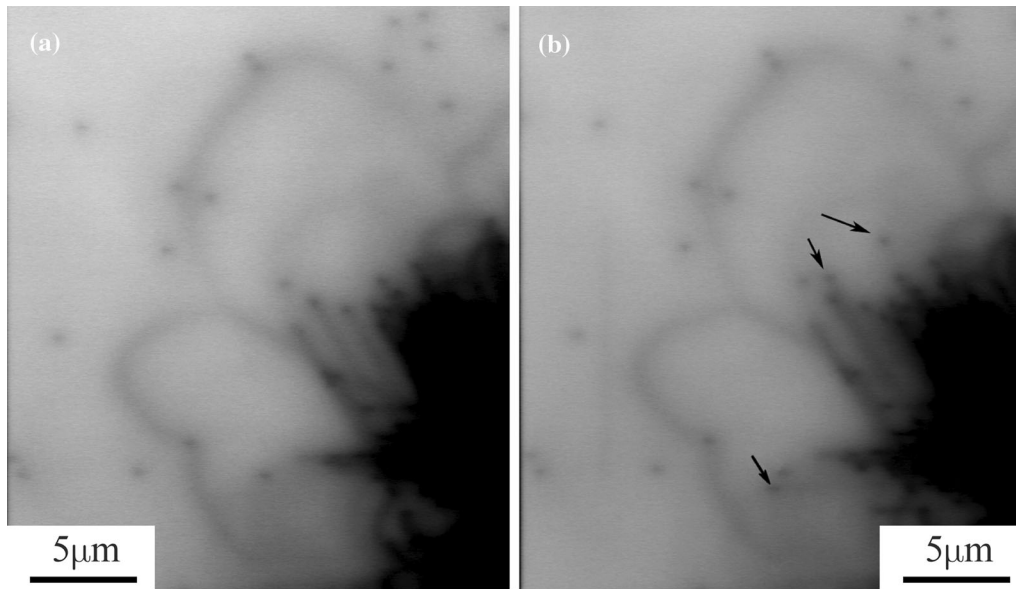
Fig. 3. EBIC images of imprint in HVPE GaN at 35 keV before (a) and after LEEBI with a dose of 1.5×10^{-2} C/cm^2 (b). Dislocations created by LEEBI are shown with arrows.
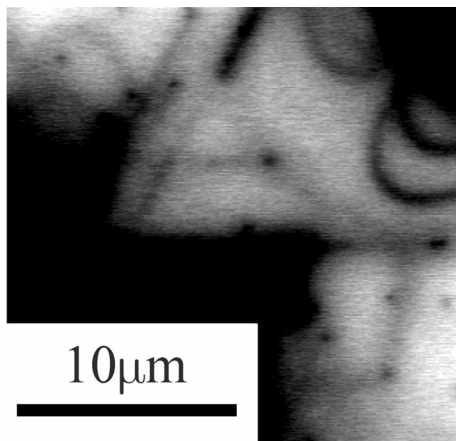

Fig. 4. EBIC image of dislocation rosette fragment in HVPE GaN at 35 keV.

dislocation half-loops gliding in the planes intersecting the surface (prismatic or pyramidal ones). The dislocation-related luminescence was shown to be unstable under LEEBI[15] and probably under high-temperature annealing.[30] That allows to assume that it is associated not with dislocations but with point defects generated by gliding dislocations. Another possible explanation of low dislocation-related luminescence stability under LEEBI can be a transformation of dissociated dislocation into perfect ones under electron beam irradiation. Such transformation was observed in Ref. 35 on basal plane screw dislocations. In our experiments the dislocation-related luminescence is most probably produced by the dislocations gliding in prismatic or pyramidal planes, nevertheless, such a mechanism allows to explain the results obtained.

Thus, it is shown that the recombination properties of freshly introduced and grown-in dislocations
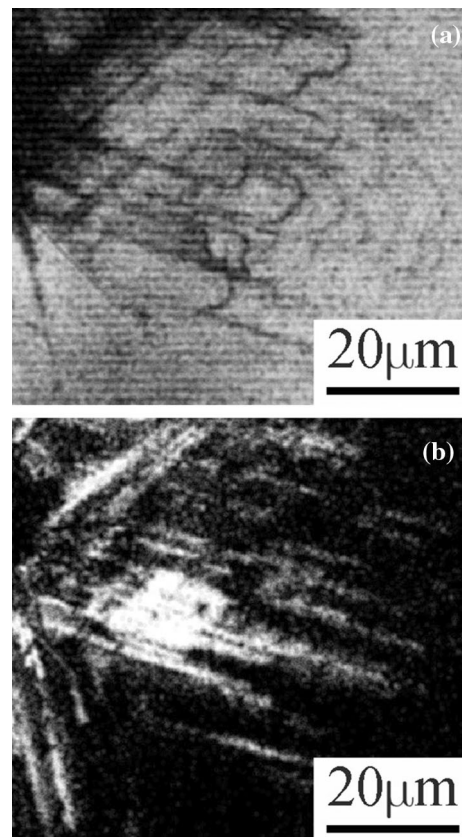

Fig. 5. EBIC (a) and 3.1 eV monochromatic CL (b) images of imprint fragment in HVPE GaN.

are very similar. That allows to assume the intrinsic nature of their activity. However, it is not the case for the dislocation-related luminescence. It is not observed in some GaN epilayers. Besides, it has been never observed on grown-in dislocations and
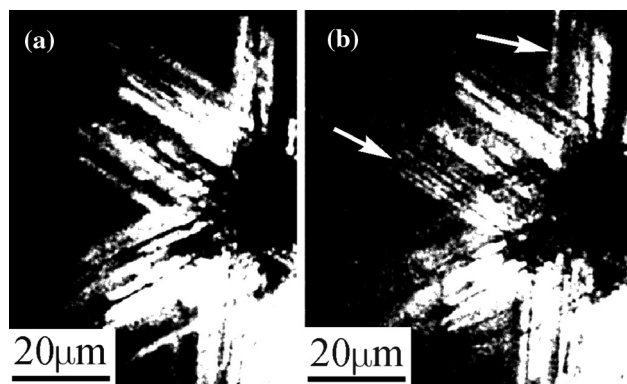
Comparative Study of Optical and Electrical Properties of Grown-In and Freshly Introduced Dislocations in GaN by SEM Methods

5177

Fig. 6. Monochromatic CL images at 3.1 eV after deformation (a) and after LEEBI with a dose of 4 mC/cm^2 (b). Dislocations generated under LEEBI were indicated with arrows.

basal plane dislocations. Thus, it can be concluded that only one or very few types of dislocations can produce such luminescence. It can be also assumed that this luminescence is extrinsic in nature.

ACKNOWLEDGMENTS

REFERENCES

1. S.C. Jain, M. Willander, J. Narayan, R. Van Overstraeten, J. Appl. Phys. **87**, 965 (2000).
2. N. Lu, I. Ferguson, Semicond. Sci. Technol. **28**, 074023 (2013).
3. S.J. Pearton, F. Ren, E. Patrick, M.E. Law, A.Y. Polyakov, ECS J. Solid State Sci. Technol. **5**, Q35 (2016).
4. H. Amano, Y. Baines, E. Beam, M. Borga, T. Bouchet, P.R. Chalker, M. Charles, K.J. Chen, N. Chowdhury, R. Chu, C. De Santi, M.M. De Souza, S. Decoutere, L. Di Cioccio, B. Eckardt, T. Egawa, P. Fay, J.J. Freedsman, L. Guido, O. Haberlen, G. Haynes, T. Heckel, D. Hemakumara, P. Houston, J. Hu, M. Hua, Q. Huang, A. Huang, S. Jiang, H. Kawai, D. Kinzer, M. Kuball, A. Kumar, K.B. Lee, X. Li, D. Marcon, M. Marz, R. McCarthy, G. Meneghesso, M. Meneghini, E. Morvan, A. Nakajima, E.M.S. Narayanan, S. Oliver, T. Palacios, D. Piedra, M. Plissonnier, R. Reddy, M. Sun, I. Thayne, A. Torres, N. Trivellin, V. Unni, M.J. Uren, M. Van Hove, D.J. Wallis, J. Wang, J. Xie, S. Yagi, S. Yang, C. Youtsey, R. Yu, E. Zanoni, S. Zeltner, Y. Zhang, J. Phys. D: Appl. Phys. **51**, 163001 (2018).
5. C. Chu, K. Tian, Y. Zhang, W. Bi, Z.-H. Zhang, Phys. Status Solidi A **216**, 1800815 (2019).
6. S.O. Kucheyev, J.E. Bradby, J.S. Williams, C. Jagadish, M. Toth, M.R. Phillips, M.V. Swain, Appl. Phys. Lett. **77**, 3373 (2000).
7. J.L. Weyher, M. Albrecht, T. Wosinski, G. Nowak, H.P. Strunk, S. Porowski, Mater. Sci. Eng. B **80**, 318 (2001).
8. S.-R. Jian, Appl. Surf. Sci. **254**, 6749 (2008).
9. I. Ratschinski, H.S. Leipner, F. Heyroth, W. Fränzel, R. Hammer, M. Jurisch, Philos. Mag. Lett. **90**, 565 (2010).
10. J. Huang, K. Xu, X.J. Gong, J.F. Wang, Y.M. Fan, J.Q. Liu, X.H. Zeng, G.Q. Ren, T.F. Zhou, H. Yang, Appl. Phys. Lett. **98**, 221906 (2011).
11. M. Fujikane, T. Yokogawa, S. Nagao, R. Nowak, Jpn. J. Appl. Phys. **52**, 08JJ01 (2013).
12. P.G. Caldas, E.M. Silva, R. Prioli, J.Y. Huang, R. Juday, A.M. Fischer, F.A. Ponce, J. Appl. Phys. **121**, 125105 (2017).
13. O. Medvedev, O. Vyvenko, E. Ubyivovk, S. Shapenkov, A. Bondarenko, P. Saring, M. Seibt, J. Appl. Phys. **123**, 161427 (2018).
14. V.I. Orlov, P.S. Vergeles, E.B. Yakimov, X. Li, J. Yang, G. Lv, S. Dong, Phys. Status Solidi A **216**, 1900163 (2019).
15. P.S. Vergeles, V.I. Orlov, A.Y. Polyakov, E.B. Yakimov, T. Kim, I.-H. Lee, J. Alloys Compd. **776**, 181 (2019).
16. K. Maeda, K. Suzuki, M. Ichihara, S. Nishiguchi, K. Ono, Y. Mera, S. Takeuchi, Phys. B **273–274**, 134 (1999).
17. E.B. Yakimov, P.S. Vergeles, A.Y. Polyakov, I.-H. Lee, S.J. Pearton, Appl. Phys. Lett. **106**, 132101 (2015).
18. E.B. Yakimov, P.S. Vergeles, A.Y. Polyakov, I.-H. Lee, S.J. Pearton, Jpn. J. Appl. Phys. **55**, 05FM03 (2016).
19. S.J. Rosner, E.C. Carr, M.J. Ludowise, G. Girolami, H.I. Erikson, Appl. Phys. Lett. **70**, 420 (1997).
20. E.B. Yakimov, J. Phys.: Condens. Matter. **14**, 13069 (2002).
21. E.B. Yakimov, A.Y. Polyakov, I.-H. Lee, S.J. Pearton, J. Appl. Phys. **123**, 161543 (2018).
22. E.B. Yakimov, Jpn. J. Appl. Phys. **55**, 05FH04 (2016).
23. I.-H. Lee, A.Y. Polyakov, N.B. Smirnov, E.B. Yakimov, S.A. Tarelkin, A.V. Turutin, I.V. Shemerov, S.J. Pearton, Appl. Phys. Express **9**, 061002 (2016).
24. I.-H. Lee, A.Y. Polyakov, N.B. Smirnov, E.B. Yakimov, S.A. Tarelkin, A.V. Turutin, I.V. Shemerov, S.J. Pearton, J. Appl. Phys. **119**, 205109 (2016).
25. C. Jiao, D. Cherns, J. Electron Microsc. **51**, 105 (2002).
26. E. Müller, D. Gerthsen, P. Bruckner, F. Scholz, Th Gruber, A. Waag, Phys. Rev. B **73**, 245316 (2006).
27. I. Arslan, A. Bleloch, E.A. Stach, S. Ogut, N.D. Browning, Phil. Mag. **86**, 4727 (2006).
28. A. Krtschil, A. Dadgar, A. Krost, J. Crystal Growth **248**, 542 (2003).
29. M. Albrecht, L. Lymperakis, J. Neugebauer, Phys. Rev. B **90**, 241201(R) (2014).
30. J. Huang, K. Xu, Y.M. Fan, J.F. Wang, J.C. Zhang, G.Q. Ren, Nanoscale Res. Lett. **9**, 649 (2014).
31. O. Medvedev, O. Vyvenko, A. Bondarenko, Phys. Status Solidi C **14**, 1700111 (2017).
32. L. Lymperakis, J. Neugebauer, M. Albrecht, T. Remmele, H.P. Strunk, Phys. Rev. Lett. **93**, 196401 (2004).
33. I. Belabbas, J. Chen, P. Komninou, G. Nouet, Comput. Mater. Sci. **79**, 118 (2013).
34. I. Belabbas, J. Chen, G. Nouet, Phys. Status Solidi C **12**, 1123 (2015).
35. I. Belabbas, I.G. Vasileiadis, J. Moneta, J. Smalc-Koziorowska, G.P. Dimitrakopulos, J. Appl. Phys. **126**, 165702 (2019).
36. T. Hino, S. Tomiya, T. Miyajima, K. Yanashima, S. Hashimoto, M. Ikeda, Appl. Phys. Lett. **76**, 3421 (2000).
37. N.M. Shmidt, O.A. Soltanovich, A.S. Usikov, E.B. Yakimov, E.E. Zavarin, J. Phys.: Condens. Matter. **14**, 13285 (2002).
38. V.M. Kaganer, K.K. Sabelfeld, O. Brandt, Appl. Phys. Lett. **112**, 122101 (2018).
39. I. Arslan, N.D. Browning, Phys. Rev. Lett. **91**, 165501 (2003).
40. M.E. Hawkridge, D. Cherns, Appl. Phys. Lett. **87**, 221903 (2005).
41. E.B. Yakimov, P.S. Vergeles, A.Y. Polyakov, N.B. Smirnov, A.V. Govorkov, I.-H. Lee, C.R. Lee, S.J. Pearton, Appl. Phys. Lett. **90**, 152114 (2007).
42. A.Y. Polyakov, N.B. Smirnov, A.V. Govorkov, A.V. Markov, E.B. Yakimov, P.S. Vergeles, N.G. Kolin, D.I. Merkurisov, V.M. Boiko, I.-H. Lee, C.R. Lee, S.J. Pearton, J. Electron. Mater. **36**, 1320 (2007).
43. E.B. Yakimov, P.S. Vergeles, A.Y. Polyakov, N.B. Smirnov, A.V. Govorkov, I.-H. Lee, C.R. Lee, S.J. Pearton, Appl. Phys. Lett. **92**, 042118 (2008).
44. Z. Liliental-Weber, D. Cherns, J. Appl. Phys. **89**, 7833 (2001).
45. H. Yoshinaga, S. Morozumi, Philos. Mag. **23**, 1351 (1971).