Wafer-scale analysis of GaN substrate wafer by imaging cathodoluminescence

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Received March 1, 2019; accepted March 7, 2019; published online April 9, 2019

With the progress in large-size high-quality GaN substrates, wafer-scale analysis is needed to evaluate homogeneity and defect distribution. We demonstrate the mapping of a 2 inch GaN substrate wafer based on the imaging cathodoluminescence technique. Macro pit defects with sizes varying from microns to millimeters are visualized and classified into three types according to their optical and structural properties. The formation mechanisms of the different types of pit defects are discussed with consideration of the facet growth involved in substrate growth.

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GaN-based materials and technologies are attracting great interest for their potential in power electronic applications.1) Lateral-structured devices such as AlGaN/GaN high-electron-mobility transistors have been practically applied to high-frequency power devices.2,3) With the progress in free-standing GaN substrates of low dislocation density and the control of impurities, vertical-structured devices with high breakdown voltage are expected.4,5) For power devices, a large area is needed to carry high current. In contrast to the rapid growth of GaN-based devices, large-size high-quality GaN substrates are still under development. Recent progress in the main technologies for bulk GaN growth has been reviewed in Ref. 6. Hydride vapor-phase epitaxy (HVPE) is expected for the fabrication of large-size GaN substrates with the advantages of high growth rates and high purity. To improve the quality of HVPE GaN substrates, special technologies have been developed such as epitaxial lateral overgrowth7) and dislocation elimination by epitaxial growth with inverse-pyramidal pits.8) These methods can help to eliminate dislocations within a large area. To realize uniform distribution and low dislocation density, new methods are employed such as HVPE based on a nano-void-assisted separation method9) and growth on a unique GaN nanowire template without dislocation or strain.6,10)

With the rapid progress in GaN growth, large-size GaN substrates with low dislocation density become possible. However, there is a need for an efficient and non-destructive characterization method which can quickly evaluate wafer homogeneity and defect distribution. Cathodoluminescence (CL) is light emission based on electron beam injection. By utilizing this phenomenon, one is able to characterize not only the nature of defects or impurities but also their distributions in the material.11) CL is extensively used for defect characterization in GaN materials, including dislocations,12) stacking faults,13) and pit defects.14,15) The CL system is usually installed in a scanning electron microscope (SEM). Benefiting from the high resolution of the electron microscope, it is suitable for the investigation of nano- and micro-structures.

Conventional CL is limited to small observation regions <200 x 200 μm². Such small observation areas are not practical for large-size wafer analysis. Recently, a new CL system termed Imaging CL capable of wafer analysis has been developed by Horiba, Ltd.16) The imaging CL system has improved light collection and transfer by means of the optimization of the ellipsoidal mirror and photomultiplier tube directly attached to the SEM. At present, the maximum observation area is 2000 x 2000 μm², which is 100 times that of conventional CL. The measurement time, depending on the display resolution, is more than 100 times as fast as that of conventional CL. It takes about 8 s to obtain an image with a display resolution of four million pixels. The mapping of a 2 inch GaN substrate wafer takes about 4–5 h. And for quick mapping with reduced display resolution, the measuring time can be shortened to 2 h. In this study, we demonstrate the application of the imaging CL technique to large-size GaN wafers. The distribution of macro defects, their optical properties and their formation mechanism are investigated.

The testing wafer is a commercially available 2 inch free-standing (0001) GaN substrate grown by the HVPE method. Dislocation is well controlled with the density less than 5 x 10⁶ cm⁻². However, there exist macro defects involved in the substrate growth. First, whole-wafer mapping is performed by imaging CL with the HORIBA imaging CL system WD-201N. Panchromatic CL images are recorded at 7 kV with beam current of 20–30 pA at room temperature. After figuring out the position of macro defects, detailed spectrum analysis is performed by a HORIBA MP32 CL system with a monochromator attached to a Hitachi SU6600 field-emission SEM at 5 kV with a beam current of 2 nA at room temperature.

Figure 1(a) shows the imaging CL mapping of a 2 inch GaN substrate. This wafer mapping is obtained by recording and tiling thousands of images automatically during measurement. In this wafer mapping, several bright spots with sizes varying from microns to millimeters are found randomly distributed in the wafer. These bright spots show stronger luminescence compared with the background. They are associated with pit defects formed during substrate growth. We classify these pit defects into three groups—A-, B- and C-type—depending on their shape and optical properties. In addition to the bright spots, in the upper part of the wafer, there is a flower-like pattern with a size of over 2 cm. The
zoomed-in CL images shown in Figs. 1(b) and 1(c) suggest that there exist triangular flakes (sizes of 100–200 microns) in the flower-like pattern’s center, while on the periphery triangular flakes extend into ribbons with lengths of over several millimeters. The contrast of these flakes and ribbons is slightly dark compared to the background. The origin of the flake and ribbon structures is not clear at present. It is speculated that these structures may have been introduced due to orientation deviation in the c-plane.

Figure 2 shows the zoomed-in CL images of the bright spots listed in Fig. 1(a). The size of these pits varies from tens of microns to 1 mm. The A-type pits are hexagonal or circular with ring-shaped doping stripes. The six edges are close to the $\langle 1\overline{1}00 \rangle$ directions. The images show dark CL contrast in the pit center, which becomes bright away from the core. There exists a dark hexagonal fan-blade-like pattern along the $\langle 11\overline{2}0 \rangle$ directions. The B- and C-type pits appear as hexagons with the six edges close to the $\langle 1\overline{1}20 \rangle$ directions. This 30° rotation suggests that the sidewall plane of the B- and C-type pits is different from that of the A-type. We can further classify the B- and C-types according to the appearance of the pit center. The B-type pits tend to contain a dark hollow core or dislocation cluster, while the C-type pits show the inner core of the A-type and a sidewall similar to that of the B-type.

Figure 3 shows the secondary electron (SE) and CL images of the three typical pits. CL spectra are taken from different sites inside pits or the background. In the SE images, the A-type pit shows a flat surface morphology suggesting that the pit is filled by overgrowth, while hexagonal sidewall morphologies remain in the B- and C-type pits. The B-type pit contains a hollow core, and the C-type pit has a flat bottom surrounded by a hexagonal sidewall. In the CL images, all the pits become visible with enhanced emission compared with the background. The background region shows weak near-band emission around 3.402–3.405 eV. For the A-type pit, the emission peak is not constant along the radial direction. It is about 3.391 eV in the dark center and is shifted to 3.379 eV at the outer bright ring. On the other hand, the emission energy from the sidewall of the B-type pit is constant at 3.376 eV, although the intensity decreases along the radial direction. The emission at 3.376 eV is also found from the sidewall of the C-type pit.

The shift of CL emission between background and pits is probably due to oxygen impurity incorporation during facet growth. The background is of (0001) facet growth, while the pit defects are formed by other facet growth (e.g. $\{10\overline{1}1\}$ and $\{11\overline{2}2\}$). Previous studies have reported that oxygen impurities incorporate into nitrogen sites and form shallow donor levels.8,9 There is an orientation dependence of oxygen incorporation in GaN, whereby nitrogen-rich $\{10\overline{1}1\}$ and $\{11\overline{2}2\}$ planes tend to incorporate more...
oxygen impurities. Reference 19 has reported that pit defects surrounded by \{10–11\} facets show stronger CL emission due to oxygen incorporation, and the emission energy from \{10–11\} facets is about 300 meV red-shifted. In this study, the sidewalls of B- and C-type pits also show red shift of about 0.26–0.29 eV; it is suggested that they are mainly formed by \{10–11\} facet growth. The red shift from A-type pits is smaller and varied along the radial direction, indicating that there are other facet growths involved in the formation of A-type pits.

Figure 4 shows the schematics of pit formation, including the top view [Figs. 4(a)–4(c)] and vertical cross-section [Figs. 4(d)–4(f)]. The B-type pit originates from imperfection regions and grows with \{10–11\} facet growth. \{10–11\} is a facet with a slow growth rate compared with others and with the presence of a hollow core, it is rather difficult to fill B-type pits by overgrowth. On the other hand, the A-type pit is structurally 30° rotated with respect to the B- and C-type pits. It is considered that the A-type pit is formed by \{11–2m\} facets with \(m\) representing multiple facets such as \{11–22\} or \{11–23\}. Compared with the slow facet \{10–11\}, \{11–2m\} are planes with fast growth rates. A previous study suggested that an A-type pit overgrows spontaneously if a fast-growing facet nucleates on its bottom.20 This may explain why the A-type pit is embedded by overgrowth. There is also the possibility of changing facet growth from the slow facet \{10–11\} to the fast facet \{11–22\}.15 The appearance of stripes and a hexagonal fan blade pattern in A-type pits may reflect the local variation of carrier concentration, which is probably introduced during fast facet growth. Finally, the C-type pit is a mixed type with two-stage growth including the A- and B-type. The first stage is the formation of an A-type pit with \{11–2m\} facet growth. If the filling of the A-type pit is not perfect, it may cause the second growth of a B-type pit.

For HVPE GaN substrate growth, the phenomena of facet growth and pit formation sometimes can be utilized to
suppress dislocation propagation and thus reduce defect density. Hence, imaging CL can assist in evaluating the overall effect of facet growth and defect control on the wafer scale. For power device applications, the existence of pit defects in GaN substrates may cause a detrimental effect of device failure, and so it is necessary to reduce the number of macro pits. In this regard, wafer screen mapping by imaging CL can give useful information on the density and distribution of macro pit defects.

In summary, we have demonstrated the application of imaging CL for the wafer-scale analysis of 2 inch freestanding GaN substrates. A variety of macro defects including flakes/ribbons and pits are visualized by panchromatic CL mapping. The pit defects are classified into three types originating from different facet growths. Imaging CL provides a quick way to evaluate the quality of GaN substrate wafers. The combination of imaging CL and spectral analysis would be a promising approach for defect characterization in large-size nitride wafers.

Acknowledgments This work was supported by MEXT’s “Program for research and development of next-generation semiconductor to realize energy-saving society” and JSPS KAKENHI Grant No. 18K04248. We are grateful to H. Akiyama, S. Horikawa, and K. Nakagawa of HORIBA, Ltd., for their great technical support.

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1) S. Fujita, “Wide-bandgap semiconductor materials: for their full bloom,” Jpn. J. Appl. Phys. 54, 030101 (2015).
2) U. K. Mishra, P. Parikh, and Y.-F. Wu, “AlGaN/GaN HEMTs–an overview of device operation and applications,” Proc. IEEE 90, 1022 (2002).
3) E. A. Jones, F. Wang, and D. Constinett, “Review of commercial GaN power devices and GaN-based converter design challenges,” IEEE J. Emerg. Sel. Top. Power Electron. 4, 707 (2016).
4) I. C. Kreibulg, A. P. Edwards, O. Aktaa, T. Prunty, and D. Bour, “Vertical power p-n diodes based on bulk GaN,” IEEE Trans. Electron Devices 62, 414 (2015).
5) H. Ohita, N. Kaneda, F. Horikii, Y. Narita, T. Yashida, T. Mishima, and T. Nakamura, “Vertical GaN p-n junction diodes with high breakdown voltages over 4 kV,” IEEE Electron Device Lett. 36, 1180 (2015).
6) K. Xu, J. F. Wang, and G. Q. Ren, “Progress in bulk GaN growth,” Chin. Phys. B 24, 066105-1-16 (2015).
7) A. Usui, H. Sunakawa, A. Sakai, and A. A. Yamaguchi, “Thick GaN epitaxial growth with low dislocation density by hydride vapor phase epitaxy,” Jpn. J. Appl. Phys. 36, L899 (1997).
8) K. Motoiku et al., “Growth and characterization of freestanding GaN substrates,” J. Cryst. Growth 237–239, 912 (2002).
9) Y. Oshima, T. Eri, M. Shihata, H. Sunakawa, K. Kobayashi, T. Ichihashi, and A. Usui, “Preparation of freestanding GaN wafers by hydride vapor phase epitaxy with void-assisted separation,” Jpn. J. Appl. Phys. 42, L1 (2003).
10) J. Liu, J. Huang, X. Gong, J. Wang, K. Xu, Y. Qiu, D. Cai, T. Zhou, G. Ren, and H. Yang, “A practical route towards fabricating GaN nanowire arrays,” CrystEngComm 13, 5929 (2011).
11) T. Sekiguchi and K. Sumino, “Quantitative electron beam tester for defects in semiconductors (CL/EBIC/SDLTS),” Rev. Sci. Instrum. 66, 4277 (1995).
12) T. Sugaheara, H. Sato, M. S. Hao, Y. Naui, S. Kurai, S. Tottori, K. Yamashita, K. Nishino, L. T. Romano, and S. Sakai, “Direct evidence that dislocations are non-radiative recombination centers in GaN,” Jpn. J. Appl. Phys. 37, L398 (1998).
13) R. Liu, A. Bell, F. A. Ponce, C. Q. Chen, J. W. Yang, and M. A. Khan, “Luminescence from stacking faults in gallium nitride,” Appl. Phys. Lett. 86, 021908 (2005).
14) W. Lee, H. J. Lee, S. H. Park, K. Watanabe, K. Kumagai, T. Yao, J. H. Chang, and T. Sekiguchi, “Cross-sectional CL study of the growth and annihilation of pit type defects in HVPE grown (0001) thick GaN,” J. Cryst. Growth 351, 83 (2012).
15) M. Zhang, D. Cai, Y. Zhang, X. Su, T. Zhou, M. Cai, C. Li, J. Wang, and K. Xu, “Investigation of the properties and formation process of a peculiar V-pit in HVPE-grown GaN film,” Mater. Lett. 198, 12 (2017).
16) © 2019 The Japan Society of Applied Physics 051005-4
17) B.-C. Chung and M. Gershenson, “The influence of oxygen on the electrical and optical properties of GaN crystals grown by metalorganic vapor phase epitaxy,” J. Appl. Phys. 72, 651 (1992).
18) S. C. Cruz, S. Keller, T. E. Mate, U. K. Mishra, and S. P. DenBaars, “Crystallographic orientation dependence of dopant and impurity incorporation in GaN films grown by metalorganic chemical vapor deposition,” J. Cryst. Growth 311, 3817 (2009).
19) W. Lee, K. Watanabe, K. Kumagai, S. H. Park, H. J. Lee, T. Yao, J. H. Chang, and T. Sekiguchi, “Cathodoluminescence study of nonuniform- ity in hydride vapor phase epitaxy–grown thick GaN films,” J. Electron Microsc. 61, 25 (2012).
20) V. Voronenkov, N. Bokshareva, R. Gorbunov, P. Latshev, Y. Lelikov, Y. Rebane, A. Tsyuk, A. Zubrilov, and V. Shreter, “Nature of V-shaped defects in GaN,” Jpn. J. Appl. Phys. 52, 08JE14-1-4 (2013).