Thin Solid Films 660 (2018) 516–520

Contents lists available at ScienceDirect

Thin Solid Films

journal homepage: www.elsevier.com/locate/tsf

Evaluation of subsurface damage inherent to polished GaN substrates using depth-resolved cathodoluminescence spectroscopy

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ARTICLE INFO

Keywords:
Subsurface damage
Chemical mechanical polishing
Non-radiative recombination
Cathodoluminescence spectroscopy
Gallium nitride

ABSTRACT

The extent of subsurface damage on (0001) GaN wafers post different polishing treatments was quantified using depth-resolved cathodoluminescence spectroscopy (DRCLS). The band edge emission spectra were obtained from CLS with different electron energies, which manifested a significant non-radiative recombination resulted from polishing-induced subsurface damage. Cross-sectional transmission electron microscopy (XTEM) was also used to diagnose the extent of the subsurface damage layer. For the GaN polished with 1.00 and 0.25 μm diamonds abrasive, the extent of non-radiative subsurface damage is about 250 and 100 nm, corresponding to the calculated electron penetration depth at the accelerating voltage for the onset of band edge emission. In this study, the depth of subsurface damage estimated from CL spectra compared well with direct XTEM measurements in GaN substrate.

1. Introduction

Gallium nitride (GaN) has been studied intensively due to its popular utilization in photonic and electronic devices such as Light-emitting diodes (LEDs), laser diodes, and high electron mobility transistor (HEMT) [1]. It was reported and widely acceptable that GaN could be grown only on dissimilar substrates due to the lacking of bulk GaN substrates [2]. However, the advent of single crystal bulk growth techniques such as the high pressure solution growth, the Na-flux method, the ammonothermal method, and the hydride vapor phase epitaxy (HVPE) made it viable to grow high-quality bulk GaN substrates. Additionally, the high quality crystallization of GaN by HVPE on ammonothermal grown GaN seed crystals was developed for an efficient production of bulk GaN substrates by Unipress group in Poland [3,4]. To use the obtained single crystal GaN materials as a substrate for semiconductor device, wafering process are required to form the material into a wafer shape. The bulk GaN substrates were then sliced normal to (0001) using a diamond saw, lapped, and polished in a similar manner to those used for polishing other substrates [5]. The resulting (0001)-exposed GaN subsurface, however, suffered from its severe damage, resulting mainly from the absence of polishing techniques proper to engineer the GaN surfaces with minimal disruption in a sub-nanoscale.

In order to fully exploit the potentials provided by the GaN surfaces, it is necessary for the GaN to possess epi-ready surfaces for avoiding the migration of defects into the GaN epilayers. Previous literature reports argued chemical mechanical polishing (CMP) methods could be an excellent methodology to evolve the epi-ready surfaces on the GaN [5–8]. The argument stated above, yet, still remains unclarified and partially convincing because an analytic technique to diagnose or quantify the extent of the subsurface damage on the GaN substrates is missing so far [9–11]. In particular, Lai et al. attempted to recover the GaN surfaces through the elimination of damage via annealing of the polished GaN, throughout which a series of cathodoluminescence (CL) images served to observe the change in the subsurface feature [9]. However, quantitative evaluation of polishing-driven subsurface damage using the CL images found it difficult due to the limitation of defects' detection at high acceleration voltage. Prior studies on indentation-driven GaN subsurface damage have suggested the CL method could be used to elucidate radiative nature of defects. In these studies, plastic deformation occurred on the surface and created a non-radiative area with 'Star-of-David-like' patterns. These patterns resulted

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https://doi.org/10.1016/j.tsf.2018.07.002
Received 22 February 2018; Received in revised form 3 July 2018; Accepted 3 July 2018

from the defects propagated along the radial component of the stress field, depending on the symmetry of the indenter and crystal system [12,13]. These studies, however, only attempted a qualitative assessment of the subsurface damage, during which the depth of the subsurface damage was not considered and therefore not used to extract the quantitative information associated with the extent of the subsurface damage [14].

Advanced version of CL denoted as depth-resolved cathodoluminescence spectroscopy (DRCLS) was developed to consider the depth of the damage and showed partial success to test a film quality in epitaxial GaN substrates [15]. In this study, we used DRCLS to further demonstrate its adaptability to the assessment of the damage layer depth and its influence on radiative recombination in the GaN substrates post polishing. The DRCLS results were also compared with the images obtained via cross-sectional transmission electron microscopy (XTEM).

2. Experimental procedure

The n-GaN structures were prepared from Lumilog, Inc. and consisted of 250 μm free standing (0001) oriented bulk GaN grown on sapphire substrates by hydride vapor phase epitaxy method. The electron concentration obtained from Hall measurements was ~ $5 \times 10^{17}\,\mathrm{cm}^{-3}$. The threading dislocation density of most of sample areas was lower than $10^{7}\,\mathrm{cm}^{-2}$.

Three (0001)-exposed GaN wafers were used and subjected to different polishing procedures: Two GaN wafers were mechanically polished with different size (1.00 or 0.25 um) of diamond particles (Buehler, MetaDi Ultra) using a Struers Co. TegraPol-35 with TegraForce-5 tabletop polisher with the polyurethane (IC1000) polishing pad. The pressure applied was 7 psi and the linear velocity was kept at 1.5 m/s. The other GaN wafer was polished via CMP process based on the use of soft colloidal silica particles of 80 nm with acidic conditions where the pH is below 3. This CMP process which is developed from Sinmat, Inc. uses the slurry with specific surfactant additives in unique chemical environment to gently polish the GaN surface, as reported elsewhere [8]. It results in removal rates of 50 to 80 nm/m and atomically smooth surface finish with no subsurface damage. Atomic force microscopy (AFM) analysis was performed to evaluate the surface morphology of the substrates post polishing. Scanning electron microscopy (SEM) coupled with cathodoluminescence (CL) analyses of the substrates were performed using FEI Nova NanoSEM 430 equipped with Gatan Mono $\mathrm{CL3}^{+}$ cathodoluminescence system, as described elsewhere [16]. CL spectra were acquired at different accelerating voltages ranging from 1 to 30 kV to probe different damage layer depths present in the substrates. The photomultiplier tube (PMT) bias of the CL system was consistently set to −1400 V for direct comparison among all spectra. Cross-sectional transmission electron microscopy (XTEM) analysis was performed using both JEOL200CX and JEOL2010F to directly observe the extent of the subsurface damages present in the substrates. All XTEM samples were prepared using FEI Strata DB235/ dual-beam focused ion beam (FIB) system with 30 keV Ga + ions, as reported elsewhere [17]. Prior to TEM specimen preparation, all implanted samples were coated with thick carbon films and 1 μm-thick Pt layers were deposited over the top surface to protect the Ga + ion damage during the ion milling process. [18]

3. Results and discussion

Fig. 1(a), (b), and (c) show the typical AFM image of HVPE GaN surface polished with 1.00 and 0.25 μm diamond particle abrasives and CMP, respectively. It is evident that the substrates polished with diamond particle abrasives exhibit many scratches and large variations in surface height, with the severity of the scratches and surface height variations increasing with increasing abrasive size.

In fact, the root-mean-squared roughness ($\mathrm{R_{RMS}}$) of the diamond-polished surfaces is 1.3 and 0.6 nm for polishing with 1.00 and 0.25 μm diamond particle abrasives, respectively. In contrast, the surface of the specimen subjected to CMP was atomically flat with no detectable scratches with $\mathrm{R_{RMS}} \sim 0.1$ nm. As expected, these observations are consistent with prior results investigating the role of polishing treatments on resulting surface morphology [8,10].

The surface morphology variations observed via AFM suggest that subsurface damage (plastic deformation) may occur as a result of polishing with diamond particle abrasives. To investigate this, SEM and CL imaging were performed as shown in Fig. 2.

In the case of SEM imaging, the contrast is related to topographical variation of the surface of the substrates, while for CL imaging, contrast is due to the presence of non-radiative recombination centers, such as dislocations as has been described previously [19,20]. SEM and CL images of substrates subjected to polishing with 1.00 μm diamond particle abrasive are shown in Fig. 2(a) and (b), respectively, and reveal the presence of a significant amount of polishing-induced damage by both surface topography variation as well as non-radiative defect generation. Similarly, SEM and CL images of substrates subjected to polishing with 0.25 μm diamond particle abrasive; shown in Fig. 2(c) and (d), respectively, also reveal a significant amount of polishing-induced damage. In contrast, substrates subjected to CMP only exhibited the presence of threading dislocations associated growth of the substrates, as shown in Fig. 2(e) and (f), and reported elsewhere [21].

CL spectra from each of the substrates were obtained at different electron acceleration voltages are shown in Fig. 3. As the accelerating voltage is increased, the electron beam effectively probes greater depths into the substrate. For the case of the substrates polished using 1.00 μm diamond particle abrasive, shown in Fig. 3(a), band edge emission at 365 nm was not observed until the acceleration voltage was 7 kV. However, in the case of substrates polished using 0.25 μm diamond particle abrasive, shown in Fig. 3(b), the onset of band edge emission was observed for an accelerating voltage of 3 kV. Finally, for the case of substrates polished using CMP, shown in Fig. 3(d), the CL spectra indicates the onset of band edge emission for an accelerating voltage of 2 kV. Additionally, in the case of accelerating voltages ≥7 kV, the band edge emission intensity is 1–2 orders of magnitude greater for the substrate subjected to CMP compared to the two substrates subjected to polishing with diamond particle abrasives. The observations that the onset of band edge emission and band edge emission intensity at higher accelerating voltages were different for each substrate subjected to different polishing treatments suggests that the extent of subsurface, non-radiative damage is different in each case, with the extent being greater in the case of the substrates subjected to polishing with diamond particle abrasives. The electron penetration depth can be determined via the expression $z = 0.052{E_0}^{1.75}/\rho$, where E_0 is the energy of the incident electrons in keV and ρ is the density of the material in g/cm^3 [22]. In the case of GaN, $\rho = 6.1\ \mathrm{g/cm^3}$, which gives $z = 29$, 100 and 250 nm for accelerating voltages of 2, 4, and 7 kV, respectively. Presumably, the penetration depth corresponding to the accelerating voltage producing the onset of band edge emission is related to the depth of polishing-induced subsurface non-radiative damage.

To quantitatively verify the extent of the non-radiative subsurface damage layer estimated from CL, XTEM was performed to directly image the extent of polishing damage for each wafer as shown in Fig. 4. For the substrate polished with 1.00 μm diamond particle abrasive, the damage layer extended 225–275 nm from the surface, as shown in Fig. 4(a); in this case, the dark lines that extend beyond the damage layer into the substrate are bend contours related to specimen preparation and are not related to polishing damage. In comparison, the substrate polished with 0.25 μm diamond particle abrasive exhibited a damage layer extending 80–120 nm from the surface, as shown in Fig. 4(b). The decrease in subsurface damage layer thickness with decreasing abrasive particle size is consistent with prior work on silicon carbide as expected [23].

For case of polishing using diamond particle abrasives, selected area