

Structural and Optical Characterization of Thick GaN Films Grown by the Direct Reaction of Metal Ga and NH₃ in CVD Reactor

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Abstract—Thick GaN films were grown on sapphire by the direct reaction of metallic Ga and ammonia in a conventional RF induction heated chemical vapor deposition reactor. The crystalline and optical qualities of the thick GaN were evaluated as functions of the distance between sapphire substrate and Ga source and growth temperature. For thick GaN grown at the position of 3.5 cm away from the Ga source, the FWHM for the (0002) peak of X-ray diffraction (XRD) curve was about 684 arcsec. The growth rate of the thick GaN film was about 18 $\mu\text{m}/\text{h}$ at this growth condition. The correlation between structural and optical properties of thick GaN films suggested that deep level yellow luminescence (YL) had a close relation with (1010) and (1011) planes developed in the growth. It was speculated that the emission of YL is mainly due to the formation of deep gap state in the band gap by Ga vacancy and impurities trapped at the domain boundary with (1010) and (1011) atomic facets.

Key words: Thick GaN, Metallic Ga, Growth, Crystal Quality, Direct Reaction

INTRODUCTION

The recent realization of efficiently bright blue, green, and yellow light-emitting diodes and laser diodes using InGaN and GaN has stimulated tremendous scientific interest in the physical properties of the group III-nitride materials [Nakamura et al., 1995]. The epilayers of the III-nitride semiconductors are usually grown on sapphire substrate by a metalorganic chemical vapor deposition (MOCVD) [Nakamura et al., 1995] or a molecular beam epitaxy (MBE) [Morcoc et al., 1995]. Although considerable investigations for the growth of high quality III-nitrides have been carried out in the past several years, the development of a suitable substrate material on which lattice-matched III-nitride epilayers can be grown is one of the most serious issues to solve in the near future.

Although various substrates such as MgO [Powell et al., 1993], GaAs [Miyoshi et al., 1992], α - and β -SiC [Steckl et al., 1997; Sitar et al., 1994], MgAl₂O₄ [Kuramata et al., 1995], Si [Hiroyama et al., 1998], etc. have been intensively examined for growing high quality GaN in the last few years, the most attractive and suitable substrate would be bulk GaN crystals. Bulk GaN crystals with the dimensions of a few millimeters have been reportedly grown using a high-pressure synthetic method [Grzegory et al., 1993]. However, these bulk crystals are still too small to be used as substrates. Another promising substrate is the thick GaN film.

Currently, three-types of growth methods for thick GaN are under development. They are (1) hydride vapor phase epitaxy

(HVPE) [Detchprohm et al., 1992; Lee et al., 1998], (2) sublimation of amorphous GaN powders [Kurat et al., 1996], and (3) the direct reaction of metallic Ga with NH₃ using high temperature vapor phase epitaxy [Fischer et al., 1996].

Most of previous studies on the growth of thick GaN have been carried out using the HVPE or sublimation methods. In the HVPE method, HCl gas first reacts with a Ga melt to form GaCl_x (x=1 to 3), which flows down to the reaction zone. On the substrate, GaCl_x reacts with NH₃ to form single crystal GaN. The sublimation method also includes two reaction steps in the growth of single crystal GaN; the formation of amorphous GaN powders by the reaction of metallic Ga with NH₃ at relatively low temperature and the sublimation of amorphous GaN under NH₃. Recently, Fischer et al. reported that thick GaN film was grown with growth rates as high as 250 $\mu\text{m}/\text{h}$ by the direct reaction of ammonia and gallium at considerably high temperatures of above 1,200 °C in a modified sublimation sandwich method (SSM) [Fischer et al., 1996]. For the growth of thick GaN films, they mounted the substrate on the upper part facing the Ga source at the distance of a few millimeters in a cylindrical graphite crucible consisting of two parts, and injected NH₃ into the gap between the Ga source and the substrate.

In this work, we grow thick GaN films on sapphire by the direct reaction of metallic Ga and ammonia in a conventional RF induction heated chemical vapor deposition (CVD) reactor. A cylindrical graphite susceptor was designed to install both substrate and Ga on a lower part of the susceptor. The sapphire substrate was a few centimeters apart from the Ga source and NH₃ gas was directed over toward the substrate from the Ga source. This growth method has all the characteristics of a conventional CVD performed at relatively low temperatures of around 1,000 °C with two sources. Thick GaN films were grown with varying growth conditions and characterized for the determina-

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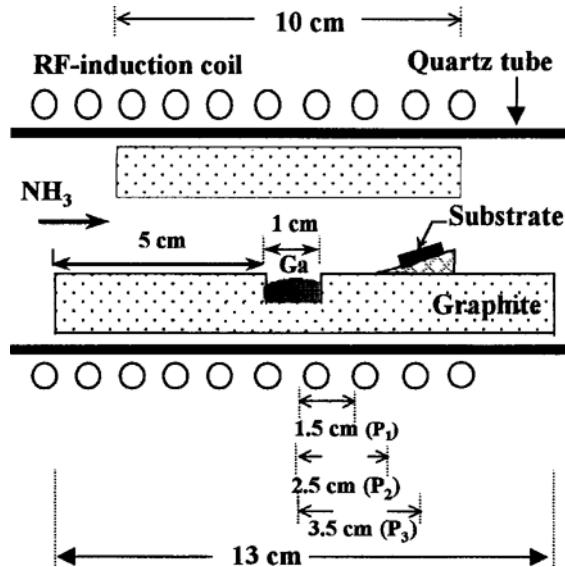


Fig. 1. Schematic diagram of RF-induction heated CVD reactor for the growth of thick GaN by the direct reaction method.

tion of structural and optical properties. We attempted to correlate the YL with the structural properties of the thick GaN films.

EXPERIMENT

Thick GaN was grown by using a modified horizontal CVD reactor as described in our previous report [Kim et al., 1998]. The cylindrical susceptor made in four parts from high purity graphite coated with SiC was heated inductively by a solid state RF generator (15 kW) operating at a frequency around 100 kHz using a solenoid coil. Fig. 1 shows the schematic diagram of the susceptor. The temperature of the susceptor was measured by using an optical pyrometer and calibrated with a Pt/Ru thermocouple.

A 10×7 mm² c-sapphire substrate was chemically cleaned by acetone, etched in 35% HCl for 20 min, and dipped in 25% HF for 5 min. The sapphire substrate was then rinsed with deionized (DI) water and mounted on the graphite susceptor. Metallic Ga and ammonia (NH₃) were used for the Ga and N sources, respectively. Ga (0.1 g) was placed in a hole dug on the lower part of the graphite susceptor, and the substrate was mounted a few centimeters apart from the Ga source on the same plane, as shown in Fig. 1. The distance of the sapphire substrate from the Ga source varied from 1.5 to 3.5 cm. The reactor was evacuated for 30 min and purged with H₂. For cleaning the sapphire surface with hydrogen, the substrate was heated at 540 °C for 10 min under a H₂ flow rate of 100 sccm and then NH₃ was introduced into the reactor with a flow rate of 500 sccm. In order to grow GaN, the temperature of the graphite susceptor was raised to a growth temperature under the NH₃ flow. The growth of thick GaN films was carried out at 0.8 torr with 500 sccm of NH₃ flow, varying the distance between the Ga source and sapphire substrate (1.5-3.5 cm) and growth temperature (1,030-1,150 °C).

XRD and Raman spectroscopy were employed to investigate

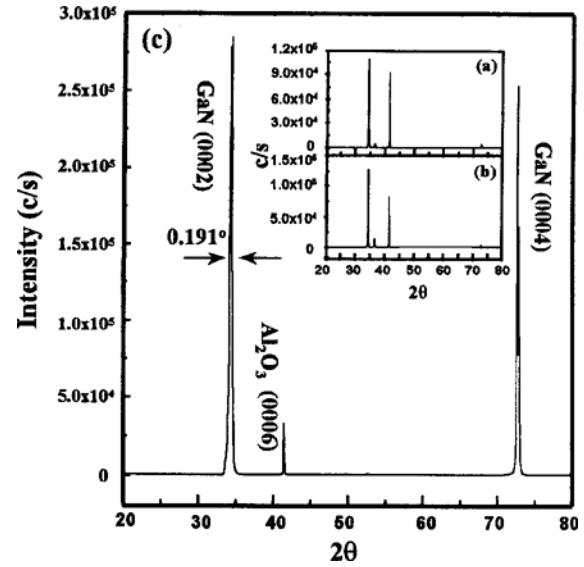


Fig. 2. XRD spectra for thick GaN grown at 1,070 °C for 60 min with 500 sccm NH₃ as a function of the distance between the Ga source and the sapphire substrate: (a) 1.5 cm, (b) 2.5 cm, (c) 3.5 cm.

the structural properties of thick GaN films. The surface and cross-sectional morphologies were examined by using a scanning electron microscope (SEM). Optical properties of the thick GaN were determined by photoluminescence (PL). XRD measurements were made using CuK α . The PL excited by a He-Cd laser at wavelength 325 nm was detected and analyzed by a photomultiplier tube, 1 m double-grating monochromator and photon counting system. The room-temperature Raman spectra were taken in the near backscattering z(xu)z geometry by using the 514.5 nm line of an Ar⁺ laser as the excitation source. The polarization of the scattered light was not analyzed.

RESULTS AND DISCUSSION

Fig. 2 shows XRD patterns for thick GaN films grown on a sapphire surface positioned at 1.5 (P₁), 2.5 (P₂), and 3.5 (P₃) cm away from the Ga source, respectively. For GaN films grown at P₁ and P₂, XRD spectra exhibit a sharp peak of wurtzite GaN (0002) at $2\theta=34.35^\circ$ with blunt peaks for GaN (1011) and GaN (0004) at $2\theta=36.75^\circ$ and 72.85° , respectively [Fig. 2(a) and (b)]. However, the XRD spectrum for GaN grown at P₃ shows diffraction only from the c-plane of GaN and the sapphire substrate [Fig. 2(c)]. This indicates that the crystal quality of the thick GaN films improves significantly when the distance between the sapphire substrate and the Ga source is increased. The uncorrected FWHM of the (0002) peak is about 0.191° (corresponding to about 684 arcsec).

The Raman spectrum for thick GaN/sapphire grown with the conditions of Fig. 2(c) exhibits the high frequency E₂ mode at 568 cm⁻¹, also indicating the growth of wurtzite GaN crystal mainly in the c-direction as verified by XRD in Fig. 2(c). No clear indication of the inclusion of cubic GaN is observed in our Raman analysis (Fig. 3). SEM observation showed that the thickness of the thick GaN film grown at the condition of Fig. 2(c)

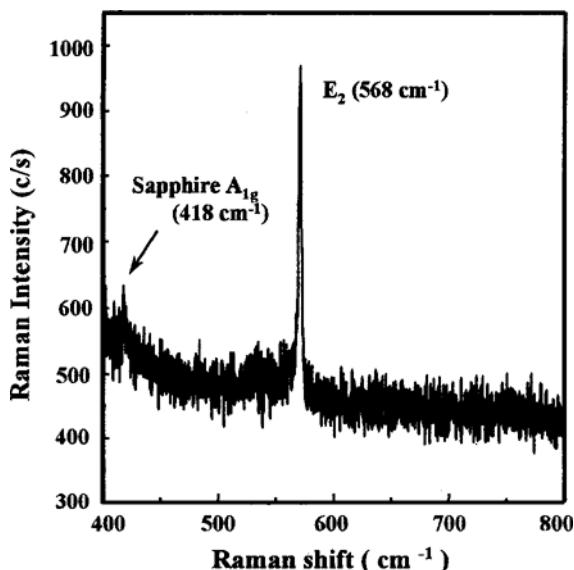


Fig. 3. A typical Raman spectrum for thick GaN films grown at the same condition of Fig. 3(c).

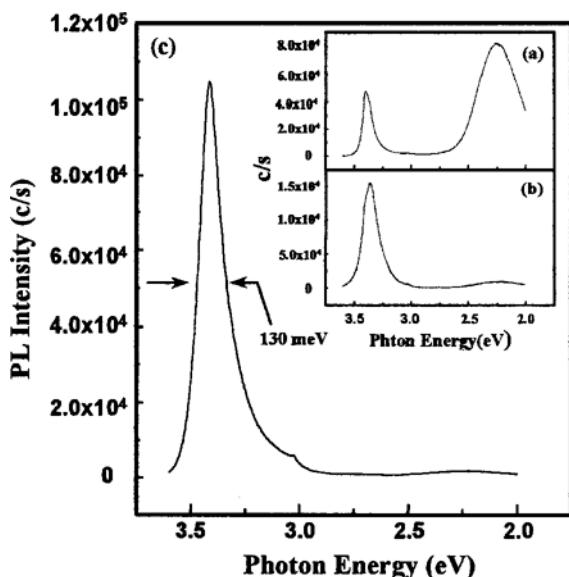


Fig. 4. PL spectra measured at room temperature for thick GaN grown at 1,070 °C for 60 min with 500 sccm NH₃ as a function of the distance between the Ga source and the sapphire substrate: (a) 1.5 cm, (b) 2.5 cm, (c) 3.5 cm.

was about 18 μm .

PL spectra were measured at room temperature for the samples grown at each condition described in Fig. 2. The PL spectrum for GaN film grown at P₁ exhibits both deep level yellow (YL) and band edge (BE) emissions at peak energies of 2.246 and 3.398 eV, respectively, with a peak intensity ratio of $I_{BE}/I_{YL}=0.5$ [see Fig. 4(a)]. For GaN films grown at P₂ and P₃, however, strong band edge emissions were observed at the energy positions of 3.358 and 3.414 eV, respectively, while a deep level yellow emission was greatly suppressed as shown in Fig. 4(b) and (c). This means that optical properties of the GaN thick films are greatly dependent on the distance of the substrate from

the Ga source. The FWHM of Fig. 4(c) is about 130 meV and the peak intensity ratio of I_{BE}/I_{YL} is 62.

In this system, the growth may be influenced by the vapor pressure of liquid Ga, the temperature profile in the graphite susceptor, and the flow pattern of NH₃ in the reactor. Metallic Ga melts at 29.8 °C to form liquid phase [Alcock et al., 1984]. The vapor pressure of liquid Ga is strongly dependent on temperature, which is represented by Arrhenius equation, and is about 1.43×10^{-2} torr at 1,070 °C [Alcock et al., 1984]. Since the growth came about at a fixed temperature and pressure with 0.1 g of Ga in this growth system, the vapor pressure of Ga should remain at a constant value. Consequently, the amount of Ga transferred to the substrate surface from the Ga source may be affected by the NH₃ flow rate and the temperature gradient generated in the graphite susceptor. The temperature of the graphite boat will decrease as it travels from the center to the edge due to the heating property of graphite with RF-induction. For high temperature gas-solid heterogeneous reactions, the growth reaction on the reacting surface is greatly influenced by both mass transport and adsorption of reacting species to and on the surface rather than the chemical reaction at the surface. At a constant flow rate of NH₃ (500 sccm), the temperature gradient in the graphite susceptor impacts significantly the amount of Ga transferred to the substrate surface since the adsorption coefficient of gaseous Ga will decrease with an increase in the surface temperature. We measured temperatures at positions of 0, 1.5, 2.5, and 3.5 cm apart from the center and observed the decrease of the temperature along the axial direction of the graphite from the Ga source to the edge. After the growth, we also found that the amount of deposited solid-products increased from the center to the edge of the graphite susceptor. Considering that the amount of Ga adsorbed on the reacting surface increases with the axial distance of the graphite boat from the center to the end, we can speculate that Ga-deficient GaN film may grow at P₁, while stoichiometric GaN film grows at P₃. This might bear some relationship to the observations from the PL spectra for the GaN films. Fig. 4 shows a deep level yellow luminescence light dominantly emitted from GaN grown at P₁, while the band edge emission prevailed in GaN grown at P₂ and P₃. Our experimental observations indirectly support the previous reports that the Ga vacancy is related to the origin of the yellow luminescence emitted from GaN [Zhang et al., 1995; Neugebauer et al., 1994]. Neugebauer and Van de Walle predicted that the gallium vacancy and related complexes such as V_{Ga}-O or V_{Ga}-Si, acting as deep acceptors, are the most favorable origin of the yellow luminescence [Neugebauer et al., 1994]. Nishida et al. have also observed the similar variation of the PL spectrum from the YL to the BE with the structural change when decreasing H₂ carrier flow rate in a two-flow horizontal MOVPE reactor [Nishida et al., 1997]. The (1011) peak on XRD spectra of GaN films grown at the positions of P₁ and P₂ is another evidence to elucidate the origin of the YL. Liliental-Weber et al. identified that a V-shaped pinhole forms between two (1011) planes which are inclined about 30° to the c-axis and the density of the pinhole defects increases dramatically with an increase of oxygen content in GaN films to persuade the (1011) GaN growth [Liliental-Weber et al., 1997]. Cathodoluminescence image also revealed that pre-

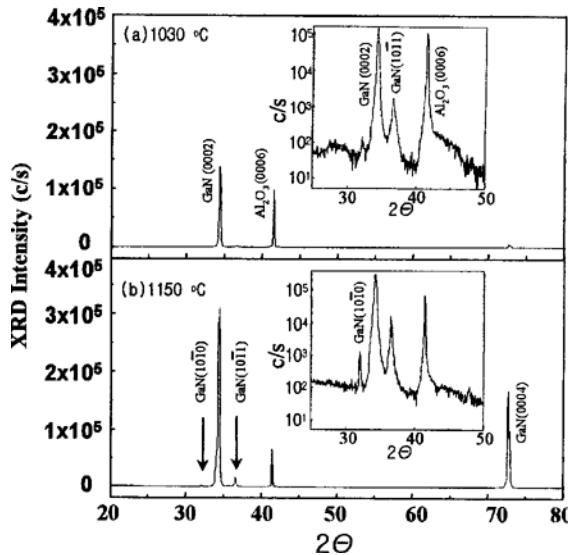


Fig. 5. XRD spectra for thick GaN grown for 60 min with 500 sccm NH_3 at (a) 1,030 °C and (b) 1,150 °C.

cipitates formed in GaN epilayers during the growth are mainly composed of O and Ga, and is responsible for the YL in the GaN epilayers [Kang et al., 1998]. These experimental observations suggest that the (1011) GaN growth bears some relation to the YL of PL spectra [Fig. 2(a) and (b)].

To investigate the effect of growth temperatures on thick GaN films, the films were grown on the sapphire positioned at 3.5 cm away from Ga source for 60 min, varying the growth temperature of 1,030–1,150 °C. Fig. 5 shows representatively XRD patterns for thick GaN films grown at 1,030 and 1,150 °C, respectively. For all films, we observed diffraction predominantly from the c-plane of GaN and sapphire, and some smaller peaks corresponding to (1010) and (1011) refractions at $2\theta=32.2$ and 36.7°, respectively. Moreover, it is interesting to see that the intensity of the smaller peaks is significantly influenced by the growth temperatures. The (1010) and (1011) peaks almost disappeared at 1,070 °C. The growth of the GaN films along [1010] and [1011] directions is mainly due to misoriented structures. It has been reported that the growth of misoriented structures is significantly affected by GaN growth condition and surface preparation condition of substrates. Hashimoto et al. observed the appearance of (1011) signal from GaN grown on 3C-SiC/Si(100) and attributed the origin of the signal to stacking faults [Hashimoto et al., 1996]. Preschilla et al., who grew GaN epilayer on GaAs, also detected (1010) and (1011) peaks in the XRD measurements [Preschilla et al., 1998]. They suggested that a gallium-poor surface favors the growth of GaN along the [1010] and [1011] direction and gallium-rich surface principally prefers the (0002) GaN growth. On the other hand, Park et al. watched the signal of (1011) plane from GaN grown on ITO glass substrates [Park et al., 1998]. In their experiments, they found that the sample with a buffer layer had the highly oriented (0002) plane parallel to the substrate surface, whereas that without the buffer layer was composed of the mixed crystalline orientation of [0002] and [1011] directions. Meanwhile, many other groups have also revealed the inclusion of (1010) and

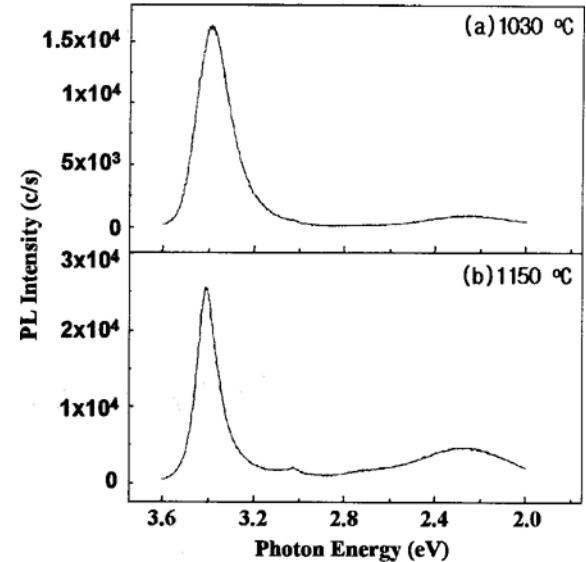


Fig. 6. PL spectra measured at room temperature for thick GaN grown for 60 min with 500 sccm NH_3 at (a) 1,030 °C and (b) 1,150 °C.

(1011) planes in the GaN films [Liliental-Weber et al., 1997].

Fig. 6 is PL spectra of the GaN films measured at room temperature. Band edge luminescence is shown at 3.414 eV. The anomalous sharp peak at 3.02 eV is not induced by sample but by measurement system. Deep level related yellow luminescence is shown at 2.25 eV. For the origin of YL, there are many candidates reported in the previous works: residual impurities such as carbon and oxygen, native defects such as vacancies and antisites, and extended defects such as dislocation and domain boundaries. But the exact origin of the YL is still debated. Our PL measurements show that the intensity of YL increases at the temperatures which grow the GaN along the [1010] and [1011] directions. The intensity varied in a similar way with that of (1010) and (1011) peaks.

To investigate the correlation between XRD and PL results, the intensity variation of (1010) and (1011) peaks and that of YL are drawn in Fig. 7. For an accurate comparison, the intensities of XRD peaks were normalized to that of the (0002) peak, and the YL intensity was normalized to that of band edge luminescence. Fig. 7 shows that the intensity of XRD peaks varies in a similar way with that of the YL. This means that the structural properties have some effects on the optical properties. When we observed cross-sectional SEM images for thick GaN films grown at 1,150 °C, it was found that the film involved many misoriented defects and micrometer-sized grain structures.

Many research groups have studied theoretically and experimentally the structural properties of (1010) and (1011) planes and some of them have attempted to correlate the structures with the optical properties of GaN. Liliental-Weber et al. observed in their experiments that nanopipes and pinholes are originated from the V-defects which are composed of (1010) and (1011) planes and the density of the structural defects increases dramatically with an increase of oxygen content in GaN films [Liliental-Weber et al., 1997]. Jones et al. proved with their theoretical study that Ga vacancy has lower formation energy

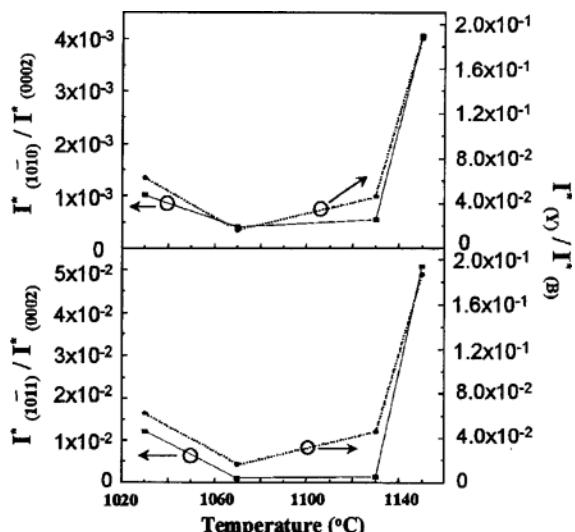


Fig. 7. The integrated peak intensity ratios of YL to BL and of (10 $\bar{1}$ 0) and (10 $\bar{1}$ 1) to (0002) as a function of the growth temperature.

near the extended defects, such as domain boundary and dislocation, and constructs more stable structure by forming the complex with oxygen [Jones et al., 1999]. Elsner et al. also insisted that the complex of Ga vacancy and oxygen trapped at threading edge dislocation act as deep acceptor [Elsner et al., 1998]. Meanwhile, Northrup et al. suggested that stacking mismatch boundary which is closely related with stacking faults induces the interface state located at 1.1 eV above valence band maximum and affiliates to the YL [Northrup et al., 1996]. Noguez et al. identified with their calculation that (10 $\bar{1}$ 0) surface has gap state of 2.7 eV [Noguez et al., 1998]. On the other hand, Kang et al. experimentally observed a strong YL emission on the precipitates composed of oxygen and Ga interstitials formed on the dislocation and grain boundary [Kang et al., 1998]. Ponce et al. also noticed the YL emission from small crystallites surrounded by extended defects formed at grain and grain boundary [Ponce et al., 1996]. Summarizing the above experimental and theoretical studies, it seems that the origin of YL has a direct relation to extended defects, native defects, and impurities included in the GaN films. Consequently, according to our observation on the consistent correlation between the YL and the structures of (10 $\bar{1}$ 0) and (10 $\bar{1}$ 1) planes, it is strongly suggested that the emission of YL is mainly due to the formation of deep gap state in the band gap by Ga vacancy and impurities trapped at domain boundary with (10 $\bar{1}$ 0) and (10 $\bar{1}$ 1) atomic facets. This result partly supports Ogino and Aki's report that a mechanism of the YL process follows shallow donor-deep acceptor transition [Ogino et al., 1980].

CONCLUSION

Wurzite GaN thick film was grown on sapphire substrate at 0.8 torr and 500 sccm NH₃ by the direct reaction of metallic Ga (0.1 g) in a conventional CVD reactor, varying the distance of the substrate from the Ga source at the range of 1.5-3.5 cm and

the growth temperature of 1,030-1,150 °C. XRD spectra for GaN grown at 1.5 and 2.5 cm exhibited a sharp peak of wurtzite GaN (0002) at $2\theta=34.35^\circ$ with blunt peaks for GaN (10 $\bar{1}$ 1) and GaN (0004) at $2\theta=36.75^\circ$ and 72.85° , respectively, while GaN grown at 3.5 cm showed diffraction only from the c-plane of GaN and the sapphire substrate. The uncorrected FWHM of the (0002) peak was about 684 arcsec. A room-temperature PL spectrum for GaN film grown at 1.5 cm exhibits both deep level yellow (YL) and band edge (BE) emissions at peak energies of 2.246 and 3.398 eV, respectively, with a peak intensity ratio of $I_{BE}/I_{YL}=0.5$, while strong band edge emissions were observed at the energy positions of 3.358 and 3.414 eV for GaN films grown at 2.5 and 3.5 cm, respectively. The growth temperature also had a tremendous effect on the structural and optical properties of thick GaN films. The correlation between the structural and optical properties of thick GaN films showed that the YL had a close relation to (10 $\bar{1}$ 0) and (10 $\bar{1}$ 1) planes developed in the GaN growth. It was speculated that the YL emits from the deep gap state formed in the band gap by Ga vacancy and impurities trapped at domain boundary with (10 $\bar{1}$ 0) and (10 $\bar{1}$ 1) atomic facets.

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