

## Reduction of wafer bow in free standing GaN grown by HVPE

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**In this study, GaN layers on sapphire were grown by hydride vapor phase epitaxy (HVPE). And free standing (FS) GaN layers were obtained after laser lift off (LLO) process. We controlled growth temperature to minimized bow of the FS-GaN after LLO process. Target thickness of GaN epilayers were over 300  $\mu\text{m}$ . GaN templates showed strong convex bowing at room temperature and the bow values showed any particular relation with growth temperature. But bows of FS-GaN substrates after LLO treatment showed mainly concave mode and those decreased according to reducing the growth temperature from 1010  $^{\circ}\text{C}$  to 1000  $^{\circ}\text{C}$  We show that reduction of bows in FS-GaN can be controlled by the growth condition of HVPE process.**

**Key words:** GaN substrate, Free standing GaN, HVPE and Bow.

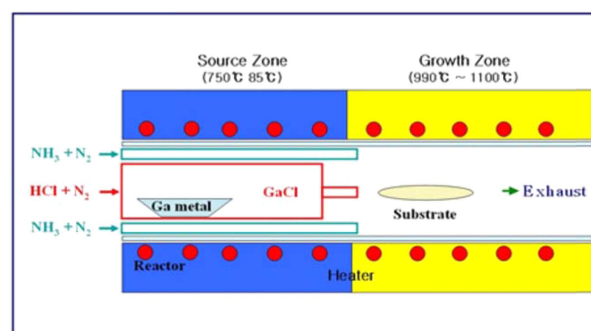
### Introduction

At present, next generation of high-efficiency lighting and high-power electronics are considered as one of the tasks for innovation technologies for energy saving in world industry. In this context, gallium nitride (GaN) has attracted a great attention for its material properties that are useful for applications in short wavelength optoelectronic and high-power electronic devices, such as white or color light-emitting diodes, blue laser diodes, UV detectors, high-power and high-frequency transistors [1]. However, since true bulk GaN substrates are not yet fully commercially available, GaN based devices are currently grown on non-native substrates, with misfit and threading dislocation densities as high as  $10^9/\text{cm}^2$ . So the device performance remains well below the theoretical capability of GaN. Therefore, native bulk crystal substrates are needed to realize the true potential of GaN-based devices. Several techniques are being used to grow GaN substrates [2]. Among them, HVPE is one of the most promising methods for the growth of bulk GaN crystals because of its high growth rate compared to other techniques [3]. But bulk GaN substrates are still a big issue despite the recent fast progress of this technology [4]. So the FS-GaN emerged as an alternative for bulk for GaN substrate. But still the large concave bow is commonly reported for crystals grown by HVPE and separated from sapphire substrates and is being assigned to large structural defects concentration gradients in the thick

HVPE GaN layers and to stress induced by lattice and thermal mismatch between GaN and  $\text{Al}_2\text{O}_3$  [5]. In this study, FS-GaN grown by HVPE was presented. We investigate the effect of growth temperature on the mechanical bow of FS-GaN with the purpose of minimizing the mechanical bow. FS-GaN substrates were attained through the LLO process and bow and stress of them were characterized.

### Experimental

Fig. 1 shows a schematic diagram of HVPE system used in this study. The process zone consisted of two parts, namely, the source zone and the growth zone. Ammonia ( $\text{NH}_3$ ) gas and hydrochloric acid (HCl) gas were used as the active gases, and metallic gallium was used as group III precursor. The substrate used was 52.5 mm (0001) sapphire. Ga boat was located in a separate tube at a temperature of 750  $^{\circ}\text{C}$ . GaCl gas was generated by reaction with HCl at source zone and was then transported by the  $\text{N}_2$  carrier gas. After the nitridation of sapphire substrate, GaN epilayers were



**Fig. 1.** Schematic diagram of the HVPE system.

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**Table 1.** The growth conditions and thickness of GaN epilayers.

Sample ID	Growth Temp (°C)	Total Growth Time (min)	Thickness (μm)	Growth Rate (μm/h)
#1	1010	135	318	141
#2	1005	135	313	139
#3	1003	125	309	148
#4	1000	125	313	150

grown in the growth zone.

Table 1 shows the growth conditions of GaN epilayers. The surface-treatment was accomplished by supplying of the mixture of NH<sub>3</sub> and N<sub>2</sub> gases at high temperature around 1020 °C for the nitridation of 52.5 mm (002) sapphire for the fabrication of 2" FS-GaN substrate. After surface treatment, GaN epilayers were grown between 1000 ~ 1010 °C without the buffer layer. Total growth time of sample #1 & #2 kept at 135 min and that of #3 & #4 was kept at 125 min considering the high growth rate at low growth temperature. The target of thickness for GaN layer was 313 μm ± 5% (15.7 μm) for each sample. FS-GaN substrates were prepared by treatment of LLO. LLO system were consisted the laser source and the lens system, furnace and moving stage. High energy pulsed laser (Quantel, YG 981E, France) at 355 nm wavelength was applied as a laser source. GaN templates were inserted in modified box furnace with quartz window on top side on the home built moving stage and the back side of GaN templates were exposed by focused laser. Sapphire were separated from GaN template by the vaporization of several μm of GaN layer at the interface of sapphire and GaN.

The surface of GaN layers were analyzed by optical microscope (OM) (Reichert, Reicher Polyvar SC, Austria) and video microscope (Sometech, Camscope ICS-305, Korea). The bow of GaN template and FS-GaN substrates were measured by 48 mm scanning of stylus surface profiler (Veeco, Dektak 150, USA). The thickness of FS-GaN and GaN templates were measured at five-points of each sample by the contact-type high precision digital measuring in-struments (Tokyo Seimitsu, Miniax DH-150, Japan). Raman spectra of the FS-GaN was measured by a micro-Raman system (NSR-3100, Jasco, Japan) with 1 cm<sup>-1</sup> resolution, 1024 × 128 pixel CCD aircooled to -65 °C, and excitation at 532 nm at room temperature. By fitting Lorentzian lines to the Raman peaks, the precision in the determination of the frequency shifts could be further increased like that spectral shifts of 0.1 cm<sup>-1</sup> can be reliably detected [6, 7]. A high-resolution x-ray diffraction (XRD) (PANalytical, X'Pert-PRO MPD and MRD, Holland) with with a CuKα line source (12x 4 mm), angle resolution 0.0001 ° and line slit (1/16 °) for the incident beam was employed to characterize the structural properties.

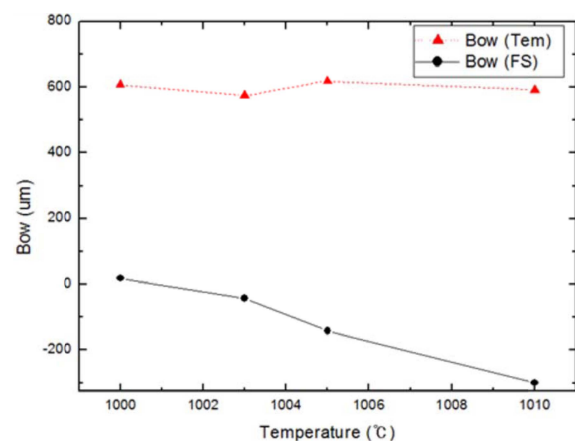
## Results and Discussion

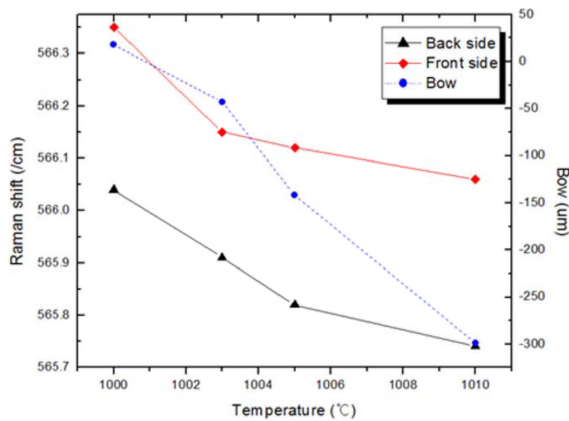
Four GaN templates were grown without wafer broken and crack inside 50.4 mm. FS-GaN substrates were accomplished successfully to remove sapphire substrate from GaN templates by LLO process. The data of bow for four GaN templates and FS-GaN substrates were shown in Table 2. The thickness of four FS-GaN substrates were shown within 1.6% run-to-run uniformity (313 μm ± 5.0 μm).

Fig. 2 shows bows of GaN templates and FS-GaN substrates with the variation of growth temperature. GaN templates showed strong convex bowing at room temperature as commonly reported [5]. Bows of them lies between 574 and 699 μm, showed any particular relation with growth temperature. But the bows of FS-GaN substrates after LLO treatment were changed from -299 μm to + 18 μm according to the variation of growth temperature from 1010 °C to 1000 °C as shown

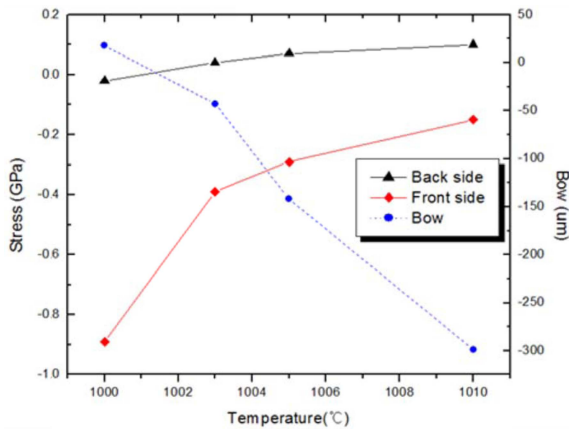
**Table 2.** Bows and thickness of GaN templates and FS-GaN substrates.

Sample ID	Bow After Growth (μm)	BOW After LLO (μm)	Thickness (μm)
#1	591	-299	318
#2	618	-142	313
#3	574	-43	309
#4	607	18	313

**Fig. 2.** Bows of GaN templates and FS-GaN substrates with different growth temperature from 1000 °C to 1010 °C.



**Fig. 3.**  $E_2$  high peak position of Raman measurements for front side and back side of FS-GaN substrates and bow of FS-GaN substrates with different growth temperatures.



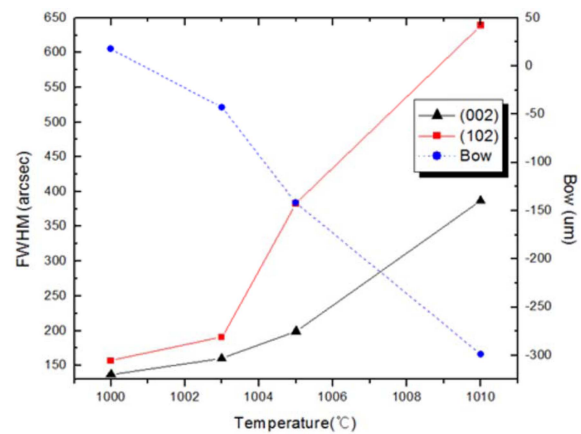
**Fig. 4.** Difference in stress between the front and back side of FS-GaN and bows of FS-GaN.

in Table 2. Sample #4 showed weak convex bowing even though others samples showed concave mode like as usual FS-GaN substrate [8]. The growth temperature is expected as an effective parameter for the control of bow for FS-GaN. The mode of bow of FS-GaN also can be controlled to convex mode even the usual case is concave mode.

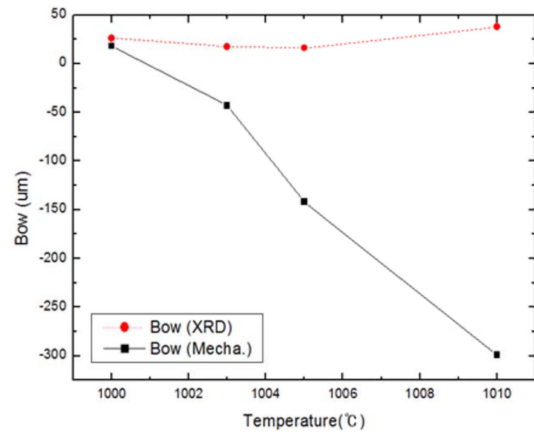
The  $E_2$  high peak at Raman mode is known to be affected by stress [9-13]. It was evaluated that  $E_2$  high peak was positioned at 566 nm for FS-GaN with null strain or stress [11].  $E_2$  high peaks were positioned at status of the compressive stress such as higher than 566 nm in Raman measurement for GaN template [10].

The gaps of  $E_2$  high peak position for front and back side of FS-GaN substrates were explained due to the difference of residual strain for each side of them, having some extent of bow. In case of front side of FS-GaN,  $E_2$  high peak shift to higher band as the bows of FS-GaN decreased. In case of back side of FS-GaN,  $E_2$  high peak also shift to higher band with the bow of FS-GaN decreased.

The stress values of FS-GaN substrates were calculated



**Fig. 5.** Relation between the Bow and FWHM values for rocking curve of GaN (002) and (102) plane of FS-GaN substrates.

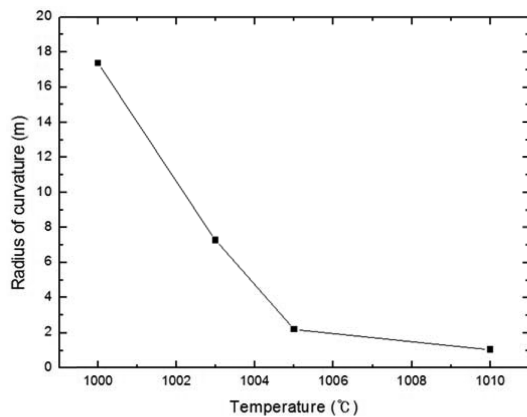


**Fig. 6.** Bows of FS-GaN substrates (a) mechanical bow (b) bow from XRD.

according to equation from the Raman shift [14]. As shown in Fig. 4, compressive stress were measured at the front side of FS-GaN and tensile stress was formed at the back side of FS-GaN except one sample grown at 1000 °C FS-GaN grown at 1000 °C showed compressive stress on both side. The compressive stress at the front side was increased from -0.15 GPa to -0.89 GPa as the bow of FS-GaN decreased from -299 to +18 μm. And the tensile stress at the back side was decreased from 0.67 GPa to 0.23 GPa with the reduction of bow and finally changed to compressive stress of -0.11 GPa with the bow of +18 μm.

Owing to difference of lattice parameter between front side and back side of FS-GaN wafer, FS-GaN showed concave mode bowing in free stress state [8]. So there seems to need some stress on both side of FS-GaN to minimized bow of it. So the large compressive stress was formed at the front side of FS-GaN which showed minimum level of bow.

Fig. 5 shows the relation between the bow and FWHM values for rocking curve of GaN (002) and (102) plane of FS-GaN substrates. FWHM values of rocking curve for GaN (0002) and (10-12) plane were



**Fig. 7.** Radius of Curvature of FS-GaN substrates calculated from mechanical bows.

effected by the absolute value of bow for each samples due to using of a  $\text{CuK}\alpha$  line source ( $12 \times 4$  mm) for sample #1, #2, and #3 such as high value of bow. In measurement of the best FWHM values for RC of GaN (0002) and (10-12) were 137.0 arcsec and 156.7 arcsec respectively in sample #4 which showed minimum bow value. As the growth temperature decreased, bows and FWHM of (002) and (102) plane of FS-GaN substrates also decreased.

The radius of curvatures and bows of FS-GaN substrates were also evaluated by 5-points measuring of (0004) rocking curve along centre line parallel to main flat plane. As shown in Fig. 6, mechanical bows do not agree well with the bow from XRD.

Fig. 7 shows the calculated radius of curvature from the results of mechanical bows of FS-GaN substrates. The radius of curvature showed in the range between 1.05 and 17.36 m. Sample #4, which showed minimum bow of  $+18 \mu\text{m}$  showed largest value of radius of curvature, 17.36 m

## Conclusions

Thick GaN layers on sapphire were grown by HVPE with the variation of growth temperature. After that, free standing GaN layers were prepared after LLO process. Bows of GaN templates with sapphire were in the range from 574 to 618  $\mu\text{m}$ . Bows of FS-GaN substrates after LLO treatment were changed from  $-299 \mu\text{m}$  to  $+18 \mu\text{m}$  according to reducing the growth temperature from 1010 °C to 1000 °C Thus, 313  $\mu\text{m}$

thick crack free FS-GaN with the low bow of  $+18 \mu\text{m}$  was successfully prepared at the growth temperature of 1000 °C The radius of curvature of FS-GaN, which showed minimum bow, was 17.36 m.

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