

Metalorganic chemical vapor deposition of GaN on Si(111): Stress control and application to field-effect transistors

H. Marchand,^{a)} L. Zhao, N. Zhang, B. Moran, R. Coffie,
U. K. Mishra, J. S. Speck, and S. P. DenBaars
*Department of Electrical and Computer Engineering and Materials, University of California, Santa
Barbara, California 93106*

J. A. Freitas
*ESTD-Electronic Materials Branch, Naval Research Laboratory, Code 6872, 4555 Overlook Avenue S.W.,
Washington DC 20375-5347*

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Two schemes of nucleation and growth of gallium nitride on Si(111) substrates are investigated and the structural and electrical properties of the resulting films are reported. Gallium nitride films grown using a 10–500 nm-thick AlN buffer layer deposited at high temperature ($\sim 1050^\circ\text{C}$) are found to be under 260–530 MPa of tensile stress and exhibit cracking, the origin of which is discussed. The threading dislocation density in these films increases with increasing AlN thickness, covering a range of 1.1 to $>5.8 \times 10^9 \text{ cm}^{-2}$. Films grown using a thick, AlN-to-GaN graded buffer layer are found to be under compressive stress and are completely crack free. Heterojunction field effect transistors fabricated on such films result in well-defined saturation and pinch-off behavior with a saturated current of $\sim 525 \text{ mA/mm}$ and a transconductance of $\sim 100 \text{ mS/mm}$ in dc operation.

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I. INTRODUCTION

Gallium nitride and its alloys are important materials for high-power, high-frequency electronic devices¹ as well as optoelectronics in the visible and ultraviolet parts of the electromagnetic spectrum.² Much progress has been made in understanding the nucleation and growth processes on sapphire³ and silicon carbide,⁴ such that most useful devices are currently fabricated using these two substrate materials. Si(111) also has attractive properties as a substrate material, in particular its wide availability, reasonable thermal conductivity, and high etch rate in common wet etchants.⁵ In spite of a large lattice mismatch (17%), GaN films with reasonable structural properties have been grown on Si(111) using a variety of buffer layers (see Ref. 6 and references therein; recent reports include Refs. 7–15). Improvements in microstructural properties were demonstrated using lateral epitaxial overgrowth⁶ and selective area epitaxy.^{16,17} Devices based on GaN/Si(111) layers have also been demonstrated, including light-emitting diodes,^{18–21} photodetectors,^{22,23} and field-effect transistors.^{24,25}

Although these advances are very encouraging, the mechanisms for dislocation generation in GaN films grown on Si(111) substrate are less understood than for growth on sapphire or silicon carbide substrates. In addition, the net stress in all GaN/Si(111) epilayers reported so far has been tensile, which often resulted in cracking (see, e.g., Refs. 6, 9, 13, 18, 19, and 26). Although the thermal expansion coefficients of GaN and Si suggest that a net tensile stress is thermal in nature, recent reports indicate that the grown-in stress

plays an important role in GaN heteroepitaxy on sapphire²⁷ and SiC²⁸ substrates. Moreover, it appears that the grown-in stress may be adjusted, to a certain extent, by carefully controlling the growth conditions during the nucleation and island formation stages.²⁹ The question then arises as to whether it is possible to reduce the dislocation density and the net stress in GaN/Si(111) epilayers by controlling the conditions in the early stages of growth. As a corollary question, one may also ask whether it is possible to achieve both objectives with the same set of growth conditions.

In this paper we discuss three nucleation schemes for growing GaN films on Si(111) substrates and report on the structural and electrical properties of representative samples. We propose and demonstrate an approach for growing compressively strained GaN films using a thick graded buffer layer which also acts as an insulator between the silicon substrate and the channel of a field-effect transistor.

II. EXPERIMENT

GaN films were grown using metalorganic chemical vapor deposition (MOCVD) on B-doped Si(111) substrates (Virginia Semiconductors, 20–80 $\Omega \text{ cm}$). Five processes were investigated as listed in Table I. Samples A–D were grown using an AlN buffer layer with a thickness varying between ~ 10 and $\sim 500 \text{ nm}$, followed by a thick (0.75–1.6 μm) layer of GaN. The AlN and GaN films were deposited at a total pressure of 76 and 760 Torr, respectively. Sample E consisted of a thick ($\sim 0.8 \mu\text{m}$) graded layer whose initial and final composition was that of AlN and GaN, respectively. This was followed by the deposition of an additional 0.2 μm of GaN. The structure was deposited without any growth interruptions. Prior to loading in the growth chamber,

^{a)}Current address: Agility Communications Inc., 600 Pine Ave, Santa Barbara, CA 93117; electronic mail: hmarshand@agilitycom.com

TABLE I. Growth parameters and structural results for the three samples under study. h_{AlN} and h_{GaN} layer thicknesses; FWHM: full width at half maximum of x-ray curves; TDD: threading dislocation density; σ_a : in-plane stress measured by laser deflection. The precision of the stress measurement is on the order of 10%.

Sample	h_{AlN} (nm)	h_{GaN} (μm)	FWHM (0002, ω) (arcsec)	FWHM (0002, $\omega-2\theta$) (arcsec)	FWHM (2021, ω) (arcsec)	FWHM (2021, $\omega-2\theta$) (arcsec)	TDD (cm^{-2})	Crack density (cm^{-1})	σ_a (MPa)
A	~ 500	1.6	~ 4500	692	> 5000	> 5000	Not measured	0 (rough)	261
B	~ 180	0.75	1971	526	3088	2673	$> 5.8 \times 10^9$	~ 21	476
C	~ 25	1.0	525	401	861	880	5×10^9	~ 5	527
D	~ 10	0.9	1497	514	803	818	1.1×10^9	0	437
E	~ 800 (grade)	0.2	(1027)	(1080)	(2538)	(2600)	$> 10^{11} \rightarrow \sim 10^{10}$	0	-270

the silicon substrates were etched in a buffered HF solution to remove the native oxide. An *in situ* hydrogen exposure at 900 °C was performed immediately prior to the AlN deposition step, which was initiated by introducing trimethylaluminum and ammonia simultaneously.³⁰ The substrate temperature was set to ~ 1050 °C for all deposition steps.

Specimens for transmission electron microscopy (TEM) were prepared by wedge polishing followed by standard Ar⁺ ion milling. Images were recorded on a JEOL 2000FX microscope operated at 200 kV. X-ray rocking curves were measured using CuK α radiation from a rotating anode source and a Philips Materials Research Diffractometer. Rocking curves (ω scans) and radial ($\omega-2\theta$) scans were acquired for 0002 (symmetric) and 2021 (asymmetric) scattering geometries using a 1.2 mm receiving slit at the detector entrance. The net stress at room temperature was measured using an *ex situ* wafer curvature measurement setup in which incident parallel laser lines are deflected off the wafer surface, similar to the technique described in Ref. 27.

In some samples a final 20-nm-thick layer of unintentionally doped Al_{0.27}Ga_{0.73}N was also deposited to form a polarization-induced two-dimensional electron gas³¹ at the GaN–AlGaIn interface. These samples were processed into heterojunction field-effect transistors (HFETs) using a standard contact lithography process with 1.25 μm gate length. The source and drain ohmic contact scheme was Ti/Al/Ni/Au annealed at 850 °C, while the gate metallization was Ni/Au/Ni. Mesa isolation for the devices was done using Cl₂ reactive ion beam etching.

III. RESULTS

A. Microstructure

For samples A–D the microstructure of the GaN film was found to be dependent on the thickness of the AlN buffer layer. In the extreme case of sample A, the surface morphology was rough and consisted of 25- to 100- μm -wide uncoalesced islands. Samples B and C exhibited a smooth surface morphology and were further analyzed by TEM as shown in Fig. 1. Sample B [Fig. 1(a)] was characterized by a large density of threading dislocations (TDs) that appeared to originate from highly defected regions in the AlN layer. Observation in plan-view TEM (not shown) revealed that the total TD density near the free surface of the GaN film was $5.8 \times 10^9 \text{ cm}^{-2}$ or larger. The TD density for sample C [Fig.

1(b)] was found to be $5 \times 10^9 \text{ cm}^{-2}$ from plan-view TEM micrographs. The AlN buffer layer in sample C appeared to be more homogeneous than in sample B, and the source of TDs could not be clearly identified (see Sec. IV).

Sample D also exhibited a smooth morphology and revealed a microstructure similar to that of sample C except that the TD density was $1.1 \times 10^9 \text{ cm}^{-2}$ as measured by plan-view TEM (not shown). However, a significant number of circular defects approximately ~ 1 mm in diameter were observed on the surface of this sample, which would possibly reduce its usefulness for device applications. These defects are discussed in Sec. III C.

Sample E [Fig. 1(c)] was grown using a thick graded buffer process. It exhibited a very high TD density at the onset of growth ($> 10^{11} \text{ cm}^{-2}$), but dislocation annihilation and fusion reactions resulted in a significant reduction of the threading dislocation density to approximately 10^{10} cm^{-2} near the free surface.

B. X-ray diffraction

For GaN grown on Si(111) the relationship between the various x-ray diffraction linewidths (symmetric versus asymmetric, ω scan versus $\omega-2\theta$ scan) and the microstructure has not been clearly established (see Refs. 32–34 for the case of GaN on Al₂O₃). To clarify this issue, a systematic study of the linewidths was conducted on samples A–E and on additional samples grown with intermediate AlN and GaN thicknesses. Figure 2 shows the relationship between the $\omega-2\theta$ and the ω linewidths for the 0002 peak. The $\omega-2\theta$ full width at half maximum (FWHM) values were in the range of 400 to ~ 600 arcsec for all samples. Using the Scherrer equation³⁵ this corresponds to a coherence length (grain size) along the *c* axis of ~ 60 nm for all samples. The 0002 ω -scan FWHM varied between 525 and < 4500 arcsec, which indicates that the out-of-plane (tilt) misorientation is strongly affected by the buffer layer thickness. Samples grown using a thinner AlN buffer layer tended to exhibit narrower linewidths. However there appears to be an optimum thickness (~ 25 nm, sample C) for which both the ω and $\omega-2\theta$ linewidths were minimized, even though the dislocation density observed by plan-view TEM was not the lowest.

For all samples analyzed so far, the FWHMs of the 2021 peak in ω scans and $\omega-2\theta$ scans were identical within 10%, such that both measurements could be used indifferently to

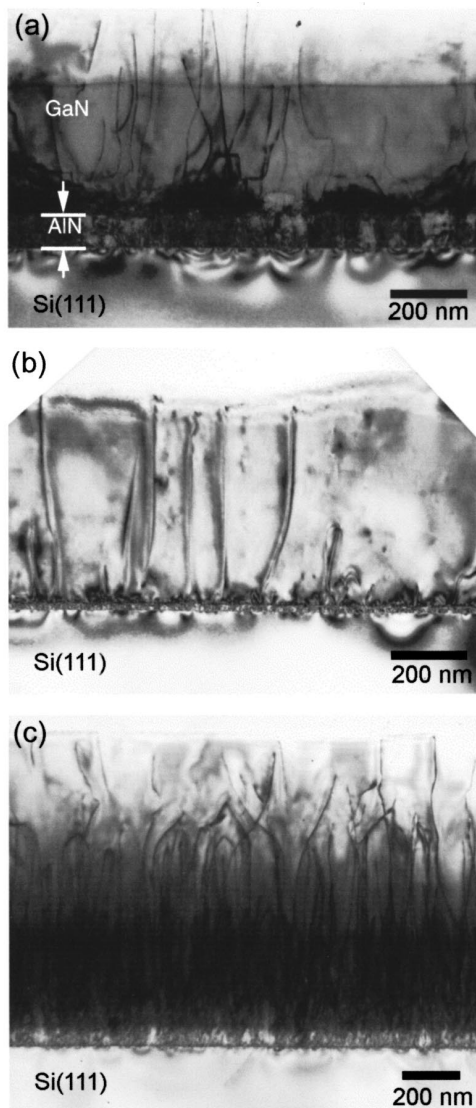


FIG. 1. Transmission electron micrographs of GaN films grown on Si(111) substrates using three buffer schemes (bright field, $g = 0002$). (a) Sample B (the top ~ 200 nm of the GaN film have a different contrast because of shadowing effects during ion milling); (b) sample C; (c) sample E.

describe the in-plane (twist) mosaicity and grain size. As suggested by the data in Table I, thinner buffer layers resulted in narrower linewidths, which is consistent with the reduction in TD density observed in TEM. The linewidths for sample E are shown in Table I for reference; these values cannot be directly compared with those for other samples because the thickness of GaN is only $\sim 0.2 \mu\text{m}$ instead of $\sim 1 \mu\text{m}$.

C. Film stress and cracking

The net film stress was measured at room temperature using a laser-deflection technique. Samples A–D were found to be under tensile stress (261–527 MPa, see Table I) whereas sample E was under compressive stress (270 MPa). Those values were obtained using the experimental results of

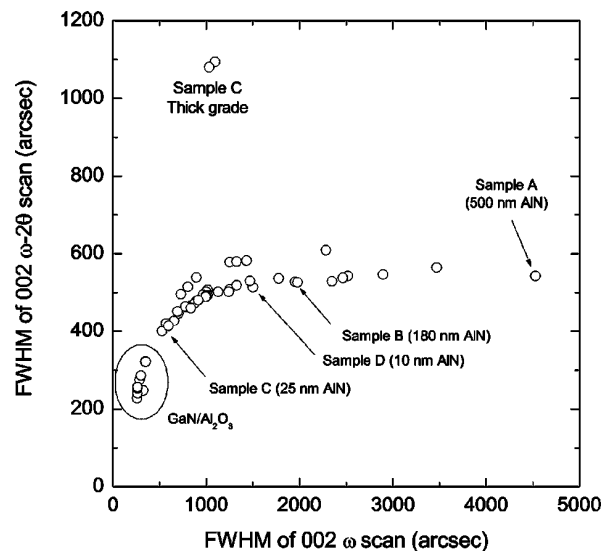


FIG. 2. Typical x-ray diffraction results for GaN/Si(111) samples. Also shown are results for typical GaN films grown on sapphire substrates using a low-temperature GaN nucleation layer.

Polian *et al.*³⁶ for the biaxial modulus (478 GPa) and assuming that the stress is purely biaxial in nature.³⁷

The GaN film in sample B was severely cracked ($\sim 21 \text{ cm}^{-1}$) while sample C exhibited only a few cracks ($\sim 5 \text{ cm}^{-1}$). The cracks were found to be parallel to the $\{1100\}_{\text{GaN}}$ planes. In some cases it was possible to achieve films similar to sample C which exhibited cracking only near the edge of the wafer (as reported in Ref. 9) but, in general, cracks were found to cross the entire wafer surface. Cracking appears to be closely related to the presence of circular growth defects found at the edge of the wafer (see Fig. 3) as well as on the wafer surface. The circular defects could act as stress concentrators and initiate the cracking.

The origin of the circular defects has been tentatively attributed to silicon outdiffusion from the substrate and poisoning of the growing film. Such an observation has already been made for the case of lateral overgrowth of GaN on Si(111) using AlN buffer layers with different thicknesses (see the discussion in Ref. 6). Cracks were occasionally ob-

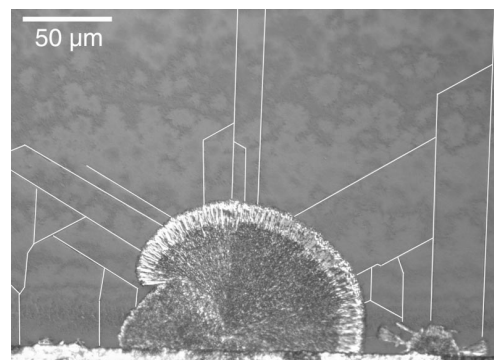


FIG. 3. Optical micrograph showing typical growth defects leading to cracking during the growth of GaN on Si(111) using a thin AlN buffer (sample C). The cracks have been emphasized for clarity. The wafer edge is at the bottom.

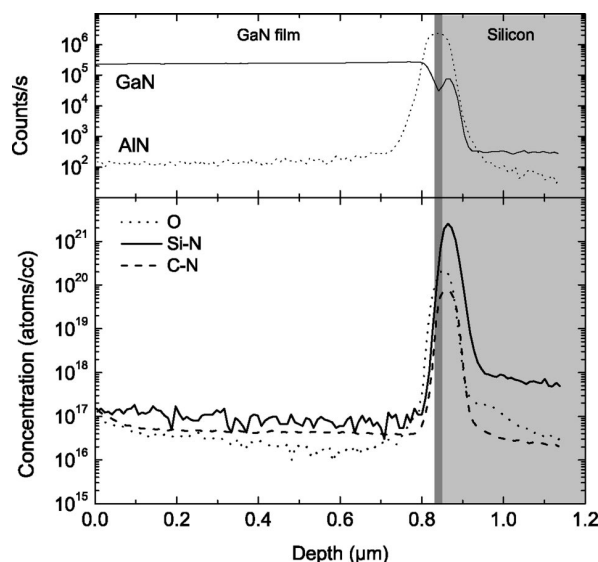


FIG. 4. SIMS results for sample C. The vertical bar indicates the position and the nominal thickness (25 nm) of the AlN layer. Silicon and carbon were detected as species bound to nitrogen.

served in cross-section TEM (not shown), in which case they appeared to have propagated into the silicon substrate by as much as $\sim 0.1\text{--}0.5\text{ }\mu\text{m}$. When samples that exhibited cracks were reloaded and regrown upon, it was found that new circular defects were created, usually accompanied by additional cracking. This suggests that cracking occurs during cooldown, as opposed to during growth.

D. Device results

Samples C and E were processed into HFETs and their I - V characteristics were measured. Sample C resulted in devices which exhibited very low current densities (100 mA/mm) with poor conductance (~ 20 mS/mm) and pinch-off characteristics (not shown). Secondary ion mass spectroscopy (SIMS) analyses were performed to investigate the sources of leakage and are shown in Fig. 4. The analysis suggests that interdiffusion at the GaN-substrate interface was significant.³⁸ The AlN thickness indicated by SIMS (~ 100 nm) was significantly larger than the nominal deposited thickness (25 nm). Ga, Al, N, O, and C were all found to have diffused as far as 50–100 nm in the substrate. As most of these elements are dopants in silicon, it seems reasonable to assume that a highly doped layer is present at the interface, which would act as a parasitic current path.³⁹ Away from the interface, silicon was found in the GaN film at a concentration of $\sim 10^{17}\text{ cm}^{-3}$. Carbon and oxygen were also present. Although the concentrations are moderate, these results suggest that it may be significantly more difficult to achieve an insulating FET buffer using a silicon substrate than using sapphire or silicon carbide.

Sample E yielded transistors with well-defined characteristics leading to complete pinch-off (Fig. 5). In dc operation the maximum saturated current was 525 mA/mm and the maximum transconductance was ~ 100 mS/mm. This is comparable to FET performance characteristics demonstrated us-

ing sapphire and silicon carbide substrates by several groups.⁴⁰ Pulsed measurements were taken to simulate bias conditions at high frequency (Fig. 5, gray curves) and show that high-frequency current collapse (dispersion) is only a minor effect in these devices. High-frequency measurements are under way and will be reported elsewhere. Hall effect measurements were carried out on sample E by etching a mesa into the substrate and depositing ohmic contacts on the AlGaN surface. At 293 K (77 K) the resulting sheet charge was $8.5 \times 10^{12}\text{ cm}^{-2}$ ($9.4 \times 10^{12}\text{ cm}^{-2}$) and the electron mobility was $1428\text{ cm}^2/\text{Vs}$ ($3923\text{ cm}^2/\text{Vs}$), the highest reported values to date for growth on Si(111).

IV. DISCUSSION

We first discuss the properties of GaN films growth using thin AlN buffer layers. The results presented in Table I and Figs. 1 and 2 indicate that, to a certain extent, it is possible to reduce the stress and the dislocation density simultaneously by adjusting the thickness of the AlN layer. For 10-nm-thick AlN buffer a dislocation density of $1.1 \times 10^9\text{ cm}^{-2}$ was observed, with some residual cracking only near the wafer edge (sample D). Schremer *et al.* (Ref. 9) have reported a similar result with a dislocation density of $3 \times 10^9\text{ cm}^{-2}$. These are among the lowest reported dislocation densities for MOCVD-grown GaN films on silicon. Threading dislocation densities of $\sim 5 \times 10^9$ and $8 \times 10^8\text{ cm}^{-2}$ have been reported for growth by molecular beam epitaxy in Refs. 18 and 12, respectively.

The results shown in Table I demonstrate that the dislocation density does not saturate as the thickness of the AlN buffer is decreased. This suggests that even lower dislocation densities could be achieved by further tailoring the properties of the AlN buffer layer. However, we note that the density ($\sim 2\text{ cm}^{-2}$) and size ($\sim 1\text{ mm}$) of the circular defects such as shown in Fig. 3 were significantly higher for sample D (10-nm-thick AlN) than for the other samples. This reinforces the hypothesis that the circular defects are caused by outdiffusion of silicon from the substrate: As discussed in Ref. 6, the AlN layer may act as a diffusion barrier such that a minimum thickness of AlN is required to preserve useful morphologies (see also Refs. 7 and 14). This could ultimately limit the dislocation density that can be achieved using the thin AlN buffer approach.

We note that the 0002 and $\overline{2021}$ x-ray diffraction line-widths exhibit a minimum at different buffer thicknesses (25 and 10 nm, respectively, see Table I). This suggests that there is a trade-off between the density of pure edge dislocations and mixed-character dislocations as the thickness of the AlN is decreased. Thus there might also be a fundamental limit to the dislocation density independent of the film morphology, although this remains a speculation at this point.

The TEM results presented in Fig. 1 demonstrate that the microstructure of GaN films is affected by the thickness of the AlN buffer layer, the mechanisms for which are currently unknown.⁴¹ As a first step toward understanding the evolution of the properties of the AlN films with varying thickness, AlN buffer layers were grown on Si(111) and their

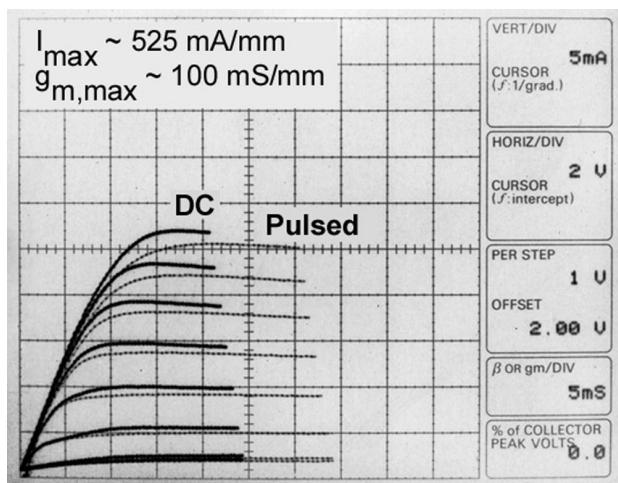


FIG. 5. I - V characteristics for devices fabricated on sample E ($L_g = 1.25 \mu\text{m}$, $L_{ds} = 3 \mu\text{m}$, and $w = 50 \mu\text{m}$).

stress measured at room temperature. It was found that (i) ~ 15 -nm-thick AlN layers were under compressive stress (3.4 ± 1.6 GPa), which corresponds to an in-plane strain of $\sim 0.7\%$; (ii) 25-nm-thick AlN films were found to be stress free within the accuracy of the curvature setup (0 ± 400 MPa); (iii) 180-nm-thick AlN films were under tensile stress (325 ± 140 MPa), even though the lattice mismatch between AlN and Si(111) is compressive by 20.4%. These results should be taken with caution since the AlN layers were not grown at the same time as the GaN films presented herein (samples A–E); several factors (e.g., oxidation of the AlN film) could affect the net stress in such thin layers. However, they suggest that the stress in the AlN layer is strongly affected by the thickness. We speculate that low dislocation densities in GaN can be obtained with either nearly stress-free or compressively stressed AlN buffers.

The results for sample E clearly show that using a thick graded layer instead of a thin AlN buffer layer results in compressive stress, as well as better FET characteristics, at the expense of higher threading dislocation density. We attribute the compressive stress in the GaN to compression in the AlGaIn graded layer. Because of the lattice mismatch between AlN and GaN the magnitude of the compressive stress increases with increasing thickness of the graded layer, that is, with decreasing aluminum composition. It is likely that the graded AlGaIn layers maintain a planar surface and grow in a layer-by-layer mode. Since there are no efficient mechanisms to introduce misfit dislocations in *planar* (0001) III-nitride films because the most common slip planes (0001) and $\{1\bar{1}00\}$ have no resolved shear stress, it is reasonable to expect that the AlGaIn graded layers have minimal relaxation during growth and subsequent GaN layers are highly compressed. Clearly, the magnitude of the compression decreases on cooling to room temperature due to the thermal expansion mismatch between the GaN and silicon. However, here we have shown that we can realize a larger compressive strain at the growth temperature than the thermal expansion mismatch strain (which normally gives tension for GaN grown on silicon substrates).

For electronic device applications the graded-buffer process offers the additional advantage of electrical insulation. Figure 5 shows that well-defined FET characteristics and reasonable power were achieved for sample E. Based on the TEM data (Fig. 1) the microstructure resulting from the graded-buffer approach is inferior to that obtained with the thin buffer approach [compare Figs. 1(b) and 1(c)]. However it has been shown that the dislocation density is not always the limiting factor in FET performance,^{42,43} such that acceptable performance can be expected for devices utilizing a two-dimensional electron gas.

V. CONCLUSION

We reported on GaN films grown on Si(111) using two nucleation schemes. Layers grown using a thin (~ 25 nm) AlN buffer were under tensile stress but exhibited dislocation densities as low as $1.1\text{--}3 \times 10^9 \text{ cm}^{-2}$. Films grown using a thick graded buffer sample exhibited a compressive stress of 270 MPa but a much higher dislocation density. Field-effect transistors fabricated using the thick graded buffer approach showed well-defined characteristics and reasonable saturated current (525 mA/mm).

ACKNOWLEDGMENTS

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