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The growth of N-face GaN by MOCVD: effect of Mg, Si, and In

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Abstract

The MOCVD growth of N-face GaN on Ga-face GaN has been investigated using excess Mg to produce inversion of the polar axis. Characterization of the inverted material by AFM, TEM, optical microscopy and chemo-mechanical polishing indicates complete inversion and the ability to grow N-face films. Growth of N-face GaN is accompanied by higher oxygen incorporation as well as higher background electron concentrations than growth of the Ga-face material under similar conditions. Silicon can be incorporated during the growth of the N-face material and acts as an n-type dopant as is the case for growth of the Ga-face. InGaN layers can be deposited on the N-face and exhibit typical emission in the 380–450 nm range with an additional emission in the red likely caused by defect levels in the InGaN. Conditions for growing high-quality inverted layers are discussed.

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1. Introduction

When GaN and its In and Al alloys are grown on sapphire and SiC substrates by MOCVD, the films have typically the (0001) orientation with the surface corresponding to the Ga-face of GaN. Because of this propensity to grow with the Gaterminated surface, there have been few studies of

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the growth on the N-face of GaN. Nevertheless, it has been demonstrated that $(0\ 0\ \overline{1})$ GaN can be grown by both MBE and MOCVD using suitable nucleation steps [1,2] and on small GaN platelets grown by high-pressure synthesis methods [3]. The resulting films have been reported to have rather poor surface quality and it has proved difficult to suppress the formation of hexagonal platelets and pyramidal surface morphologies. Although recent work has shown that N-face films can be readily chemo-mechanical polished to a superior surface finish than Ga-terminated films [4], control of the surface morphology during growth of N-face films

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is highly desirable. In this work, we describe the growth of GaN along the $[0\ 0\ 0\ \overline{1}]$ direction and the effect of Mg and Si dopants and alloys with In on the surface morphology of the films.

Both Mg and Si are commonly used as p- and ntype dopants, respectively, in the growth of (0001) GaN device structures. Mg is also of particular interest since it has been shown that under MBE growth conditions, the growth direction of GaN can be switched from the normal (0001) Ga-face orientation to the $(000\overline{1})$ N-face orientation [5]. Total energy calculations in that work predicted the formation of inversion domains when layers of Mg adatoms preferentially bonded to nitrogen causing a disruption in the stacking sequence. We have similarly found that the same growth direction inversion can be carried out under MOCVD conditions. Using the base inverted GaN:Mg layers, Si doping studies were performed. Experimental studies of Si doping along the polar [11] direction in GaAs growth by MBE has revealed amphoteric behavior of Si atoms depending on whether substitution occurs on the Ga or As site [6]. To date there have been few reports of controlled Si doping on the $(0\ 0\ 0\ \overline{1})$ N-face surface in nitrides to establish its electronic behavior.

In addition, growth on $(0\ 0\ 0\ \overline{1})$ planes leads to the incorporation of a higher concentration of oxygen. This has been suggested on the basis of density functional calculations of oxygen adsorption on GaN that showed a greater affinity for incorporation on the N-face than on the Ga-face [7]. Recent work by Ptak et al. [8] on oxygen doping using plasma-assisted MBE appears to confirm this finding.

2. Experimental details

MOCVD growths were performed with a closecoupled showerhead Thomas Swan reactor. Precursors of trimethylgallium (TMGa), trimethylindium (TMI), disilane, and ammonia were used during growth. Standard layers of GaN were grown at 1160°C with 99 μ mol/min of TMGa and 0.27 mol/min of NH₃ at atmospheric pressure, Mg doped inversion layers were grown at 1090°C

with flow rates of 99 and 0.13, respectively, at a pressure of 300 Torr. Growth conditions for In allovs will be discussed in later sections. A variety of techniques were used to characterize the crystallographic structure of the films, their surface morphology, and their photoluminescence properties. The polarity of growth was determined by convergent beam electron diffraction (CBED) studies carried out in a JEOL 2000 TEM. The samples were prepared using a FEI focused ion beam (FIB) to ensure uniformly thick TEM samples, a prerequisite for high-quality CBED patterns. Pattern matching with CBED simulations produced using EMS software¹ was used to confirm the crystallographic orientation of the films. Photoluminescence measurements were taken using pulsed excitation of a UV/Excimer laser at 248 nm from the KrF transition. A triple crystal X-ray diffractometer was used to measure rocking curve and peak positions with high resolution. Details of chemo-mechanical polishing with colloidal silica used to characterize nitrogen polar growth surfaces can be found elsewhere [4]. Hall measurements were taken at currents of 1 mA at room temperature with indium metal contacts.

3. Inversion of the GaN growth axis using Mg

Previous studies of Mg doped GaN films have revealed that both the structural and electrical properties of these films vary dramatically with very high doping levels. At incorporation levels of 5×10^{19} atoms/cm³ the surface morphology begins to degrade via the formation of hexagonal features including hexagonal hillocks and platelets. The electrical characteristics of these films also begin to suffer and the conductivity is reduced, perhaps indicative of second phase formation or amphoteric doping behavior [9]. Previous work has also associated heavily doped GaN:Mg films with the formation of inversion domains on the growth axis from (0 0 0 1) Ga-face to (0 0 0 $\overline{1}$) N-face [5].

To investigate possible conditions for forming uniformly inverted GaN films by MOCVD, we

¹P. Stadelmann, CIME-EPFL 2002, http://cimewww.epfl.ch/ people/stadelmann/jemswebsite/jems.html

grew Mg doped layers containing greater than 10²⁰ Mg/cm³ using low pressure (LP) conditions normally used in AlGaN growth. Under these conditions, the growth rate of GaN:Mg is about 50% that of standard atmospheric pressure (AP) GaN growth. In our experiments the Mg doped layers were deposited on MOCVD (0001) GaN templates, grown on sapphire with a typical low temperature GaN nucleation layer exhibiting rocking curve profiles and surface morphologies that have been discussed elsewhere [10]. The resulting LP-Mg-doped films have a dramatically different surface morphology and are covered with small hexagonal features. This morphology is shown in Fig. 1 using Nomarski imaging conditions to emphasize the height contrast. CBED TEM of the Mg-doped layers grown on GaN templates indicates that the polarity of the Mgdoped layer is reversed with respect to the underlying template. Simulated diffraction patterns, assuming a TEM foil thickness of 260 nm, confirm that the underlying GaN template is (0001) Gapolar, while the Mg-doped layer is $(000\overline{1})$ Npolar. Such a structure and the corresponding diffraction simulation is shown in Fig. 2 on a light emitting diode structure grown on the $(000\overline{1})$ Nface surface. This analysis also shows that the inverted polarity is preserved during doping and growth of multiple layers. Inversion of the growth axis was also indirectly confirmed by chemomechanical polishing. Previous work has revealed that only the nitrogen polar surfaces are removed during polishing [4], enabling this technique to check the polarity of the whole film surface by a comparison of the film thickness before and after polishing. An unintentionally doped (uid) GaN film grown on an inverted GaN:Mg layer was thinned by 0.75 µm after 10 min with colloidal silica in contrast to uid layers grown on a standard GaN template on which no thinning occurred under the same polishing conditions.

Atomic force microscopy (AFM) of the morphology of the N-face terminated samples revealed a similar stepped structure to those found on $(0\ 0\ 0\ 1)$ terminated surfaces, as documented by Weyher and co-authors [3]. The steps are crystallographically oriented. On an extremely fine scale there is evidence of considerable pitting,



Fig. 1. Nomarski optical images of Mg-doped GaN inversion layer grown at reduced pressure by MOCVD incorporating 10^{20} Mg/cm³. At higher magnification (b), the apex of the hexagonal half-platelets are seen to point towards the [1 0 $\overline{1}$ 0] direction of the GaN.

most likely the result of enhanced desorption during growth (see Fig. 3). Measurements of the X-ray rocking curves of nitrogen terminated samples reveal slightly wider rocking curve FWHM values as those observed on standard (0001) templates. For instance, the on-axis (000 $\overline{2}$) reflection had a FWHM of 375 arcsec, while the off-axis (10 $\overline{10}$) and (20 $\overline{20}$) peaks were 590 and 810 arcsec, respectively, which is slightly broader than the typical on and off-axis peak widths of 250 and 500, respectively. In plane X-ray measurements confirm the in-plane orientation dependence remains $[11\overline{20}]_{Al_2O_3}||[10\overline{10}]_{GaN}$ hence only the *c*-axis orientation is changed by Mg doping.



Fig. 2. A focused ion beam prepared cross-section bright field TEM image of an LED grown along the $[0\,00\,\overline{1}]$ direction. CBED patterns taken from various places in the crystal are shown as well as a simulated CBED pattern obtained from JavaEMS software. Layers marked inv are $(0\,00\,\overline{1})$ N-face terminated.



Fig. 3. AFM images of the surface of a $0.5 \,\mu$ m thick GaN:Mg film grown on a standard MOCVD template. The large image is a $100 \,\mu$ m² image with a Z range of 10 nm, the inset shows a $1 \,\mu$ m² image with a Z range of 5 nm. The inset was taken towards the center of a flat topped hexagonal platelet.

Taken together, the TEM, AFM, and large area polishing studies all indicate that the growth surface is uniformly $(0\ 0\ \overline{1})$ N-face terminated

after growth of a sufficiently thick GaN:Mg layer. Films with as little as 0.25 um thick GaN:Mg were found to be partially $(0\ 0\ 0\ \overline{1})$ terminated as shown in Fig. 2. However, it was observed that the thickness of the inversion layer has a significant effect on the roughness of the N-face growth following the initial Mg-doped layer. For inversion layers 0.25 µm thick, a very high density of small pyramidal hillocks was observed after N-face growth. As the thickness of this inversion layer was increased to 0.5 and 0.6 µm, the density of surface hillocks decreased to 1/mm². It is likely that thinner GaN:Mg layers may cause only partial inversion of the (0001) surface. During subsequent growth of GaN without Mg, a known surfactant [11], faceting of the surface related to the presence of inversion domains may be increased leading to the observed roughness.

4. Growth of Si-doped GaN and alloys with In in the *c*-axis direction

Previous studies of undoped GaN regrowth on the backside of polished $(000\overline{1})$ single crystals have revealed that a potential source of hillock formation is the presence of interfacial oxygen impurities [3]. In order to determine if this indeed played a role in the resulting surface morphology, an MOCVD growth on a GaN template was inverted using the technique described in Section 3 followed immediately by growth of GaN:Si under standard gallium face growth conditions, thus eliminating a potential source of oxygen contamination at a regrowth interface. The resulting surface improved dramatically and the density of hexagonal hillocks was reduced by orders of magnitude yielding surfaces with only occasional hillocks as recorded in Fig. 4. In addition to a general improvement in the overall growth morphology by a reduction in hillock density, the growth rate on the $(0\ 0\ 0\ \overline{1})$ surface approached that of standard Ga-face terminated surfaces in the presence of Si doping. Corresponding GaN:Si layers which were deposited on inverted N-face layers that had been removed from the growth chamber for study had a growth rate 44% lower than uninterrupted layers. It



Fig. 4. Nomarski images of $(0\ 0\ 0\ 1)$ terminated GaN:Si layers grown directly on GaN:Mg layers with and without interruption. Sample (a) was removed from growth chamber immediately after the GaN:Mg layer and was reinserted for the growth of the GaN:Si layer, sample (b) was not removed before the growth of the Si doped layer. A significant reduction in the number of hexagonal hillocks was observed with an accompanying increase of the growth rate, although the underlying hexagonal apex present after GaN:Mg growth can still be observed in (b), highlighted by the arrow.

appears that successful growth of N-face GaN relies on the minimization of ex situ sample processing as with other III–V layers and devices.

SIMS impurity profiles were conducted on specular $(000\bar{1})$ GaN layers in order to assess the incorporation rates of background oxygen in both (0001) Ga- and $(000\bar{1})$ N-face growth. Table 1 summarizes the incorporation of oxygen in various stages of growth and Fig. 5 shows the Table 1

Summary of SIMS detected oxygen concentration incorporated during growth, taken from the data shown in Fig. 5

Oxygen	Concentration (atoms/cm ³)	
(0001) GaN:Si	3×10^{16}	
(000Ī) GaN:Si	2×10^{17}	
(000Ī) GaN:Mg	6×10^{17}	



Fig. 5. Cs gun SIMS profile of impurities incorporated into a sample terminated with nitrogen polar GaN. The approximate sample structure is also shown below the profile for reference, see Fig. 6 for further details.

actual depth profile of a N-face terminated film doped with Si after inverting. As expected the O^{16} levels are considerably higher in the inverted layer doped with silicon as well as the Mg-doped layer which is grown at slightly reduced temperature [12]. However the concentration of oxygen in this layer is somewhat less than the order of magnitude rise expected on the basis of work done by Ptak et al, showing only a six-fold rise in oxygen incorporation in GaN:Si layers grown under the identical conditions [8].

An important question is whether silicon remains an n-type donor when films are grown along the $[000\overline{1}]$ direction. Five different test structures were grown to study the effect of silicon



Fig. 6. Structure grown to test electrical behavior of silicon doped GaN grown on N-face samples. Shading represents the region of inverted nitrogen polar growth.

Table 2 Summary of $(0 \ 0 \ 0 \ \overline{l})$ GaN Hall measurements at room temperature

Sample	Nominal Si concentration (10 ¹⁷ cm ³)	Measured n_e (10 ¹⁷ cm ³)	$\mu_{\rm e}~({\rm cm}^2/{\rm V}{\rm s})$
I	< 0.1	< 0.1	
II	< 0.1	1.2	132
III	5	6.7	240
IV	8	11	213
V	10	14	237

doping from uid to 15×10^{17} /cm³. The schematic sample structure is shown in Fig. 6. To minimize the formation of hillocks which appear at regrowth interfaces, (0001) GaN was first deposited to form a base 2 µm template and then inverted with a 0.6-0.75 µm GaN:Mg inversion layer as described above. Because of Mg doping memory effects, a 0.5 µm undoped buffer was then grown on each sample to allow the Mg concentration to return to baseline, as verified by earlier SIMS depth profiles. 0.5 µm GaN layers of varying silicon concentration were then deposited, except for sample I where the growth was terminated after deposition of the 0.5 µm thick undoped buffer. Doping levels and the measured carrier concentrations and mobilities are given in Table 2. The nominal concentration of silicon is based on a Ga-face reference sample grown under similar conditions. Sample I was too resistive at room temperature to obtain accurate Hall data due to the presence of compensating non-activated Mg doping. The extremely high resistivity of the $0.6\,\mu\text{m}$ inverted layer and the following $0.5\,\mu\text{m}$ buffer layer is sufficient to allow us to decouple any parallel conduction due to the substrate at room temperature. Further studies of the Hall effect at lower temperatures are currently underway to verify these observations. Each of the measured Hall samples is observed to have $1.7-4x10^{17}\,\text{cm}^3$ greater carrier concentration than the nominal Si doping levels expected on $(0\,0\,0\,1)$ terminated samples.

5. InGaN films on (0001)B terminated surfaces

InGaN films were grown on N-face GaN on both GaN:Mg and GaN:Si surfaces to examine the bulk properties and quantum well layers. InGaN layers were grown under flow rates of 7.7 µmol/ min at 845°C and 10.3 and 14.1 µmol/min at 910°C. The target layer thickness of the bulk InGaN was 5000 Å, quantum wells were targeted for thicknesses of 4 nm between barriers of 8 nm GaN. For comparison, growth rates of the InGaN layers on standard (0001) GaN surfaces would be expected to be 0.25 Å/s. Luminescence, X-ray, and TEM of the resulting material indicate a similar incorporation efficiency compared with Ga-face InGaN growth. However, in addition to typical In incorporation it was observed that InGaN growth on N-face GaN was prone to the formation of small polycrystalline islands under excess In precursor flow and low temperatures. TEM and energy dispersive X-ray analysis shows these small islands to be polycrystalline indium rich clusters without an epitaxial relationship to the substrate. An example of the morphology obtained during growth with In is shown in Fig. 7, and clearly shows small islands of In containing material uniformly distributed across the surface. Spatially resolved photoluminescence of this surface is shown in Fig. 8 for various optical excitation powers.

Despite the formation of these small polycrystalline InGaN features, In was also observed to incorporate as evidenced by measurements of the (0002) and (0004) X-ray diffraction peaks indicating lattice parameter change. For InGaN



Fig. 7. Surface morphology resulting from 2500 Å thick InGaN growth under excessive TMI flows on $(000\bar{1})$ surfaces taken with Nomarski polarized light. The dark specks are In-rich islands showing red luminescence.



Fig. 8. Micro-PL spectra of InGaN grown on N-face GaN under non-optimized conditions. Luminescence was stimulated with a continuous wave 514 nm Ar^+ at the various energies shown. A spectra taken over a region with no dots revealed only the Al₂O₃:Cr impurity transitions of the substrate at 690 nm.

films grown at $T_s = 845^{\circ}$ C with a trimethylindium (TMI) flow of 7.7 µmol/min, a (0004) diffraction peak is observed at 71.64°C with a corresponding broad luminescence peak centered at 489 nm. A second sample grown at $T_s = 910^{\circ}$ C with a TMI flow of 10.3 µmol/min had (0004) peak at 72.46°C and a corresponding broad luminescence centered at 418 nm. InGaN layers grown on Gaface samples under the same conditions would be expected to have similar peak emission wavelengths. Photoluminescence spectra from three different sample is shown along with the corresponding X-ray data in Fig. 9.



Fig. 9. (a) (0004) X-ray $2\theta/\omega$ scan on InGaN films deposited on (0001) GaN surfaces (b) room temperature PL measurements excited by a 248 nm pulsed UV/Excmier laser.

6. Discussion

The introduction of very high levels of Mg during MOCVD GaN growth appears to cause significant changes in film growth both structurally and electrically. As the Mg doping level is increased beyond 10^{20} /cm³, hexagonal features form on the surface accompanied by a dramatic increase in the film's resistivity. Inversion of the growth axis begins to occur under these conditions switching from a stable (0001) growth, to a (0001) growth mode. Films with as little as

 $0.25\,\mu m$ thick GaN:Mg were found to be partially $(0\,0\,0\,\overline{1})$ terminated. As the thickness of the GaN:Mg films was increased, a reduction in the number of hillocks and surface roughness was observed.

Samples which are removed from the growth chamber and exposed to atmosphere exhibited increased hillock density in conjunction with a slower growth rate during subsequent regrowth. Since hillock formation is thought to indicate (0001) inversion domains, minimizing their formation by limiting exposure to possible contaminants is an important consideration in nitrogen polar growth [3]. Furthermore, the high etching rates of $(000\overline{1})$ surfaces at growth temperature in the presence of hydrogen disallows the standard high temperature pretreatment used for many sapphire and GaN layers to ensure vicinal surfaces prior to growth and to remove impurities. Thus it appears that N-face GaN growth may be sensitive to interrupted growth processing much the same way other III-V materials are.

Apart from the structural characterization of Nface growth, analysis of the electrical properties and impurity incorporation reveals a six-fold increase in the rate of oxygen impurity incorporation along the $[0\ 0\ 0\ \overline{1}]$ direction. While our current studies did not attempt to minimize the additional oxygen incorporation observed, such a measure would obviously be desirable for future device work. Measurements of the silicon doping levels in nitrogen polar material appears to agree well with the observed behavior seen with gallium polar growth if the increased baseline carrier concentration can be attributed to the presence of higher background oxygen levels. Studies are presently underway to examine the conductivity behavior of samples at low temperature in order to extract information about the activation energy of Si and O as donors in $(000\overline{1})$ GaN. Preliminary results suggest there is little difference electrically between Si doping along the $[0\ 0\ 0\ 1]$ and $[0\ 0\ 0\ \overline{1}]$ directions in GaN in contrast to Si doping along the [1 1 1]Aand [1 1 1]B direction in GaAs [6].

InGaN structures grown on N-face GaN appears to grow similarly to their Ga-face counterparts. For a given temperature, incorporation of indium on either gallium or nitrogen polar surfaces

appears to be similar under the same precursor flow rates. A potential difference in the growth of InGaN structures, however, is the desorption of excess In. When large excess of indium precursors are introduced during growth for a given temperature, small polycrystalline clusters of material are observed uniformly decorating the surface. TEM data shows that these clusters are not epitaxial with the underlying layers, and spatially resolved photoluminescence indicates these clusters emit light at a peak wavelength of 640 nm. As the temperature of growth is increased or the injection of indium precursor is reduced, these polycrystalline clusters can be eliminated entirely. While the nature of the red emission remains ambiguous, it cannot be ruled out that the transition is simply defect level related.

7. Conclusion

MOCVD GaN films grown using Mg concentrations in excess of 10²⁰ cm³ have been shown to cause a complete inversion of the polar axis with the formation of a N-face GaN surface. CBED TEM confirms the inversion and is consistent with chemo-mechanical polishing behavior. AFM of MOCVD inverted surfaces reveals a step structure that is different from the step structures found on gallium terminated surfaces. In contrast to step morphology on the $(0\ 0\ 0\ 1)$ Ga surface, faceting and nanoscale pitting occurs, indicative of a difference in chemical potential on $(000\overline{1})$ surfaces. While the growth rate of heavily Mg doped layers is reduced by more than 50% with respect to (0001) surfaces, silicon doped layers can be grown at close to the same rate as films on (0001) surfaces. Obtaining smooth specular layers was found to be dependent on minimizing surface contamination and exposure to atmosphere.

Higher concentrations of oxygen were found in N-face growth compared with the Ga-face growth corresponding to higher background electron concentrations. Doping with Si was found to produce n-type GaN with doping efficiencies similar to those of Si doped (0001)Ga GaN films. Initial structural characterization of inverted films doped with either Mg or Si has not revealed significant differences in the dislocation content compared with gallium face growth as evidenced by TEM and X-ray characterization, but further studies are necessary to understand the microstructural influence of growth axis inversion in these films.

InGaN films have also been demonstrated on Nface surfaces with results consistent with growth on Ga-face material. Incorporation of up to 14% In was achieved using a temperature of 845°C and 7.7 µmol/min injection of TMI. Small polycrystalline clusters of InGaN were observed to form on the surface of growing films under non-ideal growth conditions. Micro-PL of these clusters reveals red luminescence at 640 nm.

While much clearly remains to be understood about growth along the $[0\ 0\ 0\ \overline{1}]$ axis in GaN and its alloys, the ability to grow high quality $(0\ 0\ 0\ \overline{1})$ GaN layers will allow for the investigation of novel polarization switched GaN based devices.

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