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Shawn D. Burnham

Gon Namkoong
Old Dominion University, gnamkoon@odu.edu

David C. Look

Bruce Clafin

W. Alan Doolittle

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Reproducible increased Mg incorporation and large hole concentration in GaN using metal modulated epitaxy

Shawn D. Burnham,^{1,a)} Gon Namkoong,² David C. Look,³ Bruce Clafin,³ and W. Alan Doolittle^{1,b)}

¹*School of Electrical and Computer Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332, USA*

²*Electrical and Computer Engineering Department, Old Dominion University, Norfolk, Virginia 23529, USA*

³*Semiconductor Research Center, Wright State University, Dayton, Ohio 45435, USA*

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The metal modulated epitaxy (MME) growth technique is reported as a reliable approach to obtain reproducible large hole concentrations in Mg-doped GaN grown by plasma-assisted molecular-beam epitaxy on *c*-plane sapphire substrates. An extremely Ga-rich flux was used, and modulated with the Mg source according to the MME growth technique. The shutter modulation approach of the MME technique allows optimal Mg surface coverage to build between MME cycles and Mg to incorporate at efficient levels in GaN films. The maximum sustained concentration of Mg obtained in GaN films using the MME technique was above $7 \times 10^{20} \text{ cm}^{-3}$, leading to a hole concentration as high as $4.5 \times 10^{18} \text{ cm}^{-3}$ at room temperature, with a mobility of $1.1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ and a resistivity of $1.3 \text{ } \Omega \text{ cm}$. At 580 K, the corresponding values were $2.6 \times 10^{19} \text{ cm}^{-3}$, $1.2 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, and $0.21 \text{ } \Omega \text{ cm}$, respectively. Even under strong white light, the sample remained *p*-type with little change in the electrical parameters. © 2008 American Institute of Physics.

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INTRODUCTION

Although Mg is the most successful *p*-type dopant for GaN, high hole concentrations have been limited by many complications, including a low solubility of Mg into GaN,¹ a tendency of Mg to accumulate and segregate at surfaces,² formation of pyramid-shaped defects,³ a memory effect of Mg in a growth chamber,^{4,5} a high vapor pressure of Mg at low temperatures,⁶ a low sticking coefficient of Mg on GaN, a deep ionization energy of Mg acceptors in GaN,⁷ unintentional hydrogen and oxygen doping,^{8,9} a significant compensation of Mg acceptors at high dopant concentrations,¹ and a drastic dependence of incorporation upon the growth regime or III-V ratio.^{10,11} As a consequence, there is a narrow window of growth conditions, which yield electrically active *p*-type GaN. Furthermore, even low or moderate hole concentrations are often not consistently obtained and are difficult to reproduce due to the Mg incorporation sensitivity to growth conditions. These complications result in large and varied resistances and mobilities in GaN-based devices that rely on *p*-type layers. If hole carrier concentrations were to be increased, and *p*-type doping of GaN standardized, these devices could benefit from better performance and better reliability.

Despite the complications associated with Mg-doped GaN, current state-of-the-art reports show promising results and give a reference for comparison. Mg incorporation as determined by secondary ion mass spectroscopy (SIMS) has been reported to be as high as $8 \times 10^{20} \text{ cm}^{-3}$ for metal or-

ganic chemical vapor deposition¹² and $3.5 \times 10^{20} \text{ cm}^{-3}$ for molecular beam epitaxy (MBE).¹³ However, above approximately $(2-3.5) \times 10^{20} \text{ cm}^{-3}$, the Mg incorporation begins to decrease with increased Mg flux¹³ and inverts Ga-polar films to N-polar.¹⁴ *M*-plane ($10\bar{1}0$) oriented substrates have been used to achieve a hole concentration as high as $p=7.2 \times 10^{18} \text{ cm}^{-3}$.¹⁵ Electrical characterization for current state-of-the-art films grown on *c*-plane substrates is shown in Table I.¹⁶⁻¹⁸ Reasonably high hole carrier concentrations are in the range of $(1-3) \times 10^{18} \text{ cm}^{-3}$. However, hole concentrations have been reported to be as high as $6 \times 10^{18} \text{ cm}^{-3}$,¹⁸ although the carrier concentration was metastable and significantly reduced upon heating during measurement.

The metal modulated epitaxy (MME) growth technique was developed to specifically address reproducibility issues.¹⁹⁻²² The MME growth technique is characterized by a group-III (metal) flux much higher than the group-V source, which would normally lead to droplets. However, the metal shutter is modulated to avoid droplet buildup and therefore build and deplete the metal bilayer on the growth surface, yielding smoother surfaces, larger grain sizes, and better reproducibility. The optimal period and duty cycle of the shutter modulation can be determined by monitoring reflection

TABLE I. Electrical characteristics of state-of-the-art Mg-doped GaN on *c*-plane substrates. (*) The hole concentration reported was metastable and was reduced by half upon annealing.

Hole concentration (10^{18} cm^{-3})	3	3	6*
Mobility ($\text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$)	9	2	0.3
Resistivity ($\Omega \text{ cm}$)	0.23	1.04	3.47
Reference	16	17	18

^{a)}Present address: HRL Laboratories, LLC, Malibu, CA 90265.

^{b)}Author to whom correspondence should be addressed. Electronic mail: alan.doolittle@ece.gatech.edu.

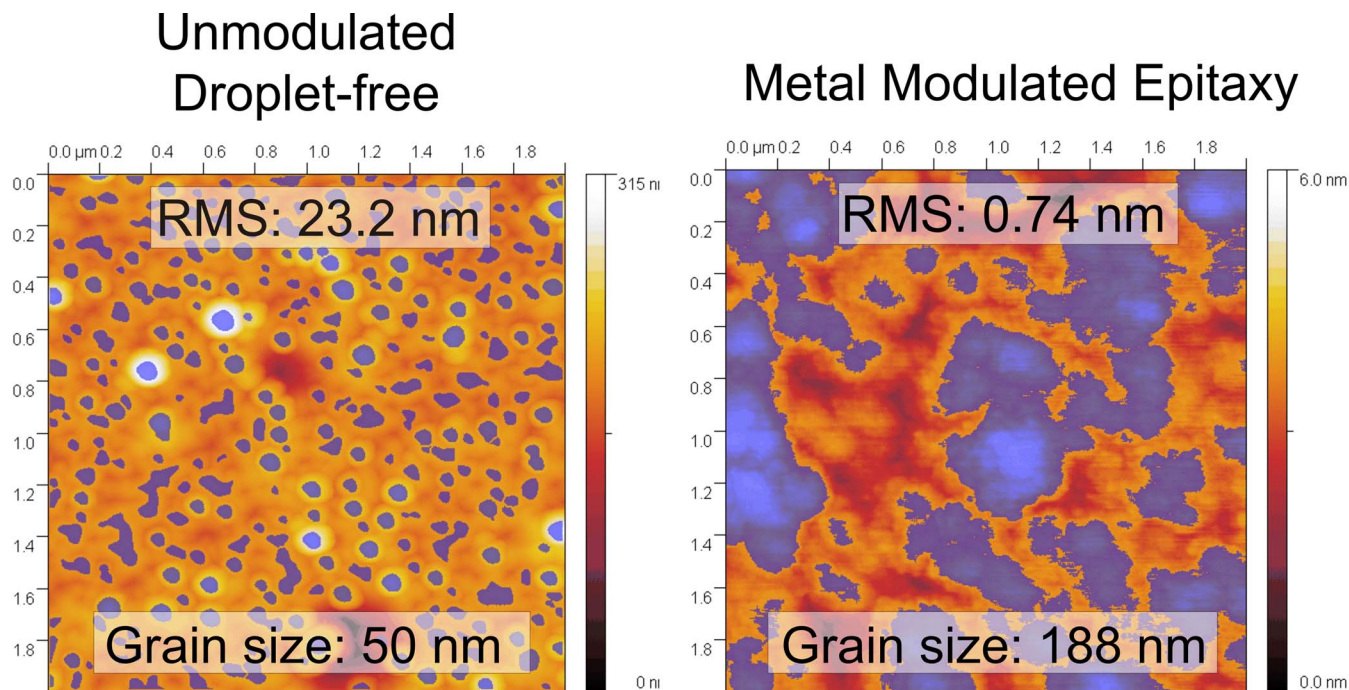


FIG. 1. (Color online) $2 \times 2 \mu\text{m}^2$ AFM scans of GaN samples grown using unmodulated droplet-free growth conditions and the MME growth technique, showing improved surface roughness and grain size as a result of metal-rich growth enhancing surface migration and lateral growth.

high energy electron diffraction intensity transients.²¹ However, until now the effects of a quantitative shutter modulation technique, such as MME, have only been reported for AlN.^{19–22} Other groups have used shutter modulation while growing GaN to improve intrarun surface uniformity, but the period and duty cycle were not experimentally determined, resulting in a significantly longer period than what was found necessary in the present work, and the effects on Mg-doped GaN were not discussed.²³ GaN surface morphology is similarly improved by the MME growth technique since it exhibits a laterally contracted metal bilayer much like AlN.^{24–27} Figure 1 shows $2 \times 2 \mu\text{m}^2$ atomic force microscopy (AFM) scans of a GaN sample grown using unmodulated droplet-free growth conditions compared to a sample grown using the MME growth technique. Both samples were droplet-free after growth and were grown with a substrate temperature of 550°C but used different Ga fluxes to avoid droplets with the unmodulated growth and to maintain droplet conditions with the MME growth. The height scale of each AFM data in Fig. 1 is different to exhibit grain size, and grains are highlighted. Surface rms roughness was reduced from 23 to 0.74 nm, or a factor of 31, and the average grain size was increased from 50 to 188 nm, or 276%, using the MME technique compared to unmodulated droplet-free growth. Furthermore, the percent variation of multiple GaN samples grown using the MME growth technique and same growth conditions was 18.7% for rms roughness and 1.57% for the average grain size, demonstrating the level of repeatability achieved with the MME growth technique. However, along with improved GaN morphology, Mg incorporation could also be affected by the shutter transitions. Therefore, this paper will report the results of Mg-doped GaN using the MME growth technique.

EXPERIMENTAL PROCEDURES

The presented epitaxial films were grown in a Riber 32 MBE system with traditional effusion cells for Ga and Al. The Mg source was supplied using a Veeco 200cc Corrosive Series Valved Cracker, as described elsewhere.^{10,28–30} A Veeco UNI-Bulb radio-frequency plasma was used for the nitrogen source with 350 W of input power and an input molecular flow rate of 1.3 SCCM (SCCM denotes cubic centimeters per minute at standard temperature and pressure). All epitaxial films in the present experiments were grown on [0001] basal *c*-plane oriented sapphire substrates, approximately $430 \mu\text{m}$ thick, with a polished growth surface and rough backside. Molybdenum was deposited on the rough backside to promote thermal absorption. To maintain high purity in the growth environment and minimize contamination, all samples were chemically cleaned to remove organics before entering the vacuum system. The samples were cleaned with a hot piranha etch, which consisted of a 4:1 mixture of $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$ heated to approximately 90°C for 10 min.³¹ They were rinsed with de-ionized (DI) water, dried with a nitrogen spray gun, and mounted onto a Veeco UNI-Block sample holder with custom-made molybdenum (Mo) retaining rings and spring plates. To further clean the samples they were then loaded into the high-vacuum introduction chamber, where they were outgassed at 700°C for 10 min.^{31,32}

To minimize the adverse effects of mismatch, low-temperature nitridation and high-temperature AlN buffer layers were grown before growing the GaN films.³³ Nitridation of the sapphire substrates was done at 200°C for 1 h.³⁴ The AlN buffer layer was grown using the MME growth technique²² with a substrate temperature of 800°C , an Al cell temperature of 1150°C [3.85×10^{-7} Torr, beam equiva-

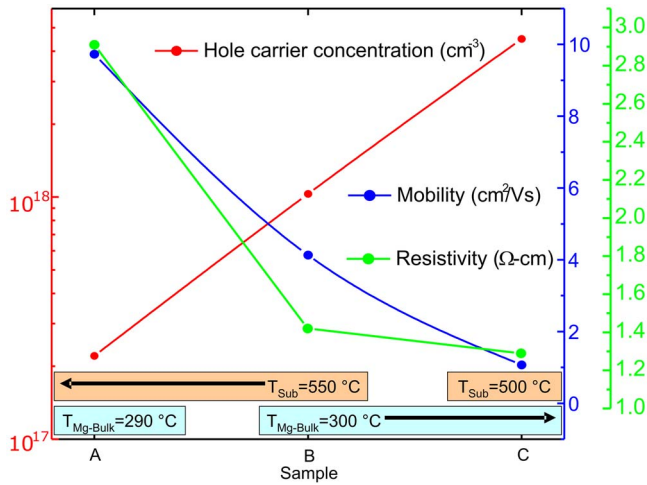


FIG. 2. (Color online) Electrical characterization values of Mg-doped GaN films. From left to right: film grown with 550 °C substrate temperature and 290 °C Mg bulk temperature, sample grown with the same substrate temperature but with a 300 °C Mg bulk temperature, and sample grown with the same Mg bulk temperature but with a 500 °C substrate temperature.

lent pressure (BEP)], and a modulation of 10 s for the shutter closed and open time. This resulted in a growth rate of approximately 0.5 $\mu\text{m}/\text{h}$. Therefore, for a target AlN buffer layer thickness of 100 nm, the AlN film was grown for approximately 12 min. For the electrically active Mg-doped GaN epitaxial films, the substrate temperature was between 500 and 550 °C. Furthermore, the Mg bulk temperature was between 290 and 300 °C. The Ga cell temperature was 990 °C (5.32×10^{-7} Torr, BEP), the Mg valve tip temperature was 900 °C, and the valve position was set to the open position of 100 mils, in the range of 0–300 mils for fully closed to fully open. To implement the MME growth technique, both the Ga and Mg shutters were modulated using simultaneous 10 s with shutters open and 10 s with shutters closed.

DISCUSSION

Electrical characterization of the electrically active films was done using the van der Pauw method for resistivity and Hall coefficient measurements,^{35,36} and the data are shown in Fig. 2. Hole carrier concentration ranged from 2.0×10^{17} to 4.5×10^{18} cm^{-3} , with corresponding mobility from 9.7 to 1.1 $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ and resistivity from 2.9 to 1.3 Ωcm . The average percentage variation of samples grown with the same target electrical properties was 7.7% for hole concentration, 23.6% for mobility, and 21.4% for resistivity. The higher hole concentration was achieved with a substrate temperature lower than that used for the other two Mg-doped GaN samples, which indicates that a lower substrate temperature allows incorporation of more Mg because of the high Mg vapor pressure. The unusually large hole carrier concentration of 4.5×10^{18} cm^{-3} can be ascribed not only to the low growth temperatures, but also to the improved crystal quality from the MME technique and an enhanced Mg incorporation by shutter modulation, described later. Furthermore, attempting to repeat the highest hole concentration resulted in remarkably good repeatability, even better than av-

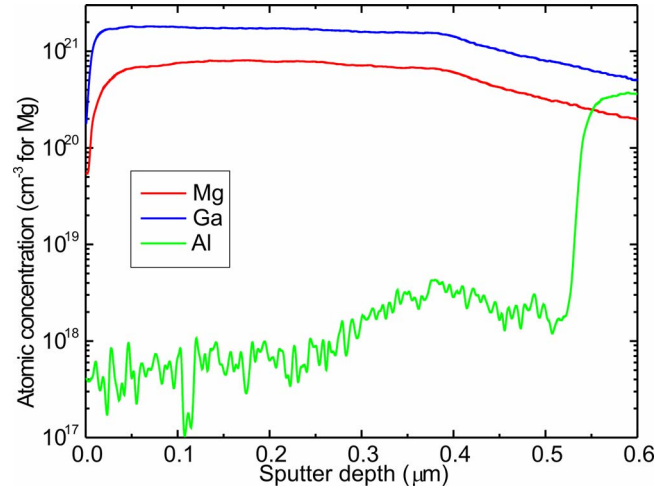


FIG. 3. (Color online) SIMS data for Mg-doped GaN sample with constant doping to illustrate uniform doping using the MME growth technique with high Mg incorporation. The average incorporation level of Mg is 7×10^{20} cm^{-3} . Mg, Ga, and Al signals mix at a depth of approximately 0.4 μm because of crater rounding at the film interface.

erages presented above. For samples grown two weeks apart, with Ga and Mg fluxes varied slightly, hole concentrations were 4.51×10^{18} and 4.42×10^{18} cm^{-3} , or less than 2% variation, regardless of sample thickness (0.38 and 0.52 μm , respectively). This enhanced repeatability is likely due to the incorporation limited growth dictated by the MME growth technique and the subsequent inability of Mg to build up to a surface concentration high enough to result in inversion of the polarity and reduction in incorporation. Further Hall coefficient measurements were taken at higher temperatures and at room temperature with variable illumination to observe the effect on electrical characteristics. At 580 K, the hole concentration was increased to 2.6×10^{19} cm^{-3} , with an associated mobility of $1.2 \text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ and resistivity of 0.21 Ωcm . Finally, strong white light produced very little change in the electrical parameters at room temperature. These results confirm the consistent *p*-type characteristic and nature of this material.

According to SIMS, enhanced Mg incorporation was demonstrated with a substrate temperature of 550 °C, and a Mg bulk temperature of 330 °C, although the Mg incorporation was much higher than that found for films with large hole concentrations. The resulting material with the highest Mg incorporation was highly resistive, beyond the measurement capability of the Hall measurement system. The uniformity of Mg doping is illustrated in Fig. 3, which shows the SIMS data for the bulk Mg-doped GaN sample with a consistent Mg incorporation level of approximately 7×10^{20} cm^{-3} , without abrupt Mg incorporation levels despite the shutter modulation approach. In the figure, the deviations at the extremities are due to non-steady-state surface effects³⁷ and the mixing of secondary ion species at the matrix change (substrate interface) at approximately 0.4 μm due to a nonflat crater bottom. In previous reports without the MME growth technique, Mg concentrations were not sustainable above approximately 3×10^{20} cm^{-3} but were decreased with further Mg exposure or increased Mg fluxes¹³ as a result of surface segregation effects.² It was found that

higher Mg fluxes eventually saturate the GaN surface with Mg, defining the saturation limit of Mg on GaN. As Mg continues to accumulate on the surface of GaN beyond the saturation limit, Ga-polar films eventually invert to N-polar GaN,¹⁴ which results in the reduction of Mg incorporation.² However, the MME growth technique results in sustained higher Mg incorporation levels, unlike that experienced with the traditional growth techniques.

This enhanced Mg incorporation effect of MME can be examined in the two distinct growth regimes below and above the Mg saturation limit of Mg and can be related to established theoretical models.³⁸ However, the established theoretical models are presently incapable of examining the lowest Mg concentration cases below the critical Mg flux because of computational limitations. Thus, the following analysis is based on phenomenological surface physics. Experimentally, below the saturation limit of Mg in GaN, the level of Mg doping is consistent between the unmodulated and MME growth techniques, and it is likely that all deposited Mg are either consumed or desorbed between MME cycles. However, at the saturation limit and above, where computational aids can be validated, there are drastic differences between the unmodulated and MME growth techniques. For Ga-polar material grown with N-rich conditions, it has been shown³⁸ that stable configurations of Mg in the second bilayer of the GaN crystal lattice form above 0.5 ML (monolayers) of Mg and that stable configurations exist without defect generation up to 1 ML Mg. However, in all of the stable configurations, the majority of Mg exists on the surface.³⁸ Furthermore, in the intermediate Ga-rich regime with a Ga bilayer, Mg atoms will replace Ga atoms in the bilayer to form Mg concentration in the metal bilayer. The resulting Mg concentration in the bilayer forms a stable configuration at the interface between the bilayer and the GaN film between 0.5 and 1 ML Mg, with Mg in the second bilayer of the GaN crystal lattice. In other words, negligible amounts of Mg are incorporated into GaN below 0.5 ML Mg, and while some Mg is incorporated into the GaN film during growth above 0.5 ML Mg, Mg also forms stable structures on the sample surface. Above 1 ML Mg, the local polarity is changed from Ga-polar to N-polar, and Mg incorporation is drastically reduced.

Therefore, a Mg adsorbed atom (adatom) is able to remain on the GaN surface in a stable configuration beyond the period of MME when Mg surface coverage is below 0.5 ML Mg. The Mg surface coverage will then continue to build up to higher levels with further cycles of MME until a coverage of 0.5 ML Mg is reached when some of the Mg will be incorporated into the film. The unincorporated Mg will remain on the surface in a stable configuration and continue to supply Mg to the growing GaN film, as with the case of the closed shutter with the MME technique, and the film will remain Ga-polar as long as the surface coverage does not reach or exceed 1 ML Mg. This would be indicated by a drastic reduction in Mg incorporation with the change to N-polar, which is not observed in Fig. 3, so it is believed that this limit is never exceeded in the current MME experiments. Therefore, the MME growth technique is able to maintain a sufficient Mg surface coverage to incorporate high Mg con-

centrations without exceeding the surface concentration limit and therefore inverting the polarity to N-polar. The ability of MME to maintain optimal growth conditions is a result of the shutter modulation approach, which allows Mg surface coverage to build between MME cycles and continuously incorporate at the optimal level.

CONCLUSION

The MME growth technique has been shown to improve the morphology of both AlN (Ref. 21) and GaN due to the increased adatom migration and improved lateral growth. For GaN grown with MME, grain size was increased to nearly 200 nm with percentage variation of less than 1.6%. The effect of the shutter modulation technique on Mg incorporation was observed for Mg-doped GaN. Consistent electrically active Mg-doped GaN was achieved with hole concentrations up to $4.5 \times 10^{18} \text{ cm}^{-3}$, with an average percentage variation of less than 8%. The SIMS analysis shows that Mg doping is uniform and consistent throughout the bulk of the film despite the transient shutter modulation, with concentrations up to $7 \times 10^{20} \text{ cm}^{-3}$. Previous studies on the energetics of Mg incorporation on GaN were used to explain the mechanism of increased and repeatable Mg incorporation and hole carrier concentration. With the increased hole carrier concentration and improved repeatability offered by the MME growth technique, GaN-based devices, which rely on *p*-type layers, can benefit from the increased performance and improved reliability.

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