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Growth rate independence of Mg doping in GaN grown by plasmaassisted MBE

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1. Introduction

Since nitride semiconductor structures were first grown, people have been looking for an efficient way to obtain p-type doping [1]. It quickly became clear that the best results could be obtain using magnesium (Mg) as a dopant. However, due to magnesium's high activation energy, only a small fraction of incorporated atoms was ionized [2]. Furthermore, when metal organic vapor phase epitaxy (MOVPE) is used for the growth of magnesium doped Ga(In, Al)N layers, neutral Mg-H-V_N complexes are formed due to a high hydrogen background pressure. These complexes need to be decomposed by either electron irradiation [3] or annealing [4] to obtain p-type doped nitride layers. Further studies on p-type doping in these technique also reported a different Mg incorporation for different Al compositions in $Al_xGa_{1-x}N$ [5], where higher Mg incorporation was followed by the formation of Mg-rich clusters.

An alternative growth technique for the growth of nitrides is plasma-assisted molecular beam epitaxy (PAMBE). Experimental work done on understanding the growth peculiarities in PAMBE enabled the growth of laser-diode-grade nitride structures from

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ABSTRACT

Doping of Ga(Al)N layers by plasma-assisted molecular beam epitaxy in Ga-rich conditions on c-plane bulk GaN substrates was studied. Ga(Al)N samples, doped with Mg or Si, grown using different growth conditions were compared. In contrast to Si doped layers, no change in the Mg concentration was observed for layers grown using different growth rates for a constant Mg flux and constant growth temperature. This effect enables the growth of Ga(Al)N:Mg layers at higher growth rates, leading to shorter growth time and lower residual background doping, without the need of increasing Mg flux. Enhancement of Mg incorporation for Al containing layers was also observed. Change of Al content from 0% to 17% resulted in more than two times higher Mg concentration.

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UV to cyan light spectrum [6]. Moreover, this technique offers two extremely interesting advantages in the growth of p-type layers. First is that due to low hydrogen background pressure, no activation of p-type layers is necessary. Second is that applying metalrich conditions during the growth, which is preferred in PAMBE, opens a possibility to grow smooth Ga(In,Al)N:Mg layers at considerably lower growth temperature (T_c) than that used in MOVPE. This approach solves one of the major difficulties in the growth of long wavelength laser structures which is the growth of ptype layers on top of the active region without decomposing it [7]. Up to now, lots of work has been devoted to explore the dependence of Mg doping on T_G [8,9] but little is known about the influence of other growth parameters and alloys composition on dopants incorporation. Recent progress in plasma sources has enabled the PAMBE growth of nitrides with growth rates as fast as 10 µm/h [10] or 7.6 µm/h [11] sustaining high crystal quality but still little is known about the impact of growth rate (V_{gr}) on the doping of crystals grown in PAMBE.

In this paper we study the influence of growth rate on Mg and Si incorporation in Ga(Al)N alloys grown by PAMBE.





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2. Experimental

Samples were grown in a custom-designed Veeco Gen20 equipped with two Mg cells. Ga and Al atoms were provided by standard sumo cells while active nitrogen is supplied by a Veeco RF plasma source. Each Mg cell had an aperture plate located at the end of its crucible which was unique for each cell. Therefore, when both cells are at the same temperature, the Mg fluxes are not equivalent. Beam equivalent pressure (BEP) as a function of operation temperature for both cells is presented in Fig. 1. From this point on all fluxes will be given in units of equivalent growth rate, assuming a sticking coefficient of 1. The active nitrogen flux, Φ_{N} , supplied by the RF plasma source was varied between 0.3 μm/h and 1.5 μm/h. The Mg flux was estimated using BEP measured by ion gauge prior to the growth by comparing it with two neighboring effusion cells (indium and gallium) taking into account different operating temperature and element-specific parameters [12]. We expect that such procedure leads to about 50% accuracy. Investigated samples were grown on bulk GaN substrates obtained by Hydride Vapor Phase Epitaxy with offcut angle equal to 0.8 degree towards m-plane and threading dislocation density 10⁶–10⁷ cm⁻². Ga(Al)N layers were grown using galliumrich conditions where the presence of excess of gallium was controlled with laser reflectometry. T_G was kept constant at 750 °C and monitored using Ga desorption from GaN surface measured before the growth [13]. The Ga desorption rates, as a function of real substrate temperature, were calibrated separately on 2 inch GaN/Al₂O₃ reference wafer using k-Space Bandit pyrometer. The composition of the Ga(Al)N:Mg layers was measured using secondary ion mass spectrometer (SIMS). Investigated samples consisted of series of 50 nm thick Ga(Al)N layers doped with increasing levels of Mg. A series of Si doped GaN layers were also prepared for comparison. Layers compositions and growth parameters were chosen to be kept in a range that was used for the growth of laser structures operating in visible light spectrum [6].

3. Mg content as a function of Mg flux

As it has been presented by many authors [14–18], two different regimes of Mg incorporation into GaN crystal can be identified. In the first regime, the increase in Mg flux leads to higher Mg content in the crystal and a subsequently higher hole concentration. In the second regime, higher Mg flux leads to either a saturated Mg concentration [15,16] or the drastic decrease in hole concentration caused by crystal quality degradation possibly related with polarity inversion and/or point defects formation [18]. To assure high quality of layers investigated in this paper, a calibration sample was grown using a constant growth rate, $V_{\rm gr}$, (determined by nitrogen flux) of 0.4 μ m/h where only Mg flux was varied and all other growth parameters were kept constant. The sample consisted of repeated stacks of GaN:Mg2/Al_{0.08}Ga_{0.92}N:Mg1/GaN:Mg1 layers where two Mg cells (Mg1 and Mg2) at different temperatures were used to dope the two GaN side layers and the sandwiched AlGaN layer. SIMS data obtained from the sample is presented in Fig. 2. Steady increasing of the Mg concentration for GaN:Mg and Al_{0.08}-Ga_{0.92}N:Mg layers with increasing Mg flux is observed. Higher temperatures for Mg1 (comparing to Mg2) cell result in lower flux due to the aforementioned faceplates. In later experiments, only the higher flux cell, Mg2, is used. It is also possible to see that for each Al_{0.08}Ga_{0.92}N:Mg layer the Mg concentration is slightly higher than that of the GaN:Mg layer grown using a Mg cell operating at the same temperature.

The Mg concentration extracted from SIMS, for Mg2, is presented in Fig. 3. While not shown, the Mg concentration using Mg1 followed the same trend as Mg2. It is important to point out



Fig. 1. Magnesium beam equivalent pressure (left-logarithmic axis) and flux in microns per hour (right-logarithmic axis) as a function of Mg cell temperatures for two different cells with different apertures.

that SIMS measurements were carefully calibrated to account for different etching rates for GaN and AlGaN lavers of different compositions, so the result presented here shows higher Mg incorporation into AlGaN layers. Fig. 3. shows that, while fixing all other growth parameters, Mg content, in both AlGaN and GaN, can be changed using different Mg fluxes and can reach ~10¹⁹ atom/cm³ for Mg1 cell temperature 440 °C. More interestingly, higher Mg incorporation for Al_{0.08}Ga_{0.92}N, compared to GaN, was observed for all used Mg cell temperatures. It is important to point out here that Mg incorporation efficiency, defined as an amount of Mg incorporated into the crystal divided by impinging Mg flux, for the growth presented in Fig. 2 can be approximated to be below 1% [19]. Such low efficiency is due to the use of a relatively high growth temperature and can be easily enhanced by lowering T_G. However, as was pointed out by several authors [14,15,18], keeping T_G high results in lower unintentional doping. That is why in the present work we concentrate on the mechanism of Mg incorporation at high T_G where strong Mg desorption and/or low sticking coefficient for Mg is present during growth.

4. Mg content as a function of growth rate

To show the counterintuitive effects of the growth rate on the incorporation of Mg into GaN layers at high T_G, a simple experiment was done. Three sets of GaN layers were grown with each set consisting of three 50 nm layers. For each set, a different nitrogen flux was used in each layer, ranging from 0.36 to 1.5 μ m/h, but the dopant flux was held constant. For all layers the gallium flux was adjusted to keep the growth conditions in the Ga-rich regime. One set of layers was grown for reference using the Si cell set to 1150 °C, while the other two sets were doped with Mg fluxes of 0.005 $\mu m/h$ and 0.013 $\mu m/h$ (resulting in 2.5 \cdot 10 18 atom/cm 3 and $7 \cdot 10^{18}$ atom/cm³ dopant concentrations for 0.4 μ m/h of nitrogen flux, as obtained in Fig. 3). Resulting dopant concentrations as a function of inversed V_{gr} are presented in Fig. 4. Silicon content in grown layers changes linearly with inverse growth rate (marked with red¹ dotted line in Fig. 4(a)). Such behavior is expected and can be simply explained by assuming that the whole Si flux, Φ_{Si} , is incorporated at the T_G used and, since Si atoms interchange Ga atoms, the Si content follows a Φ_{Si}/Φ_N relation. Mg content, on the

 $^{^{1}\,}$ For interpretation of color in Fig. 4, the reader is referred to the web version of this article.

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