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On electrical analysis of Al-rich p-AlGaN films for III-nitride UV light emitters

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Abstract

In this work, an alternative scheme to estimate the resistivity and ionization energy of Al-rich p-AlGaN epitaxial films is developed using two large-area ohmic contacts. Accordingly, the resistivities measured using current–voltage measurements were observed to corroborate the Hall measurements in the Van der Pauw configuration. A free hole concentration of $\sim 1.5 \times 10^{17} \text{ cm}^{-3}$ and low ionization energy of $\sim 65 \text{ meV}$ in Mg-doped $\text{Al}_{0.7}\text{Ga}_{0.3}\text{N}$ films is demonstrated. Nearly an order of magnitude lower hydrogen concentration than Mg in the as-grown AlGa_xN films is thought to reduce the Mg passivation and enable higher hole concentrations in Al-rich p-AlGa_xN films, compared to p-GaN films. The alternate methodology proposed in this work is expected to provide a simpler pathway to evaluate the electrical characteristics of Al-rich p-AlGa_xN films for future III-nitride ultraviolet light emitters.

Keywords: III-nitride, Mg-doped AlGa_xN, ionization energy, mobility, contact resistance

(Some figures may appear in color only in the online journal)

1. Introduction

Al-rich AlGa_xN has demonstrated applicability for ultraviolet (UV) light emitters used in applications like water sterilization, UV curing, biomedical instrumentation, etc [1, 2]. These emitters require technologically relevant n-type and p-type resistivities in Al-rich AlGa_xN films for efficient injection of electrons and holes into the quantum well(s), where they recombine. While a low resistivity of $<1 \text{ } \Omega \text{ cm}$ in Al-rich n-AlGa_xN ($>60\%$ Al mole fraction) films has been widely reported [3–5], achieving a desirable resistivity of $<10 \text{ } \Omega \text{ cm}$ in Al-rich p-AlGa_xN films is still a major challenge [6–8]. High resistivity in p-AlGa_xN has been primarily attributed to

the high acceptor ionization energy (E_A) of Mg, low solubility of Mg leading to its incorporation in electrically inactive sites and relatively low formation energy of compensating defects such as nitrogen-vacancies and Mg-nitrogen vacancy complexes [9]. The value of E_A is believed to increase linearly with Al mole fraction from $\sim 120\text{--}200 \text{ meV}$ ($x = 0$) [10–12] to $\sim 510 \text{ meV}$ ($x = 1$) [6] in p-Al_xGa_{1-x}N. The resulting low free hole concentration at room temperature (RT) leads also to a poor ohmic contact formation and a significant power loss during the device operation [13]. To overcome this difficulty imposed by high E_A in p-AlGa_xN films, a thin p-GaN layer is typically used for p-side ohmic contact formation in III-nitride UV light emitters [14, 15]. This technique not only introduces processing complexity but also promotes re-absorption of emitted light (in the p-GaN layer due to a lower bandgap), thereby reducing the external quantum efficiency

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[16]. Thus, a direct ohmic contact formation to the Al-rich p-AlGa_{0.7}N epitaxial films is necessary to achieve highly efficient UV light emitters. This necessitates the realization of Al-rich p-AlGa_{0.7}N with a low E_A that would result in a high free carrier concentration [13].

Interestingly, a resistivity of 47 Ω cm and a low ionization energy of <100 meV was reported by Kinoshita *et al* [17] for p-Al_{0.7}Ga_{0.3}N by heavy doping with Mg ($\sim 6 \times 10^{19}$ cm⁻³). The lower ionization energy was tentatively attributed to an alternate conduction mechanism such as hopping conduction and shows a pathway to achieve higher conductivity in p-AlGa_{0.7}N at higher Mg doping concentrations as long as the compensation is reduced. Two modified approaches have been recently developed to achieve a higher free carrier concentration in Al-rich p-AlGa_{0.7}N films. These include the distributed polarization doping technique, which uses a linearly, compositionally graded Mg-doped AlGa_{0.7}N layer [18–20], and the short period superlattice technique, which uses superlattice structures consisting of alternate p-Al_xGa_{1-x}N/p-Al_yGa_{1-y}N ($0 \leq x \leq 1$, $0 \leq y \leq 1$, $x \neq y$) layers [21, 22]. These techniques demand precise control over thickness and composition in the AlGa_{0.7}N epitaxial films. A few reports have highlighted the possibility of achieving a low E_A in Mg-doped bulk Al-rich p-AlGa_{0.7}N films [17, 23]. However, poor hole mobility, due to alloy scattering in Al-rich p-AlGa_{0.7}N [24], along with poor ohmic contacts restricted the determination of E_A using the traditional DC Hall measurement due to low Hall voltages.

In this work, we report on the observation of low acceptor ionization energy of ~ 65 meV in Mg-doped p-Al_{0.7}Ga_{0.3}N epitaxial films measured using AC Hall measurements. A simpler methodology (using two large area Ni/Au contacts) to estimate the E_A in Al-rich p-AlGa_{0.7}N films using temperature-dependent current–voltage (I – V) measurements is shown to corroborate the Hall measurements. The benefit of a significantly larger change in the temperature dependence of free hole concentration as compared to the hole mobility is shown to be effective in determining the E_A using I – V measurements.

2. Materials and methods

Mg-doped p-Al_{0.7}Ga_{0.3}N films were grown on sapphire substrates using low-pressure metalorganic vapor phase epitaxy (LP-MOVPE) [25, 26]. The reactor pressure was kept constant at 20 Torr throughout the growth. The growth consisted of a 400 nm thick AlN layer, followed by a 600 nm Mg-doped p-Al_{0.7}Ga_{0.3}N layer. Trimethylaluminum, triethylgallium, and ammonia gas were used as the Al, Ga, and N precursors, respectively. The film was doped with Mg using Bis-cyclopentadienyl-magnesium (Cp₂Mg) to a doping level of $\sim 2 \times 10^{19}$ cm⁻³. The p-Al_{0.7}Ga_{0.3}N films were grown at a temperature of 1100 °C in H₂ diluent/carrier gas with a total reactor flow rate of 11 slm. The V/III ratio was kept at 2000 to minimize the incorporation of V_N³⁺ self-compensating defects in p-Al_{0.7}Ga_{0.3}N [17]. After the growth, the Mg dopant activation anneal was performed either in air or N₂ ambient. Few

samples were not annealed, whereas the dopant activation temperature used for other samples used in this work will be discussed later. All the activation anneals were carried out for 20 min. Anneals under air ambient were carried out inside a tube furnace, whereas anneals under N₂ ambient were carried out inside the LP-MOVPE chamber.

For the large area ohmic contact formation, the activated p-Al_{0.7}Ga_{0.3}N wafer was cleaned using acetone, methanol, and DI water, followed by dipping in 1% HF and hot 1:1 HCl:H₂O solutions [13]. Conventional photolithography was used to pattern the contacts on p-Al_{0.7}Ga_{0.3}N layers, followed by the metal deposition and lift-off. Standard Ni (20 nm)/Au (40 nm) contacts were deposited using ultra-high vacuum e-beam evaporation (base pressure: 1×10^{-9} Torr). The contacts were annealed at 600 °C for 10 min in air ambient. The contact geometry consisted of an infinite area contact, a known diameter large-area contact, and a known separation between the contacts. In this work, the diameter of the large area contact and the separation between the contacts were chosen to be 600 μ m and 100 μ m, respectively. For AC Hall measurements, Ni/Au contacts were deposited and annealed on a square-shaped sample using the Van der Pauw geometry. AC Hall measurements were performed in an 8400 series LakeShore AC/DC Hall measurement system using a magnetic field and excitation field frequency of ~ 0.62 T and 100 mHz, respectively.

For comparing the E_A in p-Al_{0.7}Ga_{0.3}N with p-GaN, a p-GaN reference sample having a similar Mg doping concentration ($\sim 2 \times 10^{19}$ cm⁻³) was also grown on a sapphire substrate. Further details about the p-GaN growth and surface cleaning procedures can be found elsewhere [10]. Standard Ni/Au contacts with geometry similar to the p-Al_{0.7}Ga_{0.3}N were formed on p-GaN for I – V (large area contacts) and Hall measurements (Van der Pauw geometry). Temperature-dependent I – V measurements were performed using Keithley 4200 semiconductor parameter analyzer. Calculations and curve fittings were carried out using MATLAB software. Doping levels, oxygen and carbon impurity concentrations were measured using secondary ion mass spectroscopy (SIMS) performed by EAG.

3. Results and discussion

The variation of free hole concentration and hole mobility in p-Al_{0.7}Ga_{0.3}N and the p-GaN reference sample were measured using Hall measurements, as shown in figure 1(a). A RT free hole concentration of $\sim 1.5 \times 10^{17}$ cm⁻³ is obtained in p-Al_{0.7}Ga_{0.3}N, which increases to $4\text{--}5 \times 10^{17}$ cm⁻³ at temperatures of actual light emitter operation (~ 100 °C) [10]. The variation of free hole concentration with temperature (T) in p-GaN is observed to show a larger slope compared to the p-Al_{0.7}Ga_{0.3}N. The free hole concentration (p) was fitted using the following equation to extract E_A [10]:

$$\frac{p(p + N_D)}{N_A - N_D - p} = \frac{N_V}{g} \exp\left(-\frac{E_A}{kT}\right), \quad (1)$$

where N_D is the compensating donor concentration, N_A is the Mg doping concentration, N_V is the effective density of

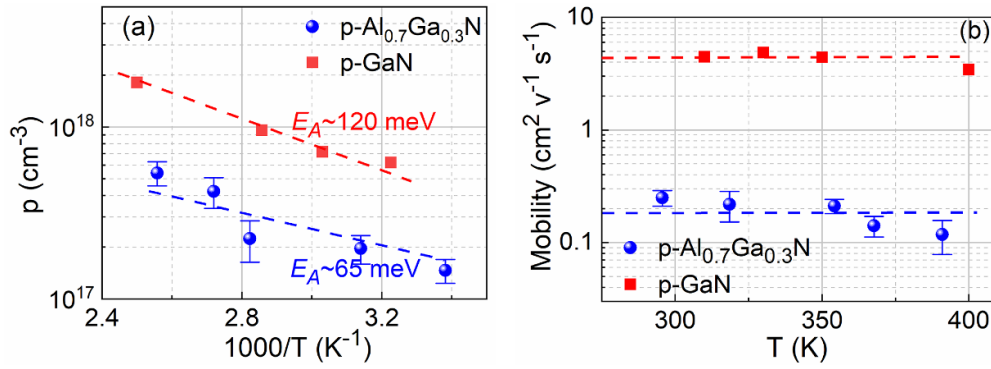


Figure 1. The variation of (a) free hole concentration and (b) mobility in p-GaN and p-Al_{0.7}Ga_{0.3}N layers as observed using Hall measurements.

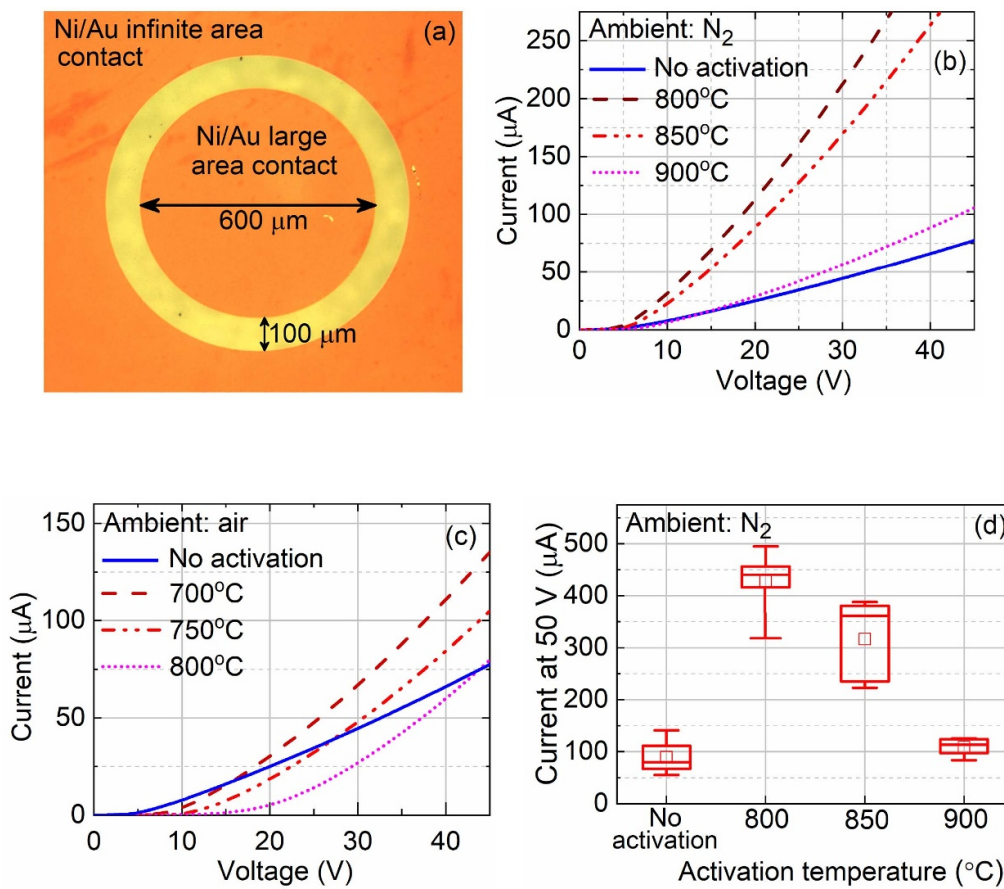


Figure 2. (a) Contact geometry used to determine E_A in p-Al_{0.7}Ga_{0.3}N; (b), (c) I - V characteristics of Ni/Au contacts to p-Al_{0.7}Ga_{0.3}N for different activation anneal schemes, and (d) current at 50 V through contacts fabricated across the wafers either non-activated or activated under N₂ ambient.

states in the valence band, g is the acceptor degeneracy factor ($g = 4$ for holes) and k is the Boltzmann constant. The ionization energy for Mg in p-GaN reference wafer was observed to be $\sim 120 \pm 5$ meV, closely resembling the previously reported values for p-GaN [11, 12]. In contrast, Mg doping in p-Al_{0.7}Ga_{0.3}N showed an E_A of $\sim 65 \pm 8$ meV for a similar temperature range. The corresponding values of N_D derived using the least-square error technique were observed to be $\sim 1.5 \times 10^{19}$ cm⁻³ and $\sim 5 \times 10^{18}$ cm⁻³ in p-AlGaN and p-GaN, respectively. The mobility follows a rather shallow trend

with temperature change, as observed in figure 1(b). From RT to ~ 85 °C, the mobility is observed to be nearly constant, while the hole concentration changes significantly. Therefore, the variation in resistivity with temperature is primarily caused by the change in free hole concentration in the epitaxial films. This indicates that the variation in sheet resistivity observed in I - V measurements can also be used to estimate the acceptor E_A . To verify this hypothesis, the resistivity was recorded using I - V measurements using the contact geometry shown in figure 2(a). Two large area ohmic contacts were fabricated

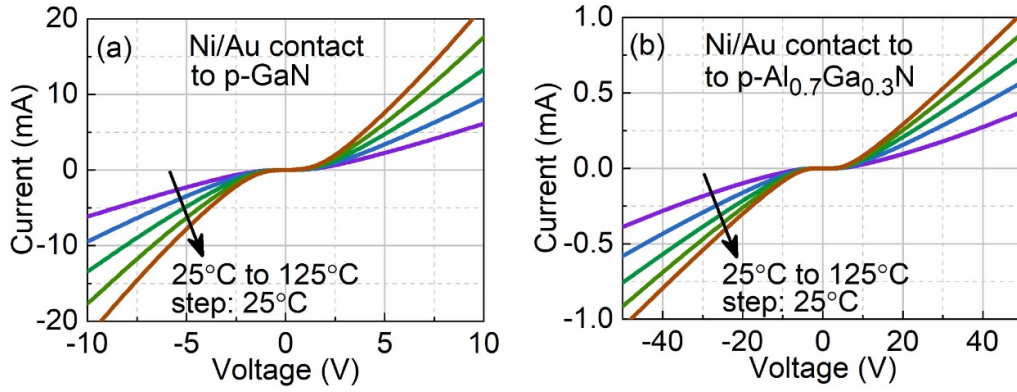


Figure 3. I - V characteristics of Ni/Au contact to (a) p-GaN and (b) p-Al_{0.7}Ga_{0.3}N having the contact geometry shown in figure 2(a).

on the p-GaN and p-Al_{0.7}Ga_{0.3}N to ensure low contact resistance.

Figures 2(b) and (c) shows the I - V characteristics of standard Ni/Au ohmic contacts to p-Al_{0.7}Ga_{0.3}N for different activation schemes. Unlike p-GaN, p-Al_{0.7}Ga_{0.3}N films showed reasonable current flow through the Ni/Au contacts even without activation anneal, as observed in figure 2(b). An increase in the current was observed after an activation anneal at 800 °C under N₂ ambient. Further increase in the activation anneal temperature in N₂ ambient resulted in a lower current through the contacts, likely due to Mg self-compensation or formation of vacancy-related complexes [9]. On the contrary, a conventional activation scheme typically performed for p-GaN (600 °C–700 °C in air ambient) was observed to result in resistive I - V characteristics for p-Al_{0.7}Ga_{0.3}N, shown in figure 2(c).

The nonlinear threshold-type I - V characteristics can be attributed to the commonly observed potential barrier at the metal-semiconductor interface [13]. Up to a certain turn-on voltage ($V_{\text{turn-on}}$), the current through the metal-semiconductor interface is restricted by the high contact resistance arising due to the barrier potential. However, once applied voltage exceeds $V_{\text{turn-on}}$, the Ni/Au contact starts conducting and the current is mostly limited by the sheet resistance of the semiconductor. A higher $V_{\text{turn-on}}$ observed for air annealed p-Al_{0.7}Ga_{0.3}N may be due to the formation of a thin AlO_x layer after the activation anneal. The observed increase in $V_{\text{turn-on}}$ at higher air anneal temperatures (in air ambient) further reaffirms the possibility of forming AlO_x. The variations in current through the contacts in different areas of the wafer either without activation anneal or activation anneal under N₂ ambient are highlighted in figure 2(d). In general, p-Al_{0.7}Ga_{0.3}N film that had an activation anneal at 800 °C under N₂ ambient showed a higher current compared to any other activation anneal schemes. Thus, the activation anneal for p-Al_{0.7}Ga_{0.3}N was fixed at 800 °C for 20 min in N₂ ambient for the remaining analyses.

Temperature-dependent I - V characteristics of Ni/Au ohmic contact to the p-GaN and p-Al_{0.7}Ga_{0.3}N samples having the large area contact geometry are shown in figure 3. Temperature range was limited to ~400 K so that mobility remains nearly constant (see figure 1(b)). Significantly

higher hole mobility in p-GaN results in lower sheet resistance and higher current values. However, a higher $V_{\text{turn-on}}$ at the metal/p-Al_{0.7}Ga_{0.3}N interface indicates a larger barrier potential. At an applied voltage much larger than the $V_{\text{turn-on}}$, the differential resistance (R_{Diff}) from the I - V characteristics at different temperatures can be written as:

$$R_{\text{Diff}} = R_{\text{CI}} + R_{\text{CL}} + R_{\text{Semi}}, \quad (2)$$

where R_{CI} , R_{CL} , and R_{Semi} are the contact resistance of infinite area outer contact, large area inner circular contact, and the semiconductor sheet resistance of p-GaN or p-Al_{0.7}Ga_{0.3}N, respectively. The values of R_{Diff} for the p-GaN and p-AlGa_{0.3}N films were calculated from I - V curves in the range 5–10 V and 40–50 V, respectively. Since the contacts occupy a large area, the $(R_{\text{CI}} + R_{\text{CL}})$ term of equation (2) can be considered much lower than the R_{Semi} . Thus, R_{Diff} can be used to estimate the resistivity of the semiconductor at different temperatures using the relation [27]:

$$\rho_{I-V} = \frac{R_{\text{Diff}} t_{\text{epi}} F}{C}, \quad (3)$$

where ρ_{I-V} is the semiconductor resistivity (in Ω cm) to be calculated from the temperature-dependent I - V characteristics, t_{epi} is the thickness of the semiconductor epitaxial film, F is the area factor, and C is a correction factor given as:

$$F = \frac{2\pi r_s}{d} \quad (4)$$

$$C = \frac{r_s}{d} \ln \left(\frac{r_s + d}{r_s} \right) \quad (5)$$

where r_s is the radius of the inner circular contact and d is the contact separation. For the geometry used in this work, $r_s \approx 330$ μm , and $d \approx 100$ μm , the values of C and F were found to be ~0.9 and ~18.7, respectively. The resistivities of p-GaN and p-Al_{0.7}Ga_{0.3}N as calculated from the I - V characteristics using equation (2) and as measured by Hall are shown in figure 4(a).

The resistivities of p-GaN and p-Al_{0.7}Ga_{0.3}N measured in the Van der Pauw geometry and calculated from the I - V curves

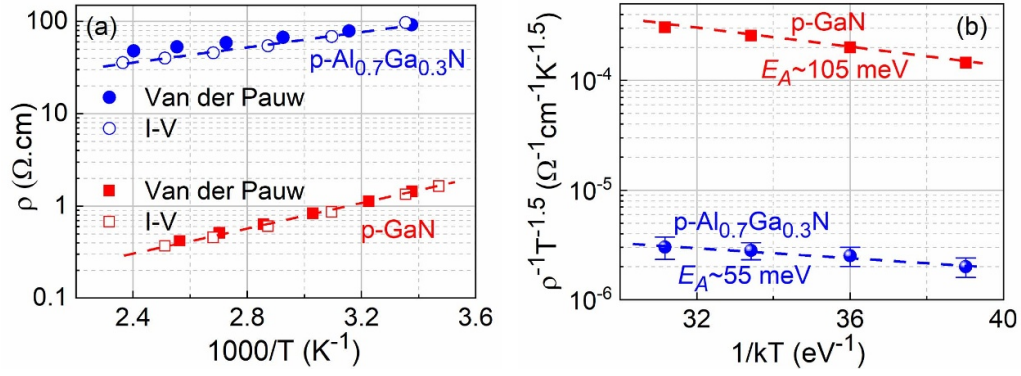


Figure 4. (a) Resistivity (ρ) vs $1000/T$ plot for p-GaN and p-Al_{0.7}Ga_{0.3}N obtained from Van der Pauw and I - V measurements and (b) $\rho^{-1}T^{-1.5}$ vs $1/kT$ plot (up to $\sim 85^\circ\text{C}$) where ρ is taken from the I - V measurements.

agree well, indicating the accuracy of the resistivity extraction methodology using large area contacts. To the 1st-order approximation (considering N_A and $N_D \gg p$), equation (1) can be rewritten as:

$$p \approx \frac{(N_A - N_D)N_V}{gN_D} \exp\left(-\frac{E_A}{kT}\right). \quad (6)$$

In the above equation, N_A , N_D , and g are independent of temperature, whereas N_V has a temperature dependence ($N_V \propto T^{1.5}$). As observed in figure 1(b), the mobility is nearly constant up to $\sim 85^\circ\text{C}$. Therefore, multiplying both sides of the equation (6) with electronic charge (q) and mobility, an equation relating the resistivity and E_A can be written as:

$$\rho^{-1}T^{-1.5} \approx \beta \exp\left(-\frac{E_A}{kT}\right), \quad (7)$$

where β is a constant term comprising the multiplication of q , mobility, and all temperature-independent terms of equation (6), and ρ is the resistivity calculated from the I - V measurements. Therefore, the slope of $\ln(\rho^{-1}T^{-1.5})$ vs $1/kT$ characteristics can be used to extract the E_A if the mobility is constant. Values of E_A derived from the temperature-dependent I - V measurements and equation (7) are ~ 105 meV and ~ 55 meV for p-GaN and p-Al_{0.7}Ga_{0.3}N, respectively. Error margin observed in both these values are within ± 5 meV. Both values are in good agreement with the values obtained using Hall measurements (figure 1(a)). Therefore, I - V measurements using large area contacts offer a simple methodology to complement the E_A in Al-rich p-AlGaIn films estimated using Hall measurements.

The low E_A observed in Al-rich p-AlGaIn films indicates a significant contribution from hole hopping or impurity band conduction mechanism instead of a pure valence band transport as suggested in [17]. Similar behavior has been observed in p-type GaN and InGaIn films, although at relatively low temperatures [28, 29]. The observation of impurity band conduction at RT in this study is possibly related to the low valence band mobility in Al-rich AlGaIn, although more investigation is necessary to quantitatively understand the

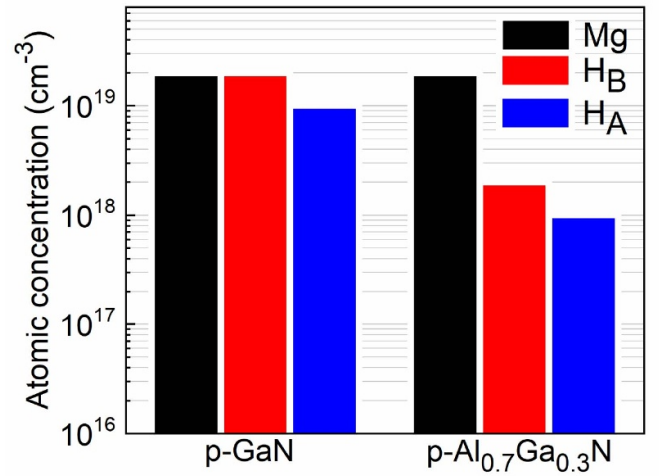


Figure 5. Atomic concentration of Mg and H in p-GaN and p-Al_{0.7}Ga_{0.3}N films; H_B and H_A represents the H concentration before and after dopant activation anneal, respectively.

individual contributions from the valence band and impurity band transport mechanisms. Interestingly, suppressed incorporation of H was observed during the growth of p-AlGaIn films. Figure 5 shows the concentration of Mg and H in p-GaN and p-Al_{0.7}Ga_{0.3}N films measured using SIMS. The concentration of H in p-GaN closely follows the Mg doping concentration (figure 5), which is believed to passivate a majority of the Mg-dopants by forming Mg-H complexes [30–33]. On the contrary, H concentration in p-Al_{0.7}Ga_{0.3}N film was observed to be nearly an order of magnitude lower for a similar Mg doping concentration. Therefore, the density of the Mg-H complexes in p-Al_{0.7}Ga_{0.3}N is likely to be much lower than that of p-GaN, indicating a higher concentration of non-passivated Mg dopants in as-grown p-Al_{0.7}Ga_{0.3}N. Accordingly, appreciable conductivity was observed for non-activated p-Al_{0.7}Ga_{0.3}N films (figures 2(b) and (d)). This contrasts with non-activated p-GaN films that are by and large highly resistive. Further reduction of H concentration in p-Al_{0.7}Ga_{0.3}N is possible by performing an activation anneal

Table 1. Recent reports on p , ρ and E_A of bulk p-AlGa_{0.3}N films at RT.

Composition	p (cm ⁻³)	ρ (Ω cm)	E_A (meV)	Reference
p-Al _{0.7} Ga _{0.3} N	$\sim 1.5 \times 10^{17}$	~ 91	~ 65	This work
p-Al _{0.7} Ga _{0.3} N	1.3×10^{17}	47	47–72	Kinoshita <i>et al</i> [17]
p-Al _{0.6} Ga _{0.4} N	8.7×10^{17}	0.7	≤ 11	Liu <i>et al</i> [9]
p-Al _{0.4} Ga _{0.6} N	3.26×10^{17}	—	46	Chen <i>et al</i> [34]
p-Al _{0.4} Ga _{0.6} N	4.97×10^{18}	0.93	39 ± 3	Qiu <i>et al</i> [8]

at 800 °C under N₂ ambient, as observed in figure 5. Despite the low ionization energy, a lower free hole concentration was observed in p-Al_{0.7}Ga_{0.3}N compared to p-GaN. This is attributed to a higher density of (non-hydrogen) donor-type compensating point defects present in the p-Al_{0.7}Ga_{0.3}N films such as nitrogen-vacancies and Mg nitrogen-vacancy complexes. Indeed, a high N_D of 1.5×10^{19} cm⁻³ was estimated from charge balance fittings to the temperature-dependent Hall measurements in figure 1(a). Firstly, this is related to relatively low formation energies of these defects in Al-rich AlGa_{0.3}N as compared to GaN. Secondly, the high density of non-passivated Mg atoms would shift the quasi-Fermi level closer to the valence band during the growth, further reducing the formation energy of these defects. Furthermore, a high current observed in non-activated films (figures 2(b) and (d)) also indicate that a large number of Mg dopants successfully substituted the group-III elements in p-Al_{0.7}Ga_{0.3}N during growth. A comparison of the values of free hole concentration (p), resistivity (ρ) and E_A obtained in this work with other literature reports for bulk Mg-doped AlGa_{0.3}N films are highlighted in table 1.

At this point, it is necessary to highlight the importance of the I - V measurement methodology developed to estimate the E_A in Al-rich p-AlGa_{0.3}N films. The low hole mobility, likely due to significant alloy scattering or contributions from hopping transport in p-AlGa_{0.3}N [17] results in relatively low Hall coefficients. This poses limitations in the accurate determination of free hole concentration and E_A using the commonly used DC Hall measurements. The AC Hall measurements employed in this work can reliably measure much lower Hall coefficients by effectively reducing the error components involved. It has to be noted that the resistivity extraction methodology may result in an overestimation of E_A if the mobility variation with temperature is large compared to the change in carrier concentration, as seen in figure 1. Furthermore, the estimation of E_A highlighted in this work may not be valid for a wider temperature range, as different ionization or conduction mechanisms dominate at different temperatures in p-AlGa_{0.3}N [9].

Furthermore, both the n-type and p-type contacts to III-nitrides are alloyed contacts where the contact resistance is a strong function of the free carrier concentration in the epitaxial film [13, 35], Hall measurements are trustable only when the contacts used for the measurement possess acceptable resistivity values. The coincidence in value of E_A estimated using Hall measurements and I - V measurements ensures

that Hall measurements performed on Al-rich p-AlGa_{0.3}N films are trustable, and vice-versa. Thus, the alternate technique (using large area Ni/Au contacts) used in this work corroborates the resistivity and E_A estimated using Hall measurements. It is important to note that low mobility is detrimental for lateral devices where the current flow experiences a high semiconductor sheet resistance in high-frequency switching devices. On the other hand, the presence of a high free hole concentration coupled with sufficient electric field can assist significant hole injection into the active region of vertical and/or quasi-vertical optoelectronic devices. Finally, while Si-doping in Al-rich AlGa_{0.3}N is already known to offer $E_A < 50$ meV [4], a low value of E_A in Al-rich p-AlGa_{0.3}N films reported is considered to be instrumental in assisting the development of III-nitride UV light emitters.

4. Conclusions

In this work, a low ionization energy (~ 65 meV) is demonstrated in p-Al_{0.7}Ga_{0.3}N bulk epitaxial films grown using LP-MOVPE. Unlike p-GaN, nearly an order of magnitude lower hydrogen concentration compared to the magnesium doping concentration is observed in p-Al_{0.7}Ga_{0.3}N epitaxial films. Furthermore, current-voltage measurements using large area Ni/Au contacts were shown to corroborate the low ionization energy observed in Al-rich p-AlGa_{0.3}N epitaxial films. Technologically relevant free hole concentration of $\sim 1.5 \times 10^{17}$ cm⁻³ observed in p-Al_{0.7}Ga_{0.3}N films in this study is promising toward the development of future III-nitride UV light emitters.

Data availability statement

All data that support the findings of this study are included within the article.

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