

# Low resistance n-contact for UVC LEDs by a two-step plasma etching process

H K Cho<sup>1,3</sup> , J H Kang<sup>1</sup>, L Sulmoni<sup>2</sup> , K Kunkel<sup>1</sup>, J Rass<sup>1</sup> , N Susilo<sup>2</sup> , T Wernicke<sup>2</sup>, S Einfeldt<sup>1</sup> and M Kneissl<sup>1,2</sup>

<sup>1</sup> Ferdinand-Braun-Institut, Leibniz-Institut für Höchstfrequenztechnik, Gustav-Kirchhoff-Str. 4, 12489, Berlin, Germany

<sup>2</sup> Technische Universität Berlin, Institute of Solid State Physics, Hardenbergstr. 36, EW 6-1, 10623, Berlin, Germany

E-mail: [hyunkyong.cho@fbh-berlin.de](mailto:hyunkyong.cho@fbh-berlin.de)

Received 11 April 2020, revised 29 May 2020

Accepted for publication 19 June 2020

Published 4 August 2020



CrossMark

## Abstract

The impact of plasma etching on the formation of low-resistance n-contacts on the AlGa<sub>0.65</sub>N:Si current spreading layer during the chip fabrication of ultraviolet light-emitting diodes (UV LEDs) emitting at 265 nm is investigated. A two-step plasma etching process with a first rapid etching using BCl<sub>3</sub>/Cl<sub>2</sub> gas mixture and a second slow etching step using pure Cl<sub>2</sub> gas has been developed. The etching sequence provides smooth mesa side-walls and an n-AlGa<sub>0.65</sub>N surface with reduced surface damage. Ohmic n-contacts with a contact resistivity of  $3.5 \times 10^{-4} \Omega \text{cm}^2$  are obtained on Si-doped Al<sub>0.65</sub>Ga<sub>0.35</sub>N layers and the operating voltages of the UVC LEDs were reduced by 2 V for a current of 20 mA.

Keywords: light emitting diode, plasma etch, ohmic contact, low resistance n-contact, high Al mole fraction n-AlGa<sub>0.65</sub>N, operating voltage

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

## 1. Introduction

AlGa<sub>0.65</sub>N-based deep ultraviolet light-emitting diodes (UV LEDs) are promising devices that could replace mercury discharge lamps in various applications including sterilization, water purification, medical diagnostics, phototherapy, and UV curing [1–3]. However, to achieve devices with a high wall plug efficiency, low operating voltages and consequently low contact resistances are required. Due to low electron affinity and to the strong Al-N bond of AlGa<sub>0.65</sub>N with high Al mole fraction, ohmic contacts are difficult to realize [4, 5]. Moreover, a buried n-AlGa<sub>0.65</sub>N layer needs to be exposed by plasma etching before the n-contact metal stack can be deposited. Plasma etching can potentially roughen the surface, change the

stoichiometry of the AlGa<sub>0.65</sub>N materials, and damage the crystal structure [6].

All these effects are known to influence the performance of the electrical contact. Various types of dry etching techniques have been used for etching mesa structures in group-III nitride LEDs or laser diodes, e.g. reactive ion etching (RIE), electron cyclotron resonance plasma, and inductively coupled plasma (ICP). ICP-RIE with Cl<sub>2</sub> or a mixture of Cl<sub>2</sub> and BCl<sub>3</sub> as the chemically reactive gases is the most commonly employed method [7–10]. For etching AlGa<sub>0.65</sub>N with high Al mole fraction in UV LEDs, achieving a sufficiently high etch rate is a further issue because of the strong bond energy between aluminum and nitrogen atoms and the high affinity of aluminum to oxidation [11, 12]. The etch rate can be easily increased by increasing the radio frequency (RF) power during the RIE process. However, a high RF power results in high energy ions which bombard the surface and potentially generate crystal defects [13, 14].

Recently, a two-step plasma etching process has been reported for GaN which involves a final etch step with BCl<sub>3</sub>/Cl<sub>2</sub> and Cl<sub>2</sub> gases at low power to reduce crystal damage

<sup>3</sup> Author to whom any correspondence should be addressed.



Original content from this work may be used under the terms of the [Creative Commons Attribution 4.0 licence](https://creativecommons.org/licenses/by/4.0/). Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.

near the surface [15–17]. Previously, our group described the effects of  $\text{Cl}_2$  plasma etching and various post etching treatments of  $\text{n-Al}_{0.75}\text{Ga}_{0.25}\text{N}$  surfaces on their chemical composition [18].

In this study, we report on the effects of the  $\text{BCl}_3$  to  $\text{Cl}_2$  gas flow ratio and RF power during the ICP-RIE plasma etching of  $\text{n-Al}_{0.65}\text{Ga}_{0.35}\text{N}:\text{Si}$  on the etch rate and the smoothness of fabricated mesa edges. A two-step etch process including a final etching with  $\text{Cl}_2$  at low RF power will be shown to provide the a smooth surface and the a low resistivity for vanadium-based contacts deposited thereon. UVC LEDs with an emission wavelength of 265 nm featuring the optimized two-step etching process exhibit lower operating voltages. To the best of our knowledge, this is the first report of the successful use of a two-step etching process in the processing of UVC LEDs. The approach described contributes to gradually increasing the low efficiency of UVC LEDs and to improve the still limited applicability of the components.

## 2. Experimental

UVC LED heterostructures with a target emission wavelength of 265 nm were grown by metal-organic vapor phase epitaxy on (0001) c-plane sapphire substrates. A 500 nm  $\text{AlN}$  layer on sapphire was patterned into stripes by photolithography and dry etching and then laterally overgrown with  $\text{AlN}$  to a total thickness of 6  $\mu\text{m}$ . Subsequently the  $\text{AlGaN}$ -based heterostructure is deposited comprised of a 900 nm thick  $\text{Al}_{0.76}\text{Ga}_{0.24}\text{N}:\text{Si}$  current spreading layer, a 100 nm  $\text{Al}_{0.76}\text{Ga}_{0.24}\text{N}:\text{Si}/\text{Al}_{0.65}\text{Ga}_{0.35}\text{N}:\text{Si}$  transition layer, 200 nm  $\text{Al}_{0.65}\text{Ga}_{0.35}\text{N}:\text{Si}$  contact layer, a threefold 5 nm  $\text{Al}_{0.62}\text{Ga}_{0.38}\text{N}:\text{Si}/1.4$  nm  $\text{Al}_{0.48}\text{Ga}_{0.52}\text{N}$  multiple quantum well active region, a 10 nm  $\text{Al}_{0.85}\text{Ga}_{0.15}\text{N}$  and 25 nm  $\text{Al}_{0.75}\text{Ga}_{0.25}\text{N}:\text{Mg}$  electron blocking heterostructure and a 230 nm thick  $\text{GaN}:\text{Mg}$  contact layer [19]. In addition, for n-contact optimization studies, single  $\text{Al}_{0.65}\text{Ga}_{0.35}\text{N}:\text{Si}$  contact layers were grown with a thickness of 500 nm.

Test structures for determining the specific n-contact resistance using the transmission line measurement (TLM) were fabricated using photolithography and ICP-RIE etching in a Sentech SI500 system. All etchings were performed with  $\text{BCl}_3$  and  $\text{Cl}_2$  at 20 °C and a pressure of 1 Pa. The gas flow ratio of  $\text{BCl}_3$  and  $\text{Cl}_2$ , the ICP power, the RF power, and the bias voltage were varied. The etched surfaces were examined by scanning electron microscopy (SEM). Etch masks made of photoresist were removed after plasma etching using a standard remover and the samples were then cleaned with piranha solution. Prior to the deposition of the n-contact metal stack, the plasma-etched surfaces were wet-chemically cleaned for 30 s in  $\text{HCl}/\text{H}_2\text{O}$  (1:1), rinsed for one minute in de-ionized water and spin-dried under nitrogen. Thereafter, a metal layer stack of  $\text{V}(15 \text{ nm})/\text{Al}(120 \text{ nm})/\text{Ni}(20 \text{ nm})/\text{Au}(40 \text{ nm})$  was deposited by electron-beam evaporation and structured by a lift-off process. The spacings between the TLM contacts were 10  $\mu\text{m}$ , 15  $\mu\text{m}$ , 20  $\mu\text{m}$ , 25  $\mu\text{m}$ , and 30  $\mu\text{m}$ , respectively. The contacts were annealed at 750 °C in nitrogen ambient for 30 s using a rapid thermal annealing (RTA) furnace.

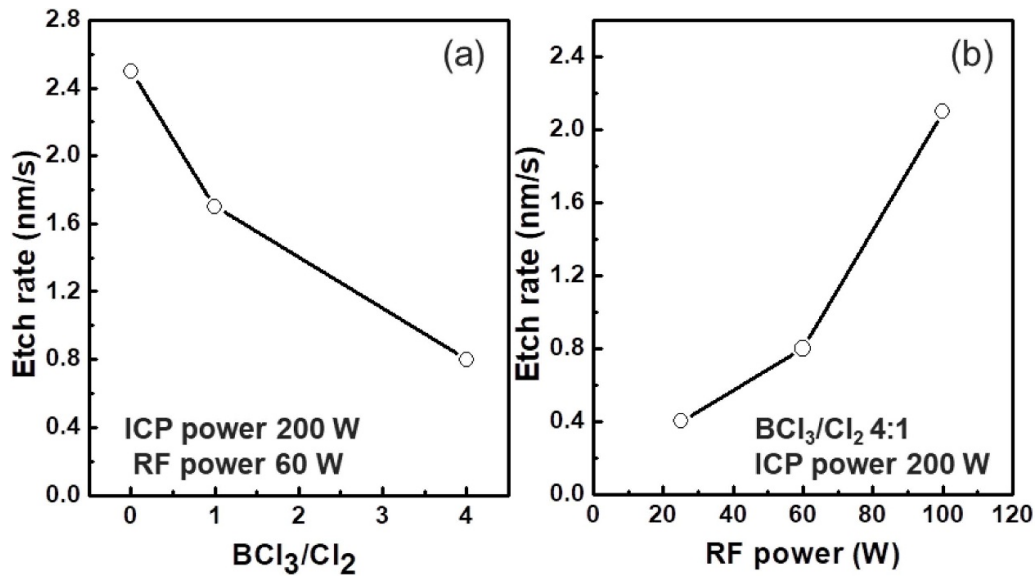
Current-voltage (I–V) measurements were performed at room temperature.

Selected wafers were processed to LEDs. Therefore, p-contacts were fabricated by depositing Pt (30 nm) and annealing at 500 °C under nitrogen for 5 min. A  $\text{SiN}_x$  passivation layer was deposited by plasma enhanced chemical vapor deposition followed by the deposition of the metal bond pads. The fabricated chips are of 1 mm  $\times$  0.6 mm size with an active emitter area of 0.15 mm<sup>2</sup>. The light output power was measured on-wafer at room-temperature using a calibrated Si photodiode.

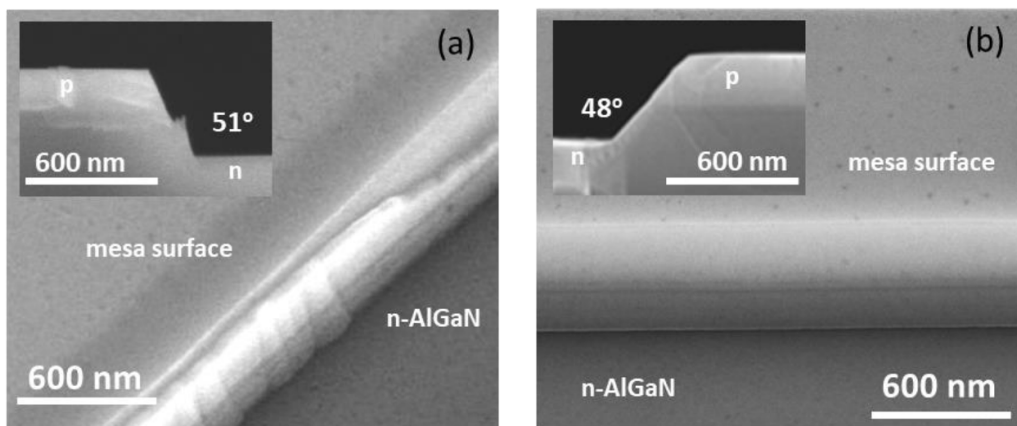
## 3. Results and discussion

Figure 1(a) shows the etch rates of  $\text{n-Al}_{0.65}\text{Ga}_{0.35}\text{N}:\text{Si}$  as a function of the ratio of the  $\text{BCl}_3$  and  $\text{Cl}_2$  flow rates. Etching was performed at an ICP power of 200 W, an RF power of 60 W and a fixed total gas flow. The etch rate decreases with increasing amount of  $\text{BCl}_3$ . This effect can be attributed to the higher dissociation threshold energy of  $\text{BCl}_3$  than  $\text{Cl}_2$  which requires a higher ICP power. With a fixed ICP power, an increased ratio of  $\text{BCl}_3$  in the total gas flow reduces the amount of available radicals thus hindering the etch rate [10]. In addition,  $\text{AlCl}_x$  which is less volatile than  $\text{GaCl}_x$  is known to form on the etched surface of high-aluminum mole fraction  $\text{AlGaN}$  [11]. With a fixed process temperature of 20 °C, the removal of  $\text{AlCl}_x$  relies on the physical bombardment by the plasma. To increase the  $\text{AlGaN}$  etch rate when using a gas flow ratio of 4:1  $\text{BCl}_3/\text{Cl}_2$ , the RF power has been varied at a constant ICP power of 200 W. The results are shown in figure 1(b). The etch rate increases steadily up to the maximum applied RF power 100 W. This effect is attributed to the increased ion energy with higher RF power which enhances the ion-assisted etching and consequently the etch rate.

Figure 2 shows the bird's eye-view scanning electron microscopy (SEM) micrographs of mesa edges etched in to the UVC LED heterostructure with different gas flow ratios: (a) pure  $\text{Cl}_2$  and (b) 4:1  $\text{BCl}_3/\text{Cl}_2$ . With the pure  $\text{Cl}_2$  plasma, the sidewalls of the etched mesa structure show a rough surface with an additional fence structure. It is assumed that the fence structure is related to the redeposition of reaction products, most likely oxides, from etching and indicates a high proportion of physical ablation. The rough sidewalls of the etched mesa were observed independent of the ICP power or the RF power (not shown here). This is assumed to be related to crystal damage. When the concentration of  $\text{BCl}_3$  increases, the mesa sidewalls become smoother and more clearly defined (figure 2(b)). This is probably due to re-deposition effects from sputtering during the plasma etch by using  $\text{BCl}_3$  based gas which generates heavier ions [10]. Furthermore, the previously observed fence is less pronounced due to the preferred etching of oxides by  $\text{BCl}_3$ . The redeposited layer protects the exposed mesa sidewalls during etching thus resulting in a smoother surface [10, 20, 21]. Images for determining the inclination angles of the mesa edges are shown as insets of figure 2. The gas flow ratio is not impacting the mesa inclination angles as 48° and 51° are measured in cross section SEM



**Figure 1.** Etch rates of n- $\text{Al}_{0.65}\text{Ga}_{0.35}\text{N}:\text{Si}$  as a function of (a) the ratio of  $\text{BCl}_3$  and  $\text{Cl}_2$  flow rates using a constant ICP power of 200 W, an RF power of 60 W, and a bias voltage of  $-100$  V, and (b) the RF power using a ratio of the gas flow rates  $\text{BCl}_3/\text{Cl}_2$  of 4:1, and an ICP power of 200 W.



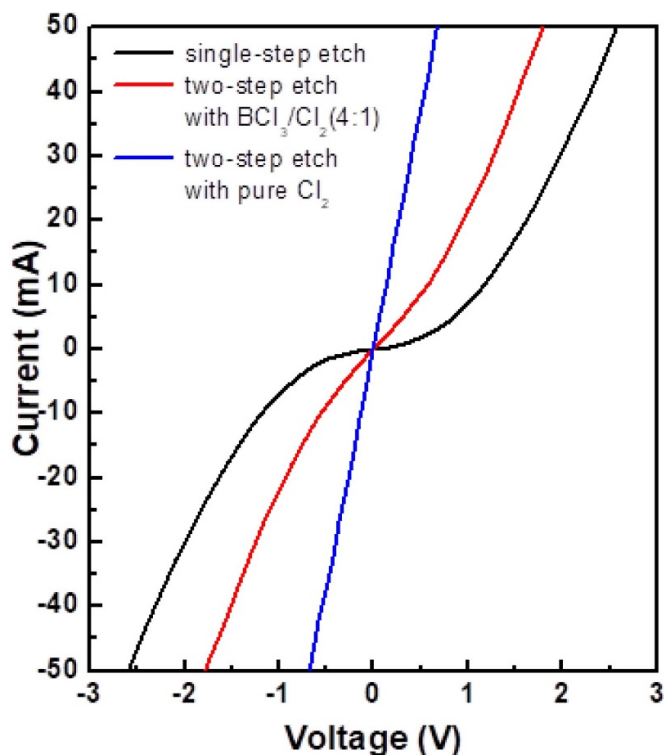
**Figure 2.** Bird's eye view SEM micrographs of the etched mesa edge of a UVC LED with (a)  $\text{Cl}_2$  and (b) a  $\text{BCl}_3/\text{Cl}_2$  gas mixture of 4:1 and using an ICP power of 200 W, an RF power of 100 W, and a bias voltage of  $-200$  V. Inset: corresponding cross section SEM micrographs with inclination angles of the mesa side walls.

micrographs for pure  $\text{Cl}_2$  plasma and for  $\text{BCl}_3/\text{Cl}_2$  gas mixture of 4:1, respectively (insets in figure 2).

A reasonably high etch rate during plasma etching requires high ICP power, high RF power, and high bias voltage. Unfortunately, plasma-induced damage often results in an etched near-surface of poor quality: generation of point defects such as Ga or N vacancies which can considerably degrade the device performance [22]; alteration of the near-surface stoichiometry through preferential loss of the more volatile element, e.g. N deficiency of the surface and subsequent oxidation [18, 23]; introduction of deep non-radiative compensating centers due to energetic ion bombardment [14, 24]. Therefore, in order to preserve the electrical contact performance of the electrodes deposited on the etched surface, a two-step etching process is proposed here. First, the target etch depth is approached with a high etch rate of  $2.1 \text{ nm s}^{-1}$  using  $\text{BCl}_3/\text{Cl}_2$ . Second, possible surface damage is removed using a low etch rate of  $0.3 \text{ nm s}^{-1}$  using either  $\text{Cl}_2$  or  $\text{BCl}_3/\text{Cl}_2$ . This is

in agreement with the results for n-contacts on n- $\text{Al}_{0.75}\text{Ga}_{0.25}\text{N}$  reported in [25]. Accordingly, the second etch step was performed with  $\text{Cl}_2$  or  $\text{BCl}_3/\text{Cl}_2$  at a low RF power of 15 W. The RF power is mainly linked to the physical etching process, i.e. the lower the RF power the smaller the expected surface damage. The ICP power was set to 100 W to ensure a stable plasma discharge at the low RF power.

The I-V characteristics of annealed contacts on plasma-etched n- $\text{Al}_{0.65}\text{Ga}_{0.35}\text{N}$  surfaces with different gas flow mixtures in the second etch step are shown in figure 3. The I-V curves are non-linear for the case of single-step etching with a  $\text{BCl}_3/\text{Cl}_2$  gas mixture of 4:1. When adding a second etch step with  $\text{BCl}_3/\text{Cl}_2$  and  $\text{Cl}_2$ , the voltage drop at the contacts decreases.  $\text{Cl}_2$  plasma is more effective in reducing contact resistivity than  $\text{BCl}_3$  plasma. An ohmic contact with a contact resistivity of  $(3.5 \pm 0.5) \times 10^{-4} \Omega \text{cm}^2$  is found when using pure  $\text{Cl}_2$  gas in the second etch step. The result supports the idea that the semiconductor surface suffers from either crystalline



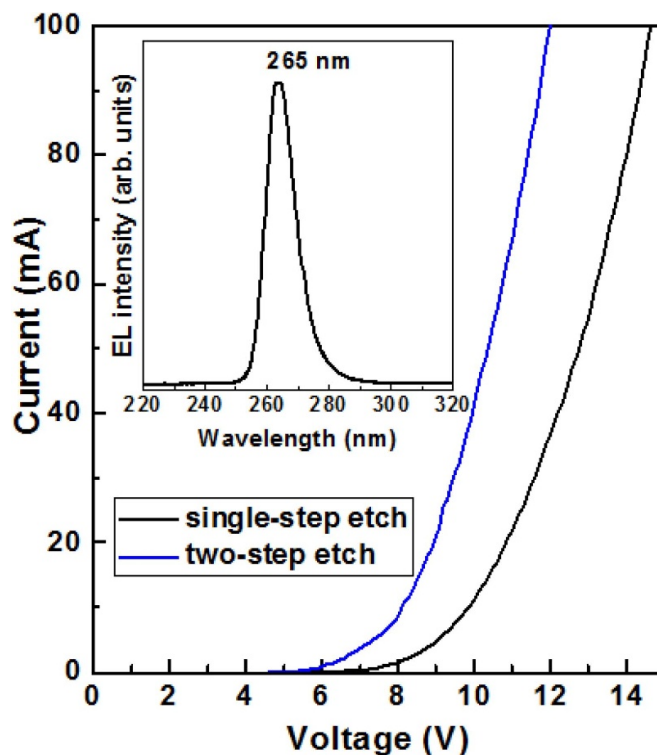
**Figure 3.** Four-point probe I–V characteristics of annealed V/Al/Ni/Au contacts with an electrode distance of 20  $\mu\text{m}$  on n- $\text{Al}_{0.65}\text{Ga}_{0.35}\text{N}:\text{Si}$  after single-step etching using  $\text{BCl}_3/\text{Cl}_2$  gas mixture of 4:1 (black curve), two-step etching using  $\text{BCl}_3/\text{Cl}_2$  (4:1) (red curve) and  $\text{Cl}_2$  (blue curve) in the second etch step.

damage or residuals induced by plasma etching which limit the contact performance. Improved contacts can be obtained by reducing this damage or these residuals through final plasma etch step where chemical etching dominates rather than physical etching.

Figure 4 shows the I–V characteristics and the emission spectrum of UVC LEDs fabricated with single-step and two-step etching process. The operating voltage of the devices with the two-step etching is lower than that of the devices with a single-step etching at a constant forward current, e.g. 8.5 V and 11 V at 20 mA, respectively. This finding agrees with the TLM data discussed before and suggests that a significant part of the operating voltage of the devices with a single-etching process drops at the n-contact. Again, the reduced resistivity of the n-contact of the LEDs is attributed to the fact that the surface damage below the metal stack is reduced during the soft final  $\text{Cl}_2$  plasma etch step.

#### 4. Conclusion

The plasma etching of n- $\text{Al}_{0.65}\text{Ga}_{0.35}\text{N}$  layers was investigated with respect to (i) the performance of V/Al/Ni/Au n-contacts deposited on the etched n-AlGaN surfaces and (ii) the feasibility of implementing the technology in the front-end processing of UVC LEDs emitting at 265 nm. A reasonable etch rate with smooth sidewalls of etched mesas was obtained when using a gas mixture of  $\text{BCl}_3$  and  $\text{Cl}_2$  and a sufficient RF power. An additional final etch step with pure



**Figure 4.** I–V characteristics of UVC LEDs fabricated with single-step etching using  $\text{BCl}_3/\text{Cl}_2$  gas mixture of 4:1 (black curve) or two-step etching using  $\text{Cl}_2$  in the second etch step (blue curve). The inset shows a typical emission spectrum of both LED types at 20 mA (0.15  $\text{mm}^2$ ). All the measurements were performed on-wafer in continuous wave operation.

$\text{Cl}_2$  at a low RF power was found to smoothen the surface and resulted in an ohmic n-contact with a contact resistivity of  $(3.5 \pm 0.5) \times 10^{-4} \Omega\text{cm}^2$ . The developed two-step etching process was successfully applied to UVC LEDs and reduced their operating voltage by 2 V at 20 mA. The superior performance of the n-contacts and the LEDs is attributed to the reduction of surface damage or residuals from plasma etching during the final soft etch step.

#### Acknowledgments

The authors would like to thank the Process Technology Department at the Ferdinand-Braun-Institut Leibniz Institut für Höchstfrequenztechnik, in general for their contributions to the developments of contacts for DUV LEDs. This work was partially supported by the German Federal Ministry of Education and Research (BMBF) in the ‘Advanced UV for Life’ consortium under contracts 03ZZ0134B and 03ZZ0134C.

#### ORCID iDs

H K Cho <https://orcid.org/0000-0001-5540-2582>  
 L Sulmoni <https://orcid.org/0000-0002-5341-7032>  
 J Rass <https://orcid.org/0000-0002-9232-0495>  
 N Susilo <https://orcid.org/0000-0002-5583-629X>



## References

- [1] Hirayama H 2005 Quaternary InAlGaN-based high-efficiency ultraviolet light-emitting diodes *J. Appl. Phys.* **97** 091101
- [2] Kneissl M and Rass J ed 2016 *III-Nitride Ultraviolet Emitters - Technology & Applications (Series on Material Science)* vol 227 (Heidelberg: Springer)
- [3] Nagasawa Y and Hirno A 2018 A review of AlGaN-based deep-ultraviolet light-emitting diodes on sapphire *Appl. Sci.* **8** 1264
- [4] Moses P G, Miao M, Yan Q and Van de Walle C G 2011 Hybrid functional investigations of band gaps and band alignments for AlN, GaN, InN, and InGaN *J. Chem. Phys.* **134** 084703
- [5] Van Daele B, Tendeloo G V, Ruythooren W, Derluyn J, Leys M R and Germain M 2005 The role of Al on ohmic contact formation on n-type GaN and AlGaN/GaN *Appl. Phys. Lett.* **87** 061905
- [6] Pearton S J 1997 Characterization of damage in electron cyclotron resonance plasma etched compound semiconductors *Appl. Surf. Sci.* **117** 597
- [7] Han Y, Xue S, Wu T, Wu Z, Guo W, Luo Y, Hao Z and Sun C 2004 Nonselective and smooth etching of GaN/AlGaN heterostructures by Cl<sub>2</sub>/Ar/BCl<sub>3</sub> inductively coupled plasmas *J. Vac. Sci. Technol. A* **22** 407
- [8] Hahn Y B, Hays D C, Conovan S M, Abernathy C R, Han J, Shul R J, Cho H, Jung K B and Pearton S J 1999 Effect of additive noble gases in chlorine-based inductively coupled plasma etching of GaN, InN, and AlN *J. Vac. Sci. Technol. A* **17** 768
- [9] Shul R J, McClellan G B, Casalnuova S A, Rieger D J, Pearton S J, Constantine C, Barrat C, Karlicek R F, Tran C Jr and Schurman M 1996 Inductively coupled plasma etching of GaN *Appl. Phys. Lett.* **69** 1119
- [10] Rawal D S, Sehgal B K, Muralidharan R, Malik H K and Dasgupta A 2012 Effect of BCl<sub>3</sub> concentration and process pressure on the GaN mesa sidewalls in BCl<sub>3</sub>/Cl<sub>2</sub> based inductively coupled plasma etching *Vacuum* **86** 1844
- [11] Smith S A, Wolden C A, Bremser M D, Hanser A D, Davis R F and Lampert W V 1997 High rate and selective etching of GaN, AlGaIn, and AlN using an inductively coupled plasma *Appl. Phys. Lett.* **71** 3631
- [12] Kim H S, Lee D H, Lee J W, Kim T I and Yeom G Y 2000 Effects of plasma conditions on the etch properties of AlGaIn *Vacuum* **56** 45
- [13] Miller M A, Mohney S E, Nikiforov A, Cargill III G S and Bogart K H A 2006 Ohmic contacts to plasma etched n-Al<sub>0.58</sub>Ga<sub>0.42</sub>N *Appl. Phys. Lett.* **89** 132114
- [14] Cao X A, Piao H, LeBoeuf S F, Li J, Lin J Y and Jiang H X 2006 Effects of plasma treatment on the ohmic characteristics of Ti/Al/Ti/Au contacts to n-AlGaIn *Appl. Phys. Lett.* **89** 082109
- [15] Ono K, Nakazaki N, Tsuda H, Takao Y and Eriguchi K 2017 Surface morphology evolution during plasma etching of silicon: roughening, smoothing and ripple formation *J. Phys. D: Appl. Phys.* **50** 414001
- [16] Tahhan M, Nedy J, Chan S H, Lund C, Li H, Gupta G, Keller S and Mishra U 2016 Optimization of a chlorine-based deep vertical etch of GaN demonstrating low damage and low roughness *J. Vac. Sci. Technol. A* **34** 031303
- [17] Qiu R, Lu H, Chen D, Zhang R and Zheng Y 2011 Optimization of inductively coupled plasma deep etching of GaN and etching damage analysis *Appl. Surf. Sci.* **257** 2700
- [18] Lapeyrade M et al 2017 Effect of Cl<sub>2</sub> plasma treatment and annealing on vanadium based metal contacts to Si-doped Al<sub>0.75</sub>Ga<sub>0.25</sub>N *J. Appl. Phys.* **122** 125701
- [19] Susilo N et al 2018 AlGaIn-based deep UV LEDs grown on sputtered and high temperature annealed AlN/sapphire *Appl. Phys. Lett.* **112** 041110
- [20] Agarwala S, Horst S C, King O, Wilson R and Stone D 1998 High-density inductively coupled plasma etching of GaAs/AlGaAs in BCl<sub>3</sub>/Cl<sub>2</sub>/Ar: A study using a mixture design *J. Vac. Sci. Technol. B* **16** 511
- [21] Tanide A, Nakamura S, Horikoshi A, Takatsuji S, Kohno M, Kinose K, Nadahara S, Ishikawa K, Sekine M and Horii M 2019 Effects of BCl<sub>3</sub> addition to Cl<sub>2</sub> gas on etching characteristics of GaN at high temperature *J. Vac. Sci. Technol. B* **37** 021209
- [22] Cho H K, Khan F A, Adesida I, Fang J Q and Look D C 2008 Deep level characteristics in n-GaN with inductively coupled plasma damage *J. Phys. D: Appl. Phys.* **41** 155314
- [23] Mouffak Z, Bensaoula A and Trombetta L 2004 The effects of nitrogen plasma on reactive-ion etching induced damage in GaN *J. Appl. Phys.* **95** 727
- [24] Jang H W and Lee J L 2003 Effect of Cl<sub>2</sub> plasma treatment on metal contacts to n-type and p-type GaN *J. Electrochem. Soc.* **150** G513
- [25] Wernicke T, Sulmoni L, Kuhn C, Tränkle G, Weyers M and Kneissl M 2020 Group III-nitride-based UV laser diodes *Semiconductor Nanophotonics (Springer Series in Solid-State Sciences)* vol 194, ed M Kneissl, A Knorr, S Reitzenstein and A Hoffmann (Berlin: Springer)