

Photoluminescence Analysis for Mitigating Sidewall Surface Nonradiative Recombination of n-GaN in Micro-LEDs Using Time-Dependent Isotropic Dry Etching

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Abstract

We show significant improvement on photoluminescence intensity of the n-type Gallium Nitride surface by using a damage-recovery isotropic dry etching technique to remove the plasma damaged layer. The average photoluminescence intensity exhibits an approximate 173% increase after 60 mins-etching, compared with the n-GaN without isotropic etching. Our findings pave the way for a significant step towards commercialization of μ LED displays for AR/VR, phones and smart watches.

Author Keywords

Sidewall/plasmas damage, nonradiative recombination, microLED, isotropic dry etching and GaN.

1. Objective and Background

Semiconductor based micro-light-emitting diodes (μ LEDs) for micro-displays has been considered as one of the most promising candidates for next-generation display applications into AR/VR smart glasses that need high-resolution, high brightness and energy-efficient (low heat emission) displays^{1,2}. To achieve the strict near-eye operating conditions of AR/VR glasses, fabricating highly efficient μ m-level μ LEDs in the low current density operating conditions is a significant challenge. In general, the reduced size of the μ LEDs helps to improve luminous efficiency, homogenise the current spreading, decrease the junction temperature, release the strain and enhance the light extraction efficiency³. However, the external quantum efficiency (EQE) for μ LEDs drops with decreasing LED sizes. Shockley-Read-Hall (SRH) nonradiative recombination appearing as the high-intensity sidewall and surface defects formed during Inductively Coupled Plasma Reactive Ion Etching (ICP-RIE) etching of μ LEDs mesas as plasma-induced damage, originating from ion bombardment and deep ultraviolet (UV) photon irradiation^{1,3-5}.

Despite the extensive efforts to reduce sidewall surface non-radiative recombination in Gallium Nitride (GaN)-based μ LEDs, low-power dry ICP etching⁶ has been applied to mitigate plasma damage in conventional LEDs. However, the potential of omnidirectional isotropic dry etching to enhance EQE of μ LED have not been well studied. Because the impact of sidewall damage is more pronounced in μ LED than in conventional LEDs, isotropic dry etching could play a critical role in improving μ LED performance. Nevertheless, its specific effects on μ LEDs are not yet fully understood, highlighting the need for further investigation into this promising approach.

In this work, we present an innovative dry etching process including a standard ICP-RIE process to create mesas and an isotropic etching process to remove the sidewall damage, and hence reduce the non-radiative recombination for the final μ LED devices. We investigated the sub-surface damage induced by ICP-RIE process and damage-recovery enabled by isotropic etching by observing the change in photoluminescence (PL) on n-GaN samples. We used the relative integrated PL intensity as a metric for changes in surface damage, which was used as an analogue for sidewall damage and damage-recovery. Due to sensitivity of

PL to the non-uniformity of n-GaN material, both pre-etching and post-etching PL measurements were carried out to show the accuracy of PL intensity improvement. Controlling the etch depth through etch time allows us to show the vertical and horizontal etching depths, as well as the etch profile. The deeper n-GaN regions are less affected by plasma damage and are used as an internal reference for PL comparison with pre-mesa-etched samples. Furthermore, we will investigate the surface band bending of the isotropically etched n-GaN surface using the Kelvin probe force microscopy (KPFM) mode of a Jupiter XRTM atomic force microscopy (AFM, Asylum Research, Oxford Instruments) to show how this is linked directly with the improvement of PL intensity. This work revealed that the non-radiative recombination caused by plasma damage can be minimised by proper sidewall treatments.

2. Results

n-GaN mesa structures were etched on commercial 1.5 μ m-thick silicon doped n-type GaN (n-GaN) wafer ($\sim 1.5 \times 10^{18} \text{ cm}^{-3}$) grown using metal-organic chemical vapour deposition (MOCVD) on photoresist (PR) patterned sapphire substrates (Enkris Semiconductor Inc.). Mesas were ICP-RIE with the depth of the mesa is approximately 600 nm. After that, nine samples were isotropic etched with different time:

1, 5, 10, 15, 20, 30, 40, 50 and 60 mins.

The effects of time-dependent isotropic etching on the UV light emission of n-GaN have been demonstrated by PL images shown in Fig. 1. The PL mapping experiment was conducted using a Renishaw inVia Raman and photoluminescence microscope (Qontor). Because of the high absorption of UV light by photoresist and a limited spatial resolution of PL measurements around 1 μ m, the PL mapping area on the etched n-GaN region were fixed at 10 μ m x 10 μ m next to a mesa. We assume that ICP-RIE process can create similar level of plasma damage on the etched regions at the sidewall and mesa-bottom. The emission wavelength was measured to be approximately 362 nm by PL excited by a 25mW, 325-nm He-Cd ultra-violet (UV) laser. The n-GaN sample without isotropic etching (mesa etched) indicated as reference, resulted in a dim and non-radiative recombination rich PL map. However, the PL intensity of the n-GaN samples show a gradual increase across nine samples as isotropic etch time increases. This demonstrates that deeper isotropic etching into n-GaN subsurface enhances the PL intensity. In other words, the plasma damage region, characterised by high density of non-radiative recombination defects generated at the sidewall and mesa-bottom after ICP-RIE process, was effectively removed layer by layer as the etching progressed deeper.

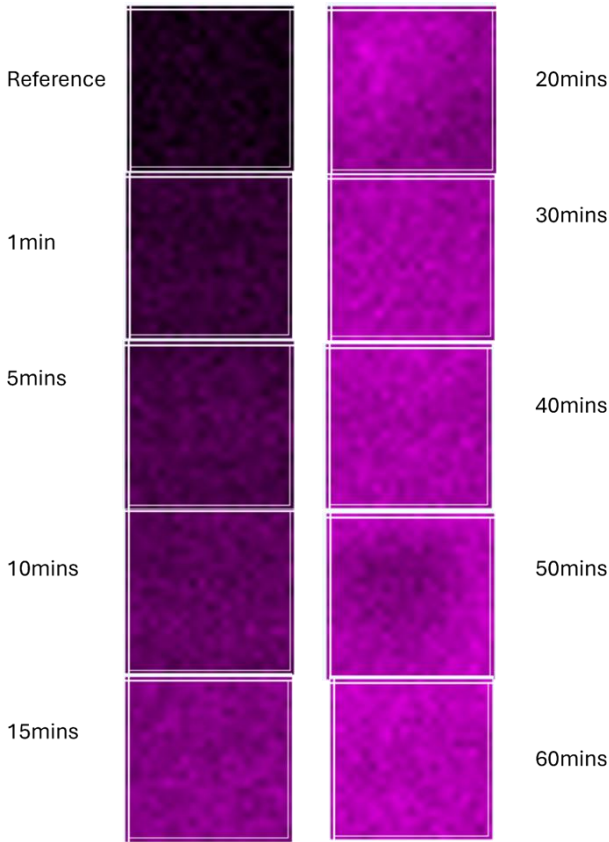


Figure 1. Photoluminescence (PL) images of n-GaN samples with varying isotropic etch time from 0 to 60 mins. The light intensity from 10 $\mu\text{m} \times 10 \mu\text{m}$ mapping area was set between 13 and 35 PL counts.

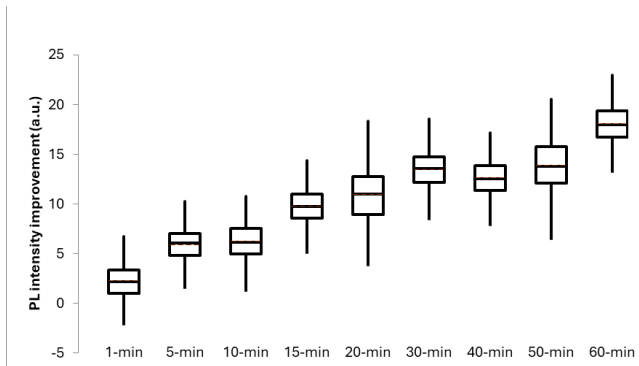


Figure 2. Box plot of the PL improvement data of n-GaN samples with varying isotropic etch times from 1 to 60 mins.

A box plot showing PL intensity improvement of isotropically etched samples, as compared to the reference sample, was generated by analysing the PL mapping data (441 points) over a 10 $\mu\text{m} \times 10 \mu\text{m}$ area as illustrated in Fig. 2. The box plot shows that the average PL intensity of isotropically etched n-GaN samples, from 1 min to 30 mins, consistently increased from 3 to 14, corresponding to an impressively average improvement of 15 to 98% as compared to the reference sample. However, the average PL intensity improvement of isotropically etched n-GaN samples, from 30 mins to 50 mins, maintained between 13 and 14, with minor difference due to over-burning of PR or other etching condition issues. Finally, PL intensity improvement of

isotropically etched n-GaN sample at 60 mins increased to 18, corresponding to a remarkable average improvement of 173%, as compared to the reference sample. This increase in average PL intensity can be explained by the effects of SRH non-radiative recombination sites caused by sidewall and surface damage and recombination.

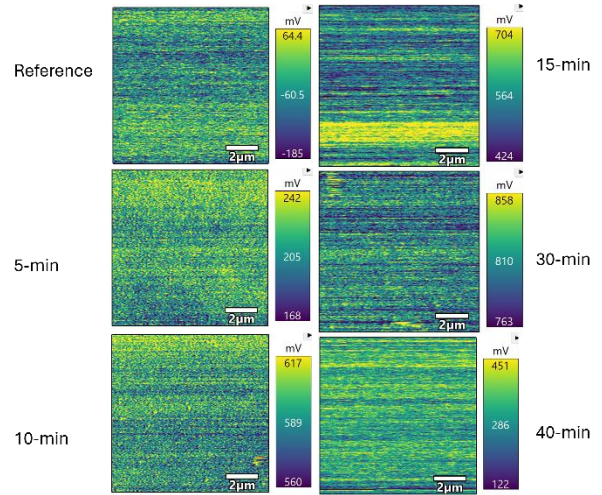


Figure 3. Kelvin probe force microscopy (KPFM) images of n-GaN samples with varying isotropic etch time from 0 to 40 mins.

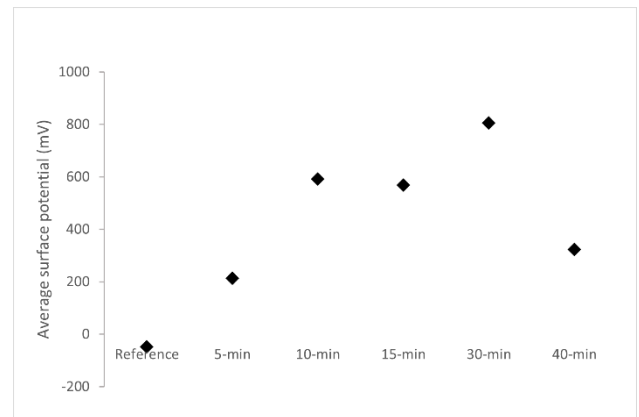


Figure 4. Average surface potential as a function of isotropic etch time ranging from 0 to 40 mins.

Changes in surface potential caused by plasma damage can be effectively analysed using KPFM, as demonstrated in previous studies^{1,8}. This technique provides valuable insights into the impact of plasma-induced modifications, enabling a deeper understanding of material behaviour at the nanoscale. In this study, the surface potentials of isotropically etched-GaN surfaces were characterised using KPFM over a 10 $\mu\text{m} \times 10 \mu\text{m}$ area as shown in Fig. 3 and 4. The KPFM images in Fig. 3 reveal that the uniformly distributed surface potentials tend to increase from the reference samples to those with longer etch time. KPFM works by measuring the contact potential difference between the AFM probe tip and the n-GaN surface. As shown in Fig. 4, the observed surface potential differences reflect variations in the surface band bending among those samples¹. Our findings reveal that the isotropically etched sample exhibited a higher surface potential, increasing by 800 mV after 30 mins of etching. This indicates that

the isotropic etching process induces a slightly greater number of surface states, resulting in more pronounced positive band bending compared to the reference sample. However, the surface potential of the sample etched at 40 mins decreased to approximately 250 mV, likely due to unintended deposition of material from the PR mask during the extended etching process. The KPFM results align closely with the PL intensity improvement data, confirming a significant reduction in nonradiative recombination centres on the isotropically etched n-GaN surface. However, further investigation is needed to fully understand the UV transparent behaviour of the materials during extended isotropic etching processes, as well as the chemical characteristics of the isotropically etched n-GaN surface.

In conclusion, we have demonstrated time-dependent PL intensity improvements with etch times ranging from 1 to 60 mins using an innovative isotropic dry etch process. For n-GaNs with 60-min isotropic etching, the PL intensity increased by 173% over a 10 μm x 10 μm area. The KPFM images revealed a progressive increase in surface potential with longer isotropic etch times. These results showed that isotropic etching is effective for reduction of SRH non-radiative recombination and surface recombination induced by plasma damage.

3. References

1. Wang, X., Zhao, X., Takahashi, T. *et al.* $3.5 \times 3.5 \mu\text{m}^2$ GaN blue micro-light-emitting diodes with negligible sidewall surface nonradiative recombination. *Nat*

Commun **14**, 7569 (2023).
<https://doi.org/10.1038/s41467-023-43472-z>

2. Matthew S. Wong *et al* 2019 *Appl. Phys. Express* **12** 097004 DOI 10.7567/1882-0786/ab3949.
3. Matthew S. Wong, Development of Device Technology for Micro-Light-Emitting Diodes, PhD thesis, UCSB, 2020.
4. Masaki Minami *et al* 2011 *Jpn. J. Appl. Phys.* **50** 08JE03.
5. Shota Nunomura 2023 *J. Phys. D: Appl. Phys.* **56** 363002.
6. Joseph G Nedy *et al* 2015 *Semicond. Sci. Technol.* **30** 085019.
7. Hori, M. Radical-controlled plasma processes. *Rev. Mod. Plasma Phys.* **6**, 36 (2022).
8. Baret, S. *et al.* Surface potential of n- and p-type GaN measured by Kelvin force microscopy. *Appl. Phys. Lett.* **93**, 212107 (2008).