

Article

Investigations of Sidewall Passivation Technology on the Optical Performance for Smaller Size GaN-Based Micro-LEDs

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Abstract: Micro-light emitting diodes (Micro-LEDs) based on III-nitride semiconductors have become a research hotspot in the field of high-resolution display due to its unique advantages. However, the edge effect caused by inductively coupled plasma (ICP) dry etching in Micro-LEDs become significant with respect to the decreased chip size, resulting in a great reduction in device performance. In this article, sector-shaped GaN-based blue Micro-LEDs are designed and fabricated. Additionally, the device performance of different size Micro-LEDs with passivation are investigated with respect to those without passivation. Several methods have been applied to minimize the etching damage near the edge, including acid-base wet etching and SiO₂ passivation layer growth. The room temperature photoluminescence (PL) results demonstrate that the light emission intensity of Micro-LEDs can be significantly enhanced by optimized passivation process. PL mapping images show that the overall luminescence of properly passivated Micro-LEDs is enhanced, the uniformity is improved, and the effective luminescence area is increased. The recombination lifetime of carriers in Micro-LEDs are increased by the usage of passivation process, which proves the reduction in non-radiative recombination centers in Micro-LEDs and improved luminescence efficiency. As a result, the internal quantum efficiency (IQE) is improved from 14.9% to 37.6% for 10 μm Micro-LEDs, and from 18.3% to 26.9% for 5 μm Micro-LEDs.

Keywords: Micro-LED; passivation; edge effect



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1. Introduction

Micro-LEDs have become one key technology of LED-based display chips for the next generation of high-resolution display. By reducing the size and pitch of LED pixels from centimeters/millimeters to micrometers, the resolution of a Micro-LED display can reach up to 3000 PPI(pixel per inch), or even more. In comparison with organic light emitting diode(OLED) technology, GaN-based Micro-LEDs also have other advantages including high contrast, high efficiency, fast response speed, high chemical and structure stability, etc., which make Micro-LEDs a current research hotspot. However, several problems are needed to be settled in the development of Micro-LEDs. One of the most important things is the efficiency of Micro-LEDs. With the reduced chip size, the edge effect of the sidewall becomes an inescapable issue. The etching process in fabrication procedure will introduce unavoidable etching damages on the side walls of each chip, which are known as non-radiative centers, resulting in degrading the device performance. Therefore, the

passivation and heal of those etching damage on sidewalls hold the great significance for the performance of Micro-LED.

At present, the edge effect and passivation of GaN-based LED on sapphire substrate have been investigated by pioneers. Kim et al. have reported that the leakage current of LED devices has a significant relationship with the size of the chip [1]. They used NO_2 plasma to passivate the sidewall to recover the etching damage. Huh and others treated the etched GaN structure with ammonium sulfide ($(\text{NH}_4)_2\text{S}$) aqueous solution and a mixture of ammonium sulfide and tert-butanol ($\text{C}_4\text{H}_9\text{OH}$), which passivated the etching damage on the side wall and reduced the reverse leakage current of the device [2]. Chevtchenko et al. and Pankove et al. respectively demonstrated that passive layer such as SiN_x can avoid direct contact between the chip and the outside environment, which reduce the density of surface state, thereby reducing the probability of surface recombination and inhibiting leakage current [3,4]. Although there are many reports on surface treatment for nitrides, most of these research objects are conventional large-scale LED. With the continuous development of technology, LED is developing towards miniaturization. Micro-LED technology emerges as the time requires. Currently, the sizes of most Micro-LEDs are tens to hundreds of microns, while we are studying Micro-LEDs with tiny size, which are less than $10\text{ }\mu\text{m}$. At this tiny size, the edge effect has a significant effect on the overall luminescent of Micro-LED, so it is necessary to study the passivation and its influence on the side wall of small-sized Micro-LED. As far as we know, there are few reports on the side wall passivation of Micro-LEDs with a tiny size less than $10\text{ }\mu\text{m}$, especially there is a lack of comprehensive and detailed optical characterization of small-sized Micro-LEDs before and after passivation. Through this research, it will be of great significance to the future academic research and industrial application of small-size Micro-LED.

In this article, we designed and fabricated sector-shaped GaN-based Micro-LEDs with a radius of $5\text{ }\mu\text{m}$ and $10\text{ }\mu\text{m}$, respectively. Several kinds of different passivation treatment were applied, including acid-base wet etching and SiO_2 passivation layer growth. Then a series of optical and electrical measurements, involving scanning electron microscope (SEM), photoluminescence (PL), PL mapping, time-resolved photoluminescence (TRPL), temperature-dependent photoluminescence (TDPL), and electroluminescence (EL) were conducted on the samples before and after passivation, in order to reveal the effect of optimized passivation process.

2. Experimental Methods

InGaN/GaN multiple quantum well (MQW) LED wafer were grown on (0001) sapphire substrates by metal-organic chemical vapor deposition (MOCVD). The structure includes $3\text{ }\mu\text{m}$ -thickness GaN buffer layer, $2\text{ }\mu\text{m}$ -thickness n-GaN layer, 15-pairs InGaN/GaN MQWs, and 100 nm -thickness p-GaN layer. A 200 nm SiO_2 layer was grown on the surface of the LED wafer as the etching mask by using plasma-enhanced chemical vapor deposition (PECVD). Typical photolithography, reactive ion etching (RIE), and inductively coupled plasma (ICP) etching process were adopted on LED wafer in order to form sector-shaped mesa. Then hydrofluoric acid was used to remove the SiO_2 mask layer. Overlay lithography was used to expose n-type and p-type electrode area, then electrodes of Ti/Al/Ni/Au were fabricated by physical vapor deposition (PVD), and rapid thermal annealing (RTA) was used to form ohmic contact. In this work, Micro-LEDs with a radius of $5\text{ }\mu\text{m}$ and $10\text{ }\mu\text{m}$ were fabricated. The samples were divided into 8 groups labeled S1 to S8, and different passivation methods were applied respectively, including acid-base wet etching and SiO_2 passivation layer growth. The details of passivation process are shown in Table 1. We first demonstrated the processing steps for S8. Firstly, the sample was placed in 1 mol/L KOH solution for 10 minutes water-bath at $40\text{ }^\circ\text{C}$; secondly, we placed the sample in $\text{HCl:H}_2\text{O} = 1:1$ solution for 10 minutes water-bath at $40\text{ }^\circ\text{C}$. Finally, a 400 nm SiO_2 passivation layer was deposited on the surface of the sample. For S5, S6, and S7, only need to omit one step of S8. Subsequently, a series of optical measurements were performed on each group of samples. The morphology of cross-section was observed by

scanning electron microscope (SEM, JEOL JSM 7000F, Japan). Photoluminescence (PL, Renishaw, UK) was used to investigate the light emission intensity of Micro-LEDs. The PL spectra were gathered by spectrometer (HORIBA iHR 320, Japan) with a spectral resolution of 1 nm and collected by the Micro-PL system with a 50× UV objective (Mitutoyo, numerical aperture 0.5, focal length 200 nm). The lateral resolution of the PL system was 5 µm. The laser beam was directed vertically onto the sample surface. The PL spectra were measured for one mesa. The PL mapping technique was employed to study the edge effect of Micro-LEDs. The lateral resolution of PL mapping system was 1 µm. Time-resolved photoluminescence (TRPL) spectra indicate the recombination lifetime of carriers in active region of Micro-LEDs. Temperature-dependent photoluminescence (TDPL) was used to estimate the internal quantum efficiency (IQE). Additionally, electroluminescence (EL) was used to study the luminescence of Micro-LEDs at different currents.

Table 1. Treatment of samples.

Sample	Methods
S1	Untreated
S2	1 mol/L KOH 40 °C water bath
S3	1:1 HCl 40 °C water bath
S4	400 nm SiO ₂ Passivation layer
S5	KOH + HCl
S6	KOH + SiO ₂ Passivation layer
S7	HCl + SiO ₂ Passivation layer
S8	KOH + HCl + SiO ₂ Passivation layer

3. Results and Discussion

As shown by the optical microscope in Figure 1a, sector-shaped GaN-based Micro-LEDs were fabricated. The structure diagram can be found in Figure 1b. The cross-section SEM image is shown in Figure 1c, where the side wall of LED was covered with a SiO₂ layer. Figure 1d shows the PL spectra of 10 µm Micro-LEDs from S1 to S8. The PL spectra mainly contained two components. The first component is the luminescence of the InGaN quantum well, which forms the main peak in the PL spectra. The other component is the luminescence of the stress-release layer below the quantum well, which forms a smaller peak in the PL spectra with a slightly longer wavelength than the main peak. The presence of the stress-release layer made the PL spectra appear to be non-symmetric. Our quantum well consists of 11 periods, each with a width of 125 Å. The theoretical luminescence is generally corresponding to the results we actually measured. According to the PL spectra, it is clearly that the improvement of luminescence of S8 is the most obvious after passivation, which means the passivation method of S8 is the best in the eight groups.

Figure 2a demonstrates the room temperature PL spectra of Micro-LED samples before and after passivation. For 5 µm-radius samples, the emission intensity was enhanced by the factor of 5.6; for 10 µm samples, the emission intensity was increased by the factor of 10.5 after passivation. It is clear that passivation treatment could obviously reduce the edge effect and significantly improve the luminous intensity of Micro-LEDs.

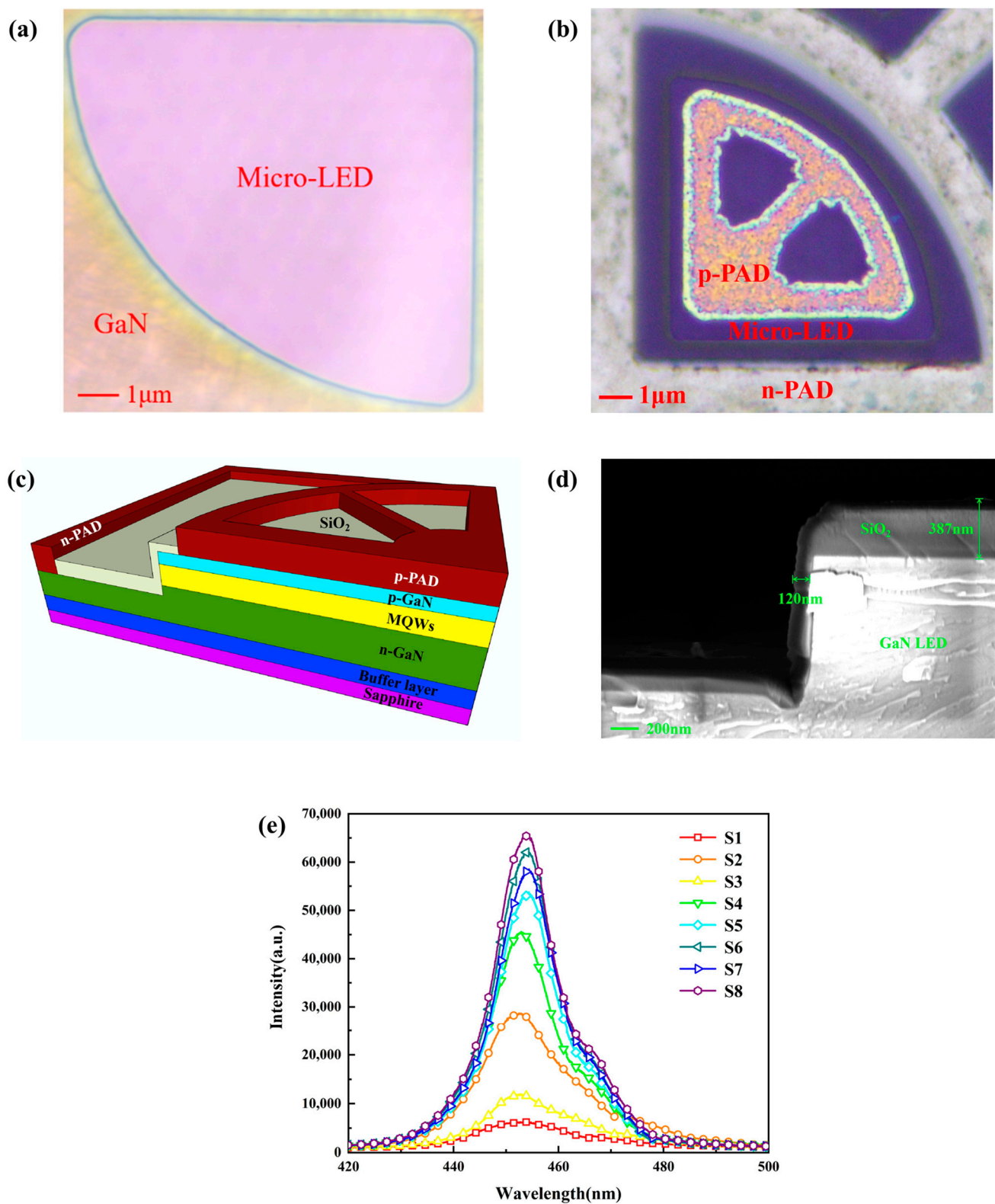


Figure 1. (a) Plan view of Micro-LED without electrodes; (b) plan view of Micro-LED with electrodes; (c) structure diagram; (d) SEM image of cross-section; and (e) photoluminescence (PL) spectra of 10 μm-radius Micro-LEDs from S1 to S8.

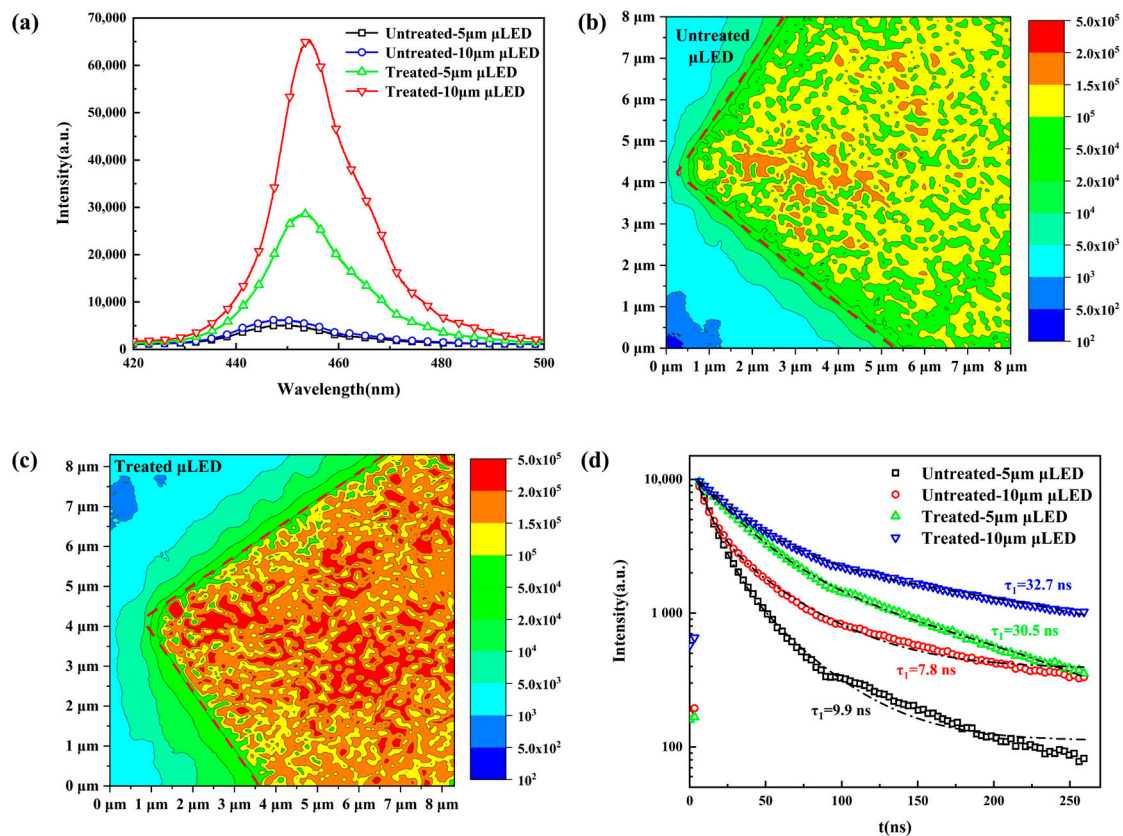


Figure 2. (a) PL spectra at room temperature; (b) PL mapping of 10 μm untreated Micro-LED; (c) PL mapping of 10 μm treated Micro-LED; and (d) time-resolved photoluminescence (TRPL) spectra at room temperature. The fitting curves are shown as black dot-dash lines in the figure.

Figure 2b,c illustrate the PL mapping images of 10 μm -radius Micro-LED with and without passivation. All samples share the same measurement parameters. The emission intensity of PL mapping images was divided into 10 levels from 100 to 500,000 in a.u. and marked with different colors. The boundaries of Micro-LED chip are marked by red dash lines in the diagram. Comparing these PL mapping images, it can be found that the passivated Micro-LED had a significantly larger area with luminous intensity above 150,000 in a.u. (orange area). Additionally, more area of high luminous intensity above 200,000 (red area) could be found in passivated Micro-LEDs. In addition, the emission of passivated Micro-LED show more uniform distribution in intensity, which was a benefit for display application. According to the integration of PL intensity, the emission of passivated Micro-LED was about 49% higher than that of the untreated samples. For the emission area with luminous intensity above 100,000 (yellow region), it was estimated that the boundary of the passivated Micro-LED had about 5% expanded area than the untreated samples. Obviously, the SiO_2 passivation layer could effectively increase the luminous intensity of Micro-LED and improve the uniformity of luminescence, on the other hand, inhibit the degradation of the luminous performance of Micro-LED devices caused by sidewall damage during ICP etching. It might be mainly attributed to the large number of dangling bonds and physical defects in the side walls of untreated samples [5]. These defects and hanging bonds are prone to become surface states, which can form leakage channels when exposed to air, resulting in a decrease in the luminescent performance of devices [6]. When the sample is treated by wet etching, the surface state and defect density are reduced, and the device luminous performance is improved. However, the sidewall stability of the sample after acid-base treatment is relatively poor, which will be easy to degenerate again when exposed to air, resulting in an insignificant actual passivation effect [7]. The usage of the SiO_2 passive layer can not only effectively reduce the defect of side wall and density of

surface states, but also can separate the device from the environment, which further protect the side wall and improve the performance and reliability of the Micro-LEDs.

Figure 2d shows the TRPL results of two size samples with and without passivation. The TRPL spectra of the sample can be fitted with a biexponential function, that is, the luminescence intensity $I(t)$ can be expressed as [8]:

$$I(t) = A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2) \quad (1)$$

Among them, A_1 and τ_1 are fast decay components and A_2 and τ_2 are slow decay components [8]. In previous studies, the carrier recombination in Micro-LED are mainly dominated by nonradiative recombination at room temperature, so τ_1 can be approximated considered as the carrier nonradiative recombination lifetime, and $1/\tau_1$ is the nonradiative composite probability [9]. The results show that the non-radiation recombination lifetime of 5 μm Micro-LED increased from 9.9 to 30.5 ns, and that of 10 μm Micro-LED increased from 7.8 to 32.7 ns after passivation treatment. This indicates that the optimized passivation process can inhibit the nonradiative recombination of carriers in Micro-LEDs due to the reduced impurities and defects density on the surface of side walls in Micro-LED. It is believed that the reduced non-radiation composite centers will improve the luminescence efficiency of Micro-LED.

For Micro-LED with a small chip size, there are many reports that demonstrated the great decrease in internal quantum efficiency (IQE), which restricts the usage of Micro-LEDs in many applications. Normally, IQE refers to the ratio of the number of photons generated in the active region of the LED per unit time to the number of electron-hole pairs injected into the LED. In general, the internal quantum efficiency η_{IQE} can be expressed as [10]:

$$\eta_{\text{IQE}} = \frac{\eta_r}{\eta_r + \eta_{\text{nr}}} \quad (2)$$

Among them, η_r and η_{nr} represent radiation recombination efficiency and non-radiation recombination efficiency, respectively [10]. In previous reports, the temperature-dependent PL was usually used to estimate the IQE of LED [11–13]. It is based on the assumption that the radiation recombination mechanism of carriers is dominant under low temperature conditions (below 20 K), where the non-radiative recombination can be ignored [14]. Under low temperature, the IQE of LED can be considered as 100% [15,16]. Therefore, IQE of LED under room temperature can be estimated by the ratio of PL integrated intensity at room temperature (300 K) and that at low temperature, that is, IQE can be expressed as [14]:

$$\eta_{\text{IQE}} = \frac{I_{\text{PL}}(\text{RT})}{I_{\text{PL}}(\text{LT})} \quad (3)$$

where $I_{\text{PL}}(\text{RT})$ represents the integrated PL intensity at room temperature, and $I_{\text{PL}}(\text{LT})$ represents the integrated PL intensity at low temperature.

Figure 3a–d shows the temperature-dependent PL (TDPL) spectra of 5 μm and 10 μm Micro-LEDs with and without passivation. For 5 μm Micro-LED without passivation, IQE is only 18.3%. After passivation, the IQE was increased to 26.9%. Meanwhile, for 10 μm Micro-LED, the IQE was increased from 14.9% to 37.6% after passivation. It is clear that the optimized passivation process could greatly increase the IQE of Micro-LEDs. It is because the passivation treatment could effectively inhibit the non-radiative recombination of carriers in the Micro-LED, leading to the improvement the radiative recombination efficiency of the carriers in the active region. Thus, optimized passivation process is very helpful for improvement of the device performance and expanding the applications of Micro-LED.

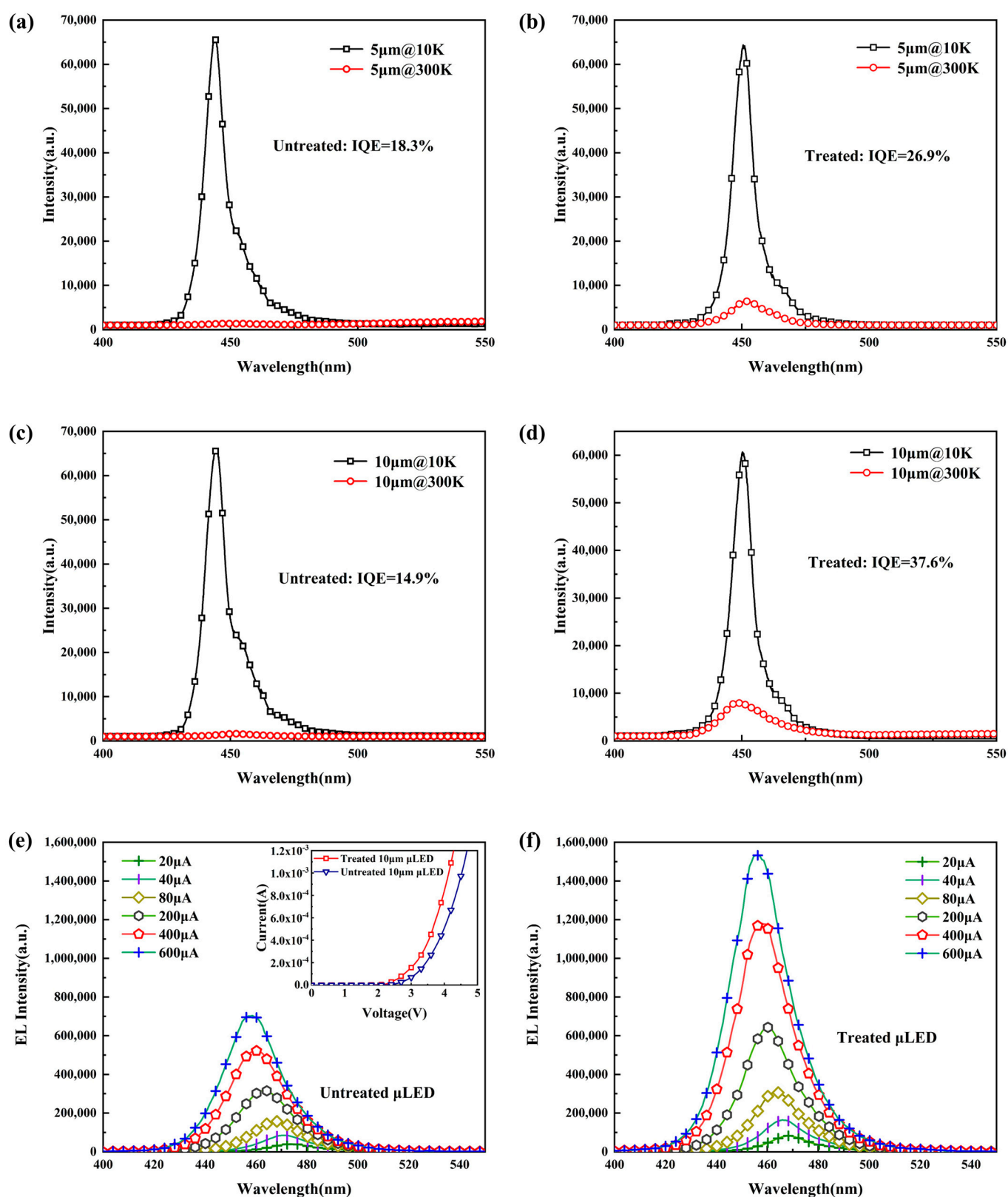


Figure 3. (a) TDPL spectra with the corresponding internal quantum efficiency (IQE) values of 5 μm untreated Micro-LED; (b) TDPL spectra with the corresponding IQE of 5 μm treated Micro-LED; (c) TDPL spectra with the corresponding IQE of 10 μm untreated Micro-LED; (d) TDPL spectra with the corresponding IQE of 10 μm treated Micro-LED; (e) electroluminescence (EL) spectra of 10 μm untreated Micro-LED, and the inset in (e) is the current–voltage (IV) curves of 10 μm Micro-LED before and after passivation; and (f) EL spectra of 10 μm treated Micro-LED.

Figure 3e–f shows the electroluminescence (EL) and current–voltage (IV) spectra of 10 μm Micro-LEDs with and without passivation. Comparing these two figures, it can be found that the EL intensity of the treated sample was approximately doubled compared to that before passivation at the same current, and the threshold voltage of Micro-LED reduced. This result indicates that the passivation treatment effectively reduced the non-radiative recombination and significantly improved the luminescence efficiency of the Micro-LED. This result was also consistent with the PL results described in the previous section. EL results once again demonstrated the importance of passivation treatment for improving the luminescence performance of small-sized Micro-LEDs from an electrical perspective.

4. Conclusions

In this article, for the sake of improving the performance of Micro-LEDs, passivation process including acid-base wet etching and SiO_2 passivation layer growth was investigated and optimized. PL spectra show that for 10 μm samples, the emission intensity was increased by the factor of 10.5 after passivation. PL mapping results show that the emission of passivated Micro-LED was about 49% higher than that of the untreated samples, and the boundary of the passivated Micro-LED had about 5% expanded area than the untreated samples. The TRPL results show that the non-radiation recombination lifetime of 10 μm Micro-LED increased from 7.8 to 32.7 ns after passivation treatment. TDPL results show that the IQE of 10 μm Micro-LED increased from 14.9% to 37.6% after passivation. EL results show that the EL intensity of the treated sample was approximately doubled compared to that before passivation at the same current. With these data, we could calculate that the non-radiative recombination is suppressed by about 20% after passivation treatment. All these results indicate that the radiation recombination efficiency of carriers was improved after passivation, and the internal quantum efficiency of Micro-LED were increased significantly. It is because passivation process can reduce the non-radiative recombination center, thereby inhibiting the non-radiative recombination of carriers in Micro-LED and improving the luminescence efficiency, which can expand the applications of Micro-LEDs.

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