Light Extraction Efficiency Enhancement of III-Nitride Light-Emitting Diodes by Using 2-D Close-Packed TiO_2 Microsphere Arrays

Xiao-Hang Li, Peifen Zhu, Guangyu Liu, Jing Zhang, Renbo Song, Yik-Khoon Ee, Pisist Kumnorkaew, James F. Gilchrist, and Nelson Tansu

***Abstract*—The enhancement of light extraction efficiency of InGaN quantum well light emitting diodes (LEDs) was achieved by employing the refractive index matched TiO_2 microsphere arrays. The optimization studies of the dipping method and rapid convective deposition (RCD) method were carried out for the deposition of TiO_2 microsphere arrays onto LEDs. The two-dimensional (2D) close-packed TiO_2 microsphere arrays were deposited by the using optimized conditions of the dipping and RCD methods, respectively. The light extraction efficiencies of LEDs under electrical injection were enhanced by 1.8–1.9 times by utilizing 520-nm diameter amorphous and anatase TiO_2 microspheres via the two deposition methods.**

***Index Terms*—III-Nitride, light-emitting diodes, light extraction efficiency, refractive index matching, TiO_2 microspheres.**

I. Introduction

THE III-Nitride semiconductors are of great importance applications in solid state lighting [1]–[17], diode lasers [18]–[23], thermoelectricity [24], [25], and solar energy conversion [26]–[29]. Blue- and green-emitting light-emitting diodes (LEDs) are primarily based on GaN based device technologies by employing InGaN quantum wells (QWs) active regions. The external quantum efficiency of LED devices is determined by both the internal quantum efficiency and extraction efficiency. Various approaches by engineering the InGaN QW structures and barrier layers have been pursued in order to enhance the internal quantum efficiency [5]–[15], which is determined by both radiative and current injection efficiencies. Novel growth methods for achieving low dislocation density InGaN/GaN material are also important for achieving high radiative efficiency from the nitride LEDs [16], [17].

The optimization of the light extraction efficiency in nitride LEDs is of great importance for achieving large external quantum efficiency in nitride LEDs. One of the main limitations related to conventional visible InGaN QW LEDs is the narrow light escape cone (24°) at the planar emitting surface that leads to low light extraction efficiency (4%) due to the large refractive index discrepancy between GaN ($n = 2.5$) and free space ($n = 1.0$). Recently, a number of approaches have been implemented for enhancing the extraction efficiency of InGaN QW LEDs, including approaches employing surface roughening [30]–[33], sapphire microlenses [34], oblique mesa sidewall [35], nanopyramid [36], photonic crystal [37]–[40], graded refractive index [41], [42], self-assembled lithography p-GaN patterning [43], GaN micro-domes [44], [45], and TiO_2 micro-pillars [46].

The surface roughening approaches utilize chemical etching, while the other approaches such as sapphire microlenses and photonic crystals employ e-beam lithography or holography lithography. Thus the non-uniformity or high-cost issues associated with these processes become obstacles for large-scale and low-cost production applicable for LEDs. Therefore, the low-cost, large-scale and easily controlled approaches for enhancing the extraction efficiency in nitride LEDs are required for commercialization.

Our recent works by employing self-assembled two-dimensional (2-D) close-packed SiO_2/polystyrene (PS) microlens arrays on top of nitride LEDs have resulted in low-cost and large-scale approach to enhance light extraction in LEDs [47]–[50]. As compared to other deposition methods such as spin coating which resulted in uniformity within relatively small region (mm $\times$ mm) scale [51], [52], the rapid convective deposition (RCD) method could easily lead to highly uniform monolayer deposition in wafer scales. In [46]–[49], the use of SiO_2/PS microlens arrays had resulted in $\sim$2.5 times increase in extraction efficiency over that of planar nitride LEDs [47]–[50]. The refractive indices of SiO_2 and PS in the visible spectrum are 1.58 and 1.46, respectively. Hence the SiO_2/PS monolayer microlens arrays on the emitting surface of InGaN QW LEDs can provide graded refractive index transition between GaN and air, which

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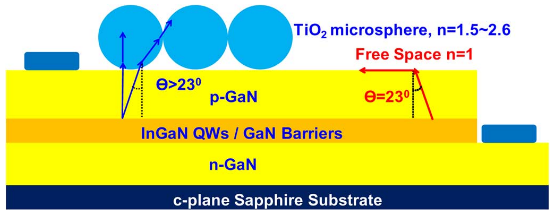

Fig. 1. Schematic of InGaN QWs LEDs utilizing TiO_2 microsphere arrays on the top surface of LEDs.

leads to the reduced Fresnel reflection and enlarged photon escape cone. In addition, a recent approach by employing concave microstructure arrays LEDs had also been demonstrated [53].

However, the refractive indices of SiO_2/PS microlens are still considerably smaller ($n \sim 1.5$–1.6) than that of GaN ($n \sim 2.5$). Thus, in order to optimize the coupling of the light from the GaN LEDs into the microlens or microstructure arrays, the microlens or microstructures are preferably formed by materials with refractive index matched to GaN. The optimized coupling of light from GaN into the microlens arrays with index matched to GaN will enable the optimization of the light extraction efficiency of the nitride LEDs.

In this work, we present detailed experimental studies on the use of self-assembled 2-D TiO_2 monolayer microsphere arrays to enhance the light extraction efficiency of InGaN QW LEDs. The refractive index of TiO_2 in the visible spectrum ranges from 1.5 to 2.6 [54], [55], depending on the crystalline phase and synthesis methods. Thus the implementation of TiO_2 microspheres provides higher indices that can match that of GaN which results in larger light coupling from GaN to the TiO_2 microspheres arrays, which in turn results in increase in LED light extraction efficiency, in comparison to those of LEDs with SiO_2 microspheres, as shown in Fig. 1. Note that the current works focus on the deposition optimization studies of 2-D TiO_2 microsphere arrays on nitride LEDs, without the use of any PS nanospheres/layers required to form the microlens arrays as described in [47]–[50].

The types of TiO_2 microspheres used in this study are amorphous ($n = 1.8$) and anatase ($n = 2.5$), respectively, with the same diameter of 520 nm. The dipping method and the rapid convective deposition (RCD) method were employed to deposit the amorphous and anatase TiO_2 microsphere arrays onto the LEDs, respectively. The electroluminescence (EL) output-power versus current density measurement was performed to investigate and compare the light extraction efficiencies of the LEDs with TiO_2 microsphere arrays deposited by the two methods. The LED far-field EL measurements were also performed to understand the influence of TiO_2 microsphere arrays on the far-field emission patterns and total output power.

The effect of refractive index of the microspheres on the extraction efficiency of the LED devices has been investigated by using 3-D finite-difference time-domain (FDTD) method [56], and these results were compared with that of planar LED. Fig. 2(a) shows the device schematics of InGaN/GaN MQW LED with hexagonal-close-packed (HCP) microsphere arrays (with diameter $d = 500$ nm) used in the simulation. The LED devices are considered as three-dimensional (3-D) structure solved by vectorial method with perfectly matched layer (PML) boundary conditions employed for the bottom and sides of the

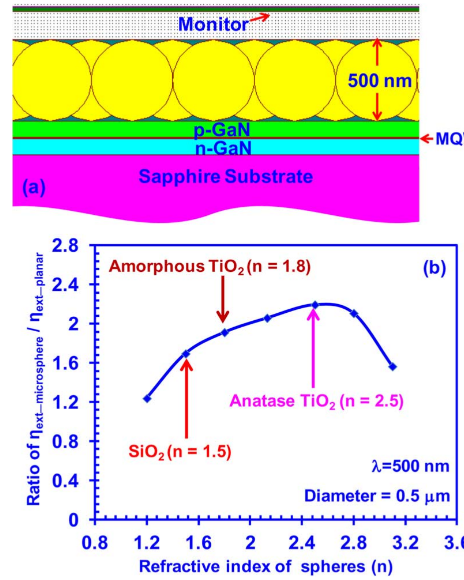

Fig. 2. (a) Schematic of InGaN/GaN MQW LED with hexagonal-close-packed (HCP) microsphere arrays. (b) Ratio of the light extraction efficiency of microsphere LEDs with various refractive indices of the microspheres. The maximum enhancement is obtained at refractive index matching condition of $n = 2.5$ for anatase TiO_2.

LEDs. The emission wavelength of the investigated LEDs is 500 nm and grid sizes are 2.4 nm and 10 nm in the MQW region and the rest of domain, respectively. The light extraction efficiency were calculated as the ratio of the optical output power collected by the monitor placed above the LED devices to the total output power generated in the InGaN/GaN MQW active region. Fig. 2(b) shows the light extraction efficiency enhancement ratio as a function of refractive index (n) of 500 nm microspheres. From the simulation, the ratio of light extraction efficiency of microsphere LEDs to that of planar LEDs was observed to reach maximum around $\mathrm{n} = 2.5$ which corresponds to the refractive index of anatase TiO_2 microspheres. This is due to the refractive index matching condition with the GaN epilayer underneath the microsphere, and the light escape cone can be significantly increased by the use of amorphous ($n = 1.8$) and anatase ($n = 2.5$) TiO_2 microsphere arrays.

II. Deposition Studies of TiO_2 Microsphere Arrays by Dipping Method

The dipping method was employed for the deposition of amorphous TiO_2 microsphere arrays. Fig. 3 shows the process flow of TiO_2 monolayer microsphere arrays deposition via the dipping method [55] on top of the III-Nitride LEDs. The deposition was performed in the class-1000 clean room to ensure the clean surface morphology. Prior to deposition, standard optical lithography was used to cover the n- and p-metal contacts of the LEDs by photoresist (Shipley 1813), such that the TiO_2 microspheres on the metal contact regions can be lifted off after the deposition. In addition, the sample surface would become hydrophilic after the photolithography process, so an uniform layer of aqueous TiO_2 suspension coated over the whole wafer can be obtained. Firstly, 520-nm diameter amorphous TiO_2 microspheres were dispersed into DI-water to form the suspension with a volume fraction of 5%, which was then immersed in the ultrasonic bath for 5 minutes. Afterwards

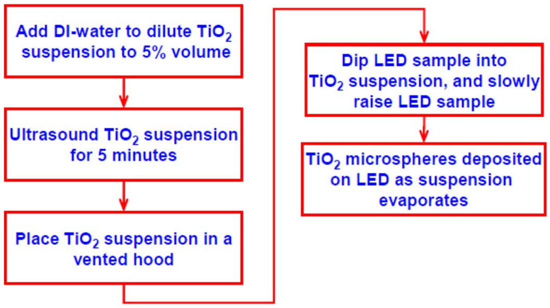

Fig. 3. Process flow of depositing 520-nm diameter amorphous TiO_2 microspheres arrays on InGaN QWs LEDs via the dipping method.

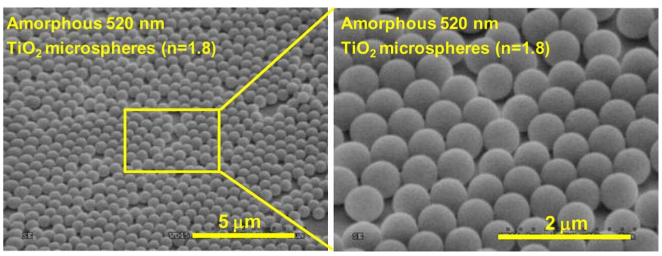

Fig. 4. SEM images of 520-nm amorphous TiO_2 microspheres deposited via the dipping method with optimized condition on the emission surface of InGaN QW LEDs with (a) lower magnification and (b) higher magnification.

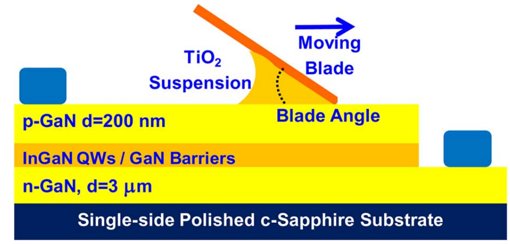

Fig. 5. Schematic of the experimental setup of rapid convective deposition method for the deposition of TiO_2 microsphere arrays on InGaN QW LEDs.

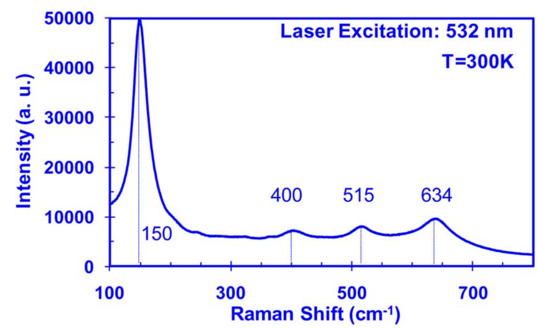

Fig. 6. Raman spectrum (532 nm) of TiO_2 microspheres used in the RCD.

the InGaN QW LED sample was dipped into the suspension and then slowly raised out. The deposition experiment was carried out under the vented hood for faster water evaporation on the LED surface. As the water evaporated, the surface pressure caused the amorphous TiO_2 microspheres to attach onto the LED surface to form the arrays. To investigate the surface morphologies of TiO_2 microsphere arrays, the scanning electron microscopy (SEM) [Hitachi 4300] measurements were performed after the deposition. Fig. 4(a) and 4(b) shows 45°-tilted SEM images of the TiO_2 microsphere arrays on top of InGaN QW LEDs with two different magnifications, indicating that the amorphous TiO_2 microspheres formed 2D monolayer and relatively close-packed arrays as shown by utilizing the optimized condition of the dipping method.

As observed in the experiments and Fig. 4(a) and 4(b), the TiO_2 microspheres deposited by the dipping method led to short-range and relatively close-packed monolayer arrays, which hinder the further improvement of light extraction efficiency. Though the relatively close-packed arrays can be accomplished by dipping method, the optimized conditions are relatively challenging for large-scale controllable process. In order to achieve long-range and close-packed monolayer arrays in controllable method, the RCD method needs to be implemented.

III. Deposition of TiO_2 Microsphere Arrays by Rapid Convective Deposition Method

To obtain long-range and close-packed TiO_2 microsphere arrays, the rapid convective deposition (RCD) method was used for the deposition of TiO_2 microspheres. Fig. 5 shows schematic of experimental setup of RCD for the deposition of anatase TiO_2 microsphere arrays. Similarly, the RCD was also performed in the class-1000 clean room and the standard optical lithography was used to cover the n- and p-metal contacts of the LEDs by photoresist for lifting off TiO_2 microspheres afterwards. Prior to deposition, the TiO_2 microsphere water suspension was immersed in the ultrasonic bath for 1 hour and then were thoroughly shaken by the vortex for 1 minute. Previously, the RCD method was employed for the deposition of SiO_2/PS microlens arrays [47]–[50], and hence the experimental setup of RCD for the TiO_2 microspheres is similar to that for the SiO_2/PS microlens arrays. However, the deposition of the TiO_2 microsphere arrays was found to require more optimization than that of the SiO_2/PS microlens arrays, which was attributed to the less mature synthesis process of TiO_2 microspheres, as compared to that of SiO_2 microspheres, leading to relatively poor monodispersity of the size of TiO_2 microspheres. The monodispersity of the microspheres was found to be critical to achieve fully close-packed and defect-free microsphere array in our experiments, therefore the future development of TiO_2 microsphere synthesis can reduce difficulty in depositing TiO_2 microspheres by RCD method.

As the synthesis of TiO_2 microspheres is less mature, the crystalline phase and the refractive index of the TiO_2 microspheres used in the RCD deposition were characterized by Raman spectroscopy measurement at room temperature. The details of the Raman spectroscopy measurement can be found in [54]. As shown in Fig. 6, the TiO_2 microspheres gave rise to strong Raman bands at 150 cm^{-1}, 400 cm^{-1}, 515 cm^{-1}, and 634 cm^{-1}, indicating TiO_2 microspheres consisted of crystals of anatase TiO_2 [58] with the index of 2.5 that matches that of GaN.

The surface morphologies of deposited TiO_2 microsphere arrays by RCD method are affected by the deposition temperature, humidity, blade hydrophobicity, volume fraction of TiO_2 suspension, blade speed, and blade angle. The RCD experiments were performed in the class-1000 clean room with invariable temperature (25 °C) and humidity (45%). The glass-based

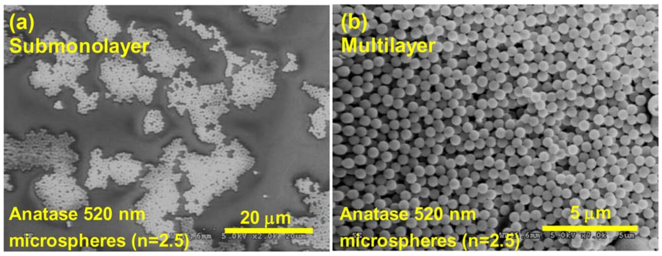

Fig. 7. SEM images of TiO_2 microspheres deposited by RCD method on the emission surface of InGaN QW LEDs with unoptimized conditions leading to (a) submonolayer and (b) multilayer of TiO_2 microsphere arrays.

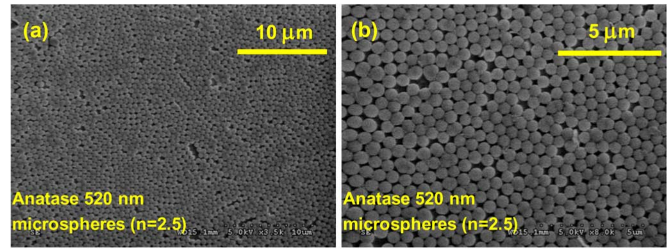

Fig. 8. SEM images of 520-nm anatase TiO_2 microspheres deposited via the RCD method on the emission surface of InGaN QW LEDs with (a) lower magnification and (b) higher magnification.

blades were covered by a layer of parafilm (Parafilm M Laboratory Wrapping Film) to ensure the hydrophobic of the surface. Therefore, the volume fraction of TiO_2 suspension, blade speed, and blade angle are the three most important factors for optimizing the deposition condition. In our RCD experiments, the blade angle was fixed at 55° while the blade speed and volume fraction were adjusted in order to obtain the optimized condition that leads to monolayer and close-packed TiO_2 microsphere array. To investigate the surface morphology of TiO_2 microsphere arrays, the SEM measurements were again performed after the deposition. Fig. 7(a) and 7(b) show the SEM images of 520-nm anatase TiO_2 microsphere array deposited by the RCD method with unoptimized deposition conditions. As shown in Fig. 7(a), the lower volume fraction or faster blade speed in comparison to the optimized conditions led to the submonolayer morphology of TiO_2 microsphere arrays. Fig. 6(b) shows that the higher volume fraction or slower blade speed in comparison to the optimized conditions resulted in the multilayer morphology of TiO_2 microsphere arrays.

Fig. 8(a) and 8(b) shows the SEM images of 520-nm anatase TiO_2 microsphere array deposited by the RCD method with optimized conditions. The optimized deposition conditions for monolayer 2-D close-packed TiO_2 microsphere arrays were obtained for the following condition: volume fraction of 10%, blade speed of 12 μm/s, and blade angle of 55°. As shown in Fig. 8(a) and 8(b), the post-deposited TiO_2 microspheres form large-scale, monolayer and close-packed arrays by employing the optimized RCD condition. The point defects in the microsphere arrays can be attributed to the poorer monodispersity of the TiO_2 microsphere diameter.

IV. Experimental Results of Electroluminescence Studies for Nitride LEDs

A. Characteristics of Nitride LEDs With Amorphous TiO_2 Microsphere Arrays

The electroluminescence studies (EL) were carried out for the following device configuration as follow: 1) planar LEDs (as reference), and 2) LEDs with 520-nm amorphous TiO_2 microsphere arrays deposited by the dipping method. To investigate the performance of InGaN QW LEDs with and without TiO_2 microsphere arrays, the on-wafer continuous wave (CW) power measurements were performed at room temperature. For the studies with amorphous TiO_2 microsphere arrays, the nitride LEDs consisting of 4 periods of InGaN QW with GaN barriers emitting at 450 nm were used. All LEDs structures studied

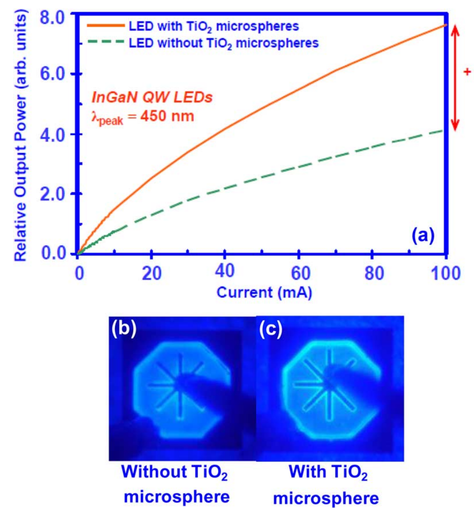

Fig. 9. (a) Light output power vs current for InGaN QW LEDs emitting at 450 nm with and without 520-nm amorphous TiO_2 microsphere arrays deposited via the dipping method. Micrograph images of LED (b) without and (c) with 520-nm amorphous TiO_2 microsphere arrays at current of 100 mA.

here were grown on 3.0 μm n-GaN template on c-plane sapphire substrates. The n-GaN was Si-doped with doping level of 5×10^{18} cm^{-3}. The p-GaN was grown utilizing 200 nm thick Mg-doped GaN with doping level of 3×10^{17} cm^{-3} at 970 °C, followed by annealing at 780 °C in the N_2 ambient. The LED devices were fabricated as top-emitting hexagonal devices with the device area of 7.5×10^{-4} cm^2, and Ti/Au as n-contact and Ni/Au as p-contact were evaporated followed by contact annealing.

Fig. 9(a) shows the light output power as a function of current injection for the 450-nm emitting InGaN QW LEDs with and without 520-nm amorphous TiO_2 microsphere arrays deposited by the dipping method. As shown in Fig. 9(a), the CW power-current measurements exhibit 1.85 times improvement in the output power of the LED device with TiO_2 microspheres arrays at the current level of 100 mA, as compared to that of planar LEDs. The significant increase in the output power can be attributed to enlarged light escape cone between GaN $(n = 2.5)$ and amorphous TiO_2 microspheres $(n = 1.8)$ as well as reduced Fresnel reflection due to the existence of amorphous TiO_2 microspheres as the intermediate index material between GaN and free space $(n = 1)$.

Fig. 9(b) and 9(c) shows the *in-situ* micrograph images of the LEDs operating at a current injection level of 100 mA without and with 520-nm amorphous TiO_2 microsphere

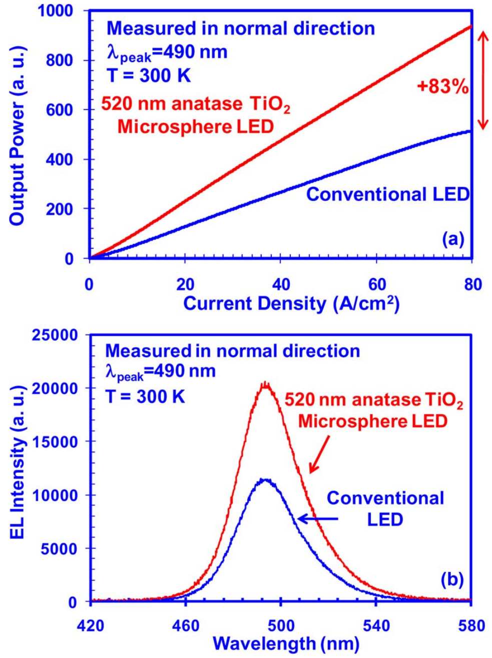

Fig. 10. (a) Light output power vs current density and (b) EL spectra ($J = 80\ \mathrm{A/cm^2}$) of InGaN QW LEDs emitting at 490 nm with and without 520-nm anatase TiO_2 microsphere arrays deposited via the RCD method.

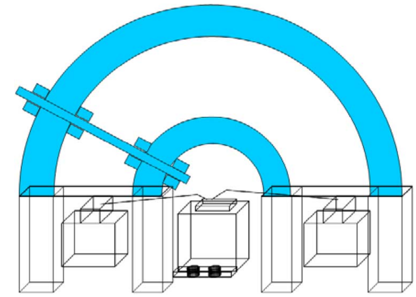

Fig. 11. Schematic of LED far-field measurement set up employed in the radial far-field EL measurements.

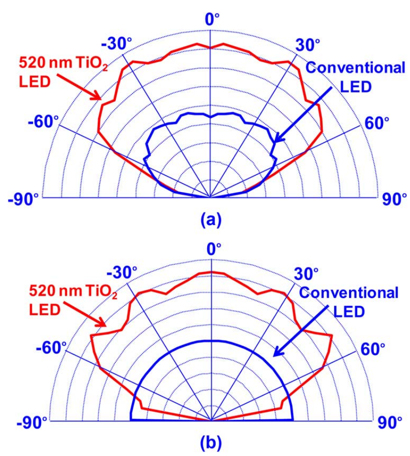

Fig. 12. (a) Far-field emission patterns of the LEDs with and without and 520-nm anatase TiO_2 microsphere arrays deposited by the RCD method, and (b) enhancement ratio of output power as a function of far-field angle. The far-field radiation patterns of the LEDs were measured from emission angle from $\theta = 0^\circ$ up to $\theta = 90^\circ$. Note that the results for $\theta = -90^\circ$ up to $\theta = 0^\circ$ range, which are presented for completeness purpose, are identical with those of $\theta = 0^\circ$ up to $\theta = 90^\circ$ range.

arrays deposited by the dipping method, respectively. The photomicrographs were taken with the same camera setting with f-number f/2.8 and shutter speed of 1/320 of a second. As shown in Fig. 9(b) and 9(c), it is clearly observed that higher intensity light emission is coupled out in the top emitting InGaN QWs LED with TiO_2 microspheres arrays.

B. Comparison of Planar LEDs and LEDs With Anatase TiO_2 Microsphere Arrays

In order to provide comparison of the EL characteristics of planar LEDs and LEDs with anatase TiO_2 microsphere arrays deposited by RCD method, the InGaN QW LEDs employing 4 periods of InGaN QW with GaN barriers emitting at 490 nm were used. All the n-GaN template and p-GaN contact layers were grown with similar growth conditions as described previously. The LED devices were also fabricated as top-emitting hexagonal devices with the device area of $7.5 \times 10^{-4}\ \mathrm{cm^2}$. The on-wafer CW power measurements were performed at room temperature.

Fig. 10(a) shows the light output power as a function of current density for the 490-nm emitting InGaN QWs LEDs with and without 520-nm anatase TiO_2 microsphere arrays. As shown in Fig. 10(a) and 10(b), the CW power-current and EL spectra measurements exhibit 1.83 times improvement in the output power of the LED device with TiO_2 microspheres arrays at the current density of 80 $\mathrm{A/cm^2}$, as compared to that without microspheres arrays. The significant increase in the output power can be primarily attributed to significantly enlarged light escape cone between GaN ($n = 2.5$) and anatase TiO_2 microspheres ($n = 2.5$).

To study the far-field emission patterns of LEDs with TiO_2 microsphere arrays, the power measurement was performed at the current density of 80 $\mathrm{A/cm^2}$ on the 450-nm emitting InGaN QW LEDs with the device area of $6.75 \times 10^{-4}\ \mathrm{cm^2}$. The far-field emission patterns of the LEDs with and without 520-nm anatase TiO_2 microsphere arrays deposited by the RCD method were measured. As shown in Fig. 11, the far-field measurement setup consisted of a semicircular structure on which the optical fiber can be rotated 180° in a single plane. The measured LEDs were placed in the center of the semicircular structure, hence the optical fiber kept equidistant away from the measured LED as the optical fiber rotated through 180° in the plane to collect the emission at varying angles (Fig. 10). The distance between the optical fiber and measured LEDs was kept at 2.5 cm which was much larger than the dimension of the measured LEDs (161 μm), in order to ensure sufficient large distance for the far-field measurements [59]. In our current studies, the far-field radiation patterns of the LEDs were measured from emission angle from $\theta = 0^\circ$ up to $\theta = 90^\circ$.

As shown in Fig. 12(a), the output power of the emission is radially plotted, with 0° as normal to the LED emitting surface. Fig. 12(a) shows that the far-field emission pattern of the LED with TiO_2 microsphere arrays exhibits overall stronger radiant intensity over that of the conventional LED. Hence TiO_2 microsphere arrays significantly increased the light extraction efficiency from widely enhanced light far-field pattern. From Fig. 12(a), the measured far-field emission of the TiO_2 coated

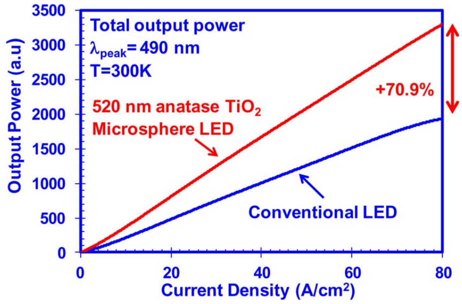

Fig. 13. Comparison of integrated light output powers as a function of current density ($J = 80$ A/cm^2) of InGaN QW LEDs emitting at $\lambda_{\rm peak} \sim 490$ nm employing 520-nm anatase TiO_2 microsphere arrays deposited via the RCD method, as compared to planar LEDs.

LEDs was much larger than that of the conventional LED. However, the enhancement of the far-field emission at varying angles was different. To illustrate this, the far-field emission of the TiO_2 coated LEDs was normalized to that of the conventional LED for obtaining the enhancement ratio of output power as a function of far-field angle, as shown in Fig. 12(b).

Fig. 12(b) shows that the far-field enhancement ratio of the TiO_2 coated LED is more significant at lower angles from 0° to 60° than that at higher angles from 60° to 90°. From Fig. 12(b), three peaks of enhancement ratio at 0°, 30° and 50° are observed, indicating that the output power enhancement brought by TiO_2 microsphere arrays is directionally dependent.

Based on Fig. 12(a), the total output power of the LEDs with and without TiO_2 microsphere arrays was obtained by integrating the far-field output power at all the angles. The total output power of the LED with 520-nm anatase TiO_2 microsphere arrays exhibits ~71% enhancement [Fig. 13], in comparison to that of the conventional LED. It is important to note that the enhancement of total output power is smaller than 83% enhancement of the output power measured in the normal direction [Fig. 10]. The decrease in the enhancement is attributed to the nonuniform enhancement ratio especially the reduced enhancement ratio at higher angles from 60° to 90°, as shown in Fig. 12(b).

V. Conclusion

In summary, the optimization studies of the deposition of 520-nm diameter amorphous and anatase TiO_2 microsphere arrays were performed by employing the dipping method and the RCD method, respectively. Both the EL in the normal direction and far-field measurements were carried out on InGaN QW LEDs with and without TiO_2 microsphere arrays. The experimental results show that the implementation of TiO_2 microsphere arrays provide a low-cost, large-scale and easily controlled process for increasing the light extraction efficiency (~1.8–1.9 times), indicating the compatibility of this approach for low-cost and high-efficiency solid state lighting.

The increase in light extraction efficiency from the use of anatase 520-nm TiO_2 microsphere arrays [Fig. 10(a)] and amorphous 520-nm TiO_2 microsphere arrays [Fig. 9(a)] on top of nitride LEDs are in the order of 1.83–1.85 times. The enhancements from the TiO_2 structures are higher in comparison to that of LED structure employing only 500-nm SiO_2 microsphere ($n = 1.5$) monolayer arrays deposited by the RCD method (~1.69 times) [48]. Further enhancement of extraction efficiency is expected with microlens structures [44]–[46] by introducing planar materials (i.e., polystyrene) which results in semi-buried TiO_2 microlens arrays, which is expected to result in higher light coupling from GaN LEDs into the TiO_2/PS microlens arrays in comparison to the SiO_2/PS microlens arrays [47], [48], [50].

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