Fabrication and Characterization of Micro-membrane GaN Light Emitting Diodes

Thesis by

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The thesis of Hsien-Yu Liao entitled “Fabrication and Characterization of Micro-membrane GaN Light Emitting Diodes” is approved by the examination committee.

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ABSTRACT

Fabrication and Characterization of Micro-membrane GaN Light Emitting Diodes

Hsien-Yu Liao

Developing etching of GaN material system is the key to device fabrications. In this thesis, we report on the fabrication of high throughput lift-off of InGaN/GaN based micro-membrane light emitting diode (LED) from sapphire substrate using UV-assisted photoelectroless chemical (PESc) etching. Unlike existing bandgap selective etching based on unconventional sacrificial layer, the current hydrofluoric acid based wet etching process enables the selective etching of undoped GaN layer already incorporated in standard commercial LED structures, thus attaining the leverage on high performance device design, and facile wet process technology. The lift-off micro-membrane LED showed 16% alleviated quantum efficiency droop under 200 mA/cm² current injection, demonstrating the advantage of LED epitaxy exfoliation from the lattice-mismatched sapphire substrate. The origin of the performance improvement was investigated based on non-destructive characterization methods. Photoluminescence (PL) characterization showed a 7nm peak emission wavelength shift in the micro-membrane LED compared to the GaN-on-Sapphire LED. The Raman spectroscopy measurements correlate well with the PL observation that a 0.86 GPa relaxed compressive biaxial strain was achieved after the lift-off process. The micro-membrane LED technology enables further heterogeneous integration for forming pixelated red, green, blue (RGB) display on flexible and transparent substrate. The development of discrete and membrane LEDs using nano-fiber paper as the current spreading layer was also explored for such integration.
ACKNOWLEDGEMENTS

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I would also like to thank my family for their constant support in my study even if I am far away from home in Saudi Arabia. Meanwhile, I am grateful for my cheering friends and badminton players here who participating extracurricular events with me to enrich my life. I would thank my dearest lady, Gaohong Yin, who has supported me when depressed and shared lots of sweet memory here at KAUST.
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<tr>
<td>CRI</td>
<td>Color Rendering Index</td>
</tr>
<tr>
<td>EL</td>
<td>Electroluminescence</td>
</tr>
<tr>
<td>EQE</td>
<td>External Quantum Efficiency</td>
</tr>
<tr>
<td>FIB</td>
<td>Focused Ion Beam</td>
</tr>
<tr>
<td>FWHM</td>
<td>Full Width at Half Maximum</td>
</tr>
<tr>
<td>IQE</td>
<td>Internal Quantum Efficiency</td>
</tr>
<tr>
<td>ITO</td>
<td>Indium Tin Oxide</td>
</tr>
<tr>
<td>LED</td>
<td>Light Emitting Diode</td>
</tr>
<tr>
<td>MBE</td>
<td>Molecular Beam Epitaxy</td>
</tr>
<tr>
<td>MOCVD</td>
<td>Metal Organic Chemical Vapor Deposition</td>
</tr>
<tr>
<td>MQW</td>
<td>Multiple Quantum Well</td>
</tr>
<tr>
<td>μPL</td>
<td>Micro Photoluminescence</td>
</tr>
<tr>
<td>PL</td>
<td>Photoluminescence</td>
</tr>
<tr>
<td>QCSE</td>
<td>Quantum Confined Stark Effect</td>
</tr>
<tr>
<td>QW</td>
<td>Quantum Well</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>SSL</td>
<td>Solid-state lighting</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission Electron Microscope</td>
</tr>
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1. Introduction

1.1 Challenges and advancement in LED technologies

In the past twenty years, the energy consumption globally has risen up to a historical climax. The idea of green energy begins to bloom. Several issues have been addressed such as, hybrid and electric vehicles are invented to reduce the oil consumption, solar panels and wind electric generators are introduced to compensate the traditional fossil-fuel power plant...etc. In this thesis, we address one of the largest energy consumption, the lighting. Nowadays, the major backlight units in display screen have changed into the lighting emitting diode (LED) to reduce the energy consumption and increase the luminous efficacy. The transparent flexible paper-based display serves as an active device to achieve less energy consumption in display for lighting in handheld consumer electronics. The history of lighting unit development is provided as follows.

Since Thomas Edison first introduced the carbon filament incandescent light bulb in 1879, this type of lighting has dominated the entire world for more than 100 years. With its high competitive advantages, such as low cost for manufacturing, convenient in tuning the light intensity, great color rendering index (CRI), and mercury-free filling etc., the incandescent light bulbs have suppressed the other lighting alternative until 1976. Even though Edward Hammer, an engineer in General Electric, invented the compact fluorescent lamp (CFL) during the oil crisis in 1973, the helical CFL only became commercially available in 1995.
While in the past ten years, solid-state lighting and display using LED has drawn much public attention. There are many different materials that can be used for electroluminescence (EL) emission. In this thesis, we focus on the group III-V (III-V) semiconductor alloys. The first III-V based visible wavelength emission (red) LED was developed at General Electric Company by Nick Holonyak, Jr. in 1962. Not until mid-90s, the first high-brightness blue LED based on InGaN was demonstrated at Nichia Corporation by Shuji Nakamura in 1994. This high-brightness blue LED is later combined with the yellow emission phosphor Y₃Al₅O₁₂:Ce (or simply YAG) to generate the white light LED. The invention of white light LED has opened up the field of solid-state lighting. With its low energy consumption, extensively long lifetime, and potential for visible light communication, high-brightness LEDs have drawn huge attention in the past ten years. We will systematically list the pros and cons of these three different types of lighting, incandescent light bulb, CFL, and LED in the following paragraph.

The major thrust of the revolution in lighting is simply the goal of reducing the unit energy consumption, i.e. increasing luminous efficacy, which is using the same amount of electricity to generate more light output. The traditional incandescent light bulb is a perfect light emitter in terms of simple device physics. At the same time, the incandescent light bulb serves as perfect heat emitter, which converts most of the electricity into heat during the lighting period. The low luminous efficacy leads to the replacement of incandescent light bulb. With the relative low wall plug efficiency, the incandescent light begins to lose its popularity in the market.
The invention of CFL is a temporary solution to energy-efficient lighting. The reason why LEDs have not yet displaced CFLs from the market are twofold: the first generation LED bulbs had a narrow and focused light beam, and the cost of the LED bulbs was too high. From Eartheasy.com [1], we clearly compare the efficiency and cost of three different types of lightings in Table 1-1. In terms of luminous efficacy, incandescent light bulb only performs 15 (lm/W) under 60 watts electricity consumption.

<table>
<thead>
<tr>
<th></th>
<th>Incandescent</th>
<th>CFL</th>
<th>LED</th>
</tr>
</thead>
<tbody>
<tr>
<td>Luminous efficacy (lm/W) (60 watts)</td>
<td>15</td>
<td>50</td>
<td>115</td>
</tr>
<tr>
<td>Watts per bulb (equiv. 60 watts)</td>
<td>60</td>
<td>14</td>
<td>10</td>
</tr>
<tr>
<td>Light bulb projected lifespan (hours)</td>
<td>1,200</td>
<td>10,000</td>
<td>50,000</td>
</tr>
<tr>
<td>Cost per bulb</td>
<td>$1.25</td>
<td>$3.95</td>
<td>$35.95</td>
</tr>
<tr>
<td>Bulbs needed for 50k hours of use</td>
<td>42</td>
<td>5</td>
<td>1</td>
</tr>
<tr>
<td>Equivalent 50k hours bulb expense</td>
<td>$52.50</td>
<td>$19.75</td>
<td>$35.95</td>
</tr>
<tr>
<td>Cost of electricity (@ 0.10per KWh)</td>
<td>$300</td>
<td>$70</td>
<td>$50</td>
</tr>
<tr>
<td>Total cost for 50k hours</td>
<td>$352.50</td>
<td>$89.75</td>
<td>$85.75</td>
</tr>
</tbody>
</table>

Table 1-1. Efficiency related comparison of incandescent light bulb, CFL, and LED.

In Mar 2014, Cree, Inc. just announced the state-of-the-art achievement of breaking 300 lumens-per-watt barrier in high-power white LED [2], which is equivalent to 20 times more efficient than the incandescent light bulb. In other aspects such as projected light bulb lifespan and total cost for 50k lighting hours of use, LED has dominantly conquered the game. In Table 1-2, the characteristics of incandescent light bulb, CFL, and LED are listed showing the benefits of LED over the traditional lighting.
<table>
<thead>
<tr>
<th></th>
<th>Incandescent</th>
<th>CFL</th>
<th>LED</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequent On/Off Cycling</td>
<td>some effect</td>
<td>shortens lifespan</td>
<td>no effect</td>
</tr>
<tr>
<td>Turns on instantly</td>
<td>yes</td>
<td>slight delay</td>
<td>yes</td>
</tr>
<tr>
<td>Durability</td>
<td>fragile</td>
<td>fragile</td>
<td>durable</td>
</tr>
<tr>
<td>Heat Emitted</td>
<td>high (85 Btu/hr)</td>
<td>medium (30 Btu/hr)</td>
<td>low (3 Btu/hr)</td>
</tr>
<tr>
<td>Sensitivity to low/high temperature</td>
<td>no / no</td>
<td>yes / yes</td>
<td>no / some</td>
</tr>
<tr>
<td>Sensitivity to humidity</td>
<td>some</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>Hazardous Materials</td>
<td>none</td>
<td>5 mg mercury/bulb</td>
<td>none</td>
</tr>
</tbody>
</table>

Table 1-2. Characteristics related comparison of incandescent light bulb, CFL, and LED.

Albeit LED has several advantages, its high unit price is still the major concern in the market. In January 2012, Osram has demonstrated high-power InGaN LEDs grown on silicon substrates commercially [3] which is speculated that the low cost of silicon wafers epitaxy processes could reduce production costs up to 90% [4].

1.2 III-Nitride (III-N) material properties and its devices

This section introduces the material properties of the crystalline III-V semiconductors in LED manufacturing. Gallium nitride (GaN) is a binary III-V compound semiconductor with a direct bandgap, which makes this material perfect in both electronics and optoelectronics application. Advanced processing of GaN electronic device, bipolar transistors, has been reported [5]. As in the optoelectronics application, III-N based materials are widely used for high-power blue and green LED with the introduction of Indium (In) to reduce the alloy bandgap energy. GaN and InGaN are the building blocks
of nitride-based optoelectronic devices. Having the Wurtzite crystal structure, this material has different polar crystalline planes as depicted in Figure 1-1.

![Figure 1-1. Wurtzite structure of GaN crystal, noted that the polar plane (c-plane) and non-polar plane (a-plane and m-plane) are colored, from [6].](image)

Due to the noncentrosymmetrical Wurtzite crystal structure, III-N based material has a strong built-in spontaneous polarization field. Especially in the c-plane grown structure, with the large lattice mismatch from sapphire substrate to AlN, GaN, InN layers, the spontaneous polarization field becomes stronger due the strain accumulation in the crystal. Therefore, the space charges induced by spontaneous and piezoelectric polarization fields exist at the interfaces of heterostructure for III-N based materials. In the c-plane InGaN/GaN multiple quantum wells (MQWs) structure, this polarization-induced electric field was called quantum-confined stark effect (QCSE). This property results in an effective bandgap narrowing and a Stark shift in the excitonic absorption [7]. The QCSE within MQWs region bends the energy bands and leads to a spatial separation
of the electron and hole wave functions, which leads to a decrease in the transition probability, and then enhances nonradiative recombination from the QW [8, 9]. Thus it has resulted in the reduction of quantum efficiency for the III-N based LEDs [10, 11].

Conventionally, III-N based devices epitaxially grow on sapphire substrate. Unlike III-As based devices, III-N based devices are short of native single crystal substrate (not cost-effective) and lack of lattice and thermal coefficient matched substrates. Many threading dislocations and strains exhibit in the c-plane grown III-N based devices on sapphire. Despite its large lattice mismatch with GaN shown in Figure 1-2, sapphire is still the most commercially available substrate. In contrast to SiC, sapphire wafers are considered cost-effective with good thermal and chemical stability, and hence is widely used for the growth of III-N LEDs. As a wide bandgap material, sapphire is transparent to the visible light suitable for back emission.

![Figure 1-2. Lattice constant and the energy gap information for III-V semiconductors.](image-url)
In III-N based LED, there is a major unsolved problem, the efficiency droop. This effect suppresses the high-power LED performance under high injection current. Before discussing this phenomenon, several terminologies in calculating the efficiency in LED field are listed below in Table 1-3:

<table>
<thead>
<tr>
<th>Type</th>
<th>Definition</th>
<th>Symbol</th>
<th>Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Injection Efficiency</td>
<td>Proportion of electrons passing through the device that are injected into the active region.</td>
<td>$\eta_{\text{INJ}}$</td>
<td>$\eta_{\text{INJ}} = \frac{I_{\text{active}}}{q}$</td>
</tr>
<tr>
<td>Radiative efficiency</td>
<td>Ratio of the radiative recombination rate to the total recombination rate.</td>
<td>$\eta_{\text{RAD}}$</td>
<td>$\eta_{\text{RAD}} = \frac{P_{\text{active}}/h\nu}{I_{\text{active}}/q}$</td>
</tr>
<tr>
<td>Internal quantum efficiency (IQE)</td>
<td>Ratio of the number of the photons emitted from the active region to the number of electrons injected into a LED.</td>
<td>$\eta_{\text{IQE}}$</td>
<td>$\eta_{\text{IQE}} = \eta_{\text{INJ}} \times \eta_{\text{RAD}} = \frac{P_{\text{active}}/h\nu}{I/q}$</td>
</tr>
<tr>
<td>Light Extraction efficiency (LEE)</td>
<td>Measure of the photon losses during the propagation from the active region into the free space.</td>
<td>$\eta_{\text{LEE}}$</td>
<td>$\eta_{\text{LEE}} = \frac{P_{\text{out}}/h\nu}{P_{\text{active}}/h\nu}$</td>
</tr>
<tr>
<td>External quantum efficiency (EQE)</td>
<td>Ratio of the number of photons emitted from the LED to the number of electrons passing through the device.</td>
<td>$\eta_{\text{EQE}}$</td>
<td>$\eta_{\text{EQE}} = \eta_{\text{LEE}} \times \eta_{\text{IQE}} = \frac{P_{\text{out}}/h\nu}{I/q}$</td>
</tr>
<tr>
<td>Voltage efficiency</td>
<td>Measure of the electrical potential energy loss during electron transport from a battery to the active region of a LED.</td>
<td>$\eta_{\text{VTG}}$</td>
<td>$\eta_{\text{VTG}} = \frac{h\nu}{qV}$</td>
</tr>
<tr>
<td>Power-conversion (wall-plug) efficiency</td>
<td>Efficiency of converting electrical power to optical power.</td>
<td>$\eta_{\text{wall-plug}}$</td>
<td>$\eta_{\text{wall-plug}} = \eta_{\text{VTG}} \times \eta_{\text{EQE}} = \frac{P_{\text{out}}}{IV}$</td>
</tr>
</tbody>
</table>

Table 1-3. Efficiency terminology in LED, from [12].
At the early stage of developing the InGaN/GaN based device, the efficiency is positively proportional to the injection current due to the relatively low injection current. The efficiency (IQE) droop phenomenon occurs under high injection current condition. In [13], the efficiency droop is discussed and investigated. Two possible mechanisms are proposed. In [14], both debating mechanisms are discussed, internal nonradiative losses (Auger recombination) and electron leakage. Auger recombination is a high-order carrier dependent term in the total recombination process, which is the most straightforward, high carrier-density, internal, nonradiative process. In electron leakage model, polarization fields in the MQW active region and the electron blocking layer (EBL) enhances the leakage of injected electrons into the p-type GaN cladding layer, and thus cause the efficiency droop [13]. Despite having been the subject of extensive research efforts for a decade, the physical origin of droop has not been clarified [14].

From this efficiency droop, a serious obstacle is raised, the green gap. Green LED suffers more severe efficiency droop than other visible LED. In Figure 1-3, the trends result in very low luminous efficacy in the green spectral range, often referred to as the green gap [15]. Thus, the gap must be closed by improving green light emission so as to develop the solid-state lighting solutions for high-quality spectral quality and promise substantial energy saving.
Figure 1-3. Luminous efficacy of InGaN based devices and AlInGaP based devices. It shows in the green region, the luminous efficacy performs poor, while Osram offers solution in the 540nm region to close the green gap [16].

In order to solve the two issues stated above, possible solutions have been proposed. H. Masui suggests that the non-polar or semi-polar plane oriented devices be the solution to better device performance [10]. Meanwhile, true bulk GaN is generally regarded as the ideal substrate for GaN-based devices [17]. In this publication, the high quality ammonothermal crystal growth for bulk GaN substrates is reported.

Investigating the spontaneous and polarization-induced field on InGaN/GaN MQWs heterostructure is an important issue currently. Many novel and useful growth techniques and structures have been developed to release the strain and reduce the separation of the wave function in the MQW region to improve the optical properties of III-N based LED
In this thesis, we have utilized the chemical lift-off process to achieve strain relaxation and proved to reduce the separation of the wave function.

1.3 Epitaxy Lift-off techniques

GaN is commonly grown on lattice-mismatched (13.8%) and thermal-mismatched sapphire substrate. In addition, sapphire has a relatively poor heat conductivity of 42 W/mK compare to that of GaN (253W/mK) [19, 20]. Thus threading dislocation nucleation and poor heat dissipation constitute to sub-optimal III-N LED device performance, when they are grown on sapphire remains an issue [21]. This drives the scientific efforts to develop versatile and high yield post-growth substrate removal or epitaxy lift-off technologies for III-N based devices and structures. Generally, epitaxial lift-off of III-N epi-structures relies on laser lift-off (LLO) and chemical lift-off (CLO) techniques. The epitaxial lift-off process smoothly de-bonds the desired layers and finally produces III-N based LED on substrates with more desirable thermal and electrical characteristics than sapphire.

In the commercial available LLO, the epi-layer is lifted off by an excimer laser sending high energy pulses to dissociate GaN into gallium droplet and N₂ gas. The high energy pulses create longitudinal pressure \( P_L \), and transverse tensile stress \( P_T \) in the material. However, the propagating shock wave may end up cracking the epitaxy if the fabrication steps are not design properly. For a lateral crack, it is acted upon by both tensile stress (\( P_T \)) and shear stress (the horizontal part of \( P_T \)) shown in Figure 1-4 [22]. Due to the high energy laser pulses, local temperature may increase up to 1000 degree Celsius. The
disadvantages of LLO are as follows, increase in dislocation density, resulting in an increase of the leakage current [23], and damaged areas containing residual droplets of material produced by the LLO process [24]. The LLO process requires careful handling and is already in production [25, 26].

![Laser lift-off process and principle using KrF 248nm excimer laser.](image)

In CLO, there are several advantages that excel the LLO process, such as low-cost, scalability, reliability in terms of process environment…etc. In this thesis, we will focus on the CLO technique which is based on a wet etching process of sacrificial layer and have proven to offer a high yield over large area [27]. By distinguishing the etching mechanisms, wet etching of semiconductors can generally be divided into two categories, namely electrochemical etching (including anodic etching, electroless etching, and photoelectrochemical (PEC) etching), and chemical etching including conventional etching in aqueous etchants and defect-selective etching in molten salts [28]. The etching
of GaN under normal conditions is found with only molten KOH or NaOH at 250 degree Celsius [21]. Thus, mostly the GaN selective etching is done under the PEC etching scheme. Currently the existing CLO techniques rely on the regrowth of the III-N device structure on extra costly sacrificial layers, such as InGaN/GaN MQW, porous GaN, Si, SiO$_2$ and ZnO [29] [30] [31] [32].

1.4 Principle of photoelectrochemical etching

The PEC etching is used as the key experimental method in this thesis. We have applied the electroless PEC etching, i.e. PEsC etching. Conventionally, PEsC etching process consists of the following three steps in etching the semiconductor: (1) Oxidation of the semiconductor surface, (2) Subsequent oxides dissolution, (3) Excess electrons reduces oxidizing agent and consumes in the electrolyte.

Neither an external voltage bias nor electrical contact to the samples is required in the PEsC etching. The chemical potential energy difference in the electrolyte and semiconductor drives the etching process. The electrolyte serves as an oxidizing agent which depletes valence-band electrons in the semiconductor with excess holes accumulated.

Gratzel et al. have reported the physics of PEsC in [33], with the discussion of the proposed model. The example is given in the case of aqueous solution of NaCl in Figure 1-5. At semiconductor-electrolyte interface, due to the energy difference of semiconductor bandgap and redox potential, three layers are formed, space charge region
(SCR), Helmholtz layer (LZ), and Gouy layer (GL). The SCR is formed inside a semiconductor when dipped into a solution. The SCR is a function of the doping level of the semiconductor and surface states. The HL is considered to be formed by two planar sheets of charges. One is due to the ions in solution adsorbed at the surface of the solid (OH$^-$ in this example), the other is due to the ions of opposite sign (Na$^+$) attracted by adsorbed ions. GL describes a region in the solution near the electrode within which there is a space charge due to an excess of free ions of one sign [34].

![Diagram of semiconductor layers](image)

Figure 1-5. Three layers formation in semiconductor dipped into solution, from [34].

In Figure 1-6 (a) (b), the band diagram of the interface of KOH (similar in HF solution) with different doping type GaN clearly shows the possibility in selectively etching of n-GaN over p-GaN. For n-type semiconductor, the electrostatic potential at the surface is negative. The energy bands bend upward, and the concentration of electrons at the surface is reduced to form a depletion layer [35]. The pinned Fermi-level ($E_F$) of n-type
A semiconductor causes an upward band bending near the surface. The surface depletion region arises due to the potential difference established by the $E_f$ at equilibrium. At the surface, electrons concentration is then reduced, and holes concentration is increased.

Figure 1-6 (c) explains the etching mechanism of photo-assisted etching, where the electron-hole pairs are generated in the semiconductor under above bandgap illumination. The photogenerated holes are accumulated at the interface between n-type GaN and electrolyte due to the energy band bending. These photogenerated holes assist in the oxidation of the semiconductor surface and the excess electrons are consumed by the reduction of the oxidizing agent in the electrolyte.

A competing process in PEsC etching is the recombination of electrons and holes. The PEsC etching only happens when the excess electrons in photo-assisted PEsC etching are consumed by the reduction reaction instead of reducing the oxidizing agents.
Figure 1-6. The band diagram at the interface of electrolyte with (a) n-GaN (b) p-GaN shows the accumulation of holes in n-GaN [36]. (c) Etching mechanism of photo-assisted etching [28].

1.5 Literature review and motivations of work on PEC etching

The selective etching of GaN has been an issue in research due to its chemical inactive characteristic. With the aid of PEC etching, the selective in GaN system was first reported by Minsky [37], later explored by Youtsey [36]. The PEC etching in GaN system is categorized into the following classes, bandgap selective, dopant selective, defect selective, and the doping concentration selective etching. Examples of the respective works are provided later in Figure 1-7. In this thesis, we modified the doping concentration selective in order to achieve the selective etching of undoped GaN over the n-type GaN.

Literature review on the development of GaN system PEC work is shown in Figure 1-7, a timeline perspective since the seminal work by Youtsey and Minsky. Highlighted work descriptions in the timeline are listed as follows:
(a) Selective etching of n-GaN over p-GaN is achieved based on dopant selective etching [36].

(b) Applying the GaN optical filter (bandgap selective etching) and increasing the KOH molar concentration, the inverted pyramids in the n-GaN layer, which is useful for light extraction [38].

(c) Nanoporous GaN can formed – the characteristic of large area / volume ratio is promising in sensing applications [39].

(d) GaN membrane prepared by bandgap selective etching, which is sandwiched between all-dielectric Distributed Bragg Reflector (DBR) microcavity for Vertical-Cavity Surface-Emitting Laser (VCSEL) fabrication [40].

(e) Air/semiconductor VCSEL with nonpolar m-plane surface using thermal decomposition of GaN [41].

(f) A 25nm thin GaN nanomembrane from 30um GaN grown on sapphire is fabricated using PEsC etching process (defect selective etching) [42].

(g) UCSB 2-step PEC for m-plane VCSEL utilizing all-dielectric DBRs. One of the PEC step is used to undercut a sacrificial layer to remove and therefore recycle m-plane substrate [43].

(h) Large area exfoliation utilizing n-GaN dopant selective etching [44].

(i) We recently develop a process to selectively etch the undoped GaN over the n-type GaN (doping concentration selective etching). Strain relief and alleviation in efficiency droop was observed [45].
Figure 1-7. Literature review and timeline perspective for the PEC related work.
Transparent flexible paper-based display is the ultimate goal in the field of hand-held display. In order to achieve the flexible membrane, we have detached the LED pixel from the low thermal and electrical conductivity sapphire substrate. The lift-off blue membrane LED fabricated in this thesis enables the possibility in manufacturing the flexible paper-based display. We fully characterized the lift-off LED to understand the device characteristics and performances. With the improvement in efficiency droop and its flexibility, the LED pixel is proposed to integrate with the nanofiber paper to form the unit pixel of flexible display. The end product opens up the field of transparent flexible paper-based display, which can be utilized for press printing, medical treatment, and substitution for the current rigid mobile phone display.

1.6 Thesis outline

A general overview of the thesis and the literature review of III-nitride material as well as research objectives, motivations and contributions are presented in chapter 1. In chapter 2, we will introduce general background information of the LED characterization tools and their working principals. The detailed fabrication process and experimental setup will be discussed with the photoelectroless chemical etching (PEsC) mechanism.

In chapter 3, the membrane LED characterization will be provided with both experimental and simulation results. The physical observation and analysis under SEM and optical microscope are discussed. The compressive strain relaxation is verified by the Raman spectroscopy; meanwhile, the device simulation under various strain schemes is completed and simulated using Nextnano. There is an interesting anomaly transition
discovered in temperature-dependent PL, power-dependent PL, and EL with discussion in this chapter. Eventually, the time-resolved PL analysis for the carrier lifetime is included. In chapter 4, the electric characteristics of membrane LED is discussed with the observation of alleviation in efficiency droop. Possible explanations are provided and discussed under the compressive strain relaxation observation in chapter 3. Besides, the concept of the membrane LED integration is claimed. With the prototype of embedded red LED in nanofiber paper, the concept is realized. The future applications and possibility are listed as well. It is possible to integrate the membrane LED for the future flexible hand-help display. In chapter 5, the conclusion and summary of the thesis is made. With the outlook and future applications, the thesis is concluded.

1.7 Significant research contributions

- Understood and investigated the origin of alleviated quantum efficiency droop in lift-off membrane LED based on correlation of PL and Raman spectroscopy.
- Fully characterized the lift-off membrane LED and reported the device performance.
- Achieved the compressive strain relaxation in lift-off membrane LED compared to the relaxed GaN condition.
- Simulated strained and relaxed LED devices to support the experiment by Nextnano.
- Measured the etching selectivity of undoped GaN over the n-GaN.
- Solved contact issue for the membrane LED with soft annealing technique.
- Embedded the red LED into the flexible, transparent, and conducting nano-fiber paper for prototype demonstration.
2. Experimental Methods and Details

2.1 Overview of GaN based light emitting diodes (LEDs) fabrication

The LED operates based on the radiative recombination of electrons and holes at the p-type and n-type semiconductor interface. The energy is released in the form of photons according to the bandgap energy of the semiconductor. In Figure 2-1, a simple model of p-n junction under different bias voltages is illustrated with the band alignment in semiconductor. Applying forward bias reduces the built-in potential in the LED; thus, electrons and holes are able to drift faster into the interface to recombine. Applying reverse bias increases the depletion region width in the p-n junction. This results in faster sweeping of the photogenerated carriers into the depletion region which is the photodiode (PD) working regime.

\[
\begin{align*}
\text{Forward bias} & \quad E_F, p, n \quad q(\phi_i - V_a) \\
\text{Reverse bias} & \quad E_F, p, n \quad q(\phi_i - V_a)
\end{align*}
\]

Figure 2-1. The band diagram of (a) Forward bias (b) Reverse bias in the LED, from [46].

q, \(\Phi_i\), Va, \(E_F\) (p,n) stands for electron charge, built-in potential, external applied bias, and Fermi level in p,n type semiconductors, respectively.
Based on the conventional p-n structure, the double heterostructure (DH) is commonly used in III-N based LED, which is a sandwich structure where one smaller bandgap material is sandwiched between two larger bandgap materials (p-i-n structure) as shown in Figure 2-2. By reducing the thickness of the intrinsic region to the dimension of de Broglie wavelength, the quantum well (QW) can be achieved. In the QW structure, larger bandgap material (GaN) serves as the barrier, while the smaller bandgap material serves as the well where electrons and holes are confined for the recombination.

![Figure 2-2. Conventional DH in InGaN/GaN LED, showing the p-type GaN, intrinsic (MQWs region), and the n-type GaN, from [47].](image)

In order to fabricate the LED, the p-type and n-type electrode have to be chosen properly. Since [48], the LED mesa structure has been established. The p-type electrode is deposited on top of the LED, while the n-type electrode is deposited on the n-type GaN. Due to the top-emitting mesa structure, the p-type electrode has to be transparent. In order to reduce the reverse-biased leakage current in metal-semiconductor interface, employing a Schottky metal with high work function, Ni (5.15 eV) and Au (5.1 eV) is a
common combination for maximizing the effective Schottky barrier height against p-GaN, which has a high Fermi energy of 7.5 eV [49, 50]. The thin Ni/Au (5nm/5nm) and Ni/ITO (5nm/60nm) for p-type electrode is investigated in [51]. Both combinations give relatively good Schottky contact formation on top of the p-type GaN, while the latter has a 15% better transparency in the blue emission region. Proper contact formation includes the metallization annealing process. During the metallization annealing process, the Ni transforms into NiO was reported as a layer with high hole concentration which leads to the reduction of contact resistance [52]. In [53], the AES and XPS studies is reported for the formation of NiO.

2.2 Electroluminescence and electrical characterization of LED

Electroluminescence (EL) by definition is the illumination from the material under the passage of an external voltage bias (electrical field) or injection current. This phenomenon is the result of radiative recombination in a material. In the direct bandgap semiconductor, the excess electrons and holes flow into the semiconductor, recombine and release their energy as photons without changing the crystal momentum. The photons are emitted as output light in the LEDs. The intensity of the output light is then collected by spectrometer under various injection currents. At the same time, the device electrical properties are also investigated, such as current versus voltage (I-V), and output light power versus current injection (L-I). In these characterizations, we can understand the device physics and carrier recombination mechanism in our membrane LED. The experiment setup in KAUST Photonics Lab is illustrated in Figure 2-3.
Figure 2-3. I-V, L-I and EL setup: (a) Schematic illustration, and (b) Experimental setup in KAUST Photonics Lab: Keithley 2400 source meter, Newport 2936C power meter and Ocean Optics QE65000 spectrometer, from [54].
2.3 Photoluminescence in GaN and related materials.

Other than the EL, another powerful characterization tool is the photoluminescence (PL) spectroscopy. The PL is the light emission which is directly excited under non-intrusive above bandgap light source or laser. The light emission in the material caused by the absorption of external photons shows various relaxation process when the re-radiated photons are emitted. PL provides information about the bandgap of semiconductor as well as many other intrinsic material properties. Studies of the PL can be various, such as time-resolved PL for estimating carrier lifetime in the material, temperature-dependent PL for eliminating thermal phonon vibration in the material. In Figure 2-4, the experimental setup is illustrated. A photo-detector is used to collect light emitted from the sample, a monochromator helps to disperse the wavelength, and the computer collects the luminescence signal from the photodetector.

Figure 2-4. Schematic illustration of the micro PL/Raman setup, from [54].

2.4 Probing epitaxy strain with Raman spectroscopy - principle and set-up

Similar to the PL spectroscopy, Raman spectroscopy utilizes the same experimental setup in Figure 2-4. The main difference is that Raman spectroscopy utilizes below bandgap
laser excitation. Raman spectroscopy relies on inelastic scattering, or Raman scattering, of the monochromatic laser light. In general a laser interacts with molecular vibrations in the crystalline structure, resulting in a shift of the laser photon energy. This shift gives information about the material strain, molecular polarization and crystallographic orientation. The strain in different material is characterized using the Raman spectroscopy. In this thesis, we will focus on the biaxial strain in the III-N based devices.

Different Raman signals are shown in Figure 2-5, elastic scattered radiation (Rayleigh scattering) at the corresponding incoming laser wavelength is filtered out, while the remaining Stokes and anti-Stokes Raman shifting signals are dispersed into the detector. The weak inelastically Raman scattered light is hard to distinguish from the intense Rayleigh scattered laser light. For Rayleigh scattered laser rejection, notch or band-pass filters are employed onto the Raman system.

For the inelastic Raman scattering, a photon interacts with the molecule in either the ground vibrational state or an excited vibrational state. This excites the molecule onto a virtual energy state for a short period of time before an inelastically scattered photon generates. The energy of the inelastically scattered photon shifts to either lower (Stokes) or higher (anti-Stokes) values compared to the incoming laser photon.
Figure 2-5. Energy-level diagram showing the states involved in Raman spectroscopy. The line thickness is roughly proportional to the signal strength from the different transitions, from [55].

GaN has the Wurtzite crystal structures. Group theory predicts eight sets of phonon normal modes exist in the hexagonal structures. In our Raman spectroscopy, we have the configuration of $Z(X, Y)\bar{Z}$ shown in Figure 2-6 (a). According to [56], only the $E_2$ are detectable under this geometries configuration. We are interested in the $E_2$(high) mode since it has relatively strong Raman signal and the vibration mode can be used to determine the strain exhibited in the horizontal direction as shown in Figure 2-6 (b).
2.5 Scanning electron microscopy and transmission electron microscopy

In analyzing the physical information of the devices, two microscopies are utilized: scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

Physical properties, such as the surface morphology and the layer information, are investigated by SEM. For SEM measurements, a focused beam of electrons is directed towards the sample and scans the surface. The signal produced by a SEM includes secondary electrons (topographical information), back-scattered electrons (atomic number and finer topographical information), characteristic X-rays (composition information), cathodoluminescence (electrical information), Auger electrons (surface sensitive composition information) and transmitted electrons. In our application, we only focus on the secondary electrons and back-scattered electrons signals. The information and the application of different signals produced by a SEM are shown in Figure 2-7. The
secondary electrons interact with atoms at or near the surface of the sample. The back-scattered electrons reflect back from the elastic interaction are collected by a detector and give information about the morphology and composition.

Figure 2-7. Information of different signals produced by a SEM, from [58].

For crystalline study and higher resolution physical properties, the TEM is an informative tool. TEM operates with electron beams transmitted through an ultra-thin specimen, interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen. Owing to small de Broglie wavelength of electrons, TEM is capable of imaging higher resolution images than SEM.

Contrast formation in the TEM depends greatly on the mode of operation. Generally, the most common mode of operation for a TEM is the bright field imaging mode. In this imaging mode, the contrast is simply formed by occlusion and absorption of electrons in
the sample [59]. Thicker regions of the sample will appear dark. In another commonly used diffraction contrast (dark field) imaging mode, the contrast is created with the diffraction. The incoming electron beam undergoes Bragg scattering. In the case of a crystalline sample, electrons are dispersed into discrete locations in the back focal plane. If the reflections that are selected do not include the unscattered beam, the image will appear dark.

2.6 Principle of selectively etching in undoped GaN

In chapter 1.4, we have discussed the PEsC etching mechanism which can be summarized into three steps. The photo-generated holes diffuse to the space charge region where they drift towards GaN/electrolyte surface. Accumulated holes in the space charge region at the interface are consumed by the oxidation reaction of the GaN:

\[
2\text{GaN (s)} + 6\text{h}^+ + 3\text{H}_2\text{O (l)} \rightarrow \text{Ga}_2\text{O}_3\ (\text{s}) + 6\text{H}^+ (\text{aq}) + \text{N}_2\ (\text{g})
\]

Subsequent dissolution of the resulting oxides at the GaN / electrolyte interface

\[
\text{Ga}_2\text{O}_3\ (\text{s}) + 6\text{HF (aq)} \rightarrow 2\text{GaF}_3 + 3\text{H}_2\text{O}
\]

Excess electron reduction at the Pt / electrolyte interface

\[
3\text{H}_2\text{O}_2 + 6\text{e}^- + 6\text{H}^+ \rightarrow 6\text{H}_2\text{O}
\]

In this thesis, we have modified the conventional PEC etching method [60] into backside-illuminated photoelectrolesschemical (BIPEsC) etching as our experimental method, using above GaN bandgap UV illumination from the substrate side to activate the selective etching process.
There are two factors affecting the BIPESC etching in our experiment. One is the minority carriers’ diffusion length in undoped GaN, and the other is the penetration depth of the incident light in undoped GaN. In [61], the carrier diffusion length of GaN is determined in the range of 100 to 260nm with different GaN film thickness, while in [62], 200nm hole diffusion length is reported in 1µm thick n-type GaN. In [63], the absorption of GaN is studied. The penetration depth of the incoming light with energy higher than 3.4 eV (above GaN bandgap) is less than 100nm. Both data from the literature are shown in Figure 2-8. Due to the shallow penetration depth and the short carrier diffusion length, the photo-enhanced carriers will only be generated at the interface where the PEsC etching happens. Thus, the selective etching of undoped GaN over n-type GaN can be achieved in this work.
Figure 2-8. (a) The carrier diffusion length in GaN versus GaN film thickness, and (b) the absorption coefficient in GaN versus energy [61, 63], the inset shows the detailed drop in absorption coefficient happens at 3.5eV in 77K condition.
2.7 Experimental set-up and considerations

In the experimental setup, the UV illumination and its spectral irradiance are illustrated in Figure 2-9 (a) and (b), respectively. The sample undergoes micro-fabrication process stated in chapter 2.8 before being immersed into the HF based solution to start the PEsC etching.

Figure 2-9. (a) Schematic PEsC etching UV illumination from Newport Arc Lamp Housing 66922 with the Newport 6293 1000W Hg-Xe lamp, and (b) the Irradiance versus wavelength of the Hg-Xe lamp, schematic graph acknowledged [44].
The LED epiwafer used in the experiment is shown in Table 2-1. The schematic graph of the LED is shown in Figure 2-10 (a) (b), while (c) and its inset are the secondary ion mass spectrometry (SIMS) result. The SIMS result is a further confirmation of the doping concentration in each layer of the blue LED. The layer information from the SIMS matches with the designed epi-structure. We can clearly observe the indium (In) concentration in the MQW region to determine the number of pairs and estimate the thickness of both well and barrier regions. Tailing in the Al and Mg profiles are due to fill-in of v-defects. SIMS result shows the evidence of high quality growth of 15 pairs MQW, which is shown in the inset TEM image.

<table>
<thead>
<tr>
<th>Material</th>
<th>Thickness</th>
<th>Dopant (cm$^{-3}$)</th>
<th>Layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>GaN</td>
<td>1 nm</td>
<td>Si, ~ 1x10$^{19}$</td>
<td>Tunneling contact</td>
</tr>
<tr>
<td>GaN</td>
<td>150 nm</td>
<td>Mg, &gt; 1x10$^{19}$</td>
<td>Capping / contact</td>
</tr>
<tr>
<td>AlGaN</td>
<td>100 nm</td>
<td>Mg, ~ 5x10$^{18}$</td>
<td>Electron blocking</td>
</tr>
<tr>
<td>InGaN/GaN (MQW)</td>
<td>300 nm</td>
<td>Undoped</td>
<td>15 pairs of MQW</td>
</tr>
<tr>
<td>GaN</td>
<td>50 nm</td>
<td>Si, &gt; 1x10$^{18}$</td>
<td>Cladding</td>
</tr>
<tr>
<td>AlGaN</td>
<td>50 nm</td>
<td>Si, &gt; 1x10$^{18}$</td>
<td>-</td>
</tr>
<tr>
<td>GaN</td>
<td>3 µm</td>
<td>Si, &gt; 1x10$^{18}$</td>
<td>Contact</td>
</tr>
<tr>
<td>GaN</td>
<td>1-2 µm</td>
<td>Undoped</td>
<td>Buffer</td>
</tr>
<tr>
<td>Sapphire</td>
<td>500 µm</td>
<td>-</td>
<td>Substrate</td>
</tr>
</tbody>
</table>

Table 2-1. Epitaxy layer structure of the blue LED.
Figure 2-10. (a) Blue (b) Green LED epiwafer layer contents from [54], and (c) SIMS result with TEM image of MQW in blue LED as inset.
In this thesis, the selective etching of undoped GaN over n-type GaN is achieved and subsequent devices are characterized. In order to excite carriers only in the undoped GaN layer, we utilize the SiO$_2$/SiN$_x$ DBR filter to filter out photons with energy above undoped GaN bandgap, which will be absorbed by the InGaN MQW layer. We shine the incoming light from the back of the sample to achieve the condition we discussed in chapter 2.6. At the same time, in order to control the experiment environment, we design the DBR filter in longer wavelength to stabilize the temperature in the solution. The characteristic of DBR filters is shown in Figure 2-11. The reflectivity of the DBR filter matches with the simulation design. The MATLAB program for simulating DBR is included in Appendix B. In Appendix C, the application of the DBR in laser facet coating work is stated.

The simulation of visible DBR filter for preventing the InGaN/GaN MQW from being etched during the PEsC process is based on 10 pairs of SiO$_2$/SiN$_x$ DBR targeting at center wavelength of 425nm. In the simulation, this filter has the peak reflectivity of 99.68% at 425nm shown in Figure 2-11 (a). The reflectivity at 445 nm, which is the emission wavelength of the blue LED, is still at a high value of 99.48%. This means most of the photon energy between the bandgap of GaN and InGaN has been reflected back without exciting the MQW region. The experiment result of the visible DBR filter from the UV-Vis spectroscopy shows high coherency compared to the simulation. The peak reflectivity is located at the same energy as in the simulation. In the 3 stacked DBR shown in Figure 2-11 (b), the experiment results also highly match the simulation.
Figure 2-11. (a) Experimental and simulated reflectivity of DBR filter for preventing the InGaN MQW etching, and (b) experimental and simulated short pass filter for stabilizing the temperature in the solution.
2.8 Process development

The blue and green micro-membrane LEDs are prepared using the PEsC etching process.

The schematic process flow is depicted in Figure 2-12.

The epi-wafer sample is cleaved into 1 cm² piece and cleaned in acetone and isopropanol alcohol (IPA) for 5 minutes, as well as hydrofluoric acid dipping for surface oxide removal. The conventional LED fabrication process ensues with 5nm/5nm Ni/Au p-contact deposition using e-beam evaporator, SiO₂ hard mask deposition using PECVD, and inductively coupled plasma reactive-ion etching (ICP-RIE) to reveal the LED undoped GaN layer. The pattern size is 90 x 90 µm in cross shape, with a total area of 4500 µm². The LED sample is then placed in the acid bath containing CH₃OH : H₂O₂(35%) : HF(48%) at a ratio of 1:2:2 under the illumination of the 750W mercury arc lamp. Once the desired etching period is reached, the samples are dip-cleaned in isopropanol alcohol (IPA), and dried using a critical point dryer (CPD). Detailed fabrication travel log is included in Appendix A.
Batch transfer of the micro-membrane LEDs onto foreign substrates is achieved by using a hardened silicone handler substrate, i.e. polydimethylsiloxane (PDMS). In order to have finer control in the membrane LED pixel transfer process, the 5µm size tungsten probe is used to perform single pixel control of membrane LED. After the PEsC process, the membrane LEDs are almost floating on top of the sapphire substrate with undoped GaN nano-whiskers supporting the membrane. When applying little force from the tungsten probe, easy Pick-and-Place membrane LED pixel can be achieved. The membrane LED is then being embedded into the nano-fiber paper to form a flexible display pixel.
3. Characterization of Membrane LEDs

3.1 Scanning electron microscope (SEM) characterization

The membrane LEDs have been examined under the optical microscope and scanning electron microscope (SEM). Different PEsC etching schemes are illustrated as follows. In Figure 3-1, the membrane LED is ready to lift-off, the undoped GaN layer has been etched into the shape of nano-whisker, by controlling the etchant concentration, different etching profile can be achieved [64].

![Figure 3-1](image)

Figure 3-1. (a) SEM image of the membrane LED after PEsC etching process, and (b) zoom-in view.

Merits of the membrane LED are shown in Figure 3-2. The batch processing in transferring the membrane LED onto the nano-fiber paper for flexible display as well as the single pixel LED transfer using Pick-and-Place technique performed by the 5\(\mu\)m tungsten probe can be achieved.
Figure 3-2. (a) Batch processing of membrane LED after the PEsC etching, and (b) single membrane LED pixel transfer with the dimension of the membrane pixel as inset.

There are several observations during the fabrication, such as the polarization enhanced PEsC etching in MQW, the GaN dead layer formation during the ICP-RIE process, and utilization of focus ion beam (FIB) micromachining for analysis of the membrane LED are shown in Figure 3-3.
Figure 3-3. (a) Polarization enhanced PEsC etching in MQW, (b) p-type GaN layer removal due to MQW being etched away, (c) FIB reveals the cross-section view of the membrane LED, (d) MQW has deeper etching while n-type GaN partially being etched, and (e) Cross-section side SEM image showing the detail in the MQW.
We have also observed the GaN dead layer after the ICP-RIE process shown in Figure 3-4. Lee et al. have reported in [65] the formation of GaN residue during the dry etching process. The residue can be intentionally used as the protection layer to protect MQW from the polarization enhanced etching during the PEsC etching.

Figure 3-4. (a) Dead layer of GaN during the ICP-RIE, (b) Threading dislocation in GaN residue after the ICP-RIE, and (c) Possible protection layer for the MQW.
After the batch transfer of membrane LEDs onto the PDMS substrate, we examine the nano-whiskers after the PE5C etching. In order to have a proper n-type electrode, the non-conducting nano-whiskers have to be removed. The SEM images and the removal of those nano-whiskers using FIB are shown in Figure 3-5.

Figure 3-5. (a) Undoped GaN nano-whiskers formation after the PE5C etching, (b) Nano-whiskers before FIB treatment, and (c) After FIB, the nano-whiskers are removed.
3.2 Strain reduction in membrane LED using, Raman spectroscopy

The PL on the lift-off LED and unetched LED is performed under the 325nm and 405nm laser excitation, in conjunction with 600 lines/mm gratings, 35µm pinhole size, 1 second integration time, and the excitation power density of 15.6 kW/cm$^2$. The device is characterized under different temperature and excitation power. Room-temperature PL experiment by 325nm laser in Figure 3-6 shows the peak emission wavelength blue shift as well as the strong yellow emission in undoped GaN nano-whiskers when exciting the lift-off LED from the n-side. Comparing to the p-side excited lift-off LED, the peak emission wavelength falls at similar wavelength, while the weaker yellow emission from the undoped GaN nano-whiskers due to the absorption of excitation laser in the LED MQW active layer.

The yellow emission of the undoped GaN nano-whiskers matches the SEM image introduced in Figure 3-5 (a). This yellow emission is due to the sub-bandgap recombination model involving shallow donors and deep donors in the undoped GaN [66]. Shalish believes electron transitions either from the conduction band [67], or from a shallow donor, to a deep state in the lower half of the band creating short path for the recombination in undoped GaN. The undoped GaN emission energy 2.2 eV reported in the literature, equivalent to emission wavelength at 563nm, matches the PL result we obtain.
Power dependent PL of the lift-off LED and unetched LED are conducted under the 405nm excitation laser, with the maximum excitation power density of 15.6 kW/cm². Various attenuating filters are applied to perform the power dependent PL experiment. The excitation power varies from low power to high power, 0.16 to 15.6 kW/cm². In the following paragraph, low excitation power density and high excitation power density are referred to 0.16 and 15.6 kW/cm², respectively.

At 77K low temperature, most of the joule heating induced phonon transition can be excluded. The peak wavelength of the unetched LED has a total 5nm shifting under various excitation power with the trend of red-shifting then blue-shifting. In the lift-off LED, the total peak wavelength shifts only 2.5nm. At low excitation power density, the...
peak emission wavelength of both unetched LED and lift-off LED are the same. Under high excitation power density 15.6 kW/cm$^2$, the peak emission wavelength of these two devices has a 2.3nm (15 meV) difference. This peak emission wavelength has the similar trend reported in [68]. The less peak wavelength shifting provides the indication of strain relaxation in the MQW region [69]. The trend of shifting in the lift-off LED performs a constant blue-shifting, which differs from the trend in unetched LED.

The full width at half maximum (FWHM) also shows an interesting difference between the unetched LED and the lift-off LED. In the unetched LED, the FWHM performs an increase under low excitation power, while a decrease under high excitation. The trend matches its peak wavelength shifting. In the lift-off LED, the band-filling effect is significant under high excitation power. Under high excitation power, radiative recombination from higher energy states will occur because there are more injected carriers to fill up the quantum well. It causes the PL emission blue shift and FWHM broader as the excitation power increases [70]. Being dominated by the band filling effect, the FWHM of the lift-off LED increases as the excitation power increases. The FWHM of the unetched LED has a comparative larger but consistent value compares to the lift-off LED. At low excitation power, the lift-off LED has a small FWHM. Under high excitation power, the FWHM is comparable to the unetched LED, which shows the excitation power dependent broadening exhibited in the lift-off LED. In the unetched LED, the peak emission wavelength shifts, while the FWHM keep almost constant under various excitation powers. The 77K power dependent PL experiment data are all shown in Figure 3-7.
Figure 3-7. 77K power dependent PL, FWHM and peak wavelength versus excitation power density in unetched LED (a) (b), and lift-off LED (c) (d).

Under room temperature 297K excitation, both unetched and lift-off LED performs differently. In the unetched LED, although the peak wavelength indeed performs a similar shifting trend as under low temperature excitation, the degree of shifting is only 2nm shift. We can clearly see that the peak wavelength has similar value at both high power and low power. This is the evidence of balancing of joule heating effect and the band filling effect. In the lift-off LED, the peak wavelength has a clear red shift towards longer wavelength when increasing the excitation power. This indicates the strain relaxation in the MQW which will be further supported in the following Raman
spectroscopy measurement to determine the degree of strain relaxation. This as well shows the enhanced joule heating effect in the lift-off LED, which requires a proper contact and heat sink to conduct the heat flow. The peak wavelength in the lift-off LED has a drastically red shift after the $0.78 \text{ kW/cm}^2$. This interesting transition phenomenon has drawn our attention and we have further explained with the temperature dependent PL experiment in the later chapter.

For the FWHM in both devices at high temperature, the trends are similar to the one at low temperature. The FWHM broadening is more significant compares to the one at low temperature. In both unetched LED and lift-off, the FWHM has a continuous broadening as the excitation power increases. The degree of broadening is stronger in the lift-off LED, which shifts from $14.3 \text{ nm}$ to $34 \text{ nm}$. This significant shift tells us the more severe band filling effect in the lift-off LED. The $297K$ power dependent PL experiment data are all shown in Figure 3-8.
Figure 3-8. 297K power dependent PL, FWHM and peak wavelength versus excitation power density in unetched LED (a) (b), and lift-off LED (c) (d).

Due to the lattice and thermal mismatch between GaN and c-plane sapphire substrate, the GaN grown on c-plane sapphire exhibits a biaxial compressive strain with the value of 1.2 GPa [71]. The 30° rotation of the lattice in the epitaxial growth of GaN on sapphire leads to a reduction in the lattice constant (2.75 Å) of substrate parallel to the a vector of GaN [72]. Consequently, GaN layer is expected to be compressively strained. Using the reported Poisson ratio [73], the changes in GaN lattice constant a and c under compressive strain can be determined. As shown in Figure 1-2, the lattice constant of InGaN is larger than the one in GaN. Thus, compressive strain from the GaN layer
propagates to the MQW region. As we relax the strain within the GaN layer, the strain in
MQW region also gets relaxed. The strain induced peak emission energy shifting of GaN
is investigated \[74\]. The bandgap of GaN at room temperature can be expressed
according to \( E_g = 3.4285 + 0.0211\sigma_{xx} (\text{eV}), \sigma_{xx}: \text{biaxial stress} \). The peak emission
energy preforms a blue shift when compressively strained. In the MQW region, the
unetched LED exhibits a relatively large compressive strain. After the PEsC process, we
have observed a red shift of the peak emission energy in the lift-off LED, which provides
us the information of compressive strain relaxation. As we have observed the indication
of strain relaxation in the power dependent PL experiment, we conduct the Raman
spectroscopy to determine the degree of strain relaxation in the lift-off LED.

The Raman spectroscopy is conducted under the 473nm laser excitation laser wavelength,
100X objective lens, 2400 lines/mm gratings, 100 µm laser pinhole size, and 10 s
integration time. In \[75\], the unstrained GaN has the \( E_2(\text{high}) \) phonon peak at 567.6 cm\(^{-1}\).
The unetched LED on sapphire grown by MOVPE has the \( E_2(\text{high}) \) phonon peak at 570.6
± 0.5 cm\(^{-1}\) in the experiment, while the lift-off LED has the peak at 567.5 ± 0.5 (cm\(^{-1}\)).
The equation to calculate biaxial strain from the Raman shift is as follows:
\[
\sigma = \frac{\Delta \omega}{k_R} \quad \omega: \text{wavenumber}, \ k_R: \text{Raman stress factor} = -3.6 (\text{cm}^{-1}/\text{GPa}). \ [76]
\]
From the above equation, the calculated compressive strain reduction is 0.86 GPa. The
reported strain free GaN has the \( E_2(\text{high}) \) phonon peak at 567.6 cm\(^{-1}\), so the lift-off LED
has completely relax the compressive strain, as shown in Figure 3-9. This experiment
further indicates consistency and assumption with results, such as the observation of
shorter peak emission wavelength in PL measurement, reduction of carrier lifetime in
time-resolved PL measurement, and the reduction in the efficiency droop. The supported data will be provided in the following section.

![Graph showing Raman spectroscopy data](image)

**Figure 3-9.** Raman spectroscopy for measuring the strain in the membrane LED.

### 3.3 Simulating strain with Nextnano

Due to the strong polarization field in the MQW region, electron and hole wavefunctions in the this region have a spatial separation. The spontaneous polarization field becomes stronger due the strain accumulation from the lattice mismatch between GaN and c-plane sapphire substrate. In the lift-off membrane, the compressive strain in the MQW region is relieved. Thus, it increases the electron and hole wavefunctions overlapping. The radiative recombination becomes more efficient after relaxing the strain.
In order to simulate the difference in compressively strained and relaxed devices, we utilize Nextnano as our simulation tool. The simulated device is a simplified version of 3-pair of MQW, with the 0%, -1%, -2%, -3% compressive biaxial strains value presented in the GaN layer (minus indicates compressive strains). The compressive strain in the GaN is done by changing the GaN lattice constants during the simulation. The reported GaN lattice constant $a=3.8192\ \text{Å}$, $c=5.185\ \text{Å}$, from [77]; with the Poisson ratio in GaN of 0.212 [78]. As discussed in chapter 3.2, the strain will propagate to the MQW region, where the simulation is in Figure 3-10. As from the simulation, we can tell that the MQW has a higher degree of strain due to the larger lattice constant in InGaN layer. When increasing of strain in the GaN layer, the strain in the MQW increases accordingly.

![Figure 3-10. Elastic energy density under various strain conditions.](image-url)
The energy band diagram is calculated under zero external bias and 2.8V forward bias voltage shown in Figure 3-11 (a) (b), respectively. The simulated energy band structure of the whole device is shown in the inset of Figure 3-11. The more strain in the device, the more severe band bending occurs, i.e., the bottom of the quantum well declined. As discussed in chapter 1.2, the electron and hole wave functions separate spatially due to the QCSE, resulting in the reduction of radiative recombination and IQE. When the device is relaxed, the band bending is reduced as well.

At the same time, the electron and hole wavefunctions are simulated using 1D Schrödinger-Poisson simulator provided in Nextnano. This simulator calculates the eigenvalues and eigenfunctions distribution in the device. The overlapping factor in the first quantum well has been calculated in Figure 3-12. The overlap of electron and hole wavefunctions and the probability of radiative recombination are simultaneously increased. The relaxed device has a highest electron and hole wavefunctions overlapping factor of 16.19%. In Figure 3-13 (a), we shows the spatial separation of electron and hole wavefunctions under different strain conditions. The hole wavefunction doesn’t show much difference as long as the device is strained, while the electron wavefunction shows a shift towards the edge of quantum well (outside quantum well).

Finally, we have seen a strong current density presented in the relaxed device shown in Figure 3-13 (b). This also provides the evidence of the increase in radiative recombination in the MQW region. In the simulation, we have observed the red shifting in the peak wavelength when relaxing the strain in the GaN layer shown in Figure 3-14.
This red shifting of peak wavelength matches the experimental data we obtained in the temperature dependent PL. This could be attributed to the decrease of effective bandgap when relaxing the device [79].

Figure 3-11. Band structure (first quantum well) (a) without bias, and (b) 2.8V bias.
Figure 3-12. Electron and hole wavefunctions overlapping in first quantum well.

Figure 3-13 (a) Spatial separation of electron and hole wavefunctions in first quantum well, and (b) Current density distribution in first quantum well.
3.4 Anomaly in temperature dependent PL and EL transition characteristics

The temperature dependent PL conducts under various temperatures from low temperature 77K to room temperature 297K, under the 405nm excitation laser, with the excitation power density of 15.6 kW/cm$^2$. The peak emission wavelength red shifted in the unetched LED showing the typical Varshni empirical equation, i.e. the reduction in transition energy with temperature. The lift-off LED follows the Varshni equation at low temperature region. The lift-off device shows clearly a faster peak energy shift at temperature $> 200$ K, which indicated a significant heating effect. The transition at 200 K in the lift-off LED corresponds to the previous EL and power dependent result. The sudden drop in the measurement indicates that 200K being the threshold temperature or transition temperature. If we fit only the data $< 200$K in lift-off LED, the trend will show...
the similar shifting trend as the unetched LED. The plots of temperature dependent PL of both unetched LED and lift-off LED are shown in Figure 3-15 (a) (b).

In the Figure 3-15 (c), we have extracted the information of peak energy in unetched LED and lift-off LED. In the typical Varshni empirical fitting, the parameters are explained as follows:

\[ E_g(T) = E_0 - \frac{\alpha T^2}{T + \beta} - \frac{\sigma^2}{k_B T} \]

\( E_0 \): energy gap at 0K; \( \alpha, \beta \): Varshni coefficients; \( \sigma \): degree of localization effect

In our experimental data, the \( \alpha \) and \( \beta \) fitted values match the literature [68]. Thus, the \( \sigma \) values in our fitting show the increased in carrier localization effect in the lift-off LED. As the difference in peak energy can be clearly seen in the Figure 3-15 (c), the lift-off LED exhibits a red-shift in peak energy compares to the unetched LED. It is due to the compressive strain relaxation of InGaN, which is been simulated and shown in Figure 3-14.
Figure 3-15. Temperature dependent PL of (a) unetched LED, (b) lift-off LED. (c) The Varshini equation fitting in lift-off LED and unetched LED.

3.5 Time-resolved PL for carrier lifetime measurement

In the time-resolved PL, 120 µW 355nm excitation laser, and the integration time 0.5ns are applied. Streak camera is applied in the single wavelength TR-PL experiment. As for the wavelength sweeping TR-PL using CCD, we apply the 400nm excitation laser with the 100uW excitation power. The samples we used for the time-resolved PL is the
circular 10 µm in diameter lift-off LED (with the roughen surface for light extraction) and the same size in unetched LED. Optical microscope and SEM images of the device are shown in Figure 3-16.

Figure 3-16. (a) Microscope image, and (b) SEM image of the circular lift-off LED.

In the experiment result, the reduction in radiative recombination lifetime is observed in the lift-off LED compares to the unetched LED. In Figure 3-17 (a), the TR-PL experiment data is fitted with the exponential decay model [80] to determine the radiative recombination lifetime. The recombination lifetime in the lift-off LED has a short 1.7ns value compares to the unetched LED. In Figure 3-17 (b) (c), the CCD captured TR-PL signals are plotted for the lift-off LED and the unetched LED. We observe that the signal in the lift-off LED is much weaker than the one of unetched LED. The strong signal of the unetched LED has spread over the entire scanning window of 50ns, which shows the long recombination lifetime. From the single wavelength TR-PL experiment, we know that the lift-off has very short radiative recombination lifetime. Thus, the signal in the lift-off LED decays fast in a short period of time. The peak energy in the lift-off LED has a
2nm red shift to the unetched LED, which matches the previous PL experiment data and the Raman spectroscopy.

Figure 3-17. (a) TR-PL data using single shot wavelength detection. Wavelength scanning TR-PL using CCD in (b) unetched LED, and (c) lift-off LED.
3.6 Summary

In summary, the lift-off LED has been characterized under various experimental methods. The lift-off LED can only perform the high performance with the proper heat sink. As we have separated the LED membrane from the poor heat conductive sapphire, there creates the potential to transfer the lift-off LED onto the metal heat sink for high injection current. However, we have also observed the excitation power dependent transition in the lift-off LED for both electrical and optical injection, which will be discussed in the next chapter.

Understanding the device characteristics, we have more confidence in utilizing the lift-off LED and integrated it with other material to make the potential flexible LED display. In the next chapter, the EL characteristics of the lift-off LED and preliminary integration of the nano-fiber paper are investigated and reported.
4. Membrane LED with Ag-nanowire Coat Nanofiber Paper

4.1 Electroluminescence (EL) and electrical characteristics

Before integrating the lift-off LED, we have performed the EL measurement to understand the electrical characteristics of the lift-off LED. Comparison of lift-off membrane LED and unetched LED is illustrated with the efficiency improvement in the membrane LED. The current versus voltage (I-V) graph in Figure 4-1 (a) shows the electrical characteristics in the membrane LED, with the turn-on voltage at 3.25V. In the output power versus current (L-I) graph in Figure 4-1 (b), the lift-off LED shows lower light output at lower injection current. With the increase injection current, the lift-off begins to show stronger light output. The transition value of injection current is 3.75mA. An improvement in relative EQE is shown in Figure 4-1 (c) with a reduction in efficiency droop at high injection current (200 mA/cm²). The improvement in efficiency droop clarifies the transition point in the L-I graph. With higher efficiency in the lift-off LED, the recombination rate in the lift-off LED is higher so that the light output is stronger under high injection current. The alleviated droop effect is due to the strain relaxation in the n-type GaN layer after the PEsC process. The strain relaxation is investigated by the Raman spectroscopy in the previous chapter. The GaN compressive strain relaxation also relax the strain in the MQW region, which increases the overlapping of electron and hole wavefunction as simulated in chapter 3.3. The radiative recombination in the MQW becomes more efficient so as to reduce the droop effect.
Figure 4-1. Electrical characteristics comparison of lift-off LED and unetched LED, (a) I-V characteristic, (b) L-I characteristics, and (c) EQE versus current graph.

During the EL measurement of the unetched and lift-off LED shown in Figure 4-2 (a) (b), we have observed the blue shifting in the peak emission wavelength at low injection current, which is due to the band filling effect in the GaN material system [81]. While under high injection current, the joule heating effect dominates to show the red shifting in peak emission wavelength of the lift-off LED [82]. The competition of band filling and joule heating effect leads to a transition point at 10 mA injection current (222 A/cm²).
This transition phenomenon is also observed and discussed in the previous power dependent PL and temperature dependent PL sections. In the unetched LED, the transition is not observed, which means the shifting is only toward the short wavelength. Interesting color tenability in the lift-off LED is also shown in Figure 4-2 (c). With the peak emission wavelength shifting, the emission wavelength can be tuned from pure 450nm blue emission to 500 nm cyan color.

![Figure 4-2](image.jpg)

Figure 4-2. EL spectra of the (a) Lift-off LED, (b) Unetched LED under different injection current, and (c) Emission wavelength transition from blue to cyan color.

4.2 Nanofiber paper characteristics

The transferrable characteristic of the lift-off LED enables many integration works. In the recent work [83], the LLO LED is implemented on the graphene-coated flexible substrates. Instead of using graphene for the integration, we have proposed another
promising material, the Cellulose Nanofibril Nanofibers [84]. The nanofiber papers are provided and fabricated by the research assistant Ms. Xuezhu Xu from North Dakota State University, while the silver nanowire is prepared with the assistance of postdoctoral fellow Dr. Jian Zhou in Composite and Heterogeneous Material Analysis and Simulation Laboratory (COHMAS) at King Abdullah University of Science and Technology. This transparent and conductive nanofiber paper with the active lift-off LED embedded inside can be made for the flexible display as well as serving as the current spreading layer. In contrast to the graphene deposition, the nanofiber paper has advantage of simple fabrication process. In order to characterize the embedded device, we manage to transfer the lift-off LED pixel onto the nanofiber paper using *Pick-and-Place* technique. In the following section, the embedded red LED is a proof-of-concept for the demonstration of using nanofiber paper as the electrode. The single pixel of the flexible display module using lift-off LED is provided as well. The electrical characteristic of the nanofiber paper is measured with the resistance measurement. The experiment shows the nanofiber paper has the unit resistance $R/cm=1700\, (\Omega/cm)$. The nanofiber paper is shown in Figure 4-3.

![Image](image_url)

Figure 4-3. (a) Nanofiber paper pixel unit, and (b) probing the nanofiber paper for the electrical characteristic.
4.3 Red LED proof-of-concept implementation and characterization

In order to prove the possibility of integrating membrane LED into the nanofiber paper, a crude end product using commercial InGaAs/GaAs 633nm red LED as active component is made with the aid of nanofiber paper. The embedded sample is soft-annealed under 150 degree Celsius for 10 min to achieve better contact conduction. The microscopic view of the red LED from the top side is shown in Figure 4-4 (a). This red LED has the top-bottom contact design, so the nanofiber paper can easily get contacted with the LED chip. The embedded red LED pixel shown in Figure 4-4 (b) is characterized under traditional LED characterization.

Figure 4-4. (a) Top-view (n contact side) of the red LED, and (b) the red LED chip embedded into the nanofiber paper with the red LED lit.

We have characterized the embedded red LED pixel using similar characterization method introduced in chapter 4.1. In Figure 4-5, the (a) I-V characteristic, (b) L-I characteristics, and (c) EQE versus current graph have been reported. It shows the embedded red LED pixel has relatively bad electric contact from the I-V characteristic.
The contact resistance of the embedded pixel has larger values compares to the one directly test on the metal substrate. While in the L-I characteristics and EQE graph, the embedded pixel performs acceptably well in terms of the light output. This fact convinces us the possibility in integrating the blue membrane LED into the nano-fiber paper to establish the flexible paper-based display.

![Graph](image)

Figure 4-5. Electric characteristics of the embedded red LED pixel, (a) I-V characteristic, (b) L-I characteristics, and (c) EQE versus current graph.
4.4 Summary

With the alleviation of the efficiency droop effect, the lift-off LED has a better performance compares to the unetched LED. The lift-off LED can then be utilized in the high power devices once the heat sink issue is solved. The lift-off LED can be transferred to any substrate with better heat conductivity and flexibility. We have proposed the integration of the lift-off LED using nano-fiber paper to manufacture the flexible paper-based display. Prototype is made by integrating the 1mm x 1mm red LED. The performance of the prototype, although relatively poor, has the room for improvement. This embedded red LED pixel prototype proves the possibility in integrating the lift-off LED into nano-fiber paper.
5. Conclusions, and Recommendations

5.1 Conclusions

In the thesis, we utilize the PEsC etching process to fabricate the lift-off membrane LED. This etching process shows the capability of selective etching of undoped GaN. The lift-off membrane LED is fully characterized with photoluminescence, electroluminescence, SEM, TEM, SIMS to understand the device after the PEsC etching process. The lift-off membrane LED shows an improvement in alleviating the efficiency droop. In order to understand the origin of the alleviation of the efficiency droop, we have characterized the strain exhibiting in the GaN layer of the lift-off LED using Raman spectroscopy. The compressive strain has 0.86 GPa relaxation. In the lift-off LED, the inherent strain in the GaN layer has been fully relaxed after the PEsC etching. We have run the simulation on simplified device with different strain conditions. The simulation supports the increase in electron and hole wavefunctions overlapping in the MQW region. The radiative recombination has increased in the MQW region to support the fact of alleviation of the efficiency droop.

Secondly, the lift-off membrane LED has enabled the flexible and high-efficiency application in flexible paper-based display. With the aid of nano-fiber paper, the prototype of embedded red LED pixel is realized. The decent performance of the embedded pixel supports the possible integration of the lift-off membrane LED into the nano-fiber paper. The possible flexible paper-based display can be realized using the PEsC etched lift-off membrane LED.
5.2 Outlook and future applications

Future works and applications can be done to further utilize and strengthen this PEsC etching process. The outlook and future applications are stated as follows:

Side-wall protection: In order to prevent the MQW region from the polarization enhanced etching, the simplest way is to cover the side-wall of the device before the PEsC etching. The possible solution to this issue is to utilize the SiNx thin film as the side-wall protection. At the same time, the etchant shall be changed into KOH as it does not etch the SiNx thin film. The side-wall protection work is ready for experiment and currently ongoing.

Large-area exfoliation: Smaller device has the simplicity as an advantage in device fabrication. For future application such as the flexible display, the large-area LED has to be fabricated. In order to make the large-area LED from the PEsC etching process, we have to reverse the pattern design so that the etchant can then flow into the area where to etch the undoped GaN. Large-area (1mm x 1mm) LED membrane has many advantages for further applications.

Nanofiber paper integration: As the large-area LED is completed, the integration with nanofiber paper can be accomplished. With a larger device, it is possible to handle the lift-off membrane with tweezers and other tools. The top-bottom contact design of the lift-off LED is sandwiched as we demonstrated in chapter 4.3, which can be further applied as the paper-based flexible display.
PUBLICATION LIST

• Chao Zhao, Tien Khee Ng, Hsien-Yu Liao, Aditya Prabaswara, Michele Conroy, Shafat Jahangir, Pallab Bhattacharya, , and Boon S. Ooi, “Surface Passivation of Molecular Beam Epitaxy Grown InGaN/GaN Quantum-Disk Nanowires Light Emitting Diodes” Nanoscale (Under submission)

• Ahmed Ben Slimane, Hsien-Yu Liao, Tien Khee Ng, Pawan Mishra, Damian Pablo San-Romn-Alerigi, and Boon S. Ooi, “Complete Green p-i-n LEDs Lift-off by Selective Area Illumination” Appl. Phys. Lett. (Under submission)


• Hsien-Yu Liao, Ahmed Ben Slimane, Tien Khee Ng, and Boon S. Ooi, “High Performance Epitaxial-Lifted-Off Micro-InGaN-LEDs for Optoelectronics Integration”, In proceeding of SPIE Photonics West 2015, California, USA. (7-12 Feb. 2015)


## Appendix.A  Membrane LED fabrication device travel log

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Step</th>
<th>Resource</th>
<th>Check/Inspection/Remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wafer Cleaning</td>
<td>Acetone / IPA rinsing, blow dry using N₂ gun.</td>
<td>Solvent bench, LIT-016</td>
<td></td>
</tr>
<tr>
<td></td>
<td>HCl:H₂O = 1:1 cleaning at 90 degree Celsius, 15 minutes</td>
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<td></td>
</tr>
<tr>
<td>E beam evaporator p-contact deposition</td>
<td>Ni 5nm / Au 5nm</td>
<td>DEP-008</td>
<td></td>
</tr>
<tr>
<td>RTP ohmic contact formation</td>
<td>RTP 600 degree Celsius 1min</td>
<td>No gas introduced</td>
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</tr>
<tr>
<td>PECVD Dielectric hardmask deposition</td>
<td>1μm SiO₂</td>
<td>PECVD (DEP-009)</td>
<td>27min deposition</td>
</tr>
<tr>
<td></td>
<td>Dep. Rate = 40nm / min</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>SiH₄ 6sccm, N₂O 850sccm, N₂ 162.5sccm, Pressure 1000mT, RF power 20W, Table Temperature 300 degree Celsius</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ICP-RIE Chamber cleaning</td>
<td>30min cleaning</td>
<td>PE-004B</td>
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</tr>
<tr>
<td></td>
<td>O₂ 5sccm, Pressure 10mT, RF power 100W, ICP power 1200W, Table Temperature 10 degree Celsius</td>
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<td></td>
</tr>
<tr>
<td>Patterning</td>
<td>Resist: ECI3027 4um, bake 1min @ 100 degree Celsius</td>
<td>LIT-001 (JST Resist Spin/Bake)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Step1: speed 700 rpm, ramp:1000 rmp/s and time 3 sec</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>Step2: speed 1200 rpm, ramp:1500 rmp/s and time 3 sec</td>
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<tr>
<td></td>
<td>Step3: speed 1750 rpm, ramp:3000 rmp/s and time 30 sec</td>
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<tr>
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<td>Softbake on hotplate 100 degree Celsius for 60 sec</td>
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<tr>
<td>Photolithography</td>
<td>MASK: Bensou array plus sign</td>
<td>LIT-022</td>
<td></td>
</tr>
<tr>
<td></td>
<td>UV exposure for 200mJ/cm²</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Develop sample using AZ726 MIF solution for 60s</td>
<td>LIT-017</td>
<td></td>
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<tr>
<td>ICP-RIE SiO₂ pattern transfer</td>
<td>C₄F₄ 40sccm, O₂ 5sccm, Pressure 10mT, RF 100W (DC self-bias = 300V), ICP power 1200W, Table Temperature = 10 degree Celsius</td>
<td>PE-003A</td>
<td>Use tiny amount of Fomblin oil for heat dissipation.</td>
</tr>
<tr>
<td>Process</td>
<td>Description</td>
<td>Details</td>
<td></td>
</tr>
<tr>
<td>-------------------------------</td>
<td>-----------------------------------------------------------------------------</td>
<td>------------------------------------------------------------------------</td>
<td></td>
</tr>
<tr>
<td>Sample cleaning</td>
<td>Dip the sample in acetone for 5min to clean out the remaining photoresist</td>
<td>Check under the profiler, trench thickness should match the SiO2 thickness measured previously under reflectometer</td>
<td></td>
</tr>
<tr>
<td>GaN ICP-RIE etching</td>
<td>Ar 3sccm, Cl₂ 15sccm, Pressure 10mT, RF 100W, ICP power 500W, Table Temperature = 10 degree Celsius</td>
<td>Etch time: 6min + 1min (break) *3 loop; 1 min break for the sample cool down; Selectivity(SiO₂:GaN) ~ 1:5</td>
<td></td>
</tr>
<tr>
<td>Pt deposition</td>
<td>thickness: ~10nm</td>
<td>DEP-003</td>
<td></td>
</tr>
<tr>
<td>deposition time: 15s</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>UV BIPEsC etching</td>
<td>HF/CH₃OH/H₂O₂ (2:1:2)</td>
<td>UV Hg-Xe lamp</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>After the UV etching, the sample should be rinsed with IPA and then kept in IPA for transferring.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>750W 11min (Green), 12min (Blue)</td>
<td></td>
</tr>
<tr>
<td>Critical point dryer</td>
<td>automatic drying process</td>
<td>WP-010</td>
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</table>
Appendix.B  Transfer matrix method MATLAB for reflectivity simulation

The DBR simulation is done with the transfer matrix method (TMM). The physics model of the DBR is analyzed with each matrix representing a layer in the DBR structure. A DBR is made of alternating two different refractive index layers with sharp interface. Thus, the periodic structure in the DBR can be modeled with the simple matrix form calculation as follows [85]:

Assume a plane wave is impinging to a DBR from the $K^{th}$ layer, as shown in Figure B-1,

![Figure B-1. Schematic of a periodic layered media](image)

The net reflectivity at layer $i$ is represented by $\Gamma_i$,

$$\Gamma_{i+1}(\lambda) = \frac{r_{i+1}(\lambda) + \Gamma_i(\lambda)e^{-j2\beta_i(\lambda)l_i\cos(\theta_i)}}{1 + r_{i+1}(\lambda)\Gamma_i(\lambda)e^{-j2\beta_i(\lambda)l_i\cos(\theta_i)}}, \text{ where}$$

$$r_i = \frac{n_i - n_{i-1}}{n_i + n_{i-1}}; \beta_i(\lambda) = \frac{2\pi n_i(\lambda)}{\lambda}$$

The variable $r_i$ is the local reflectivity between layers $i$ and $i-1$, $\beta_i(\lambda)$ is the propagation constant of the $i^{th}$ layer, $l_i\cos(\theta_i)$ is the effective thickness of the $i^{th}$ layer, $n_i$ is the refractive index of the $i^{th}$ layer, and $\theta_i$ is the propagation angle in the $i^{th}$ layer [86].
With the formulas above, a MATLAB program is then created to simulate the reflectivity spectrum of a DBR. The MATLAB program could be changed for either ideal simulation or the comparison with the actual experimental data. The light is incident normal to the DBR surface. With inputting the actual thickness of the alternating layers, the exact reflectivity of a DBR can be obtained. In the ideal case, each layer thickness of the DBR should be exactly optically equal to the quarter of multiple targeted wavelength, i.e. $m\lambda/4n$, m, n are integers. If this restriction is not satisfied, the peak reflectivity will decrease and shift horizontally with respect to the thickness variation.

The reflectivity spectrum of a DBR is also dependent on the refractive index difference of the two materials. The higher the difference, the higher the reflectivity will be at smaller wavelengths. This is crucial for designing a target wavelength in the green light spectrum. The reflectivity also depends on the number of pairs. If we increase the number of alternating layer pairs in our DBR, then the net reflectivity will increase. This is due to the fact that there will be more constructive interference throughout the DBR heterostructure.

In the MATLAB program, we not only simulate single DBR but also the full cavity to visualize the emission dip in the DBR pairs. With the powerful beauty of the TMM, we can easily add the cavity in between two DBR pairs to see the full device simulation in order to fabricate the VCSEL and the further work on InGaP/InAlGaP diode laser facets coating. In the cavity design, the light will emit from the lower reflectivity DBR side, and the simulation results are provided in Figure B-2.
Figure B-2. SiO$_2$/SiNx DBR mirror (a) 10 pairs for 445 nm InGaN/GaN LED device, and (b) 9/10 pairs (bottom/top) for 633 nm InGaP/InAlGaP diode laser facet coating.
Appendix.C  DBR facet coating on red laser

C.1 Introduction

Research on improving the performance of semiconductor laser has been an on-going effort since its invention in 1960s. For practical semiconductor lasers and optical amplifiers, the application of high-reflection (HR) and anti-reflection (AR) coatings on the device facets to engineer the facet reflectivity is conventionally achieved by RF sputtering [87] or electron-beam evaporation [88] techniques. These processes are typically performed in high vacuum using complex and costly equipment. Although coating of HR and AR mirrors using plasma-enhanced chemical vapor deposition (PECVD) technique has been attempted [89], the process is primarily developed for 1.55µm devices that required less thickness precision control.

A reliable PECVD process technology for HR and AR coatings with high thickness precision control for light emitting devices in visible spectrum using a simple PECVD system is still lacking. In this study, we demonstrated the coating of a high-reflection SiO$_2$/SiN$_x$ [90] Distributed Bragg Reflector (DBR) mirror on the cleaved facet of InGaP/InAlGaP red-emitting diode lasers emitting at 633 nm using a low film stress and accurate thickness control PECVD process.

C.2 DBR Design and Fabrication

Alternating SiO$_2$/SiN$_x$ dielectric layers with optical thickness of $\lambda/4$ have been used in our high-reflection DBR mirror. With regards to the dielectric deposition process, dielectric stack with precise thickness control and compositionally abrupt interface are two critical requirements for achieving high reflection at the targeted wavelength of 633 nm.
nm. In our case, these process requirements were achieved in the PECVD process, in which an intermediate gas line purging is introduced before switching gases to deposit the alternating SiO$_2$/SiN$_x$ layers in the DBR mirror. The SiN$_x$ film is deposited with a mixture of SiH$_4$, NH$_3$ and N$_2$ gases with flow rates of 23, 20 and 980 sccm, at chamber pressure of 850 mTorr and growth temperature of 300 °C. The RF power is 20 W at alternating low and high frequencies in a 6 and 14 sec interval to achieve a low stress film, and thus consistent refractive index can be assured and film cracking can be eliminated. The SiO$_2$ film is deposited with SiH$_4$, N$_2$O and N$_2$ with flow rates of 6, 850 and 162.5 sccm, with chamber pressure of 1000 mTorr at the same growth temperature as SiN$_x$ film, while the RF power is 20 W throughout the film deposition thickness. The films are then examined using ellipsometer and reflectometer to confirm the film thickness and refractive index. The desired thickness for SiN$_x$ ($n=1.99$) and SiO$_2$ ($n=1.44$) were calculated to be 79.52 nm and 109.9 nm for 633 nm diode laser, respectively.

Figure C-3. (a) Scanning electron microscopy (SEM) image of 10-pair SiO$_2$/SiN$_x$ DBR, and (b) simulation (R$_{sim}$) versus experimental (R$_{exp}$) reflectivity for 10-pair DBR.
Figure C-3 (a) shows the SEM image of the 10-pair SiO$_2$/SiN$_x$ DBR stack deposited on (100) Si substrate, while Figure C-3 (b) exhibits the comparison of reflectivity between MATLAB simulation based on transfer matrix method and UV-VIS spectrophotometer measurement, demonstrating accurate thickness control in our process development using the PECVD technique with low stress dielectric thin film. The 10-pair DBR has a central wavelength at 633 nm with reflectivity reaching 98.29%.

C.3 Laser Fabrication

The 633 nm laser consists of a 6 nm In$_{0.47}$Ga$_{0.53}$P quantum well sandwiched between 80 nm In$_{0.5}$Al$_{0.5}$Ga$_{0.2}$P barriers. The buffer layer consists of a 200 nm n-GaAs and the active region is cladded by 1µm n-In$_{0.5}$Al$_{0.5}$P layer and 1µm p-In$_{0.5}$Al$_{0.5}$P layer. The highly doped p-contact layer consists of a 75 nm p-In$_{0.5}$Al$_{0.5}$P layer and capped by a 200 nm p-GaAs layer. The details of the wafer structure are given in Table C-1. The broad area diode lasers were fabricated using the standard UV contact lithography and inductively-coupled plasma (ICP) etching techniques.
The dominant optical losses for a diode laser are the internal loss and mirror loss. Here, DBR mirrors are deposited, using the PECVD technique, on the laser facets to increase reflectivity, thereby reducing the mirror loss and threshold current.

C.4 Results and Discussion

The diode laser bar with dimensions of 100 µm × 1220 µm × 243 µm (corresponding to stripe width × cavity length × laser bar thickness) is shown in Figure C-4. Three sets of devices were studied. These are: (i) as-cleaved, (ii) single-facet coated with 3-pair DBR ($R_{sim}=72.1\%$), and (iii) two-facet coated (3-pair DBR, $R_{sim}=72.1\%$ at facet 1, and 4-pair DBR, $R_{sim}=84.3\%$ at facet 2). The light-current (L-I) characteristics of these lasers are given in Fig. 3 (a). A significant improvement in threshold current of about 6% and 20% has been observed from a single facet coated and two facets coated devices, respectively.

<table>
<thead>
<tr>
<th>Material</th>
<th>Thickness</th>
<th>Dopant</th>
<th>Remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>p-GaAs layer</td>
<td>200 nm</td>
<td>Zn ($&gt;2\times10^{18}$)</td>
<td>Contact layer</td>
</tr>
<tr>
<td>p-In$<em>{0.5}$Al$</em>{0.3}$P</td>
<td>75 nm</td>
<td>Zn ($&gt;3\times10^{18}$)</td>
<td>-</td>
</tr>
<tr>
<td>p-In$<em>{0.5}$Al$</em>{0.3}$P</td>
<td>1000 nm</td>
<td>Zn ($1\times10^{18}$)</td>
<td>Upper Cladding</td>
</tr>
<tr>
<td>u-In$<em>{0.5}$Al$</em>{0.5}$Ga$_{0.5}$P</td>
<td>80 nm</td>
<td>Undoped</td>
<td>Barrier</td>
</tr>
<tr>
<td>u-In$<em>{0.47}$Ga$</em>{0.53}$P</td>
<td>6 nm</td>
<td>Undoped</td>
<td>Quantum well</td>
</tr>
<tr>
<td>u-In$<em>{0.5}$Al$</em>{0.5}$Ga$_{0.5}$P</td>
<td>80 nm</td>
<td>Undoped</td>
<td>Barrier</td>
</tr>
<tr>
<td>n-In$<em>{0.5}$Al$</em>{0.3}$P</td>
<td>1000 nm</td>
<td>Si ($&gt;1\times10^{18}$)</td>
<td>Lower Cladding</td>
</tr>
<tr>
<td>n-GaAs</td>
<td>200 nm</td>
<td>Si ($&gt;2\times10^{18}$)</td>
<td>Buffer layer</td>
</tr>
<tr>
<td>n-GaAs substrate</td>
<td>350 ± 25 µm</td>
<td>Si ($&gt;1\times10^{18}$)</td>
<td>2” diameter, 10° offcut</td>
</tr>
</tbody>
</table>

Table C-1. 633nm InGaP/InAlGaP Diode Laser Structure
In Figure C-4 (a), the top-view microscope image showed the two-facet coated diode laser with dielectric film covering part of the metal contact. There is unintended film deposition on the p-contact surface that can be eliminating by improving the PECVD deposition process and applying proper protection to the contact layer [91]. The comparisons of the facet view before and after DBR coating are shown in Figure C-4 (b) and (c), respectively. Figure C-4 (c) showed the reddish color reflection, indicating the intended operating wavelength in the red regime.

Figure C-4. Microscope images of the two-facet coated 633nm diode laser: (a) top-view, (b) facet view before DBR coating, and (c) facet view after DBR coating.

To this end, we also note that consistent and repeatable device threshold current improvement can be achieved using our PECVD process, as shown in Figure C-5. The improvement of 2-10% and 19-24% were achieved for single-facet coated and two-facet coated devices, respectively. The vast reduction in two-facet coated sample is due to the fact that more photons are reflected back into the resonance cavity, which simultaneously enhances lasing action and reduces the relative threshold current.
Figure C-5. (a) Diode laser L-I curves showing improvement in threshold current under different coating conditions. (b) Device number versus threshold current improvement percentage demonstrating consistency and repeatability in either single-facet coated or two-facet coated PECVD process.

C.5 Summary

High-reflection SiO$_2$/Si$_x$N$_y$ DBR mirrors have been successfully fabricated on 633nm diode lasers using a PECVD process with accurate thickness control and low thin film stress. A significant improvement of device characteristics have been observed from the facet coated devices. The simple dielectric DBR coating technology will be useful for HR and AR coatings of light emitting and semiconductor optical amplifier devices in visible spectrum.
REFERENCE


85. Michael McCoy, Curry Taylor, and Gary Swenson, *Design and Fabrication of Distributed Bragg Reflectors for use in RCLEDs and VCSELs*. May 1, 2001: University of Illinois at Urbana-Champaign.

