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ABSTRACT

We report on research results in this project which synergize advanced material science approaches with fundamental optical physics concepts pertaining to light-matter interaction, with the goal of solving seminal problems for the development of very high performance light emitting diodes (LEDs) in the blue and green for Solid State Lighting applications. Accomplishments in the duration of the contract period include (i) heteroepitaxy of nitrogen-polar LEDs on sapphire, (ii) heteroepitaxy of semipolar (11 $\bar{2}$ 2) green LEDs on sapphire, (iii) synthesis of quantum-dot loaded nanoporous GaN that emits white light without phosphor conversion, (iv) demonstration of the highest quality semipolar (11 $\bar{2}$ 2) GaN on sapphire using orientation-controlled epitaxy, (v) synthesis of nanoscale GaN and InGaN medium, and (vi) development of a novel liftoff process for manufacturing GaN thin-film vertical LEDs. The body of results is presented in this report shows how a solid foundation has been laid, with several noticeable accomplishments, for innovative research, consistent with the stated milestones.

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TABLE OF CONTENTS

FINAL SCIENTIFIC/TECHNICAL REPORT	1
ABSTRACT	1
TABLE OF CONTENTS	2
EXECUTIVE SUMMARY	4
I INTRODUCTION	5
II EXPERIMENTAL	5
III RESULTS AND DISCUSSION	6
III.A Heteroepitaxy of nitrogen-polar GaN and LED	6
1. Growth on N-polar GaN	6
2. Demonstration of blue LEDs on N-face GaN	7
III.B Heteroepitaxy of nonpolar (11 $\bar{2}$ 0) GaN	7
III.C Heteroepitaxy of semipolar (11 $\bar{2}$ 2) GaN	9
III.D Hybrid QD/Nanoporous GaN white LEDs	12
III.E Semipolar (11 $\bar{2}$ 2) LEDs on stripe-patterned sapphire	14
III.F Selective Growth of InGaN/GaN on semipolar and nonpolar planes	19
III.G Nanostructured light emitting medium	20
III.H Development of epitaxial liftoff process for vertical LEDs	23
IV MILESTONE SUMMARY AND COMPARISONS	28
V LIST OF PUBLICATIONS AND PRESENTATIONS	29
REFERENCES	Error! Bookmark not defined.

LIST OF GRAPHICAL MATERIALS

All graphical materials are imbedded within the main body of this report.

EXECUTIVE SUMMARY

The aim of this research project at Yale and Brown Universities is to synergize advanced material science approaches with fundamental optical physics concepts pertaining to light-matter interaction, with the goal of solving fundamental efficiency problems so as to enable the development of very high performance light emitting diodes (LEDs) in the blue and green for Solid State Lighting applications, covering the spectral regime of approximately 430 to 550 nm. Our work is directed towards novel, highly adaptable device concepts that enable their flexible utilization and matching to the broad spectrum of approaches and requirements that pertain to contemporary solid state lighting approaches. The material base of the light emitters is formed from nanostructured gallium nitride and related semiconductor heterostructures, which are enclosed by mesoscopic optical confinement and light emitting enclosures for efficient extraction of optical energy. The ultimate goal of the research is to reach the goal of a highly wall-plug efficient, high optical power device by concentrating on two specific, closely coupled performance dictating elements within the LED.

The research program has been organized in the following way: First, in ongoing work we are synthesizing nanostructured active media to enhance the internal radiative efficiency utilizing special concepts in epitaxial growth. Second, the research has also focused focus on the design and fabrication of advanced photonic confinement structures, which encase the nanostructured active medium for enhancing the spontaneous emission by strengthening light-matter interaction at a fundamental level and for efficiently extracting and distributing the photons for delivery into specific geometrical radiation patterns by design.

Below we summarize the progress made during the course of this project. The body of results is presented clearly indicates that a solid foundation has been laid, in addition to several noticeable accomplishments, for reaching the stated milestones in the coming years. Specifically we have made substantially advances in (i) heteroepitaxy of nitrogen-polar LEDs on sapphire, (ii) heteroepitaxy of semipolar (11 $\bar{2}$ 2) green LEDs on sapphire, (iii) synthesis of quantum-dot loaded nanoporous GaN that emits white light without phosphor conversion, (iv) demonstration of the highest quality semipolar (11 $\bar{2}$ 2) GaN on sapphire using orientation-controlled epitaxy, (v) synthesis of nanoscale GaN and InGaN medium, and (vi) development of a novel liftoff process for manufacturing GaN thin-film vertical LEDs.

I INTRODUCTION

A revolution in lighting regarding energy saving, component lifetime, and fixture versatility is anticipated if the traditional tube-based (incandescent and fluorescent) lamps can be replaced by solid state LEDs. For this vision to become reality, a substantial gap in terms of light output between a standard incandescent bulb (1000~2000 lumens) and a single III-N LED chip (10~50 lumens) needs to be bridged. Engineering issues such as metal contacts, current spreading, and light extraction have to be addressed in scaling up the device area for increased optical output from a single chip. Of paramount importance is the identification and realization of an active medium capable of more efficient conversion of electron-hole pairs into photons, especially under higher-level current injection ($J=10^3\sim10^4$ A/cm²). Carrier localization due to In-related compositional fluctuations in InGa_N is attributed to the constraint of in-plane carrier diffusion, thus preserving and preventing the injected carriers from recombining at nonradiative dislocation sites. Such a benefit of localization diminishes at high-level injection as more carriers acquire a nature of extended electronic states and are consumed by nonradiative processes. A recent observation of a substantial decay in quantum efficiency at a modest injection level from state-of-the-art high power AlGaInN LEDs served as a reminder of the necessity of innovative and creative material research for an efficient light-emitting medium.

In this report we summarize the progress and accomplishment made during the DOE Contract entitled “*Multicolor, High Efficiency, Nanotextured LEDs*”. Details of the experimental facilities and methodology are described in experimental section (II). The body of results is presented in Section III which clearly indicated that a solid foundation has been laid, in addition to several noticeable accomplishments, for reaching the stated milestones in the coming years. Specifically we have made substantially advances in (i) heteroepitaxy of nitrogen-polar LEDs on sapphire, (ii) heteroepitaxy of semipolar (11 $\bar{2}2$) green LEDs on sapphire, (iii) synthesis of quantum-dot loaded nanoporous GaN that emits white light without phosphor conversion, (iv) demonstration of the highest quality semipolar (11 $\bar{2}2$) GaN on sapphire using orientation-controlled epitaxy, (v) synthesis of nanoscale GaN and InGa_N medium, and (vi) development of a novel liftoff process for manufacturing GaN thin-film vertical LEDs.

II EXPERIMENTAL

MOCVD growth was carried out in a horizontal reactor (Aixtron 200/4 HT-S) using trimethylgallium (TMGa), triethylgallium (TEGa), trimethylaluminum (TMAI), trimethylindium (TMIIn) and ammonia (NH₃) as sources with hydrogen and nitrogen as carrier gases. Growth was monitored by in-situ reflectance with 550nm and 900nm light sources. Atomic force microscopy (AFM) was conducted using a Veeco Nanoscope IIIA AFM with tapping mode. Scanning electron microscopy (SEM) was performed using a Hitachi SU-70 FE-SEM, 1nm resolution. Chemical composition is determined by energy dispersive x-ray spectroscopy (EDS) that is embedded in the SEM. High-resolution transmission electron microscopy (TEM) and electron diffraction (ED) were performed using a FEI Tecnai 20 Field Emission TEM. Scanning energy dispersive x-ray spectrometry (EDS) was carried out in a Philips CM-12 TEM with a nominal beam diameter of 50 nm. Thin film material characterization is performed by a triple-axis x-ray diffractometer (Bede D-1), for microstructural analysis and μ -Raman spectroscopy

(Horiba-Jobin Yvon HR-800). Photoluminescence measurements were performed by optical pumping using 30 ns pulsed frequency-quadrupled 263 nm (solid state laser), 325 nm (HeCd laser) and 405nm (solid-state laser), and signal was collected and analyzed with an Acton 3100i grating spectrometer and photomultiplier tube. LED measurements were performed by a probe station setup and Keithley 2400 sourcemeter for I-V, Ocean Optics HR-4000 spectrometer for electroluminescence, and optical power measurement.

III RESULTS AND DISCUSSION

III.A Heteroepitaxy of nitrogen-polar GaN and LED

1. Growth on N-polar GaN

With the well established properties and limitations of Ga-polar [0001] III-Nitride, there has been an increasing interest in exploring alternative crystallographic planes including non- and semi-polar directions. N-polar [000 $\bar{1}$] offers a distinctly (diametrically) different atomic configuration (stoichiometry of cations and anions, dangling bonds and reconstruction), allowing an access to the kinetic space in epitaxy not normally attainable on Ga-polar surface. Already there have been reports regarding contrasts between Ga- and N-polar surfaces in adatom diffusion barrier, 3-dimensional (3D) and 2-dimensional (2D) nucleation behavior, and indium incorporation efficiency in InGaN. During the first eight month of this project, smooth high quality N-polar GaN has been grown using a LT-GaN buffer on appropriately nitridized sapphire. The nucleation evolution of N-polar GaN possesses distinctly different characteristics from the two-step growth of Ga-polar

Fig 1 In-situ reflectance traces of 2-step growth of Ga-polar (a) and N-polar (b) GaN epilayers on sapphire. SEM images on the right show surface morphology at point “B” for Ga-polar (upper) and N-polar (lower) samples.

GaN. With the homogeneous template of densely packed tiny grains formed after the annealing of LT-GaN buffer, N-polar HT-GaN growth starts in a quasi 2D mode with no roughening-recovery process. Compared to Ga-polar GaN, N-polar GaN shows a higher background electron concentration and a lower mobility, which are probably due to the enhanced oxygen incorporation and the more dislocation/impurity scattering, respectively.

Under appropriate growth condition and with the use of offcut wafers, we have demonstrated that the N-polar GaN can have a microstructural quality comparable to that of Ga-polar GaN (Fig 2, left panel).

2. Demonstration of blue LEDs on N-face GaN

Fig 2 (Left) X-ray rocking curves of Ga- and N-polar GaN epilayers. (Center) TEM micrographs showing the nonuniformity of InGaN MQWs grown on N-polar GaN. (Right) A photo of LED operation of a N-polar LED.

In May 2008 we began the growth of InGaN LEDs on non-polar m-GaN and N-polar GaN surfaces. We discovered a combined usage of high-temperature AlN buffer layer and a 2° offcut wafer could lead to optically smooth N-GaN with rocking curve linewidths comparable to (if not better than) those from Ga-polar GaN. Using a similar p-n junction structure, we demonstrated light emission from N-polar GaN (Fig 2, right panel). Important observations include (1) a higher thermal stability for InGaN on N-polar surface, (2) a red shift emission wavelength, possibly due to the formation of indium-rich regions judging by the much more broadened PL linewidth, and (3) a much reduced EL intensity. To the best of our knowledge, this is the first time light emission is reported from N-polar device.

III.B Heteroepitaxy of nonpolar (11 $\bar{2}$ 0) GaN

In year 2 of this project we explored the growth of nonpolar a-GaN on r-plane sapphire as a viable pathway to large-area nonpolar LEDs for solid state lighting applications. We noted that by controlling the morphological evolution (see in-situ reflectance traces in Fig 4) we were able to modify and epitaxial dynamics. We have also demonstrated that x-ray diffraction rocking curve linewidth, a measure of the abundance of structural imperfections due to dislocations and stacking faults, is much reduced as we artificially induce a three-dimensional surface roughening step.

As a continuation of this work, we examined the microstructural properties of a-GaN using transmission electron microscopy (TEM). Given the high anisotropy in nonpolar GaN, we examined the microstructural along m-axis (Fig 3) and c-axis (Fig 4 and Fig 5) to obtain a clear picture regarding the impact of modification of epitaxial dynamics. All the TEM micrographs on the left correspond to conventional one-step growth while the ones on the right correspond to two step growth. Due to the nature of line direction and Burger's vectors, the imaging condition in Fig 3 is sensitive to the presence of basal

stacking faults (BSF) and mixed/screw-type dislocations. Clearly the use of 3D island growth reduces the defect density near the interface.

Fig 3 TEM micrographs with $[1\bar{1}00]$ zone axis and $[1\bar{1}20]$ two beam condition, near GaN/AlN interface.

Fig 4 TEM micrographs with $[0001]$ zone axis and $[1\bar{1}00]$ two beam condition, near GaN/AlN interface.

By rotating the imaging axis of 90° (to c-axis) and using the $[1\bar{1}00]$ diffraction, we examined the microstructures at nucleation point (Fig 4) and after coalescence (Fig 5). It was discovered that the dislocation density is indeed greatly suppressed. Comparative study shows that the majority of defects in the improved a-GaN samples are now purely edge-type dislocations, this will become a focus for our future study.

Fig 5 TEM micrographs with [0001] zone axis and [1 $\bar{1}$ 00] two beam condition, near top GaN surface.

We have also demonstrated the growth of InGaN quantum wells. Fig68 shows a representative photoluminescence trace from an InGaN MQW on a-GaN. Our initial study indicates that InGaN a-plane quantum wells are dimmer in luminescence intensity; reasons may be due to a high density of stacking faults and partial dislocations, as well as possibly difference in surface kinetics during the growth of a-plane InGaN. Based on the initial results, we made a strategic plan to switch and begin to explore semipolar (11 $\bar{2}$ 2) plane.

PL 325 nm laser excitation power density: $\sim 0.35 \text{ W/cm}^2$

Fig 6 PL from a-plane InGaN QWs.

III.C Heteroepitaxy of semipolar (11 $\bar{2}$ 2) GaN

In addition to the heteroepitaxy of nonpolar a-plane GaN, we have made significant progress in the growth of semipolar (11 $\bar{2}$ 2) GaN on r-plane sapphire. Knowledge of v -plot (Fig 3) is instrumental in expediting our control and rational design of the heteroepitaxial evolution. By dividing growth procedures into two stages, beyond LT GaN buffer growth, we explored the possibility of tailoring and promoting the initial island formation, followed by a process of rapid lateral coalescence. The need to promote

individual island growth is based on the consideration that this will (1) help to bend the threading dislocations during island growth, (2) reduce the density of low-angle grain boundary, and (3) create geometric configuration for the blocking of planar defects (stacking faults).

Fig 7 On-axis $2\theta/\omega$ -scans of the semipolar $(11\bar{2}2)$ InGaN/GaN MQWs samples A-C. The data of samples A and B were up-shifted vertically for a better illustration. The X-ray scans were measured when the surface striation direction $[11\bar{2}3]$ was parallel to the X-ray beam plane ($\phi = 0^\circ$).

Fig 8 Room temperature internal quantum efficiency from Ga-polar and semipolar InGaN MQWs. The IQE for Ga-polar MQWs are determined from temperature-dependence PL. Note that semipolar samples are scaled up by 8 times.

So far we discovered that higher pressure is effective in enhancing the growth rate along $[11\bar{2}2]$ direction while a low pressure and high V/III ratio tend to reduce the $[11\bar{2}2]$ growth rate. With a low pressure and high V/III ratio, we can reproducibly obtain semipolar epilayers on sapphire with featureless surface. Introducing a step of high growth pressure induces a significant reflectance roughening, but the surface can be recovered back to reasonable smoothness if the 2nd-step is conducive to lateral growth. An important observation is that there is a substantial improvement with the structural quality revealed by x-ray diffraction. We determine the dislocation density of our heteroepitaxial semipolar GaN is *within a factor of five* to that from c-plane commercial

Fig 9 PL spectra (upper row) and photo images (lower row) of InGaN MQWs grown on semipolar GaN on m-sapphire.

LEDs.

Using the 2-step condition, we have built semipolar InGaN MQWs. Fig 9 shows x-ray diffraction from three InGaN quantum well samples grown on semipolar GaN on *m*-plane sapphire substrates. For this initial demonstration, we fixed the reactor pressure (100 mbar) and In% in the gas phase (60%), only reducing the growth temperature from 760°C to 725°C. The x-ray diffraction indicates that the microstructural quality of MQWs tends to deteriorate with decreasing temperature due to reduced surface diffusion of adatoms. As the growth temperature decreases, there is a corresponding shift of PL peak from 470 to 550 nm (Fig 9), following a change of approximately 2nm/°C slope. From these initial growths, the room-temperature integrated PL intensity decrease by less than two from 470 to 510nm, and it then decreases by a factor of 3.5 to 550 nm. Photos of these samples are shown in the insets in Fig 9. The internal quantum efficiency is estimated (Fig 8) by comparing integrated PL intensity between semipolar and (calibrated) Ga-polar samples.

Fig 10 LED spectrum with peak emission at 525 nm. The blue emission is likely due to electron overshoot and recombine in the p-GaN region.

Fig 11 I-V characteristics and a photo image (inset) of semipolar InGaN LED grown on semipolar GaN on *m*-sapphire.

This PL result is of telltale significance. Our previous effort in heteroepitaxial growth of nonpolar *m*-GaN and *a*-GaN and the InGaN MQWs, with a comparable defect density to the present heteroepitaxial semipolar GaN, has not produced much noticeable luminescence. The InGaN MQWs on semipolar GaN has yielded from beginning very reasonable PL intensity which is about a factor of seven lower than the best *c*-plane InGaN QWs grown by our group. This is especially remarkable given the dislocation density of the semipolar GaN with planar heteroepitaxy is about ten times higher than the typical *c*-GaN template, with additional stacking faults on the order of 10^5cm^{-1} . As a further confirmation, we have grown by MOCVD complete semipolar InGaN PN diodes using the InGaN QW condition described above. While the p-contact growth is still under optimization, we have observed good electroluminescence (Fig 12) at 520 nm with a quick “flash test” procedure at Yale using In to contact scribed n-GaN and p-GaN surface. To the best of our knowledge, this is the first report of a green LED operation of semipolar GaN on sapphire substrates. With this encouraging result as a foundation, we

anticipate that the OCE procedure will drastically enhance the LED performance at green and yellow wavelengths.

III.D Hybrid QD/Nanoporous GaN white LEDs

In this part of the project, we have demonstrated and developed a new, unique strategy in producing multicolor, high efficiency, nanotextured light emitting diodes. This is done by an electrochemical etching process of GaN films. This results in a structure with properties that can be tailored and utilized in various optoelectronic configurations. In this case for hybrid QD/nanoporous GaN, by using the pores of the GaN in embedding highly efficient colloidal quantum dots, we can utilize the increased light scattering within the NP GaN to produce efficient light emission. By controlling the composition of QDs in the NP GaN, multicolor emission can be achieved.

Fig 12 Embedding of QDs into nanoporous GaN (left: PL as a function of dipping time, right: optical image of GaN with embedded QDs)

Here, we demonstrate the initial feasibility of this idea by dipping NP GaN into a colloidal QD solution, consisting of a highly engineered colloidal quantum dot chemistry to produce high density of quantum dots. The nanoporous structure of the GaN can be controlled by changing the parameters of electrochemical etching of GaN, with pore sizes ranging from 30nm to 60nm. The different pore sizes allow a different density of quantum dots to be encompassed by the NP GaN, with maximum density shown by 60nm pore sizes. Photoluminescence with optical pumping of the quantum dots show a large intensity increase above 4 hours, with largest intensity shown with the 60 nm pore size samples.

Fig 13 Photoluminescence spectrum of QD embedded NP GaN samples

In Figure 13, the PL spectra of the QD embedded NP GaN samples are shown. In this case, single size QDs are embedded emitting at $\sim 620\text{nm}$. The QD emission is order of magnitudes higher than the bandedge emission of GaN. The next step is to produce a QD mixture in order to embed multiple sizes of QDs to produce multicolor emission, producing white light.

Continuing from the previous demonstration of successfully embedding QDs into the nanoporous GaN matrix we have prepared by electrochemical etching, we tune the QD incorporation in order to demonstrate multicolor emission. The large light scattering that occurs in the NP GaN matrix allows for multiple transits of optical pumped light to excite the embedded quantum dots, improving the light output of the composite system. The difficulty of utilizing this for white light emission is the unequal optical efficiencies of QDs at different wavelengths, which necessitates a careful preparation of QD solution composed of the RGB QDs. Figure 14 shows a successful QD loading with white light emission by optically pumping.

Figure 14 PL spectrum (left) and optical emission (right) from white light emission from multiple QD incorporation embedded into NP GaN matrix

The next stage in this project is to demonstrate an electrically pumped device. This involves the porosification of an LED, and the loading of such device with QDs. This would allow the QDs to be optically pumped by emission of the GaN LED, resulting in a high-efficiency, multiple wavelength, nanotextured light emitting device.

III.E Semipolar (11 $\bar{2}2$) LEDs on stripe-patterned sapphire

Continuing from the work on semipolar heteroepitaxy, we adopt a novel growth method of orientation controlled epitaxy (OCE) in producing high quality semipolar GaN templates on patterned sapphire substrates. In parallel with our efforts in producing semipolar GaN on planar m-sapphire using a 2step 3D-2D roughening recovery growth methodology for significant reduction of defects and stacking faults, we continue this avenue of growth due to its potential to possibly eliminate all stacking faults. It is believed that this may be the limiting feature of our semipolar templates in producing bright green and longer wavelength emission from MQWs and LEDs. Shown in Figure 15, the depiction of this growth method, new epitaxial relations are highlight to be possible by this method.

Figure 15 (a) Growth method on stripe etched r-sapphire along [1-100] direction, two step shown of two neighboring semipolar (11-22) oriented crystals grown off of the c-plane sapphire sidewall (b) epitaxial relation of semipolar GaN on m-sapphire (c) new epitaxial relation of semipolar GaN on etched r-sapphire using OCE

We have studied a procedure to pattern sapphire substrates using a Cl-based reactive ion etch. We are able to etch sapphire into grooves of 2 μm wide spacing with 2 μm terraces and with a depth of 0.5 μm . Building on our success in producing a coalesced semipolar template, we investigate the important parameters in producing a high quality template. In this growth process, there are three important growth stages which may impact the microstructural quality of the device layers above the GaN template – (1) selectivity – including the buffer used, and initial growth conditions to ensure single orientation GaN.

(2) Crystal shaping – using our knowledge of the kinetic Wulff plot, the crystal growth off of the c-plane sidewall can be tuned by advancement and suppression of different facet growth. This can enable geometric defect reduction, notably stacking fault blocking between neighbouring crystallites. (3) coalescence of the stripes to produce a flat template such that InGaN MQWs are deposited on single orientation, and are uninterrupted with smooth interfaces. During this month, we perform a detailed study of the buffer used in order to reproducibly, over entire substrate, have selective growth of the semipolar GaN.

Semipolar (11-22) GaN on patterned sapphire substrates has been produced already demonstrating improved microstructural quality due to the intrinsic features of the unique growth. The conditions of selectivity and choice of buffer have been explored to optimize the further growth (LT-AlN buffer, low supersaturation), and the idea of stacking fault blocking has been investigated through experiment. We have designed a set of controlled growth interruptions to predict and verify the relations between growth conditions and crystal shape. Here, we used the kinetic Wulff plots measured previously to correlated facet evolution with growth condition to model and extrapolate coalescence configurations of neighboring crystal islands. The importance of this is to ensure an optimal coalescence for defect reduction and surface morphology for semipolar (11-22) GaN on patterned sapphire substrates. The results are shown in Figure 16:

Fig 16 (a)-(c) growth condition dependence and (i)-(iii) growth progression of semipolar GaN grown selectively from c-plane sapphire sidewall from etched r-plane sapphire substrate

The results show a large tunability in crystal shape with the modification of growth conditions. Starting from the initial growth conditions for buffer and selective growth, we vary the important growth parameters (temperature, pressure and V/III ratio) in order to determine the modification of relative facet growth rates. Using a low pressure growth, we see unselective growth off of the r-plane sapphire. This is speculated to be because of increased diffusion through the boundary layer increases the mass transport of precursors

to the substrate, increasing the supersaturation for nucleation. For the high pressure, and low and high V/III ratios, we continue selective growth with differing speeds of the a-plane and c-plane facets. Low V/III ratio increases the speed of the c-plane facet, which results in the crystal growing over neighboring crystals. However, coalescence is delayed due to decreased amount of gas phase transport reducing the precursors reaching between the facets. It is found that an optimal coalescence configuration is shown in row (c), and XRD rocking curve linewidths confirm the microstructural quality of the films, with on axis (0002) diffraction of 0.1 deg, similar to commercial c-plane GaN.

High quality GaN growth on sapphire substrates were enabled by two types of buffer growth – low temperature GaN and low temperature AlN. The properties of these two buffers have been studied extensively, and its consequences on the properties of the final GaN layer has been analyzed in detail. Deposition of LT GaN buffer at 500 C is characterized by nucleation of highly dislocated, mixed cubic and wurtzite phased islands, and upon subsequent anneal undergo a selective dissociation and reforming in a large high quality wurtzite islands. On the other hand, LT AlN buffer is characterized by a columnar structure, of which GaN follows. It is seen that a SiO₂ mask is necessary to be used for selective growth off of the sidewall. However, the large island size and the SiO₂ mask will negatively influence the final GaN layer, due to misorientation of crystals and stress and impurities effects, respectively. Figure 17 shows the effect of using a LT AlN buffer.

Fig 17 (a) LT AlN buffer on sapphire SEM, (b) AFM, (c) inclined and (d) top view of selective growth using LT AlN buffer

Studying the effects of selectivity with growth condition enables scientific insight on the conditions favoring selectivity, shown to be relying on surface diffusion and near thermodynamic equilibrium conditions. Work will continue in the following months studying the next steps of growth for optimum semipolar film growth for LED performance.

On-axis diffractions at various azimuths, as well as (n0-n0) and (000n) series of diffractions are measured for n=1-3 are compared against semipolar (11-22) GaN on

planar m-sapphire by a two-step growth method, as shown in Figure 18a. All on and off-axis diffractions have FWHM of 280 to 550 arcsec, representing a two to five fold decrease in FWHM over semipolar (11-22) GaN on planar m-sapphire, indicating a remarkable improvement in crystalline quality. In fact, the narrow linewidths of this template are similar to those in commercial c-plane GaN LEDs. This shows great potential for this template to be used for high brightness light emitters.

Fig 18 (a) High resolution XRD rocking curve linewidths of semipolar films grown by the two methods, (b) functional dependence on Bragg angle showing linear correlation for patterned sapphire linewidths, indicating a low presence of stacking faults

It is also noted that the difference in linewidths are difference depending on the diffraction plane that is considered. Further analysis of the diffraction linewidths yield information about the character of defects that make up the microstructure of the films.

From classic XRD theory of dislocation broadening of rocking curve linewidths, it can be shown if instrument broadening, curvature and surface imperfection effects are negligible, that the functional dependence of linewidth on Bragg angle is $\beta^2 \propto \tan^2(\theta)$. As shown in Fig 18b, this follows for the semipolar GaN on patterned sapphire. However, for semipolar GaN on planar sapphire, there is a large variation with the ϕ and χ of the diffraction, which can be explained by the large densities of stacking faults present in the material. This indicates a much decreased density of stacking faults in the patterned sapphire case. This could be of huge benefit in optical devices, since the associated partial dislocations are known to be non-radiative recombination centers, and has been shown to limit the optical efficiency of non-polar GaN LEDs.

We have proceeded in using the semipolar GaN on patterned sapphire as a template for light-emitting diode growth. Here, we use an active region similar to c-plane growth, which is unoptimized with respect to the semipolar surface, but will give an idea of the improved in performance over semipolar growth on planar m-sapphire. On-wafer testing is done to characterize electroluminescence, light output power, voltage, wavelength and

linewidth as a function of injection current. The results are compared to semipolar on planar LED, as fabricated earlier, shown in figure 3.

Fig 19 Optical characterization of semipolar LEDs (Top row: semipolar LED on planar m-sapphire, Bottom row: comparison with LED on patterned sapphire)

From this characterization, we can see a marked improvement in light output characteristics over the semipolar LED on m-sapphire. These results show the highest reported output power of any heteroepitaxial semipolar (11-22) LED. Overall light output power has an increase of 250%, as seen in the LI curve. This is due to the improved microstructural quality of the template. The dual wavelength emission of the semipolar LED on planar sapphire is eliminated, and replaced by a very narrow linewidth emission, comparable to that of c-plane GaN LEDs. This indicates an improves InGaN homogeneity or other factor related to the sub band gap emission. As well, the wavelength shift at low currents is not evident. These properties of semipolar LEDs show some similarity, and particularly in the low current regime have some interesting properties. The light emission characteristics will be analyzed in the next coming months to determine how performance can be further increased.

The semipolar GaN LEDs have shown promise with respect to the other orientations of GaN produced. Semipolar GaN grown on patterned sapphire has been shown to have very good microstructural quality by TEM and x-ray diffraction analyses. In effort to further improve and understand the light emission characteristics of LEDs grown on semipolar (11-22) surfaces, we examine the light emission patterns of the LEDs under high magnification optical microscope, as shown in Figure 20. In particular, emission at low injection is carefully studied, to elucidate the green emission and wavelength shift that occurs for sub mA injection.

Fig 20 Left: emission pattern under no injection (top), and low injection (<1mA) at 100x (middle row) and 500x (bottom row) magnification, Right: emission pattern for semipolar LED on planar m-sapphire.

III.F Selective Growth of InGaN/GaN on semipolar and nonpolar planes

Our study of different GaN facets as candidates for light emitting devices has allowed us to investigate numerous semipolar and nonpolar planes. This expertise, especially in growing nanostructures consisting of a multitude of facets, including concave and convex facets, using a donut shaped selective area growth, allows us to demonstrate both facet dependency on light emission as well as fundamental differences in InGaN growth on different semipolar and nonpolar facets in a single growth. Here, InGaN/GaN MQWs are grown on SAG mesas on c-plane and a-plane templates. The light emission is characterized by CL measurements, as shown in Figure 21.

Fig 21 CL spectroscopy and maps of InGaN/GaN MQWs grown on c-plane SAG (left) and a-plane SAG (right)

Growth on c-plane and a-plane SAG islands show clear peaks in the CL spectrum, showing either distinct In incorporation on different facets, different InGaN growth rates, or quantum confinement effects at edges and corners. It is shown that strong light emission comes from the semipolar facets on both c-plane and a-plane SAG islands. Cross-sectional SEM or TEM will be able to differentiate the effects of QW thickness versus In incorporation.

III.G Nanostructured light emitting medium

In this part of the project we have explored several versions of nanoemitters listed in Fig 22. It is important to stress that in our study of nanoemitters the compatibility with solid-state lighting technology and manufacture is always considered. After three years of study, we came to the conclusion that Gen IV in Fig 22 would offer us the best chance of success in making flexible, dislocation free, and high efficiency nanoemitters.

Gen I: Top down e-beam lithography with dry etching to form nano-emitters.

Gen II: Bottom-up catalytic growth of nanorods using catalytic nanoparticles.

Gen III: Top down self-formed masking with dry etching to form nano-emitters.

Gen IV: Bottom-up selective growth of nanoemitters through nano-patterning.

Fig 22 SEM images of nanotextured, nanoemitters synthesized over the course of this project.

Exploration and implementation of GaN light emitting structures in the nanoscale emerge as an exciting frontier that could address long standing issues such as "efficiency droop" and "green gap", producing more boosts to the performance of GaN LEDs. Through Generations I-III we have learned that a few drawbacks need to be considered in designing a nanoemitters: 1) E-beam lithography (Gen I) is time consuming and not practical for mass production, 2) Top-down dry etching (Gen I and III) at nanoscale could produce damage and compromise IQE at high injection levels, 3) catalytic growth (Gen II) suffers from uniformity and contamination issues. In this quarter we study the possibility of a "Gen IV" design through the integration of several concepts including (1) nanoscale lithography, (2) selective area growth (SAG, Fig 2), (3) crystallographic templating, and (4) metamorphic epitaxy, all of which have been investigated at Yale, to realize high-performance, manufacture-friendly nanoLEDs for next generation lighting applications. We believe that the ensembled arrays of nanoLEDs, while retaining compatibility with conventional post-growth fabrication and packaging procedures, will offer the much needed freedom in material and device innovations. The ability to prepare InGaN active regions in a non-planar and nano-tailored fashion will provide unprecedented opportunities in defect reduction, polarization control, heteroepitaxial stress relaxation, and a new way to manage the incorporation of Indium in InGaN.

Fig 23 Concept schematic drawing of a nanoLED enabled by nano-lithography and selective area growth.

Along these lines, we have succeeded in the growth of GaN and InGaN nanorods (Fig 22, Gen IV image) using conditions specially developed for selectivity. Circular mask openings of 100 nm diameter and 400 nm pitch was prepared by interferometric lithography. Cross-sectional TEM reveals that the nanorods are dislocation free (Fig 24, left), x-ray diffraction confirms that the nanorods are InGaN having an In-content of 14%, RT PL also confirms the bandedge emission at around 475 nm. We note that this is the first report of InGaN nanorods. In general an InGaN epilayer of comparable thickness on GaN will suffer from strain-induced roughening or relaxation. The observed high-quality InGaN also serves as a new nano-template that will be suitable for the construction of longer-wavelength InGaN nanorod LEDs.

Fig 24 Left: Cross-sectional TEM of an InGaN nanorod showing no dislocations; middle: RT photoluminescence of an array of InGaN nanorods grown by nanoSAG; right: x-ray diffraction radial scan showing that the In composition in the nanorods is about 14%.

III.H Development of epitaxial liftoff process for vertical LEDs

In the direction of improving performance of LEDs, we have worked on an alternative wafer separation process using nanoporous GaN. The incentives in converting the LED configuration from lateral (Fig 25, bottom left) to vertical (Fig 25, bottom middle) include much improved heat and light extractions, a better electrical injection efficiency, and a substantially better packing density. There is a clear trend in the SSL community (Fig 25, upper right) to migrate from lateral or vertical LEDs. Currently the separation of GaN LED layers is accomplished through laser liftoff.

Fig 25 Upper left: list of all mechanisms of losses, upper right: the trend and benefit in EQE associated with different device configurations, lower left: a schematic drawing of a lateral LED having both front contacts, , lower middle: a schematic drawing of a vertical LED; lower right: the use of a textured surface for enhanced light extraction.

What we discovered and implemented is that with the use of nanoporous GaN (Fig 26) which can enable the formation of airgap (or GaN on air) structures that can facilitate a lateral separation of GaN device layers through the transformation of GaN cylindrical pores into bubbles (Fig 26b) and then into elongated airgap (Fig 26d)

Fig 26 Morphological evolution of a 2-layer nanoporous sample growth temperature. It was found that the low-porosity layer (upper) is transformed into bubbles, which the high-porosity layer (lower part) is transformed into elongated airgap.

Overgrowth, layer characterization were performed to verify this method. Wafer bonding schemes were investigated to attach the LED layer to a target substrate (a glass slide in Fig 27). We found that we can remove a large area ($>1\text{cm}^2$) of LED layers from a sapphire substrate *without* the use of UV laser liftoff

Fig 27 Top: A photograph showing the epitaxial liftoff of more than 1cm² LED layer being transferred from sapphire (left) to a glass slide (right). The dark color on the right is due to the presence of Ga droplets (bottom right) on the N-polar surface. The Ga droplets can be etched away with HCl.

It is important to stress that the epitaxial lift-off process does not adversely affect the microstructural quality of GaN overlayers. Fig 28 shows the microstructural characterizations including Raman (left) and x-ray diffractions (right) before and after the transfer. It was shown that after the transfer the GaN layer becomes stress-free based on the Raman peak. X-ray diffraction indicated that the material quality, indicated by the linewidth of rocking curves for symmetric and asymmetric scans, is comparable and (slightly better) after transfer.

Fig 28 Raman (left) and x-ray rocking curves (right) of GaN device layers before and after transfer.

The most direct benefits of removal of sapphire from the GaN device structure is the possibility for improving the device performance by moving to a vertical LED configuration, and bonding to a substrate with more desirable thermal characteristics. A rise in junction temperature by 10°C has been shown to decrease the luminous efficacy by 5%, which under high injection current with a thermally insulating substrate such as sapphire is of concern. A vertical device configuration will improve the injection efficiency, eliminate current-crowding, increase the available surface area for irradiance, and improvement in device scaling considerations. Initial device results of transferred LED layers are shown in Fig 29 (on glass slide) and Fig 30 (on p-type silicon). From Fig 29, we have a vertical LED on Ag-epoxy bonded to glass slide. In this case the turn-on voltage is fairly low (<2.2 V), possibly because of various leakage paths present in the vertical thin-film device. Fig 30 shows another vertical device that is now bonded through a multilayer scheme (Fig 30, left) to a p-type silicon wafer so the wafer can be

used as the p-type contact. One of the advantages of this technique over the prevalent LED liftoff method, LLO, is the ability to bond to any arbitrary substrate in a simple way. This may enable an unprecedented range of envisioned applications incorporating nitride LEDs into a variety of different host materials. Examples of the utility of this are the recent demonstrations of inorganic LEDs transferred to flexible and transparent materials. Sophisticated transfer methods have been developed to pick-and-place micron scale InGaP LEDs onto plastic substrates, for a variety of applications. However, with the introduction of nitride based liftoff LEDs, white LED and full color RGB applications can now be considered. Transfer of LEDs using our liftoff method to a transparent, flexible substrate in various configurations is demonstrated to show the feasibility of these applications.

Fig 29 Left: Photograph of the lit-up LED on glass slide, middle: electroluminescence spectrum, upper right: L-I-V plot, lower right: peak wavelength and emission peak FWHM versus injection current.

Fig 30 Left: Photograph of the lit-up LED on p-silicon, middle: electroluminescence spectrum, upper right: L-I-V plot, lower right: peak wavelength and emission peak FWHM versus injection current.

IV MILESTONE SUMMARY AND COMPARISONS

- Fabricate InGaN nanostructures for SSL:green emission at 520 nm
Status: Demonstrated, IQE ~15% (determined by temperature-dependent PL)
- Growth of semipolar InGaN (on SiC or m-AlN): Dislocation below 10^9cm^{-2}
Status: Demonstrated, Dislocation density on orientation-controlled semipolar (11 $\bar{2}$ 2) GaN is below 10^8cm^{-2} , confirmed by TEM and XRD.
- Low-loss contact to nanostructures: Determination of interface ΔE_G
Status: Achieved by XPS measurement performed at Princeton for GaN with a set of inorganic and organic layers.
- Demo of super-efficient from optical pumping : Stimulated emission
Status: Demonstrated at 380 nm.
- Self-assembly of GaN nanorods for SSL: Nanorod arrays (0.5 μm long, 10^{10}cm^{-2})
Status: Demonstrated, nanorods were fabricated by top-down dry-etching, as well as bottom up selective area growth.
- Non-polar and semi-polar InGaN MQWs: $\lambda=520\text{nm}$, 590nm $\eta_{\text{int}} > 0.6$
Status: We have demonstrated PL from 500 to 600 nm, and EL from 480 to 530 nm for semipolar GaN LEDs. Using orientation controlled epitaxy has led to record-low dislocation density on sapphire. The result has received much attention and led to a sponsored research by an LED manufacture.
- Superluminescence from electrical injection: $\lambda = 410\text{ nm}$, $\eta > 150\text{ lm/W}$
Status: NYR*
- Study of light extraction from nanotextured medium: $\eta_{\text{extr}} > 60\%$
Status: We have demonstrated that light extraction from nanotextured medium typically exhibits three- to five-fold enhancement.
- InGaN non-polar green and amber LED: $\lambda=520, 590\text{ nm}$, $\eta > 100\text{ lm/W}$
Status: Emission wavelength has reached 520 ad 590nm, luminous efficacy has not reached 100 lm/W
- Technology transfer: Tech transfer and IP license
Status: This project has led to one tech transfer (orientation controlled growth) and one IP licensing (nanoporous layer separation).

*Not yet reached.

V LIST OF PUBLICATIONS AND PRESENTATIONS

Presentations

1. Q. Sun, Y. S. Cho, B. H. Kong, H. K. Cho, T. -S. Ko, C. D. Yerino, I.-H. Lee, and J. Han; “A comparative study of InGaN Quantum Wells on Ga- and *N-polar Surfaces*”, Electronic Materials Conference, Santa Barbara, June, 2008.
2. Q. Sun, C. D. Yerino, I. -H. Lee, and J. Han, “A Mesoscopic Model Toward Omni-Directional GaN Epitaxy”, Electronic Materials Conference, Santa Barbara, June, 2008.
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4. Q. Sun, Y. S. Cho, T. -S. Ko, C. D. Yerino, I.-H. Lee, and J. Han; “N-polar GaN and InGaN Quantum Wells by MOCVD”, Second International Symposium on Growth of III-Nitrides (ISGN-2), Tokyo, Japan, July, 2008.
5. Q. Sun, T. -S. Ko, C. D. Yerino, I.-H. Lee, and J. Han; “N-polar GaN and InGaN Quantum Wells by MOCVD”, International Workshop on Nitride Semiconductors, Montreux, Switzerland, October, 2008.
6. J. Han, Q. Sun, I. -H Lee, “Nonpolar and Semipolar GaN for SSL, Prospects and Challenges”, 2009 Solid State Display and Lighting Conference, Seoul, South Korea, January 2009 (Invited).
7. Q. Sun, B. Leung, and J. Han; “*High Resolution X-Ray Diffraction of III-Nitride Wide Bandgap Semiconductors*”, The Inaugural Yale-Rigaku Symposium on X-Ray Diffraction, Yale University, New Haven, USA, May 2009.
8. C. D. Yerino, Q. Sun, T. S. Ko, I.-H. Lee, and J. Han; “*The Origin of Pits and Striations on the Surface of Nonpolar a-Plane GaN*”, The 51th Electronic Materials Conference, Pennsylvania State University, USA, June 2009.
9. C. D. Yerino, Q. Sun, T. S. Ko, I.-H. Lee, and J. Han; “*Kinetic Wulff Diagram: Toward Rational Design of Nonpolar and Semipolar Heteroepitaxy*”, The 51th Electronic Materials Conference, Pennsylvania State University, USA, June 2009.
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12. J. Han, “Epitaxial Science of GaN, nanowires, quantum dots, and mesoscopic morphology”, SPIE Symposium of Nanoepitaxy, San Diego, CA, August 2009 (Invited).
13. Y. Zhang, S.-W. Ryu, C. D. Yerino, B. Leung, Q. Sun, and J. Han; “*A conductivity-based selective etching for next generation GaN devices*”, The 8th International Conference on Nitride Semiconductors, Jeju, Korea, October 2009.
14. C. D. Yerino, Q. Sun, T. S. Ko, B. Leung, I.-H. Lee, and J. Han; “*Kinetic Wulff Diagram: Toward Rational Design of Nonpolar and Semipolar GaN Heteroepitaxy*”,

- The 8th International Conference on Nitride Semiconductors, Jeju, Korea, October 2009.
15. Q. Sun, C. D. Yerino, B. Leung, T. S. Ko, Y. Zhang, I.-H. Lee, J. Han, B. H. Kong, and H. K. Cho; “*Heteroepitaxy of C-plane (0001), Nonpolar A-plane (11 $\bar{2}$ 0) and Semipolar (11 $\bar{2}$ 2) GaN: Similarities and Differences*”, The 8th International Conference on Nitride Semiconductors, Jeju, Korea, October 2009.
 16. Q. Sun and J. Han; “*Kinetic Wulff plot: understanding and controlling GaN heteroepitaxy*”, Invited talk at Peking University, Beijing, China, November 2009.
 17. A. Nurmikko and J. Han; “*Nanostructured Nitrides in Augmenting Light Emission and Detection*”, Invited talk at MRS fall meeting, Boston, USA, December 2009.
 18. J. Han; “*Prospects of Nonpolar and Semipolar Green LEDs for Solid State Lighting*”, Invited talk at The Second International Conference on White LEDs and Solid State Lighting, Taipei, Taiwan, December 2009.
 19. J. Han, Nanostructured Nitride for Solid State Lighting – where is the “Nano” help?, seminar given at YINQE meeting, January 2010.
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 21. J. Han, *Heteroepitaxial Pathways to Nonpolar and Semipolar GaN LEDs*, The 4th International Conference on LED and Solid State Lighting (LED 2010) COEX, Seoul, Korea February 2010. (Plenary)
 22. J. Han, *Frontiers of Epitaxial Research for Solid State Display and Lighting*, Seminar at Seoul National University, Korea, February 2nd 2010. (Invited)
 23. J. Han, *Let there be light*, talk given at Yale IEEE Student Chapter Meeting, April, 2010.
 24. Q. Sun, Y. Zhang, B. Leung, C. Yerino and J. Han., *Heteroepitaxial Semipolar GaN-based Light-Emitting Diode*, the 19th Connecticut Microelectronics and Optoelectronics Symposium University of Connecticut April 7th 2010
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 29. J. Han, *Heteroepitaxial Pathways to Nonpolar and Semipolar GaN LEDs*, International Union of Materials Research Societies - International Conference on Electronic Materials 2010, KINTEX, Seoul, Korea, August 22-27, 2010. (Invited)
 30. J. Han, *Heteroepitaxial Pathways to Nonpolar and Semipolar GaN LEDs*, Seminar at Korea University, Seoul, Korea, August 22-27, 2010. (Invited)

31. Y. Zhang, Q. Sun, B. Leung, and J. Han; *Large area, free standing GaN by a novel nanoetching process*, International Workshop on Nitride Semiconductors (IWN), Tampa Florida, September 2010.
32. Y. Zhang, Q. Sun, B. Leung, C. D. Yerino, and J. Han; *Nanoporous III-nitrides Structures: Fabrication and Characterization*, International Workshop on Nitride Semiconductors (IWN), Tampa Florida, September 2010.
33. Y. Zhang, B. Leung, Q. Sun, C. D. Yerino, H.G. Chen, S. R. Jianand and J. Han; *High Quality Semipolar (11-22) GaN and InGaN/GaN MQWs grown on Nanoporous GaN by Metal Organic Chemical Vapor Deposition*, International Workshop on Nitride Semiconductors (IWN), Tampa Florida, September 2010.
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35. C. Dang, Y. Zhang, J. Han and A. Nurmikko, A Wavelength Engineered Emitter Incorporating CdSe-based Colloidal Quantum Dots into Nanoporous InGaN/GaN Multiple Quantum Well Matrix, International Workshop on Nitride Semiconductors (IWN), Tampa Florida, September 2010.
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38. Y. Zhang, B. Leung, C. Yerino, Q. Sun, S-W Ryu, H. Cao and J. Han, III-Nitride Semiconductors for Photonic Applications, the first annual Yale Photonics poster session Yale University, Jan 1st 2010
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40. J. Han, Solid State Lighting: Economics, Science and Technology The 3rd International workshop on Energy, Environment, Water and Sustainability, Daejeon, Korea, October 2010 (Plenary)
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43. C. Dang, Y. Zhang, J. Lee, J. Han, A. V. Nurmikko, C. Breen, J. S. Steckel, S. Coe-Sullivan, Wavelength Engineered Luminescent Material Incorporating Colloidal Quantum Dot within a Nanoporous Gallium Nitride Matrix, Conference on Lasers and Electro-Optics, Baltimore, Maryland (May 2011)
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46. J. Han, GaN Material Research for SSL: Heteroepitaxial Science and Nanostructured Medium, Invited seminar at Samsung LED, Korea (2011)
47. B. Leung, Y. Zhang, C. Yerino, J. Han, B. Kong, H. Cho, Q. Sun, Z. Chen, S. Lester, K.-Y. Liao, Y.-L. Li, *Optical Emission Patterns in Semipolar (11-22) GaN Light Emitting Diodes on Planar m-Plane and Etched r-Plane Sapphire*, Electronic Materials Conference, Santa Barbara, California. June 2011
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51. J. Han and M. E. Coltrin, Heteroepitaxial science of polar, semipolar and nonpolar (In)GaN, DOE PI meeting, Washington DC, August, 2011
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53. D.Chen, H.Xiao,Y. Zhang and J. Han; *Morphology and formation mechanism of porous GaN via HF electrochemical etch and its application in liftoff of GaN based thin layers*, 2011 Materials Research Society Fall Meeting and Exhibit, Boston MA, November 2011.

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Patents and disclosures:

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