InGaN/GaN Multiple Quantum Wells Grown on Nonpolar Facets of Vertical GaN Nanorod Arrays

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Abstract: Uniform GaN nanorod arrays are grown vertically by selective area growth on ⟨0001⟩ substrates. The GaN nanorods present six nonpolar $\{1\bar{1}00\}$ facets, which serve as growth surfaces for InGaN-based light-emitting diode quantum well active regions. Compared to growth on the polar {0001} plane, the piezoelectric fields in the multiple quantum wells (MQWs) can be eliminated when they are grown on nonpolar planes. The capability of growing ordered GaN nanorod arrays with different rod densities is demonstrated. Light emission from InGaN/GaN MQWs grown on the nonpolar facets is investigated by photoluminescence. Local emission from MQWs grown on different regions of GaN nanorods is studied by cathodoluminescence (CL). The core−shell structure of MQWs grown on GaN nanorods is investigated by crosssectional transmission electron microscopy in both axial and radial directions. The results show that the active MQWs are predominantly grown on nonpolar planes of GaN nanorods, consistent with the observations from CL. The results suggest that GaN nanorod arrays are suitable growth templates for efficient light-emitting diodes.

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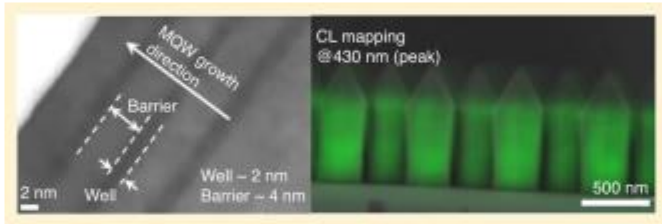

Key words: GaN nanorods, GaN nanowires, selective area growth, multiple quantum well, light-emitting diodes, nonpolar

InGaN/GaN is a promising material for highly efficient solid-state lighting. However, two inherent difficulties with this material limit its potential as an efficient alternative light source. The large lattice mismatch between the GaN materials and the sapphire substrates upon which they are grown results in strain and high dislocation densities, and the presence of large polarization and piezoelectric fields caused by the strain inside the active region effects the efficiency of quantum wells used for light emission. The high dislocation density of GaN based materials, around $10^9 - 10^{10}$ cm^{-2}, is attributed to the 16% lattice mismatch between GaN and sapphire. The piezoelectric field inside the multiple-quantum-well active region causes spatial separation of the electron and hole wave functions. Consequently, thin quantum wells, approximately 3 nm thick, are grown to increase the radiative recombination efficiency.[1,2] The latter choice leads to inefficient electron capture and high carrier concentrations at the operating current that can lead to Auger recombination. Both of these effects have been implicated in the high current efficiency "droop" observed in blue light-emitting diodes (LEDs).[3–5] Growth on nonpolar or semipolar GaN substrates has been explored as methods for reducing the piezoelectric field and increasing the quantum well thickness to increase the radiative recombination efficiency in the active region of InGaN/GaN LEDs.[6–8] However, the high cost of these specialized substrates has prohibited widespread adoption.

Recently, GaN nanostructures have been studied due to their interesting characteristics, such as large surface-to-volume ratio and the exposure of different facets, other than the typical polar facet,

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c-plane. These nanostructures are fabricated on easily accessible substrates by different approaches: etching,[9,10] regrowth,[11] vapor−liquid−solid (VLS),[12−14] and selective area growth (SAG) by metal organic chemical vapor deposition (MOCVD).[15,16] In addition, GaN nanorods/nanowires exhibit unique physical properties that release strain at the growth heterointerfaces on the nonlattice-matched sapphire substrates by virtue of their nanometer scale size and the nanorods grow without dislocations.[17−19] Nanorods are also amenable to growth on lower cost substrates, such as silicon.[20−22] Core−shell InGaN/GaN multiple quantum wells (MQWs) have been demonstrated on GaN nanowires/nanorods for application to LEDs and lasers.[23−25] Electroluminescence has been observed in single nanowire LEDs with the core−shell structure.[26,27] Also, InGaN/GaN MQWs embedded in nanowires/nanorods along or around axial directions have been applied to LEDs.[28−31] However, less emphasis has been placed on the elimination of piezoelectric fields in the MQWs resulting from the growth orientations or different epitaxial growth techniques. In this study, semipolar and nonpolar facets are exposed on GaN nanorods grown vertically from the basal plane of the polar substrates. These exposed semipolar/ nonpolar facets can be utilized as growth templates for InGaN-based LED active regions to reduce or eliminate the piezoelectric fields in the active regions. The exposed sidewall nonpolar $\{1\bar{1}00\}$ facets are available for the growth of InGaN/GaN heterostructures with no piezoelectric fields and increased radiative recombination efficiency. MOCVD-grown GaN nanorods are expected to be high quality, dislocation-free templates for the subsequently grown structures.

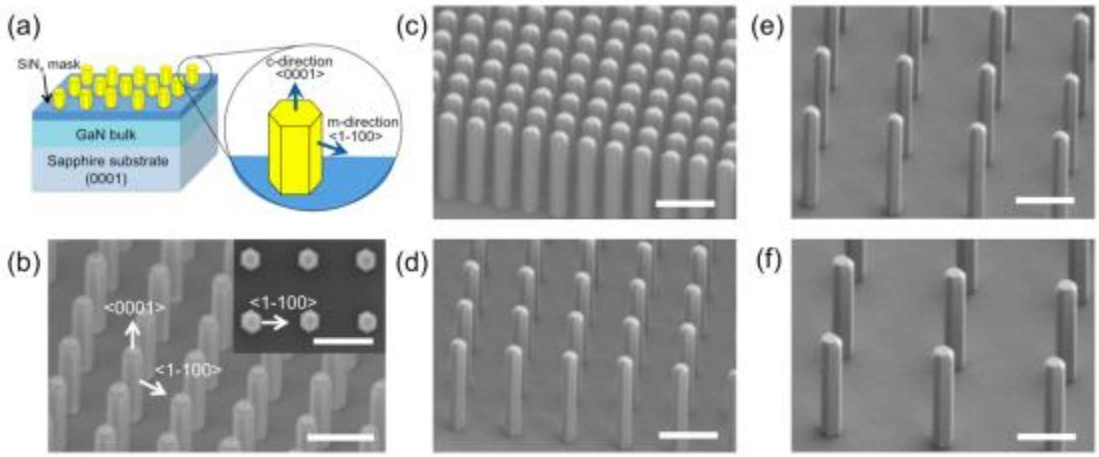

Figure 1. (a) Schematic diagram of a GaN nanorod array grown vertically from it substate. The enlarged diagram shows two dominant facets formed by GaN nanorods. (b) Uniform GaN nanorod array is grown by SAG by MOCVD. The arrows indicate the polar and nonpolar factes of the nanorods. In the inset, a top view of hexagonal GaN nanorods shows the six vertical sidewalls. Ordered GaN nanorod arrays grown on 250 nm, 500 nm, 750 nm, and 1 μm center-to-center spacings are demonstrated in (c−f), respectively. The scale bars are 500 nm in all figures. All the FE-SEM images were recorded at a 45° angle.

GaN substrates consisting of an epitaxial layer of c-plane GaN grown on sapphire were prepared for nanorod growth by patterning the substrate with a dielectric film containing a dot array of nanoscale openings prepared by electron beam lithography and reactive ion etching. A 20 nm thick SiNx layer was deposited onto Si-doped GaN layers by plasma enhanced chemical vapor deposition (PECVD). Poly(methyl methacrylate) (PMMA) was used as an electron beam resist on the dielectric mask prior to electron beam writing. The hole diameter was controlled between 70 and 250 nm by varying the writing parameters, and dot arrays with 250 nm to 1 μm center-to-center spacing were prepared for GaN nanorod growth. The pattern was transferred from the PMMA resist into the SiN_x dielectric mask using a CF_4 based reactive ion etching process, and the surface was then cleaned with solvents and further treated with oxygen plasma to remove any resist residue. The samples were then loaded into a close-coupled showerhead MOCVD system for GaN nanorod growth.

Semipolar or nonpolar planes have also been demonstrated in micrometer-scale stripe structures grown by modulating NH_3 injection to enhance lateral growth.[32,33] To achieve isolated nanorod

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structure with nonpolar surfaces, a pulsed growth mode was applied to enhance the vertical growth along ⟨0001⟩ directions.[15] Trimethylgallium (TMG) and ammonia (NH_3) were used as the precursors for the nanorod growth. The growth pressure was 200 Torr for the entire pulsed growth mode process. Various growth parameters, including growth temperature, gas flow rate, and precursor flow period, were adjusted to achieve GaN nanorods with high aspect ratios. The flow rates of TMG and NH_3 were 17.7 μmol/min and 67 mmol/min, respectively. InGaN/GaN multiple quantum wells were grown subsequently on the GaN nanorods using trimethylindium (TMI) and triethylgallium (TEG) as the indium and gallium sources, respectively. The flow rate of TEG was 20 μmol/min and that of TMI ranged between 3.4 and 17.0 μmol/min for different MQW growth tests. The growth pressure was increased to 300 Torr in nitrogen ambient to grow InGaN quantum wells and GaN barriers under continuous growth conditions. The growth temperature varied between 730 and 815 °C for different experimental purposes. NH_3 was injected into the chamber until the temperature decreased below 400 °C.

After nanorod growth, the surface morphology was evaluated by scanning electron microscopy (Hitachi S-4800 field emission FE-SEM) and the facet orientations of the GaN nanorod were deduced from the orientation of the underlying GaN material. The GaN nanorods were removed from the GaN bulk and dispersed on a transmission electron microscope (TEM) grid. The crystal structure of the GaN nanorods was investigated by TEM (JEOL 2100LB). GaN nanorods with InGaN/GaN MQWs were also investigated by FE-SEM and TEM. Because of the thickness of the nanorods, samples were sectioned by a focused ion beam milling system (JEOL MultiBeam JIB-4500), and the resulting slices were transferred to a TEM grid by a liftout process (Omniprobe Autoprobe 200). Photoluminescence measurements were performed by illuminating the samples with a focused 325 nm HeCd laser source to measure the light emission from the MQWs. Local

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emission from MQWs grown on GaN nanorods was studied by cathodoluminescence (CL) measurements (Horiba Scientific CLUE series equipped in the FE-SEM).

The schematic diagram of a GaN nanorod array grown by SAG is shown in Figure 1a. The enlarged nanorod shows two dominant facets, c- and m- planes, which are polar and nonpolar, respectively. For simplicity, semipolar planes, also observed in GaN nanorods, are not shown in the schematic diagram, but the detailed results are discussed later. From the FE-SEM results, all of the GaN nanorods grew normal to the c-plane and exhibited six nonpolar sidewalls with $\langle 1\bar{1}00 \rangle$ normals, as shown in Figure 1b. The hexagonal shapes of the GaN nanorods are clearly seen from the top in the inset of Figure 1b. GaN nanorods with diameters of ∼150 nm or less are grown on dot arrays with 250 nm to 1 μm center-to-center spacings. Uniform arrays of nanorods grown for 300 growth cycles with center-tocenter spacing from 250 nm to 1 μm are shown in Figure 1c−f. The height of the nanorods depends on several factors in the pulsed growth mode, such as the number of pulsed growth cycles, the fill factor of the growth mask, and so forth. The detailed growth mechanism will be published elsewhere.

The crystal strucure of the GaN nanorods without MQW growth was revealed by diffraction patterns and lattice images recorded in the $\langle 1\bar{1}00 \rangle$ and $\langle 11\bar{2}0 \rangle$ zone axes as shown in Figure 2.

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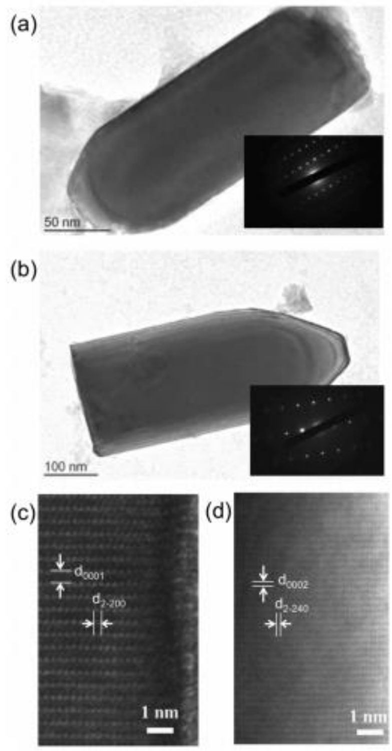

Figure 2. TEM images of GaN nanorods taken from $\langle 11\bar{2}0\rangle$ and $\langle 1\bar{1}00\rangle$ zone axis are shown in (a,b), respectively. The insets show wurtzite diffraction patterns in both zone axes. High-resolution images of (a,b) are shown in (c,d) respectively.

These GaN nanorods were removed from the substrate by sonication and dispersed on a TEM grid. The wurtzite crystal structure in the GaN nanorods was confirmed in both diffraction patterns and high resolution TEM images. No stacking faults or dislocations were observed in the GaN nanorods, so they may be suitable templates for subsequent MQW growth.

Typically, GaN nanorods 500 nm in height are grown over a period of 120 growth cycles. The nonpolar surfaces that form the vertical sidewalls are exposed for subsequent InGaN/GaN MQW growth. Under the continuous MQW growth InGaN/ GaN tends to grow laterally resulting in MQWs predominantly grown on the sidewalls of the GaN nanorods. When the MQW growth temperature is 815 °C and the TMI flow rate is 3.4 μmol/min, the emission peaks are located in the 380−420 nm wavelength region caused by low indium incorporation. The emission wavelength shift to 437 nm

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is achieved by reducing the growth temperature of MQWs from 815 to 730 °C. This result shows the controllability of emission wavelength from the MQWs grown on the sidewalls by increasing the indium content of the MQWs.

Changes in emission wavelengths with respect to opening diameters in the mask are observed in the MQWs grown on GaN nanorods. MQW growth was performed at 770 °C with 17.0 μmol/min TMI injection.

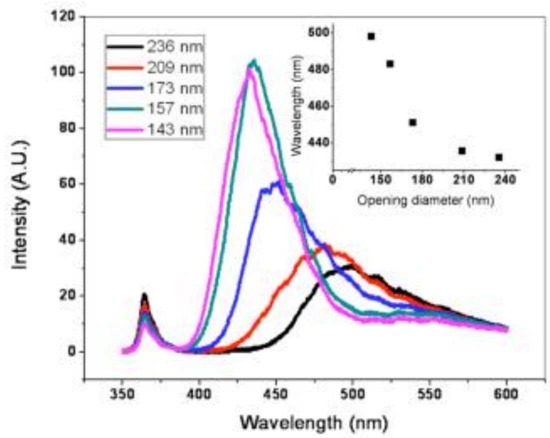

Figure 3. The emission wavelengths mesaured from nanorod samples grown with different opening sizes are shown in the spectra. The inset shows the emission wavelength decreases as the opening size on the dielectric mask increases.

By varying the opening size in the patterned dielectric growth mask, emission wavelengths from 432 to 498 nm were achieved, as shown in Figure 3. The variation of the peak wavelength with opening size is shown in the inset. The emission wavelengths are closely related to the indium composition and the quantum well width. Varying the opening size affects the enhancement of the growth rate that occurs in selective area growth which may affect both the indium composition and the QW thickness. However, the MQW thickness cannot be determined directly from the changes of the nanorod diameters, and extensive TEM studies of sectioned samples are required to determine the cause of the wavelength change. The portions of the sample from which these spectra are taken are grown simultaneously on different regions of the same wafer. The apparent reduction of the efficiency with increasing wavelength may be a consequence of unoptimized growth conditions for the longer wavelength portions of the sample rather than a fundamental effect. Despite the need for further studies, these results suggest that the emission wavelength may be controlled by careful mask design and control of the nonpolar plane surface area. They also suggest that precise design will be necessary to control the wavelength of emission in LEDs.

GaN nanorods with three pairs of MQWs were sectioned by focused ion beam and transferred onto a TEM grid for structure analysis. GaN nanorods are sliced in two different orientations to demonstrate the core−shell structure of InGaN/GaN MQWs. Bright-field TEM images of sectioned GaN nanorods with MQWs are shown in Figure 4. The surfaces of the nanorods were covered with a carbon layer to protect them from damage from the focused ion beam. The GaN nanorods sectioned along $\langle 11\bar{2}0 \rangle$ are shown in Figure 4a, and three boxed regions indicate the locations where magnified images were recorded and shown in Figure 4c−e. Three pairs of MQWs are clearly distinguishable on nonpolar planes. The exposure of three main facets in the nanorod resulted in different growth rates on the various planes.

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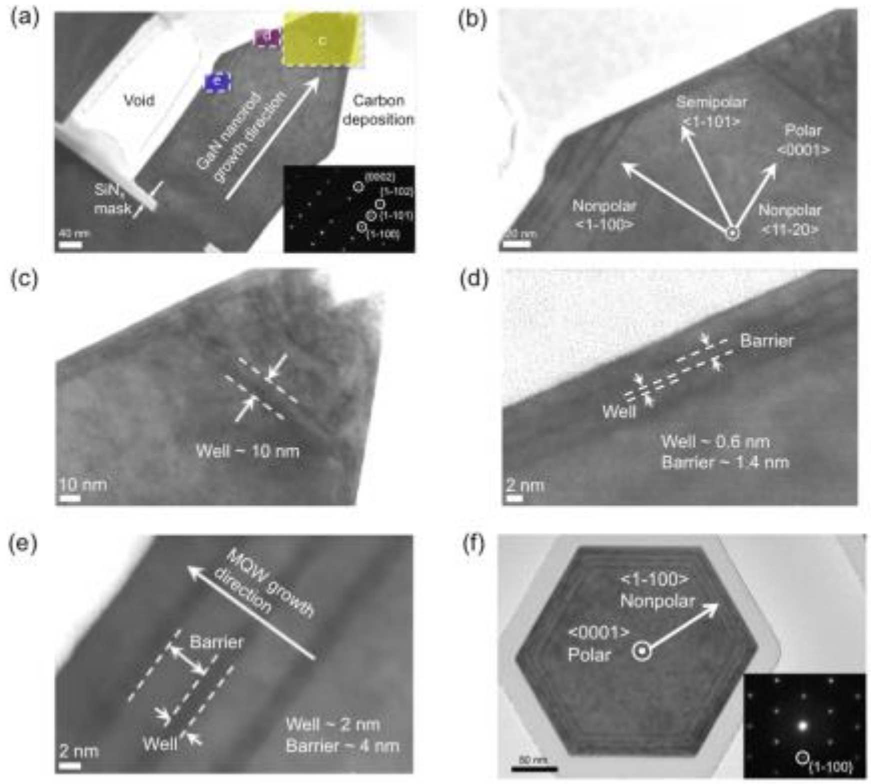

Figure 4. (a) The nanorod is sectioned along ⟨$11\bar{2}0$⟩-direction to investigate the MQW growth on three different planes. The inset shows the diffraction pattern of the ⟨$11\bar{2}0$⟩ zone axis and a beam blocker blocks the center beam. Three colored boxes indicate the locations where magnified images are taken in panels (c−e). (b) InGaN/GaN MQWs are grown on three different planes as indicated in the arrows. (c) TEM image shows a thick quantum well is grown on the c-plane, polar plane. (d) Thin MQWs are grown on the semipolar plane. (e) MQWs are grown on the nonpolar plane. (f) The nanorod is sectioned along ⟨0001⟩-direction to investigate the MQW growth on the periphery of the GaN nanorod. Three pairs of concentric hexagonal rings of InGaN are clearly seen in the image. The inset shows the diffraction pattern of the ⟨0001⟩ zone axis.

Figure 4b designates the orientations of MQWs grown on polar, semipolar, and nonpolar planes. The MQWs grown at the edge between semipolar and nonpolar are slightly thicker possibly due to the migration of growth species from the semipolar plane to the nonpolar plane. Because of the hexagonal pyramid shapes of the GaN nanorod tips after MQW growth and the nanometer-scale dimension of the tip, interfaces between barriers and wells are difficult to distinguish on this plane. In addition, the interface between well and barrier is not as abrupt as for other orientations presumably due to the fast growth rate on the c-plane and possibly due to migration of species from

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the slow growth semipolar facet. In Figure 4c, only the first well is clearly distinguishable at the nanorod tip. The QW grown on the c-plane is considerably thicker than typically used for efficient light emitters. This results in poor emission efficiency due to the large spatial separation between electron and hole wave functions.

MQWs grown on the semipolar and nonpolar planes are shown in Figure 4d,e, respectively. In Figure 4d, the MQWs grown on semipolar planes are perhaps too thin to capture the electrons and holes efficiently for radiative recombination. Similar to the results for GaN nanosheet structures, the growth rates for each plane were estimated from TEM images, resulting in the following sequence: polar (0001) > nonpolar $\{1\bar{1}00\}$ > semipolar $\{1\bar{1}01\}$.[34] The nanorod sectioned along ⟨0001⟩-direction is shown in Figure 4f. Three pairs of MQW are clearly seen and are equally distributed on the six nonpolar planes.

MQWs grown on the c-plane may affect the light emission when the c-plane is present on the nanorod template. To avoid or minimize MQWs grown on the polar plane, the c-plane surface area must be eliminated. Differences in growth rates on the polar, semipolar, and nonpolar planes are utilized, and the cplane is terminated by applying a continuous GaN growth for 10 min after the pulsed GaN nanorod growth. The slow growth rate of inclined semipolar planes pinches off the c-plane eliminating it after 10 min of continuous growth, as shown in Figure 5a. The sharp tips of the nanorods are shown in the inset. After the sample was cleaved to observe the emission from the sidewalls, a CL spectrum was recorded in the region outlined in the image of Figure 5b. CL mappings recorded at 430 and 480 nm are shown in Figure 5c,d. The peak emission at 430 nm is attributed to the MQWs grown on the nanorod sidewalls. The longer emission wavelength is especially observed at the edges between semipolar and nonpolar planes. This result is consistent with observation from the crosssectional TEM image in Figure 4b. No light emission is observed from the nanorod tips due

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to exlusion of MQWs grown on the c-plane. In this way, piezoelectric fields in the MQWs can be greatly reduced or eliminated due to only the presence of semipolar and nonpolar planes from the GaN nanorods serving as growth templates for MQWs.

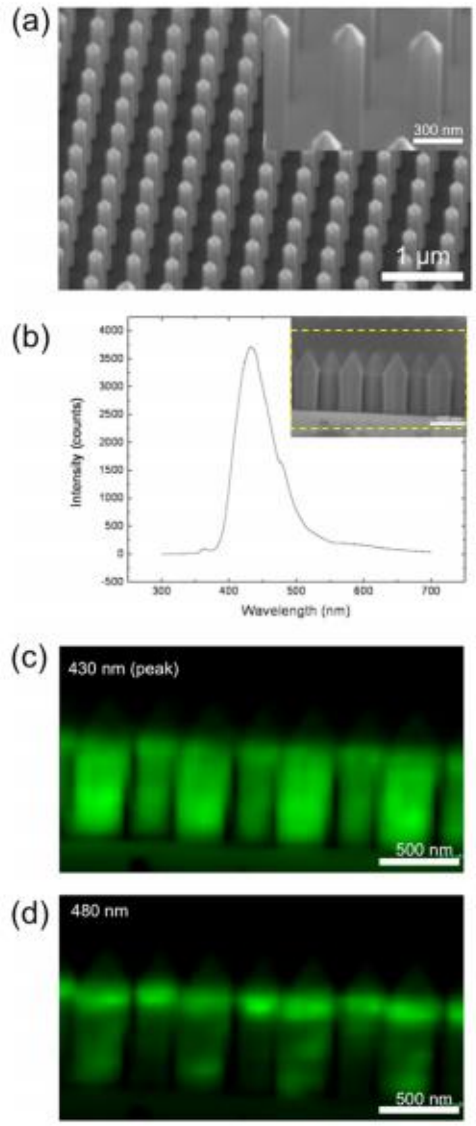

Figure 5. (a) An uniform array of GaN nanords with sharp and termianted c-plane is achieved after 10 min of continuous GaN growth. The inset shows the c-plane is minimized or terminated on top of the nanorods. The FE-SEM image was recorded at a 45° angle. (b) CL spectrum is recorded at the boxed region in the inset. CL mapping results at 430 and 480 nm are show in (c,d), respectively.

InGaN/GaN MQWs on uniformly grown GaN nanorod arrays are produced by selective area growth by MOCVD. Tunable emission over a 60 nm wavelength span from near UV to blue green

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is demonstrated by varying the mask design. From cross-sectional TEM images, the InGaN/GaN MQWs are predominantly grown on the $\{1\bar{1}00\}$ nonpolar planes. The growth of MQWs on c-plane can be minimized or eliminated by terminating the c-plane before MQW growth. No light emission from c-plane under these conditions is observed in the CL mapping, confirming that MQWs grow only on semipolar and nonpolar planes. The elimination of piezoelectric fields is expected to improve radiative recombination. Therefore, the exposed nonpolar sidewalls of GaN nanorods are potential templates for the InGaN/GaN MQWs growth to reduce the strain-induced piezoelectric field in the active regions of InGaN/GaN light-emitting diodes. Reduction of the piezoelectric fields may mitigate the causes of efficiency droop in LEDs.

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