Epitaxial Growth of GaN Core and InGaN/GaN Multiple Quantum Well Core/Shell Nanowires on a Thermally Conductive Beryllium Oxide Substrate

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ABSTRACT: Beryllium oxide (BeO) belongs to a very unique material family that exhibits the divergent properties of high thermal conductivity and high electrical resistivity. BeO has the same crystal structure as GaN, and the absolute difference in the lattice constants is less than 17%. Here, the growth of GaN nanowires (NWs) on the polycrystalline BeO substrate is reported for the first time. The NWs are grown by a vapor−liquid−solid approach using a showerhead-based metal−organic chemical vapor deposition. The growth direction of NWs is along the m-axis on all planes of the substrate, and it is confirmed by transmission electron microscopy (TEM) and selected area electron diffraction (SAED) patterns. The vertical and tilted growth of NWs is due to the different planes of the substrate such as the m-plane, a-plane, and semipolar planes and is confirmed by X-ray diffraction. Subsequently, the GaN shell and InGaN/GaN multiple quantum wells (MQWs) are coaxially grown using a vapor−solid approach in the same reactor. A very high crystal quality is verified by TEM and SAED and is also confirmed by measuring the photoluminescence. The optical emission is tuned for the entire visible spectrum by increasing the indium incorporation in InGaN quantum wells. The conformal growth of InGaN/GaN MQW shells and the defect-free nature of the structure are confirmed from spatially resolved cathodoluminescence. This study will provide a platform for researchers to grow GaN NWs on the BeO substrate for a range of optical and electrical applications.

INTRODUCTION

GaN is known to exhibit superior characteristics such as small Auger effects, high radiative recombination rate, high electron mobility, biocompatibility, and a tunable band gap from near-infrared (InN = 0.7 eV) to deep ultraviolet (AlN = 6.12 eV) by alloying it with indium and aluminum, respectively. Such unprecedented characteristics make GaN a promising material for electrical and optical applications such as high electron mobility transistors, light-emitting diodes (LEDs), photodetectors (PDs), photoanodes, and piezoelectric nanogenerators. Conventionally, GaN is grown on a sapphire substrate, which is a thermal and electrical insulator. Such properties hinder sapphire to become the most promising candidate to be used for a high-power device because an extra arrangement would be needed to manage the heat dissipation from the device. Therefore, a substrate with high thermal conductivity and high electrical resistance is essential to fabricate efficient high-power devices. The materials exhibiting these two divergent properties are beryllium oxide (BeO), diamond, aluminum nitride, silicon carbide, and single-crystal boron nitride.

Among all the above-mentioned materials, we have opted for BeO because it has the same crystal structure as GaN and is least studied yet. In general, good electrical insulators such as plastics or ceramics exhibit a low range of thermal conductivities, while electrical conductors such as metals show high thermal conductivity. The thermal conductivity of BeO is similar to that of aluminum metal, while the electrical resistivity is comparable to the best of the plastics. In addition to these two properties, BeO exhibits a high bulk modulus (212 GPa, the hardest known material), is the lightest among the wurtzite-structured materials, exhibits a high melting point (2532 ± 10 °C), is the lightest among the wurtzite-structured materials, exhibits a high melting point (2532 ± 10 °C), exhibits a high band gap (10.6 eV, direct), exhibits low dielectric losses, and is transparent to X-
ray/ultraviolet/infrared light. Owing to a very high thermal conductivity (330 W/mK), it has been used for a diverse range of heat transport applications such as heat dissipation in nuclear reactors, heat transfer in cryogenic systems, and refractory materials. Therefore, it could be the best material to transport the heat of high-power electrical and optical devices.

The prerequisite of the substrate for the epitaxial growth of GaN includes the hexagonal crystal structure and very low lattice mismatch (<7% is preferable). The crystal structure of BeO is the same as that of GaN, while the lattice parameters are $a = 2.698 \, \text{Å}$ and $c = 4.380 \, \text{Å}$, exhibiting a lattice mismatch of 15−17%. This lattice mismatch is relatively small compared with that of sapphire and GaN (∼30%). BeO could be employed as a substrate for the growth of GaN, but a single-crystal wafer is not available. It is available in a polycrystalline form exhibiting crystalline domains with different crystal orientations. Therefore, the best possible approach could be the growth of single-crystal nanowires (NWs) with identical growth directions. The growth direction of GaN NWs can be controlled with the help of either catalyst composition or substrate orientation during the vapor−liquid−solid (VLS) growth mode. To circumvent the possible doping of the metal catalyst as an impurity into the GaN-based active region, the active regions could be grown radially in the vapor−solid (VS) growth mode. To the extent of our knowledge, it is the first report on the GaN-based NW growth by metal−organic chemical vapor deposition (MOCVD) on the BeO substrate. Therefore, the study would provide a fundamental understanding of the growth approach and would lead the way for further studies and applications.

Herein, we report the growth of GaN-based NWs on the BeO substrate using MOCVD by adopting a two-step VLS−VS growth technique. In this work, a polycrystalline BeO is used as a substrate, followed by the deposition of a Au thin film as a catalyst. Then, the GaN core NWs are grown in the MOCVD reactor using the VLS growth approach. To avoid the possible Au contamination into the active region, the growth is switched from axial to radial by shifting to the VS mode from the VLS mode. The morphology and structural properties of single-crystalline GaN-based NWs are studied by SEM and transmission electron microscopy (TEM). The optical emission is tuned for the entire visible spectrum by increasing the indium composition in the InGaN MQW shells. The demonstration of the single-crystalline GaN NW growth onto the BeO substrate will pave the way to fabricate GaN-based structures onto the thermally conducting and electrically insulating BeO substrate for optical and high-power electrical applications.

**RESULTS AND DISCUSSION**

To grow the single-crystalline GaN on polycrystalline BeO, the best approach is the growth of single-crystalline GaN NWs with identical crystal orientation. The GaN NWs were grown on the BeO substrate using Au as a catalyst by the VLS technique. After the deposition of the Au thin film, the samples were loaded into the reactor, and in situ low-temperature deposition of indium and gallium was conducted, followed by annealing for the agglomeration of the Au/Ga/In metal−alloy catalyst as illustrated in Figure 1a. The scanning electron micrograph of a bare BeO substrate is shown in Figure 2a. The image reveals that the substrate is composed of several grains depicting a polycrystalline nature of the BeO substrate. The inset shows the bird’s eye view of the BeO substrate. As the precursors were introduced to the reactor, the GaN NWs were grown and their schematics are shown in Figure 1b. Figure 2b(i),(ii) shows the low- and high-magnification images of the GaN core NWs, respectively. It is revealed that the NWs are uniformly grown on all grains irrespective of the host crystal orientation. The NWs were circular having a diameter, length, and surface density of 19 ± 2 nm, 4.4 ± 0.1 μm, and (3.4 ± 0.1) × 10^9 cm^-2, respectively.

It is important to mention that the GaN NWs grown using Au or Ni as a catalyst show superior quality as compared to self-catalyzed NWs. They claimed that the optical performance was also degraded because of the defects originated from the metal catalyst incorporation into epitaxial NWs. To suppress the possible contamination of the metal catalyst in the active region of optical devices, we switched to the VS mode for the radial growth of InGaN/GaN multiple quantum well (MQW) shells, as shown in the schematic in Figure 1c. First, the GaN shell is grown around the GaN core NW, which acts as a host for the growth of InGaN/GaN MQW shells. Subsequently, the InGaN/GaN MQW coaxial shells are grown, and their low- to high-magnification SEM images are shown in Figure 2c(i),(ii). Interestingly, GaN NWs are aligned in two different directions, that is, vertically aligned and tilted NWs. The degree of alignment depends on the orientation of the domains of the BeO substrate. Approximately, half of the domains of the BeO substrate are covered with vertically aligned NWs, as shown in Figure 2d(i),(ii). Figure 2d(i) shows a high-magnification image of vertically aligned NWs, and a very clear boundary between the vertically aligned and tilted NWs can be seen in Figure 2d(ii). The low- and high-magnification images of tilted NWs are shown in Figure 2e(i), (ii), respectively.
The crystal quality and the growth direction were examined by conducting TEM. Figure 3a−c shows the low- to high-magnification TEM images of the GaN core NWs, indicating that the NWs are straight and smooth with a regular diameter of 19 ± 2 nm. Figure 3d shows a high-magnification image that depicts the planes up to the atomic level. To have an insight, a very clear image is shown in Figure 3e, in which the interplanar distance perpendicular to the growth direction is 2.8 Å, which is assigned to the m-axis. The high-magnification image in Figure 3e shows the planes up to the atomic level, indicating the growth direction of GaN core NWs along the m-axis. The selected area electron diffraction (SAED) patterns are recorded from the same orientation, and the diffraction patterns are regular, which confirms the monocrystalline nature of GaN NWs. The zone axis of the SAED patterns is (0001), and the hexagonal shape of the SAED patterns confirms the growth direction of GaN core NWs along the m-axis; hence, the SAED patterns verify our interpretation constructed from interplanar spacing.

To utilize the core NWs for optical applications such as LEDs or PDs, the InGaN/GaN MQW core/shell structure was grown laterally. Prior to the growth of the InGaN QW shell, the GaN shell was grown around the GaN core that converted the geometry of NWs from circular to triangular. The triangle-shaped GaN shell was annealed in NH3 at the same temperature, adopting the same approach as reported in our previous work. The grown GaN shell acted as a host for the monocrystalline growth of InGaN/GaN MQW shells. High-quality InGaN/GaN MQW NWs were grown.
crystal quality InGaN/GaN MQWs are expected to grow around the triangle-shaped GaN shells. After the InGaN/GaN MQW coaxial shell growth, TEM measurement was conducted, and the low- to high-magnification images of InGaN/GaN MQW coaxial NWs are shown in Figures 4a–c. The diameter of NWs increased to 86 nm after the growth of InGaN/GaN MQW shells, which is the evidence of shell growth. Figure 4d confirms the growth direction along the same axis, that is, $m$-axis, as GaN core NWs because the 2.8 Å interplanar spacing is attributed to the growth along the $m$-axis, as shown in Figure 4e. Additionally, the high-resolution TEM lattice fringes are well spaced, which indicates the high crystal quality of GaN NWs with fewer dislocations and defects. The diffraction spots from the SAED patterns are regular and correspond to the diffraction direction of [0001]. The growth direction is parallel to the $m$-axis, the same as the GaN core NWs as confirmed by the SAED patterns shown in Figure 4f. To evaluate the cross-sectional geometry of NWs after VS growth, the line profile of the NW cross section is conducted, and the scanned area is shown in Figure 4g. Figure 4h shows the geometrical profile of NWs that is measured as a distance from the camera over the cross-sectionally scanned width of NW, which shows the triangular shape of the NWs. The $y$-axis shows the height from the NW surface to the camera, and the $x$-axis shows the line scanning perpendicular to the GaN core NW growth direction. Hence, the profile validates the triangular cross section of the NWs. The cross-sectional evolution of NWs from circular to triangular during the VS growth can be understood using the Wulff plots known as the $\nu$-plots.\textsuperscript{31−33} During the VS growth mode, the growth occurs only at the sidewalls of the NWs without catalyst assistance. The VS growth starts at different growth rates on all the possible perpendicular planes to the $m$-axis. Within the convex geometry, slow-growing facets define the surface shape, and fast-growing facets disappear with growth; consequently, the $\nu$-plot of the crystallographic plane dictates the facet evolution. Here, the slowest perpendicular plane is (0001$^\parallel$), and the second slowest plane is (112$^\parallel$$. Therefore, the triangle-shaped NWs are surrounded by the (112$^\parallel$) planes with the basal plane of (0001$^\parallel$). Hence, this is the most appropriate approach to understand the sidewall evolution of shell growth around the GaN core NWs.

Figure 4. Transmission electron micrographs of InGaN/GaN MQW coaxial NWs: (a) low-magnification image, (b) increased width of NWs due to InGaN/GaN MQW shell growth, (c,d) high-magnification image and plane orientation, respectively, (e) interplanar spacing of 2.8 Å along the growth direction, confirming the growth direction along the $m$-axis, and (f) SAED patterns verifying the growth direction along the $m$-axis. (g) Area investigated by the profiler to check the geometrical shape and (h) triangular shape verification by the profiler.

Figure 5. (a) XRD of the BeO substrate using the grazing angle of incidence of 5° to record the 2$\theta$ scan. The demonstration of the growth evolution of a variety of substrate planes: (b-i) growth on the (101$^\parallel$0) plane, (b-ii) corresponding SEM micrograph, (c-i) growth on the (101$^\parallel$1) plane, (c-ii) corresponding SEM micrograph, (d-i) growth on the (112$^\parallel$0) plane, and (d-ii) corresponding SEM micrograph.
It is worth mentioning that the GaN NWs grow along the m-axis on all grains of the BeO substrate. To confirm the m-axis growth on all grains, TEM was conducted for several samples, and all the time, the result was identical, that is, m-axis growth direction. During the VLS growth mode, it is possible to control the growth direction of GaN core NWs on any substrate such as the growth of m-axial, a-axial, or c-axial NWs using the same substrate by controlling the catalyst composition.27,34

To evaluate the BeO substrate crystal structure, grazing angle X-ray diffraction (XRD) is conducted while the detector was moved between 20 and 80°, as shown in Figure 5a. The XRD patterns reveal that the dominant plane of the substrate is (1010), followed by (1011) and (1120) planes. The minor planes are (0002), (1012), and (1013). Here, only the growth is discussed on major dominant planes, that is, (1010), (1011), and (1120). The m-axial NW growth on the (1010) plane is vertical, having an angle of 90° with the substrate, as shown in Figure 5b(i) and the corresponding SEM image is shown in Figure 5b(ii). In the case of (1011) plane, the m-axial growth will be tilted on the substrate, as illustrated in the schematic of m-axial growth on the (1011) plane exhibiting the possible angle of 62°, as shown in Figure 5c(i), and the SEM image of m-axial growth on the (1011) plane is shown in Figure 5c(ii). The third major plane is (1120), and the possible m-axial growth direction on the (1120) plane will have a tilting angle of 60° as illustrated in the schematic of Figure 5d(i), and its SEM image is shown in Figure 5d(ii). The growth of m-axial GaN NWs on the (0002) plane will not take place because the m-axis has 90°. The m-axial growth angles with the substrate having (1012) and (1013) planes will be 43 and 32°, respectively. The underlying reason for the uniform and single-crystalline growth direction of m-axial GaN NWs on a specific plane of the BeO substrate can be explained by the help of controlled catalyst composition. To designate the precise mechanism exactly for the growth direction, which is governed by the catalyst composition, is very difficult. By using the in situ TEM measurements of nitride NWs, a recent report claims that there are a variety of parameters by which the growth mechanism is governed, such as supersaturation of the catalyst, the nucleation step at the interface of all the three phases, and the interface of the catalyst and NW.35 Additionally, the interfacial energy at the NW–catalyst interface that can be controlled by the catalyst composition plays a key role in controlling the growth direction of NWs.27

Following agglomeration, the precursors are introduced into the reactor; the catalyst absorbs the precursors and super-saturates, and nucleation of GaN nanocrystals starts at the liquid–solid interface. The GaN nanocrystals start to grow in random directions at this stage, but the m-plane facet grows faster. The rest of all domains start to decline and are eliminated from the growth front because of geometrical constraints inflicted by the dimensions of the catalyst. Consequently, the NW with the m-axis prevails, and single-crystal m-axial GaN NWs on the polycrystalline substrate are achieved.

To employ the GaN NWs for real optical applications in the visible spectrum, the InGaN/GaN MQWs are of primary interest. Therefore, the InGaN/GaN MQWs were grown coaxially on triangular sidewalls. The band gap can be tuned spanned between blue and red emissions by growing InGaN-based QWs, either by varying the thickness or by controlling the indium composition in InGaN QWs.29,30 Here, the optical emission was tuned for the entire visible spectrum by controlling the indium incorporation in InGaN QWs. To evaluate the optical properties of InGaN/GaN MQW NWs, the room-temperature PL was conducted on the NW ensemble for all the three samples, and the spectra are shown in Figure 5. The center of the peak emission of samples (LIn), (MIn), and (HIn) are found at 477, 539, and 586 nm, respectively. The full width at half-maximum (fwhm) increased with increased indium incorporation into InGaN QWs as 117, 158, and 217 nm for samples (LIn), (MIn), and (HIn), respectively. The increased fwhm with In incorporation is due to the increased defects and In segregation. Further, the band-to-band transition of pristine GaN was not observed in all the three samples, indicating the conformal growth of InGaN/GaN MQW coaxial shells from the top to the bottom of the NWs. The local emission from InGaN/GaN MQW core/shell NWs is examined by performing cathodoluminescence (CL). To prepare samples for CL spectroscopy, the InGaN/GaN MQW core/shell NWs were cut by a blade from the BeO substrate, sonicated in ethanol, and few drops of the solution were poured on the Si substrate. The SEM and the corresponding CL images of NWs of the samples (LIn), (MIn), and (HIn) are shown in Figure 6a(i), (ii), c(i), (ii), d(i), (ii), respectively.

Figure 6. (a) Photoluminescence (PL) emission spectrum of InGaN/GaN MQW coaxial NWs as a function of indium ratio inside the InGaN QWs. (b-i, b-ii), (c-i, c-ii), and (d-i, d-ii) SEM images and the corresponding panchromatic CL emission of samples Lin, Min, and Hin, respectively. The scale bar is 500 nm.

The panchromatic CL image shows the overall emission from the NWs, and it is revealed that the GaN core is covered with InGaN/GaN MQW shells from top to bottom. The uniform emission is attributed to the defect-free growth of NWs. The InGaN/GaN MQWs grow on the semipolar facet as the GaN core NWs grow along the m-axis.29,30 The overlap of electron and hole wave functions increases because of the suppressed piezoelectric and polarization charges and leads to higher radiative transitions from InGaN QWs. Therefore, it results in higher internal quantum efficiency (IQE). Hence, we have demonstrated for the first time the growth of GaN NWs on the BeO substrate using a two-step growth approach of VLS and VS, which exhibits very high crystal quality and can be used for a variety of optical and electrical applications such as LEDs, PDs, and NW-based transistors.
CONCLUSIONS

In summary, we have demonstrated the growth of high crystal quality m-axial GaN NWs on the polycrystalline BeO substrate by MOCVD (showerhead-based). The growth of GaN core and InGaN/GaN MQW coaxial shells is conducted by adopting a two-step growth approach with sequential VLS and VS modes. A very smooth surface of GaN core and InGaN/GaN MQW NWs is confirmed by SEM. The single crystallinity of core and core/shell NWs is confirmed by the TEM measurements. SAED patterns are used to confirm the growth direction along the m-axis, and the same is confirmed for InGaN/GaN MQW shells; further, the InGaN/GaN MQWs are grown on semipolar growth facets, which reduced the polarization and piezoelectric charges inside the QWs and resulted in suppressed quantum-confined start effect, which lead to high IQE. The vertical and tilted growth is explained with the help of XRD and the NW growth angles. The optical measurements, the indium incorporation in InGaN QWs. Finally, the spatial emission properties were examined by conducting CL measurements. The demonstration of GaN-based NWs on the thermally conducting BeO substrate underlines the potential of this scalable, facile, and efficient approach to use such structures to fabricate a variety of optical and electrical devices.

EXPERIMENTAL PROCEDURE

To use the polycrystalline BeO substrate for the growth of single-crystalline GaN NWs, a 0.8 nm thick Au thin film was evaporated on the substrate by an e-beam evaporator. Subsequent to the Au deposition on the BeO substrate, the samples were loaded into the MOCVD reactor to deposit indium and gallium for the formation a Au/Ga/In metal—alloy catalyst. The low-temperature in situ deposition of gallium and indium was conducted using trimethylgallium (TMGa) and trimethylindium (TMIn) as respective precursors for gallium and indium. Then, the samples were annealed at 840 °C for 1400 s in the H2 environment at 25 Torr. As a result, the thin films of Au, Ga, and In agglomerated, and an alloy catalyst of Au/Ga/In was formed in a spherical shape and acted as a catalyst for VLS growth.

Subsequent to the successful agglomeration of the Au/Ga/In alloy catalyst, the precursors for gallium and nitrogen were turned open to the MOCVD reactor; TMGa and NH3 were used as precursors for gallium and nitrogen, respectively. The flow rates of TMGa and NH3 were maintained as 55.4 μmol·min⁻¹ and 3.57 mmol·min⁻¹, respectively, with a V/III ratio of 64. The growth time was fixed for 3000 s, and the temperature and pressure were set as 850 °C and 25 Torr, respectively.

After the growth of GaN core NWs, the goal was to demonstrate the shell growth by the VS approach. To grow the GaN shell, the V/III ratio was escalated to 11,300 with the flow rates of TMGa and NH3 as 31.6 μmol·min⁻¹ and 357 mmol·min⁻¹, respectively, and the pressure was fixed at 200 Torr during the VS growth mode. After the growth of the GaN shell, a postgrowth nitridation was conducted to improve the crystal quality up to the atomic level as reported in our previous work.30 Subsequent to the shell growth of GaN, the InGaN-QW/GaN-barrier shells were grown, and the flow rates of TMGa and NH3 were maintained as 31.6 μmol·min⁻¹ and 357 mmol·min⁻¹, respectively. To grow the InGaN QW, the flow rates of TMGa and NH3 were fixed at 27.6 μmol min⁻¹ and 357 mmol min⁻¹, whereas the flow rate of TMIn varied as 17.3, 34.6, and 51.9 μmol min⁻¹ for samples LIn, MIn, and HIn, respectively. The ratio of the flow rates of TMIn/(TMIn + TMGa) for samples LIn, MIn, and HIn varied as 0.35, 0.52, and 0.62, respectively. The total number of pairs of InGaN/GaN MQWs was fixed as 0.5 for all samples. Subsequently, the postgrowth nitridation was conducted at 850 °C for 500 s in the NH3 environment; then, the reactor was cooled down to 80 °C while the NH3 flow was maintained up to 250 °C to avoid the crystal degradation.

A showerhead-based commercial MOCVD (CCS-FT 19 × 2 in., AIXTRON) is used for NW growth. Thereafter, the morphology of the NWs is investigated by a field emission scanning electron microscope (JSM-6700, JEOL). A field emission transmission electron microscope (JEM-2100F, JEOL) is used to determine the interplanar spacing using a high-resolution micrograph; additionally, SAED patterns were also recorded using the same machine. The specimen was prepared by cutting GaN NWs using a blade, followed by their sonication in ethanol. Then, a couple of solution drops were poured on the TEM grid, and the specimen was dried at 55 °C using a hot plate. The samples were kept under vacuum before loading into the TEM chamber. The crystal structure of the BeO substrate was characterized by performing XRD using an incident grazing angle of 5° while the 2θ of detector ranged between 20 and 80°. The room-temperature PL spectroscopy of the InGaN/GaN MQW NWs is conducted using a spectrometer (f = 0.5 m, Acton Research Co. Spectrograph 500i) and an intensified charge-coupled device (P1-Max3) (Princeton Instruments) with a diode-pumped solid-state laser (Ekpla), where the excitation wavelength of the laser was 266 nm with an optical power of 30 mW. Spatially resolved CL spectroscopy (Gatan MonoCL4) is carried out to evaluate the local emission and the uniformity of the InGaN/GaN triangle-shaped MQW shells by Gatan MonoCL4 installed with the scanning electron microscope (FEI, XL 30S FEG) while the acceleration voltages were fixed at 15 KV.

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https://dx.doi.org/10.1021/acsomega.0c02411
ACS Omega 2020, 5, 17753–17760
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Notes

The authors declare no competing financial interest.

Acknowledgments

This work was supported by the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science, and Technology (2018R1A6A1A03024334 and NRF-2019R1A2C1006360). The samples were grown by using MOCVD (Aixtron-CSI5276) at the Energy Convergence Core Facility in Chonnam National University.

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