Comparison of GaN nanowires grown on c-, r- and m-plane sapphire substrates

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Abstract

Gallium nitride nanowires were grown on c-plane, r-plane and m-plane sapphire substrates in a showerhead metalorganic chemical vapor deposition system using nickel catalyst with trimethylgallium and ammonia as precursors. We studied the influence of carrier gas, growth temperature, reactor pressure, reactant flow rates and substrate orientation in order to obtain thin nanowires. The nanowires grew along the $\langle 10\overline{1}1 \rangle$ and $\langle 10\overline{1}0 \rangle$ axes depending on the substrate orientation. These nanowires were further characterized using x-ray diffraction, electron microscopy, photoluminescence and Raman spectroscopy.

Keywords: GaN, nanowire, growth, MOCVD, nickel catalyst, sapphire substrates

1. Introduction

Gallium nitride (GaN) nanowires (NWs) have been of interest for nanoscale photonic [1, 2, 3, 4, 5, 6, 7] and electronic [8, 9, 10] device applications. The growth of GaN NWs by catalyst mediated [8, 9, 11, 12, 13] as well as catalyst free [6, 14, 15, 16] methods have been reported. Unlike InAs, where $\langle 111 \rangle$ oriented wires are usually obtained [17, 18, 19], III-nitride NW growth has been observed along different crystal directions and is strongly dependent on growth conditions, the substrate and its orientation.

In most of the reports of GaN NW growth the axis along the nanowire length (or the growth direction) is along either the polar $\langle 0001 \rangle$ direction (i.e. c-axis) or the non-polar directions $\langle 11\overline{2}0 \rangle$ (a-axis) and $\langle 1\overline{1}00 \rangle$ (m-axis). On (0001) c-plane sapphire, Gottschalch et al. [11] reported the

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growth of vertical wires growing along c-axis using gold as catalyst, while Ji et al. [20] obtained wires growing along the a-axis with nickel catalyst. On (1102) r-plane sapphire using nickel-catalysed metalorganic chemical vapor deposition (MOCVD), Zhou et al. obtained wires tilted on the substrate and growing along m-axis. [12] On the other hand, Wang et al. [13] and Qian et al. [4] reported the growth NWs along the a-axis on r-plane sapphire with nickel catalyst. The growth of inclined GaN nanorods along the c-axis using m-plane (1100) sapphire as substrate without any foreign catalyst was reported recently. [16, 21] There are very few reports on the growth of GaN NWs along semipolar directions. The growth along the semi-polar <1011> direction has been reported on c-plane sapphire by Park et al. by reaction of metallic Ga and gaseous NH$_3$ by using nickel as the catalyst. [22] Peng et al. also reported growth along <1011> but on graphite substrates, using a mixture of Ga$_2$O$_3$ and C along with NH$_3$ as precursor, by a hot-filament CVD, without any catalyst. [23] Tessarek et al. obtained GaN wires via a self-catalyzed method by MOCVD, on different planes of sapphire, namely c-, r-, a- and m-plane. These wires grew invariably along the c-axis with diameters more than 200 nm. [21] There are, however, hardly any reports that compare GaN NW growth on different substrate orientations in the same run. We report the growth of thin, nickel-catalysed, GaN NWs on c-, r- and m-plane sapphire substrates under identical conditions. We studied the dependence of growth direction on the substrate orientation with other growth conditions kept similar, and characterized the samples using electron microscopy, x-ray diffraction, photoluminescence and Raman spectroscopy.

2. Growth of nanowires

GaN NWs were grown using MOCVD with nickel catalyst and trimethyl-gallium (TMGa) and ammonia (NH$_3$) as precursors. The substrates were cleaned, drop-coated with nickel nitrate hexahydrate solution (∼ 0.01 M), blow-dried with N$_2$ gas and annealed in hydrogen to form metallic nickel nanoparticles which subsequently served as the catalyst particle. [13, 24] This method to form the catalyst particles is more convenient than techniques involving evaporation of nickel or gold. The size of the catalyst particle can be controlled by the annealing time and temperature (See Supplementary Information section II for details). Since the exact composition and phase (solid/liquid) of the nickel-gallium alloy that serves as the catalyst during growth is unknown, the NW growth mechanism could be either vapour-liquid-solid (VLS) or vapour-solid-solid (VSS). From a post-growth compositional analysis of the catalyst particle (not presented here), we believe that at the growth temperatures used in our experiments (∼ 840 °C) GaN NW growth
happens via a VSS process. We also tried to grow GaN NWs using gold nanoparticles as catalyst. Gold catalysed wires grew slower than their nickel assisted counterparts under similar growth conditions, as reported by Zhou et al.\cite{12}

We varied the temperature, reactor pressure and rate of flow of precursors in order to obtain thin and non-tapering NWs. The growth of NWs was sensitive to the reactor conditions. We first discuss the effect of carrier gas on growth. After annealing the nickel nitrate hexahydrate coated sapphire substrates in a hydrogen environment to produce nickel nanoparticles, we could grow GaN NWs in a pure nitrogen ambient. The scanning electron micrograph (SEM) of the NWs is shown in Figure 1(a). The wires typically had a triangular cross section (Figure 1(b)). However, under similar reactor conditions in an H$_2$ ambient or in a mixture of equal volumes of N$_2$ and H$_2$, we did not obtain any wires. Instead, we obtained just hemispherical gallium droplets (Figure 1(c),(d)), unlike earlier reports.\cite{10}

NWs were grown on different orientations of sapphire namely c-plane, r-
plane and m-plane. The NWs obtained from the same growth run at 840 °C and 150 torr with \( \sim 45 \, \mu\text{mol/min} \) (\( \sim 1 \text{sccm} \)) NH\(_3\) flow and 7.8 \( \mu\text{mol/min} \) (2 sccm) TMGa flow on these substrates are shown in Figure 2. On these substrates we obtained NWs as thin as 20 nm diameter with a triangular cross section. The growth temperature was varied between 820 °C and 1020 °C. Experiments carried out at different reactor pressures between 50 torr and 200 torr (Figure 3) showed that long thin NWs were obtained at 150 torr. At a relatively low pressure of 100 torr icecream-cone shaped structures were formed. A lot of wires grown at 175 torr had kinks or had a zig-zag morphology. At 200 torr there were very little NW growth, and those few were highly tapered and short. Very low flow of precursors (7.8-9.7 \( \mu\text{mol/min} \) of TMGa and \( \sim 45 \, \mu\text{mol/min} \) of NH\(_3\)) and small V/III ratio (\( \sim 5 \)) was used to facilitate anisotropic growth. At large V/III ratio the growth is slower, while at low V/III ratio the NWs obtained are more tapered. (For SEM images refer Supporting Information Section I.) We found that a pressure of 150 torr in N\(_2\) environment, \( \sim 840 \, ^\circ\text{C} \) and a V/III ratio of \( \sim 5 \) was optimum for obtaining thin non-tapering wires.

A comparison of the NWs obtained on different planes which are shown in Figure 2 is given in Table 1. All these samples compared are from the same growth run (at 840 °C and 150 torr with \( \sim 45 \, \mu\text{mol/min} \) NH\(_3\) flow and 7.8 \( \mu\text{mol/min} \) TMGa flow). The length of the NWs were determined by imaging cross sectional samples. The NWs obtained on c-plane sapphire are narrower than the NWs on r-plane. On m-plane sapphire we see two distinct set of NWs – one which is narrow (thickness \( \sim 30 \text{ nm} \)) and one which is thicker (thickness \( \sim 90 \text{ nm} \)). Since the NWs on m-plane sapphire are not erect, it is difficult to measure its exact length, but they are longer than NWs grown on c- and r-plane.
3. Structural characterization of nanowires

The crystal structure and the crystallinity of the NWs were analyzed using x-ray diffraction (XRD) and transmission electron microscopy (TEM).

3.1. XRD analysis

The grazing incidence XRD pattern the GaN nanowires grown on c-plane, r-plane and m-plane sapphire substrates is shown in Figure 4. The peaks are quite sharp indicating good crystal quality. The peak positions have
Table 1: Comparison of NWs grown on different orientations of sapphire under similar conditions. The average length and diameter of the NWs given in the table are from the same run. (Growth conditions: 840 °C and 150 torr with ∼45 μmol/min NH₃ flow and 7.8 μmol/min TMGa flow. The SEM images of these wires has been shown in Figure 2.)

been indexed to the wurtzite crystal structure of GaN. The lattice constants obtained by least square fitting from the peak positions were a = 3.188 Å and c = 5.179 Å which agrees with values reported in literature.²⁵ ²⁶

Figure 4: X-ray diffraction pattern with the planes indexed showing that the GaN wires have crystallized in the wurtzite phase. (XRD plots are displaced for clarity.)
3.2. TEM analysis

The crystallinity and growth direction of the wires grown on c-, m- and r-plane sapphire were determined using TEM. Since the NW thickness is less than 100 nm they are electron transparent and do not require further thinning down for TEM studies. The sample on which wires were grown was ultrasonicated in methanol to make a suspension. This suspension was slowly dropped multiple times onto a TEM grid and the solvent allowed to evaporate. The presence of the catalyst nanoparticle seen at the end of the wire in the TEM image (Figure 5(a)) confirms that the growth was catalytic. From energy dispersive X-ray spectroscopy the presence of Ga in the catalyst along with Ni was confirmed. The lattice planes seen in the high resolution TEM images (Figure 5(b)) and the well-defined electron diffraction pattern obtained (Figure 5(c)) confirm the single crystal nature of these wires. The diffraction pattern was indexed to find the lattice planes that gave rise to the diffraction spots. By comparing the diffraction pattern and the low magnification TEM image of the wire, the growth axis was determined. An indexed electron diffraction pattern obtained with a TEM is shown in Figure 6 showing that the growth direction is \(<10\bar{1}1>\) for this wire. The wires grew along the m-axis (\(<10\bar{1}0>\)) on both c-plane (Figure 5(c)) and r-plane sapphire substrates. On m-plane sapphire most of the NWs grew along the \(<10\bar{1}1>\) (Figure 6) and \(<10\bar{1}0>\) directions (Table 1).

4. Optical characterization

The optical properties of the GaN NWs were studied using photoluminescence (PL) spectroscopy from ensembles of NWs. The sample was excited
Figure 6: Electron diffraction pattern of a NW grown on m-plane sapphire when viewed along the [1101] zone axis. The line indicates axis of NW growth. This particular wire is oriented along the <1011> direction.

with a 266 nm frequency-quadrupled Nd:YAG laser. The luminescence from the sample was dispersed through a 0.55 m monochromator and detected with a thermoelectrically cooled Si-CCD. The PL spectra of GaN NWs at 10 K

Figure 7: PL from GaN NWs: PL spectra of ensembles of NWs at 10 K that were grown on c-plane, r-plane and m-plane sapphire substrates and mechanically transferred to an Si/SiO$_2$ substrate. (Spectra are displaced for clarity.)

grown on c-plane, r-plane and m-plane sapphire are shown in Figure 7. The spectra are normalized such that the near-band-edge emission peak is of the same intensity. PL spectra from these NWs peak around 3.50 eV (354 nm),
which corresponds to the near-band-edge emission of GaN. The FWHM of the PL peaks are $\sim 22$ nm (220 meV), $\sim 42$ nm (390 meV) and $\sim 37$ nm (350 meV) for NWs grown on c-plane, r-plane and m-plane sapphire respectively. These FWHM values are comparable to those reported in literature. The width of these spectra could be due to the variation in thickness and properties of the different wires of the ensemble, or due to defect-related luminescence. An additional peak is observed at $\sim 3.1$ eV (400 nm) which might be due to strongly localized excitons. Yellow luminescence from these samples is relatively small indicating good crystal quality.

5. Raman spectroscopy of NWs

Raman spectroscopy was used to study the phonon modes of GaN NWs. A Witec alpha 300R confocal Raman microscope was used with a 532 nm frequency doubled Nd:YAG laser for excitation. Figure 8 shows the Raman spectra for single GaN NWs, that were transferred to an aluminium foil which serves as the substrate. Al foil was chosen as substrate since it provides much lesser background signal in the region of interest than conventional crystalline substrates like Si, GaAs, sapphire, etc and other amorphous substrates. The intensity of the Raman peaks depends on the direction of both the incident and scattered polarization vectors relative to the crystal orientation. In order to observe as many phonons as possible from NWs from all samples the incident electric field was set at an angle of 45° with the NW axis, and the scattered light of all polarizations were detected simultaneously.
shown is after background correction.) Difference in relative intensity may
due to difference in growth direction of NW and/or because the incident
light might be falling in different direction relative to the crystallographic
directions.

At around 144, 567 and 734 cm$^{-1}$ the E$_2$ (low), E$_2$ (High) and E$_1$ (LO)
are seen.$^{32, 33, 34}$ These peaks are allowed by group theory in an infinitely
large sample with wurtzite crystal structure belonging to the space group
C$_{6v}^4$ (P6$_3$mc), and has been observed experimentally in first order Raman
spectra. But, the other peaks observed in the spectrum shown (Figure 8)
arise due to the finite dimensions of the NWs. The acoustic phonons, and
also the combination of both acoustic and low-lying optical branches at the
M symmetry point in the Brillouin zone gives rise to the spectral peak around
256 cm$^{-1}$.35 The peak at $\sim$ 308 cm$^{-1}$ arises from the overtone process of
acoustic phonons and the energy position matches the flat phonon branch
at the H-point in the Brillouin zone. The peak observed in the spectra at
$\sim$ 418 cm$^{-1}$ also is an acoustic overtone corresponding to the M-point.36
37, 35, 33 The Raman peaks are broader compared to those reported in
bulk samples due to the finite size effects in accordance with the phonon
confinement model, where the spatial confinement of phonons in nanocrystals
leads to an uncertainty in wave vector and hence a broadening of the Raman
spectral lines.38, 39, 35

6. Conclusions

In conclusion, we have grown GaN NWs on c-plane, m-plane, and r-plane
sapphire substrates using nickel catalyst and TMGa and NH$_3$ as precursors
by MOCVD. A pressure of 150 torr in N$_2$ environment and $\sim$ 840 °C, with
low precursor flow rates and V-III ratio, was optimum for growing thin, non-
tapering GaN NWs. The wires had a triangular cross-section and grew along
the $<10\overline{1}1>$ and $<10\overline{1}0>$ directions. Low temperature PL shows near-band-
edge emission from the NWs and small yellow luminescence indicates good
crystalline quality. Raman spectra reveals the good crystal quality of these
NWs and effects due to finite size.

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