GaN nanowires (NWs) have emerged as superior nanomaterials suitable for various energy harvesting applications, including solar cells,\textsuperscript{1,2} nano-lasers,\textsuperscript{3,4} NW-based light emitting diodes,\textsuperscript{5,6} piezoelectric nanogenerators,\textsuperscript{7,8} and photoelectrochemical water splitting (PEC-WS).\textsuperscript{9,10} Compared to thin films or nano-sized powders, one-dimensional (1D) GaN NWs with high aspect ratio may be considered good candidates to enhance the photocatalytic activity due to the remarkable improvement of the effective surface-to-volume ratio.\textsuperscript{11,12} The transportation of photo-generated carriers may also be enhanced in the GaN NWs due to the reduced traveled distance of the carriers to the electrolyte.\textsuperscript{13} Furthermore, GaN NWs showed high chemical stability against the harsh PEC experimental conditions in the presence of corrosive acidic electrolytes.\textsuperscript{14,15} Consequently, using GaN NWs as a photoanode for the production of clean and renewable hydrogen energy by PEC-WS is worth studying.

Recently, considerable efforts have been made in the enhancement of the photocatalytic properties of GaN NWs through the bandgap engineering, metal decoration or the deposition of InGaN to form heterostructure photoanodes. In this regard, heavily Si-doped GaN NWs were reported to enhance the photocurrent density in the PEC-WS due to the GaN surface band bending and its variation with Si doping.\textsuperscript{12} It was also found that the decoration of GaN NWs with Rh/Cr$_2$O$_3$ core/shell nanoparticles will promote the hydrogen evolution during the PEC-WS process.\textsuperscript{15} Additionally, the PEC-WS performance was enhanced by employing a double-band In$_x$Ga$_{1-x}$N/GaN core/shell NW photoanode due to the enhanced light absorption.\textsuperscript{15} In this study, we introduce a simple way to enhance the PEC-WS performance and the hydrogen evolution through the control of the NW density and aspect ratio of undoped GaN NWs without any metal decoration or complex structures. To the best of our knowledge, density- and aspect ratio-dependent PEC-WS of GaN NWs have rarely been reported.\textsuperscript{10}

Owing to the large degree of randomness in the vapor-liquid-solid (VLS) growth process, the controllable synthesis and reproducibility of VLS grown GaN NWs is essential to meet the prerequisites of PEC-WS applications. In this context, several approaches have been applied to understand the influence of different growth parameters on the morphology and properties of GaN NWs grown using the VLS technique. For example, it was reported that the growth mode and growth direction can be changed with the growth temperature,\textsuperscript{16,17} the crystallographic alignment and growth orientation can be controlled by selecting the appropriate substrate type,\textsuperscript{18} and the diameter of GaN NWs can be controlled by the size of the starting catalytic particles.\textsuperscript{19} Here, we present a guided procedure for controlling the NW density and aspect ratio of GaN NWs by optimizing the different growth parameters of the VLS mechanism. The effect of the growth temperature, V/III ratio, Ni thickness, and growth time on the morphology, NW density and aspect ratio of GaN NWs will be addressed in this study.

### Experimental

The fabrication of GaN NWs.— The GaN NWs investigated in this study were synthesized by Ni/In/Ga catalyzed VLS growth mechanism on c-sapphire substrates in a vertical metallorganic chemical vapor deposition (MOCVD) reactor. To fabricate a low melting point Ni/In/Ga alloy catalyst, a 1 nm Ni thin film was deposited on c-sapphire substrate using E-beam evaporator, which was then loaded to the MOCVD reactor for the deposition of In and Ga layers. The formation of In and Ga layers was performed by the flow of 0.64 μmol/min of TMIn at 500 °C for 20 sec and then 9 μmol/min of TMGa at 600 °C for 30 sec, in the absence of NH$_3$. Nanocatalytic particles with an average diameter of approximately 100 nm were produced after the annealing of Ni/In/Ga alloy in the MOCVD reactor at 800 °C for 10 min in a hydrogen environment. The growth of GaN NWs began immediately after the annealing in the same MOCVD reactor. Trimethylgallium (TGMa) and NH$_3$ were used as the Ga and N precursors, respectively. The growth pressure was fixed at 75 Torr, while the hydrogen was used as the carrier gas. The growth temperature, V/III ratio, and Ni thickness were all altered to obtain the highest NW density of GaN NWs. After the optimization of these growth parameters, the aspect ratio of GaN NWs was improved by increasing the growth time.

The surface morphology of the as-grown GaN NWs was monitored using field emission scanning electron microscopy (FE-SEM) (JSM-6700F, JEOL, Japan). The crystal quality at the atomic scale was visualized using field emission transmission electron microscopy (FE-TEM) (TECNAI G2 F20, FEI).

The PEC-WS of GaN NWs.— All PEC-WS experiments were carried out in a 1 M HCl water-based electrolyte in a home made two-electrode PEC cell connected with a current source (Keithley) capable of applying variable potentials and detecting very weak currents. GaN NWs were served as the photoanode, while Pt wire was used as the counter electrode. GaN NWs photoanode was mounted on a Teflon cell, where the sample surface was directly in contact with the electrolyte through a hole with a diameter of around 0.5 cm. A highly conductive Cu wire was bonded to the sample surface using indium contact to collect the photogenerated electrons from the photoanode and transport them to the counter electrode for the generation of the hydrogen. In front of the photoanode, a transparent window was fitted through which the sample was illuminated via a 300 W xenon lamp.
The stimulated solar light was perpendicular to the sample surface with a light irradiance of 100 mW/cm² where the exposed sample area was approximately 0.5026 cm².

Results and Discussion

Catalyst optimization for the VLS growth of GaN NWs.— In the catalyst mediated growth methods, the catalyst size and morphology defines the final shape and dimensions of the as-grown NWs. Consequently, the production of catalytic particles with uniform shape and well defined size is highly desirable. In the early stages of the VLS technique, single transition metals, mainly Ni and Au, were widely employed to initiate the growth of 1D NWs. However, these metals sometimes acted as a source of contamination for the host material, especially at elevated growth temperatures, which may work as undesired impurities in the nanometer scale that can weaken the electrical and optical properties. A high resolution FE-TEM lattice resolved image was used to investigate the favorable incorporation of the group-III element into the metal catalytic particle on the tip of III-N and III-As NWs, which revealed the Ni-element and another weak peak related to Ga. These results are consistent with the previous investigations made spatially on the catalytic particle on the tip of III-N and III-As NWs, which revealed the favorable incorporation of the group-III element into the metal catalyst. A high resolution FE-TEM lattice resolved image was also taken around the stem of the GaN NW, which showed a highly ordered crystal lattice with no structural defects such as dislocations or stacking faults at the atomic-scale. The selected area electron diffraction pattern (SAED) taken along the [0001] zone-axis, shown in the inset of Figure 2d, also revealed a regular diffraction spot pattern with a hexagonal symmetry, which can be indexed to the wurtzite GaN crystal structure. The detailed FE-TEM results may indicate that the as-grown GaN NWs exhibit a high quality single crystalline wurtzite structure with no defects, which would be advantageous for the proposed PEC-WS application. The carrier transportation in the single crystalline NWs will be significantly enhanced than that of the polycrystalline materials, which will be contributed to a high photocurrent with low recombination loss.

The controllable synthesis of GaN NWs.— The controllable synthesis of nanostructures is extremely desirable for their integration in various applications. Here, we present guided routes for controlling the NW density and aspect ratio of VLS grown GaN NWs by simple and high-yield approaches, in which only the growth parameters, namely, growth temperature, V/III ratio, Ni-thickness, and growth time will be optimized. Figure 3 shows the calculated number density of GaN NWs as a function of the different growth parameters.
Figure 2. The microstructure of GaN NWs. (a) Low magnification TEM image of the as-grown GaN NWs as an indication of the VLS growth. (b) HR-TEM image of the nanocatalytic particle and the GaN NW stem. (c) HR-TEM image of the catalytic particle (point 1) and the corresponding TEM-EDX spectrum. (d) Lattice-resolved TEM image taken approximately at point 2 of (b). The inset shows the corresponding SAED pattern taken along the [0001] zone-axis at the same point.

Effect of the growth temperature.—The growth temperature may be considered the most critical parameter in determining the morphological and physical properties of the VLS grown NWs. To study the influence of the growth temperature on the morphology of GaN NWs, NH₃ flow rate, TMGa flow rate, Ni thickness, and growth time were fixed at 13.4 mmol/min, 7.64 μmol/min, 1 nm, and 30 min, respectively, while the growth temperature was altered from 780 to 810 °C. We observed a temperature-sensitive growth where the size and shape as well as the NW density of GaN NWs were significantly altered with a small temperature window (780–810 °C). At the lowest growth temperature (780 °C), irregular 1D shapes were produced with a low density of uniform GaN NWs. As the temperature was increased to 795 °C, better uniformity and higher density of GaN NWs were achieved, which disappeared with the regular increase of temperature to 810 °C. The NW density of the resulting GaN NWs was estimated and plotted in Figure 3a as a function of the growth temperature. The highest density was obtained at 795 °C, which may be attributed to the enhancement of the NH₃ cracking efficiency that enables denser nucleation sites and promotes Ga-adatoms diffusion lengths. At elevated growth temperatures, the effective V/III ratio will be increased, inducing a 2D growth due to the enhancement of the lateral growth rate over the vertical growth rate, producing GaN islands rather than NWs.

Effect of the V/III ratio.—GaN NWs were reported to be grown under both Ga-rich and N-rich conditions; however, the size and shape differ. In our study, we performed two sets of experiments, during which the growth of GaN NWs was governed by changing the V/III molar ratio through varying either the TMGa or NH₃ flow rates. To study the influence of the NH₃ flow rate on the morphology of GaN NWs, growth temperature, TMGa flow rate, Ni thickness, and growth time were adjusted at 795 °C, 7.64 μmol/min, 1 nm, and 30 min, respectively, while the NH₃ flow rate was altered from 13.4 to 22.3 mmol/min. We observed that N-rich conditions were essential in producing higher NW density of GaN NWs than that of Ga-rich conditions. This result may be explained by the fact that the formation of GaN NWs by VLS growth is ruled by the diffusion of Ga-adatoms and their preferential incorporation inside the catalyst. Consequently, under Ga-rich conditions or in the lack of N-atoms, the surface diffusion of Ga-adatoms and their axial diffusion on the side walls of GaN NWs to the tip will be considerably reduced, leading to the formation of conical shaped and thick GaN NWs. To confirm this observation, we fixed the NH₃ flow rate at 13.4 mmol/min and gradually increased the TMGa flow rate. During this experiment the growth temperature, Ni thickness, and growth time were fixed at 795 °C, 1 nm, and...
30 min, respectively. Figure 3c shows the severe drop of the NW density of GaN NWs with increased TMGa flow rate. With decreasing the V/III ratio through increasing the Ga-flux, the growth of GaN NWs almost ceased, which was not observed when the NH₃ flow rate was increased. Additionally, the GaN NWs mostly kept their shape and size with increasing the N-flux, while their shape was changed from a uniform diameter to a cone-like shape with increasing the Ga-flux. These results may suggest that the density of VLS grown GaN NWs can be enhanced by increasing the available N-atoms that can induce Ga-adatoms diffusion lengths, leading to better nucleation sites and higher growth rates.24,26

Effect of the Ni thickness.—It was reported that the thickness of the Ni catalyst has a significant role in determining the morphology as well as the NW density of the VLS grown GaN NWs.27 Therefore, after the optimization of growth temperature, NH₃ flow rate, TMGa flow rate, and growth time at 795 °C, 17.9 mmol/min, 7.64 μmol/min, and 30 min, respectively, we changed the Ni thickness in order to change the density of GaN NWs. In the VLS growth mechanism, the starting catalyst morphology and composition are essential for the production of high quality NWs. In this study, we used a ternary catalytic alloy composed of Ni-metal film and two successive layers of In and Ga deposited by MOCVD. The rule of using foreign metals such as In and Ga was to enhance the catalyst reliability by improving its morphology after agglomeration.16 Keeping the same composition of the In and Ga layers, the Ni-metal thickness varied between 1, 1.5, and 2 nm. At 1 nm, we observed a dense formation of GaN NWs on the surface of the sapphire substrate, but they appeared as two different shapes; i.e. uniform diameter GaN NWs and tapered GaN nano-pyramids with a conical cross-section. Increasing the Ni-thickness to 1.5 nm provided an increased number density and also improved the morphology of GaN NWs, where mostly all GaN NWs possessed a uniform cross-section with an average diameter of approximately 114 nm. Tapered GaN nano-pyramids were reproduced at 2 nm and the density was reduced to 20% from the previous value calculated at 1.5 nm. It is worth mentioning that, as the Ni thickness was increased over 2 nm, the growth of GaN NWs under similar growth conditions was vanished and only a 2D layer with a rough surface was produced. Furthermore, we did not observe any catalytic particles at the apices of the tapered GaN nano-pyramids, which may be attributed to the growth mode switching from VLS to vapor-solid mechanism. We may attribute the formation of the tapered GaN nano-pyramids to the lack of catalyst size uniformity with changing the Ni layer thickness, where large size catalysts may cause this kind of diameter increase. In addition, the tapered nature of GaN nano-pyramids may be attributed to the gradual solidification and shrinkage of liquid droplets during the MOCVD growth.28 The effect of the Ni thickness on the density and morphology of GaN NWs is summarized in Table I. We may conclude from this Table that the average length of all GaN NWs was almost the same, while the average diameter increased with increasing the Ni thickness. This may be consistent with the previous reports, which implied that changing the thickness of the Ni catalyst will affect the density and diameter of GaN NWs without causing any significant increase in their length.27

| Table I. Summary of the modifications shown in the cross section, length, diameter, and NWs density as a function of the Ni thickness and growth times. The sample grown at 1.5 nm Ni thickness and 30 min was used as the reference for increasing the growth time. |
|-----------------|-----------------|-----------------|-----------------|
| Growth Parameters | Average Length (μm) | Average Diameter (nm) | NWs Density (μm⁻²) | Cross Section |
| Ni Thickness (1 nm) | 4.3 | 76 | 3.706 | ▲♦ |
| Ni Thickness (1.5 nm) | 4.1 | 114 | 4.627 | ▲ |
| Ni Thickness (30 min) | 4.3 | 150 | 4.566 | ▲♦ |
| Growth Time (40 min) | 8.2 | 290 | 5.306 | ▲ |
| Growth Time (60 min) | 8.5 | 500 | 2.665 | ▲♦ |

▲ triangular cross section ♦ conical cross section

The effect of the growth time.—To investigate the effect of the growth time on the aspect ratio of GaN NWs, we fixed the growth temperature, NH₃ flow rate, TMGa flow rate, and Ni thickness at 795 °C, 17.9 mmol/min, 7.64 μmol/min, and 1.5 nm, respectively, and increased the growth time from 30 min to 40 and 60 min. Figure 4 displays the morphology evolution of the GaN NWs as a function of the growth time. As the growth time was increased to 40 min, the GaN NWs were grown with higher NW density and better size uniformity than those grown for 30 min. As summarized in Table I, the density was increased by about 15% and the length of GaN NWs was almost doubled with increasing the growth time to 40 min, indicating a considerable enhancement of the vertical growth rate and hence the aspect ratio. A regular increase of the growth time to 60 min led to a significant increase of the GaN NWs diameter, while they kept relatively the same length. The longer NW lengths and the improved density obtained at 40 min may be attributed to the higher degree of supersaturation achieved with increasing the growth time, which may increase the chemical potential of the melted alloy composed of the catalyst and Ga-adatoms.29 Consequently, due to the free energy minimization, the growth rate of GaN NWs at the catalyst-NW interface will be increased causing the length to be also increased. For the same reason, the surface diffusion of Ga-adatoms is enhanced, leading to the formation of dense nucleation sites, which in turn improved the NW density of the GaN NWs. On the other hand, it was reported that increasing the growth time may induce the vertical growth rate of NWs to a limit at which the lateral growth rate will significantly exceed that in the vertical direction, leading to the expansion of the NW diameter,29,30 which may explain the noticeable increase of diameter at 60 min. It is worth noticing that the NW density was significantly reduced in the case of using thick Ni catalyst or increasing the growth period over 40 min, as shown in Table I, which may be associated with the evolution of tapered GaN NWs with large diameters in both cases. The formation of tapered GaN NWs may be attributed to the sudden decrease of the growth temperature during the cooling step after finishing the MOCVD growth, which can significantly limit the surface

![Figure 4](Image 304x72 to 548x267)
diffusion length of the precipitated Ga adatoms. Consequently, the growth is restricted to the base of the NW on the substrate surface, which led to the expansion of the NW base and the shrinkage of the NW apex.

PEC-WS of GaN NWs.—The development of a simple structure and well-designed semiconductor photoanode to improve the PEC-WS performance may be a challenge. In this study, we introduce a new approach based on the enhancement of the PEC-WS performance by controlling the NW density and aspect ratio of the GaN NWs photoanode without any additional doping or complex structures.

To investigate the PEC properties of the nanoscale GaN NWs photoanode, a two electrode home-made PEC cell was designed, as shown in Figure 5a. The GaN NWs were acting as the working photoelectrode to absorb the illuminating light energy and convert it into electron-hole pairs, which then can start the water splitting half reactions. The conventional semiconductor band bending may lead to the separation of the photogenerated electron-hole pairs, where the holes will be drifted to the surface of the GaN NWs to strip the oxygen from the water (the water oxidation) and the electrons will be migrated to the counter electrode (Pt wire) to reduce the hydrogen ions into molecular hydrogen gas (the hydrogen reduction), as shown in Figure 5b. This process efficiency can be improved with enhanced light absorption and higher densities of the photogenerated electron-hole pairs. Figure 6a shows a set of linear sweeps of the photocurrent density as a function of the applied potential for the VLS grown GaN NWs photoanode with variable Ni thicknesses of 1, 1.5, and 2 nm, which corresponding to different NW densities of approximately 3.706, 4.627, and 4.566 $\mu$m$^{-2}$, respectively. We observed a NW density-dependent PEC-WS performance, where the photocurrent was directly proportional to the NW density of the GaN NWs. The anodic behaviors of the photocurrent suggest that the working photoelectrode was unintentionally n-type semiconductor. The dark signals were negligible for all samples, even at high applied potential, indicating a good quality with insignificant leakage currents. The stability of the GaN NWs photoanode was also investigated for the same samples for a prolonged time. During these experiments, a small bias voltage...
of 0.5 Volt was applied between the two electrodes to overcome any external losses of the measuring system. As shown in Figure 6b, the accumulated ionic charges were increased in the case of denser GaN NWs, suggesting a longer lifetimes of the photoanode to work in the PEC-WS experiment. Higher photocurrent density and longer term stability may suggest an improved PEC-WS with increasing the NW density, which may be addressed as follows. As the NW density of the GaN NWs was enhanced by the optimization of the Ni catalyst, the illuminating light will suffer multiple scattering when incident on the dense forest-like GaN NWs photoanode, as shown in the schematic illustration in Figure 5b. As a result, the absorbed light portion will be increased due to the improved light trapping effects, leading to better driving force for the liberation of charge carriers from the semiconductor photoanode. Consequently, the overall PEC-WS performance will be significantly improved.

In order to confirm the observed NW density-dependent PEC-WS performance as well as to investigate the effect of the increased aspect ratio of the GaN NWs by longer growth times, the samples grown for 40 and 60 min were also used and compared with the reference sample grown for 30 min (1.5 nm Ni thickness). As depicted in Figure 6c, the photocurrent density was further enhanced with approximately 34% at 1 Volt by extending the growth time to 40 min, which was degraded again at 60 min. Since these samples were grown under identical growth conditions, except the growth time, the observed change of the photocurrent densities can be directly correlated with the significant morphological evolution of the GaN NWs. On the other hand, the poor size uniformity may play an important role in the observed drop of the photocurrent density at 60 min. The variation of the measured photocurrent density was also studied as a function of the irradiation time at the same bias voltage of 0.5 Volt, as depicted in Figure 6d. Interestingly, although the sample grown for 60 min showed lower photocurrent density than that grown for 30 min, the former was saturated earlier, which may reveal the role of GaN NWs size in the improvement of the PEC-WS performance. Additionally, the sample grown for 40 min, which possessed the highest NW density together with the longest GaN NWs, exhibited the optimum PEC-WS performance, which may be attributed to the enhancement of the surface-to-volume ratio by the increased NW density and the aspect ratio of GaN NWs. It was generally accepted that the aspect ratio of the NW-based photoanode will play an important role in the enhancement of the overall PEC-WS performance. Furthermore, the diameters of the GaN NWs grown for 40 min were also smaller than those grown for 60 min, which can significantly affect the PEC-WS performance. The small diameter of the NWs can effectively facilitate the charge separation and improve the transportation of the separated charge carriers to the desired water splitting half reaction interfaces. Consequently, the supply rate of the photogenerated charge carriers to the water splitting redox reaction sites will be increased due to the reduced travel distance caused by the small diameters of the GaN NWs. Additionally, we believe that the photocurrent density produced by the GaN NWs grown on c-plane sapphire substrates can be enhanced by improving the lateral conductivity of the substrate, which can improve the charge collection efficiency. This can be achieved by the growth of GaN NWs in a conductive layer such as n-type GaN or replacing the dielectric sapphire substrate with a conductive one such as Si. This study is currently under investigation in our group.

To fully evaluate the PEC-WS performance of the GaN NWs photoanode, we further measured the applied bias photon-to-current efficiency (ABPE), which can allow diagnostic measurements represent the development of the GaN NWs performance as a function of the applied potential. The measured ABPE values for GaN NWs grown with different Ni thicknesses and different growth times was calculated by using equation 1:

\[
ABPE(\%) = \frac{J_p (mA/cm^2)(1.23 - V_{\text{app}}(volt))}{P_{\text{light}}(mW/cm^2)} \times 100
\]

where \(J_p\) is the measured photocurrent density, 1.23 is the standard state reversible potential of water, \(V_{\text{app}}\) is the applied potential during the measurement of the photocurrent density, and \(P_{\text{light}}\) is the illuminating light power density. As displayed in Figure 7a, as the applied potential was increased, the ABPE efficiency attained its maximum value and then decreased again when the applied potential was approaching to the thermodynamic WS potential (1.23 Volt). The maximum efficiency was shifted to higher applied potential values for the sample of the highest GaN NWs density (1.5 nm). Interestingly, the optimal applied potential for the PEC-WS process was decreased as the NW density and aspect ratio of GaN NWs were further improved by increasing the growth time to 40 min. As shown in Figure 7b, the sample grown for 40 min recorded the highest efficiency of approximately 0.1% at 0.35 Volt compared to 0.06% at 0.55 Volt for the 30 min growth time, which may represent a promising modification of the GaN NWs performance in the PEC-WS process. Since the photocurrent density, process stability, and ABPE efficiency were all improved with the NW density and aspect ratio of the GaN NWs, we may address the value of fabricating a dense NW-based photoanode with high aspect ratio to promote the overall PEC-WS performance.

Conclusions

In conclusion, we introduced a guided scheme for the facile control of the NW density and the aspect ratio of the single crystalline GaN NWs through a simple route based on the matching between the different growth conditions. Single crystalline GaN NW arrays grown using a VLS mechanism were then employed as efficient photoanodes for PEC-WS applications. The results revealed that the photocurrent density can be effectively improved by increasing the NW density of the GaN NW photoanode; however, the process stability may be degraded with prolonged time. As the aspect ratio of the GaN NWs was increased by extending the growth time from 30 to 40 min, the photocurrent density was improved with about 34% at 1 Volt and 30 min growth time, which may represent a promising modification of the GaN NWs performance in the PEC-WS process. Since the photocurrent density, process stability, and ABPE efficiency were all improved with the NW density and aspect ratio of the GaN NWs, we may address the value of fabricating a dense NW-based photoanode with high aspect ratio to promote the overall PEC-WS performance.
samples, indicating an interesting modification with the increased GaN NWs aspect ratio. The improved overall PEC-WS performance of the dense and high aspect ratio GaN NWs were attributed to the enhancement of the light trapping effects that can increase the driving force of the semiconductor photoelectrode for the creation of electron-hole pairs.

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