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Modulating the ZnO NR shape to enhance the luminescence efficiency for optoelectronic applications

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Abstract

It is essential to control the size of zinc oxide (ZnO) nanorods (NRs) for the improvement of tunable optoelectronic applications. In this paper, we present the results of a systematic and extensive study that explored the effect of growth parameters on the morphology and optoelectronic properties of ZnO NRs. We found that the length and width of ZnO NRs can be efficiently tuned by carefully controlling the growth parameters and by modulating the ZnO seed with reduced graphene (rGO). These results could give us a better understanding of the growth behavior of ZnO NRs and could contribute to the control of the morphology of these NRs for nano-device applications. Moreover, the effect of growth parameters on the optoelectronic properties of ZnO NRs was thoroughly investigated by fabricating light-emitting diodes (LEDs) with different morphologies of the ZnO NRs and the correlation between the morphology and the luminescence efficiency of ZnO was established. These investigations illustrate a viable and highly promising approach to enhance the luminescence intensity of ZnO NR-based LEDs by 500 times. The present work will guide researchers in the production of low-temperature, size-controlled, and aligned ZnO NRs for tunable highly luminescent optoelectronic applications.

Introduction

Recently, zinc oxide (ZnO) has been the most extensively studied material owing to its superior electrical, optical, and piezoelectrical properties. ZnO nanostructures have received considerable attention due to the simplicity of their synthesis process and to their immense potential in different applications such as solar cells, sensors, and optoelectronic devices. ZnO possesses diverse hierarchical nanostructures such as nanowires, nanorods (NRs), nanodots, nanotubes, nanosheets, nanobelts, nanoribbons, and nanorings [1–3]. Several growth techniques have been employed to create an array of ZnO nanostructures, and these include the vapor-liquid-solid [4], vapor-solid [5], metal organic chemical vapor deposition [6], chemical bath deposition [7], and hydrothermal methods [8, 9]. However, most of the techniques are limited by their high-temperature process that cannot be scaled up over a large device area at a very low cost. Comparatively, the low-temperature hydrothermal method is attractive for realizing industrially scalable applications owing to its low cost and ability to produce ZnO nanostructures on various substrates including amorphous, single-crystalline, polycrystalline, and flexible substrates [10, 11]. In the hydrothermal method, the morphology, growth density, and diameter can be controlled by adjusting the reaction parameters such as the precursor concentration, growth temperature, as well as growth time. This method uses a low-cost, low-temperature process that can be scaled up for industrial production on any substrates, and the properties of the final product can be tuned by adjusting the synthesis parameters. Wang et al [12] reported that seed layer and precursor concentration can change the morphology of ZnO NRs. Recently, some researchers have made efforts to control ZnO nanostructures by putting different elements such as polyethyleneimine molecules, surfactants, and graphene-related materials into growth solutions or into the seed layer [13–15]. Yu et al [15] and
Pruna et al [16] have grown ZnO NRs by introducing reduced graphene oxide (rGO) into the ZnO seed layer using a sol-gel hydrothermal method. Recent studies are focused mostly on the correlation of nanoarchitecture morphology with growth parameters to modify the electrical and optical properties of ZnO NRs [11, 17–19]. However, achieving control over the synthesis process to tune the width and length of ZnO NRs is a very important subject of research worldwide; it is still a challenging task and requires further explanation. In this paper, we report the results of systematic studies on the effect of reaction parameters on the morphology and optoelectronic properties of ZnO NRs and we investigate the correlation between morphology and luminescence intensity. Furthermore, we present the strategies we have developed for controlling the morphology during the growth process, which can lead to enhanced optoelectronics properties.

**Experimental details**

Zinc nitrate hexahydrate [Zn(NO$_3$)$_2$·6H$_2$O], hexamethylenetetramine (HMT) (C$_6$H$_{12}$N$_4$), zinc acetate (Zn(CH$_3$COO)$_2$·2H$_2$O), and potassium hydroxide (KOH) were purchased from Sigma-Aldrich. Single-layer GO was purchased from Angstrom Materials (Dayton, OH, USA) and was synthesized by using a modified Hummers method according to the manufacturer’s datasheet. The ZnO seed was prepared by dissolving zinc acetate and KOH in methanol with molar concentrations of 0.05 and 0.025 M, respectively. The KOH solution was added dropwise into the zinc acetate solution [20]. After conducting an appropriate cleaning procedure, we spin coated a ZnO seed layer on n-SiC substrates. The ZnO seed coated substrates were heated at 100 °C for 20 min. The substrates were placed carefully into the aqueous growth solution of ZnO NRs prepared from the reactant precursors [Zn(NO$_3$)$_2$·6H$_2$O] and HMT (C$_6$H$_{12}$N$_4$), and heated at 90 °C for 3 h. The diameter of the ZnO NRs was controlled by varying the molar concentration of [Zn(NO$_3$)$_2$·6H$_2$O] from 0.04 M to 0.12 M, whereas the molar concentration of HMT was fixed to 0.08 M. To study the effect of the molar concentration of HMT, we changed the concentration from 0.04 M to 0.1 M, whereas the molar concentration of [Zn(NO$_3$)$_2$·6H$_2$O] was fixed to 0.08 M. The length of ZnO NR arrays was controlled by varying the growth duration from 2 h to 9 h.

The GO was reduced by adding hydrazine hydrate and heating the solution at 120 °C under magnetic stirring. Then, the rGO was exfoliated in methanol with ultrasonic treatment. Then, the ZnO seed was modulated by incorporating various amounts of rGO; a 1-ml seed solution was added with one, two, and three portions (one portion = 0.1 ml in volume) of rGO. The modulated seed layer solution was spin coated on the n-SiC substrates at 2000 rpm for 30 s followed by heating at 130 °C for 5 min. The substrates were placed carefully into an aqueous growth solution of 0.08 M [Zn(NO$_3$)$_2$·6H$_2$O] and HMT (C$_6$H$_{12}$N$_4$) and heated at 90 °C for 3 h.

n-ZnO NRs/n-SiC Schottky light-emitting diodes (LEDs) were fabricated according to the different diameters (65, 129, and 175 nm) and lengths (0.383, 1.2, and 3.9 μm) of the ZnO NRs and of the ZnO NRs grown on an rGO-modulated seed solution. After the ZnO growth, the top and bottom metal contacts were deposited. Aluminum (200 nm) was evaporated on the n-SiC substrate to make ohmic contact on a small portion of the substrate, which was covered before the ZnO NR growth. Then, 100-nm-thick Au Schottky contacts of diameter 2.0 mm were evaporated onto a group of NRs. The morphology of the ZnO NRs was characterized by scanning electron microscopy (SEM), and the optical properties were investigated by fluorescence spectroscopy and electroluminescence (EL) spectroscopy.

**Results and discussion**

The growth process for a single-crystal ZnO is called the Ostwald ripening process, in which kinetically favored small crystallites nucleate and grow to larger particles owing to the energy difference between the large and the smaller particles based on the Gibbs-Thomson law [21, 22].

Decommission reaction:

\[
\text{C}_6\text{H}_{12}\text{N}_4 \rightarrow 4\text{NH}_3 + 6\text{HCHO} \quad (1)
\]

\[
\text{Zn(NO}_3)_2 \rightarrow \text{Zn}^{2+} + 2\text{NO}_3 \quad (2)
\]

\[
\text{Zn}^{2+} + 4\text{NH}_3 \leftrightarrow \text{Zn(NH}_3)_4^{2-} \quad (3)
\]

Hydroxyl supply reaction:

\[
\text{Zn}^{2+} + \text{OH}^- \rightarrow \text{Zn(OH)}_2^{2-} \quad (4)
\]

\[
\text{NH}_4 + \text{H}_2\text{O} \rightarrow \text{NH}_4^+ + \text{OH}^- \quad (5)
\]
Figure 1. Cross-sectional FESEM images of ZnO NRs grown on an n-SiC substrate at a fixed molar concentration of zinc nitrate (0.08 M) but at different molar concentrations of HMT: (a) 0.04, (b) 0.06, (c) 0.08, and (d) 0.1 M.
ZnO NR synthesis reaction:

\[ \text{Zn(OH)}_2 \rightarrow \text{ZnO} + \text{H}_2\text{O} \]  

The aqueous solutions of \([\text{Zn(NO}_3]_2 \cdot 6\text{H}_2\text{O}\) and HMT can produce the following chemical reactions. The HMT begins to decompose into ammonia (equation (1)), and Zn(NO\(_3\))\(_2\) decomposes into Zn\(^{2+}\) and NO\(_3\)\(^-\) (equation (2)) [23, 24]. Subsequently, a Zn\(^{2+}\) cation reacts with an OH\(^-\) anion to form ZnO nuclei under certain growth conditions (equations (4) and (6)) [24]. Controlling the chemical reaction parameters and conditions could play a significant role in the morphology and optoelectronic properties of ZnO NRs.

In the ZnO NR growth process, the HMT concentration is the main factor that supplies the OH\(^-\) anions, which play a vital role in the growth of ZnO NRs [23, 24]. The function of HMT during the ZnO NRs growth is to bridge two Zn\(^{2+}\) ions and it also plays two critical roles: it produces a basic environment for the formation of Zn(OH)\(_2\) and coordinates with Zn\(^{2+}\) to decompose the Zn(OH)\(_2\) into ZnO. The resultant ions (Zn\(^{2+}\) and OH\(^-\)) are used to promote the growth of ZnO NRs along the c-axis direction and to increase the ZnO NRs. Therefore, ZnO NRs with various lengths can be prepared by simply adjusting the OH\(^-\) concentration [11]. In general, the precursor concentration determines the size of ZnO NRs. The HMT solution with molar concentrations of 0.04, 0.06, 0.08, and 0.1 M produces ZnO NRs with various lengths. The average lengths of ZnO NRs prepared with molar concentrations of 0.04, 0.06, 0.08, and 0.1 M were 0.383, 0.810, 1.172, and 2.165 \(\mu\)m, respectively, as shown in figures 1(a)–(d). Figure 1 shows that a higher HMT concentration produces longer ZnO NRs without any big change in diameter. These results indicate that long ZnO NRs with roughly the same diameter size can be achieved with higher HMT concentrations. The data presented in table 1 show the average lengths and diameters of ZnO NRs according to the HMT concentrations. One reason for this result is that the HMT generated OH\(^-\) ions that bonded with Zn\(^{2+}\) ions to form the ZnO NRs. The ZnO NRs with a limited diameter size were due to the small amount of HMT solution, which resulted in a discontinuous ZnO growth in the lateral plane and in the growth in the longitudinal direction instead [25]. Figure 2 shows that the length of the ZnO NRs increases exponentially with the increase in HMT molar concentration. The first regime of growth belongs to an early random growth along the fast growth axis of the NRs, based on the fact that a large number of very short rods with a random growth orientation and shape lie at the base near the seeded surface [17, 26, 27]. The random

| HMT concentration (M) | Diameter (nm) | Length (\(\mu\)m) |
|-----------------------|--------------|------------------|
| 0.04                  | 124          | 0.383            |
| 0.06                  | 125          | 0.810            |
| 0.08                  | 129          | 1.172            |
| 0.1                   | 137          | 2.165            |

**Figure 2.** Length of ZnO NRs at different molar concentrations of HMT.
growth is then followed by an aligned growth arising from the space constraints imposed on the growing rods owing to the dense packing of the seeds. Shuzi et al. reported that there are many small ZnO NRs at the bottom, which means that the ZnO crystallite rods amalgamate with each other at the later growth stage because...
the growth velocity of ZnO is faster along the c-axis than in the other directions. For the cross section SEM image the samples are cut due to which some rods are broken and damaged therefore some rods have different shapes.

For the formation of Zn(NO$_3$)$_2$, Zn$^{2+}$ and OH$^{-}$ are required, which are derived from Zn(NO$_3$)$_2$ and (CN)$_6$N$_4$, respectively. Then, Zn(OH)$_2$ will be dehydrated and will precipitate on the substrate, leading to the formation of ZnO NRs. The growth rate of ZnO NRs is determined by the relative growth rate of the polar and nonpolar surfaces. The increase in the Zn(NO$_3$)$_2$ concentration increases the Zn(OH)$_2$ amount, which will increase the growth rate. This process is endothermic and will hinder the growth rate of ZnO NRs in the $\langle 000 1 \rangle$ direction; as a result, thicker ZnO NRs were obtained with increasing Zn(NO$_3$)$_2$ concentration.$^{[25, 29]}$ Therefore, it is possible to control the thickness of ZnO NRs by adjusting the Zn(NO$_3$)$_2$ concentration. It can be clearly seen in figures 3(a)–(e) that a higher Zn(NO$_3$)$_2$ concentration produces thicker ZnO NRs with approximately the same length. These results indicate that thicker ZnO NRs with the same length can be achieved by changing the Zn(NO$_3$)$_2$ molar concentrations. The data presented in table 2 show the average lengths and diameters of the ZnO NRs according to the Zn(NO$_3$)$_2$ molar concentrations. We found that, by optimizing the Zn(NO$_3$)$_2$ molar concentration, we can tune the diameter of the ZnO NRs. Thus diameter can be increased by increasing the Zn(NO$_3$)$_2$ molar concentration. In the chemical reaction, Zn(OH)$_{4-x}$(ONH$_4$)$_x$ complexes are formed, which are converted into Zn(OH)$_{4-x}^-$, NH$_4^+$ ions bind to the Zn(OH)$_{4-x}^-$ (a growth unit of NRs), which helps to increase the growth speed. These processes are endothermic and will hinder the growth of ZnO NRs in the $\langle 000 1 \rangle$ directions, making the NRs thicker. Therefore, the diameter of ZnO NRs can be tuned from thinner to thicker by controlling the Zn(NO$_3$)$_2$ molar concentration.$^{[25]}$ Furthermore, figure 4 shows that the diameter of the ZnO NRs increases exponentially with the increase in Zn(NO$_3$)$_2$ molar concentration.

Figures 5(a)–(e) shows that the average length of the ZnO NRs increases from 0.474 nm, 1.1 $\mu$m, 3.9 $\mu$m, and 4.32 $\mu$m as the immersion time increases from 2, 3, 5, 7, and 9 h, respectively, at a precursor concentration of 0.08 M. Hence, as the time increases, the ZnO nuclei will continuously grow.$^{[30]}$ A longer

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### Table 2. Morphological parameter of ZnO grown at an HMT concentration of 0.08 M.

| Zn(NO$_3$)$_2$ concentration (M) | Diameter (nm) | Length ($\mu$m) |
|---------------------------------|----------------|-----------------|
| 0.04                            | 65             | 1.15            |
| 0.06                            | 102            | 1.3             |
| 0.08                            | 129            | 1.2             |
| 0.1                             | 175            | 1.2             |
| 0.12                            | 590            | 1.4             |

![Figure 4. Average diameter of ZnO NRs at different molar concentrations of zinc nitrate.](image-url)
growth time would result in a longer rod due to the Ostwald ripening process \[21\]. The axial growth rate was found to be more pronounced than the lateral growth rate, which can be attributed to the low surface free energy within the c-axis \[31, 32\]. Figure 6 shows that the length-growth rate (length/time) increases first then drops as
the deposition time increases, which is associated with the consumption of the $(\text{Zn}^{2+}, \text{OH}^{-})$ species with time; similar results have been reported previously [25, 33]. The morphological parameters are listed in table 3.

Figures 7(a)–(c) shows the average diameters of the ZnO NRs, i.e., 48, 58, and 78 nm, as the rGO concentration in the seed solution increases from one, two, and three portions, respectively, where a precursor concentration of 0.1 M was used at $90^\circ\text{C}$ for 3 h. The results indicate that the diameter of ZnO NRs decreases as the concentration of rGO increases in the seed solution, which suggests that the diameter of ZnO NRs could be changed by modifying the ZnO seed solution with rGO, as illustrated in figure 7. The morphological parameters are listed in table 4. The inclusion of rGO into the ZnO seed solution might cause higher hydrophobicity; therefore, smaller clusters of ZnO NRs might be formed and the diameter of these NRs might become smaller [34]. The rGO in the ZnO seed could enhance the growth of ZnO NRs in the c-plane and inhibit the lateral growth. The ZnO structure has two polar planes, namely, (002) and (00–2), and a charged surface that could cause a faster growth rate along the (002) direction; additionally, rGO enhances the reaction with OH and a Zn-terminated c-plane [15]. The results of the material analyses indicate that the diameter of ZnO NRs can be effectively modulated by adjusting the rGO compositions, which may have the potential to optimize the ZnO NRs according to the device performance.

Besides studying the effect of morphology on the optical properties of ZnO NRs, we also fabricated Schottky diodes and performed EL emission measurements. Figure 8 shows the EL spectrum of ZnO NRs with different diameters (65, 129, and 175 nm); the EL spectrum reveals that there are three types of emission peaks: a violet peak at 430 nm, a blue peak at 460 nm, and a broad deep band emission centered at 530 nm. There is no big change in the emission intensities. Figure 9 shows the EL spectrum of ZnO NRs with different lengths: 0.383, 1.2, and 3.9 $\mu\text{m}$. The EL spectra of Schottky diodes with ZnO NR lengths of 0.383 and 1.2 $\mu\text{m}$ reveal that there are three types of emission peaks: a violet peak at 430 nm, a blue peak at 460 nm, and a broad deep band emission centered at 530 nm. The EL spectra of Schottky diodes with a ZnO NR length of 3.9 $\mu\text{m}$ have two emission peaks:

![Figure 6. Growth rate of the length and diameter of ZnO NR at various growth times.](image)

### Table 3. Morphological parameter of ZnO grown at different growth durations and at an equal molar (0.08 M) concentration of HMT and Zn(NO$_3$)$_2$

| Time (h) | Length ($\mu\text{m}$) | Growth rate ($\mu\text{m h}^{-1}$) | Diameter ($\mu\text{m}$) | Growth rate ($\mu\text{m h}^{-1}$) |
|---------|----------------------|-------------------------------|----------------------|-------------------------------|
| 2       | 0.474                | 0.237                         | 0.122                | 0.061                         |
| 3       | 1.1                  | 0.366                         | 0.110                | 0.036                         |
| 5       | 3                    | 0.6                           | 1.4                  | 0.28                          |
| 7       | 3.9                  | 0.55                          | 1.5                  | 0.21                          |
| 9       | 4.32                 | 0.48                          | 1.5                  | 0.16                          |
a violet-blue peak at 455 nm and a broad deep band emission centered at 560 nm. By comparing the emission bands of the Schottky LEDs, we can clearly see that, with increasing length of the ZnO NRs, the relative intensity of the visible deep band emission increases. Figure 9 shows that the Schottky LED with 1.2-μm ZnO NRs has 10 times higher emission intensity than that of one with 0.383-μm ZnO NRs and that the Schottky LED with 3.9-μm ZnO NRs have 300 times higher emission intensity than that of one with 0.383-μm ZnO NRs. It was previously reported that the deep band emission intensity depends on the size of the ZnO and that the extraction

Figure 7. Cross-sectional FESEM images of ZnO nanorods grown on an n-SiC substrate at an equal molar concentration of HMT and zinc nitrate (0.1 M) with an rGO-modulated seed solution: (a) with one portion of rGO, (b) with two portions of rGO, and (c) with three portions of rGO (one portion = 0.1 ml of rGO).

Table 4. Morphological parameter of ZnO grown at an equal molar concentration (0.1 M) of HMT and Zn(NO₃)₂ on an rGO-modulated seed solution.

| GO concentration (ml) | Diameter (nm) | Length (nm) |
|-----------------------|---------------|-------------|
| 0.1                   | 48            | 407         |
| 0.2                   | 58            | 548         |
| 0.3                   | 78            | 607         |
factor for ZnO is low due to its high absorption coefficients ($\sim 2 \times 10^5$ cm$^{-1}$), which increases the probability of ultraviolet (UV) reabsorption within the crystal itself \[32\text{–}34\]. This reabsorbed UV can excite the defect states in the structures, resulting in visible deep band emission because the defects responsible for this emission are not confined near the surface but are ubiquitous inside the ZnO NRs \[29\]. Previously, we have reported that the UV emission from ZnO NRs can be internally reabsorbed within 2 $\mu$m \[35, 36\].

Figure 10 shows that the EL spectra of ZnO NRs grown on a rGO-modulated seed exhibited two emission peaks: a violet peak at 430 nm and a green emission centered at 530 nm. The green emission at 530 nm could be attributed to the OH groups attached to the surfaces of the ZnO NRs. Figure 7 shows that the emission intensity from rGO-modified Schottky LEDs is 2 times stronger than that from LEDs with 3.9-$\mu$m-long ZnO NRs and is 500 times stronger than that from LEDs with 0.383-$\mu$m-long ZnO NRs. This behavior of the rGO-modified Schottky LEDs may be attributed to the surface defects in the ZnO caused by oxygen groups being adsorbed into the rGO and, more probably, to the resonant excitation of graphene plasmons \[37\text{–}40\]. The enhanced luminescence in the rGO-modified Schottky LEDs is due to the missing oxygen functional groups, which can provide percolation pathways between sp2 clusters \[40\text{–}42\]. To elucidate the rGO contribution to the ZnO emission spectra, we performed a fluorescence spectroscopy analysis because this method is recognized as an influential method that can indicate the transfer and trapping of a charge carrier in a material. The fluorescence
emission spectrum of rGO at an excitation wavelength of 300 nm was investigated. Figure 11 shows the fluorescence emission spectra of rGO dissolved in methanol. The spectra exhibit a UV emission peak at 370 nm and a green emission band centered at 540 nm; the emission at 300 nm is the excitation pulse. The result of the fluorescence spectroscopy analysis confirmed that rGO contributes to the emission of ZnO NRs and showed the enhancement in the EL emission spectra. This could be an efficient approach to enhance the optical efficiency of ZnO NRs.

These results indicate that the size of ZnO NRs can be effectively tuned by adjusting the growth parameters and by modulating the seed. Modulating the size of ZnO NRs may have the potential to optimize ZnO NR-based optoelectronic devices.

Conclusions

We have demonstrated the effect of precursor concentration, growth time, and modulation of ZnO seed on the morphology and optoelectronic properties of hydrothermally grown ZnO NRs. The effect of synthesis conditions on the growth of ZnO NRs was systematically studied by SEM, and the optoelectronics properties...
were investigated by EL spectroscopy. Moreover, we have demonstrated a highly promising approach to enhance the luminescence intensity of ZnO NR-based Schottky LEDs by using the appropriate selection of growth parameters. It has been clearly shown that different sizes of ZnO NRs can be obtained by manipulating the growth parameters; this manipulation is aimed at modifying the electrical and optical properties, which could facilitate the continuing research efforts on tunable highly luminescent LEDs.

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