

# UV-Blue and Green Electroluminescence from Cu-Doped ZnO Nanorod Emitters Hydrothermally Synthesized on *p*-GaN

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Aqueous solution synthesis of ZnO nanorods on *p*-GaN(0001) is a low-temperature (< 100 °C) and cost-efficient growth technique of high quality emitters for LED applications. We present morphological, optical and structural properties of zinc oxide nanorod arrays grown by a hydrothermal seed layer-free and rapid synthesis (15 min) on *p*-GaN(0001). We found that the epitaxial layer possesses a close packed hexagonal nanorod morphology and lateral facets are oriented in the same direction for the various nanorods. The effect of Cu-doping on the optical and electroluminescence properties of Cu–ZnO nanorod arrays on GaN substrate is discussed in details. The UV/Blue and green (near-white) emissions were found in both photoluminescence and electroluminescence spectra indicating the possibility to use the synthesized Cu–ZnO/*p*-GaN hetero-structures in white LED applications. The emissions started at relatively low forward voltage of 4.9 V and the intensity of the emission increased with increasing the biasing voltage. We propose for further exploration an efficient, seed layer-free and low temperature hydrothermal synthesis technique to fabricate Cu-doped ZnO/*p*-GaN heterojunction light-emitting devices-LEDs.

**Keywords:** Cu–ZnO Nanorods, ZnO, Hydrothermal, Epitaxy, Photoluminescence, UV-Light Emitting Diode, Green Emission, ZnO/*p*-GaN Heterojunction.

## **1. INTRODUCTION**

In the last few years, light-emitting diodes (LED) based on heterojunctions ZnO nanorods/nanowires grown on *p*-GaN attracted increasing interest based on enhancement of light output intensity and their possible applications in lighting.<sup>1–5</sup> ZnO and GaN have the same wurtzite crystal structure, similar lattice parameters, a small in-plane lattice mismatch ( $\sim 1.9\%$  for the *a* parameter), the same stacking sequence (2H),<sup>6–7</sup> a strong exciton binding energy of 60 meV for ZnO compared to 25 meV for GaN.<sup>5,8</sup> Such properties favor the development of high quality LED based on ZnO/GaN-structure.<sup>9</sup> Nanostructures based on these semiconductors offer the added benefit of material quality leading to improved device efficiency.<sup>10</sup> However, it is known that heterojunctions of n-ZnO/p-GaN-based LED structures emits light in the near-UV range at both low and room temperatures.<sup>5, 11–13</sup> For practical applications it is important to develop white LEDs by using cost-effective technological approaches.

Previous reports demonstrated the bandgap tuning of ZnO films by addition of dopants.<sup>12–19</sup> However, several issues have to be clarified, such as the possibility of doping nanorods through a cost-effective and efficient process, and to tune its properties by incorporation of dopant in

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ZnO lattice. Cu and Zn have similar configurations of their outer electron shells and a small difference in atomic radii (0.057 and 0.074 nm, respectively). Copper is a prominent luminescence activator in II-VI semiconductors.<sup>17,18</sup> The effect of Cu doping on the structural, optical and electroluminescence properties of ZnO nanorods grown by hydrothermal technique could be useful for green LED diodes.<sup>19</sup> Previous works suggested that high concentrations of Cu could be incorporated into ZnO for band-gap engineering.<sup>20-23</sup> Previous theoretical reports<sup>21-24</sup> demonstrated that new bands can be formed inside the ZnO bandgap by Cu-doping, narrowing the bandgap significantly. However, experimental works showed that Cu substituted at the Zn site exhibited defect states located at -0.17 eV below the bottom of the conduction band<sup>25</sup> and at +0.45 eV above the valence band.<sup>23</sup> In recent reports, a greenish-blue shift in the emission peak was observed for Cu-doped ZnO film-based LED device fabricated by plasma-assisted molecular beam epitaxy,26 and by the filtered cathodic vacuum arc technique.<sup>27</sup> One of the most intensively studied approaches to the synthesis of Cudoped nano-ZnO is the vapor deposition technique.28,29 In this case, the dopant concentration depends on gas phase transport which is difficult to control precisely.<sup>30, 31</sup> Another approach is to grow Cu:ZnO nanowires by electrochemical deposition<sup>24, 32, 33</sup> on *p*-GaN which allows to integrate it in LEDs. The effect of different growth regimes, which affect the density of zinc oxide nanowire arrays and respectively the light extraction efficiency, was also experimentally investigated.24, 32, 33

Hydrothermal synthesis of nano-ZnO is an efficient and green procedure of ZnO nanorod growth on many types of surfaces. Our previous reports on the hydrothermal synthesis technique of ZnO nanorods will serve as a reference for this work.<sup>34-36</sup> A recent paper reported on hydrothermal synthesis of single-crystalline Cu-doped ZnO nanorods.37 Xu et al.<sup>38</sup> reported on successfully synthesized single crystal Cu-doped (0.8-2.5 at.%) ZnO nanowires through a facile solution process at a low temperature (< 100 °C). Sharma et al.<sup>39</sup> reported green photoluminescent Cu:ZnO nanophosphors using a simple hydrothermal and suggested applications for white-light LEDs. The origin of bluegreen emission in zinc oxide has remained controversial and it has been assumed that defects, transition-metal doping, and oxygen vacancies are responsible for the green emission in ZnO.<sup>40</sup> Kumar et al.<sup>40</sup> reported a higher intensity of the green emission in Cu doped ZnO nanorods grown by co-precipitation method and suggests that the copper impurity creates deep level defect state in the band gap of zinc oxide. Xing et al.<sup>41</sup> reported on charge transfer dynamics in Cu-doped ZnO nanowires and time resolved photoluminescence measurements showed that the UV decay dynamics coincide with the build-up of the Cu-related green emission. Herng et al.<sup>27</sup> demonstrated the blue and green bands electroluminescence from an heterojunction LED fabricated using the conductive Cu:ZnO layer as an electron injector and a *p*-type GaN as hole injector. These work clearly indicate on the possibility to develop a white LED by using Cu:ZnO/GaN heterojunction.

In this paper, we report on the hydrothermal growth conditions which give the desired Cu:ZnO nanorods on *p*-GaN surface and improved optical quality to achieve high light extraction. The results show that violet–blue emission is comparable to our previously reported ZnO-NR/NW layers grown on *p*-GaN. In the present paper hydrothermal synthesis demonstrated that short wavelength emission can be shifted in the blue range with Cu-doping of ZnO with the occurrence of a green emission, which paves the way for a cost-effective path to fabricate white-LEDs.

## 2. EXPERIMENTAL DETAILS

The ZnO nanorod arrays were grown hydrothermally on p-GaN substrates (TDI, Inc. Corporation) according to a procedure described in our previous papers.<sup>34–36</sup> The *p*-type GaN layer was  $\sim 2.5 \ \mu m$  thick on sapphire, with a crystal miscut of  $\sim 0.59^\circ$  and a Mg-dopant concentration of  $\sim 4 \cdot 10^{18}$  cm<sup>-3</sup>. Before hydrothermal growth, the p-GaN(0001) substrate was cleaned according to Refs. [5, 11] and finally rinsed with high purity water (resistivity  $\sim 18.2 \text{ M}\Omega \cdot \text{cm}$ ). 0.10–0.15 M Zn(SO<sub>4</sub>)  $\cdot$  7H<sub>2</sub>O and 2 M ammonia solution NH<sub>4</sub>OH (Fisher Scientific reagent grade, without further purification) precursors were used for the synthesis of ZnO nanoarchitectures. Manipulation and reactions were carried out inside a fume hood. For one set of samples CuCl<sub>2</sub> (99.99%+, Alfa Aesar) was added in the bath at 4  $\mu$ M to perform doping. All samples were exposed to post-deposition annealing at 300 °C in air for 10 h. Precursors were mixed with 100 ml DI-water until complete dissolution at 20 °C and solution became colorless.<sup>34–36</sup> Details on synthesis procedure can be found in previous works.<sup>34–36,42</sup> Afterwards, samples were characterized by X-ray diffraction (XRD) using a Rigaku 'D/B max' X-ray diffractometer (CuK $_{\alpha}$  radiation source with  $\lambda = 1.54178$  Å). The operating conditions were 40 kV and 30 mA at a scanning rate of 0.02 °/s in the  $2\theta$  range from 25 to 130°, and for enlarged view there were studied in detail regions 34 to 35° and 72 to 75°. The morphologies of the heterostructures were studied by a scanning electron microscope (SEM) Hitachi S800.

The micro-Raman spectra were investigated by using a Horiba Jobin-Yvon LabRam IR system in a backscattering configuration (632.8 nm line of a He–Ne laser). The continuous wave (cw) photoluminescence (PL) was excited by the 325 nm line of a He–Cd Melles Griot laser. The emitted light was collected by lenses and was analyzed with a double spectrophotometer providing a spectral resolution better than 0.5 meV. The signal was detected by a photomultiplier working in the photon counting mode. The samples were mounted on the cold station of a LTS-22-C-330

optical cryogenic system. The LED device, integrating the ZnO-NR/*p*-GaN and Cu:ZnO-NR/*p*-GaN heterostructures, was maintained by a bulldog clip and was biased with a Keitley 2400 source. The electroluminescence (EL) was collected by an optical fiber connected to a CCD Roper Scientific detector (cooled Pixis 100 camera) coupled with a SpectraPro 2150i monochromator. The monochromator focal lens was 150 mm, grating of 300 gr/mm blazed at 500 nm in order to record the emission of the ZnO in the whole near-UV-visible range.

#### 3. RESULTS AND DISCUSSION

Figure 1 presents a SEM top-view of the Cu:ZnO NRs hydrothermally grown on the *p*-type GaN(0001)-substrate. The deposition was made of close-packed hexagonal ZnO rods. The mean radius was 180 nm and the rods had a rather flat top with hexagonal shape. We observed that the lateral facets are oriented in the same direction for the various NRs (Fig. 1) and the top aspect is typical of an epitaxial growth with all the NRs having the same in-plane crystallographic orientation.<sup>5, 11, 43</sup> The inset in Figure 1 exhibits tilted side view of the heterostructure Cu:ZnO NRs/ *p*-GaN/sapphire used in the material characterizations and for integration in LED structures. Also, in Figure 1 (inset) one can see that the Cu:ZnO-NR/GaN interface is smooth and the nanorods are perpendicular to the p-GaN layer/sapphire. It can be suggested that ZnO NRs are epitaxially grown directly on the (0001) p-type GaN:Mg. Quantitative elemental analyses (EDX) were done to estimate the atomic Cu content in the deposition prepared in the presence of copper chloride. The molar ratio between copper and zinc in the ZnO NR arrays was found about 1.9% for samples Cu:ZnO/GaN.



Fig. 1. SEM top-view image of epitaxial HT Cu–ZnO nanorods hydrothermally grown on *p*-GaN substrate at 98 °C in 15 min. Insert shows side-view of ZnO/*p*-GaN/sapphire heterostructure, scale bar is 1  $\mu$ m.

The  $\theta/2\theta$  XRD patterns of the heterostructure (Cu:ZnO NRs/p-GaN) is dominated by both ZnO and GaN peaks (Fig. 2). It is obvious that single crystalline p-GaN film is highly oriented with the c-axis perpendicular to the sapphire substrate. On the enlarged view (Fig. 2(b)), one observes the ZnO(0002) and GaN(0002) diffraction peaks. The XRD pattern matches the lattice spacing of ZnO wurtzite (space group:  $P6_3mc(186)$ ). The data are in good agreement with the Joint Committee on Powder Diffraction Standards (JCPDS) card for ZnO (JCPDS 036-1451). This means that the dopant did not change significantly the wurtzite structure of ZnO and that Cu atoms were in the ZnO NRs. The effect of Cu-doping on the crystallinity of the ZnO nanorods can be seen from a small shift (~0.034°) to a higher  $2\theta$  angle value of the (0002) XRD diffraction peaks for Cu-ZnO as compared with those of ZnO (Fig. 2(b)). The lattice constant c is calculated at 5.2045 Å for pure ZnO NWs and 5.1980 Å for Cu-doped ZnO. A lattice deformation of Cu-doped ZnO was discussed previously in details.<sup>24, 32</sup> The full width at half maximum of the (0002) peak (Fig. 2(b)) increased for Cu-doped samples from 0.06° (pure ZnO) to 0.10° (Cudoped ZnO) suggesting the incorporation and disorder in lattice due to Cu dopant. Such changes in crystallinity might be the result of changes in the atomic environment due to extrinsic doping of ZnO NRs. Due to low formation energy of Cu in ZnO at O-rich conditions, high concentration of dopants can be easily achieved with copper.<sup>21</sup> Our experimental XRD data showed only ZnO peaks at concentration of Cu in the bath of 4  $\mu$ M CuCl<sub>2</sub> which suggests its good incorporation into the lattice, in agreement with previous data.24, 32, 33

Figure 2(c) shows the enlarged views of the ZnO(0004) peak on the left-side of the GaN(0004) reflection of Cudoped ZnO. XRD data confirm that GaN and HT-ZnO have the same out-of-plane orientation. The patterns are typical of a well textured zinc oxide nanomaterial. The full width at half maximum of (0002) peak for ZnO and GaN are  $0.10^{\circ}$  and  $0.07^{\circ}$ , respectively.

Figure 3 presents room temperature micro-Raman spectrum of the Cu:ZnO/GaN heterostructure, indexed with GaN and ZnO emission modes. The Raman peaks located at 100 and 439 cm<sup>-1</sup> are attributed to the ZnO lowand high- $E_2$  modes, respectively.<sup>31–33</sup> The high- $E_2$  mode is clearly visible at 439 cm<sup>-1</sup> with a FWHM of 8 cm<sup>-1</sup>, while the line-width of the peak corresponding to  $E_2$  (low) mode is about 3 cm<sup>-1</sup>, corroborating the high quality of HT-Cu:ZnO.<sup>31–33</sup>

Figure 4 presents the PL spectra of a sample doped with Cu measured at low (10 K) and room temperatures. The spectra are dominated by the near-bandgap UV emission. This emission comes predominantly from the recombination of donor bound excitons ( $D^0X$ ) at low temperature, and is centered at 368.9 nm (3.360 eV). The room temperature near-bandgap PL spectrum represents a structured



Fig. 2. XRD pattern of the HT-Cu–ZnO-NRs/p-GaN/Al<sub>2</sub>O<sub>3</sub>: (a) (0001) structure; (b) enlarged view of the ZnO(0002)/GaN(0002) region (compare pure ZnO and Cu-doped ZnO NRs); and (c) enlarged view of the ZnO(0004)/GaN(0004) region.

band resulting from the superposition of the band originating from the recombination of free excitons (FX) with the maximum at 375.6 nm (3.30 eV) with two LO phonon replica.

Two visible PL bands are present in the spectrum at low temperature. The band located around 1.85 eV is supposed to be associated with a deep unidentified acceptor with the energy level situated close to the middle of the bandgap.44 Another broad band with a maximum around 490-510 nm (2.4-2.5 eV) is observed in the low temperature spectrum. An emission band in this spectral range is often observed in different ZnO samples. Studenikin and Cocivera<sup>45</sup> assigned the green luminescence to a donoracceptor transition (D, A) from oxygen vacancy  $(V_0)$  to Zn vacancies  $(V_{Zn})$ . Kang et al.<sup>46</sup> ascribed the green luminescence to transitions involving deep levels within the band gap associated with oxygen vacancies. We believe that the green emission band observed in our samples can be associated with the Cu impurity. Usually, two types of bands related to the Cu impurity are observed in this spectral

range in Cu-doped ZnO samples. A structured luminescence band has been assigned to the internal transition of a hole in Cu<sub>Zn</sub> center from the excited state at  $\sim E_V + 0.4$  eV to the ground state at  $\sim E_C - 0.2$  eV [Refs. [44, 47, 48] and refs. therein]. The fine structure of the emission spectrum is due to multiple phonon replicas associated with LO and local or pseudolocal vibration modes. Another structureless green luminescence band was attributed to transitions from a shallow donor to the Cu<sup>+</sup> state of a neutral Cu<sub>Zn</sub> acceptor with a level approximately 0.5 eV above the top of the valence band.49 The PL band observed in our sample is structureless. It was previously shown that the structureless band can be transformed into the structured band by annealing the samples at temperatures above 800 °C,<sup>49</sup> and this transformation was attributed to the conversion of the Cu<sup>+</sup> state into the Cu<sup>2+</sup> state. The temperature increase to 300 K leads to the quenching of the PL band at 2.4-2.5 eV (see Fig. 1) which is typical for the Cu-related luminescence from Cu:ZnO.<sup>50</sup> Herng et al.<sup>27</sup> observed in



Fig. 3. Raman spectrum measured at room-temperature of Cu–ZnO NRs hydrothermally grown on p-GaN thin film/sapphire. Samples were annealed at 300 °C for 30 min.



**Fig. 4.** PL spectra of a ZnO sample doped with Cu measured at 10 K (curve 1) and 300 K (curve 2).

ferromagnetic and highly conductive copper-doped ZnO films that the PL spectrum is dominated by the green luminescence, whereas the blue luminescence dominates the electroluminescent (EL) spectra. It was suggested<sup>41</sup> that this difference in the PL and EL spectra is due to the influence of interface defects, and the green band comes from multiple energy levels in the forbidden band due to Cu or/and Zn<sub>i</sub>.

The electroluminescence of the Cu:ZnO NRs/p-GaN LED structure was studied at forward bias at room temperature (RT). A threshold for the violet-green-EL was detected at a remarkably low forward voltage of about 4.9 V and the violet-green-EL signal increased with the applied forward bias. No signal was detected under reverse bias. Figure 5(a) (Curve 1) shows EL spectra measured at 6.4 V from sample #1 (HT-ZnO/p-GaN). Figure 5(a) (Curve 2) shows EL from hydrothermally grown sample #2(HT-Cu:ZnO NRs/p-GaN), which is characterized by an emission peak centered at 417 nm and a broader peak around 520 nm mainly due to Cu-related emission in ZnO. The maximum of the EL wavelength (curve 2) is red-shifted compared to the PL emission of ZnO by about 21 nm. We can observe that the general shape of both Cu:ZnO-PL and EL near-bandgap emissions is similar with the presence of a tail in the violet-blue region. The inset displays the chromaticity coordinate of the spectrum (x = 0.31, y = 0.35 and z = 0.34) and illustrates the near-white color of the emission. Figure 5(b) shows that at lower forward voltage of 6.9 V the electroluminescence emission peak at 417 nm is lower than the maximum wavelength emission around 520 nm. Interestingly, Herng et al.<sup>27</sup> observed green emission only in PL spectra, while EL emission spectra were dominated by the blue luminescence. In contrast to this, the EL emission in our samples, especially at low forward voltage, is dominated by the green luminescence. Apart from that, the green emission in the PL spectra was quenched with increasing temperature up to 300 K, while the green emission is persistent in the EL spectra up to room temperature. On the other hand, in samples with a similar design, but prepared by electrodeposition,<sup>24</sup> a weak red emission band was observed in the EL spectra in addition to the strong near-band-edge emission. All these observations suggest that the luminescence spectrum is strongly influenced by both the excitation conditions and the technological conditions of the ZnO deposition. These issues need additional investigations.

The low emission threshold (< 5 V) and RT UV-blueemission at low voltage (< 10 V) demonstrate that the interface between the two semiconductors is of good quality with a low density of defects and that the developed hydrothermal technique along with previously reported electrodeposition procedure<sup>24, 33</sup> are effective to produce such excellent interfaces.

The hydrothermally grown LED structure possesses improved performances (turn-on voltage  $\sim$  5 V) compared



**Fig. 5.** (a) Electroluminescence spectra of the ITO/Cu:ZnO-NRs/*p*-GaN/In-Ga heterojunction light emitting diode (LED) structure under forward bias voltage of 7.9 V and comparison with pure ZnO EL spectrum at 6.4 V. Curves denotes: 1—pure-ZnO/*p*-GaN at 6.4 V and 2—HT-ZnO:Cu/*p*-GaN. The inset shows the chromaticity coordinate of spectrum (2) (b) Electroluminescence spectra of the ITO/Cu:ZnO-NRs/*p*-GaN/In-Ga heterojunction LED structure under forward bias voltage of 6.9 V. All measurements were done at 20 °C.

to those reported in the literature since in most previous works a forward bias beyond 5–10 V had to be applied to observe a significant EL emission. Moreover, in most cases visible emissions were found to be due to defects or doping levels in the emitting material (e.g., Mg-deep levels in *p*-GaN, intrinsic defects in ZnO, etc.).<sup>51–54</sup>

#### 4. CONCLUSIONS

We report on structural, optical and electroluminescence properties of heterojunctions ZnO nanorods on p-type GaN layers and the effects of the Cu-addition during hydrothermal growth. The developed technological approach permits to synthesize good quality epitaxial material by seed layer-free low-temperature solution growth methods. The Cu:ZnO NRs were vertically oriented with their *c*-axis perpendicular to the (0001) oriented GaN substrate. A comparison of their PL and EL emission properties has been done. The PL spectra were dominated by an UV near-band-edge emission and a broad band located in the green region at low temperature. The PL band located around 1.85 eV is associated with a deep unidentified acceptor with the energy level situated close to the middle of the bandgap.<sup>44</sup> A broad PL band with a maximum around 490–510 nm (2.4–2.5 eV) is observed in the low temperature spectra.

These heterojunctions were used to construct light emitting diode structures. For HT-ZnO, a narrow UVemission peak centered at 399 nm was measured at 20 °C above an applied forward bias of about 4.0 V, and about 5 V for Cu–ZnO/GaN. The electroluminescence intensity increased with the applied forward voltage. For HT-Cu:ZnO NRs/*p*-GaN heterostructures were measured an emission peak centered at 417 nm and a broader peak around 520 nm mainly due to Cu-related emission in ZnO. Our results state the effectiveness of hydrothermally grown ZnO as an active layer in solid state near-white lighting device.

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