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Controlled electroluminescence of *n*-ZnMgO/*p*-GaN light-emitting diodes

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Effective control of room-temperature electroluminescence of *n*-ZnMgO/*p*-GaN light-emitting diodes (LEDs) over both emission intensity and wavelength is demonstrated. With varied Mg concentration, the intensity of LEDs in the near-ultraviolet region is increased due to the effective radiative recombination in the ZnMgO layer. Furthermore, the emission wavelength is shifted to the green/yellow spectral region by employing an indium-tin-oxide thin film as the dopant source, where thermally activated indium diffusion creates extra deep defect levels for carrier recombination. These results clearly demonstrate the effectiveness of controlled metal incorporation in achieving high energy efficiency and spectral tunability of the *n*-ZnMgO/*p*-GaN LED devices. © 2012 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4773367>]

Light emitting diode (LED) devices generally consist of oppositely doped semiconducting materials in contact with each other to form the *p-n* junction, where electrons and holes recombine to emit light when a bias voltage is applied. In recent years, LEDs have attracted very strong interest in numerous optical and optoelectronic applications owing to their exceptional properties such as lower energy consumption, longer lifetime, and lower cost compared to the incandescent bulbs.^{1,2} Various semiconducting materials have been implemented in LED devices, including gallium arsenide (GaAs),³ tin oxide (SnO₂),⁴ zinc oxide (ZnO),^{5,6} aluminum nitride (AlN),⁷ and conductive polymers such as poly(3,4-ethylenedioxythiophene).⁸ Among them, ZnO nanofilms are very promising as ZnO is a direct band-gap semiconductor ($E_g \sim 3.3$ eV), has a large exciton binding energy (~ 60 meV), and can produce visible photoluminescence (PL) at and even above room temperature.^{6,9}

The electroluminescence (EL) of most ZnO-based LED devices, however, currently remains unsatisfactory due to their low carrier concentrations, inefficient carrier injection and recombination, and limited ability to tune the emission wavelength. These limitations severely hamper the versatility and potential of ZnO in nanophotonic and nanoelectronic applications.¹⁰ To solve this, a variety of ZnO-based structures with engineered nanoscale features have been reported, including the asymmetric double heterojunction structure,¹¹ the multi-quantum wells of ZnO/ZnMgO,¹² the layered ZnO/ZnMgO nanorods,¹⁰ and the rib-like waveguide structures.⁵ Unfortunately, the growth and fabrication processes of these nanostructures are rather complicated and the integration of these materials into functional devices remains a significant challenge.

In this work, we demonstrate the simultaneous control over both the intensity and wavelength of electroluminescence of *n*-ZnO/*p*-GaN LEDs by controlling the active ZnO thin films with custom-selected metal ions. The heterostructural

n-ZnO/*p*-GaN system is one of the mostly studied ZnO-based LED devices. Alivov *et al.* and Rogers *et al.* have pioneered in the fabrication of *n*-ZnO/*p*-GaN LEDs and demonstrated their room-temperature EL in the blue-UV region, which was directly related to the band structure of ZnO.^{13,14} Chen *et al.* and Zhang *et al.* inserted an additional layer at the interface of *n*-ZnO and *p*-GaN to increase the emission intensity,^{15,16} whilst we recently fabricated a rib-like waveguide structure and observed an increase of ~ 5 times.⁵ On the other hand, a variety of different metal dopants and the post-annealing technique have been adopted to tune the EL spectral wavelength.^{17,18} Nevertheless, the efficiency of these methods remains relatively low for tailoring the intensity and wavelength of the *n*-ZnO/*p*-GaN LEDs.

We show that by first incorporating magnesium (Mg) ions, the emission intensity of *n*-ZnMgO/*p*-GaN LEDs in the near-UV spectral region can be substantially increased by more than two orders of magnitude compared to the undoped *n*-ZnO/*p*-GaN device. Furthermore, the emission wavelength can be shifted from the near-UV to the green/yellow (~ 550 nm) spectral regions by doping with indium ions. We propose that the incorporation of these metal ions changes the band structure and the defect levels within the energy band gap of the ZnO layer, which subsequently affect the EL properties of the LED devices. Our results are promising for fabricating energy-efficient solid-state lighting, flexible organic-inorganic lighting,¹⁹ quantum computing,² and a variety of optoelectronic devices, which are important for the development of a sustainable, green, and energy-efficient future.²⁰

To prepare the LED devices, 200 nm thick ZnMgO film was first deposited onto one half of the *p*-GaN/sapphire substrate (Semiconductor Wafer Inc.) using filtered cathodic vacuum arc (FCVA) technique.²¹ As compared to the conventional ion implantation technique,^{22,23} the FCVA technique offers several advantageous features such as uniform doping and good film quality arising from the plasma-based deposition environment.^{24,25} The metal targets used in FCVA were ZnMg alloys with different Mg contents (nominal 0–15 wt. %), made

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by compressing the two metal powders using hydraulic press.²¹ The *p*-GaN layer was used as a hole injection layer because both ZnMgO and GaN show a similar band gap energy (~ 3.4 eV), and a low lattice constant mismatch of 1.9%.²⁶ The oxygen partial pressure was set at 2×10^{-5} Torr during the deposition. When the desired thickness was obtained, the films were annealed at 600 °C in air for 30 min to reduce defects and improve the film quality. Finally, a Ni (20 nm)/Au (50 nm) contact was deposited onto the other half of the *p*-GaN:Mg/sapphire substrate using the electron-beam evaporation technique (Edwards AUTO 306).

Figure 1(a) shows a schematic of the fabricated ZnMgO-based LED devices and Figs. 1(b)–1(d) show the field-emission scanning electron microscopy (FE-SEM; Zeiss Leo Gemini 1550) images of the ZnMgO-based nanofilms obtained by ZnMg alloys with 0%, 4%, and 15% Mg content, respectively. As the Mg level in the nanofilms could be significantly different from the nominal content in the metal alloys, we probed the surface composition using X-ray photoelectron spectroscopy (XPS; Specs Sage 150). The XPS measurements indicated that when 4% and 15% Mg were used, the actual Mg contents on the surface of thin films were 30% and 50%, respectively, i.e., forming $\text{Zn}_{0.7}\text{Mg}_{0.3}\text{O}$ and $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ compounds.²⁷ This systematically larger value in the as-deposited nanofilms was attributed to the large vapor pressure of ZnO and Zn, which caused Zn-related species to easily desorb from the substrate surface.²⁸ However, since XPS is mostly a surface-analysis technique and the crystal structure of ZnO normally changes when the Mg content increases, we used X-ray diffraction (XRD; Siemens) technique to further investigate the film structure. It was shown that all films were polycrystalline and of hexagonal structure, with the prominent peaks from the (002) plane.²⁷ Moreover, a shoulder peak was observed near the $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ (002) peak, possibly relating to the ZnO phase. Therefore, we noted that the $\text{Zn}_{0.7}\text{Mg}_{0.3}\text{O}$ thin film had $\text{Zn}_{0.96}\text{Mg}_{0.04}$ in source material and the $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ thin film had $\text{Zn}_{0.85}\text{Mg}_{0.15}$ in source material. For simplicity, the expressions of $\text{Zn}_{0.7}\text{Mg}_{0.3}\text{O}$ and $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ are used hereafter.

One can also see from Figs. 1(b) to 1(d) that the surface of intrinsic ZnO nanofilm consisted of uniformly distributed nanocrystalline ZnO grains, which became slightly non-uniform after the incorporation of Mg content. We intentionally used Mg because the size of Mg ions is very close to that of Zn ions, resulting in a minimal lattice mismatch between ZnO and MgO.¹⁰ More importantly, the formed ZnMgO compound shows a tunable energy band gap up to 0.8 eV and an enhanced carrier mobility, which are highly desirable for the color-tuned LED applications.^{29,30}

The room-temperature EL was measured using a custom-built system (a monochromator coupled to a PDS-01 PMT detector). During the measurements, the positive electrode was connected to the *p*-GaN substrate while the negative electrode was connected to an indium-tin-oxide (ITO)-coated quartz mechanically pressed against the top of the $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ thin film.³¹ Light was then collected from the uncoated side of the quartz substrate through an objective lens. The typical room-temperature EL spectra of the ZnO and $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ LEDs at varied forward biases are shown in Figs. 2(a) and 2(b), respectively. In both cases, broad-band emissions were observed in the wavelength range from 350 to 500 nm. The dominant peaks of each spectrum, located at 410 and 398 nm for the ZnO and $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ films, respectively, were in the near-UV region. The EL spectra of $\text{Zn}_{0.7}\text{Mg}_{0.3}\text{O}$ LEDs showed a similar shape with a peak centered at the wavelength of 403 nm.²⁷ This emission wavelength is higher compared to the ZnO nanofilm but lower than $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$. It was noted that the emission from all the LED devices was strong enough to be observed clearly with a naked eye.

In general, the light emission of *n*-ZnO/*p*-GaN LEDs has a strong peak in the near-UV region and also a weak and broad band in the blue/green region.^{9,17} The former was attributed to the radiative recombination between electrons in the conduction band of the *n*-ZnO layer and holes injected from the valence band of the hole-injection layer *p*-GaN, so called the *near-band-edge* (NBE) recombination.^{6,17} The broad band in the blue/green region, on the other hand, was

FIG. 1. (a) Schematic diagram of the *n*-ZnMgO/*p*-GaN heterojunction LED device. SEM images of ZnMgO thin films with the Mg contents of (b) 0%, (c) 4%, and (d) 15%.

FIG. 2. The EL spectra of the (a) n -ZnO/ p -GaN and (b) n -Zn_{0.5}Mg_{0.5}O/ p -GaN (Zn_{0.85}Mg_{0.15} in source material) LED devices under various bias voltages. The corresponding PL spectra of the (c) ZnO and (d) Zn_{0.5}Mg_{0.5}O films.

normally assumed to be due to the transition between the electrons trapped in the defect levels of the ZnO layer and the holes injected from the GaN layer. The current observation of only strong peaks in the near-UV region but not in the blue/green region confirmed that the nanofilms were of high quality.

We then probed the optical band gaps of ZnO and Zn_{0.5}Mg_{0.5}O by the PL measurements, where the sample was pumped at an excitation power of ~ 1 mW/cm² by a 355 nm frequency tripled Nd-YAG pulsed laser (6 ns pulse width and 10 Hz repetition rate). The dominant PL peaks were found at 380 and 376 nm for ZnO and Zn_{0.5}Mg_{0.5}O, respectively [Figs. 2(c) and 2(d)], arising from the NBE recombination. As the EL wavelengths of both devices were red-shifted by ~ 25 meV compared to the PL peaks, it led us to assume that the light emission was generated through a two-stage radiative recombination process.^{4,7,32} In this process, the electrons are first captured by the shallow surface energy levels of the n -ZnO and n -ZnMgO thin films and then recombine with the injected holes from the p -GaN layer to emit light.

Figures 2(a) and 2(b) also show that the EL peak positions of the Zn_{0.5}Mg_{0.5}O nanofilm-based LED device slightly blueshifted compared to the ZnO LED device. The observed dependence of the EL spectra on the elemental composition of the ZnMgO nanofilms clearly indicates that the EL emission occurred predominantly from the n -ZnMgO side; otherwise, the wavelength would remain relatively constant if it was due to the NBE recombination at the p -GaN side (the PL peak of p -GaN was at 363 nm).²⁷ The blueshift in the emission can be related to the Mg incorporation, which broadens the intrinsic energy band gap of ZnO films.^{33,34} Indeed, incorporating different metals has been demonstrated as an effective way of bandgap engineering of semiconducting nanomaterials.³⁵ For example, the bandgap of Zn_{0.9}Mg_{0.1}O is ~ 0.25 eV wider compared to the stoichiometric ZnO.³⁵ Thus, the recombination energy of charge carriers

is expected to be higher in the ZnMgO nanofilms, which is consistent with our observations of a shorter wavelength of light emission generated.

Another prominent feature observed from Fig. 2 is that the EL intensity of Zn_{0.5}Mg_{0.5}O LED device is ~ 120 times (i.e., two orders of magnitude) higher than that of the n -ZnO/ p -GaN heterojunction at a similar bias voltage (e.g., at 10 V). The turn-on voltage, defined as the voltage at which the LED current begins to increase in an exponential manner, is ~ 8 V for the n -ZnO/ p -GaN heterojunction and ~ 4 V for the n -Zn_{0.5}Mg_{0.5}O/ p -GaN heterojunction. The mechanism and difference between the mentioned heterojunctions and previous report of ~ 6 V will be reported later.⁵ Such a significant increase in the EL intensity and a low turn-voltage are very promising for applying the LED devices in *low energy-consumption lighting* and *reducing the inevitably associated greenhouse gas emissions*.²⁰ We relate these experimental observations to several quantum effects arising from Mg incorporation:

- (i) the broadening in the energy band gap could induce energetic shifts of both the conduction and valence bands of the ZnMgO nanofilm, leading to a lower band offset and a higher concentration of electrons and holes injected to the nanoscale junctions;²⁶
- (ii) the incorporation of Mg content could induce quantum spatial carrier confinement at the interface of n -ZnMgO/ p -GaN, which greatly increased the recombination rates of electrons and holes;³⁶
- (iii) the carrier mobility is larger in the ZnMgO nanofilm compared to the ZnO film.³⁰

To further enhance the light emission by reducing the contact resistance, we deposited a 150 nm thick ITO film onto the Zn_{0.5}Mg_{0.5}O nanofilm using radio-frequency (RF) sputtering. The ITO film was then rapidly annealed at 400 °C for 20 min in air to obtain a good ohmic contact.²⁷ Figure 3(a) shows the current-voltage (I-V) characteristics of the

FIG. 3. (a) The current-voltage (I-V) characteristics of $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ before and after the ITO deposition and annealing at 400°C . The inset shows the leakage current at reverse bias voltage conditions. (b) The electroluminescence and (c) photoluminescence spectra of the annealed $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ LED device under various bias voltages. Inset in (b) is a photo taken from the device under a bias voltage of 15 V. (d) The electroluminescence spectra of the ITO/ $n\text{-Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ /p-GaN heterojunction-based LED device further annealed at 500°C . The emission peak in (d) has shifted back to the near-UV region.

$p\text{-ZnMgO}/n\text{-GaN}$ heterojunction before and after the annealing, which has indeed led to a remarkable improvement in the conductivity of the heterojunction. The leakage current (at reverse bias) was low for the LED both before and after the ITO deposition (inset of Fig. 3(a)), clearly indicating the diode-like rectifying characteristics.³⁷

An interesting feature was observed in the EL spectra of the annealed $n\text{-ZnMgO}/p\text{-GaN}$ LED device, as shown in Fig. 3(b). Using the same EL measurement configuration, the emission wavelength has shifted from the near-UV [$\sim 398\text{ nm}$; Fig. 2(b)] to the green/yellow ($\sim 550\text{ nm}$) spectral range. This yellowish light emission can be clearly seen from the whole surface of the $\text{Zn}_{0.5}\text{Mg}_{0.5}\text{O}$ device [inset of Fig. 3(b)], evidencing a good uniformity of the light-emitting film.³¹ Additionally, the corresponding PL measurement showed a dominant peak at $\sim 536\text{ nm}$ [Fig. 3(c)], further confirming that the wavelength shift was an intrinsic property of the ZnMgO film. The light emission at 550 nm is in the middle of the simulated AM1.5 solar spectrum. Thus, this LED device can be used in solar simulator and spectrophotometers.

We now discuss the mechanism of the observed emission features. At high annealing temperatures, Indium can easily diffuse from the ITO layer to the ZnMgO nanofilm due to the low eutectic temperature (143.5°C) of the In-Zn system.^{38,39} This thermally activated diffusion may create extra deep defect levels in the ZnMgO film. These levels form below the conduction band minimum of ZnMgO and can be used to either enhance the EL intensity or tune the EL wavelength.^{31,35,37} When operated under the forward bias conditions, the electrons in the conduction band are captured by these deep defect levels and recombine at the $n\text{-ZnMgO}/p\text{-GaN}$ interface with the holes injected from the $p\text{-GaN}$ layer. As the recombination energy becomes smaller, a larger emission wavelength was obtained. Figure 4 illustrates the energy

band diagram of the $n\text{-Zn}_{0.5}\text{Mg}_{0.5}\text{O}/p\text{-GaN}$ heterojunction at a forward bias voltage.^{6,9} In general, the energy band gap for the ZnMgO and GaN thin films is ~ 3.3 and 3.4 eV , respectively.^{6,21} When the heterojunction is formed, the bands of $n\text{-ZnMgO}/p\text{-GaN}$ exhibit a type-II alignment with a small band offset voltage ($\sim 0.1\text{ eV}$). The energy level of these deep defects, as obtained from the EL peaks, is $\sim 2.2\text{ eV}$ above the valence band of ZnMgO (Fig. 4). This value is consistent with the results obtained by other authors.³⁵

It is worth noting that although a much higher current density (under the same bias voltage) was obtained after the deposition of the ITO film [Fig. 3(a)], the deep defect levels in the annealed ZnMgO film remained limited. A smaller number of electrons were involved in the deep defect-related radiative transitions compared to the radiative recombination

FIG. 4. The energy band diagram of the $n\text{-Zn}_{0.5}\text{Mg}_{0.5}\text{O}/p\text{-GaN}$ heterojunction at forward bias conditions. At equilibrium, electrons are captured by the indium-induced deep defect levels ($\sim 2.2\text{ eV}$ above the valence band of ZnMgO) and then recombine with the holes injected from the valence band of GaN to emit light.

before annealing.³¹ This is why the annealed *n*-ZnMgO/*p*-GaN heterojunction featured a larger turn-on voltage and a lower EL intensity [Figs. 2(b) and 3(b)].

Finally, we further annealed the ITO/*n*-ZnMgO/*p*-GaN LED device at 500 °C for another 20 min. Interestingly, the strong EL peak has shifted back to the near-UV wavelength range (~386 nm), as shown in Fig. 3(d). This observation is attributed to the release of indium ions from the ZnMgO film to, possibly, the bottom GaN layer. Ling *et al.* recently observed a similar phenomenon that less Cu dopants remained in the ZnCuO film at higher temperatures.³⁷ The release of indium ions also reduces the shallow surface vacancy levels,³² which could account for the shorter EL wavelength [at 386 nm; Fig. 3(d)] as compared to that of the LED device without the ITO electrode [at 398 nm; Fig. 2(b)]. The thermally activated diffusion and release of metal dopants in the present case are quite similar to the case of thermally doped and de-doped single-walled carbon nanotubes.⁴⁰

In conclusion, we have demonstrated that the emission intensity of *n*-ZnMgO/*p*-GaN LEDs can be enhanced by two orders of magnitude through Mg incorporation. The thermally activated indium diffusion could further create extra deep defect levels and shift the wavelength emission from the near-UV to the green/yellow spectral region. The growth and metal incorporation processes of ZnMgO thin films reported here were simpler yet more efficient as compared to other procedures. The ability to tune the emission wavelength of LED devices could be of critical importance for the practical implementation of the full-color light sources. Our results clearly demonstrate the generality and efficiency of deterministically incorporating different metal ions to improve the electroluminescence properties of LED devices, which are highly promising for many advanced and energy-efficient electronic and optoelectronic applications.⁴¹

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