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Fabrication and properties of p-type K doped $Zn_{1-x}Mg_xO$ thin film

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ABSTRACT

A series of K doped $Zn_{1-x}Mg_xO$ thin films have been prepared by pulsed laser deposition (PLD). Hall-effect measurements indicate that the films exhibit stable p-type behavior with duration of at least six months. The band gap of the K doped $Zn_{1-x}Mg_xO$ films undergoes a blueshift due to the Mg incorporation. However, photoluminescence (PL) results reveal that the crystallinity decreased with the increasing of Mg content. The fabricated K doped p-type $Zn_{0.95}Mg_{0.05}O$ thin film exhibits good electrical properties, with resistivity of 15.21 Ω cm and hole concentration of 5.54×10^{18} cm^{-3}. Furthermore, a simple ZnO-based p–n heterojunction was prepared by deposition of a K-doped p-type $Zn_{0.95}Mg_{0.05}O$ layer on Ga-doped n-type ZnO thin film with low resistivity. The p–n diode heterostructure exhibits typical rectification behavior of p–n junctions.

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1. Introduction

ZnO is a novel II-VI compound semiconductor material with direct wide band-gap (3.37 eV) and large exciton binding energy (60 meV) [1–4]. As it owns these merits, its application in light-emitting device in ultraviolet wavelength is promising. Being a native n-type semiconductor, p-type ZnO is relatively difficult to realize and usually it has an aging effect [5], which restricts the application of ZnO-based LED devices. To date, regarding the acceptor dopants, much attention has been paid to the group-V elements such as N [6,7], P [8,9], As [10,11], Sb [12], etc., and some p-type conversions have been achieved. Recently, group-I elements such as Li and Na have been explored, which can substitute Zn^{2+} and give rise to one hole in ZnO film likewise. Park et al. and Janotti and Walle [13,14] reported that group-I elements were identified to be shallow acceptors to create p-type conduction in ZnO compared with group-V elements. As for group-I dopants, Li has been studied for a long time [15]. However, as Li ion has a small radius, it tends to stay in the interstitial position and acts as donor. Recent investigations have suggested that doping Na into ZnO is a promising p-type conversion method [16,17]. Little attention has been paid to another important element K in group-I and its related alloys [18–22], which behaves analogously to Na. In previous study, co-doping methods have been tried to realize ZnO p-type conversion by using Al–N, In–N, Na–H, Mg–Li, Li–N, etc., and the goal is to facility the solubility of acceptor and improve the p-type stability [23–27]. In this paper, we will focus on the fabrication and properties of K doped $Zn_{1-x}Mg_xO$ ($Zn_{1-x}Mg_xO$:K) alloy thin films. When Mg is introduced into ZnO, the band gap gets wider than the pure ZnO, and the conduction level is enhanced, giving rise to a deeper native dope energy level [17,28]. Moreover, element K owns a larger size, so the forming of interstitials is suppressed and it has a relative low acceptor level, which benefits to the p-type conversion as well. The fabricating of $Zn_{1-x}Mg_xO$:K alloy thin film method achieves not only p-type conversion but also band gap engineering simultaneously without disturbing the wurtzite structure. Our motivation is to clarify the behavior of K in $Zn_{1-x}Mg_xO$ films based on the structural, electrical, and optical properties, which we will elucidate in the following.

2. Experimental details

A series of K doped p-$Zn_{1-x}Mg_xO$ thin films were fabricated by pulsed laser deposition (PLD). The ceramic target ($Zn_{1-x}Mg_xO$:K) was prepared by mixing ZnO (5 N), MgO (4 N), and K_2CO_3 (5 N) powders and agate balls with ethanol milled for 24 h. In the $Zn_{1-x}Mg_xO$:K targets, the Mg content is 5 at.%, 10 at.%, 15 at.% respectively and the K content is 1 at.%. While the ZnO:K target was made by mixing ZnO and K_2CO_3 powders with 1% K in mole percent. After a thermal treatment at 50 °C for 24 h, the powder was molded into pellets with diameter of 4 cm. In the end, the pellets were sintered at 800 °C for 2 h with further keeping at 1300 °C for another 2 h. Afterwards, it cooled down in ambient conduction. During the film growth, the substrates were kept at 500 °C and ultrapure O_2 (99.9999%) with a working pressure of 40 Pa was introduced into the chamber. A KrF excimer laser (Compex102, 248 nm, 25 ns) was used as the ablation source. The laser repetition rate was 5 Hz and the energy per pulse was 340 mJ.

The film structure and crystalline quality were characterized by Bede D1 X-ray diffraction (XRD) system with a Cu Kα radiation (λ = 0.15406 nm). The electrical properties were investigated by using a four-point probe van der Pauw configuration (HL5500PC) at room temperature. The field-emission scanning electron microscope (FE-SEM) and high-resolution transmission electron microscope (HRTEM) were employed for morphological and microcosmic structures studies. Energy-dispersive

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Table 1
Hall measurement results of $Zn_{1-x}Mg_xO$:K films fabricated with different Mg content and different oxygen pressure. The electrical properties were measured after six months for comparison. ('*' Means the signal is out the range of Hall measurement, '+' means the film is p-type, '–' means the film is n-type, '/' means the film thickness is not measured.).

Sample	Atmosphere (Pa)	μ ($cm^2\ V^{-1}\ S^{-1}$)	ρ (Ω cm)	N (cm^{-3})	Thickness (nm)
ZnO:$K_{0.01}$	40	0.31	82.50	$+2.44 \times 10^{17}$	226
ZnO:$K_{0.01}$ (6 months)	40	0.04	17.43	-9.80×10^{18}	/
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$	40	0.08	15.21	$+5.54 \times 10^{18}$	212
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$ (6 months)	40	0.11	11.72	$+4.77 \times 10^{18}$	/
$Zn_{0.90}Mg_{0.10}O$:$K_{0.01}$	40	2.16	931.90	$+3.10 \times 10^{15}$	234
$Zn_{0.90}Mg_{0.10}O$:$K_{0.01}$ (6 months)	40	0.68	80600	$+1.14 \times 10^{14}$	/
$Zn_{0.85}Mg_{0.15}O$:$K_{0.01}$	40	*	*	*	231
$Zn_{0.85}Mg_{0.15}O$:$K_{0.01}$ (6 months)	40	*	*	*	/
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$	0.04	7.85	0.03	-2.73×10^{19}	223
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$ (6 months)	0.04	0.14	0.04	-2.03×10^{20}	/
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$	0.4	2.25	0.15	-1.87×10^{19}	240
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$ (6 months)	0.4	2.90	0.16	-1.35×10^{19}	/
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$	4	2.47	0.21	-1.20×10^{19}	245
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$ (6 months)	4	2.39	0.21	-1.26×10^{19}	/
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$	20	0.10	2.85	$+2.27 \times 10^{18}$	238
$Zn_{0.95}Mg_{0.05}O$:$K_{0.01}$ (6 months)	20	0.56	1.56	-2.27×10^{18}	/

This work discussed the effect of Mg and K codoped into ZnO. The $Zn_{0.95}Mg_{0.05}O$:K film obtained at 500 °C with 40 Pa O_2 exhibits a better p-type property, which has an electrical resistivity of 15.21 Ω cm, and a carrier concentration of $5.54 \times 10^{18}\ cm^{-3}$. The rectification of the p-$Zn_{0.95}Mg_{0.05}O$:K/n-ZnO:Ga heterojunction device also confirms the p-type property of the $Zn_{0.95}Mg_{0.05}O$:K film. Our results indicate that p-type $Zn_{1-x}Mg_xO$:K films may show a promising future in the fabricating of p-type ZnO film.

X-ray spectroscopy (EDX) was used to study the element compositions. The optical transmission through the film was measured in the wavelength range from 300 to 800 nm by a Varian Cary-300 spectrophotometer. X-ray spectroscopy (XPS) was used to study the element compositions. A $Zn_{0.95}Mg_{0.05}O$:K/n-ZnO:Ga heterojunction was made by depositing p-type $Zn_{0.95}Mg_{0.05}O$:K thin film on the Ga-doped n-type ZnO thin film. The Ga-doped n-type ZnO thin film was prepared on Al_2O_3 substrate, with electron concentration higher than $\sim 10^{19}\ cm^{-3}$ and resistivity of $\sim 10^{-3}$ Ω cm. The electrodes of the $Zn_{0.95}Mg_{0.05}O$:K/n-ZnO:Ga heterojunction were made by depositing Ni (30 nm) and Au (70 nm) on the p-type $Zn_{0.95}Mg_{0.05}O$:K film by magnetron sputtering, while Ti (30 nm) and Au (70 nm) electrodes were deposited on the prepared Ga-doped n-type ZnO thin film respectively. Good ohmic contacts on both p-type $Zn_{1-x}Mg_xO$:K and Ga-doped n-type ZnO thin film were made by rapid thermal annealing at 600 °C for 1 min in N_2 atmosphere [29]. Agilent E5270B parameter analyzer was used to analyze the current–voltage (I–V) characteristics of the heterojunction. Photoluminescence (PL) measurements were performed on a FLSP920 (Edinburgh Instruments) fluorescence spectrometer at room temperature.

3. Results and discussion

After 30 min deposition, a series of $Zn_{1-x}Mg_xO$:K films were grown by varying the Mg content from 0 to 15%. Referring to our previous study, it seems that the films obtained at a suitable high oxygen pressure are more likely to realize the p-type conversion [16]. Excess oxygen atoms in atmosphere not only decrease the concentration of V_O greatly, but also increase that of O_i and V_{Zn}, both of them being acceptors and contributing to the p-type conductivity in a certain degree. Thus, p-type ZnO films are easier to be obtained in oxygen rich environment [30]. Therefore, we chose the films deposited under 40 Pa O_2 ambient to study. The films obtained at lower oxygen pressure were found to be n-type and the related results are shown in Table 1.

The θ–2θ X-ray diffraction (XRD) patterns of the as-grown $Zn_{1-x}Mg_xO$:K films are shown in Fig. 1, indicating that no peaks originate from other phases such as MgO or metal Mg are detected within the sensitivity of XRD measurements. Although Mg and K are co-incorporated into the ZnO, the $Zn_{1-x}Mg_xO$:K films are well c-axis preferentially oriented. From the XRD pattern, it can be seen that all the (0 0 0 2) peaks of $Zn_{1-x}Mg_xO$:K samples could be fitted with the hexagonal wurtzite structure having slightly decreased lattice parameter values (or increased 2θ values) in comparison to that of pristine ZnO sample ($2\theta = 34.45°$). The decreased lattice parameter values indicate the incorporation of Mg^{2+} (0.66 Å) at the replacement sites of Zn^{2+} (0.74 Å). As the Mg content is increased from 0 to 15%, the (0 0 0 2) peak of $Zn_{1-x}Mg_xO$:K moves from 34.53° to 34.64°.

Fig. 2a–d shows the evolution of surface morphologies of $Zn_{1-x}Mg_xO$:K films deposited at 500 °C with 40 Pa O_2 as the ambient gas. It is clear that when the Mg content is 5% or below, the grain size is small and the surface of the film is smooth and dense without macroscopic defects. When Mg content is increased to above 10%, obvious grain boundaries and macroscopic defects both emerged obviously. In previous report, the MgO can be formed when the Mg content reached nearly 35% [31]. While in the fabricated the $Zn_{1-x}Mg_xO$:K films, the highest Mg content is limited to 15%, thus the possibility of forming MgO clusters is negligible. In order to rule out the possibility of existence of MgO clusters, HRTEM image (taken randomly throughout the film) together with EDX mapping were carried out (see the supporting information material) on the $Zn_{0.85}Mg_{0.15}O$:K film. These results confirm that Mg is uniformly doped into the $Zn_{0.85}Mg_{0.15}O$:K film. Therefore, it is proven that for all the films with Mg content less than 15% are surely without existing the MgO clusters.

The transmission spectra of $Zn_{1-x}Mg_xO$:K films in the wavelength from 300 to 800 nm are measured, as shown in Fig. 3. It shows that the optical transmittance of the film is over 85% in the visible wavelength region. The inset shows the relationship

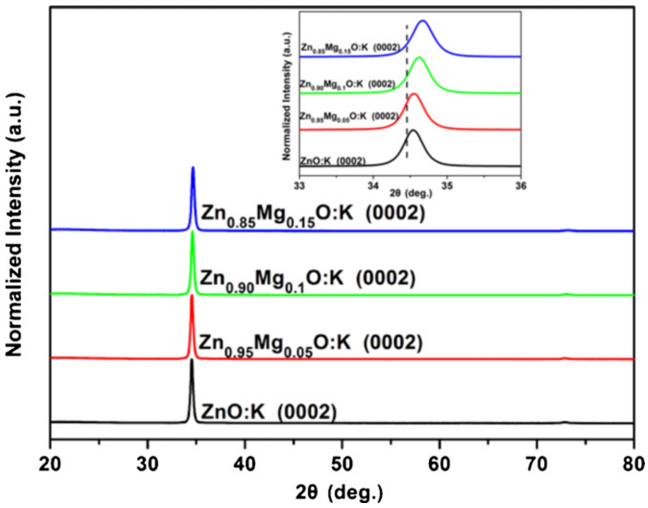

Fig. 1. XRD patterns of $Zn_{1-x}Mg_xO$:K films deposited with various Mg contents. The inset is the (0 0 0 2) peak of $Zn_{1-x}Mg_xO$:K films in a large scale.

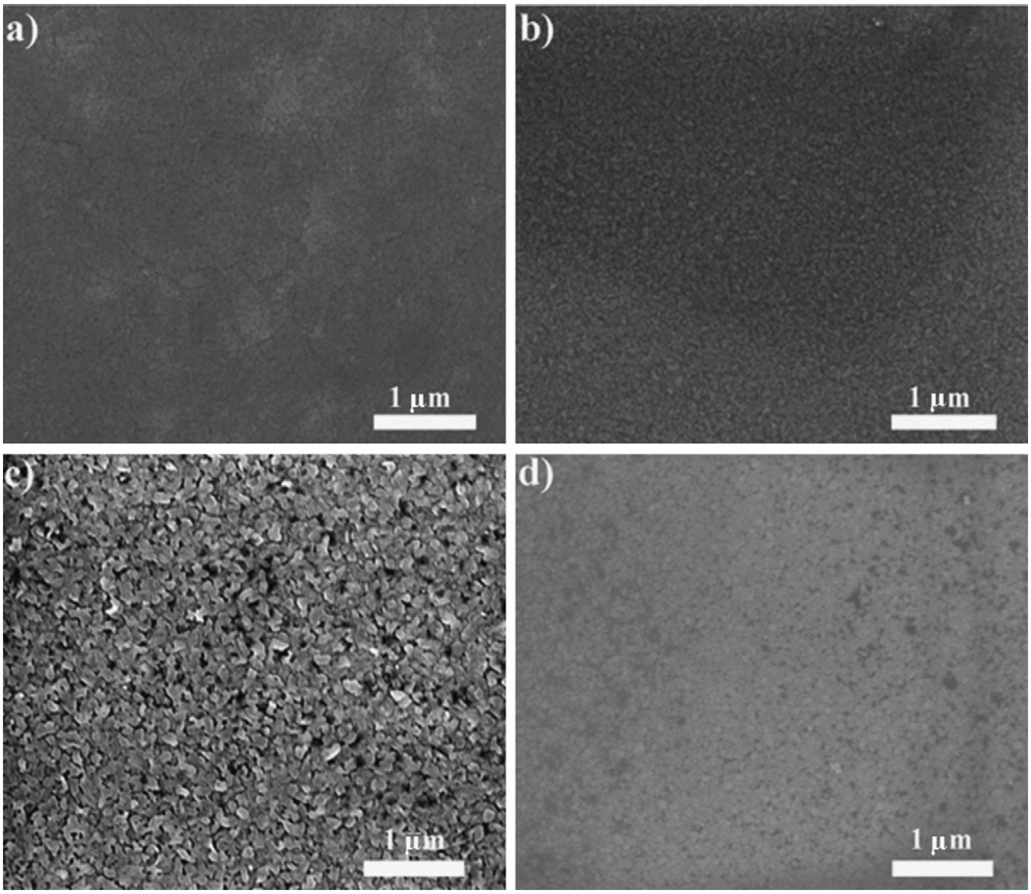

Fig. 2. SEM images $Zn_{1-x}Mg_xO$:K films deposited on quartz substrates with different Mg contents (a) $x = 0$, (b) $x = 0.05$, (c) $x = 0.10$, (d) $x = 0.15$.

between the optical adsorption band gap of $Zn_{1-x}Mg_xO$:K film versus the Mg content. The band-gap (E_g) value was evaluated by extrapolation of the linear portion of the $(\alpha h\nu)^2$ versus $h\nu$ relation, where α is the absorption coefficient and $h\nu$ is photon energy. From Fig. 3, it is evident that the band gap of the $Zn_{1-x}Mg_xO$:K films undergoes a blueshift. The optical gap of $Zn_{0.85}Mg_{0.05}O$:K thin film is much lager than that of pristine ZnO:K thin film. Mg–K co-incorporating realized band gap engineering, modifying the band gap from 3.25 to 3.5 eV.

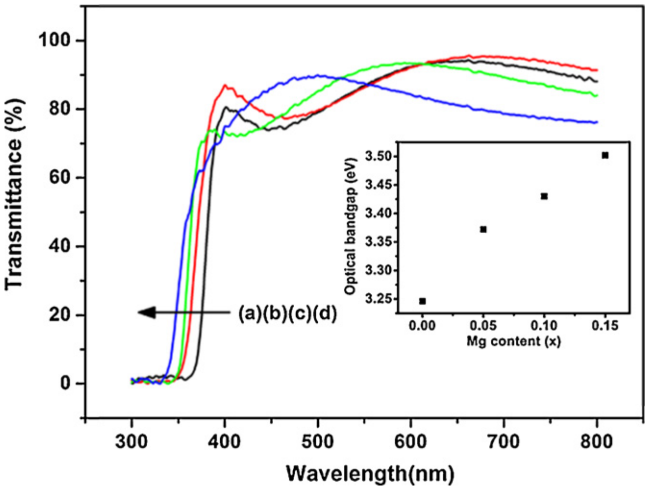

Fig. 3. Transmittance spectra of $Zn_{1-x}Mg_xO$:K films deposited on quartz substrates with various Mg contents. The inset shows the variation of optical band gap (E_g) of $Zn_{1-x}Mg_xO$:K films with different Mg contents.

Table 1 shows the electrical resistivity (ρ), carrier concentration (n), mobility (μ) and thickness as a function of Mg content and oxygen pressure. The values in each below are the Hall results measured after six months for comparison. ('*' Means the signal is out the range of Hall measurement '+' means the film is p-type, '−' means the film is n-type, '/' means the thickness is not measured.) When the Mg content increases from 0 to 15%, the electrical properties change from p-type to high resistivity (or insulating). It is found that the film doped with 5% Mg and 1% K has a relative low resistivity and more stable p-type behavior compared with other samples. When Mg is introduced into ZnO, the band gap of $Zn_{1-x}Mg_xO$:K film gets wider than the pure ZnO, therefore the conduction band level is raised, thus increasing deep energy level of the native dopant, and consequently, facilitating the p-type conversion. The motivation for considering the (Zn,Mg)O alloy resides in the potential to reduce the residual n-type conductivity due to shallow defect donor state. The addition of Mg moves the conduction band edge up in energy and potentially away from the intrinsic shallow donor state, thus increasing the activation energy of the defect donors. [32,33] This interpretation will be responsible for the higher hole concentration observed in the $Zn_{0.95}Mg_{0.05}O$:K than that in ZnO:K fabricated with identical condition. The realization of p-type conversion indicates the activation of K acceptor. However, with the increasing of Mg content (>5%), the electrical properties of the films degraded, similar with previous reported in Li doped p-type ZnMgO [34], which can be explained by the acceptor level getting too deep to activate in such a wide band-gap.

To study the oxygen pressure influence on the electrical properties, we further fabricated a series of $Zn_{0.95}Mg_{0.05}O$:K with various oxygen pressures. The electrical measurement results are shown in Table 1. The change of conduction behavior with respect to oxygen

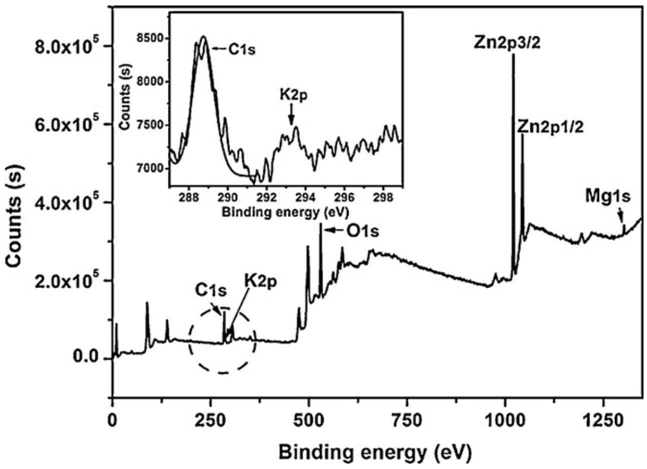

Fig. 4. XPS spectrum of $Zn_{0.95}Mg_{0.05}O$:K film obtained at 500 °C with 40 Pa O_2. The inset is the select area in a large scale, showing the appearance of K2p.

pressure can be understood as follows: as the oxygen pressure is far below 40 Pa, the strong self-compensation effect from the shallow native donor level takes the major role, and consequently, the films show n-type behavior. When the oxygen pressure increases up to 40 Pa, there will be enough oxygen to compensate the oxygen defects and finally realizes a p-type conduction. It is found from Table 1 that $Zn_{0.95}Mg_{0.05}O$:K thin film fabricated at 500 °C and 40 Pa O_2 has relatively the optimized p-type properties and can stay stably for more than six months without degrading. Thus, in the following section, all investigations are based on the $Zn_{0.95}Mg_{0.05}O$ film obtained at 500 °C with 40 Pa O_2.

As K content is relatively low in the ceramic target and there is concentration discrepancy between the target and the deposited film, it is necessary to confirm the existence of K in the thin films after fabrication. Thus, XPS measurement on $Zn_{0.95}Mg_{0.05}O$ thin film is shown in Fig. 4. The concentration of Zn, Mg, O, K is 39.3%, 6.5%, 54%, 0.2% respectively. The peak centered at 293.5 eV is assigned to originate from K incorporated into ZnO. The observed K related peak (see inset) supports the presence of K in the film. As the XPS detective depth is only about several nanometers during the experiment, it cannot exclude the possibility of other adsorptions on the surface without Ar^+ ion cleaning, so the C1s peak coexists with the K2p peak in the pattern. Herein, ZnO always exhibits n-type conductivity naturally, and the introducing of Mg^{2+} does not offer extra electrons or holes. Consequently, the probable reason of the p-type conversion is attributed to K^+ substituting of Zn^{2+} or Mg^{2+} in the film. As the film shows p-type, it is therefore suggested that there existed more K^+ substituting Zn^{2+} playing as the acceptor than the native donors, as a result, the film shows p-type conductivity, which is consistent with the Hall measurement result.

To further confirm the p-type behavior of $Zn_{0.95}Mg_{0.05}O$:K thin film, a simple ZnO-based p–n junction was fabricated, as shown in Fig. 5. The p–n junction was prepared by depositing p-type $Zn_{0.95}Mg_{0.05}O$:K thin film on a pre-obtained Ga-doped low-resistance n-type ZnO (ZnO:Ga) thin film with Al_2O_3 as the substrate. The inset of Fig. 5 shows the schematic structure of the p-$Zn_{0.95}Mg_{0.05}O$:K/n-ZnO:Ga heterojunction. The electrodes Ni/Au (30 nm/70 nm) on p-type $Zn_{0.95}Mg_{0.05}O$:K and the Ti/Au (30 nm/70 nm) electrodes on n-type ZnO:Ga thin film were respectively made by magnetron sputtering. From the inset of Fig. 5, the good linear properties of the electrodes indicate they have good ohmic contacts with the films after rapid thermal annealing at 600 °C for 1 min in N_2 atmosphere. As the pre-obtained Ga-doped n-type ZnO thin film with a low-resistance, only when the obtained $Zn_{0.95}Mg_{0.05}O$:K film behaves good p-type property can the heterojunction shows intrinsic rectifying behavior. The current–voltage measurement at room temperature shows a typical diode characteristic. The device showed remarkable rectification characteristics after measuring for several times, which further confirms the p-type property of the $Zn_{0.95}Mg_{0.05}O$:K thin film.

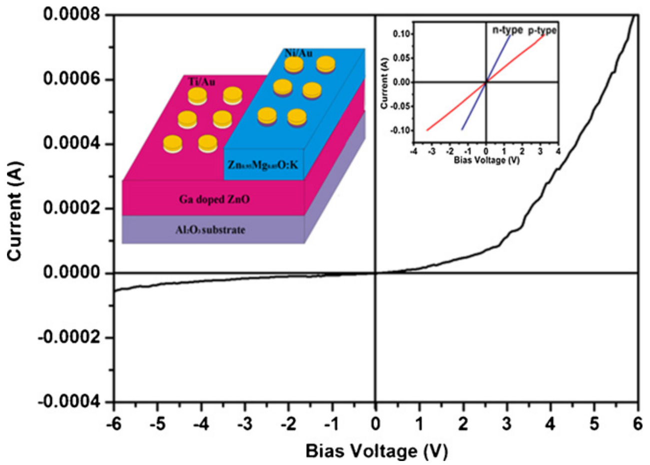

Fig. 5. The current–voltage (*I*–*V*) characteristics of $Zn_{0.95}Mg_{0.05}O$:K/n-ZnO:Ga heterojunction taken at room temperature. The inset shows the schematic cross-sectional view of the heterojunction device and the *I*–*V* curve between the electrodes and the ZnMgO:K and ZnO:Ga films respectively. (The red line shows the ohmic contact between the Ni/Au electrode and the p-type ZnMgO:K film, while the blue line shows the ohmic contact between the Ti/Au electrode and the n-type ZnO:Ga film). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

Fig. 6 shows the PL spectra of the $Zn_{1-x}Mg_xO$:K thin film fabricated on quartz substrate. From the spectra, it can be seen that there is an ultraviolet emission peak for each sample. As the Mg content increasing, the emission peak located at the ultraviolet range moves to shorter wavelength range, indicating a broader band gap between the valance band and conduction band, which is consistent with the optical adsorption result. Thus, the incorporated Mg realizes the band gap engineering in ZnO. Furthermore, as the $Zn_{1-x}Mg_xO$:K thin films were obtained on quartz substrate, there is not any hetero-epitaxial growth relationship between the films and the substrate. Moreover, at a relatively fast growing rate, it cannot get rid of forming some defects in the film during depositing. This explains the broad peaks observed locating in the visible range, which can be ascribed to the defects existed in ZnO [35,36].

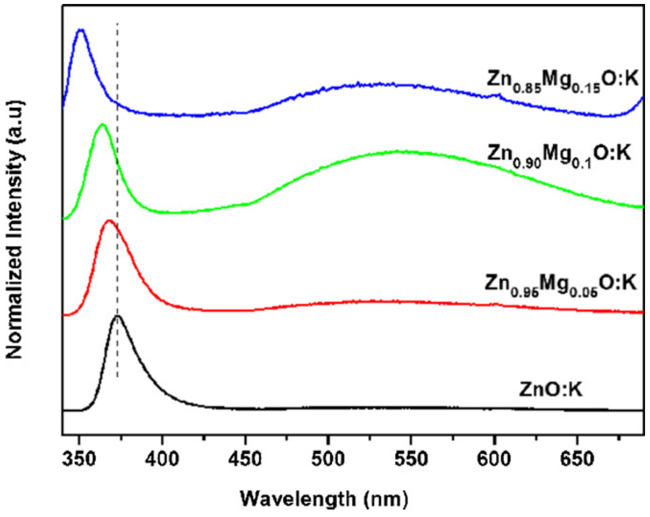

Fig. 6. Room-temperature PL spectra of the $Zn_{1-x}Mg_xO$:K thin film fabricated.

4. Conclusions

In conclusion, we have demonstrated the fabrication of $Zn_{1-x}Mg_{x}O$:K films by PLD. Although Mg and K are co-incorporated into ZnO, the films still maintain wurtizite structure. The $Zn_{0.95}Mg_{0.05}O$:K film obtained at 500 °C with 40 Pa O_2 exhibits a better p-type conduction, with resistivity of 15.21 Ω cm, and carrier concentration of 5.54×10^{18} cm^{-3}. The rectification behavior of the p-$Zn_{0.95}Mg_{0.05}O$:K/n-ZnO:Ga heterojunction device further confirms the p-type properties of the $Zn_{0.95}Mg_{0.05}O$:K film. Our results indicate that p-type $Zn_{1-x}Mg_{x}O$:K films may show a promising future in the fabricating of p-type ZnO film.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jallcom.2011.04.047.

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