Optical and electrical properties of gallium-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$

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In this study, the optical and electrical properties of epitaxial single crystal gallium-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films grown on c-plane sapphire substrates by pulsed laser deposition were investigated. In these films, the Ga content was varied from 0.05 to 7 at. % and the Mg content was varied from 5 to 15 at. %. X-ray diffraction showed that the solid solubility limit of Ga in $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ is less than 3 at. %. The absorption spectra were fitted to examine Ga doping effects on bandgap and band tail characteristics. Distinctive trends in fitted bandgap and band tail characteristics were determined in films with Ga content below 3 at. % and Ga content above 3 at. %. The effects of bandgap engineering on optical transparency were evaluated using transmission spectra. Carrier concentration and Hall mobility data were obtained as functions of Ga content and Mg content. The electrical properties were significantly degraded when the Ga content exceeded 3 at. %. Correlations between conduction mechanisms and gallium doping of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films were described. In addition, the effect of bandgap engineering on the electrical properties of epitaxial single crystal gallium-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films was discussed. © 2010 American Institute of Physics. [doi:10.1063/1.3271415]

I. INTRODUCTION

Transparent conducting oxide (TCO) materials are used in a variety of optoelectronic devices, including flat panel displays and thin film solar cells.¹ Indium doped tin oxide is the most widely used TCO material. In recent years, concerns regarding the availability of indium have risen due to the fact that indium is a limited natural resource.¹ As a result, significant research efforts have been undertaken to develop alternative TCO materials, including Nb doped TiO_2 as well as ZnO-based materials.² Among alternative TCO materials, ZnO (bandgap=3.4 eV), ZnO doped with Al, and ZnO doped with Ga have received significant attention.^{3–6} For example, Jin *et al.*⁴ deposited Al-doped ZnO thin films using dc magnetron sputtering of Al along with rf magnetron sputtering of ZnO; they prepared films with ~85% thermal infrared reflectance.⁵ ZnO has several other advantages, including nontoxicity and low cost.⁷

Zinc oxide can be alloyed with other oxides, such as MgO ,⁸ CdO ,⁹ and BeO .¹⁰ The bandgap of ZnO alloys can be tailored by varying the alloy composition. For example, the bandgap of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ alloy can be engineered between the bandgap values of ZnO (3.4 eV) and MgO (7.8 eV). The crystalline structure of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ can change from hexagonal to cubic when the Mg content exceeds 40 at. %.¹¹ Single crystalline hexagonal $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ films containing magnesium content up to 36 at. % without phase separation have been prepared using pulsed laser deposition (PLD); films with bandgap values up to 4.0 eV were prepared using this method.¹² Mimemoto *et al.*¹³ demonstrated use of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films as transparent window layers for

Cu(In,Ga)Se_2 (CIGS) solar cells. The conduction band offset values of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ and CIGS were controlled by changing the bandgap, enabling optimization of CIGS solar cell conversion efficiency.

As with ZnO, the bandgap of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ can be modified by doping this material with various elements. Several investigators have examined the electrical and optical properties of doped Al-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$.^{14,15} For example, Matsubara *et al.*¹⁴ demonstrated use of Al-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ as a bandgap tunable TCO material. The reported resistivity increased from 3×10^{-4} to $1 \times 10^{-3} \Omega \text{ cm}$ as the bandgap increased from 3.5 to above 3.9 eV. Fewer studies have been performed on Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ materials. For example, Harada *et al.*¹⁶ examined the electrical properties of Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films grown by molecular beam epitaxy. Phase separation was observed when the gallium content exceeded $1 \times 10^{18} \text{ cm}^{-3}$, which restricted their evaluation of the electrical properties of single crystal Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ in the carrier concentration range lower than $2 \times 10^{17} \text{ cm}^{-3}$.

In this study, a systematic investigation of the optical and electrical properties of Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ was performed, in which the Ga content was varied from 0.05 to 7 at. % and Mg content was varied from 5 to 15 at. %. Absorption spectra and transmission spectra were obtained in order to determine the effect of gallium doping on optical properties. Carrier concentration and Hall mobility data were examined as functions of Ga content and Mg content.

II. EXPERIMENTAL PROCEDURE

Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films were grown on double-side polished c-sapphire substrates by pulsed laser deposition

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using a substrate temperature of 650 °C; films with thickness values of ~ 200 nm were obtained. The target-to-substrate distance was maintained at 45 mm during the deposition process. The oxygen partial pressure was maintained at 2×10^{-5} Torr during the deposition process. A COMPex 205 KrF excimer laser (Lambda Physik, Fort Lauderdale, FL, USA) was used for ablation of the Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ targets; this laser was performed using a wavelength and a pulse duration of 248 nm and 25 ns, respectively. A pulse repetition rate of 10 Hz was used in this study. The energy density of the laser beam was ~ 3.5 J/cm². The stoichiometries of thin films prepared using PLD closely correspond with those of the target materials if the energy density of laser beam is high enough to enable formation of a stoichiometric plume from the target material and if the substrate is aligned with the center of the plume.¹⁷ Recent work by Bhosle *et al.*¹⁸ used x-ray photoelectron spectroscopy in order to demonstrate that the dopant concentration in Ga-doped ZnO prepared using PLD corresponded with that of the target material.

The multicomponent targets were prepared by solid-state sintering method. 99.99 at. % purity ZnO (Alfa Aesar, Ward Hill, MA, USA), 99.99 at. % purity MgO (Alfa Aesar, Ward Hill, MA, USA), and 99.99 at. % purity Ga₂O₃ (Sigma Aldrich, St. Louis, MO, USA) powders were mixed in methanol solution using a magnetic stirring plate. The methanol solvent was subsequently removed by heating on a hot plate. The mixture was pressed into a 1 in. pellet. The pellet was sintered at 1000 °C in air for 24 h. The crystalline structure of the Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films was investigated using x-ray diffraction (XRD) (Rigaku, The Woodlands, TX) with Cu K α radiation and a nickel filter. The optical spectra were measured with a U-3010 UV-Vis dual-beam spectrometer (Hitachi, Tokyo, Japan). Hall measurements were obtained with an Ecopia HMS-3000 system (Bridge Technology, Chandler Heights AZ, USA) using the van der Pauw configuration at room temperature.

III. RESULTS AND DISCUSSION

A. X-ray diffraction studies of Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films

Figure 1 shows the XRD θ - 2θ curves for $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$ thin films with Ga content ranging from 0.05 to 7 at. %. (0006) diffraction of the c-sapphire substrate was assigned to the peak with a 2θ value of 41.6°. The peak shown at $\sim 34.6^\circ$ was attributed to (0002) diffraction of the wurtzite $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ structure. In films containing a gallium content of 3 at. % and higher, a small peak at 29.4° was observed; this feature was attributed to (220) diffraction of the ZnGa_2O_4 spinel structure.¹⁹ The presence of the spinel phase indicates that the solid solubility for Ga in $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ is close to 3 at. %. This solubility limit is considerably higher than the reported thermodynamic solubility value (0.5 at. %), which was obtained from samples prepared by solid state reaction of Ga₂O₃ and ZnO powders.¹⁸ The relatively high Ga concentration in PLD-deposited films was attributed to the non-equilibrium nature of excimer laser ablation. The average kinetic energy of depositing species produced by exci-

FIG. 1. XRD profile for gallium-doped $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$. The gallium content in these films varies from 0.05 to 7 at. %.

mer laser ablation is 100–1000 times higher than the thermodynamic equilibrium value (kT , in which k is the Boltzmann constant and T is the temperature). The XRD data also revealed that Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films were textured along the c-axis. Previous work by Jin *et al.*²⁰ involving Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films grown on c-plane sapphire indicates that film growth occurs by means of domain matching epitaxy.²¹

B. Optical properties of Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films

Figure 2 shows optical transmission spectra of $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$ thin films with Ga content between 0.05 and 7 at. %. The overall transmission values varied in the range of $\sim 80\%$ – 95% for wavelengths between ~ 370 and 800 nm, showing that all of the films were highly transparent in the visible wavelength range. The variation in transmittance was attributed to thin film interference. Figure 3 shows the absorption spectra of $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$ thin films with Ga content between 0.05 and 7 at. %. The absorption spectrum for the $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$ thin film containing 0.05 at. % Ga showed two excitonic peaks near the fundamental optical absorption

FIG. 2. (Color online) Transmission spectra for gallium-doped $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$. The gallium content in these films varies from 0.05 to 7 at. %.

FIG. 3. (Color online) Absorption spectra for gallium-doped $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$. The gallium content in these films varies from 0.05 to 7 at. %.

edge. Only one excitonic peak was observed as the Ga content increased to 0.1 at. %, which was attributed to broadening of excitonic states caused by dissociation of excitons by impurity atoms.²² This dissociation process was continuously enhanced as the Ga content was increased from 0.1 to 1 at. %. The exponential band tail (the Urbach tail) became prominent when the Ga content was further increased from 3 to 7 at. %, which indicates that the films were highly disordered. Considering that the ZnGa_2O_4 spinel phase forms in films with Ga content greater than 3 at. %, the absorption spectra demonstrate good agreement with XRD data.

The absorption spectra were fitted for examining bandgap and Urbach tail values in order to further investigate the effect of Ga doping on the optical properties of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ films. The optical bandgap E_g was determined using the following equation:

$$\alpha(E) = C \cdot (E - E_g)^n, \quad (1)$$

in which C is a constant and n is a power factor. In this case, $n = \frac{1}{2}$ is used for curve fitting since $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ is a direct band semiconductor and the band to band direct allowed transition is dominant.¹⁹ The absorption coefficient can be determined from original absorption spectra using the following equation:

$$I = I_0 \cdot \exp(-\alpha \cdot t), \quad (2)$$

in which α is the absorption coefficient and t is the thickness. Fitting was performed by plotting the square of the absorption coefficient as a function of the photon energy and extrapolating the linear region of the plotted curve to the photon energy axis. The bandgap values derived from this data fitting are listed in Table I.

The band tail was determined by fitting the absorption coefficient in the vicinity of the fundamental absorption edge using the Urbach equation:

$$\alpha(E) = \alpha_0 \cdot \exp\left(\frac{E - E_g}{E_u}\right), \quad (3)$$

in which α is the absorption coefficient, α_0 is the pre-exponential factor, and E_g is the optical bandgap, and E_u is the Urbach tail.²³ The Urbach tail (E_u) is used to describe exponential dependence of the absorption coefficient at the optical absorption edge. The Urbach tail is caused by disorder

TABLE I. The fitted values of bandgap and Urbach tail for gallium-doped $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$.

Gallium content (at. %)	Bandgap (eV)	Urbach tail (meV)
0.05	3.45	100
0.1	3.44	109
0.5	3.47	100
1	3.48	91
3	3.39	134
5	3.38	155
7	3.35	164

der in materials, which leads to an extension of the parabolic density of states into the band edge. Disorder generally includes thermal disorder, which reflects thermal occupation of phonon states, or structural disorder, which is associated with impurities and defects in the material.²⁴ In this case, the Urbach tail was mainly attributed to the effects of Ga doping on the fundamental optical absorption. The Urbach tail values determined by fitting the absorption spectra in the wavelength range from $\lambda = 345$ to $\lambda = 450$ nm are listed in Table I.

The bandgap and band tail values derived from curve fitting show two different types of correlations. Below a Ga content of 3 at. %, the bandgap showed a blueshift and increased from 3.45 to 3.48 eV; the Urbach tail was approximately constant and the fitted value was ~ 100 meV. Above a Ga content of 3 at. %, the bandgap showed a redshift and decreased from 3.48 to 3.35 eV; the fitted Urbach tail showed a continuous increase from 91 to 164 meV. This band gap narrowing can be understood by considering the widening of doping levels as a function of Ga content. Below a Ga content of 3 at. %, the Ga doping level was located below the conduction band and was separated from the conduction band. It should be noted that the doping level may have become broader as the Ga content was increased. In this case, there was no significant change in the density of states near the conduction band bottom, resulting in the almost constant Urbach tail values. The blueshift of bandgap was attributed to the Burstein–Moss effect since the electrons occupied the lower energy states in the conduction band and the band to band direct transition only occurred to higher energy states.²⁵ Above a Ga content of 3 at. %, the doping level became so broad that it was incorporated into the conduction band bottom. This incorporation of doping level as well as the formation of ZnGa_2O_4 spinel caused an extension of the density of states into the bandgap, resulting in the band tail at the band edge and the redshift in the bandgap.²⁶ As additional Ga dopants were incorporated in the $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin film, the Urbach tail became larger and the bandgap was reduced. Figure 4 shows the effects of Mg on the optical properties of Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films. As the Mg content was increased, the fundamental absorption edge shifted to the lower wavelength region due to the increase in bandgap. This shift improved optical transparency in the shorter wavelength region. As shown in Fig. 3(a), the onset of the absorption edge changed from $\lambda = 355$ to $\lambda = 340$ nm when the magnesium content was increased from 5 to 15 at. %, respectively.

FIG. 4. (Color online) Transmission spectra for 1 at. % gallium-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$. The magnesium content in these films varies from 5 to 15 at. %.

C. Electrical properties of Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films

Figure 5 shows the carrier concentrations of Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films as a function of Ga content. For films with Mg content of 5 and 10 at. %, the carrier concentration generally increased as the Ga content was continuously increased from 0.05 to 1 at. %. At 5 at. % Mg, the carrier concentration increased from 8.2×10^{18} to $1.1 \times 10^{20} \text{ cm}^{-3}$. For films with 10 at. % Mg, the carrier concentration increased from 5.2×10^{18} to $9.2 \times 10^{19} \text{ cm}^{-3}$. It should be noted that the carrier concentration became significantly lower when the Ga content was increased from 3 at. %. At 5 at. % Mg, the carrier concentration decreased to $2.1 \times 10^{19} \text{ cm}^{-3}$. At 10 at. % Mg, the carrier concentration decreased to $1.4 \times 10^{19} \text{ cm}^{-3}$. As the Ga content was increased from 3 to 7 at. %, the carrier concentration showed a very small increase, indicating that Ga doping of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ was saturated in this doping content range. For films with 15 at. % Mg, the maximum carrier concentration ($3 \times 10^{18} \text{ cm}^{-3}$) was achieved at 0.5 at. % Ga. The carrier concentration continuously decreased as the Ga content was further increased. For $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ films with 5 and 7 at. % Ga content, the carrier concentration exceeded the measurement capabilities of the Hall measurement system. The significant reduction in carrier concentration for $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ films with

FIG. 5. (Color online) Carrier concentration data for gallium-doped $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$.

FIG. 6. (Color online) Hall mobility data for gallium-doped $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$.

high Ga content region indicated that a considerable fraction of Ga dopants were not electrically active. This was mainly attributed to the formation of the ZnGa_2O_4 spinel phase when the Ga content exceeded 3 at. %.

Figure 6 shows Hall mobility data for Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films as a function of Ga content. Hall mobility values increased as the Ga content was increased from 0.05 to 0.5 at. %. The highest Hall mobility value ($34 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$) was obtained from the 0.5 at. % Ga-doped $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$ thin film. The measured Hall mobility data show very good agreement with Hall mobility values for doped ZnO that have been described in the literature.²⁷ The tendency of the Hall mobility values to increase with increasing Ga content indicates that the dominant scattering mechanism below a Ga content of 3 at. % is grain boundary scattering.²⁸ For electron transport by thermionic emission across the grain boundary region, the effective mobility μ_{eff} is determined by the grain barrier with potential energy E_b ,

$$\mu_{\text{eff}} = \frac{eL}{\sqrt{2\pi m^* kT}} \cdot \exp(-E_b/kT). \quad (4)$$

In this equation, L is the grain size, m^* is the effective mass, T is the sample temperature, and k is the Boltzmann constant. This model has been applied to epitaxially grown Al- or Ga-doped ZnO thin films in the $\leq 10^{19} \text{ cm}^{-3}$ carrier concentration range.²⁹ The Hall mobility continuously decreased as the Ga content was increased from 0.5 to 7 at. %. For $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$ thin films, the Hall mobility dramatically decreased from 34 to $0.84 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. The rapid decrease in Hall mobility in the heavy doping region cannot be explained by well-known theoretical models such as Conwell–Weisskopf and Brooks–Herring–Dingle models, in which substitutional dopant atoms are assumed to be distributed homogeneously as scattering centers. It should be noted that this assumption is not valid due to the existence of the ZnGa_2O_4 spinel phase. It is more likely that the ZnGa_2O_4 spinel phase can form clusters that are distributed throughout the films. These clusters may play a critical role in the scattering process, which results in the rapid decrease in Hall mobility values. Lu *et al.*³⁰ also reported a similar decrease in Hall mobility values for heavily Al-doped (up to 10 at. %)

ZnO, which was attributed to formation of Al–Al and Al–O clusters in the intragrain region; an intragrain cluster scattering mechanism was proposed. Our experimental results suggest that structural inhomogeneity is an important factor in determining electron mobility for heavily doped semiconductors, particularly ZnO-based TCO materials, since ZnO can easily form spinel phases with Al_2O_3 and Ga_2O_3 .¹⁷ Masetti *et al.*³¹ proposed an empirical methodology for modeling mobility as a function of doping content. Theoretical models are needed to describe the dramatic decrease in electron mobility to very low values for heavily doped ZnO.

The Mg content has a significant influence on the electrical properties of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films.³² At a given Ga content, higher carrier concentration values were observed in $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films with lower Mg content. In 1 at. % Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$, the carrier concentration dropped significantly from 1.1×10^{20} to $2.1 \times 10^{19} \text{ cm}^{-3}$. At a given gallium concentration, Hall mobility (μ) values were shown to decrease in $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films with higher magnesium concentration values. These results can be attributed to several factors. The electron effective mass can affect mobility, which can be defined using the following equation:

$$\mu = \frac{q\langle\tau\rangle}{m^*}. \quad (5)$$

In this equation, $\langle\tau\rangle$ is electron average scattering time and q is the electron charge. In films with higher magnesium content, the electron effective mass is increased, which in turn decreases the mobility.³³ In addition, the incorporation of magnesium in the $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ film introduces an alloy scattering mechanism, which decreases the electron mobility.¹⁴ The carrier concentration is also decreased with increasing magnesium concentration, which indicates that the addition of magnesium can lower the Ga doping efficiency.

IV. CONCLUSIONS

Gallium-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films, in which the Ga content was varied from 0.05 to 7 at. % and Mg content was varied from 5 to 15 at. %, have been epitaxially grown on sapphire substrates by PLD. XRD θ - 2θ data revealed that the solid solubility of Ga in $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ is less than 3 at. %. In order to evaluate the effect of Ga doping on the optical properties of $\text{Mg}_x\text{Zn}_{1-x}\text{O}$, absorption spectra were fitted to determine bandgap and Urbach tail values. When the Ga content was lower than 3 at. %, the bandgap showed a blueshift and Urbach tail remained approximately constant with an increase in Ga content. When the Ga content was higher than 3 at. %, the bandgap showed a redshift and Urbach tail continuously increased with an increase in Ga content. The transmittance values for these films were in the range of 80%–95% in the wavelength range from $\lambda \sim 370$ to $\lambda = 800 \text{ nm}$ for $\text{Mg}_{0.05}\text{Zn}_{0.95}\text{O}$. This optical transparent window can be extended to the shorter wavelength region through modulation of the Mg content. The onset of the ab-

sorption edge extended from approximately $\lambda = 320$ to $\lambda = 340 \text{ nm}$ as the Mg content was increased from 5 to 15 at. %. However, the carrier concentration and Hall mobility values were decreased in films with high Mg content. These results suggest that optical transparency and conductivity values were compromised due to bandgap engineering in Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films. The carrier concentration and Hall mobility values were significantly degraded in films with a Ga content greater than 3 at. %. A systematic evaluation of the electrical and optical properties of Ga-doped $\text{Mg}_x\text{Zn}_{1-x}\text{O}$ thin films provides support for the use of this material in TCO applications.

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