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Properties of shallow donors in ZnMgO epilayers grown by metal organic chemical vapor deposition

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High quality $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers have been grown by means of metal organic chemical vapor deposition technique on top of ZnO templates. The grown samples were investigated by x-ray photoelectron spectroscopy and photoluminescence. The magnesium (Mg) concentration was varied between 0% and 3% in order to study the properties of shallow donors. The free and donor bound excitons could be observed simultaneously in our high quality $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers in the photoluminescence spectra. The results indicate that both built-in strain and Mg-concentration influence the donor exciton binding energy. It clearly shows that the donor exciton binding energy decreases with increasing Mg-concentration and with increasing built-in strain. Furthermore, the results indicate that the donor bound exciton transition energy increases with decreasing strength of the built-in strain if the Mg-concentration is kept the same in the $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4902007>]

INTRODUCTION

Due to the large free exciton binding energy (60 meV) and excellent optical properties, the wide-bandgap ZnO related materials have attracted significant attention both in fundamental research and for device applications. The major research efforts have been focused on investigations and fabrication of ZnO related nanostructures, the p-type doping, and optoelectronic devices based on ZnO. The ZnO epilayers and nanostructures have been grown on various types of substrates by different techniques, such as molecular beam epitaxy (MBE),^{1–4} metal-organic vapor phase epitaxy or metal organic chemical vapor deposition (MOCVD),^{5–10} pulsed laser deposition (PLD),^{11,12} the vapour-liquid-solid (VLS) catalytic growth technique,^{13–15} magnetron sputtering,^{16,17} and low temperature chemical solutions.^{18,19} There are also great efforts to grow p-type layers of ZnO and ZnO based heterostructures (such as ZnMgO/ZnO, ZnBeO/ZnO, and ZnMnO/ZnO). The growth of the latter heterostructures by means of MBE and MOCVD has been reported.^{1–10,20} The fabrication of p-type ZnO has also been reported by employment of different growth techniques. However, the realization of light emitting diodes (LEDs) has only been demonstrated for epilayer materials grown by MBE,^{21,22} MOCVD,²³ and hybrid beam deposition.²⁴ The nitrogen (N)-as acceptors in ZnO have been questioned recently,²⁵ and also the efficiency of N-as acceptor dopant in ZnO layer is very low. The quality of the p-type doping is still well below the required level for device performance, in addition to the instability of p-type doping. Another factor, which influences the realization to achieve a high hole concentration in p-type ZnO epilayer, is the high un-intentional n-type background impurity concentration. To solve the problem, this fact may suggest for seeking other ZnO-related alloys with a larger bandgap energy and possible lower un-intentional background impurity concentration in comparison with ZnO.

Recent investigations of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers indicate that the conductivity of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ layer deteriorates with increasing Mg-concentration.²⁶ It was concluded that the deterioration of the electrical properties with increasing Mg content may be caused by a combination of precipitation of the rocksalt MgZnO second phase, a decreasing crystallinity level, and bulk alloying effects. However, to draw a firm conclusion about the conductivity deterioration in ZnMgO layer, the properties of $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ layer, such as effective mass and dopant ionization energy, should also be investigated thoroughly but not achieved yet.

In this study, we have investigated one important property, the shallow donor ionization energy in $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers, by using photoluminescence (PL) spectroscopy. Furthermore, relative information on the background impurity concentration has been derived from a comparison among different samples from PL spectra. An AIXTRON shower-head type MOCVD has been used to grow $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers with various Mg-concentrations. It has been shown that the ionization energies obey the Haynes' rule, i.e., the ionization energy of shallow impurities is linearly correlated to the binding energy of the exciton bound to the corresponding shallow impurity²⁷ for the ZnO materials. We have deduced the shallow donor bound exciton (DX) binding energy versus the Mg-concentration in $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers from the measured photoluminescence spectra. In order to gain this information on the donor ionization energies, it is required that both the free exciton (FX) and donor bound exciton can be observed simultaneously, which accordingly require high quality $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers with low background doping levels. Such high quality $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers have been grown by means of the metal organic chemical vapor deposition technique on ZnO templates. The magnesium (Mg) concentration was varied between 0% and 3%. The grown samples were investigated by X-ray photoelectron spectroscopy (XPS) and PL. Our

FIG. 1. Illustration of the ZnMgO epilayer grown on top of the ZnO template.

results indicate that both the built-in strain and the Mg-concentration will influence the binding energy of the donor bound exciton: The exciton binding energy will decrease with increasing Mg-concentration and with increasing built-in strain.

EXPERIMENTS AND SAMPLES

An advanced AIXTRON closed coupled showerhead (CCS) MOCVD system was used to grow ZnO and Zn_{1-x}Mg_xO epilayers on 2 in. sapphire wafers in our work. Prior to any layer deposition, a 2 in. sapphire wafer was annealed at 1100 °C for 5 min in a N₂ ambient. Subsequently, a 300 nm low growth temperature ZnO buffer layer was first deposited on the substrate at a temperature of 490 °C, and

after that a 500 nm ZnO layer at 950 °C. Subsequently, the temperature was lowered to the growth temperature, T (between 700 and 950 °C), for the Zn_{1-x}Mg_xO epilayer with a thickness of 220 nm (see Fig. 1)

For all layer depositions, diethyl zinc (DEZn, (C₂H₅)₂Zn), bis-cyclopentadienyl magnesium (Cp₂Mg), and N₂O were used as the Zn, Mg, and O precursors, respectively (except for the low growth temperature ZnO buffer layer, where instead tBuOH was used as O precursor), for which high-purity nitrogen gas (at a flow rate of 9 l/s) was employed as carrier gas. During the layer growth, the morphology evolution of the film surface was monitored by an *in situ* reflectance monitor system that comprises a modulated 635 nm diode laser beam. The typical growth rate was about 3–8 nm/min. XPS was used to investigate the Mg composition in the Zn_{1-x}Mg_xO layers, since the x-ray diffraction measurements were not able to determine the Mg concentration for low levels than 6% in the Zn_{1-x}Mg_xO epilayers. XPS measurements were performed using a Scienta ESCA200 spectrometer in ultra-high vacuum (UHV), with a base pressure of 10⁻¹⁰ mbar. The measurement chamber is equipped with monochromatic Al (Kα) X-rays in such a way that the full width at half maximum (FWHM) of the clean Au 4f_{7/2} line was 0.65 eV. All spectra were measured at room temperature with a photoelectron take-off angle of 0° (normal emission). The XPS spectra from all samples are shown in Fig. 2. The concentration of Mg was deduced according to the spectral weight ratio between Mg 2p and Zn 3p after the calibration taking into account of the corresponding atomic sensitivity factors. The gained information on the samples employed in this investigation is summarized in Table I.

PL measurements were carried out in a variable temperature cryostat, in which the temperature could be continuously regulated from 1.8 K up to room temperature. A monochromator HR460 (Jobin Yvon-SPEX) equipped with a

FIG. 2. XPS spectra from different ZnMgO epilayers.

TABLE I. Sample information on the $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers used in this study. The donor ionization energy E_{i0} is estimated according to $E_B/E_{i0} = 0.3$ (Ref. 27) by using the measured DX binding energy E_B .

	Growth temperature T ($^{\circ}\text{C}$)	Deduced Mg concentration (x) (%)	Deduced DX binding energy (meV)	DX energy (eV)	Estimated E_{i0} (meV)
Z05	950	0.38		3.36810	
Z08	900	0.55	16.61	3.37525	55.4
Z07	850	0.84	17.13	3.38023	57.1
Z04	800	1.30	15.24	3.38742	50.8
Z06	750	1.86	13.65	3.40138	45.5
Z03	700	3.01	11.76	3.40987	39.2
Z11	750	1.10	14.76	3.38277	49.2
Z10	750	2.80	13.08	3.41519	43.6

CCD detector (Spectrum One) was used to disperse and detect the emissions from the epilayers. A frequency-doubled Ar-ion laser with a wavelength of 266 nm was used as the excitation source for the normal PL measurements, while a frequency doubled/tripled sapphire:Ti laser (with an operation frequency of 76 MHz) with continuously tunable wave lengths between 230 nm and 400 nm was used for the selective PL (SPL) measurements.

RESULTS AND DISCUSSION

In order to achieve high quality $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayers with the lowest possible background impurity concentration, we have optimized the growth conditions by using two ZnO buffer layers as a template and low growth rate. Fig. 3 shows PL spectra for various $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ samples (as listed in Table I). These PL spectra show that typically three PL peaks near the bandgap are predominant for all Mg-concentrations. The PL peak at 368.76 nm can be identified as the DX in the ZnO layer. The donor, which binds an electron-hole pair (to form the DX), can be assigned as aluminum (Al) impurity based on the energy position and the exciton binding energy (the energy separation between the free exciton and the DX). Since sapphire wafers have been employed for the ZnO and $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ epilayer growth, it is reasonable that Al is the predominant donor impurity. The two other peaks in the PL spectra can be interpreted as the FX and DX in the $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ band edge emission region. These peaks exhibit a blue-shift with increasing Mg-concentration, as expected for the excitons in the $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ layer. There will be an additional experimental outcome expounded below to support the interpretation of the FX and DX transitions as being originating from the free exciton and the donor bound exciton in the $\text{Zn}_{1-x}\text{Mg}_x\text{O}$ layer, respectively. Fig. 3(a) shows PL spectra of samples with varying Mg-concentrations (0.38%, 0.84%, and 1.86%), which were grown at different temperatures (950 $^{\circ}\text{C}$, 850 $^{\circ}\text{C}$, and 750 $^{\circ}\text{C}$, respectively). On the other hand, Fig. 3(b) shows PL spectra of samples with altered Mg-concentrations (1.1%, 1.86%, and 2.8%) but grown at the same temperature, 750 $^{\circ}\text{C}$. These results imply that the FX emission gains PL intensity relative to the DX emission as the Mg-concentration increases.^{28,29}

In order to draw conclusions on the origin of the FX and DX in the ZnMgO band edge emission region, Figure 4

FIG. 3. PL spectra from various ZnMgO epilayers, measured at 5 K with a laser excitation of 266 nm with an excitation power of 1 mW.

shows the PL and SPL spectra of the sample with a Mg-concentration of 0.84% (Z07). With the excitation resonant with either the DX peak in ZnO or the DX in ZnMgO , we can clearly identify the LO-phonon-replica related to the donor bound exciton in both ZnO and ZnMgO . The energy separation between the two LO-phonon-replicas is exactly the same energy separation as between the DX transitions in the ZnO and ZnMgO layers. With above bandgap excitation, the PL shows a similar structure although different intensity levels as the PL spectrum with excitation resonant with the DX transition, as shown in Fig. 4. Furthermore, the intensity of the LO-phonon-replica from both ZnO and ZnMgO epilayers shows a similar dependence on the excitation energy, i.e., when the excitation energy is resonant with the DX transition, the corresponding LO-phonon intensity is enhanced in comparison with the non-resonant case. These facts support the interpretation of the DX transition in ZnMgO as being the donor bound exciton. To find further support that the FX and the DX peaks in ZnMgO layer really are originating from the free exciton and the donor bound exciton, the temperature dependence of the FX and DX peaks in PL spectra is performed. As shown in Fig. 5, the FX in the ZnMgO layer becomes predominant at temperatures above 70 K. Such temperature dependence is consistent with the interpretation of the FX and DX transitions as being originating from the free exciton and the donor bound exciton. The similar temperature dependence has been reported earlier³¹ in ZnMgO epilayer.

FIG. 4. PL (black line) and SPL (color lines) spectra, measured at 5 K. The PL spectrum was measured with a laser excitation at 266 nm, while the SPL spectra were measured with a resonant excitation at wave lengths as indicated by vertical arrows in the figure.

A common method to determine the ionization energy of shallow impurities is to measure the free to bound transition using PL spectroscopy or to determine excited states using selective PL spectroscopy. However, these methods cannot be applied in our case, since the free to bound transition and the excited states cannot be detected even in our high quality ZnMgO epilayers (see Figs. 3 and 4) due to strong emission from ZnO layer. We can roughly estimate the ionization energy of donors by application of Haynes' rule, i.e., the correlation observed between the ionization energy (E_{io}) of the shallow impurity and the binding energy (E_B) of the exciton localized by the same shallow impurity. Meyer *et al.* have shown that the ratio E_B/E_{io} in ZnO for excitons bound to ionized donors, neutral donors, and acceptors is about 0.06–0.3, 0.3, and 0.1–0.15, respectively.²⁷ Consequently, if we know the tendency of E_B for the bound excitons in our ZnMgO epilayers, we can apply the Haynes' rule to gain information on the dependence of the ionization energy E_{io} on the Mg-concentration in the ZnMgO structure, since they can be assumed to vary correspondingly. The estimated ionization

FIG. 5. Temperature dependence of the PL spectra of the ZnMgO sample with a Mg concentration of 1.86% (sample Z06), measured with an excitation wavelength of 350 nm.

energies were also summarized in Table I for various ZnMgO epilayers.

In Fig. 6, the binding energies of the donor bound excitons versus the Mg-concentration and the donor bound exciton energies are summarized. Since there is a lattice mismatch between ZnO and MgO, the built-in strain in thin ZnMgO epilayers cannot be avoided. In addition, ZnMgO epilayers with the same Mg concentration, but grown at different temperatures, have different built-in strain, with a smaller built-in strain for structures grown at higher temperatures due to strong Mg-diffusion into the buffer ZnO layer.³⁰ In order to get an idea of both the built-in strain and the Mg-concentration effects, we have investigated two sets of ZnMgO epilayer samples. For one set, the Mg-concentration was changed, while the growth temperature was kept constant. For the other set, both the Mg-concentration and the growth temperature were varied. Fig. 6 clearly illustrates the effect of the built-in strain and the Mg-concentration on the exciton binding energy. By comparing these experimental results shown in Fig. 6, the following observations can be made; the donor exciton binding energy (or donor ionization energy) decreases with (1) increasing Mg-concentration and (2) increasing built-in strain. We would like to point out that the similar investigations were reported earlier by Neumann *et al.*³¹ Their samples were grown by using plasma-assisted molecular beam epitaxy and using different buffer layers and growth temperatures.

FIG. 6. The exciton binding energy versus (a) the Mg-concentration and (b) the transition energy of the DX.

They find that the binding energy of line I₀ (donor related bound exciton) is unchanged with increased Mg-concentration in ZnMgO layer.

In summary, we have grown and investigated high quality ZnMgO epilayers. Both the free exciton and donor bound exciton were observed simultaneously in the photoluminescence spectra of the high quality Zn_{1-x}Mg_xO epilayers. The results indicate that both the built-in strain and the Mg-concentration influence the donor exciton binding energy. It clearly shows that the donor exciton binding energy decreases with increasing Mg-concentration and with increasing built-in strain. Furthermore, the results indicate that the donor bound exciton transition energy increases with a decreasing strength of the built-in strain if the Mg-concentration is kept the same in the Zn_{1-x}Mg_xO epilayers. Our results imply that the decrease of the conductivity in ZnMgO epilayer is not due to the fact that the electronic structure of the shallow donors is changed. Our results also indicate that the background n-type doping concentration decreases in ZnMgO epilayers in comparison with the corresponding ZnO layers.

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