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Influence of Codoping with Ga on the Electrical and Optical Properties of N-Doped ZnO Films

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This study reports on the dependence on Ga concentration of the electrical and optical properties of ZnO films doped at a N concentration of  $10^{18}$  cm<sup>-3</sup>. For Ga concentrations below 0.5 mol %, the N concentration was kept at  $10^{18}$  cm<sup>-3</sup>. However, as the Ga concentration increased to 5 mol %, the N concentration suddenly increased from  $10^{18}$  to  $10^{19}$  cm<sup>-3</sup>. Furthermore, the donor-acceptor pair emission and yellow luminescence in N single-doped ZnO films were suppressed significantly by doping with Ga at a concentration of 0.1 mol %. However, doping with Ga concentrations above 0.5 mol % resulted in increased red luminescence originating from deep levels generated through the excessive incorporation of Ga atoms. An evaluation of the crystalline quality and electrical properties indicated that the Ga atoms located on the Zn sites changed largely from shallow donor sources to compensating defects trapping electrons at Ga concentrations in the 0.5 to 5 mol % range. These results suggested that the increased N concentration and the enhancement of ultraviolet (UV) emission could be attributed to the role played by the Ga atoms located on the Zn sites in the N single-doped ZnO films.

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Zinc oxide (ZnO) is widely known as a wide-gap semiconductor that can display novel ferroelectric<sup>1</sup> and magnetic properties<sup>2,3</sup> because it is possible to incorporate a wide variety of monovalent, divalent, and trivalent ions into the lattice. Additionally, ZnO has a large exciton binding energy (60 meV), an attractive feature that can be used to facilitate the design of novel optical devices utilizing  $\operatorname{excitons.}^{4,5} A$  variety of dopants and related optical properties for ZnO have been anticipated for use in new technological applications including the use of (Zn,Mg)O and (Zn,Cd)O alloys in the field of bandgap engineering.<sup>6,7</sup> Accordingly, great importance is placed on controlling the electrical activity and optical properties of ZnO using various dopant types and a variety of doping methods such as chemical doping, ion implantation, and plasma irradiation. In particular, enhancement of the optical properties of H<sub>2</sub>-plasma irradiated ZnO single crystals is a major area of study concerned with the control of deep levels.8

We found recently that the donor-acceptor pair (DAP) emission in the UV region, and the yellow luminescence of N single-doped ZnO (ZnO:N) films were suppressed significantly by doping with Ga at a concentration of 0.1 mol %. The suppression of deep-level emission through the use of codoping has been observed for various wide-gap semiconductors. Leem et al. reported that the reduction in yellow luminescence of GaN:Mg, Si films was attributed to a charge compensation between Mg<sup>2+</sup> and Ga vacancies.<sup>9</sup> Watanabe et al. found that a complex of Na and O affected the suppression of deeplevel emission in CuInS<sub>2</sub> films.<sup>10</sup> Codoping methods using two different atoms for wide-gap semiconductors have been employed in an effort to minimize the formation of compensating native defects and to increase the solubility limits of either dopant, an approach that has been strongly supported by first-principle computer stimulations.<sup>11,12</sup> For example, it is well known that the high hole concentration in ZnSe films codoped with Li and N is due to the increased N concentration resulting from the simultaneous incorporation of Li atoms.<sup>13</sup> We have also observed an increased N concentration at a Ga concentration of 5 mol % in Ga and N codoped ZnO (ZnO:Ga, N) films.<sup>14</sup>

This paper reports on the interesting finding that the increase in N concentration and the suppressive effect on luminescence from DAP transitions and deep levels are controlled by the doping concentration of Ga in the ZnO:Ga, N films. This study provides new information toward our understanding of the codoping characteristics of ZnO.

### Experimental

All ZnO films used in this work were grown by pulse laser deposition (PLD) at a base pressure of  $2 \times 10^{-7}$  mbar. An ArF excimer laser ( $\lambda = 193$  nm, 20 ns, and 3 Hz) with an energy density of 1 J/cm<sup>2</sup> was focused on a ZnO single crystal (Eagle-Picher: 6 N), and Ga<sub>x</sub>-doped ZnO ( $0 \leq x \leq 5 \mod \%$ ) ceramic targets were fabricated using a conventional solid-state reaction method. The target-to-substrate distance was fixed at 4.5 cm. The growth rate and substrate temperature were kept at 10 Å/min and 400°C, respectively. Glass (Corning no. 7059) was chosen as the substrate. The film thickness was approximately 300 nm as measured by a surface profiler (Alpha-step 500). Nitrogen and oxygen were supplied simultaneously by a radio frequency (rf) radical source (at 13.56 MHz) with a rf power of 250 W through the cracking of high purity N<sub>2</sub>O gas (6 N). The operating conditions of the rf radical source were fixed in all experiments.

The chemical species generated in the rf plasma cell were characterized using optical emission spectroscopy. A typical optical emission spectrum of the N<sub>2</sub>O plasma with an applied rf power of 250 W and a pressure of  $1.0 \times 10^{-3}$  mbar is shown in Fig. 1. The spectrum shows three types of emissions originating from the  $\gamma$ -transition ( $A^2\Sigma^+$ - $X^2\Pi$ ) of the excited NO molecule (NO\*) and the second positive ( $C^3\Pi_u$ - $B^3\Pi_g$ ) and first positive ( $B^3\Pi_g$ - $A^3\Sigma_u^+$ ) transitions of the excited N<sub>2</sub> molecule (N<sub>2</sub><sup>\*</sup>).<sup>15,16</sup> Oxygen was supplied by the rf plasma cell as O<sub>2</sub> molecules in an unexcited state. This was confirmed by quadrupole mass spectroscopy.

The crystalline quality of the films was characterized by X-ray diffraction (XRD) using CuK $\alpha$  radiation (RINT-2000, Rigaku). Optical properties were measured by photoluminescence (PL) spectroscopy using a He-Cd laser (325 nm) excitation source with a light intensity of 20 mW/cm<sup>2</sup> and 1.5 mm slit width. The incorporation of N and Ga atoms into the ZnO films was estimated by secondary ions mass spectroscopy (SIMS). All SIMS results were calibrated by the simultaneous measurement of N and Ga ions implanted into a ZnO single crystal. Electrical properties were measured at room temperature using the Van der Pauw method.

#### **Results and Discussion**

Figure 2 shows the relationship between the Ga and N concentrations for the ZnO:Ga, N films. The Ga concentration doped into the films agreed well with the Ga<sub>2</sub>O<sub>3</sub> content of the ablation targets. The initial concentration of N atoms incorporated into the ZnO: N films under the N<sub>2</sub>O plasma conditions and growth temperature used in this work was approximately  $10^{18}$  cm<sup>-3</sup>. The N concentration did not change at Ga concentrations below 0.5 mol %, but suddenly increased from  $10^{18}$  to  $2.2 \times 10^{19}$  cm<sup>-3</sup> with increasing Ga concent

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Figure 1. Optical emission spectrum from rf excited plasma of N2O.

trations up to 5 mol %. This result indicates that the incorporation of N atoms into ZnO is dependent on the Ga doping concentration. The increase in N concentration through the use of a codoping strategy is also suggested theoretically by first-principle calculations.<sup>11</sup>

Figure 3 details the dependence of electrical properties on Ga concentration for the ZnO:Ga, N films. Although the ZnO:N films displayed high resistivity above  $10^3 \Omega/cm$ , the free electron density increased to  $1.6 \times 10^{19} \text{ cm}^{-3}$  with increasing Ga concentration due to the increased number of Ga atoms acting as shallow donor sources. The origin of the high resistivity in the ZnO:N films may be attributed to the deep levels related with the yellow luminescence around 2.2 eV for the PL spectra shown in Fig. 4b. These are discussed later. The free electron density, on the other hand, decreased in the ZnO:Ga, N film doped with a Ga concentration of 5 mol %. Some of the Ga atoms doped into the ZnO created lattice defects



Figure 2. Relationship between Ga and N concentration for ZnO:Ga, N films.



**Figure 3.** Dependence of Ga concentration on the electrical properties of ZnO:Ga, N films. Black and white circles indicate resistivity and free electron density, respectively.

involving vacancies and their complexes. Wang *et al.* proposed that complexes such as  $(Ga_{Zn}-O_{interstitial})$  and  $(Ga_{Zn}-V_{Zn})$  must be formed in highly Ga-doped ZnO.<sup>17</sup> In the case of Ga-doped ZnSe, complexes such as  $(Ga_{Zn}-V_{Se})$  and  $(Ga_{interstitial}-V_{Zn})$  have been found and are known to act as compensating native defects, as determined from the results of ion channeling measurements and positron beam analysis.<sup>18,19</sup> Therefore, it is considered that the decrease in free electron density at a Ga concentration of 5 mol % is due to the presence of vacancies and complexes created by the excessive incorporation of Ga atoms into ZnO.

Figure 5 shows the dependence of Ga concentration on the full width half maximum (fwhm) value for the  $\omega$ -rocking curves of the (0002) diffraction peak of the ZnO:Ga, N films. A typical XRD pattern for the ZnO films is shown in the inset of Fig. 5. The XRD pattern contains only one strong peak at the (0002) plane of ZnO, indicating that the films were highly c axis oriented and that second phases such as ZnGa<sub>2</sub>O<sub>4</sub> and GaN were not observed within the measurement limits. The fwhm value underwent large changes with increasing Ga concentration, while the smallest fwhm value was obtained in the ZnO:Ga, N film doped with Ga at a concentration of 0.1 mol %. Broadening of the fwhm value for the  $\omega$ -rocking curve is generally observed in samples doped with impurities and may result from the increased local strain around the impurities or from lattice defects generated by the impurities.<sup>20</sup> Thus, doping with Ga at a concentration of 0.1 mol % should lead to a reduction in the formation of local strain and lattice defects in the ZnO:N films. On the other hand, the fwhm value broadened significantly at a Ga concentration of 5 mol %, due to the formation of vacancies and complexes resulting in a decrease in the free electron density. Additionally, the N concentration remained almost constant up to a Ga concentration of 5 mol %, at which point vacancies and complexes were formed through the excessive incorporation of Ga atoms. Thus, it is suggested that the increased N concentration is closely related to the presence of vacancies and complexes created by the excessive incorporation of Ga atoms. Recently, Nakahara et al. reported that ZnO:Ga, N films showed a remarkable increase in the N concentration under excessive supplement conditions of Ga atoms at 8  $\times 10^{20}$  cm<sup>-3</sup>.<sup>21</sup> Their report and our work indicate that the increase in N concentration is an essential phenomenon that is dependent on



Figure 4. PL spectra for ZnO:Ga, N films at (a) room temperature and (b) 77 K.

neither the substrate type nor the thin film fabrication technique used.

Figure 4a and b show the PL spectra at room temperature and 77 K, respectively, for the ZnO:Ga, N films doped with various Ga concentrations. All PL spectra were normalized against the maximum emission peak in order to visually compare the relative intensity ratio of the UV and deep-level emissions. In the case of the ZnO:N films, the UV emission at 77 K can be divided further into the near band-edge emission ( $I_{\rm NBE}$ ) at 3.350 eV and the broad peak at 3.245 eV. The temperature dependence of each emission peak in the temperature range from 10 K to room temperature was investigated in an effort to clarify the origin of the  $I_{\rm NBE}$  at 3.350 eV and the

broad peak at 3.245 eV (Fig. 6). The  $I_{\text{NBE}}$  shifted to a lower energy from 3.356 to 3.246 eV with increasing temperature. This shift occurred as a result of the decrease in bandgap with increasing temperature, indicating that the  $I_{\text{NBE}}$  is produced by the recombination of excitons. On the other hand, the broad peak at 3.245 eV with the longitudinal optical (LO) phonon replicas of 3.171 eV at intervals of 74 meV moved to slightly higher energies, followed by the complete



**Figure 5.** Dependence of Ga concentration on the fwhm value for X-ray  $\omega$ -rocking curves of the (0002) peak of ZnO:Ga, N films. The insert figure is a typical XRD pattern for ZnO:Ga, N films.



**Figure 6.** Temperature dependence of the near band-edge emission  $(I_{\text{NBE}})$  and DAP emission in the temperature range from 10 K to room temperature. The insert details the PL spectra at 10 K in the ultraviolet region for ZnO:N films grown on a ZnO (a) single crystal and (b) on a glass substrates.

disappearance of the emission peak above 190 K. The inset in Fig. 6 shows that these emission peaks at 10 K were similar to the DAP emission with clear LO phonon series of ZnO homoepitaxial film on a ZnO substrate doped with a N concentration of 1.0  $\times$  10<sup>19</sup> cm<sup>-3</sup>. Thonke *et al.* reported recently that the zero phonon peak of the DAP emission for a ZnO single crystal containing few N atoms (2 ppm) was located at 3.220 eV at 5 K.<sup>22</sup> These results indicate that acceptor levels are formed in the ZnO:N films grown on the glass, although the detailed mechanism of the DAP transition requires further investigation.

It should be noted that the phenomenon of deep-level emission for ZnO is quite complex and our understanding remains controversial. It has been suggested that the blue and green emissions located at around 2.8 and 2.4 eV are related to Zn vacancies<sup>23</sup> and Cu impurities,<sup>24</sup> respectively. The PL intensity and the fwhm value of the emission peak of the yellow luminescence in the ZnO:N film, shown in Fig. 4b gradually increased and narrowed, respectively, with decreasing temperature. This behavior is similar to that of the localized luminescence centers observed in ZnS and ZnSe films.<sup>25,26</sup> Iwata et al. also reported that ZnO:N films showed yellow luminescence at around 2.2 eV at low temperatures.<sup>27</sup> The localized luminescence is produced by an internal transition of the localized centers created by the trapping of electrons at anion vacancies. The luminescence efficiency always decreases at room temperature since the probability of nonradiative transitions is higher than that of radiative transitions with increasing temperature. We consider that the intense vellow luminescence at 77 K in the ZnO:N film may be associated with an increase of intrinsic defects in ZnO due to the incorporation of N atoms.

The optical properties of ZnO:N films were enhanced greatly by doping with Ga. In Fig. 4a and b, the DAP emission and yellow luminescence were suppressed significantly at both room temperature and 77 K as the Ga concentration increased up to 0.1 mol %. However, as the Ga concentration exceeded 0.5 mol %, the  $I_{\text{NBE}}$ declined and a red luminescence at around 1.9 eV was observed at room temperature. With a further increase of Ga concentration, the PL spectrum could not be measured due to the very weak emission intensity. The increase of red luminescence along with the reduction of  $I_{\text{NBE}}$  is thought to be the result of luminescence produced by deep levels related to excessive incorporation of Ga atoms. Moreover, we confirmed that the PL properties depended on the doping concentration of Ga given the increase in the fwhm value for the emission peak of  $I_{\rm NBE}$  with increasing Ga concentration. Broadening of the emission peak by the incorporation of donor impurities is generally attributed to a screening effect of the excitons caused by the random distribution of donor impurities.<sup>28</sup> Accordingly, we conclude that Ga doping at 0.1 mol % assisted in reducing the formation of acceptor levels and localized centers in the ZnO:N films.

Figure 7 shows the dependence of the relative PL intensity ratio of  $I_{\rm NBE}$  to  $I_{\rm DL}$  ( $I_{\rm NBE}/I_{\rm DE}$ ) on the Ga concentration for the ZnO:Ga, N films. As the Ga concentration increased, the  $I_{\rm NBE}/I_{\rm DE}$  ratio gradually increased at both room temperature and 77 K. The maximum  $I_{\rm NBE}/I_{\rm DE}$  ratio was obtained for the ZnO:Ga, N film with a Ga concentration of 0.1 mol %. As shown in the inset of Fig. 7, a  $I_{\rm NBE}/I_{\rm DE}$  ratio as high as 95 was achieved even at room temperature, which is equivalent to the  $I_{\rm NBE}/I_{\rm DE}$  ratio of a high quality ZnO single crystal (Eagle-Picher, 6 N). We then investigated the PL properties of ZnO films codoped with N and other dopants aside from Ga to further clarify the effect of the suppression of the yellow luminescence by codoping. As shown in Fig. 8, suppression effects were also observed in ZnO films doped with Al and N, but they were not seen in ZnO films doped with Li and N. This result confirmed that trivalent ions such as Ga<sup>3+</sup> and Al<sup>3+</sup>, which act as donor sources in ZnO, and have an opposite charge to anions, play an important role in suppressing the DAP emission and the yellow luminescence.

In recent years, many researchers have reported that the suppression of deep-level emission and the increase in free electron density were observed at the same time in  $H_2$ -plasma irradiated ZnO.<sup>8,29-31</sup>



**Figure 7.** Dependence of Ga concentration on the PL intensity ratio of  $I_{\text{NBE}}$  to  $I_{\text{DE}}$  at room temperature and 77 K. The insert shows the PL spectra for ZnO:Ga, N film doped with a Ga concentration of 0.1 mol % on glass (filled line) and the undoped ZnO single crystal (empty circles).

C. G. van de Walle suggested that H atoms could, theoretically, form shallow donor levels in ZnO and thus introduce large distortions of the tetrahedral structure centered around the Zn ions due to the formation of strong H-O bonds.<sup>32</sup> Ohashi et al. proposed that the suppression of deep-level emission by H2-plasma irradiation arises from a charge compensation between H ions and lattice defects or residual impurities.<sup>33</sup> We measured the *d*-value of the (0002)-plane for the ZnO:Ga, N films using a standard Si substrate. In the ZnO:N films the d-value was 2.607 Å, which resembles the d-value of nondoped ZnO films grown in an O2/O3 atmosphere. On the other hand, ZnO:Ga, N films doped with Ga concentrations of 0.1 and 5 mol % displayed d-values of 2.612 and 2.616 Å, respectively, indicating that the length of the c axis increased despite that the ionic radius of  $Ga^{3+}$  ions (0.61 Å) is smaller than that of  $Zn^{2+}$  ions (0.72 Å). Furthermore, lattice distortion resulting from the doping of Ga into ZnO has been verified by nuclear magnetic resonance measurements for ZnO:Ga and ZnO:Al powder samples.<sup>34</sup> Therefore, the increase in length of the c axis must be related to the local lattice distortion due to the replacement of Zn ions by Ga ions. We propose that suppression of the DAP emission and yellow luminescence in ZnO:Ga, N films are closely related to the mechanism involved in the enhancement of UV emission of H<sub>2</sub>-plasma irradiated ZnO.

## Conclusions

The codoping characteristics of ZnO were demonstrated using various ratios of Ga and N. The increase in N concentration observed in the highly doped region at Ga concentration of 5 mol % was attributed to the formation of vacancies and complexes associated with compensating centers of electrons. On the other hand, the N concentration remained almost unchanged in the doped regions consisting of Ga concentrations below 0.5 mol %, where Ga can act as a shallow donor source at Zn sites. Furthermore, a significant



Figure 8. PL spectra at 77 K for ZnO: N film and ZnO films codoped with N and  $M_e$  ( $M_e = Ga$ , Al, and Li).

suppressive effect in the ZnO:N film was found when using a Ga concentration of 0.1 mol %. A suppressive effect was also observed in ZnO films codoped with Al and N, indicating that trivalent ions play an important role in suppressing the formation of acceptor levels and localized centers in the ZnO:N films. Finally, the codoping of Ga into N single-doped ZnO that led to an enhancement in UV emission and the formation of shallow donor levels was similar to the electrical and optical behavior observed with H-doped ZnO.

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