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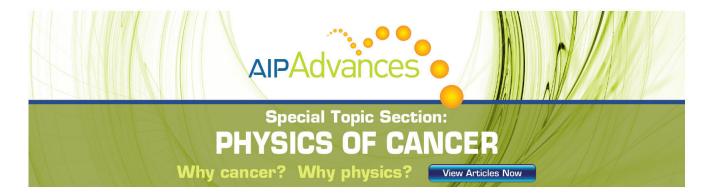
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Nitrogen-related recombination mechanisms in *p*-type ZnO films grown by plasma-assisted molecular beam epitaxy

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The recombination mechanisms of nitrogen-related emissions in *p*-type ZnO films were investigated by photoluminescence (PL) measurements. The enhanced peak at 3.260 eV was confirmed as a donor-acceptor pair (DAP) transition and the emission around 3.310 eV was assigned to the free electron to acceptor (FA) recombination by studying the temperature evolution of DAP and FA luminescence, calculating the energy level of the corresponding nitrogen acceptor, and measuring the decay time of DAP transition. By comparing the PL spectra of the samples with various hole concentrations, it can be found that as the hole concentration increases, the DAP peak significantly dominates the spectra. In addition, the acceptor binding energy is estimated to be about 120 meV from the FA transition, which is in good agreement with the value calculated by a hydrogenic acceptor model. © 2007 American Institute of Physics. [DOI: 10.1063/1.2772581]

I. INTRODUCTION

With a wide band gap of 3.370 eV and a large exciton binding energy of 60 meV at room temperature, ZnO has attracted considerable attention as a promising material for optoelectronic devices, especially in short-wavelength light emitting diodes (LEDs) and laser diodes durable at hightemperature operation. However, it is well known that the fabrication of p-type ZnO is rather difficult because of a strong self-compensation effect arising from the presence of native defects² or hydrogen impurities.³ Nitrogen (N) has been proved to be the most promising dopant for the growth of p-type ZnO, especially since the observation of the electroluminescence (EL) from the ZnO homojunction. 4 We have also obtained the reproducible N-doped p-type ZnO films grown by plasma-assisted molecular beam epitaxy (P-MBE) on sapphire using radical NO as oxygen and nitrogen sources, ⁵ leading to the observation of blue-violet EL from a homojunction LED.⁶ Recently, other groups have also obtained the EL from ZnO homojunctions using N as p-type dopant.^{7,8} However, these EL spectra are always dominated by the defect-related emissions, 4,6-8 due to the poor quality of p-type ZnO layer of the LEDs. Therefore, for improving its quality and further obtaining exciton-related EL emission

from ZnO homojunction LEDs, it is very important to understand the recombination mechanisms of N-related emissions in p-type ZnO films. The possible emissions associated with N acceptors are attributed to donor-acceptor pair (DAP), neutral acceptor-bound exciton (A^0X) , and free electron to acceptor (FA) transitions. Many works have concentrated on this issue, but controversies on basic optical properties associated acceptors still remain. First, the DAP emissions, as a direct probe of the acceptors in ZnO, have been observed by many groups. 9-14 However, according to their reports, the observed DAP peaks varied from 3.160 to 3.270 eV, with various acceptor energies such as 266, 209±3, 10 195±10, 11 $177,^{12}$ $165 \pm 40,^{13}$ and 135 meV. ¹⁴ Second, the emission near 3.310 eV has been observed by several groups and attributed to different recombination processes such as A^0X , 15,16 DAP, ^{17,18} and FA transitions. ^{19,20} Obviously, the origin of this line is still being argued. Thus, a systematic investigation is required to clarify the acceptor-related recombination properties in p-type ZnO.

In this work, the luminescence properties in N-doped *p*-type ZnO films have been investigated by PL measurements. The enhanced peak at 3.260 eV is confirmed as a DAP transition and the emission around 3.310 eV is assigned to the FA recombination by studying the temperature evolution of DAP and FA luminescence, calculating the energy level of the corresponding nitrogen acceptor, and measuring the decay time of DAP transition. Especially, the possible

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TABLE I. Electrical properties at room temperature of N-doped p-type ZnO films grown by different NO flow rates.

Sample	NO flow rate (ml/min)	Carrier concentration (cm ⁻³)	•	Carrier type
A	0.2	6.88×10^{16}	0.96	p
B	0.5	4.35×10^{17}	0.22	p
C	1.0	1.22×10^{18}	0.45	p

recombination mechanism of the emission near 3.310 eV is discussed. It is strongly suggested that this peak in N-doped p-type ZnO should originate from the FA transition.

II. EXPERIMENTS

The epitaxial ZnO films were grown on c-plane sapphire (c-Al₂O₃) by P-MBE. The detailed cleaning process of the substrate was described in the previous work.⁵ Pure zinc (99.999%) was supplied by means of a Knudsen effusion cell. For the growth of undoped ZnO films, research grade O₂ was introduced into an Oxford Applied Research Model HD25 rf (13.56 MHz) plasma source. For the growth of N-doped ZnO films, Oxygen and nitrogen were obtained from the NO with 99.999% purity, flowing through the same plasma source, operated at a power of 300 W. During growth, Zn beam partial pressure and substrate temperature were fixed at 2.5×10^{-4} Pa, and 425 °C, respectively. The NO flow rate for the N-doped samples can be found in Table I. The thicknesses of the N-doped samples measured by ellipsometry were varied from 560 to 850 nm.

X-ray diffraction (XRD) was carried out on a Rigaku O/max-RA x-ray system using Cu $K\alpha$ ($\lambda = 1.5418$ Å). X-ray photoelectron spectroscopy (XPS) was measured by VGES-CALAB MK II XPS system (VG Co., East Grinstead, U.K.). Hall-effect measurements were carried out for the p-type ZnO films in van de Pauw configuration by Lakeshore's 7707 Hall measurement system. The Ohmic contacts for electrical measurement were made by vacuum-sintering $(\le 5 \times 10^{-3} \text{ Pa})$ small indium dots at the four corners of square samples. PL spectra were measured employing 325 nm He-Cd laser as the excitation source and collected with a microprobe system made by Jobin-Yvon Co. in France. The decay curve of PL was measured by using the timecorrelated single-photon counting technique with FL920fluorescence lifetime spectrometer.

III. RESULTS AND DISCUSSIONS

Figure 1 shows XRD θ -2 θ patterns of the three N-doped ZnO films with various NO flow rates, which are summarized in Table I. Besides the diffraction peak of $Al_2O_3(006)$, only ZnO (002) peak can be observed at 34.44° for all samples, indicating that the N-doped ZnO films have a high c-axis orientation with a wurtzite structure. To identify the composition and chemical state of the N-doped ZnO films, XPS measurement was conducted after Ar⁺ etching the surface of the film. Figure 2 shows a typical N 1s XPS spectrum of the N-doped ZnO film grown with NO flow rate of 0.5 ml/min. As can be seen, a clear peak centered at 396 eV is observed. It is believed that nitrogen occupies at least two

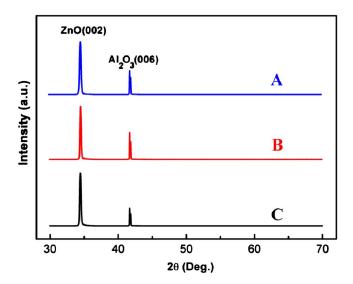


FIG. 1. (Color online) XRD spectra of the N-doped ZnO films grown with NO flow rates of 0.2 (a), 0.5 (b), and 1.0 ml/min (c), respectively.

chemical environments in N-doped ZnO films.²¹ One is formed by substitution of N atoms on O sites to act as acceptors, donated by NO, and the other by substitution of N2 molecules on O sites to act as double donors, donated by (N₂)₀. It has been justified that the binding energy around 396 eV is attributed to the signal of N_O and the peak at 404 eV to the signal of $(N_2)_0$. As shown in Fig. 2, the only peak centered at 396 eV clearly demonstrates that nitrogen is incorporated in the form of N_O, being the desired acceptor, in our N-doped ZnO films.

The electrical results at room temperature for the N-doped ZnO films are summarized in Table I. All the samples exhibit the p-type conduction. It can be seen that by increasing NO flow rate from 0.2 to 1.0 ml/min, the hole from 6.88×10^{16} concentration increases to $\times 10^{18}$ cm⁻³. To further study the properties of these N-doped p-type ZnO films, 80 K PL spectra for the undoped ZnO and sample A are compared in Fig. 3. The spectrum of the undoped ZnO is typically dominated by a free exciton

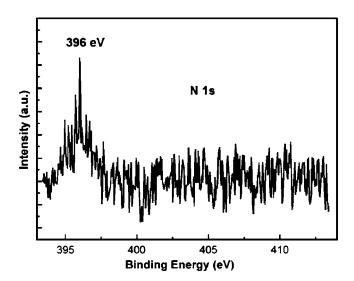


FIG. 2. N 1s XPS spectrum of the N-doped ZnO film grown with NO flow rate of 0.5 ml/min.

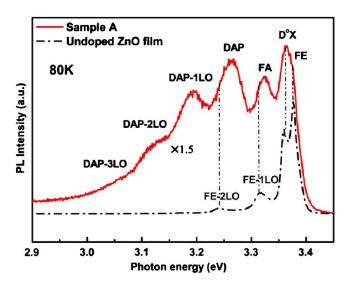


FIG. 3. (Color online) PL spectra of undoped ZnO and N-doped p-type ZnO (sample A) at 80 K.

(FE) peak at 3.377 eV and a neutral donor-bound exciton (D^0X) at 3.363 eV, with two weaker broad lines centered at 3.313 and 3.240 eV which are assigned to the longitudinal optical (LO) phonons, respectively.²² By comparing the undoped and N-doped samples, the most obvious difference is that the latter has a strong line at 3.263 eV, with three gradually weaker broad lines centered at 3.191, 3.119, and 3.047 eV, respectively. Since the emission at 3.263 eV exhibits an obvious blueshift with increasing excitation intensity in our previous study, we attributed it to a DAP transition. Below, it will be discussed in detail. The three shoulders located at lower-energy side of DAP are assigned to DAP-LO, DAP-2LO, and DAP-3LO, respectively, since the energy difference between their peak positions is close to the LO phonon energy of ZnO.²³ The FE and D^0X emissions from the undoped ZnO can also be found at the higher-energy side of emission peaks from sample A.

Significantly, another peak at 3.322 eV should necessarily be considered because it always appears in N-doped samples. (This peak slightly shifts to 3.317 eV for samples B and C, as shown below.) By comparing with the undoped sample, it can be believed that this peak should be associated with the nitrogen acceptors rather than the recombination of FE-LO. Moreover, the line around 3.310 eV always appears in p-type ZnO, not only in N-doped 15 but also in As-doped, 19 and P-doped²⁰ samples. However, the origin of this peak is always controversial up to now. It has been suggested the line at 3.315 eV represents an A^0X recombination in N-doped ZnO films. 15 Indeed this assignment is questionable because of the unusually large binding energy to the acceptor (62 meV), which implies a rather large value of Haynes factor. 17,24 Due to the above difficulty, some authors have suggested that this line is a DAP transition rather than an A^0X transition. ^{17,18} But this assignment also has its difficulties in explaining a much smaller value of nitrogen acceptor energy.²⁴ (The DAP emission at 3.315 eV would imply an acceptor energy of <100 meV.²⁴) In this regard, it should be noted that the lines at 3.322 eV in As-doped p-type ZnO (Ref. 19) and at 3.310 eV in P-doped samples²⁰ have been

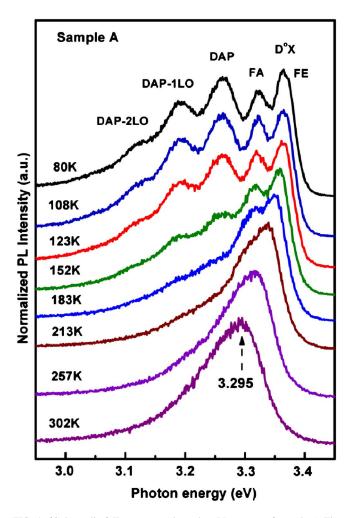


FIG. 4. (Color online) Temperature-dependent PL spectra of sample A. The spectra were normalized and shifted vertically for clarity.

assigned to FA transitions. These identification results are more reasonable, given the difficulties arising from assignments of this emission to A^0X or DAP transition. Then, according to these analyses, the peak at 3.322 eV in sample A is tentatively attributed to a FA transition. A further confirmation of this assignment will be demonstrated below.

It has been demonstrated that the DAP emission in ZnO always quenches quickly and gradually transforms into FA with increasing temperature due to the smaller donor binding energy than acceptor energy. 9-12 Figure 4 shows the temperature-dependent PL spectra of sample A from 80 to 302 K. It is evident that with increasing temperature, the DAP peak quenches rapidly and the FA intensity gradually exceeds the intensity of DAP. To illustrate this, the intensity ratio of FA to DAP (FA/DAP) as a function of temperature is shown in Fig. 5(a). It can be seen that the FA/DAP value increases as the temperature increases until 198 K. This temperature evolution is a typical transformation from DAP to FA emission, when the donors are thermally ionized. At temperatures above 213 K, the FA peak gradually loses its intensity because the acceptors are thermally ionized. Also shown in Fig. 4, the D^0X peak in sample A rapidly quenches and the FE emission dominates the higher-energy side. The broad FE luminescence eventually buries the FA peak when tempera-

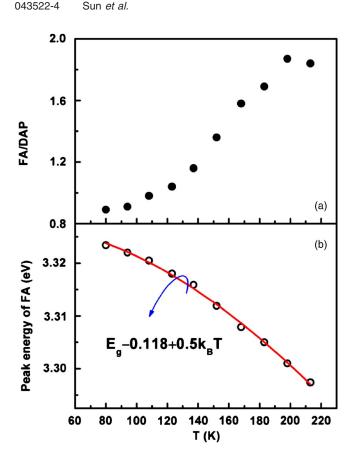


FIG. 5. (Color online) (a) The intensity ratio of FA emission to DAP emission at various temperatures for sample A. (b) Temperature-dependent peak energy of FA and the fitting curve using Eq. (1).

ture goes up, as seen in Fig. 4. At room temperature, the FE emission centered at 3.295 eV dominates the spectrum of sample A.

To further confirm our assignment, the temperaturedependent peak position of the FA emission is analyzed, as shown in Fig. 5(b). It can be seen that the FA transition energy shows a continuous redshift with increasing temperature. The experimental data fit well in an equation for a free electron to acceptor transition given by 10

$$E_{FA}(T) = E_g(T) - E_A + \frac{1}{2}k_B T,$$
 (1)

where $E_g(T)$ and $E_{FA}(T)$ are the temperature-dependent band gap energy and FA energy, respectively, E_A is the acceptor binding energy, k_B is the Boltzmann constant. Here, $E_g(T) = E_g(0) - \alpha T^2 / (T + \beta)$, where α and β are constants and $E_{\varrho}(0)$ is the band gap energy at T=0 K. The acceptor energy level is found to be 118 meV. The fitted values of $E_{\sigma}(0)$, α , and β are equal to 3.440 eV, 8.6×10^{-4} eV/K, and 793 K, respectively, which are in good agreement with those reported by Wang et al. 10 The perfect match between the experimental data and the fitting curve further supports our assignment of the FA transition.

Figure 6 shows the normalized PL spectra at 80 K of the three N-doped p-type ZnO films. For sample A with the hole concentration of 6.88×10^{16} cm⁻³, the intensity of the DAP peak is comparable to the intensity of the D^0X . However, for sample B with the hole concentration of 4.35×10^{17} cm⁻³,

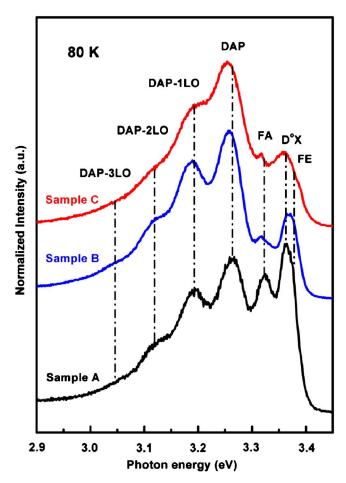


FIG. 6. (Color online) Normalized PL spectra at 80 K for the N-doped p-type ZnO films with various hole concentrations (shown in Table I).

the DAP peak significantly dominates the spectrum and slightly shifts to 3.258 eV. For sample C with the hole concentration of 1.22×10^{18} cm⁻³, the DAP peak is further enhanced and shifts to 3.255 eV. It can be obviously seen that when the hole concentration increases, the DAP peak gradually dominates the spectra and shows a slight redshift. Tamura et al. reported that the DAP peak in N-doped ZnO shifts to lower energy with increasing nitrogen concentration. Moreover, the redshift of the DAP is always observed in p-type ZnSe:N by increasing nitrogen concentration. ^{25,26} Interestingly, as the hole concentration increases, the FA peak also shifts from 3.322 (sample A) to 3.317 eV (samples B and C), almost the same value of redshift as occurred in DAP. This probably indicates that the transitions of DAP and FA are associated with the identical acceptors, i.e., No. On the other hand, the same tendency of redshift for DAP and FA further confirms that our assignments are very reasonable for both of them. Comparing the spectra in Fig. 6 also shows that when the DAP intensity is gradually enhanced the D^0X intensity is correspondingly decreased. In addition, the FE lines in samples B and C are too weak to identify due to the heavy doping of nitrogen. The DAP dominance over the D^0X is expected from the fact that donors are more likely to be close to acceptors due to higher N concentration and thus will contribute to emission through a DAP process rather than an excitonic process. 15

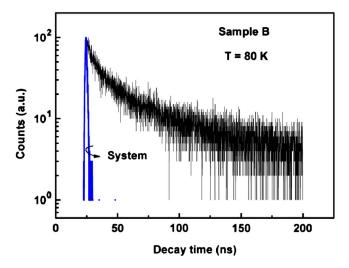


FIG. 7. (Color online) Temporal response of the DAP emission at 80 K for sample B.

As shown in Fig. 6, the appearance of the FA emissions enables us to calculate the acceptor binding energy at 80 K according to Eq. (1). With the FE energy of 3.377 eV and its binding energy of 60 meV, the value of E_A is calculated to be about 120 meV for the N-doped p-type ZnO films, consistent with the value of 165±40 meV reported by Zeuner et al. 13 This is slightly smaller than 140 meV based on the DAP transition in our previous study.²⁷ However, this is possible because the energy position of the DAP can only give a rough estimate of the acceptor binding energy due to the inaccurate determination of the donor to acceptor distance. Thus, it can be concluded that N acts a shallow acceptor in ZnO. So far, several groups have confirmed the N on oxygen sites in ZnO by electron paramagnetic resonance measurements. 18,28 Then, by using a hydrogenic acceptor model, ¹⁹ a value of E_A = 132 meV can be obtained assuming an effective hole mass of $m_p = 0.64m_0$ (m_0 is the free electron mass) and low-frequency dielectric constant $\varepsilon_0 = 8.12^{24}$ This value favorably agrees with that obtained by the PL spectra, implying that N in ZnO behaves as a hydrogenic acceptor. Base on the above analyses, it should be emphasized that the assignment of the emission around 3.310 eV to FA transition here is rather reasonable because it can avoid the difficulties arising from the assignments of this band to DAP or A^0X .

Since the DAP emissions always dominate the PL spectra of the N-doped p-type ZnO films, their dynamical behaviors should be studied to explore a more detailed recombination mechanisms. Figure 7 shows the typical PL temporal response of the DAP band measured at 80 K. It is obvious that the decay curve is nonexponential. The decay kinetics for DAP recombination was thoroughly analyzed by Thomas et al.,²⁹ and this analysis was applied to other materials such as CdS, ²⁹ ZnSe, ³⁰ GaN, ³¹ and ZnO. ¹³ Based on their theory, when an electron bound to a donor recombines with a photon-generated hole bound to an acceptor, the radiative recombination rate W depends exponentially on the separation, R, between the donor and acceptor involved 29

$$W(R) = W_{\text{max}}e^{-(2R/a_D)},\tag{2}$$

where $W_{\rm max}$ is the transition probability in the limit $R\!\to\!0$ and a_D is the Bohr radius for a more weakly bound particle. It is evident from Eq. (2) that the lifetime of the bound hole, $\tau = W^{-1}$, is much longer for distant pairs than for close pairs. This results in an increase of the instantaneous lifetime of the measured PL with the time delay. Thus, DAP emission generally exhibits a nonexponential decay due to the random distribution of donors and acceptors. Here, the nonexponential decay of DAP in Fig. 7 indeed supports our assignment of this band to DAP transition.

IV. CONCLUSIONS

In conclusion, the luminescence properties in N-doped p-type ZnO films have been investigated by PL measurements. The enhanced peak at 3.260 eV is confirmed as a DAP transition and the emission around 3.310 eV is assigned to the FA recombination. The temperature evolution of the spectra shows a gradual transformation from DAP to FA emission. The room temperature spectrum of p-type ZnO is dominated by the broad FE emission with the weak FA transition at its lower energy side. Moreover, it can be believed that the DAP and FA transitions are associated with the identical nitrogen acceptors. The acceptor binding energy is estimated to be about 120 meV from the FA transition, which is in good agreement with the value calculated by a hydrogenic acceptor model.

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