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Photoluminescence study of p-type vs. n-type Ag-doped ZnO films

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Silver doped ZnO films have been grown on sapphire (0001) substrates by pulsed laser deposition. Hall measurements indicate that p-type conductivity is realized for the films deposited at 500 °C and 750 °C. Transmission electron microscopy images show more obvious and higher density of stacking faults (SFs) present in the p-type ZnO films as compared to the n-type films. Top view and cross sectional photoluminescence of the n- and p-type samples revealed free excitonic emission from both films. A peak at 3.314 eV, attributed to SF emission, has been observed only for the n-type sample, while a weak neutral acceptor peak observed at 3.359 eV in the p-type film. The SF emission in the n-type sample suggests localization of acceptor impurities nearby the SFs, while lack of SF emission for the p-type sample indicates the activation of the Ag acceptors in ZnO.

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I. INTRODUCTION

The semiconductor properties of ZnO, including its wide-bandgap (3.37 eV at room temperature) and large exciton binding energy (60 meV), are favorable comparing to current materials used for optoelectronic device applications.^{1–3} Benefit from its outstanding semiconductor properties, ZnO was widely studied in various applications including photoelectrocatalysis,^{4,5} photocatalysis,^{6,7} light emission devices,⁸ ion batteries,⁹ and solar cells.¹⁰ Many of the prospective applications for ZnO require both n- and p-type ZnO. However, p-type ZnO has been proven difficult to realize. A major obstacle towards obtaining stable and reliable p-type conductivity is the limited knowledge on intrinsic and dopant-induced defects in ZnO. It is known that structural, electrical, and optical properties of ZnO films are strongly influenced by deposition parameters, post treatment, and dopant elements.^{11–23} Understanding the doping nature of impurities through investigation of microstructural and optical properties of ZnO is important for overcoming the bottleneck in realizing ZnO-based devices.

Photoluminescence (PL) is a powerful technique for studying the light emitting properties of semiconductors in order to characterize a variety of material parameters. PL emission spectrum provides useful information for identifying defect and impurity levels. Stacking faults (SFs) are one of the main types of extended defects that occur in II-VI and III-V semiconductors.²⁴ Extended defects are known to affect the electronic properties of semiconductors by introducing energy levels in the band gap, which can hinder the quantum efficiencies and lifetime of devices.^{25,26} In particular, both polar (*c*-plane) and non-polar (*a*-plane) GaN films tend to possess a high-density of SFs.^{27–30} Luminescence

peaks in the 3.29–3.42 eV range have been attributed to SF features observed in GaN by transmission electron microscopy.^{31–33} Although SFs are commonly observed in ZnO films as well, the luminescence energy associated with them has not been well correlated.^{24,34,35} The 3.31 eV emission band commonly observed in undoped and doped ZnO has been suggested as being related to SFs,³⁶ but also been reported to be attributed to various electron-acceptor pair and exciton transitions.^{37–40}

Most reports of p-type ZnO have been for group V elements including N,^{41–44} P,^{45,46} As,^{47–49} and Sb.^{50,51} dopants. The low temperature (LT) PL of the p-type samples utilizing these dopants includes emissions in the energy range of 3.3–3.35 eV. These emissions are typically attributed to free electron to neutral acceptor (e, A⁰), neutral acceptor bound exciton (A⁰X), or donor-acceptor pair (DAP) transitions. As a p-type dopant, Ag has been theoretically suggested to be a promising candidate for ZnO.^{52,53} Experimental results show that doping ZnO with Ag can lead to enhanced band edge emission.^{21,54–57} In this work, Ag-doped ZnO (SZO) films were grown by pulsed laser deposition (PLD). The effects of Ag-doping on the electrical, microstructural, and optical properties of ZnO thin films were assessed. In particular, PL spectroscopy was used to determine possible signals of SFs in ZnO, which were observed by transmission electron microscopy (TEM). Comparison between n- and p-type SZO samples was investigated and showed differences in the defect emissions. The role of SFs in the doping process of ZnO:Ag films is discussed.

II. EXPERIMENTS

The ZnO:Ag target was synthesized using high-purity ZnO (99.999%) and Ag₂O (99.99%) powders. ZnO and 1 at. % Ag₂O were mixed and ball-milled for 90 min. After a conventional ceramic press, the target was calcined and sintered

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in flowing oxygen at 500 °C for 5 h and 1100 °C for 1 h, respectively. SZO films were deposited on single-crystal sapphire (0001) substrates by PLD with a KrF excimer laser (Lambda Physik 210, $\lambda = 248$ nm, 10 Hz). Single-layer films were deposited at substrate temperatures of 400 °C, 500 °C, and 750 °C. For all depositions, the laser energy was set at 340 mJ and the base pressure was 1×10^{-6} Torr before oxygen was introduced. The oxygen partial pressure during depositions was controlled at 200 mTorr, 270 mTorr, and 270 mTorr for the 400 °C, 500 °C, and 750 °C depositions, respectively. Post-deposition annealing was performed at 400 °C in an oxygen pressure of 150 Torr for all depositions. Deposition time was controlled to maintain comparable film thickness between samples grown at different substrate temperatures.

Microstructural characterization was performed using TEM (a JEOL2010 analytical electron microscope with a point-to-point resolution of 0.23 nm). Hall measurements were conducted at room temperature (RT) using a commercial physical-property measurement system (Quantum Design PPMS 6000) at a magnetic field of both 1 T and -1 T to account for the possible offset between voltage contacts. Ohmic contacts, as confirmed by PPMS and four-point probe, were pressed indium solder onto 80 nm sputter deposited Au contacts.

The peculiarities of light emission of the samples were studied by micro-photoluminescence setup. Excitation was performed by frequency doubled Nd:YVO laser as continuous wave excitation source, giving a wavelength $\lambda = 266$ nm. The Nd:YVO laser beam was focused by UV lens, providing the excited area around $1.5 \mu\text{m}$ in diameter. The emitted luminescence was collected and mirrored into a single grating 0.45 m monochromator equipped with a liquid nitrogen cooled Si-CCD camera with a spectral resolution of ~ 0.1 meV. Via control of the laser transmittance, the power excitation density was ranged from 0 to 400 W/cm², enabling the power dependent PL study. The low temperature PL study was performed at 4–10 K by helium cooling of the cold-finger where the samples were placed. Via decreasing the liquid He flow and local heating of the sample holder, the temperature dependent PL study was performed for the range from 4 to 300 K. Two types of samples were used for PL analysis: p-type ZnO films and similar n-type ZnO as a reference for comparison. The samples were studied in two different geometries/modes: (i) the surface of the films was irradiated (top-view mode) and the PL signal was collected from it and (ii) the samples were cleaved and both the excitation and signal collection were performed on the cross-sections of the samples (cross-section mode).

III. RESULTS AND DISCUSSION

A. Electrical properties

The electrical properties of the films discussed in this work are shown in Table I. Based on our previous work, the 500 °C and 750 °C samples were deposited under the optimized growth conditions (e.g., laser energy and partial oxygen pressure) for achieving p-type conductivity.⁵⁸ For comparison, the 400 °C sample was deposited under conditions to obtain

TABLE I. Electrical properties of the n-type and p-type SZO films.

Temperature (°C)	Resistivity (Ω cm)	Carrier concentration (cm ⁻³)	Mobility (cm ² /V s)	Carrier type
400	2	7.78×10^{17}	4.03	n
500	0.9	2.3×10^{18}	3.03	p
750	200	5.4×10^{15}	5.74	p

n-type conductivity. The Hall measurements confirmed that the charge carrier type of each sample is consistent with the original designs. As seen in Table I, the carrier concentration decreases and the resistivity increases by three orders of magnitude for the 750 °C sample compared to the 500 °C sample. This may be due to the formation of compensating defects in the 750 °C sample.

B. Microstructural characteristics

To understand the microstructural properties of the SZO films, a detailed comparative cross-sectional TEM study of the 500 °C p-type and 400 °C n-type sample was conducted. Fig. 1 shows the TEM images and the corresponding selected area electron diffraction (SAED) patterns of (a) the 500 °C p-type sample and (b) the 400 °C n-type sample. The SAED patterns indicated epitaxial growth of ZnO in both samples. SFs were observed in both films. As seen in Fig. 1(a), more than 14 clear SFs, indicated by arrows, present throughout this area. In comparison, within the same size of imaging area in the 400 °C n-type sample [Fig. 1(b)], only 7 SFs were observed, and most of those do not show clear strain contrast. The result indicates that the p-type SZO film has a higher SF density and contains larger local strain nearby the SFs. High resolution STEM (HR-STEM) was carried out to further investigate the role of Ag in the SF behavior. Fig. 1(c) shows the HR-STEM image of the 500 °C p-type sample. No obvious clustering of Ag atoms was observed near the SF features, which suggests a uniform distribution of silver in this sample. The SFs formation is a way of relaxing the local strain in the sample, which is not necessarily directly related to the Ag deficient or rich conditions. Therefore, SFs exist in both p- and n-type SZO samples. However, with silver more uniformly doped in the p-type sample, the lattice strain is larger compared to the n-type sample, which leads to a higher SF density and larger strain accommodated by each SF in the p-type sample.

Fig. 1(c) also shows perfect epitaxial growth of the 500 °C p-type sample, which is free from grain boundaries and other obvious growth defects except the SFs. This result is confirmed by the high-resolution TEM (HRTEM) image and inverse fast Fourier transform (IFFT) image in Figs. 2(a) and 2(b). The IFFT in Fig. 2(b) was taken from the HRTEM image in (a), as indicated by boxes, with only the (0002) reflections selected. Three SFs, marked by circles, are shown in the IFFT image. No other obvious defects were observed and the lattice distortion is minimal. Figs. 2(c) and 2(d) show the HRTEM image and the corresponding IFFT image of the 400 °C n-type SZO film. Compared to the p-type sample, more boundaries and local contrast variation are present in

FIG. 1. (a) TEM image of 500 °C p-type sample shows high density of SFs features, indicated by arrows. The SAED pattern in the inset indicates are highly texture growth of the ZnO thin film. (b) TEM image of 400 °C n-type sample shows a lower density of SFs with less strain contrast. The SAED pattern in the inset indicates highly texture growth of the ZnO thin film. (c) HR-STEM image of 500 °C p-type sample shows high epitaxial quality and uniform distribution of Ag in lattice.

the n-type sample. In the IFFT image, only one SF was observed. The contrast variation is mainly caused by local disorder and large lattice distortion. The local disordered and distorted areas in the n-type sample could play as traps for Ag atoms and keep them nonactivated. Thus, it is suggested that higher epitaxial quality with reduced disorder of the film is favorable for enabling p-type conductivity.

C. Top view PL study of n-type vs. p-type SZO films

The top view RT PL results of the p-type films deposited at 500 °C and 750 °C on *c*-cut sapphire are shown in Fig. 3. The intensity of the near-band-edge emission (NBE) peak of the films are comparable; however, the 500 °C sample is located at 372 nm (3.33 eV), while the 750 °C sample is located at 376 nm (3.3 eV). In addition, the deep level emission (~ 500 nm) in the 750 °C sample suggests the presence of defects. It is known that the UV NBE emission (376–380 nm) in ZnO is due to exciton transitions⁵⁹ and the broad deep level emission (500–550 nm) is due to intrinsic structural defects and impurities.⁶⁰ It is worth noting that such deep level emission is not present in the 500 °C sample. Suppression of deep level emission compared to NBE for Ag-doped films has previously been observed and suggests that Ag is not located at interstitial sites or present as antisite defects.⁵⁴ The presence of defects in the 750 °C sample may be the cause for higher resistivity and lower carrier concentration compared to the 500 °C sample (Table I).

In order to further understand the doping nature of the p-type films, the p-type 500 °C sample was compared to the n-type 400 °C sample. The top view LT PL study revealed two spectra shown in Fig. 4, containing peaks both different in spectral location and intensity. The peak of free excitonic (FX) emission was found for both samples with some small difference. The experimental data have been fitted by the Varshni expression⁶¹

$$E_g = E_g(0) - \frac{\alpha T^2}{\beta + T},$$

where $E_g(0)$ is band gap at $T = 0$ K, α is dE_g/dT , and β is a constant correlated with the Debye temperature, θ_D . The values were obtained for FX energies at 0 K: $FX_p = 3.3777$ eV, $FX_n = 3.3792$ eV. Such a difference between two samples may be explained as (i) local/general strain effects in the p-doped ZnO and/or (ii) fitting correctness affected by nearby peak. Additional to FX peaks, one more peak was clearly detected for every spectrum. For n-type ZnO, the peak at 3.3597 eV was attributed to neutral donor bound exciton (D^0X). While for p-type ZnO, the peak observed at 3.3710 eV was attributed as due to ionized donor bound exciton recombination (D^+X). It is likely that the weak evidence of this peak exists even for n-type ZnO sample; however, for p-type doped ZnO this peak is much more stronger (in fact dominant) due to self-compensation effect of the material.

In Fig. 5, the energy positions of the peaks as a function of temperature were fitted by the Varshni expression and the values of $E_g(0)$ are found to be 3.381 eV, 3.379 eV, and 3.371 eV for the n-type FX, p-type FX, and p-type AX peaks, respectively. The fitting results in reasonable fitting parameters. The value of α , calculated to be $9\text{--}12 \times 10^{-4}$ eV/K, agrees well with that reported for free excitons.⁶² The value of β is important due to its relation with θ_D . Until now, this value is reported to be in a wide range between 305 and 900 K. In our case, based on the fitted value β , the reasonable Debye temperature of 750 K was obtained, which agrees well with that for ZnO thin films.⁶³ It is also noted that,

FIG. 2. (a) HRTEM image of the 500 °C p-type sample, (b) an inverse Fourier transform of the boxed area in (a) with only the $\pm(0002)$ reflections selected, (c) HRTEM image of the 400 °C n-type sample, and (d) an inverse Fourier transform of the boxed area in (c) with only the $\pm(0002)$ reflections selected. SFs in (b) and (d) are indicated in the circled areas.

FIG. 3. Room temperature PL of p-type SZO samples deposited at 500 °C and 750 °C on *c*-cut sapphire.

FIG. 4. Low Temperature (10 K) PL spectra, acquired from the top of the n- and p-type SZO samples. The Gaussian multi fit has been applied for peaks spectral location determination (not shown here).

FIG. 5. Peaks energy shift with the temperature for the n- and p-type SZO samples.

compared with the trend of other peaks, the n-type D^0X peak energy varied differently. For this n-type Ag-doped ZnO sample, the donor sources could be un-activated Ag dopants,⁶⁴ Zn interstitials,¹⁴ oxygen vacancies,⁶⁵ and other impurities. In this case, the mixed contributions from various n-type charge carriers lead to the complex D^0X energy trend, i.e., first slightly decrease (10 K–40 K), then increase (40 K–100 K), and finally decrease following the same trends as others (100 K and higher). Thus, the complex energy-temperature trends of the n-type D^0X peak actually reflect the complex nature of the n-type conduction mechanisms in the Ag-doped ZnO film, which has not yet been previously reported.

Evolution of the n-type and p-type SZO samples as a function of increasing temperature is shown in Figs. 6(a) and 6(b). It is noted that the expected SFs related emission does not exist in either the n-type (Fig. 6(a)) or p-type (Fig. 6(b)) SZO samples from the top of the samples. This may be due to several effects: (i) the penetration depth of the laser is rather small (~ 50 up to 100 nm), thus not many SFs are irradiated, or (ii) the SFs related emission is polarized and light is emitted along SFs planes, perpendicular to *c*-axis, therefore undetectable from the top of the film. This light emission anisotropy is of interest, since it may shed light on the number of previously reported confusing PL data of the ZnO nanostructures.^{66–75} Such ZnO nanostructures could demonstrate different PL spectra not due to different SFs concentrations, but due to different geometries of PL data obtaining, different nanowires texture and orientation, or different degree of *c*-axis texture for ZnO films. Therefore, the PL signals from the cross-section of the n- and p-type SZO samples were also acquired.

D. PL spectra of n-type vs. p-type SZO films. Obtained from the cross-section of the samples.

LT PLs were obtained from the cross-section of the n- and p-type samples as seen in Fig. 7. Unfortunately, it was difficult to perform the temperature dependent study with the cross-sectional geometry since the position of the sample is sensitive to the temperature change and the irradiation area cannot be kept the same.

FIG. 6. Evolution of the PL spectra with temperature increase for p- vs n-type SZO samples.

FIG. 7. Low temperature (10 K) PL spectra of SZO, acquired from the cross-section of the samples. The respective peaks are indicated. Peak at 3.314 eV, attributed to basal plane SFs emission is observed only for n-type SZO sample, which indicates the localization of nonactivated acceptors nearby SFs.

The LT (10 K) PL spectra of both n- and p-type SZO possessed peaks of FX recombination: $FX_p = 3.3801$ eV, $FX_n = 3.3785$ eV. Once again, such a difference in peak positions between the samples may result from the different strain (presumably local strain) values due to the doping. In addition, the deviation from the FX values obtained from the top-view PL study may be due to expected substrate lattice mismatch (LM) and thermal expansion coefficient (TEC) induced strain gradient along the film thickness.

Additional to the FX peak, the n-type sample demonstrates a neutral donor bound exciton peak (D^0X) at 3.3611 eV and ionized donor bound exciton emission peak (D^+X) at 3.3672 eV. The p-type sample possesses strong D^+X at 3.3709 eV. Moreover, some weak evidence of the peak at 3.359 eV can be observed, which may be attributed to neutral acceptor bound exciton (A^0X). Another peak at 3.334 eV is visible, which can be attributed to two electron satellite (TES) of the dominant peak, being separated by ~ 36 meV.

Interestingly, a weak peak at 3.314 eV has been observed for the n-type sample. This exciton emission could be attributed to SFs. In fact, Schirra *et al.* have also attributed the commonly observed 3.31 eV emission peak in ZnO to acceptor state transitions caused by SFs rather than intentional impurity acceptors.³⁶ However, no SF related emission was observed for p-type ZnO. For the n-type sample, it was originally grown aimed to be p-type, but turned out to have insufficient activated acceptor dopants. The rest of acceptor dopants is nonactivated. They could be localized nearby SFs in ZnO and contribute to SF emission. This, in fact, may additionally favor even more SF creation, as has been reported for GaN.^{32,76–78} For the p-type sample, most of the acceptor dopants are activated and uniformly distributed in the ZnO lattice. There is no obvious concentration of dopants on SFs and thus no SF related emission visible.

We have also studied power dependent PL at 10 K for the cross-sections of n- and p-type SZO (not shown here). The excitation power (P_{exc}) was ranged from 0.1 to 400 W/cm². The peak positions did not vary with the excitation power for all peaks in the PL spectra, while the peak intensity followed the power law $I = P_{exc}^n$, where $n = 1.2–1.4$. This confirms that the observed emission of all peaks is of excitonic type.

From the above comparison study, it is obvious that the electrical properties of SZO thin films are strongly influenced by the SFs. The p-type film possesses a high density of SFs, as observed by TEM, but no SF emission was detected by PL. In this case, the SFs have limited influence on the dopants distribution in ZnO, and thus the function of SFs is mainly for strain relaxation. In contrast, n-type SZO film shows lower SFs density and more local disorder in TEM, but PL emission attributed to SFs was observed. In this case, the SFs not only function for strain relaxation but also as dopants traps. As acceptors are localized nearby SFs and remain nonactivated. In order to enhance the electrical properties of p-type ZnO, further work is needed to understand how the function of SFs is related to their size, distribution, and other properties and how they can be manipulated by film growth and processing.

IV. CONCLUSION

SZO films were synthesized by PLD on *c*-cut sapphire substrates at different temperatures. P-type conductivity was realized for the 500 °C and 750 °C samples while the 400 °C sample showed n-type conductivity. According to TEM analysis, the p-type SZO film possessed a higher density of basal plane SFs compared to the n-type film. PL study of the SZO

films was performed in both top-view and cross-section mode. It was shown that PL study of SFs had a more prominent effect and was more informative in cross-section mode. Both n- and p-type SZO samples demonstrated FX emission, ionized donor bound exciton emission, and followed by neutral donor/acceptor bound exciton emission. A peak at 3.314 eV was solely observed for n-type SZO. This peak was attributed to the exciton emission of the SFs. In the n-type SZO, a large amount of nonactivated acceptor impurities were localized near the SFs, providing their visibility in PL spectrum. While in the p-type SZO, the acceptors were activated and more uniformly distributed. Thus, despite of even higher SFs density, no signal was observed. The PL study demonstrates a link between microstructural characteristics observed by TEM and n-type vs. p-type behavior in SZO thin films.

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