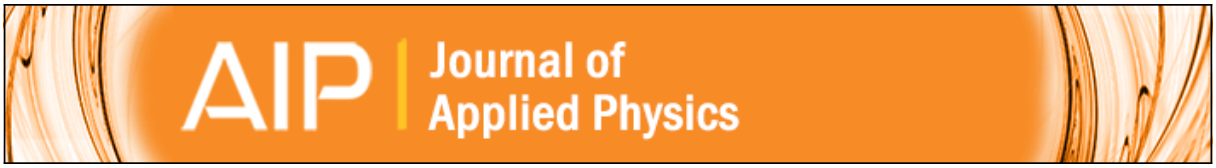

Ultrafast relaxation and absorption saturation at near exciton resonance in a thin ZnO epilayer

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Ultrafast relaxation and absorption saturation at near exciton resonance in a thin ZnO epilayer

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We observed ultrafast free exciton thermalization time of 700–900 fs and obtained the magnitude of maximal differential absorption to be 1.8×10^4 cm^{-1} with the pumping fluence of 10 $\mu J/cm^2$ by measuring transient differential transmission in a thin ZnO epitaxial layer at room temperature. The largest induced transparency occurs near exciton resonance associated with absorption saturation by comparing the excitation from the above band-gap to band-tail states. The pumping dependent transient absorption reveals transition of excitonic relaxation from exciton-phonon scattering to exciton-exciton scattering or to an electron-hole plasma. © *2011 American Institute of Physics*. [doi:10.1063/1.3525993]

I. INTRODUCTION

There has been great interest in optical properties of ZnO with the wide band-gap energy of 3.37 eV for the development of ultraviolet (UV) photonic devices.[1] Due to its large exciton binding energy of 60 meV as compared with other wide band-gap semiconductors,[2] such as ZnSe and GaN, free excitons exist in ZnO even at room temperature (RT). Besides, its large nonlinear absorption has been investigated by the Z-scan method near two-photon exciton resonance in near IR and near exciton resonance in UV range.[3,4] RT optical pumped UV spontaneous emission (SPE), stimulated emission, and lasing have been reported in ZnO thin films grown on sapphire substrates.[5,6] The SPE is attributed to both the free exciton and near-band-edge emissions, whereas, the stimulated emission and lasing are related to exciton-exciton scattering and electron-hole-plasma (EHP) depending on the optical pumping intensity.

Transient absorption measurements have been commonly performed in GaN and related III-V materials on time scales of femtosecond (fs) to nanosecond (ns).[7–9] For example, from the nondegenerate ns pump-probe technique, GaN thin films reveal large optical nonlinearities associated with exciton saturation under high interband excitation.[7] There are few studies on carrier dynamics in ZnO thin films and nanostructures by using transient absorption[10,11] and time-resolved PL (Refs. 12–16) (TRPL) spectroscopy. Yamamoto *et al.* measured the carrier dynamics in ZnO thin films,[10] and observed the saturation of exciton absorption as well as the optical gain on the lower energy side of free exciton by using the nondegenerate pump-probe method with high band-to-band excitation fluence of 90 $\mu J/cm^2$. On the other hand, by using the optical Kerr gate (OKG) method to study the TRPL in ZnO thin films with exciton resonance pumping fluence of 70 $\mu J/cm^2$, Takeda *et al.* reported that the formation of EHP takes less than 1 ps to build up.[12]

For the best performance of ZnO-based UV photonic devices, such as high-speed photonic devices and optical switches, quantitatively investigating the dynamical behavior and optical nonlinear absorption near free exciton resonance is necessary. In this work, besides the dynamics of the above band-gap states, the fs degenerate pump-probe technique under resonant excitation was used to investigate free exciton thermalization by measuring the time-resolved differential transmission in a thin ZnO epitaxial layer at RT. An ultrafast relaxation time of 700–900 fs and maximal absorption saturation can be observed at exciton resonance. The investigation of pumping dependent transient absorption suggests transition of excitonic relaxation from exciton-phonon scattering into exciton-exciton scattering or into an EHP under increase in pumping density.

II. EXPERIMENT

Our c-axis oriented ZnO thin film with thickness of 70 nm was grown on a c-plane sapphire substrate by high-vacuum pulsed laser deposition with KrF excimer laser. The detail growth apparatus and condition can be found in Ref. 17. The absorption spectrum was measured at RT (~295 K) by using the spectral photometer (Jasco V-670) with resolution of 0.5 nm. The time-integrated PL excited by a frequency doubled mode-locked Ti:sapphire laser with central wavelength of 360 nm was recorded by a single grating monochoromator (iHR320) at RT. Similar to the system setup in previous studies,[8,11,18] the measurement of transient absorption was carried out using the frequency doubled mode-locked Ti:sapphire laser with 82 MHz repetition rate (Tsunami, Spectral Physics Inc.), equipped with a frequency doubler (Model 3980), as the light source to provide UV

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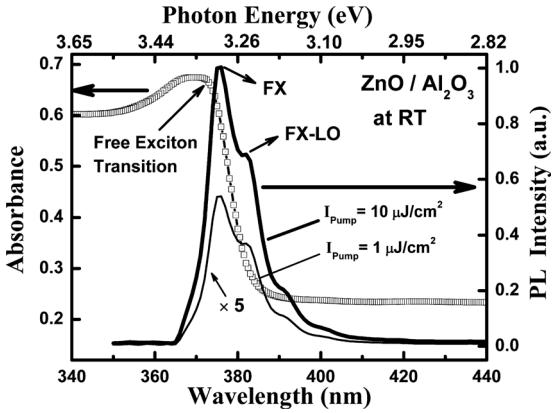

FIG. 1. Absorption spectrum (hollow squares) and TIPL spectra (solid lines) with excitation of 1 $\mu J/cm^2$ (thin line) and 10 $\mu J/cm^2$ (thick line) from a thin ZnO epitaxial layer at RT.

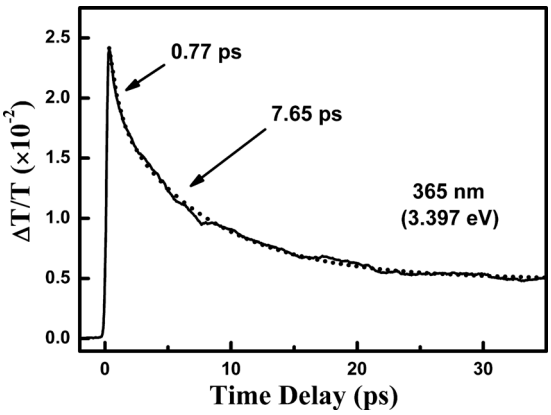

FIG. 2. Measured normalized transient differential transmission as a function of time delay with photoexcited energy above band-gap states. Dotted line is a fitting curve.

excitation with wavelength ranging from 360 nm (3.444 eV) to 383 nm (3.238 eV). The laser output was divided by a beam-splitter into the pump and the probe beams. The pump beam was chopped at 1 KHz by a chopper. The pump and probe beams made a small angle of 8° were focused on the sample by a 5 cm focal length lens with an intensity-contrast ratio of pump to probe larger than 20:1. The transmitted probe signal was detected by a photodiode connected to a lock-in amplifier. A time delay between the pump and the probe beams was introduced by an optical delay line with a stepping motor. In order to reduce the coherent artifact, we kept the pump and the probe polarizations orthogonal to each other by using a half-wave plate and put a linear polarizer with the polarization parallel to the probe beam before the detector.

III. RESULTS AND DISCUSSION

Figure 1 shows the absorption and time-integrated photoluminescence (TIPL) spectrum of the 70 nm ZnO epitaxial layer grown on c-plane sapphire at RT. The absorption edge for photon wavelength at 373 nm (3.324 eV) in Fig. 1 is corresponding to free exciton transition. We determined the band-gap energy to be around 3.384 eV by considering the exciton binding energy of 60 meV at RT. The linear absorption coefficient of free exciton transition is estimated to be around 2.2×10^5 cm^{-1}, also comparable to the value measured in ZnO single crystals.[19] The TIPL spectrum at RT under 1 and 10 $\mu J/cm^2$ excitation reveals the emission peak around 376 nm (3.298 eV) corresponding to free exciton emission with FWHM of 102 meV. It is comparable to previous result,[10] in which the peak position of PL spectra is located at 3.299 eV due to free exciton radiation at RT. It is also consistent with the result in Fig. 3 of Ref. 20, in which the PL peak is around 3.3 eV at RT. Due to large exciton and LO-phonon coupling in ZnO,[21] several peaks on the lower energy shoulder are the LO-phonon replica. The excitation carrier density is determined by $N_{exc}=I_{exc}\alpha/\hbar\omega_{exc}$ where I_{exc} is pumping fluence (or energy flux density), α represents the absorption coefficient, and $\hbar\omega_{exc}$ is photon energy. The focused diameter of pump and probe beams were measured to be 42–45 μm. We obtained the carrier density $N_{exc}\sim2.32\times10^{18}$ cm^{-3} with $I_{exc}=10$ $\mu J/cm^2$. We also normalized the measured TIPL spectra under 1, 10, and 20 $\mu J/cm^2$ excitation, and found they are almost the same except for slightly increasing around the dip, about 43 meV below FX emission. It may be due to the line-width broadening induced by heating under high pumping or the emergence of EHP luminescence as reported previously by Klingshirn *et al.*[20] They had shown the EHP emission in the range from 3.34 to 3.28 eV at 10 K, redshifting with increasing excitation density. Because the used sample is only 70 nm thick, the surface trapping will decrease the excited carrier density and the calculated carrier density directly from pumping may be overestimated.

The free carrier dynamics pumped by photon wavelength at 365 nm (3.397 eV) under 10 $\mu J/cm^2$ excitation, corresponding to the near band-gap state, is shown in Fig. 2. At the beginning of the pulse excitation, an instantaneous rise around zero time delay can be seen, and then the photogenerated free carriers quickly occupy the above band-gap states to quench the further absorption that is what the band-filling (BF) is observed. The trace after zero time delay can be found consisting of a fast and a slow decay. Generally speaking, the photoexcited free carriers are scattered out of their initial states through carrier-carrier scattering, which is comparable to the time scale around pulse duration, and then quickly relax to reach the quasiequilibrium with the lattice system through carrier-phonon interaction. Thus, the decrease in transient differential transmission can be attributed to the free carriers consume their kinetic energy via carrier-carrier and carrier-phonon scattering to the band-edge or to form excitons.

The normalized transient differential transmissions ($\Delta T/T$) near free exciton transition as a function of time delay for excitation wavelengths at 372 nm (3.333 eV) and 373 nm (3.324 eV) with 10 $\mu J/cm^2$ excitation are shown in Fig. 3. Their temporal behavior is similar to that performed at near band-gap (3.397 eV) excitation. Because the excitonic states are simply occupied by the resonantly generated excitons, the absorption saturation by the BF can be achieved.[22] The absorption saturation due to the occupancy of nonequilibrium excitons is sufficient to produce decrease in absorption resulting in a measurable differential transmission even with spatially averaging over the probe beam pro-

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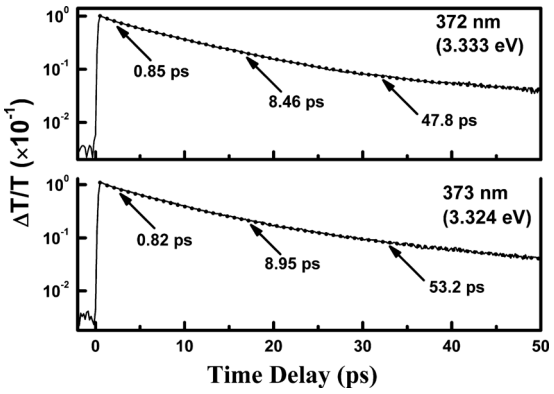

FIG. 3. Measured normalized transient differential transmission as a function of time delay for photon wavelength at 372 and 373 nm. Dotted lines are fitting curves.

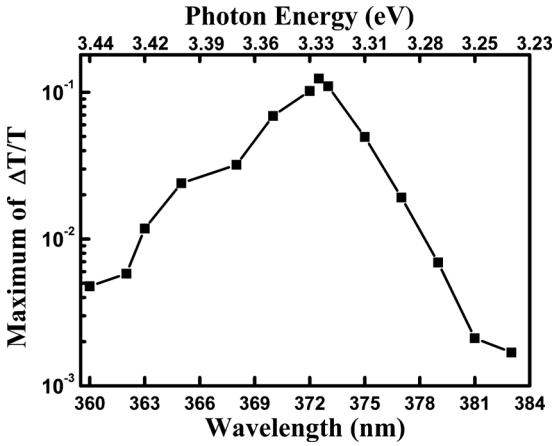

FIG. 4. The maximal differential transmission as a function of photon wavelength under pumping density of 10 $\mu J/cm^2$.

file, which is just slightly smaller than the pump one. The fast recovery of absorption saturation can be attributed to excitonic thermalization process through phonon emission. After the thermalization, the decrease in exciton population via either radiative or nonradiative recombination causes the slow recovery.

In order to extract the time constants of free carrier relaxation, the response function of $A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2)$ was used to fit the trace in Fig. 2. Here, the coefficient A_1 and A_2 is corresponding to the fast and slow exponential decay component contributed to the BF effect, respectively. The fast decay time τ_1 of 0.77 ps is consistent with the hot carrier cooling time on the order of 1 ps reported by Yamamoto *et al.* in ZnO thin films.[10] Besides, the obtained slow decay time τ_2 of 7.65 ps is close to the nonradiative recombination time on the order of 10 ps in ZnO nanorods.[11] On the other hand, the best fitting for the excitation at 372 and 373 nm in Fig. 3 were achieved in use of three decay times as: $A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2) + A_3 \exp(-t/\tau_3)$. An ultrafast time constant τ_1 of 0.82–0.85 ps can be obtained due to excitonic thermalization. According to previous TRPL studies of EHP dynamics in ZnO thin films by using OKG method,[12] the redshift in PL emission was observed with a time constant less than 1 ps during the build-up stage of EHP state. Because the formation of an EHP is concerned about photoexcited enough carriers to achieve the Mott transition, the screening of Coulomb interaction and the band-gap renormalization (BGR) effects need to be considered during the relaxation process. However, we pumped the sample here with lower excitation density and monitored the intrinsic nonequilibrium excitonic state. Thus, the reported ultrafast relaxation time of 0.82–0.85 ps is mainly due to the thermalization process through exciton-phonon scattering. Furthermore, the time constant τ_2 of 8.46–8.95 ps should be predominately caused by the nonradiative channels, such as trapping by defects, multiphonon emission, or Auger recombination. And the time constant τ_3 of 47.8–53.2 ps is attributed to the longer radiative lifetime, which is comparable to the previous TRPL studies that the SPE time is on the order of 46 to 110 ps in ZnO films grown by MBE and 30 to 74 ps in rf-sputtered films.[15,16]

The maximal normalized differential transmission as a function of photon energy from the above band-gap to the band-tail states was plotted in Fig. 4. The largest induced transparency due to absorption saturation occurs at 372.5 nm (3.329 eV), corresponding to the free exciton transition, is consistent with the absorption spectrum in Fig. 1. It can be obviously observed that the maximum of $\Delta T/T$ near the free exciton transition (372.5 nm) is five-times larger than the value near the band-gap state (365 nm) in Fig. 4, although their linear absorption coefficient is almost the same. Under exciton-resonant excitation, part of excited free excitons can be ionized into free electrons through the assistance of optical phonon, which the free electron-hole pairs and free excitons coexist in this system. The access free electrons cause the screening of Coulomb attractive potential to introduce the so-called BGR. According to previous studies, the investigation of transient absorption in GaAs and ZnTe quantum wells at RT,[22,23] that the bleached exciton absorption by excited resonant excitons is stronger than the same density of free electron-hole pairs. Thus, we can mainly attribute the negative differential absorption to the BF of resonantly excited excitons, while the screening effect may play a minor role.[22,23] Because the measured differential reflectance ($\Delta R/R$) is on the order of 10^{-3}, whereas the measured differential transmission ($\Delta T/T$) is on the order of 10^{-1}, we neglected the change in reflectance and deduced the differential absorption ($\Delta\alpha$) from the differential transmission based on the equation of $\Delta\alpha = -(1/d)\ln(1+(\Delta T/T))$ with d being the thickness of sample. The value of $\Delta\alpha$ is estimated to be -1.8×10^4 cm^{-1} near exciton resonance under 10 $\mu J/cm^2$ excitation that is comparable to previous results in GaN at 10 K and in GaAs at 15 K measured with differential transmission spectrum by using the pump-probe technique.[7,24]

In order to realize the mechanism of free exciton thermalization, we performed the pumping dependent dynamics and nonlinear absorption near exciton resonance. Figure 5 shows the free exciton dynamics for three different pumping fluence. We found the ultrafast relaxation time on the order of 800–900 fs is almost independent of the pumping fluence under 1–20 $\mu J/cm^2$. This ultrafast relaxation time is responsible for excitonic thermalization via exciton-phonon

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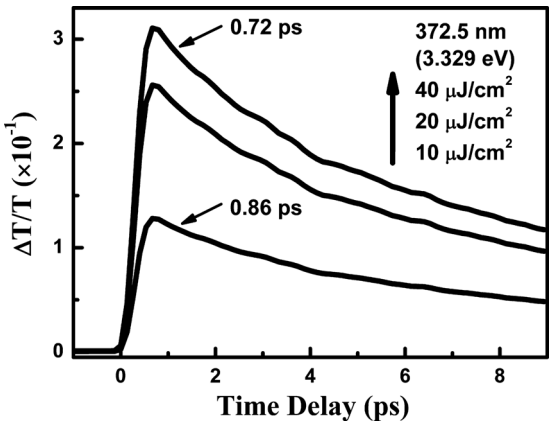

FIG. 5. Measured normalized transient differential transmission as a function of time delay for photon wavelength at 372.5 nm under pumping fluence of (from bottom to top) 10, 20, and 40 $\mu J/cm^2$.

scattering. However, the relaxation time slightly decreases to 700–800 fs as increasing the pumping fluence to 30–50 $\mu J/cm^2$. By using the OKG measurements, previous TRPL studies reported the build-up of P-band emission with a time constant of 800 fs through exciton-exciton scattering in ZnO thin films.[13] Consequently, the decrease in relaxation time with increasing pumping fluence supports the transition of excitonic thermalization from exciton-phonon scattering at low pumping into exciton-exciton scattering at high pumping.

The negative differential absorption maximum as a function of pumping fluence is shown in Fig. 6. Because the temporal regime of the measured differential absorption on the order of pulse duration is shorter than the relaxation process, the negative differential absorption maximum is mainly due to the absorption saturation by BF effect. To estimate the saturation intensity, we fit the experimental results by using the absorption saturation model[25]

$$\left|\frac{\Delta\alpha}{\alpha_0}\right| = \frac{(1-x)(I/I_S)}{1+I/I_S}, \quad (1)$$

where $\Delta\alpha$ is the differential absorption maximum within the pulse duration, α_0 is the absorption coefficient without pumping, x is the nonsaturated portion of absorption, and I_S is the saturation intensity. We obtained $x=0.65$ and $I_S=25\ \mu J/cm^2$. It means that about 35% of resonantly excited excitons contribute to the BF. As increasing the pumping fluence up to 50 $\mu J/cm^2$, a constant value of $\Delta\alpha \sim -4.7\times10^4\ cm^{-1}$ will be reached. In addition, the maximal differential absorption reveals a linear dependence of pumping fluence ($<20\ \mu J/cm^2$) corresponding to exciton-phonon scattering. It can be observed the emergence of nonlinearity as running off the linear dependence with pumping fluence around 20 $\mu J/cm^2$ (corresponding to $N_{exc}\sim4.6\times10^{18}\ cm^{-3}$) as shown in Fig. 6. When performed excitation larger than 20 $\mu J/cm^2$, it reveals a nonlinear dependence of pumping fluence that indicates the saturation of BF due to transition from exciton-phonon scattering dominant to exciton-exciton scattering or transition to an EHP. Similar results were reported in GaN thin films at 4 K under exciton resonant excitation.[9]

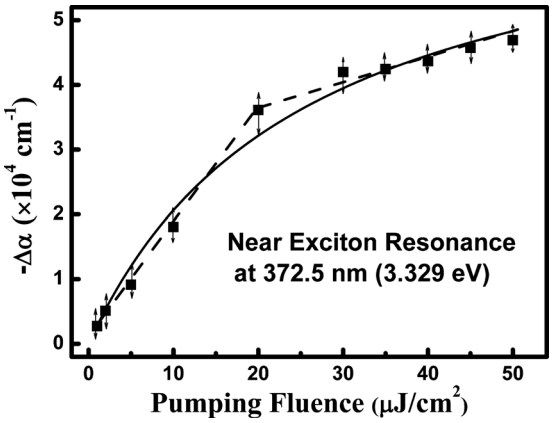

FIG. 6. A plot of negative differential absorption maximum as a function of pumping fluence (solid squares) with excitation near exciton resonance for wavelength at 372.5 nm. The solid curve represents the fitting curve to Eq. (1) and the dashed lines plotted to guide the eyes revealing a turning point at pumping fluence of 20 $\mu J/cm^2$.

IV. CONCLUSION

In conclusion, the energy dependent dynamics in a thin ZnO epitaxial layer have been investigated by using the fs pump-probe technique at RT. Transient absorption measurement shows an ultrafast relaxation time of 800–900 ps due to free exciton thermalization, and the differential absorption ($\Delta\alpha\sim-1.8\times10^4\ cm^{-1}$) under pumping fluence of 10 $\mu J/cm^2$. When the excited excitonic density increases, the relaxation time decreases to 700 fs and the magnitude of $\Delta\alpha$ gradually approaches a constant value. In addition, the largest induced transparency associated with absorption saturation occurs at near exciton resonance. The pumping dependent dynamics reveals transition of free exciton thermalization from exciton-phonon scattering into exciton-exciton scattering or into an EHP.

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