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REVIEW ARTICLE

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Multi-photon excitation in ZnO materials

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Abstract A brief introduction on the advance in the fabrication technology of ZnO materials was given. Related research on the multi-photon excitation processes in several kinds of ZnO materials under intense pump conditions by fs pulses were reviewed. Stimulated emission properties in ZnO microtubes and nanowires have also been dealt with. Possible nonlinear effects that emerged under the extremely intense field were discussed.

Keywords ZnO, multi-photon excitation

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1 Introduction

ZnO is a kind of II-VI compound wide band gap semiconductor with a direct band gap of 3.37 eV. This makes it a candidate for ultraviolet (UV) light-emitting devices. In particular, the large exciton binding energy of about 60 meV and the developed crystal-growth technology makes ZnO advantageous over GaN in the application of room-temperature (RT) UV laser diodes [1, 2]. ZnO also shows attractive properties in other aspects; it has been demonstrated to exhibit sensitivity to various gas species, which makes it suitable for sensing applications [3, 4]. Its resistance to high-energy radiation makes ZnO a suitable candidate for space applications [5, 6]. High Curie temperature >300 K for doped ZnO has been predicted and n-type doped ZnO was predicted to stabilize high-Curie-temperature ferromagnetism [7, 8]. ZnO nanostructures that exhibit piezoelectric properties have been synthesized, which implies their use as nanoscale traducers and resonators [9, 10]. ZnO also shows conventional chemical wet etching applications, making it feasible to get large area substrates at relatively low material costs [11, 12].

The above-mentioned properties have caused much attention for ZnO's potential application in various fields such as solar cells, sensors, varistors, transparent high power electronics, and optical waveguides [13]. In the past few decades, numerous researches have been drawn on ZnO materials [14−18]. Figure 1 shows the number of published articles on ZnO searched in the Web of Science from 1995 to 2007. As a result, great progress has been made in the fabrication technology of ZnO materials [19]. High-quality crystals and various kinds of nanostructures have been prepared at relatively low tem-

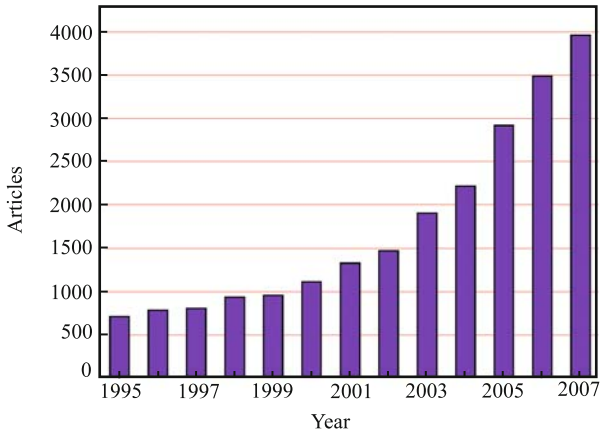

Fig. 1 The number of published articles on ZnO from 1995 to 2007.

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perature [20−23]. Exploring the excitation processes, especially the lasing behavior in ZnO nanostructural materials under intense pump conditions, is now possible.

Research on the photoluminescence (PL) properties of ZnO nanostructures have shown that under high excitation conditions, exciton-exciton collision, as well as the recombination of the electron-hole plasma (EHP), is a key process leading to stimulated emission and lasing [24−28]. Nanostructures could serve both as active gain material and optical cavity [29−31]. The nonlinear optical properties of ZnO are also attractive. Efficient second-harmonic generation (SHG) has been observed in ZnO thin films [32], while the high conversion efficiency of third-harmonic radiation was also achieved [33]. These studies demonstrate the potential of using ZnO to fabricate RT UV lasers. Using the well-developed visible to infrared laser source through multi-photon excitation at room temperature, we could obtain the UV emission and lasing from ZnO materials.

In this review, we will first give a brief introduction on the advancement of fabrication technology of ZnO materials. Then, we focus on multi-photon excitation processes under intense pump in several kinds of ZnO materials including single crystal, microtubes and nanowires. Stimulated emission and lasing properties in ZnO microstructures were also dealt with.

2 Fabrication techniques

ZnO is a II−VI compound semiconductor that exhibits the same crystal structure and tiny lattice mismatch to GaN. This made it a suitable substrate for epitaxial growth of GaN in earlier years. High-quality ZnO materials have been fabricated by methods that include hydrothermal, molecular-beam epitaxy (MBE) [34], metal-organic chemical-vapor deposition (MOCVD) [35], RF magnetron sputtering [36, 37], and pulsed-laser deposition [38].

Various kinds of nanostructures have been obtained hydrothermally from different substrates [39−46]. The growth process in hydrothermal has also been widely investigated [47, 48]. Properly polished hydrothermal ZnO exhibits optical properties similar to vapor-phase-grown materials [49]. It seems that hydrothermal is suitable for fabricating ZnO nanostructures on a large scale. However, alkali metals and metallic impurities in the solution will inevitably become mixed in the outcome. The sizes of the fabricated nanostructures are also relatively bigger compared with those of other fabrication techniques (usually more than 100 nm in diameter for nanotubes and nanorods).

Another method applicable to large-scale production is MOCVD. Significant progress has recently been made with improved reactor design [50]. One advantage is the feasibility of catalyst-free growth of ZnO nanostructures. Controlled growth mode has been demonstrated by adjusting the growth parameters [51].

Compared with CVD, the advantages of sputtering are its low cost, simplicity and low operating temperature [52]. It was found by Kim *et al.* that high substrate temperature is beneficial for improving the crystal structure, and the adjustment of RF power is necessary for the appropriate growth rate [36, 37, 53].

Due to precise control over the deposition parameters and *in situ* diagnostic capabilities, MBE is widely applied to fabricate ZnO films with high-quality. It has been demonstrated that a buffer layer and pretreatment have important effects on the growth of ZnO besides that of substrates [34, 54−57].

Besides fabrication techniques of pure ZnO, doping properties of ZnO are another highlight of research on this material. Doped ZnO materials have been fabricated by sputtering, hydrothermal, sol-gel and other techniques [58−60]. Their electronic, magnetic and optical properties have been widely researched. Thermal treatment effects on the structure and emission properties of fabricated ZnO materials have also been researched [61, 62].

Although much progress has been made in the fabrication field, there still exists a bottleneck for developing a reliable and reproducible high-quality p-type conductivity ZnO fabrication technology [19]. Furthermore, research on special nanostructures are still needed to prepare novel devices. Large-scale synthesis nanostructures with unique properties and high-yield, high-purity are necessary before this kind of material shifts from laboratory research to applications.

Recent progress in the fabrication technology of ZnO materials are reviewed in detail in Refs. [17, 19, 63].

3 Study on multi-photon excitation in ZnO materials

An important application for ZnO is serving as a potential candidate for RT UV emitters. However, in most related works, electron hole pairs are generated via linear optical excitation processes and the requirement of a laser source in the deeper ultraviolet range puts some limitations on its applications. Further study on the multi-photon excitation process may reveal a novel route to generate PL in ZnO materials. The SEM images of ZnO materials used to explore the multi-photon excitation processes are shown in Fig. 2(a) and (b).

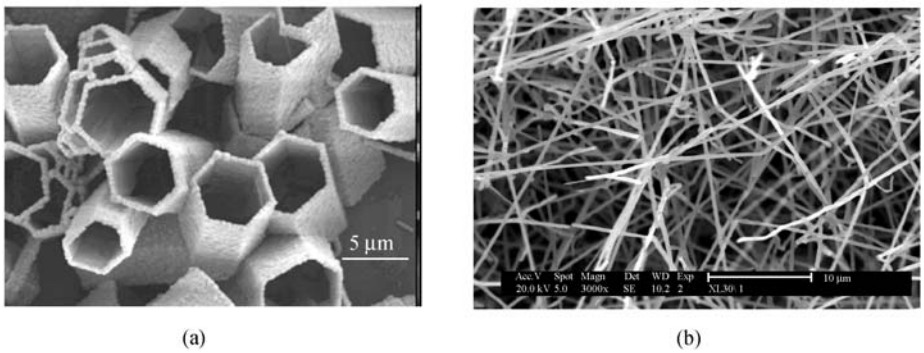

Fig. 2 The SEM image of the microtubes **(a)** and nanowires **(b)**.

3.1 Multi-photon excitation in ZnO crystals

He and Dai *et al.* reported research on the multi-photon excitation process in ZnO single crystals [64, 65]. Under intense excitation of high repetition fs pulses, three-photon absorption (3PA) was observed to be dominant in the excitation process at near infrared, while 2PA (2PA) was responsible for band edge emission when the photon energy was above half of the band gap of ZnO. Optical nonlinearity in the ZnO single crystal has been studied, and the effects of defect states have also been discussed.

Further research on the excitation process were carried out by applying Ti:sapphire laser (Spectra-Physics, Spitfire) with low repetition in our group [66]. The PL properties in ZnO single crystals under excitation intensity of 1 TW/cm^2 at wavelength of 800 nm are shown in Fig. 3. There is a sharp peak in the UV region located at 392 nm and a broad peak at about 550 nm, which is caused by defects in the ZnO crystal [67]. From the power dependence shown in Fig. 4, although the photon energy of the pump is still less than half of the band gap of ZnO, 2PA is found to be dominant in the excitation process under the action of an extremely intense field. Also, the transition from 2PA to 3PA was obtained when the wavelength of the pump was tuned from 780 to 820 nm. The discrepancy in the excitation process of ZnO single crystals under different pump intensities was regarded as possibly related to other effects that emerged under an extremely intense field induced by fs pulses with low repetition. This will be discussed in Section 3.4 in detail.

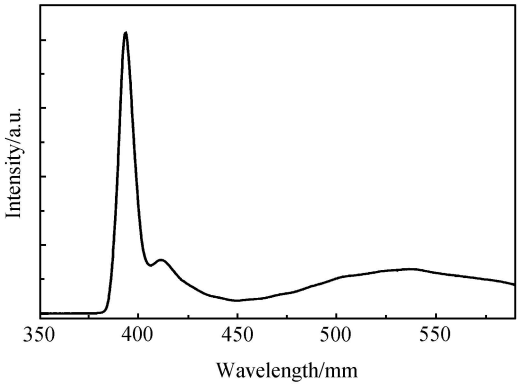

Fig. 3 The PL spectra in a ZnO single crystal excited by 800 nm fs pulses under excitation intensity 1 TW/cm^2.

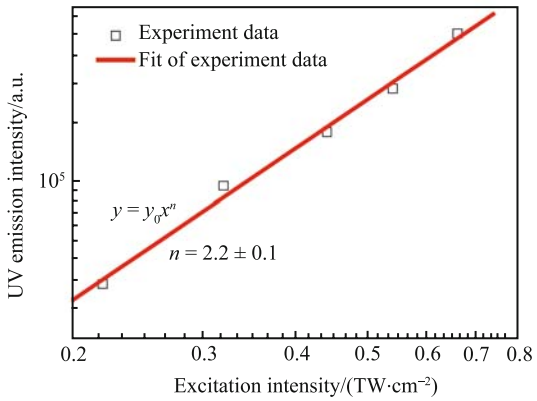

Fig. 4 Dependence of the UV peak intensity on excitation intensity on a logarithmic scale. The dots are experimental data and the solid line represents the fitting result.

3.2 Multi-photon excitation in ZnO microtubes

Since Iijima discovered carbon nanotubes, many efforts have been made to fabricate the tubular structured materials and study their optical and physical properties due to their special morphology [68−70]. 2PA luminescence in ZnO microtubes was first reported by Zhang *et al.* by applying nanosecond lasers [71]. Figure 5 shows the PL properties in ZnO microtubes irradiated by a nanosecond laser at 527 nm. The quadratic dependence of PL intensity on the excitation power shown in Fig. 6 confirms the dominant role of 2PA in this excitation process.

Excited by fs pulses at near infrared region, the band edge emission from a ZnO microtube was also observed.

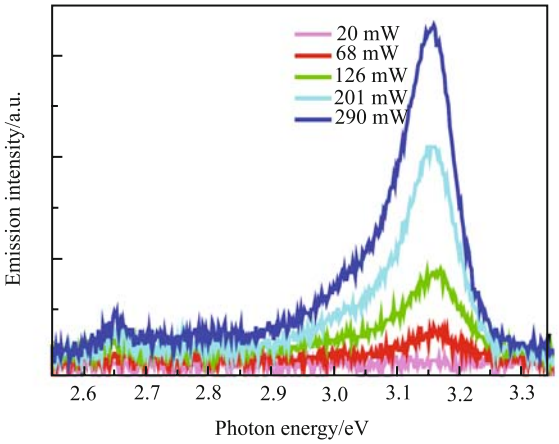

Fig. 5 The PL spectra in ZnO microtubes excited by 527 nm nanosecond pulses under different excitation powers.

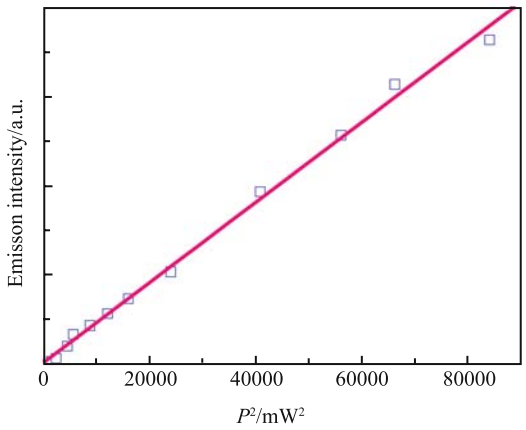

Fig. 6 The intensity of PL emission at 3.15 eV vs. the square of the excitation power.

Figure 7 is the PL spectra of ZnO microtubes excited by Xe lamp at 325 nm, nanosecond laser at 527 nm and fs pulses at 808 nm, respectively [72]. The small sharp peak at 404 nm is attributed to a second harmonic generation (SHG) signal, since this peak is located at half of the incident wavelength and shifts with the tuning of excitation pulses. The RT UV emission in a ZnO microtube can be generated through different excitation processes, including multi-photon absorption (MPA) as shown in this figure.

When increasing the excitation intensity, the transition from spontaneous band edge emission to stimulated emission in microtubes emerged and is shown in Fig. 8(a). When the excitation fluence exceeded the lasing threshold, the density of generated carriers was high enough for Mott transition [73] and the stimulated emission is obtained. The emission with peak at 388 nm is related to the exciton-exciton collision, while the peaks around 393 nm reflect the recombination process of EHP.

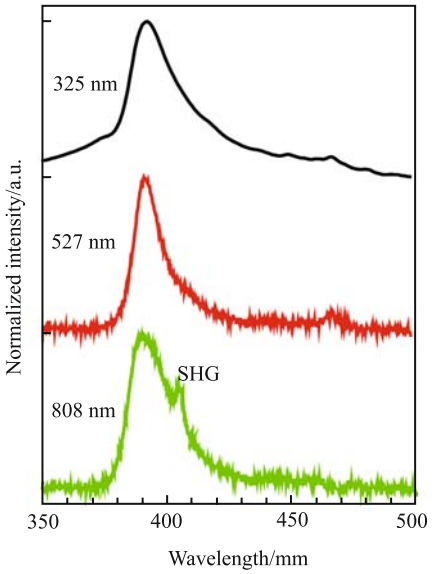

Fig. 7 PL spectra from ZnO microtubes excited by Xe lamp via single photon absorption at wavelength of 325 nm, MPA processes using nanosecond laser pulses at 527 nm and femtosecond pulses at 808 nm, respectively.

Based on the power dependence shown in Fig. 8(b), EHP is suggested to contribute to the gain of the stimulated emission, since the carrier density induced by MPA is superlinearly dependent on the excitation intensity. Mott transition may occur when the excitation is extremely intense. Before the emergence of the stimulated emission, the power dependence for band edge emission is quite similar to that of SHG, indicating how 2PA is important in the excitation process of ZnO microtubes under intense pumping at 808 nm.

When stimulated emission occurred, several well separated peaks can be seen in the PL spectra, as shown in Fig. 8(a). These peaks are attributed to different propagating modes in ZnO microtubes and have been verified by examining the linewidth and spacing between neighbor peaks through calculation.

3.3 Multi-photon excitation in ZnO nanowires

Since various nanostructures have been fabricated with high quality, small-diameter ZnO nanowires are expected to further lower the lasing threshold both because of the density enhancement of states near the band edge due to quantum effects and radiative recombination results from carrier confinement. An efficient SHG by 2PA process was observed by Prasanth *et al.* [74] in a single ZnO nanowire irradiated by fs pulses, while tuning the 2PA pump wavelength below band gap; resonant enhancement of the SHG was also observed.

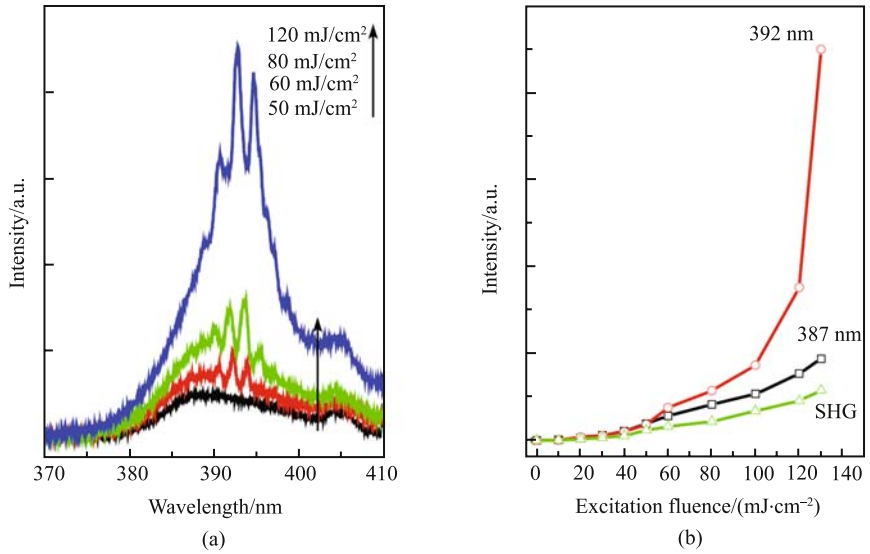

Fig. 8 **(a)** MPA induced PL spectra from ZnO microtubes excited by 808 nm femtosecond laser with different pumping fluences. **(b)** Emission intensity at 388 and 393 nm and SHG peak vs the excitation fluence.

The excitation processes in ZnO nanowires under high excitation intensity were explored in Refs. [75, 76]. Figure 9 is the PL spectra of ZnO nanowires excited by intense fs pulses at 806 nm with excitation intensity of 400 GW/cm^2. The UV band edge emission at 385 nm, SHG signal at 403 nm and the broad peak around 500 nm can clearly be distinguished before the appearance of the stimulated emission. When the excitation intensity exceeded a threshold, a sharp peak at 392 nm with FMHM of only 0.5 nm emerged as shown in Fig. 11, indicating lasing formation in the nanowires. The power dependence shown in Fig. 10 confirms that the signal at 403 nm is caused by SHG. Even at low pumping intensity, 2PA is also important in the excitation process in spite of the fact that the photon energy of excitation pulses is still less than half of the

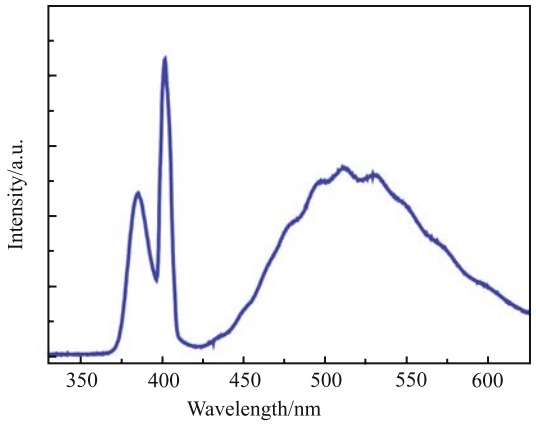

Fig. 9 PL emission spectrum of ZnO nanowires excited by intense femtosecond pulses at wavelength of 806 nm under excitation intensity of 400 GW/cm^2.

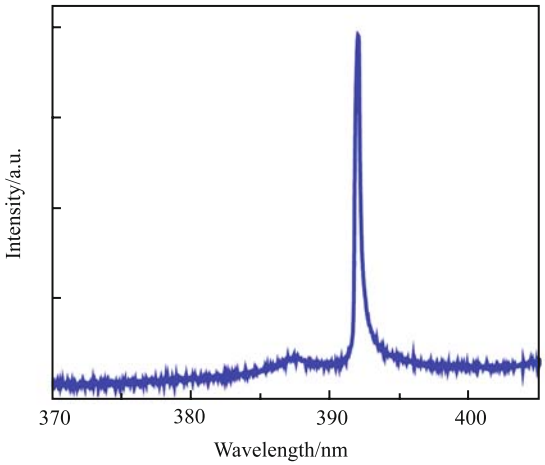

Fig. 10 Lasing emission spectrum from the ZnO nanowires with excitation intensity of 2.7 TW/cm^2.

band gap of ZnO. The red shift of the UV peak position with the increase of excitation intensity can be clearly seen in the inset of Fig. 11, indicating that the recombination of EHP is also responsible for the stimulated process in ZnO nanowires.

Although RT UV lasing in single ZnO nanowires has been obtained by one-photon absorption [1], the demand for a deeper UV pump source would put some limitations on its applications. Nonlinear optical processes would probably offer a new way to pump nanolasers.

3.4 Discussions on the excitation process in ZnO materials

It is well known that the band gap of ZnO is 3.37 eV at room temperature (a larger band gap should be obtained

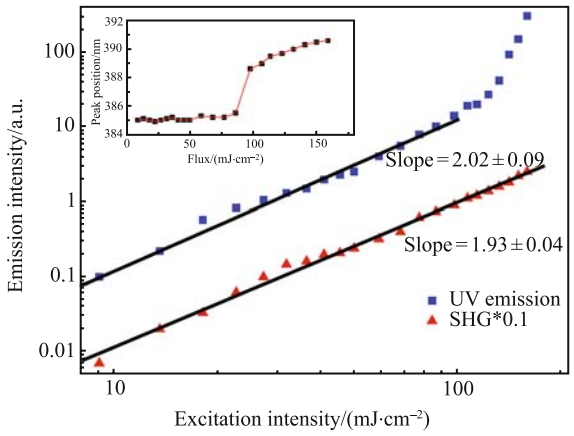

Fig. 11 Intensity of UV emission and SHG (multiply by 0.1 for clarity) on the excitation fluence shown in the logarithmic scale. The solid lines are the linear fitting results in the logarithmic scale. The inset shows the emission peak position at different excitation intensities.

in ZnO nanostructures due to quantum effects). 2PA should be responsible for the generation of carriers when the excitation photon energy lies between the band gap and its midpoint, while 3PA should be dominant when the photon energy is less than half of the band gap, as demonstrated by some researchers for ZnO materials when the excitation intensity is under TW/cm^2 [64, 65, 74]. However, 2PA absorption was found to be important in the excitation process in various ZnO materials under extremely intense femtosecond pulse excitation, even though the excitation photon energy is below half of the band gap of ZnO.

Nonlinear effects, such as MPA, Stark and Rabi effects that emerged under an intense field have been observed in various kinds of materials, including molecules and semiconductors [77−80]. These effects can make the band gap of the semiconductor varied by up to hundreds of millielectrovolts under an extremely high laser field [81, 82], which would dramatically reduce detuning effect and would enhance excitation via the 2PA process, similar to the case of exciting ZnO materials by applying intense fs pulses around 800 nm. The Rabi oscillation-assisted 2PA processes have already been demonstrated in many molecular two-level systems [77]. Since two-photon Rabi oscillation has recently been validated by the experiments in semiconductor quantum dots [78], it is reasonable to extend the Rabi oscillation in the 2PA process to ZnO materials, as the direct band gap semiconductor could be approximately taken as a two-level system.

Below are the motion equations of density matrix elements for two-level system in the presence of relaxation derived from the quantum optical theory:

$$\begin{aligned}
\frac{\mathrm{d}}{\mathrm{d}t}\rho_{11} &= -\frac{\mathrm{i}}{4}\Omega(t)(\rho_{12}-\rho_{21})+\gamma_{sp}\rho_{22}\\
\frac{\mathrm{d}}{\mathrm{d}t}\rho_{22} &= \frac{\mathrm{i}}{4}\Omega(t)(\rho_{12}-\rho_{21})-\gamma_{sp}\rho_{22}\\
\frac{\mathrm{d}}{\mathrm{d}t}\rho_{12} &= \frac{\mathrm{d}}{\mathrm{d}t}\rho_{21}^{*}=-(\gamma_{ph}-\mathrm{i}\Delta)\rho_{12}+\frac{\mathrm{i}}{4}\Omega(t)(\rho_{22}-\rho_{11})
\end{aligned} \tag{1}$$

where ρ_{11} and ρ_{22} are the population probabilities of the lower and upper states, respectively; ρ_{12} is the interference term; γ_{sp} and γ_{ph} are the spontaneous emission rates between the two states and the dephasing rate, respectively; and $\Delta = \omega_{21} - \omega_0 - \omega_0$ is the two-photon detuning. Two-photon Rabi frequency is proportional to the intensity of the laser pulse and it could be expressed as $\Omega(t) = \langle 1|E(t)\alpha E(t)|2\rangle = \Omega_0 \mathrm{e}^{-t^2/t_{\mathrm{p}}^2}$ where the laser pulse is assumed to have a Gaussian temporal profile with the pulse duration t_{p}.

Figure 12 shows the simulation results of the time-dependent population of the upper state (shown in red curves) and the image part of the interference term (shown in blue curves) under two-photon resonance excitation. The figure considers $\gamma_{\mathrm{sp}} = 0$ meV at different dephasing rates with $\Omega_0 = 50$ meV and $t_{\mathrm{p}} = 150$ fs, similar to the case of ZnO excited by fs pulses [83, 84]. The calculation results show that the dephasing effect is really important for the population of the upper state under resonant condition. When the dephasing effect is taken into consideration, the population left in the upper states after the fs laser duration decreased monotonously. This is attributed to dephasing weakening of the interference effect, as it can be clearly seen from the decrease of the blue curves in Fig. 12(b), (c) and (d).

But under detuning conditions, the results are very interesting. Figure 13 shows the simulation results at similar conditions except with $\Delta = 200$ meV. There is no population in the upper state after the pulse duration if dephasing doesn't take into consideration under this detuning condition, as all the generated carriers in the upper state fall back immediately to the lower state when the excitation pulse elapses. However, the population remains in the upper state if dephasing is considered. This can be clear seen from the blue curves in Fig. 13, as the image part of the interference term determines the evolution of carriers falling back to the lower state even if the spontaneous emission is neglected. Also, for its damage to interference effect, too large dephasing is found of no use to enhance the population in the upper state.

Furthermore, under extremely intense excitation, oscillation can be found in the time-dependent population of the upper state as shown in Fig. 14. Because the two-photon Rabi frequency is increased under intense excita-

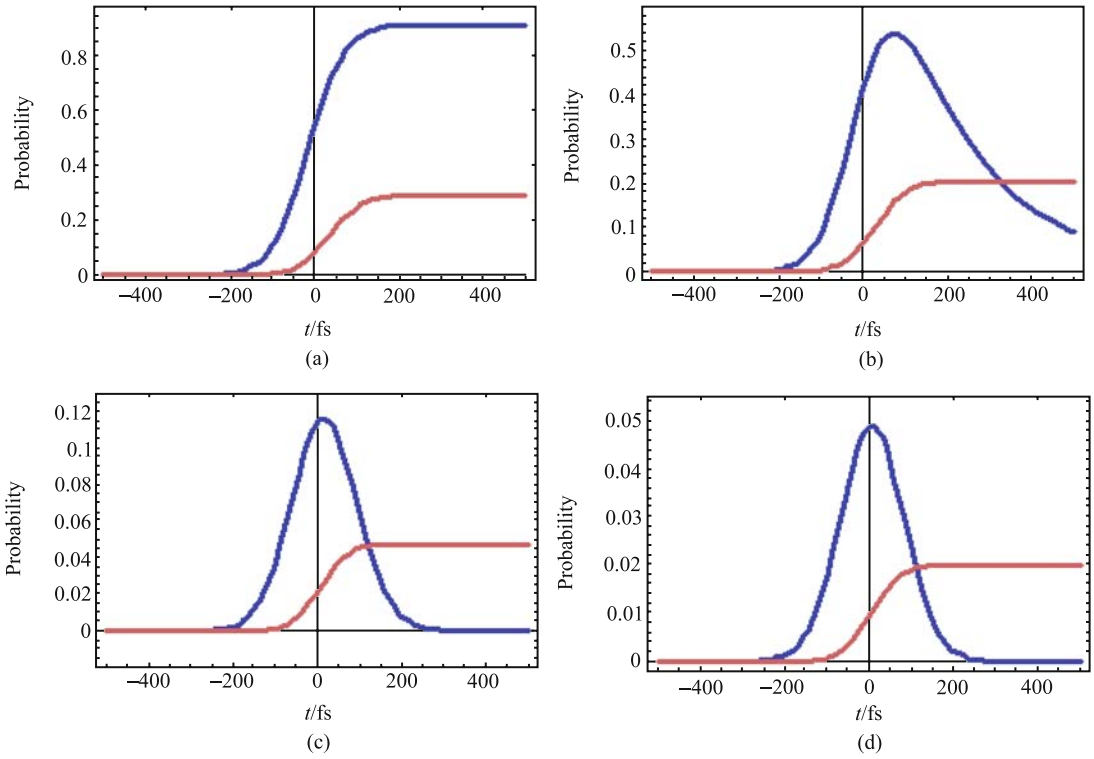

Fig. 12 Simulation results of time-dependent population of the upper state (shown in red curves) and image part of the interference term (shown in blue curves) with two-photon resonance and $\Omega_0 = 50$ meV after irradiation by fs pulses with FWHM 150 fs at different dephasing rates **(a)** $\gamma_{\rm ph}$=0 meV, **(b)** $\gamma_{\rm ph}$=20 meV, **(c)** $\gamma_{\rm ph}$=200 meV, **(d)** $\gamma_{\rm ph}$=500 meV.

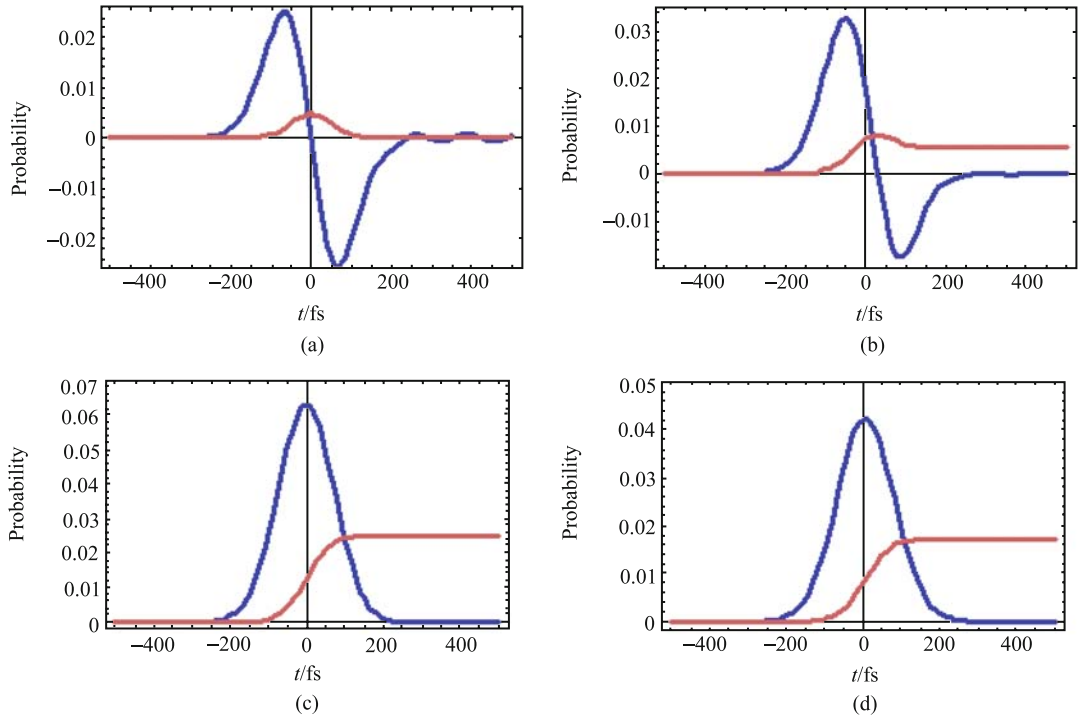

Fig. 13 Simulation results with two-photon detuning at 200 meV and $\Omega_0 = 50$ meV under similar conditions at different dephasing rates **(a)** $\gamma_{\rm ph}$=0 meV, **(b)** $\gamma_{\rm ph}$=20 meV, **(c)** $\gamma_{\rm ph}$ = 200 meV, **(d)** $\gamma_{\rm ph}$=500 meV.

tion, it is not surprising that the oscillation of population can be seen in the duration of the excitation fs pulse under extremely intense excitation conditions. A similar result has been observed directly by Schülzgen *et al.* in semiconductors [79].

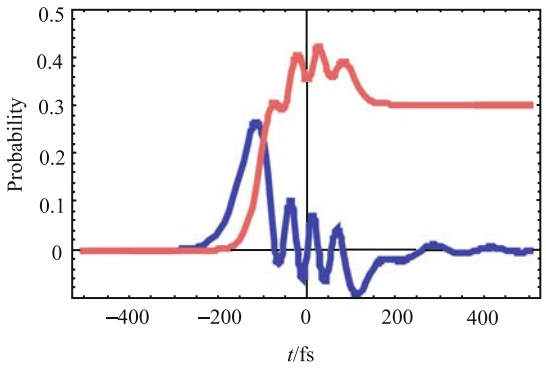

Fig. 14 Simulation result of the population of the upper state with two-photon detuning at 200 meV at dephasing rate γ_{ph}=20 meV after irradiation by extremely intense fs pulse Ω_0 =1 eV.

Figure 15 shows the probabilities left in the upper state after the irradiation of fs laser pulses at different excitation powers and different detunings. In the resonant case, the probability shows "oscillation" with increasing excitation power, which is confirmed by to the photocurrent result of the single photon excitation case in semiconductor quantum dots [85]. However, with the existence of detuning, the population probability in the upper state will grow monotonously with increasing excitation power. It can also been seen that the probability in the upper state decreases monotonously with increasing detuning under a given excitation intensity.

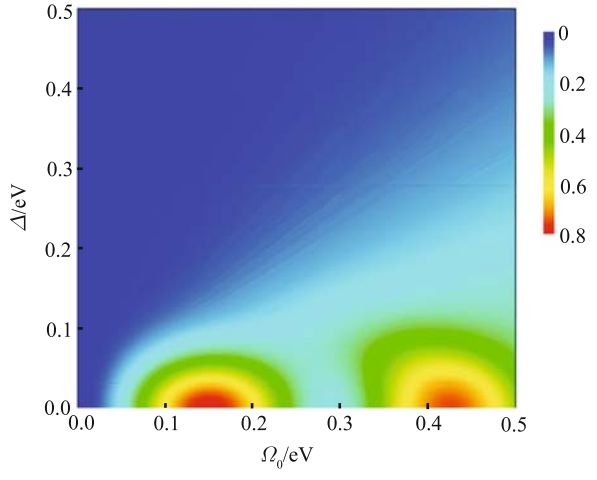

Fig. 15 The probabilities left in the upper state after the irradiation of fs laser at different excitation powers and different detunings.

All of the above-mentioned simulation results and observed experimental phenomena show that under the extremely high optical field generated by fs pulses, the Rabi effect may benefit 2PA in the excitation process even under large detuning. This may provide a new nonlinear optical way to generate RT UV PL in ZnO materials under the intense field of fs pulses.

4 Summary

A brief review on the progress in the fabrication technology of ZnO materials is presented. Developing a reliable and reproducible high-quality p-type conductivity ZnO fabrication technology for large scale application remains a challenge for its future applications. The researches on the multi-photon excitation process in ZnO single crystals, microtubes and nanowires, with successful results have been introduced. In particular, the excitation processes in ZnO materials under an extremely intense field of fs pulses have been discussed. Nonlinear effects that emerged in these conditions are theoretically responsible for the unusual excitation processes. Stimulated emission properties in ZnO microtubes and nanowires have also been considered. Based on the simulation of excitation processes in this material, we have confirmed that even at detuning conditions, there would be a definite population in the excited state and PL emission from ZnO material under extremely intense excitation. The effective excitation of ZnO by nonlinear optical absorption (2PA and 3PA etc.) is proven to be a realistic mechanism of the excitation for wide band-gap semiconductors.

Researches on the multi-photon excitation process reveal a novel NLO route to effectively excite the carriers and generate the PL emission in ZnO materials, and it is meaningful to explore RT UV lasers using ZnO materials in near future.

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