Ultraviolet luminescence enhancement of ZnO two-dimensional periodic nanostructures fabricated by the interference of three femtosecond laser beams

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New Journal of Physics 13 (2011) 023044 (14pp)
Received 2 September 2010
Published 28 February 2011
Online at http://www.njp.org/
doi:10.1088/1367-2630/13/2/023044

Abstract. We have developed a simple and rapid method for fabricating ZnO periodic nanostructures. By changing the laser polarization combinations, we fabricated different types of two-dimensional (2D) nanostructures on ZnO crystal surfaces by the interference of three femtosecond laser beams. The 2D nanostructures became more regular and uniform when increasing the cross angles between any two laser beams. Compared with the case of the plane surfaces of ZnO crystals, the 2D nanostructures revealed an ultraviolet (UV) luminescence enhancement excited by an 800 nm femtosecond laser beam. We studied the photoluminescence properties of the 2D nanostructures and the mechanisms of the UV luminescence enhancement. Our results indicated that the enhancement was caused by an increase in optical absorption with respect to that of the unaltered ZnO plane surface and by the formation of surface defect states.
1. Introduction

ZnO nanostructures have attracted considerable attention owing to their wide band gap (3.37 eV) and high excitonic binding energy (60 meV). Various methods for fabricating different kinds of micro- and nanostructures have been reported, such as chemical vapor deposition [1]–[3], the vapor–liquid–solid process [4]–[6] and biocatalysts [7].

Laser-induced periodic ripples in semiconductors and dielectrics have been studied intensively in the last four decades. The periods were usually close to the laser wavelengths, and these long periodic ripples (LP-ripples) were attributed to the interference between the incident laser and the surface scattered light field [8, 9]. Recently, nanoripples with periods much shorter than the laser wavelengths were reported in semiconductors and dielectrics after irradiation of linearly polarized femtosecond laser pulses [10]–[13]. These short periodic ripples (SP-ripples) were perpendicular to the laser polarization. If the laser beam was circularly polarized, nanoparticles would be induced on the sample surfaces [14]. To understand the mechanisms of the formation of SP-ripples, several explanations have been proposed, such as interference, self-organization and the enhancement of local electric field [10]–[14].

Femtosecond laser-induced nanostructures on ZnO have also been studied intensively [15]–[19]. The authors of [15]–[17] have reported on the formation of SP ripples and their Raman properties. Huang et al [18, 19] studied the fabrication of uniform ZnO nanosquares ablated alternately by two femtosecond laser beams with polarizations orthogonal to each other. Dufft et al [13] reported the formation of LP- and SP-ripples. They found that the periodic ripples depended on the laser fluences and pulse numbers, and attributed the formation of SP-ripples to the surface second harmonic generation (SHG) [13].

Due to the high efficiency and low cost, holographic lithography (HL-technology) becomes an important technology for fabricating nanostructures [20]–[22]. Combining the fabrication of short-periodic nanostructures induced by femtosecond laser irradiation with HL technology, we have fabricated two-dimensional (2D) complex micro-/nano-structures on the surfaces of ZnO crystals [23, 24].

In this paper, we change the laser polarization combinations and fabricate several types of 2D nanostructures on the surfaces of ZnO crystals by the interference of three femtosecond laser beams. The 2D nanostructures become more regular and uniform with increasing cross angles between any two laser beams. Then, we make further investigations into the photoluminescence (PL) properties of the 2D nanostructures induced by multi-photon absorption.
2. Experimental set-up

Figure 1 shows the experimental set-up for the interference of three femtosecond laser beams [24]. A linearly polarized laser at a wavelength of 800 nm was delivered from a commercial Ti : sapphire regenerative amplifier (Hurricane, Spectra-Physics). The pulse duration was 50 fs, and its repetition rate could be changed in the range of 1–1000 Hz. The laser beam was passed through a half-wave plate (HF1) and a Glan polarizer (GZ) to adjust the pulse energy and polarization. Then the laser beam was split into three beams using two beam splitters. Three half-wave plates (HF2, HF3 and HF4) were used to rotate the polarization of the three laser beams, respectively. The inset in figure 1 shows that the spatial positions of the three beams formed a regular triangle. A, B and C represent the positions of the three beams on the P-plane, and O the superposition point on the sample. Laser pulses from the three beams arrived at the sample surface simultaneously. The zero temporal delay point was determined by measuring the signal of sum frequency via a BBO crystal. We changed the laser polarizations and the cross angles $2\theta$ between any two laser beams and fabricated several kinds of 2D nanostructures. Commericially available c-cut ZnO single crystal with a size of 10 mm $\times$ 10 mm $\times$ 1 mm was used in the experiments. The two surfaces of the ZnO crystal were both optically polished.

PL micrographs of the 2D micro-patterns were acquired using a Nikon microscope (Eclipse 80i) following excitation with 800 nm laser pulses. For the 2D microstructures with a period of 3.36 $\mu$m, the 800 nm laser beam focused on the patterns at an incident angle of 45°, with a focus diameter of 3 mm. However, to acquire clear PL micrographs of the 2D patterns with a period of 1 $\mu$m, the 800 nm laser beam was normally focused on the sample surface using a 100× objective microscope. Here, no color filter was used in the experiment.

To investigate the PL properties of the 2D nanostructures, we fabricated a large area with a size of 1.7 $\times$ 1.3 mm$^2$ by ablating the sample spot by spot. The ablation area was excited by an
800 nm laser beam at an incident angle of 45°. The PL spectra were collected in the normal direction of the sample surface and measured using a spectrometer with a charge-coupled device. We compared the PL spectra of the 2D nanostructures with those of the plane surfaces of the ZnO crystals.

To comprehend the mechanisms of ultraviolet (UV) luminescence enhancement, we measured the optical reflectivity, transmissivity and absorptivity of the 2D nanostructures and the plane surfaces of the ZnO crystal. The 800 nm laser beam was focused on the ablation area and the plane surfaces of the ZnO crystal at an incident angle of 45°. The reflected light was collected using a silica lens with a diameter of 78 mm. The lens was set at a position 56 mm away from the sample, and the collection angle was calculated to be 70°. Considering the scattering effect of the 2D nanostructures, we measured the scattering spectra using a fiber optic spectrometer (USB2000, Ocean Optics). The measurements were carried out on the circumference of a semicircle with a radius of 100 mm.

3. Results and discussion

3.1. Periodic micro-/nanostructures

Figure 2 shows the SEM images of the 2D nanostructures. Three types of polarization combinations are shown in the insets in figures 2(a), (c) and (e), respectively. For the cross angle of $2\theta = 13.6^\circ$, the 2D spots were formed in a hexagonal arrangement with a period of 3.36 $\mu$m. Meanwhile, short periodic nanostructures were embedded in the hexagonal microstructures. In figure 2(a), the nanoripples with a period of 200 nm formed on each ablation spot, and they were perpendicular to laser polarization. In figures 2(c) and (e), radially orientated and circularly distributed nanoripples appeared around each bulgy microspot, respectively. Also, there were six deeply ablated nanopits, which are shown by the white circles in figures 2(c) and (e). We performed theoretical calculations of the intensity pattern and the polarization pattern for the three types of laser polarization combinations. The results accord well with the 2D periodic nanostructures [24].

As the cross angles $2\theta$ increased to $47^\circ$, the periods of the hexagonal patterns decreased to 1 $\mu$m. The 2D periodic structures became more regular and uniform, as shown clearly in figures 2(b), (d) and (f). In addition, we observed that the fabricated patterns were obviously different from the ones of $2\theta = 13.6^\circ$, especially for the two laser polarization combinations shown in figures 2(c) and (e). In figures 2(d) and (f), the six deeply ablated nanopits around each bulgy spot disappeared. Meanwhile, for the laser polarization combination shown in figure 2(c), the patterns evolved into hexagonal nanoflowers composed of radial nanoripples (see figure 2(d)). For the laser polarization combination shown in figure 2(e), the patterns evolved into regular nanoring arrays (see figure 2(f)). Using this method, we could obtain several kinds of regular and uniform nanostructures, which would periodically modify the photoelectric properties of the sample.

3.2. Photoluminescence of the two-dimensional (2D) nanostructures

Figure 3 shows clearly bright blue light emitting from the 2D nanostructures. For the parallel laser polarization combination, the hexagonal pattern with blue light emission dots shown in figures 3(a) and (b) accords well with the 2D nanostructures shown in figures 2(a) and (b),
Figure 2. SEM images of the ablation areas induced by the interference of three laser beams. The laser fluences and pulse numbers of a single laser beam are as follows: (a) 3.74 J cm$^{-2}$, 40; (b) 0.05 J cm$^{-2}$, 260; (c) 0.20 J cm$^{-2}$, 500; (d) 0.10 J cm$^{-2}$, 500; (e) 3.5 J cm$^{-2}$, 50 and (f) 0.43 J cm$^{-2}$, 100. The insets of (a), (c) and (e) show the polarization geometries of the three laser beams. The laser intensity of the 50 fs pulse is equal to $2 \times 10^{13}$ W cm$^{-2}$ if the laser fluence is 1 J cm$^{-2}$.

respectively. For the polarization combination with a cross angle of 120°, the luminescence from six deep nanopits around each bulgy spot was very strong (see figure 3(c)). But for the 2D nanostructures with a period of 1 µm, figure 3(d) shows that the six individual emission nanopits evolved into nanorings. The result accorded well with the SEM images shown in figure 2(f).

The PL micrograph in figure 4(a) shows clearly that strong blue light is emitted from the interference pattern. In particular in the points with deeper ablation depths, the PL intensity was much stronger, which was proved by the PL intensity measurements shown in figure 4(b). The pixel numbers are the data on the inset line in figure 4(a). Figure 4(b) shows that the PL intensities on these ablation dots were 10–16 times higher than those on the plane surface surrounding the whole ablation spots. The diameter of the excitation laser was about 3 mm, which was much larger than that of the ablation area. Therefore, the weak PL intensities around the ablation area in figure 4(a) were not caused by the lower intensities of the local excitation laser field.

The energy structures of the ZnO crystal have been studied in detail [25]–[27]. For a better understanding of the PL spectra in the following, we will now describe them briefly. The band gap of the ZnO crystal is of 3.37 eV, and it red-shifts to 3.25 eV at room temperature.
Figure 3. (a–d) PL micrographs of the two-dimensional (2D) nanostructures corresponding to the patterns shown in figures 2(a), (b), (e) and (f), respectively.

Figure 4. (a) PL micrograph of the interference pattern; (b) the PL intensities on the inset line in (a).

The excitonic energy is lower than the conduction band edge by 50–100 meV. The energy of oxygen defect states is peaked at 2.35 eV and the Zn interstitial energy state is very close to the conduction band edge. Here, the valence band edge is taken as 0 eV.

We studied the PL spectra of the plane surface of the ZnO crystal and the 2D nanostructures excited by 800 nm laser pulses with a repetition rate of 1 kHz. For the plane surface, figure 5(a) shows that the PL spectra consist of a broad visible band peaking at 2.35 eV and an ultraviolet (UV) band. The green light emission was mainly attributed to the defect states of oxygen vacancies. The UV emission band of the plane surface is shown in detail in figure 6(a). The peak position is at 3.11 eV for the excitation laser intensity of 16.2 GW cm$^{-2}$. It shifts gradually to 3.15 eV as the laser intensity increases to 90.0 GW cm$^{-2}$. As pointed out in [28], this UV band is a result of the superposition of the band-gap emission and the SHG emission. The band-gap

New Journal of Physics 13 (2011) 023044 (http://www.njp.org/)
Figure 5. (a, b) The PL spectra of the plane surface of the ZnO crystal and the 2D microstructures. The insets of (a, b) show the optical micrographs of the plane surface and the 2D nanostructures, respectively.

Figure 6. The PL spectra of the plane surfaces (a) and the 2D nanostructures (b). The excitation laser intensities of lines 1–9 in (a) are 16.2, 20.1, 25.1, 34.4, 41.4, 46.8, 60.4, 70.0 and 90.0 GW cm\(^{-2}\), whereas in (b) they are 10.5, 14.0, 16.2, 20.1, 25.1, 27.0, 34.4, 41.4 and 55.1 GW cm\(^{-2}\), respectively.

emission is usually attributed to the excitons bound to neutral donors (D\(_0\)X) and the excited states [26, 29, 30].

For the 2D nanostructures, figure 5(b) shows that the green emission band was well suppressed. Many previous works have demonstrated that the density of oxygen vacancies decreased greatly after an annealing process in an oxygen-enriched environment, and the green light emission band was greatly depressed, too [29, 31, 32]. The 2D nanostructures were induced by femtosecond laser ablation in air, which also had a laser annealing effect on the ablation area. So the suppression of the green emission band of the 2D nanostructures was caused by the reduction of the density of oxygen vacancies.

In contrast to the green emission band, figure 5(b) shows that the UV emission band of the 2D nanostructures was greatly enhanced. The dependence of the UV band on the excitation laser intensities is depicted in figure 6(b). The UV emission band peaks at 3.17 eV for the
excitation laser intensity of 10.5 GW cm\(^{-2}\), and it does not shift as the intensity increases to 55.1 GW cm\(^{-2}\). The papers [1, 6] compared the UV luminescence and the SHG signal of ZnO nanowires excited by an 800 nm femtosecond laser beam. The UV luminescence dominates the spectra as the excitation laser intensity is higher than 9.6 GW cm\(^{-2}\) [6]. The two peaks are hard to resolve as both the wavelength and the bandwidth are very close [28].

Excited by femtosecond laser pulses at wavelengths of 650 and 330 nm, the UV emission bands of the 2D nanostructures were at 3.20 and 3.25 eV, respectively. The peaks changed slightly with the excitation laser wavelengths, which was similar to the results reported in [33, 34]. Excited by the laser with photon energy less than half of the ZnO band gap, the peak of UV emission was 3.15 eV, which could be attributed to D\(_{0}\)X and the excited states. If the photon energy of the excitation laser is larger than half of the band gap, the peak of the UV emission band was at 3.30 eV, which could be attributed to the free exciton emission.

In general, the UV emissions of the plane surfaces and the 2D nanostructures were slightly broader and red-shifted compared with the results reported in [35], which was mainly caused by the higher temperature in our experiments (laboratory temperature). The authors of [30] have also reported that the width of the UV emission band increases and the peak red-shifts with increasing temperature. In addition, the red-shift of the UV emission band was also dependent on the excitation laser wavelengths and intensities.

Figure 7 plots the PL intensities of the UV luminescence and the SHG signals as a function of 800 nm laser intensities \(I\). For the 2D nanostructures, the slope of the fitting line of the UV luminescence at a wavelength of 391 nm was 2.4, whereas for the SHG signals it was 2.03, which was similar to the results reported in [1]. At room temperature, the ZnO crystal could linearly absorb light with photon energies in the range of 2.95–5 eV. For photon energies in the range of 2.95–3.25 eV, it was the band-tail absorption. In our experiments, the photon energies of the excitation laser were in the range of 1.48–1.61 eV. The sample can absorb the excitation laser via the two- and three-photon processes. Therefore, the dependence of \(I^{2.4}\) indicated...
that the UV luminescence was caused by the two- and three-photon absorptions. The authors of [6] have reported on the UV luminescence of ZnO nanowires and found a dependence of $I^{2.13}$ on the excitation laser intensities, which revealed a two-photon absorption process as the excitation mechanism responsible. In addition to the slight difference between the samples, the main reason for the different slopes is the excitation laser spectra. In [6], the laser photon energies were in the range of 1.38–1.75 eV. For the plane surface of the ZnO crystal, the slope obtained by fitting the intensity dependence at a wavelength of 400 nm is 2.22. Figure 6(a) shows clearly that the UV band peaks shift gradually from 400 nm (3.11 eV) to 393 nm (3.15 eV) as the excitation laser intensities increase from 16 to 90 GW cm$^{-2}$. Therefore, the deviation in the slope of the 400 nm signal from 2.0 is mainly caused by the superposition of the UV emission band.

Many previous works have reported various methods for fabricating ZnO nanostructures. The enhancement factors of the UV emissions of doped nanostructures were reported to be up to $10^2$–$10^4$ [2, 4, 5, 36, 37]. These results were very interesting and exciting. However, the authors of [5, 36, 37] also reported that before being doped, the UV emissions of the nanorods and nanowires were very weak.

As a typical direct wide-band-gap semiconductor, the ZnO crystal itself has a strong blue emission band. In our experiments, we chose the ZnO crystal as the comparison object, and found that the UV emission was enhanced by 7–9 times after the 2D nanostructures were fabricated (see figure 7). The result was similar to the ZnO nanorods reported in [3].

The chemical methods for fabricating ZnO nanostructures are usually complicated. Compared with these methods, the interference of multi-femtosecond laser beams has some advantages. These luminescence dots have a regular arrangement, with the patterns and periods adjustable by changing the arrangement of the spatial positions and polarizations of laser beams. The whole fabrication process was conducted in a laboratory environment and lasted for only several seconds.

3.3. Discussions on the enhancement of the UV emission

Figure 8 shows the scattering light intensities of the 2D nanostructures. The results indicated that the main scatting light was in the direction of the reflection angle of 45°. Summing the scattering light intensities in the collection angle of 15°–75° and in the semicircle (5°–175°), a collection efficiency of 65% was obtained. So we made a correction factor of 0.65$^2$ for the hemispherical scattering light.

Figure 9(a) shows that the relative reflectivities and transmissivities of the 2D nanostructures decreased, respectively, to 18–25% and 10–15%, where the reflection and transmission of the plane surface of the ZnO crystal were normalized to 1. For each power of the incident laser, the absorptivity of the plane surface of the ZnO crystal was 18%, and for the 2D nanostructures it was 90% (see figure 9(b)). Namely, the optical absorption was enhanced by five times after the 2D nanostructures were formed.

The formation of the 2D nanostructures was one reason for the enhancement of absorptivity. The increase in surface area enhanced the light absorption, and the multiple reflections of the incident light among these nanostructures reduced the reflection. In addition, the nonlinear absorption could be improved by the light localization effect of nanostructures [38]–[40]. A theoretical study indicated that the light intensity can be enhanced by four times in the surface of nanostructures [41].
Another reason for the enhancement of absorptivity was attributed to the change in crystalline structure on the sample surface [42, 43]. Micro-Raman experiments were conducted with a Raman spectrometer (Jobin Yvon T64000) excited by an argon ion laser at a wavelength of 514 nm and measured in backscattering geometry. The results are shown in figure 10. For the 2D nanostructures, the main Raman-shift peaking at 437 cm$^{-1}$, namely $E_2$ mode, decreased obviously compared with the plane surface. Meanwhile, the peak at 572 cm$^{-1}$, which is attributed to $A_1$ (LO) phonon, was also observed [25]. The results indicated that the crystalline structure in the sample surface was greatly damaged during the formation of the 2D nanostructures. The authors of [44] also studied the changes in crystalline structures during the formation of nanoripples. Cross-sectional transmission electron microscopy showed that the depths of nanoripples were in the range of 200–400 nm. The electron diffraction patterns...
revealed that the irradiated surfaces were capped by a layer (50–100 nm thick) of amorphous material, while in the inner region it was still of crystalline structure.

Compared with the crystalline structures, the energy state densities of the band tail in the amorphous materials increased and the tail moved deeper into the band gap. This increased the two-photon absorption process of the 2D nanostructures excited by the 800 nm femtosecond laser pulses. Therefore, the slope of the UV luminescence of 2D nanostructures shown in figure 7 was less than that of the plane surface of the ZnO crystal.

During the change from crystalline to amorphous structures, there were different kinds of defect states formed on the sample surface. As we discussed above, the laser ablation had an annealing effect on the 2D nanostructures. The density of oxygen vacancies could not increase obviously because the experiments were conducted in air. The main surface defect states were zinc interstitials and zinc vacancies. In addition, the defect states of zinc interstitials could enhance the UV emission, which was caused by the coupling between excitons and the donor zinc interstitials. Therefore, the enhancement of UV emission of the 2D nanostructures resulted from the enhanced optical absorption and the surface defect states of zinc interstitials.

4. Conclusions

In summary, we fabricated the 2D nanostructures on the surfaces of the ZnO crystal by the interference of three femtosecond laser beams, and obtained regular and uniform nanostructures by increasing the cross angles between any two laser beams. Compared with the plane surface of the ZnO crystal, the 2D nanostructures revealed an enhancement of the UV emission excited by 800 nm femtosecond laser pulses. The intensity of the UV emissions was comparable to the nanorods fabricated by the chemical vapor deposition method. We studied the mechanisms of the enhancement of UV emissions and found that they were mainly caused by the increase in optical absorption and the formation of surface defect states of zinc interstitials.

Recently, Shimotsuma et al [45] and Beresna and Kazansky [46] reported the birefringence of self-assembled nanostructures in glass induced by femtosecond laser pulses, and they
further demonstrated the potential applications of the nanostructures in the optical storage and the polarization-sensitive diffractive elements. We have fabricated different types of complex nanostructures by changing the laser polarization. In addition to the enhancement of optical absorption and the UV luminescence, these structures could also be used as uniaxial, biaxial and azimuthal micro-crystals, and have potential applications in the optical storage and complex birefringence elements in 2D and 3D geometries.

Acknowledgment

This work was supported by the National Natural Science Foundation of China (10874044, 10904038 and 11004060), the Shanghai Municipal Science and Technology Commission (09142200500, 09JC1404700, 08JC1408400 and 09ZR1409300) and the Twilight Project sponsored by the Shanghai Education Committee (07SG25).

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