Modified threshold of two-photon-pumped random lasing of ZnO nanorods by femtosecond laser ablation

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We investigated systematically the modification of the two-photon-pumped random lasing (TPPRL) behavior of ZnO nanorods through femtosecond (fs) laser ablation. The excitation wavelength dependence of the TPPRL behavior of ZnO nanorods was measured and the threshold of TPPRL before and after fs laser ablation was compared. With increasing excitation wavelength, a rapid increase in the threshold of TPPRL was observed at ~720 nm and it offers us the opportunity to significantly modify the two-photon luminescence from ZnO nanorods by fs laser ablation. The excitation wavelength dependence of the threshold was attributed to the wavelength dependence of the mean free path of photons in ZnO nanorods. When the fs laser ablation was carried out at 720 nm, it was found that the improvement in TPPRL could be achieved at excitation wavelengths longer than 620 nm while the TPPRL behavior became deteriorated for excitation wavelengths shorter than 620 nm. If the ablation and characterization wavelengths were chosen to be the same, then no obvious change was found for wavelengths shorter than 680 nm. However, a significant improvement in TPPRL could still be observed at 720 nm. The effects of ablation time and ablation method on the TPPRL behavior were also studied. It was revealed that the reduction in the threshold and the enhancement in the internal quantum efficiency could be realized by increasing ablation time and using continuous ablation. The results presented in this paper will be useful for the modification of the TPPRL behavior of random lasing media. © *2012 American Institute of Physics*. [http://dx.doi.org/10.1063/1.4752273]

I. INTRODUCTION

Random lasers have attracted great interest in the last decade due to their potential applications in the fabrication of optoelectronic functional devices.[1] Different from a conventional laser that is usually constructed with a gain medium and a cavity, a random laser relies on the feedback provided by the multiple scattering in a disordered medium. The initial studies on random lasers were carried out more than 20 years ago[2–4] and the term of random lasing was first introduced in 1995 by Wiersma, van Albada, and Lagendijk.[5,6] In general, random lasers can be classified into two types,[7] depending on the scattering strength or the coherence of light. The random lasing with coherent feedback was first reported by Cao *et al.* in 1998.[8,9] In practice, random lasing can be easily achieved by using nanosecond or picosecond lasers.[10–16] In comparison, the observation of random lasing is more difficult when femtosecond (fs) lasers are employed because of the short duration of pulses or gain.[17,18] From another point of view, the high peak power of fs lasers offers us the opportunity to realize two-photon-pumped random lasing (TPPRL) although the threshold may be higher than that obtained by using single-photon pumping. A detailed theoretical analysis based on the diffusion approximation model revealed, however, that the threshold of two-photon pumping could be smaller than that of single-photon pumping.[19] The underlying physical mechanism is that a smaller absorption coefficient leads to a larger gain volume. Thus, the thresholds in both cases are independent of absorption coefficient but dependent on the mean free path of photons at the pump or excitation wavelength. Basically, the threshold for two-photon pumping can be expressed as follows:[19]

$$I_{th} = \frac{\pi^2}{6}\frac{D(\lambda_{em})}{\eta(\lambda_{ex})D(\lambda_{ex})}. \quad (1)$$

Here, $D(\lambda_{\mathrm{em}})$ and $D(\lambda_{\mathrm{ex}})$ represent the diffusion coefficients of photons at the emission (λ_{em}) and excitation (λ_{ex}) wavelengths, $\eta(\lambda_{\mathrm{ex}})$ is the proportionality coefficient between the absorbed power and the gain rate. By using the relationship between the diffusion coefficient D and the mean free path of photons l_t which is given by[19]

$$D \approx \frac{v l_t}{3}. \quad (2)$$

Equation (1) can be easily rewritten as follows:

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$$I_{th} = \frac{\pi^2}{6} \frac{l_t(\lambda_{em})}{\eta(\lambda_{ex}) l_t(\lambda_{ex})}. \quad (3)$$

Here, the dispersion of the gain material is neglected and it is assumed that the speed of light in it is independent on wavelength, i.e., $v(\lambda_{em}) \approx v(\lambda_{ex})$. From Eq. (3), it can be seen that the threshold of two-photon pumping depends strongly on the mean free path of photons at both the emission and excitation wavelengths. It implies that one can modify or control the threshold of random lasing by changing the mean free path of photons. Basically, the mean free path of photons is inversely proportional to the scattering cross section σ and the concentration of randomly distributed scatters N, i.e., $l_t \propto 1/\sigma N$.[20] In practice, the modification of particle size can be easily achieved by using fs laser ablation.[21,22] Very recently, we demonstrated a significant enhancement in two-photon luminescence (TPL) by using fs laser ablation of ZnO nanorods.[23] It was shown that the significant enhancement in TPL after fs laser ablation could be utilized to realize optical data storage. A dramatic reduction in the threshold of TPPRL is responsible for the significant increase in TPL after fs laser ablation and it was suggested that the reduction in the threshold of TPPRL originates from the change in the mean free path of photons at both the emission and excitation wavelengths. So far, very few studies have been devoted to TPPRL and the validity of the excitation wavelength dependent threshold for two-photon pumping (i.e., Eq. (3)) has not been experimentally demonstrated. In addition, a systematic investigation of the modification of the threshold of TPPRL is still lacking. Also, it is interesting to find out whether the dramatic reduction in the threshold of TPPRL observed previously at 720 nm is a popular phenomenon or a special case.

In this article, we investigate systematically the excitation wavelength dependence of the threshold of TPPRL for ZnO nanorods before and after fs laser ablation. The effects of ablation method, including ablation wavelength, ablation time, and ablation interruption, on the threshold of TPPRL are clarified. The results and conclusions obtained in this work are useful for the control and modification of the thresholds of random lasers through fs laser ablation.

II. EXPERIMENTAL

The ZnO nanorods used in our study were synthesized by using the method reported previously.[24] The synthesis process for ZnO nanorods can be described in three steps. First, 250 ml aqueous solutions of zinc acetate ($ZnAc_2 \cdot 2H_2O$) and triethanolamine (TEA, $N(CH_2CH_2OH)_3$) with molar ratio of 1:2 were prepared and put inside two dropping funnels. Then, the two solutions were dropped within 2 h into a three-neck flask filled with water and subjected to a bath at 90 °C. Subsequently, the above suspension was continuously stirred for 1 h. Finally, the suspension containing ZnO seeds was aged for 12 h at room temperature. After filtration, the residue was washed three times with a lot of acidic and nonionic water and dried at 80 °C for 24 h to obtain ZnO nanorods. The SEM image of the synthesized ZnO nanorods is shown in Fig. 1. The average diameter and length of ZnO nanorods are measured to be ~250 nm and ~2 μm, respectively.

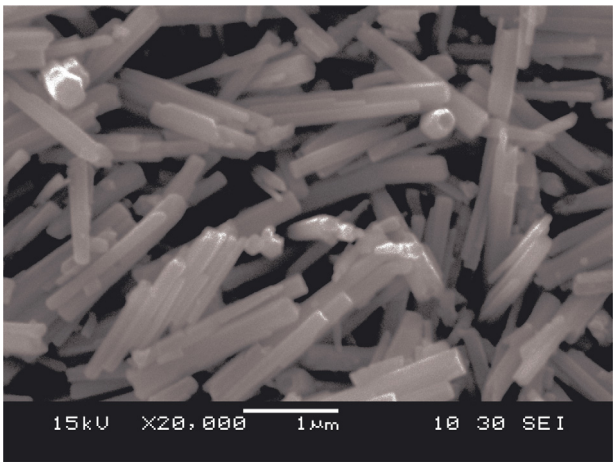

FIG. 1. SEM image of the synthesized ZnO nanorods.

For the measurements of TPL, ZnO nanorods were densely packed into a 100-μm-thick sample cell made of two cover glass slides. The tunable laser light from an optical parametric amplifier (OperA Solo, Coherent) with a duration of 100 fs and a repetition rate of 1 kHz was focused normally on the sample cell by using a lens with a focusing length of 150 mm. The sample cell was placed 20 mm away from the focus. The TPL from ZnO nanorods was detected at 45° with respect to the normal by using a fiber spectrometer with a resolution of 0.37 nm (USB2000+, Ocean Optics). The pump energy density was derived by accurately measuring the intensity distribution of the laser spot at the sample position by using a laser beam analyzer (HV1300UM, CDHC-Optoelectronics).

For the measurements of the mean free path of photons in the sample containing ZnO nanorods, we used a solid-state laser (Verdi-5, Coherent) and a He-Ne laser. A charge-coupled device (CCD) was employed to record the coherent backscattering (CBS) cone of the sample.

III. TPPRL OF ZnO NANORODS AT DIFFERENT EXCITATION WAVELENGTHS BEFORE fs LASER ABLATION

In experiments, we first measured the TPL of ZnO nanorods at different excitation wavelengths (λ_{ex}) ranging from 580 to 720 nm, as shown in Fig. 2(a). A sharp increase of TPL with increasing pumping energy density was observed for all excitation wavelengths except 720 nm. It was also confirmed that the sharp increase in TPL was accompanied with a dramatic decrease in the linewidth of TPL. This behavior indicates that TPPRL of ZnO nanorods can be achieved at excitation wavelengths shorter than 720 nm. The threshold of TPPRL for each excitation wavelength can be easily determined. For comparison, the dependence of the threshold of TPPRL on excitation wavelength is plotted in Fig. 2(b). It can be seen that the threshold of TPPRL increases with increasing excitation wavelength. For $\lambda_{ex} = 700$ nm, the intensity of TPL saturates rapidly at a pump energy density of ~15 mJ/cm^2. At

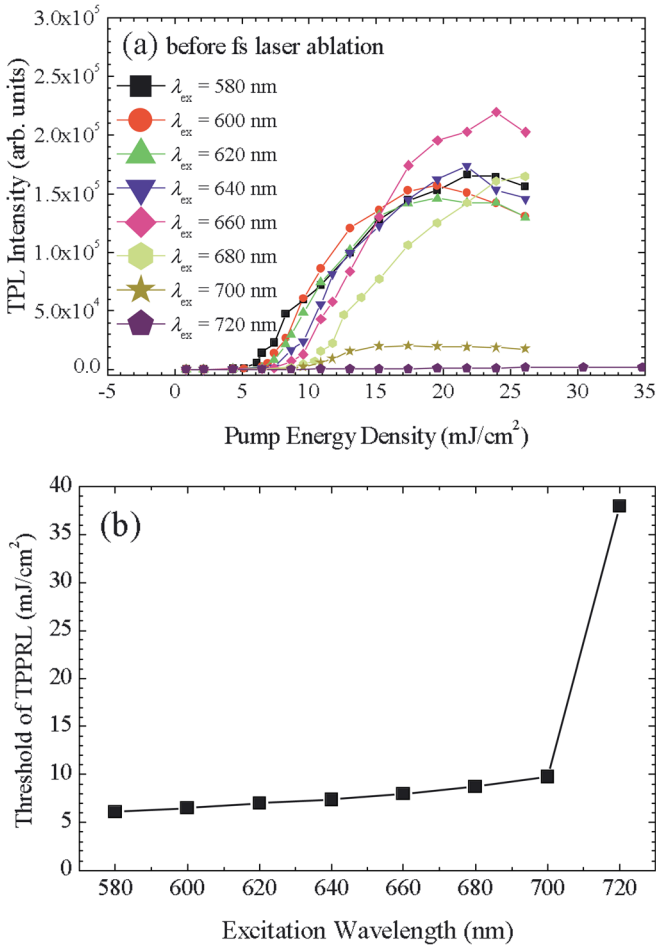

FIG. 2. (a) TPL intensity of ZnO nanorods as a function of pump energy density measured at different excitation wavelengths before fs laser ablation. (b) Wavelength dependence of the threshold of TPPRL of ZnO nanorods before fs laser ablation. Since the threshold of TPPRL at 720 nm is larger than the ablation threshold of ZnO nanorods which is estimated to be ~38 mJ/cm^2, we use 38 mJ/cm^2 as the minimum threshold of TPPRL at 720 nm in order to show the sharp increase of the threshold at this wavelength.

720 nm, we do not see TPPRL for pump energy density smaller than 38 mJ/cm^2, implying that the threshold of TPPRL at this wavelength is larger than 38 mJ/cm^2. For pump energy densities larger than 38 mJ/cm^2, a decrease in TPL was observed, indicating the ablation of ZnO nanorods. According to the theoretical analysis presented at the beginning, the difference in the threshold of TPPRL originates mainly from the wavelength dependence of the mean free path of photons at the excitation wavelength, as manifested in Eq. (3). The proportional coefficient $\eta(\lambda_{ex})$ in Eq. (3) characterizes mainly the internal quantum efficiency of ZnO nanorods at the excitation wavelength. The effect of this parameter is reflected in the slope of TPL intensity above the threshold. Although the absorption coefficients at different wavelengths are different, the theoretical analysis given in Ref. 13 clearly indicated that the threshold is independent on absorption coefficient even when single-photon pumping and two-photon pumping are compared. Therefore, the increase in the threshold of TPPRL with increasing excitation wavelength should reflect the wavelength dependence of the mean free path of photons in ZnO nanorods. It means that the mean free path of photons decreases slightly for wavelengths shorter than 700 nm and sharply at 700 nm. In order to confirm the validity of this conclusion, we have measured the mean free path of photons at two typical wavelengths (532 and 632.8 nm) that are easily accessed by employing a solid-state laser and a He-Ne laser. In Fig. 3, the CBS cones of the sample containing ZnO nanorods recorded by using a CCD are compared for the two wavelengths. The mean free paths of photons derived from the full width at the half maximum of the CBS cones are 5.5 ± 0.2 μm at 532 nm and 4.3 ± 0.2 μm at 632.8 nm, respectively. The mean free path of photons exhibits a weak dependence on the measurement point due to the nonuniformity of the sample. The ratio of the mean free path of photons at 532 nm to that at 632.8 nm is calculated to be 1.28. This value is very close to the ratio of the threshold at 640 nm to that at 580 nm that is estimated to be 1.22. In experiments, we did not measure the threshold of TPPRL at 532 nm. If we assume that the threshold at 532 nm is slightly smaller than that at 580 nm according to the excitation wavelength dependence of the threshold shown in Fig. 2(b), then the validity of Eq. (3), which implies an inverse proportion relationship between the threshold and the mean free path of photons at the excitation wavelength, is experimentally verified. Basically, the mean free path of photons is inversely proportional to the scattering cross section of ZnO nanorods.[20] From the wavelength dependence of scattering cross section calculated for ZnO nanorods, one does observe a sharp increase at ~700 nm but only for the case when the ZnO nanorod is parallel to the incident light.[23] This characteristics can be employed to interpret the sharp decrease of mean free path of photons at ~700 nm, which leads to the dramatic increase in the threshold of TPPRL. However, ZnO nanorods contained in the sample are expected to orient randomly and the sharp increase in scattering cross section at ~700 nm will disappear for other orientations. Therefore, more experiments are needed to clarify the sharp increase in the threshold of TPPRL or the dramatic reduction in the mean free path of photons observed at 720 nm.

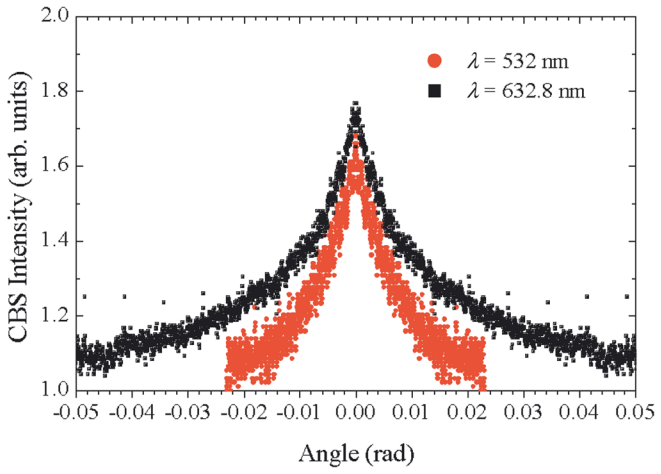

FIG. 3. CBS cones of ZnO nanorods measured at two different wavelengths (532 and 632.8 nm). The corresponding mean free path of photons can be derived from the full width at the half maximum of the CBS cone.

IV. TPPRL AT DIFFERENT EXCITATION WAVELENGTHS AFTER fs LASER ABLATION AT 720 nm

In Sec. III, we have compared the threshold of TPPRL at different excitation wavelengths. Previously, we have shown that the threshold of TPPRL can be significantly reduced at 720 nm through fs laser ablation of ZnO nanorods.[23] It is interesting to know whether this behavior can be observed at other excitation wavelengths. For this reason, we have performed fs laser ablation of ZnO nanorods at 720 nm with a laser fluence of ~114.25 mJ/cm^2 and an ablation time of 10 s which corresponds to the irradiation of 10 000 pulses. After that, we measured the TPPRL behavior of the ablated ZnO nanorods at different excitation wavelengths ranging from 580 to 720 nm. The results are presented in Fig. 4. For all wavelengths, one can clearly see TPPRL behavior with different thresholds ranging from 4.75 to 9.50 mJ/cm^2. The lowest threshold (~4.75 mJ/cm^2) was observed at 680 nm while the largest threshold was found at 720 nm (~9.50 mJ/cm^2). For wavelengths shorter than 680 nm, a monotonous increase in the threshold was observed with decreasing excitation wavelength. At 580 nm, the threshold of TPPRL approached the value at 720 nm.

As discussed in our previous paper, the dramatic reduction in the threshold of TPPRL mainly originates from the change in the mean free path of photons occurring at both the emission (390 nm) and excitation (720 nm) wavelengths after fs laser ablation.[23] Therefore, the decrease or increase in the threshold of TPPRL depends strongly on the change in the mean free path of photons induced by fs laser ablation. A detailed comparison of the thresholds of TPPRL before and after fs laser ablation is presented in Fig. 5 for different excitation wavelengths. It is noticed that there is no change in the threshold at 620 nm. For wavelengths longer than 620 nm, a reduction in the threshold is found after fs laser ablation. However, a reduction in the threshold of TPPRL is not observed for all wavelengths. Instead, a slight increase in the threshold is found at 600 and 580 nm. This behavior is in sharp contrast with what we observed previously at 720 nm where a significant reduction in the threshold was achieved after fs laser ablation. This wavelength dependence of threshold implies that a significant modification in the threshold of TPPRL can only be achieved at wavelengths where a dramatic change in scattering cross section or mean free path of photons occurs after fs laser ablation. In addition, it indicates that an improvement in TPPRL is not expected for all wavelengths and the effect of fs laser ablation on the threshold of TPPRL becomes less pronounced at short wavelengths.

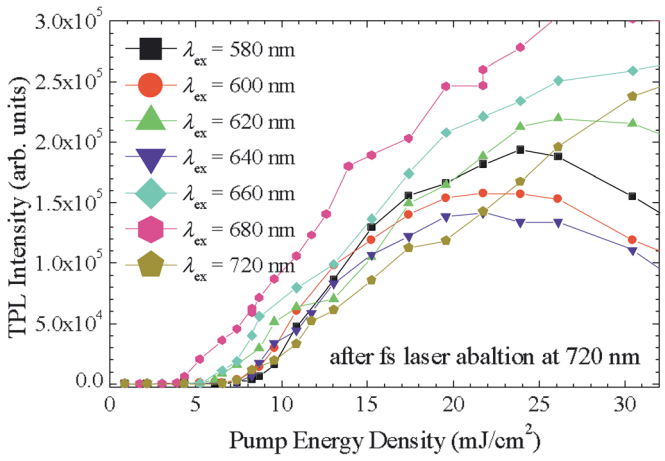

FIG. 4. TPL intensity of ZnO nanorods as a function of pump energy density measured at different excitation wavelengths after fs laser ablation at 720 nm.

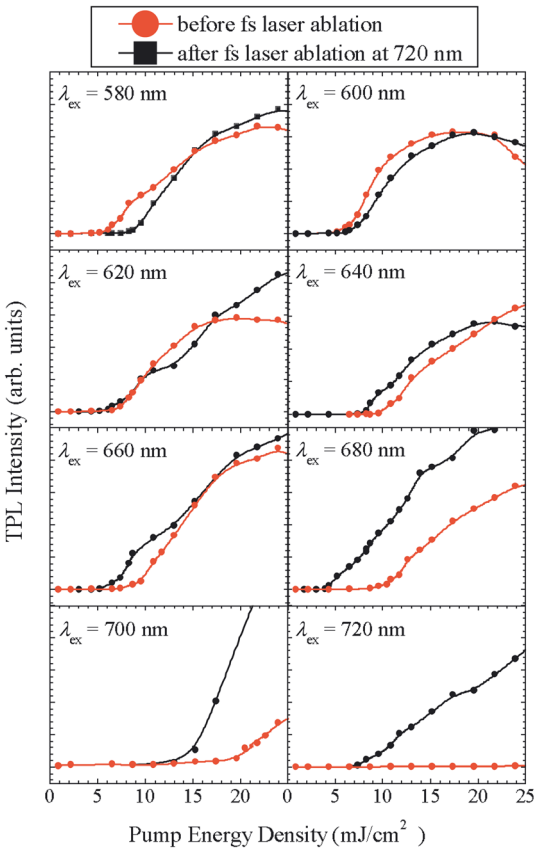

FIG. 5. Comparison of the TPPRL behavior of ZnO nanorods at different excitation wavelengths before and after fs laser ablation at 720 nm.

V. TPPRL AT DIFFERENT EXCITATION WAVELENGTHS AFTER fs LASER ABLATION AT THE SAME WAVELENGTH

We have found in previous studies that the length of short ZnO nanorods (more precisely referred to as nanodisks) obtained by fs laser ablation exhibits a linear dependence on the ablation wavelength.[23] Thus, ZnO nanodisks with a smaller size will be obtained if a shorter ablation wavelength is used. In experiments, we first ablated ZnO nanorods with fs laser pulses of different wavelengths (λ_{ab}) ranging from 580 to 720 nm. Then, the TPPRL behavior of the ablated ZnO nanorods was characterized by exciting the ablated region of the sample with the same wavelength. We have compared the TPPRL of ZnO nanorods before and after fs laser ablation at several typical wavelengths, as shown in Fig. 6. As compared with the results shown in Fig. 5, it is found that no obvious change in the threshold of TPPRL is observed for most wavelengths except for 720 nm. This

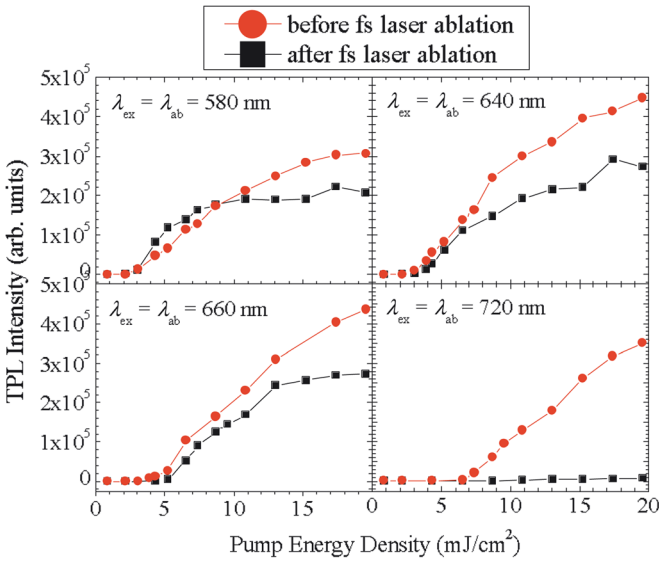

FIG. 6. Comparison of the TPPRL behavior of ZnO nanorods at different excitation wavelengths before and after fs laser ablation at the same wavelength.

behavior indicates that the improvement in TPPRL can be easily achieved by using fs laser pulses with a long wavelength. When fs laser pulses with a short wavelength is used, the size of the resulting ZnO nanodisks is small, leading to a small scattering cross section at the emission wavelength of ZnO (~390 nm) and a large threshold.

VI. EFFECTS OF ABLATION TIME AND ABLATION METHOD ON THE THRESHOLD OF TPPRL

In the experiments described above, the ablation time was chosen to be 10 s, corresponding to the irradiation of 10 000 pulses. Under this condition, the ablation of ZnO nanorods is not complete, implying that some ZnO nanorods remain unablated. The SEM image of ZnO nanorods after the irradiation of 10 000 pulses is shown in Fig. 7. One can easily find some unablated ZnO nanorods. In this case, the mean free path of photons in the mixture of ZnO nanorods and nanodisks (ablated ZnO nanorods) is difficult to estimate through the calculation of scattering cross section. It is reasonable to assume, however, that the mean free path of photons in the mixture is between the value of ZnO nanorods and that of ZnO nanodisks. The reduction in the threshold of TPPRL originates from the increase in the mean free path of photons after fs laser ablation. If we fix the laser fluence and increase the ablation time, a further reduction in the threshold of TPPRL is expected due to the complete ablation of ZnO nanorods.

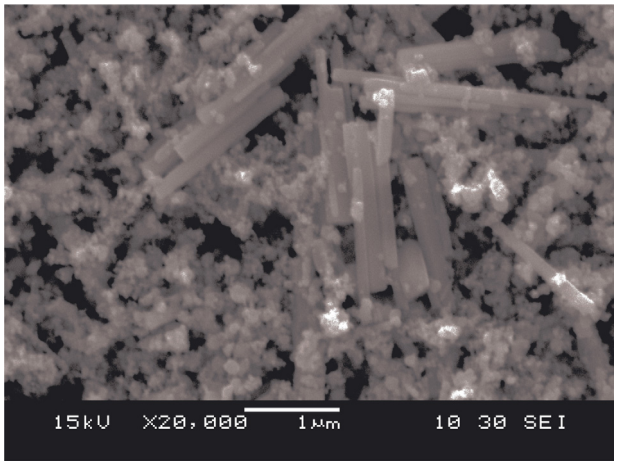

FIG. 7. SEM image of ZnO nanorods after irradiation of 10 000 fs pulses with a fluence of ~114.25 mJ/cm^2.

In order to find out the effects of ablation time and ablation method on the threshold of TPPRL, we have characterized the TPPRL behavior of ZnO nanorods after experiencing different ablation processes. The results for two wavelengths of 660 and 720 nm are presented in Figs. 8(a) and 8(b), respectively. In both cases, we fixed the laser fluence at 118.75 mJ/cm^2 and ablated ZnO nanorods in three different ways. In the first experiment, we irradiated ZnO nanorods with 10 000 pulses and characterized the TPPRL behavior at low laser fluences which are smaller than the ablation threshold (~38 mJ/cm^2). In order to see the effects of ablation time, this process was repeated six times, corresponding to a total ablation time of 60 s. For the second experiment, we chose to irradiate 30 000 pulses on a new measurement point and characterized the TPPRL behavior at low laser fluences. This process was performed two times,

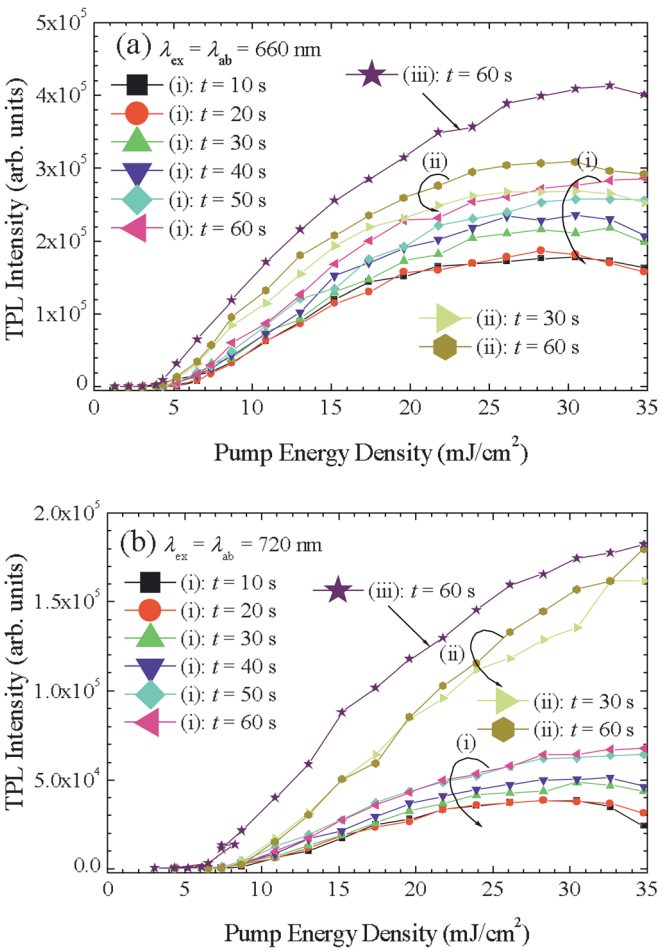

FIG. 8. Pump energy density dependence of the TPL intensity of ZnO nanorods ablated at 660 nm (a) and 720 nm (b) with three different methods which are indicated by (i), (ii), and (iii). The total ablation time for each ablation method is chosen to be the same (60 s).

giving a total ablation time of 60 s. In the final experiment, we characterized the TPPRL behavior of ZnO nanorods on a different measurement point after irradiating them with 60 000 pulses. From Fig. 8(a), it can be seen that a slight decrease in the threshold is observed after ablating ZnO nanorods six times. It is noticed that the increase of TPL with increasing pump energy density becomes faster (or the slope becomes larger) and the saturated TPL becomes larger after each ablation process. It implies the enhancement in the internal quantum efficiency. This effect becomes more obvious when the second ablation method is used and the largest improvement in both the threshold and the internal quantum efficiency is achieved when the third ablation method is employed. When the ablation time is increased from 10 s to 60 s with no interruption, the threshold of TPPRL is reduced from 5.70 to 4.28 mJ/cm^2 while the saturated TPL is enhanced more than two times. In comparison, the improvement is not significant when the ablation process is separated into several steps.

For $\lambda_{ex} = 720$ nm, we can find similar phenomena but the effects of ablation time and continuous ablation appear to be more significant, as shown in Fig. 8(b). A six-fold increase in the saturated TPL is observed when the ablation time is increased from 10 s to 60 s. In addition, the threshold is reduced from 9.50 to 7.13 mJ/cm^2.

VII. CONCLUSION

We have systematically investigated the modification and control of the threshold of TPPRL of ZnO nanorods through fs laser ablation. The excitation wavelength dependence of TPPRL behavior was measured and a rapid increase in the threshold of TPPRL was observed at ~700 nm. This phenomenon offers us the opportunity to significantly modify the TPL from ZnO nanorods through fs laser ablation. The wavelength dependence of the threshold was attributed to the wavelength dependence of the mean free path of photons. When the fs laser ablation was carried out at 720 nm, it was found that the improvement in TPPRL could be achieved at wavelengths longer than 620 nm while the TPPRL behavior became deteriorated for wavelengths shorter than 620 nm. If the ablation and measurement wavelengths were chosen to be the same, then no obvious change was found for wavelengths shorter than 680 nm. However, a significant improvement could still be observed at 720 nm. We have also studied the effects of ablation time and ablation method on the TPPRL behavior. It was revealed that the reduction in the threshold and the enhancement in the internal quantum efficiency could be realized by increasing ablation time and using continuous ablation. The results presented in this paper will be helpful for modification and control of the TPPRL behavior of random lasing media.

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