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The photoluminescence of ZnSe bulk single crystals excited by femtosecond pulse*

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This paper reports on the photoluminescence spectra of ZnSe single crystal with trace chlorine excited by the femtosecond laser pulse. Three emission bands, including second-harmonic-generation, two-photon-excited peak and a broad band at 500–700nm, were detected. The thermal strain induced by femtosecond pulse strongly influences the photoluminescence of ZnSe crystal. The corresponding strain ε in ZnSe crystal is estimated to be about 8.8 $\times 10^{-3}$ at room temperature. The zinc-vacancy, as the main point defect induced by femtosecond pulse, is successfully used to interpret the broad emission at 500–700nm. The research shows that self-activated luminescence possesses the recombination mechanism of donor–vacancy pair, and it is also influenced by a few selenium defects and the temperature. The rapid decrease in photoluminescence intensity of two-photon-excited fluorescence and second-harmonic generation emission at lower temperature is attributed to the fact that more point defects result in the thermal activation of the two-photo-absorption energy converting to the stronger recombination emission of chlorine–zinc vacancy in 500–700nm. The experimental results indicate that the femtosecond exciting photoluminescence shows a completely different emission mechanism to that of He–Cd exciting luminescence in ZnSe single crystal. The femtosecond laser exhibits a higher sensitive to the impurity in crystal materials, which can be recommended as an efficient way to estimate the trace impurity in high quality crystals.

Keywords: photoluminescence, femtosecond pulse, ZnSe crystal, defects

PACC: 7280E, 7855E, 7865K

1. Introduction

ZnSe semiconductor has recently attracted substantial attention due to its technological importance for optoelectronic applications such as blue-light semiconductor lasers and light-emitting diodes.[1,2] Practical ZnSe-based devices seriously depended on not only high-quality ZnSe crystals but also the mechanism of degradation process of relative luminescence,[3,4] and the latter caused a great enthusiasm such that a large number of studies were devoted to the photoluminescence (PL) of ZnSe.[5−8] According to the luminescence mechanism deduced from the interaction of ZnSe polycrystals with a continuous laser, some parameters, such as temperature,[5,9] excited intensity,[10] photon energy[11−13] and crystal purity[14] were recognized as important factors to influence the luminescence. However, when an ultrashort pulse laser with high intensity was employed as the exciting source, the various luminescence mechanisms and new physical effects would rise in the focus volume because of the basic quantum mechanical formulation rather than the semiclassical Boltzmann equation.[15] Recently, two aspects of the interaction of infrared laser ultrashort pulses with transparent materials have received a great deal of research attention. The first is the nonlinear optical phenomenon, which opens a novel way for ZnSe crystal in the luminescence application.[16−18] Secondly, ultrashort laser pulses could induce structure changes or modification, defect formation, and physical damage.[19−22] However, the actual mechanism and effects of the interaction with a femtosecond laser pulse remain unclear to many crystals, such as ZnSe crystals. The PL spectrum, as a sensitive tool to defect and impurity in crystals, should help us to reveal some details of the

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interaction.

In this paper, the ZnSe single crystal will be pumped by a near-infrared femtosecond periodic-pulse laser. The PL property of ZnSe bulk crystal and some effects on the interaction of femtosecond pulses with ZnSe crystal will be revealed.

2. Experimental

The ZnSe bulk single crystal used in this study was grown by chemical vapour transport with the assisting of $Zn(NH_4)Cl_5$.[23] The as-grown ZnSe has (111) orientation surface and 1.42–1.52mm in thickness. The PL measurements were performed at various temperature using a mode-locked Ti:Sappohire laser (Spectra Physics spitfire amplifier) with centre wavelength of 800nm, repetition rate of 1kHz and pulse width of 50fs. The laser spot on the sample had a diameter of about 50μm, yielding an incident laser excitation intensity of about 4GW/cm^2. The emission from the ZnSe sample was measured by a monochromator with a 600 grooves·mm^{-1} grating. A band pass filter was used to prevent the laser light from entering the spectrometer. The electric signal from the photomultiplier of the spectrometer was sent to the computer spectra acquisition system. As a contrasting investigation, He–Cd laser and Ar ion laser were also used as the exciting sources.

3. Results and discussions

The emission spectra of ZnSe single crystal at room temperature is shown in Fig.1. Figure 1(a) illuminates the PL spectrum excited by the He–Cd laser with 30W, which is dominated by the blue emission band with two branches. The PL peak located at 439nm is associated with the excitonic emission line,[24] which is connected with free excitation and neutral donor bound excitation of Cl^0X[25,26] The emission at 418nm (3.0eV) is analogous to the broad line described in the literature.[27] This is the important fingerprint of high crystalline quality of ZnSe sample.[25] An interesting point is that we do not detect the self-activated (SA) emission resulted from Cl_{Se}-V_{Zn}-complexes. Considering that the compound $Zn(NH_4)_3Cl_5$ containing chlorine element serves as the transport agent in the crystal growth process, we find that some chlorine atom could ineluctably incorporate into the lattice, so a conclusion can be preliminarily made that ZnSe sample does not contain zinc-vacancy. According to Fig.1(b), when the femtosecond pulse is used as the excited source, ZnSe single crystal exhibits completely different emission from Fig.1(a). The femtosecond PL spectrum consists of three main emission bands peaking at 403.5nm, 472.5nm and about 603.5nm respectively, which will be discussed in detail in the following text.

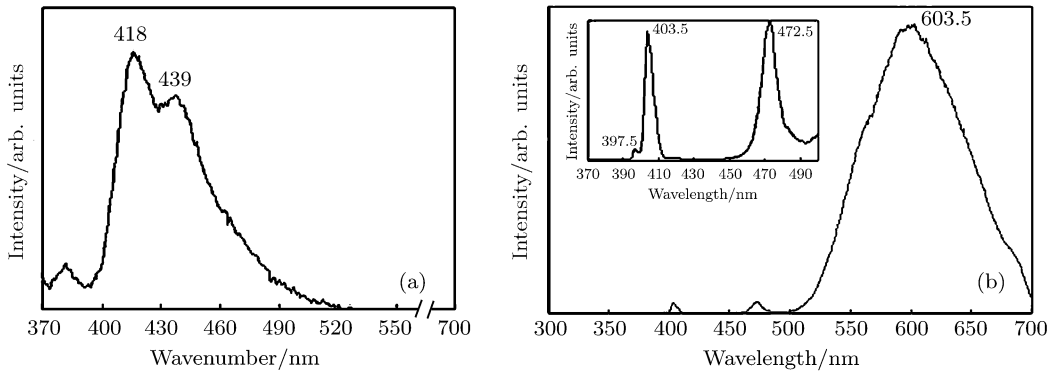


Fig.1. PL spectrum of ZnSe single crystal at room temperature: (a) He–Cd laser with 30W as the excited source, (b) femtosecond laser pulse as the excited source. Insert is detail in the region 370–500nm.

3.1. Two-photon-excited fluorescence

Two-photon-excited fluorescence (TPEF), which peaks at 472.5nm, can be easily observed in Fig.1(b) because the two-photon absorption coefficient in ZnSe is fairly large.[28] In Ref.[29], the TPEF spectra of a ZnSe single crystal consists of a band with two maxima near 2.690 and 2.610eV at 300K, while in Ref.[30], the TPEF spectra exhibits a wide asymmetric band with the peak of 475nm in the region of 460–490nm at room temperature. In our case, the TPEF spectrum is almost a symmetric band in the region of

460–480nm at 300K. Moreover, it exhibits a particular character with the temperature. Fig.2 shows the typical TPEF emission in ZnSe sample at various temperature ranges from 300K to 10K. According to Fig.2, we can find several interesting case about the TPEF emission. Firstly, a shift to higher energies at lower temperature is observed in Fig.2. In order to reveal the origin of this shift, the temperature dependence of the peak position for TPEF emission are obtained by monitoring PL spectra at 440–480nm. A linear fit to TPEF peaks position data corresponding to the temperature of 300–80K yields a solid line with slop of -4.2×10^{-4} (see Insert A in Fig.2). It should be noted that the line slop of -4.2×10^{-4}eV·K^{-1} is in accordance with the temperature dependence of ZnSe energy gap ($\mathrm{d}E_\mathrm{g}/\mathrm{d}T = -4.0 \times 10^{-4}$eV·K^{-1}, $T = 77 - 300$K[31]). This indicates that the temperature behaviour of TPEF emission is completely dependent on the temperature effects of ZnSe energy gap. Secondly, all TPEF are almost the symmetric band at the temperature range of 80–300K. However, at the temperature lower than 80K, for example 60K, 20K and 10K, the TPEF splits into two branches. The insert B of Fig.2 reveals that TPEF band at 10K can be fitted to two Gaussian peaks (see the broken line in insert B of Fig.2) with maxima at 447.6nm (2.77eV) and 455.6nm (2.72eV). According to Ref.[29], the splitting peaks are attributed to excitons bound to zinc vacancies. In our experiment, the lower temperature and the higher incident laser intensity promote the emergency of zinc vacancies in zinc crystals. Moreover, starting from high to low temperature, we find that the TPEF exhibits a decreasing trend in its intensity. This will be discussed together with the emission at 500–700nm in the following text.

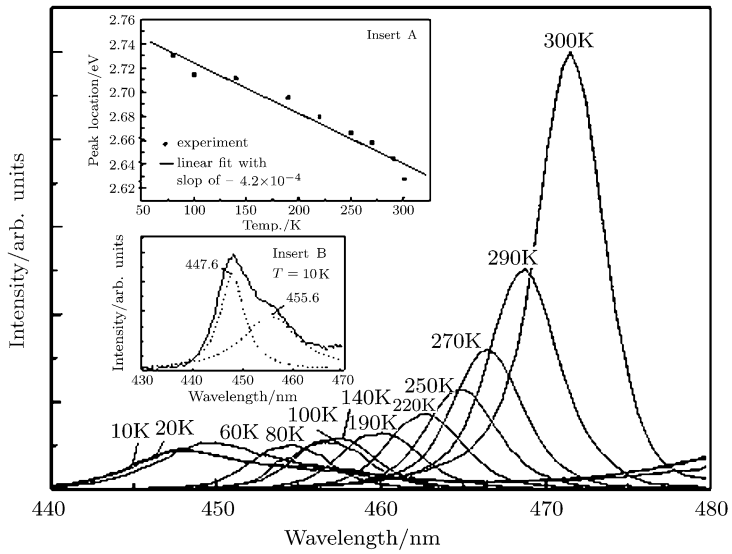


Fig.2. TPEF spectra of ZnSe single crystal at various temperature. Insert A: temperature dependence of TPEF position at 80–300K. Insert B: the splitting peaks of TPEF at 10K.

According to Fig.2, the higher intensity of TPEF can be detected at room temperature. This means that the laser pump power can easily be converted into TPEF emission at higher temperature. The coefficient of the exciting radiations conversion into TPEF equals about 10^{-7} at 300K, being the same as the report in Ref.[30]. The similar result indicates that the species of defects induced by femtosecond pulse possibly can be similar to that in the sample in Ref.[30] because the two-photon emission is sensitive to bulk defects.[31] The induced bulk defects in our sample will be revealed further by PL spectra in Section 3.3.

3.2. Second-harmonic-generation emission

Because the polarization selection rulers for the second-harmonic-generation (SHG) is the same as those for two-photon absorption,[33] the second-harmonic emission spectrum emerges, as showed in the insert of Fig.1(b), which consists of two peaks located at 403.5nm (3.073eV) and 397.5nm (3.119eV)

with explicitly different intensity. We believe that the two peaks should be attributed to the internal strain in bulk crystal caused by the heat effect from the interaction of femtosecond laser pulse with crystal. According to recent theoretical analysis, the principal effect of a femtosecond laser pulse is multiphoton ionization in the materials, associating with the strong electric field,[34,35] resulting in free electron heating and energy absorption in wide-band-gap solid.[35,36] Thus, the laser energy is absorbed by the sample although ZnSe crystal is transparent to the exciting laser radiation. Then, a significant fraction of the absorbed energy is likely to be converted into heat, resulting in an increase of the lattice temperature in the laser exposed region, and the internal stress in ZnSe crystal is induced. Because of the induced stress, the 2P exciton state splits into two states, i.e. the heavy-hole (HH) and light-hole (LH) 2P exciton states.[33,37] This is in accordance with the fact that the softness of ZnSe materials makes it susceptible to strain. Therefore, the two SHG peaks are related to the HH and LH 2P exciton states. Because the stress could easily relax in the particle, ZnSe power sample could not generate two branches of SHG signal.[18]

Considering the law of conservation of energy and the adjustment of gap, when the excitation wavelength is 800nm (1.54eV), we find that the HH would be the dominant transitions.[38] Therefore, the high-intensity peak corresponds to the HH 2P exciton and the weak-intensity peak to the LH one in the insert of Fig.1(b). According to the position of two SHG peaks, the splitting energies $\Delta E_{\mathrm{LH}}^{\mathrm{2P}}$ between the HH and LH excitons can be determined to be 46meV. Further, we can estimate the strains value by using the following relation:[39]

$$\Delta E_{\mathrm{LH}}^{\mathrm{2P}} = 5.2\varepsilon(\mathrm{eV}), \tag{1}$$

So, the strain ε in ZnSe bulk crystal caused by femtosecond laser pulse is estimated to be about 8.8×10^{-3} at room temperature. The result indicated that the heat strain is larger by about 8.7 times than that in ZnSe/GaAs film caused by the lattice mismatch.[40] The induced strain is so large that it easily caused the emergence of defects in the ZnSe crystal.

3.3. Physical nature of the emission at 500–700nm

In Fig.1 (b), a broad emission band in 500–700nm dominated the PL spectrum of ZnSe single crystal. According to the energy position, it is easy to attribute to this emission to chlorine donor-vacancy recombination.[16] But it was not detected in the He–Cd laser excited PL spectrum (Fig.1(a)). This affords us the two following important points of information on the ZnSe sample.

1) The ZnSe single crystal used in this measure is a slightly Cl doped sample because the chlorine element, as the donor from the transport agent $Zn(NH_4)_3Cl_5$, could partially incorporate into the lattice during the growth.[23] In fact, further experimental results indicate that the trace Cl content could not be detected through the inductively coupled plasma (ICP) and the energy dispersive analysis of x-rays (EDAX) energy spectra. This reveals that the ZnSe sample had a high crystalline quality with trace chlorine and almost does not contain zinc vacancy and other impurity. This is in accordance with the PL spectrum of the Cl doped ZnSe film with high quality, where SA emission was inhibited seriously.[41,42]

2) The strong emission at 500–700nm wavelength indicates that zinc vacancy is induced by the laser irradiation, so that it recombines with the trace Cl donors existing in the lattice of ZnSe crystal to produce a strong emission. This interpretation is supported by recent reports that defect, vacancy, colour centres, and structural changes can be easily created by femtosecond laser[19−22]. This dominant emission indicates that, in our case, the zinc vacancy is the main point defect induced by femtosecond laser pulse. The formation of zinc vacancy is related to two aspects. One is its lower formation energy, the other is that zinc vacancy is the most promising defect centre to relax the lattice strain and distortion in ZnSe:Cl crystal[43].

In order to reveal the characterization of the emission at 500–700nm, the luminescence spectrum of ZnSe crystal is measured at various temperatures. This result is shown in Fig.3. In Fig.3(a), a peculiar feature of the emission at 500–700nm is observed that there is a shift to short wavelength (blue shift) at higher temperature, which is opposite to the red shift of the band gap of ZnSe. This emission exhibits a slight increase in intensity with decreasing temperature. This is attributed to the contribution of donor–acceptor transitions caused by thermal exciton dissociation enhancing at lower temperatures due to laser-induced zinc vacancy.[44] In our case, at about 20K, the emission starts to decease due to the thermal emptying of the zinc vacancy and Cl donor levels, which is

a typical characterization of donor-acceptor transition of V_{Zn}-Cl_{Se}-complexes. This further confirms that zinc vacancy is the dominative defect in ZnSe crystal under the experimental conditions. Figure 3(b) shows that the temperature dependence of the integrated intensity of 500–700nm emission is an inverse function of temperature, and the relationship of the emission intensity depending on the inverse temperature is agreeable to the Arrhenius plot. This indicates that the temperature dependent behaviour of this emission is the nonradiative recombination mechanism.[4,42,45] The broken solid line in Fig.3(b) is the best fit to the experimental data with the model described by Eq.(2):[42,45]

$$I = I_0[1 + C\exp(-E/k_{\mathrm{B}}T)]^{-1}, \qquad (2)$$

where I_0, C are the constants, E is the activation energies, k_{B} is the Boltzmann constant and T is the temperature. The best fit yields C values of 42.5 and an activation energy of 68.8meV respectively. It should be noted that the estimated value of E is in agreement with the energy difference between the band gap and the energy position TPEF spectrum in ZnSe, for example 61meV (2.67–2.61eV) at room temperature and 60meV (2.74–2.68eV) at 190K respectively. This implies that the temperature dependent behaviour of the emission in 500–700nm is relative to TPEF and the valence band. But the difference between 61meV and the estimation energy of 68.8meV at room temperature indicates to us that there must be some factors influencing this emission, as revealed in the following text.

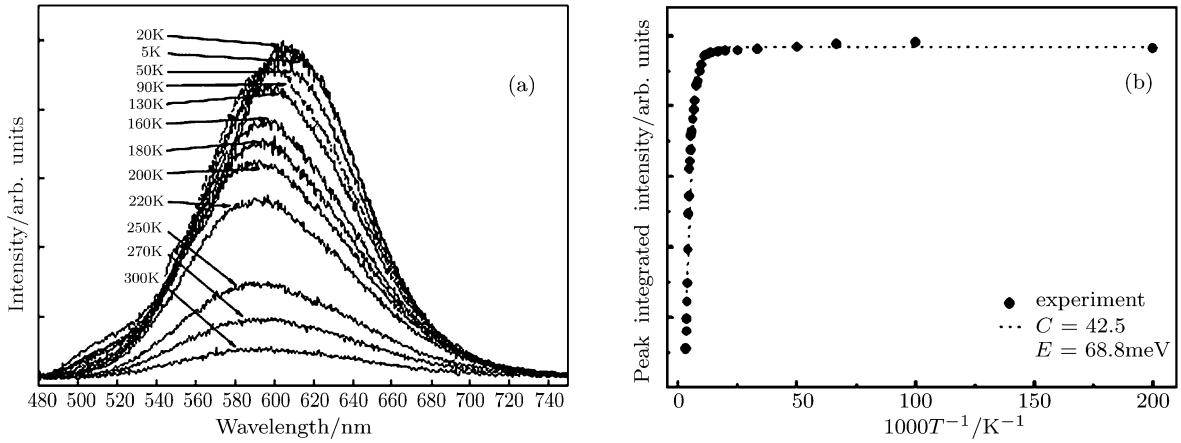


Fig.3. (a) Luminescence spectra of ZnSe single crystal at 500–700 wavelength in various temperatures. (b) The intensity of 500–700 emission as a function of inverse temperature.

Figure 4 shows the full width of half maximum (FWHM) of the emission in 500–700nm at various temperatures. The FWHM of this emission band exhibits a gradual increase with the increasing temperature. The following theoretical relation Eq.(3) is used to estimate the FWHM temperature dependence of this emission band.

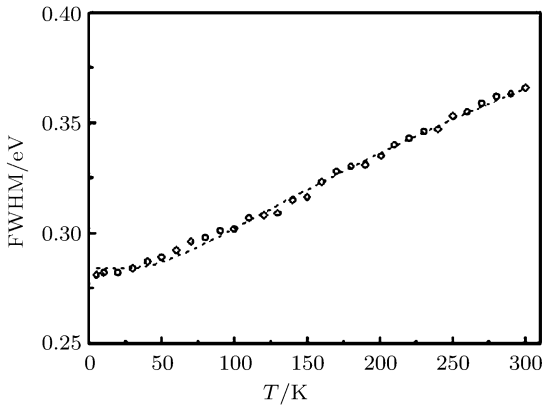


Fig.4. The FWHM of the emission in 500–700nm at various temperatures.

$$\mathrm{FWHM} = A + B\coth(h\nu/2k_{\mathrm{B}}T)^{1/2}, \qquad (3)$$

where A, B are the constants, $h\nu$ is the vibrational energy, k_{B} is the Boltzmann constant and T is temperature. The best fit yields a vibrational energy of 14.6meV and A, B values of 0.19, 0.09 respectively. The fitting curve is shown by a broken line in Fig.4. A little estimated vibrational energy of 14.6meV in the above relation for ZnSe:Cl indicates that the variety of FWHM with temperature is very weak.

The above results indicate that the emission band at 500–700nm exhibits a special mechanism and influ-

ences both TPEF and SHG, being different from the SA emission of Cl doping ZnSe/GaAs film in Ref.[41]. In our experiment, with the decreasing temperature and the continuance of laser radiation, at some temperature (∼200K for this sample), the strong emission of SHG become very weak so that it could not be detected. TPEF also exhibits a lower intensity, and the emission of 500–700nm dominated the whole spectrum with decreasing temperature. Based on above analysis about the difference between the activation energy (61meV) at room temperature and its estimated value (68.8meV), we believe that the increase in intensity of 500–700nm emission band and the degradation of TPEF with decreasing temperature can be attributed to two aspects. One is the special origin of the point defect of zinc vacancy deduced by femtosecond laser pulse; the other is related to the energy conversion between SA emission and TPEF. In our case, the amount of zinc vacancy varies with different temperature and the radiating time in the laser spot, and the intensity of the strain induced by the laser pulse at low temperature is likely to be higher than that at room temperature, because at lower environment temperature, the temperature difference between the laser spot and its around sample body is larger than that at a higher environment temperature. This is in agreement with the experimental fact that the higher SA emission intensity emerges at lower temperature. The rapid decrease in TPEF intensity at lower temperature is due to that the more point defects result in the thermal activation of the two-photon-absorption energy converting to the stronger recombination emission of chlorine–zinc vacancy via the conduction band in 500–700nm than TPEF emission. In the process of this energy conversion, the conduction band plays an important role. This result has been approved by the fact that the TPEF emission exhibits a gradual decrease with the decreasing temperature in Fig.2, being a different trend from SA emission shown in Fig.4. Due to the same polarization rules for both SHG and TPEF, SHG emission would also become weak with the rapid decrease two-photon-absorption energy at lower temperature.

According to experimental data, zinc vacancy is the important defect induced by femtosecond laser pulse to dominate the PL spectra of ZnSe single crystal. In order to understand if zinc vacancy can exist permanently in femtosecond-irradiated ZnSe sample, further studies were carried out. Figure 5 shows the PL spectra of the femtosecond-irradiated ZnSe crystal excited by He–Cd laser and Ar ion laser. It is easily

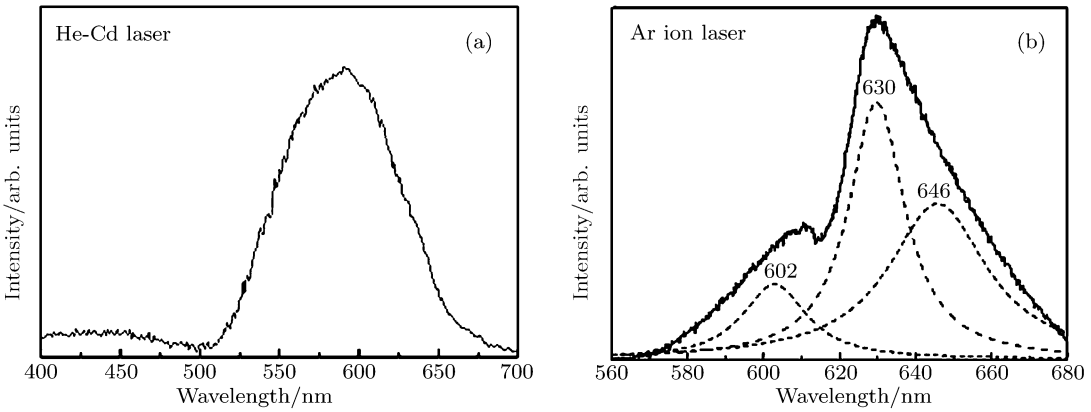


Fig.5. PL spectrum of the femtosecond-irradiated ZnSe single crystal: (a) He–Cd laser as the excited source, (b) Ar ion laser as the excited source.

found that SA emission in the range of 500–700nm dominate the PL spectrum excited by 325nm wavelength, which is distinct from Fig.1(a), revealing the permanent existence of zinc vacancy and the serious damage in irradiated ZnSe crystal. Because the femtosecond pulse damnifies the quality of ZnSe crystal, the exciton emission is inhibited in Fig.5(a). Moreover, Fig.5(b) offers an evidence of other defect in irradiated ZnSe crystal, and Ar ion laser is used as the exciting source at room temperature. The PL spectrum consists of three Gaussian peaks locked at 602nm, 630nm and 646nm respectively. The three peaks are covered in Fig.5(a) because the irradiated ZnSe crystal possesses a broad emission at 500–700nm

when He-Cd laser serves as an excited source. The peak at 602nm is attributed to the emission of Cl-V_{Zn} complex, and the peak at 630nm is related to a complex donor (V_{Se}-V_{Zn})- V_{Zn},[46] while the origin of the peak at 646nm can be linked with the interaction of V_{Se} and V_{Zn}. Therefore, some selenium-related vacancy can also be induced in irradiated ZnSe sample. Considering the higher formation energy,[43] we find that a small quantity of selenium-related defects can appear in the irradiated ZnSe crystal. Considering the determination of SA activation energy, we can deduce that a few selenium defect is also a factor of influencing SA emission and expanding the difference between the activation energy (61meV) and its estimated value (68.8meV) at room temperature.

According to above data, an important suggestion is that the PL of ZnSe crystals excited by femtosecond laser pulse exhibits a higher sensitivity to impurity, such as chlorine in crystals materials, while the nature of this higher sensitivity is the recombination of impurity–vacancy pair. Therefore, the femtosecond laser can be used as a tool to estimate the trace impurity of high quality crystals.

4. Conclusion

The PL spectrum of high quality bulk ZnSe single crystal was measured, and the femtosecond laser pulse was used as the excitation source. Three emission bands, including SHG, two photon excited peak and a broad band at 500–700nm, were detected and investigated. The thermal strain induced by femtosecond pulse strongly influences the PL of ZnSe crystal. The strain ε in ZnSe crystal is estimated to be about 8.8×10^{-3} at room temperature. The zinc-vacancy, as the main point defect induced by femtosecond pulse, is successfully used to interpret the broad emission at 500–700nm, and the nature of this emission is the recombination of donor–acceptor pair. A few selenium defects and the temperature are proved to be the important factor of influencing SA emission. The rapid decrease in PL intensity of TPEF and SHG emission at lower temperature is attributed to that the more point defects result in the thermal activation of the two-photo-absorption energy converting to the stronger recombination emission of chlorine–zinc vacancy in 500–700nm than TPEF emission via the conduction band.

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