The effects of La^{3+} doping on luminescence properties of PbWO$_4$ single crystal

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Abstract

X-ray-excited luminescence (XEL), thermoluminescence (TL), photoluminescence under excitation of UV light and X-ray photoelectron spectroscopy (XPS) measurements were conducted on PbWO$_4$ (PWO) single crystals doped with La^{3+} at a series of concentration levels. With the increase of doping levels, the intensity of PWO in the XEL or the excitation–emission spectra was found to decrease accordingly, especially in the case of heavy doping. TL peaks in the range from room temperature to 200°C disappeared after the doping with La^{3+}. A splitting of the La3d XPS peak in the heavy La^{3+}-doped samples was observed and proposed to be responsible for that. At a high doping level, La^{3+} would occupy W-sites besides Pb-sites and thus induce a self-compensation via pairs $[(La_W^{3+})''' - (La_{Pb}^{3+})^\bullet] - (V_O)^{\bullet\bullet}$ or clusters $[3(La_{Pb}^{3+})^\bullet - (La_W^{3+})''']$, where nonradiative recombination was previously reported to occur. The mechanism of the influence on luminescence was also discussed in this paper.

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1. Introduction

Lead tungstate single crystal, PbWO$_4$ (PWO), has revealed itself as a good scintillating materials for usage in high-energy physics detectors, especially in compact muon solenoid (CMS) detector at large hadron collider (LHC) in CERN, because of its excellent features, such as high density, short radiation length and fast decay time [1,2]. In order to improve its scintillating properties, a large number of researches, especially doping treatments, have been carried out [3]. Kobayashi et al. first reported radiation hardness and transmittance in short wavelength region (320–450 nm) could be significantly improved by La^{3+} doping [4]. Since then, trivalent ions have become the most intensively studied dopants [5,6] mainly because they are considered to replace the Pb^{2+} ions and to compensate the Pb^{2+} deficiency taking account their radius (La^{3+} 1.15 Å, Pb^{2+} 1.20 Å) and electronegativity. Our previous works have demonstrated that trivalent ion La^{3+} would occupy

Pb^{2+}-site and induce excess positive charge into Pb-sublattice, which is compensated by V_{Pb} (Pb vacancy) through the formation of defect complexes $[2(La_{Pb}^{3+})^\bullet - V_{Pb}'']$ [7]. Researches also confirmed that such La-doped PWO crystals show complete absence of slower decay components and only a prompt one is present in scintillation decay (decay time = 1 ns), resulting in likely usage as subnanosecond scintillator emitting in the blue spectra region [8]. Subsequently, Kobayashi et al. proposed a new heavy and radiation-hard Cherenkov radiators based on PbWO$_4$ doped with appropriate trivalent ions (such as La^{3+}, Gd^{3+}, Y^{3+}). Heavy doping with trivalent ions considerably reduces scintillation light intensity to a level as or lower than the Cherenkov light intensity, by keeping the excellent radiation hardness and causing no significant degradation in the short wavelength [9]. Recently, a beam test of this PWO Cherenkov radiator has been carried out [10]. This is a very valuable result because doping with appropriate trivalent ions might bring PWO crystals to be employed in a new practical usage in high-energy physics as an alternative promising Cherenkov radiators crystal material because of its less expensive cost, high

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radiation hardness, and well-developed growth technique of large-volume single crystal.

The aim of this paper is to present the luminescence characteristics of PWO single crystals doped with La^{3+} in different concentration levels, such as X-ray-excited luminescence (XEL), thermoluminescence (TL) and photoluminescence (PL). Based on microstructure of PWO, different coordinate environment and doping mechanism of La^{3+} are discussed, especially heavy doping ones. X-ray photoelectron spectroscopy (XPS) measurements were employed to elucidate this doping mechanism.

2. Experimental

All the investigated PWO crystals were grown from an initial mixture of stoichiometric PbO and WO_3 powders ($>99.999\%$) in a platinum crucible with the equivalent conditions using modified Bridgeman method. Colorless and crack-free PWO crystals including pure and doped by La^{3+} in a concentration of 50, 150, 200, 800 at.ppm, 1.0 and 2.5 mol% were obtained, respectively. PWO crystals were cut into plates of about $15 \times 15 \times 2$ mm^3, all facets were polished and their opposing large faces were set perpendicular to the c-axis of the crystal for XEL and PL measurements. Excitation and emission spectra were taken with a fluorescence spectrophotometer, a Perkin-Elmer LS-55, which has an Xe lamp as an excitation source. XEL spectra were measured by using an X-ray-excited spectrometer, FluorMain, where an F-30 movable X-ray tube (W anticathode target) was used as the X-ray source, and operated under the same condition (80 kV, 4 mA) at room temperature (RT). A layer of Tyvek paper wrapped the crystals with only an exposed facet coupled to the gating of monochromator. The luminescence spectra were obtained by 44 W plate grating monochromator and Hamamatsu R928-28 photomutiplier with the data acquired by computer.

Samples in XPS and TL experiments were cut into plates of about $10 \times 10 \times 1$ mm^3 in dimension at the same position from parent bulk PWO crystal with two facets polished. In order to avoid or minimize contamination, it is critical for all samples to be kept fresh in this experiment. The XPS measurements were carried out on Micro-Lab 310-F made by VG Scientific Ltd., using AlKa X-ray. A hemispherical analyzer and a multi-channel detector obtained energy spectra of the emitted electrons. The TL glow curves measurement after UV light irradiation were made at RT by FJ-427A thermoluminescence spectrometer with a heating rate of $2°C/s$. A 1000 W high-pressure mercury lamp was used as UV light irradiation source to irradiate the samples for 15 min.

3. Results and discussion

TL glow curves of pure PWO and PWO : La^{3+} after UV irradiation were measured (Fig. 1). There exist at least two TL peaks between RT and 200°C in the pure PWO. These peaks disappeared for all PWO : La^{3+} studied here, which is a significant lowering sign of intrinsic lattice defects in this doped PWO crystals. The suppression of these peaks indicates disappearance of the color centers with a result of inhibiting the absorption bands and improvement of the performance (e.g. transmittance or radiation hardness). The same effect of La^{3+} doping has been confirmed by Baccaro et al. [11] in PWO with La^{3+} doping 100–150 ppm.

XEL spectra of samples were given in Fig. 2. For further studies, each XEL spectrum could be defined as a superposition of at least two Gaussian components
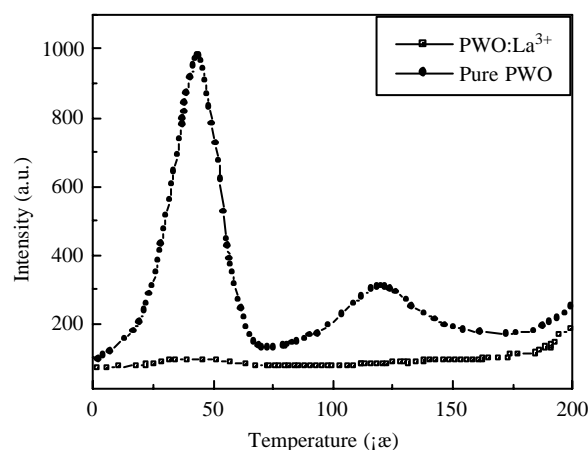

Fig. 1. The TL glow curves of pure PWO and La^{3+}-doped PWO crystals between RT and 200°C after UV irradiation for 15 min.
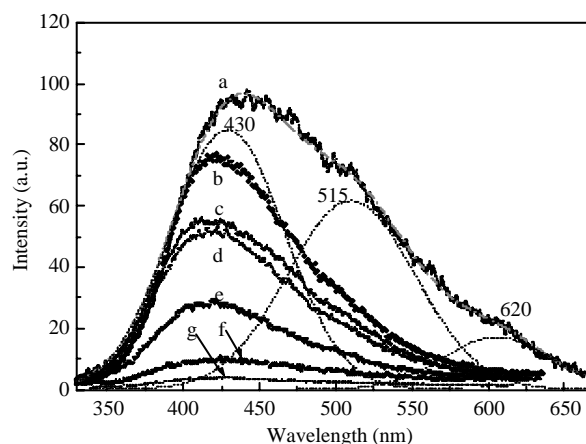

Fig. 2. The X-ray excited luminescence spectra of pure PWO crystal and La^{3+}-doped PWO samples at RT. (a) Pure PWO, (b) PWO doped with 50 at.ppm La^{3+}; (c) 150 at.ppm; (d) 200 at.ppm; (e) 800 at.ppm; (f) 1.0 mol% and (g) 2.5 mol% of La^{3+}. XEL spectra of pure PWO are also shown with Gaussian decomposition (dot lines).
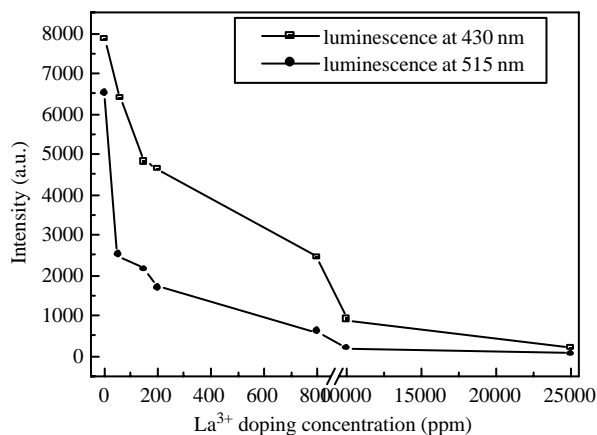
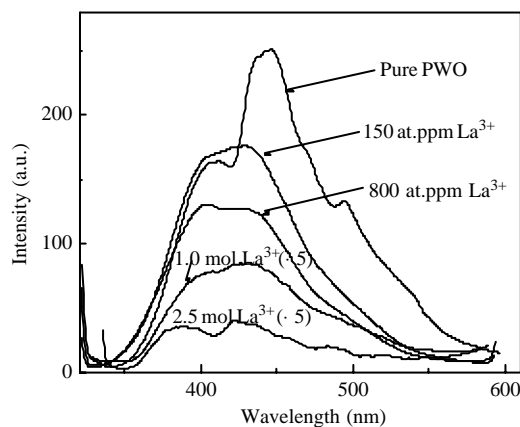Fig. 3. Dependence of X-ray excited luminescence intensity on the La^{3+}-doping concentration in PWO : La^{3+}.

Fig. 4. The emission spectra (excited by UV light 310 nm) of pure PWO single crystal and La^{3+}-doped PWO samples at RT.

Fig. 5. The excitation spectra (monitoring 420 nm emission) of pure PWO crystal and PWO : La^{3+} samples at RT.

with the same full-width at half-maximum (FWHM) and approximate peaking at 430 nm (blue luminescence) and 515 nm (green luminescence) wavelength. Dependence of XEL intensity on doping concentration is plotted in Fig. 3 by the data of Gaussian components. XEL spectrum of pure PWO in Fig. 2a can be defined as a superposition of three Gaussian components, peak 1 at 430 nm, peak 2 at 515 nm and peak 3 at 620 nm (red luminescence). With increasing La^{3+}-doping concentration from 50 at.ppm to 1.0 and 2.5 mol%, the luminescence of PWO : La^{3+} crystal became weaker and weaker and were dominated by blue luminescence components. The PWO : La^{3+} 1.0 mol% decreased its luminescence intensity by a factor of about 8 compared to that of pure PWO. Luminescence of PWO : La^{3+} 2.5 mol% presented nearly a straight line and was so weak that can hardly be detected under XEL measurement. At the same time, in all La^{3+}-doping PWO, the green luminescence was suppressed more heavily with respect to the blue one and the red one that disappeared completely.

Emission spectra under 310 nm UV light and excitation spectra by monitoring 430 nm emission are shown in Figs. 4 and 5, which have a variety of results similar to that of XEL. In the emission spectra of pure PWO, a stronger emission at 450 nm bands and a weaker one at around 400 nm bands were observed. With the La^{3+} doping, emission bands from 450 to 600 nm were greatly suppressed and emission around 420 nm became the dominated component on the emission spectra of PWO : La^{3+}. When La^{3+}-doping concentration increases up to 2.5 mol%, two separate emission peaks in blue luminescence region were presented. So we could imagine that in heavily La^{3+}-doped PWO, there might be some changes to regular tungsten groups $[WO_4]^{2-}$, which are assumed to be the blue luminescence centers in tungsten scheelites [12]. On excitation spectra of 430 nm emission, excitation peak at 310 nm shifts to
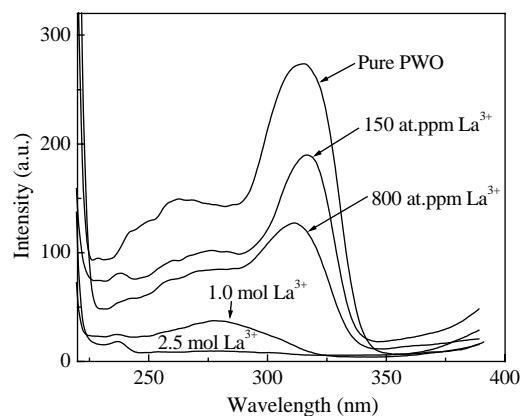
short wavelength, becomes weaker and weaker and almost disappears on that of PWO doped with La^{3+} 1.0 and 2.5 mol%. This indicates heavy doping La^{3+} annihilate intrinsic lattice defects and intensively suppresses exciton luminescence efficiency. The excitation mechanism might be changed in the heavy doped PWO : La^{3+} due to probable new trap centers in the PWO lattice, which are involved in the energy transfer and storage process in this crystal [13]. As mentioned later, in heavy PWO : La^{3+}, La^{3+} could substitute the Pb-sites first and further occupy the W-sites. So this new substitution might badly disturb the creation of the exciton, which was based on $[WO_4]^{2-}$ groups, and result in restraining the corresponding absorption and emission. However, further experiments were necessary to elucidate the excitation mechanism.

La $3d$ XPS measurements in La^{3+}-doped samples of 150 at.ppm, 1.0 and 2.5 mol% were carried out. The spectra results are shown in Figs. 6 and 7. The spectrum of PWO doped with 150 at.ppm La^{3+} has a nearly straight line (Fig. 6) because of its lower doping
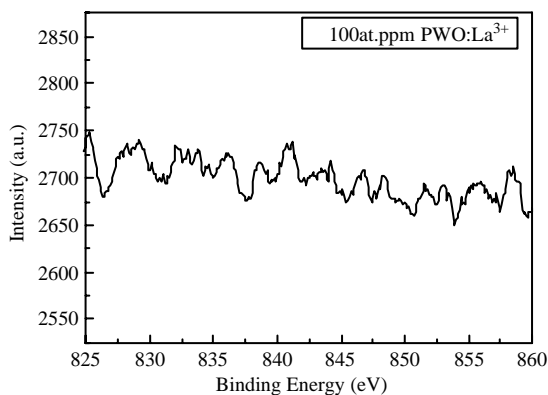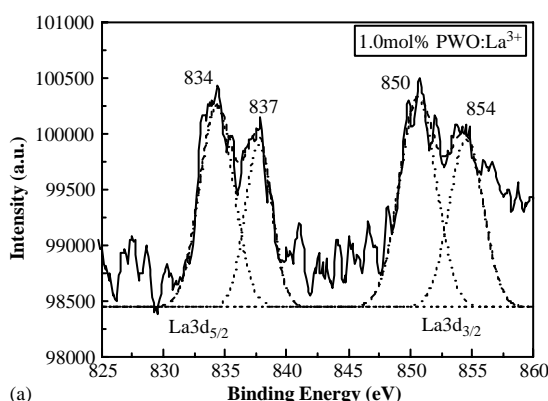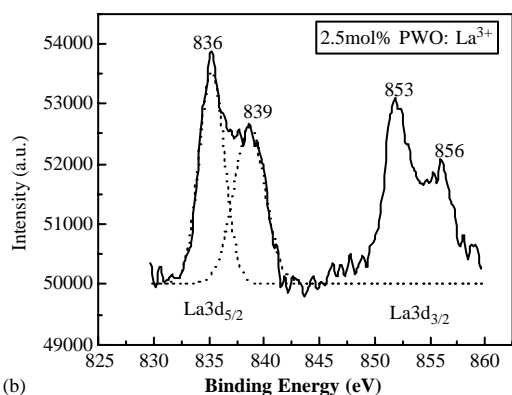
Fig. 6. The XPS spectra of La$3d_{5/2}$ and La$3d_{3/2}$ electrons of PWO : La^{3+} 150 at.ppm.

Fig. 7. The XPS spectra of La$3d_{5/2}$ and La$3d_{3/2}$ electrons of PWO : La^{3+} 1.0 mol (a), La$3d_{5/2}$ and La$3d_{3/2}$ were fitted by two Gaussian curves (dash lines); and XPS spectra of La$3d_{5/2}$ and La$3d_{3/2}$ electrons of PWO : La^{3+} 2.5 mol. La$3d_{5/2}$ was fitted by two Gaussian curves (dash lines).

concentration. But that of 1.0 and 2.5 mol% doped samples present two separate peaks at about 836 and 853 eV (Fig. 7), attributed to the two spin orbits of La$3d_{5/2}$ and La$3d_{3/2}$, respectively [14]. Either peak can be defined as a superposition of two Gaussian components. We could suppose that the splits of XPS La$3d_{5/2}$ and La$3d_{3/2}$ might be caused by the following three

cases: (1) La^{3+} standing in the interstitial site; (2) existence of interstitial oxygen nearby the La^{3+}, and (3) La^{3+} replacing W-site except that of Pb-site. However, it is impossible for the La^{3+} standing in the interstitial site taking account of its large ionic radius (1.16 Å) that is similar to that of Pb^{2+}. Moreover, this interstitial site needs other ions to combine with the interstitial La^{3+}. Hence bad deformation of PWO lattice can occur. In the tetragonal stolzite PWO crystal, the distance of Pb–O (2.580 or 2.637 Å) is by far greater than that of W–O (1.795 Å), so if the interstitial oxygen truly stands in the W–O shell, it is impossible for about 3.3 eV chemical shift of La $3d_{5/2}$ caused by the interstitial oxygen considering the shift of interstitial oxygen O $1s$ XPS (~ 0.8 eV) [15]. So it seems to be reasonable that La^{3+} should occupy the W-site with the exception of Pb-sites to explain the chemical shift of La $3d_{5/2}$ and $3d_{3/2}$ in XPS experiments. Moreover, similar evidence of (La$_W^{3+}$) was also obtained by our co-operator in the radial structure function (RSF) measurement of La^{3+} during the extended X-ray absorption fine structure (EXAFS) experiment of PWO : La^{3+} (from private communication, unpublished). Esaka [16] has found that conductance of Pb$_{1-x}$La$_x$WO$_{4+x/2}$ increases rapidly with increase of x up to a value ($x = 0.2$) and then falls down when x is larger to some extent ($x = 0.4$), This might also be caused by the substitution of La^{3+} in W-site.

It is well known that during crystal growth, evaporation of PbO may result in lead deficiency. In general, upon lower La^{3+}-doping concentration, for example 50, 150, 200 at.ppm, La^{3+} ion is supposed to occupy the Pb^{2+}-site and formed dipole complexes $[2(La_{Pb}^{3+})^{\bullet} - V_{Pb}'']$ [7]. While increasing La^{3+}-doping concentration, more and more V_{Pb} could be produced to keep the charge balance in PWO lattice. However, PWO keeps the Scheelite structure well even after heavily doping with La ion by 15 mol% [17] or in Pb$_{0.7}$La$_{0.3}$WO$_{4.15}$ ceramics [16]. Upon heavier doping, more and more V_{Pb} should be produced to keep the charge balance, which might exceed the ability to accommodate for the Scheelite structure PWO crystal [18]. So in order to keep Scheelite structure well, La^{3+} ions could occupy the other coordinate site except for the Pb site in PWO lattice, e.g. W-site.

Taking into account of the large difference of ionic radius between La^{3+}(1.16 Å) and W^{6+}(0.63 Å), is it possible that La occupies the W-site? It seems difficult for a random or irregular substitution of La in W-sites in heavy La-doping PWO. As the large La ions certainly behave differently from the higher valent W ions, a corresponding structural reorganization seems reasonable. The as-grown PWO crystal has a Scheelite-type structure (Fig. 8) with a and c values of 5.456 and 12.020 Å, respectively. The Scheelite structure is so flexible that it has the ability to easily adjust itself to
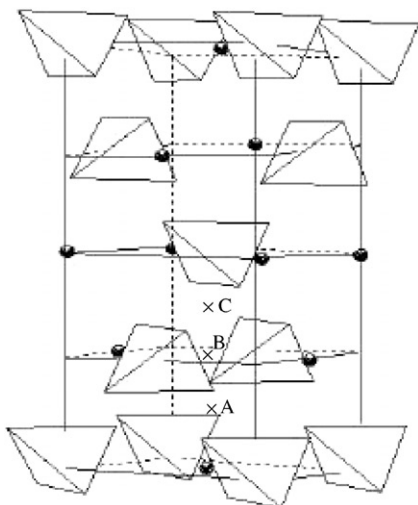
Fig. 8. The unit cell of $PbWO_4$ crystal structure. The black balls are Pb sites; X: A, B, and C show empty positions in the lattice.

tolerate different kinds of defects (exintrinsic, or nonstoichiometry defects). From the viewpoint of plane structure, all the oxygen atoms can be regarded to form multiple O ion planes perpendicular to *c*-axis. The distance of W–O (bond length 1.795 Å) in the isolated tetrahedral along *c*-axis is $1.795/\sqrt{3} \sim 1.04$ Å. Two adjacent tetrahedra is approximately $12.02/4 \sim 3.0$ Å, which means that the distance between two O planes may be at least $3.0 - (2 \times 1.04) \sim 0.9$ Å. With regard to the peculiarity of the structure, there exists great permissibility for the Sheelite structure to accommodate the large ions, e.g. La, standing at the W-sites by relaxing itself, where the V_O might be created and the other three oxygen ions shifted outwards from the original position in the tetrahedra, resulting in a badly deformed coordination environment. For example, 1 mol% La-doped PWO get the PLO8 polyhedron and WO_4 tetrahedron rotate anticlockwise by about $57.8°$ along their S4 axis compared with the pure PWO [19]. The deformed tetrahedra could be compensated and stabilized by the adjacent La–O polyhedron preexisting due to substituting of La at the Pb-sites through the junction of V_O. The negative charge created by the assumptive substitution of La in W-site might be stabilized and compensated by the positive preexisting La_{Pb} and V_O introduced by means of long distance compensation.

So the La ions occupying the W-sites like that of Pb-sites might come into another stable state by means of lattice relaxation. The PWO crystal lattice could get a relaxation to reduce defect-induced lattice strains due to heavy La doping, in which a local superstructure within the Scheelite structure could be formed. In our previous works, we have observed this superstructure in 1 mol% La doping PWO [20]. This superstructure is embedded in the PWO lattice just like "island", which is favored by

the local La ion aggregates and might get charge balance mainly in the form of self-compensation. In fact, PWO with heavy La doping could be grown with good optical quality.

We would like to give another well-known example of $LiNbO_3$: Mg. In lower doping, Mg^{2+} ions could enter the Li^+-sites. However, in higher doping levels (> 5 mol%), the self-compensation solution modes would be significantly preferred [21]. Mg^{2+} would occupy the Nb^{5+}-sites despite the large differences of the charge numbers and electronegativity, $+2$, 1.17 and $+5$, 1.2, respectively. The computer simulation [22] also confirmed this substitution, where the Mg_{Nb} could be in small defect aggregates.

In fact, the first introduced positive center $(La_{Pb}^{3+})^{\bullet}$ would also stabilize and compensate partly the $(La_W^{3+})'''$ defect. Heavy La^{3+} doping gives much more chance for the La^{3+} ions to close up each other, thus forming self-compensation pairs (dimer) $[(La_W^{3+})''' - (La_{Pb}^{3+})^{\bullet}] - (V_O)^{\bullet\bullet}$ or cluster (La small aggregates) $[3(La_{Pb}^{3+})^{\bullet} - (La_W^{3+})''']$. The so-called self-compensation pairs or clusters might act as "killing sites" [13] where effective nonradiative recombination of free electrons and holes occurs, hence intensively suppress the luminescence of PWO crystal by enormously decreasing its light yield. With increasing La^{3+} dopant in PWO crystal, effective nonradiative recombination augments resulting in awfully feeble luminescence intensity, as was verified in the results of XEL and emission of UV excitation (Figs. 2 and 4).

Moreover, it is well known that when the rare earth (RE) ions are doped in crystals with a sufficiently high concentration, it might lead to creation of RE–RE self-compensation ion pairs by means of an electron exchange force and result in the creation of nonradiative center that influences luminescence properties. This phenomenon occurs commonly in many kinds of rare earth ions doped laser, phosphate crystals and silicate scintillating glasses [23,24] and scintillation crystals [25,26]. For example, in scintillation crystal $Bi_4Si_3O_{12}$ (BSO) with Ce-doping level of 0.4 at.%, Ce-doped BSO crystal presents intensive depression of the light output and shortening of the decay constant, and other luminescence properties due to the local aggregation of Ce ions or the creation of more extended "Ce-rich" defects [25].

From these viewpoints, we would like to suggest that the substitution of La to W-site seems more reasonable. However, many more experiments and calculations of computer simulation are really needed.

4. Conclusions

La^{3+} doping in PWO could dramatically suppress the luminescence intensity of PWO crystals. Doping at a low

concentration level, the trivalent ion La^{3+} may substitute for the Pb^{2+}, with the excess charge compensated by intrinsic V_{Pb} for the formation of defect complexes $[2(La_{Pb}^{3+})^{\bullet} - V_{Pb}'']$, and this La-doped PWO shows a lower luminescence intensity compared with pure one. However, heavy La^{3+} doping into the PWO crystals might also cause La^{3+} self-compensation by substituting for the W-site (La_W^{3+}) and create La self-compensation pairs $[(La_W^{3+})''' - (La_{Pb}^{3+})^{\bullet}] - (V_O)^{\bullet\bullet}$ or clusters $[3(La_{Pb}^{3+})^{\bullet} - (La_W^{3+})''']$, at which new effective recombination takes place, and thus severely degrades the luminescence.

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