

Luminescence Properties of Phosphate Phosphor: Barium Tungstate Doped with Dy

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Abstract

A series of barium-tungstate-based phosphors doped with different concentrations of Dy³⁺ were synthesized by solid-state reaction method. Photoluminescence properties and decay lifetime of Dy³⁺-doped BaWO₄ samples were studied. The results indicated that luminescent properties of BaWO₄:Dy³⁺ depended on the Dy³⁺ concentration, and the inner energy could transfer from WO₄²⁻ to Dy³⁺. The quality of the light was checked by estimating CIE parameters, and the results showed that BaWO₄:Dy³⁺ was a potential candidate as blue-green luminescent materials in white LED because of its excellent emission spectrum excited by UV light.

Keywords

LED, BaWO₄:Dy³⁺, Phosphors, Decay Lifetime, Luminescent Properties

1. Introduction

Recently, metal tungstates have received much attention due to its luminescence properties, structure performance and its wide application in various fields [1]-[3]. As matrix, tungstate is rather promising for optoelectronic application in which it can emit blue light. Among these metal tungstates, barium tungstate with a tetragonal scheelite-like structure possesses good chemical and thermal stability [4]-[6]. The BaWO₄ crystallites with good crystal properties can be prepared by a simple and convenient electrochemical method [7] [8]. For example, BaWO₄ luminescent materials are applied as luminescent matrix by doping lanthanide ions, which have been widely investigated and show excellent luminescence properties [9].

The BaWO₄ luminescent materials doped with Tb³⁺ or Eu³⁺ have been reported that they can be synthesized by a simple hydrothermal route, and show potential applications in the field of phosphor-converted white LEDs [10] [11]. Alkaline-earth metal tungstate AWO₄ (A = Ca²⁺, Sr²⁺, Ba²⁺) was used to study as phosphor self-excited under UV excitation, exhibiting blue or green light [12]. Up to now, Rare-earth (Nd³⁺, Tm³⁺, Er³⁺, Ho³⁺ and

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Yb³⁺) doped AWO₄ (A = Ca²⁺, Sr²⁺, Ba²⁺) phosphors have been successfully synthesized and studied the energy transfer mechanism in the matrix as well as the connection between luminescence properties and doped ion [13]-[15]. Otherwise, Dy³⁺ ions have two dominant emission bands and the yellow and blue emission intensities can be adjusted. Hence it is possible to obtain white light emission with Dy³⁺-activated luminescence materials [16] [17].

The existence of the inner energy transfer from the host to Dy³⁺ ions with BaWO₄ as matrix, including the luminescence properties and its internal energy transfer mechanism, is attracting more research interests. In this paper, a series of BaWO₄ phosphors with different Dy³⁺ doping concentrations are prepared by solid state method and their photoluminescence properties are investigated under UV excitation.

2. Experimental

A series of Dy³⁺-doped barium tungstate phosphors were prepared by the conventional solid-state-reaction method. The reagents including BaCO₃ (A. R.), WO₃ (A. R.) and Dy₂O₃ (99.9%) were used as the raw materials without any further purification. Raw materials with stoichiometric ratio (Ba_{1-x}WO₄: xDy³⁺ (x = 0, 0.01, 0.02, 0.03, 0.04, 0.05, 0.06)) were weighed, sufficiently mixed and ground together in an agate mortar. The mixture was annealed for 2 h at 500°C and re-annealed at 1000°C for 8 hours, and then was slowly cooled down to room temperature. As a result, white phosphor samples were produced.

The phase structure of the product was characterized by using XD-2 X-ray powder diffractometer (CuK α , 36 KV, 20 mA). A scan rate of 0.02°/s was applied to record the patterns in 2 θ range 10° - 70°. The excitation and emission spectra of as-synthesized phosphors BaWO₄:Dy³⁺ were recorded on Japan's Hitachi F-7000 fluorescence photometer. Luminescence decay curves were measured with a PLS920P spectrometer, using nanosecond flash-lamps for excitation. All measurements were performed at room temperature.

3. Results and Discussion

The XRD patterns of the Ba_{1-x}WO₄:xDy³⁺ (x = 0 ~ 0.06) prepared by solid state reaction method were shown in **Figure 1**. It can be seen that the XRD diffraction peaks of the samples matched well with the standard card of BaWO₄ (PDF 43-0646), and the diffraction peak intensity were high without any other additional peak of Dy₂O₃ [18]. Therefore, the as-prepared BaWO₄ samples were crystallization with pure phase that is tetragonal phase structure, belonging to the I41/a (88) space group. The result shows that the Dy³⁺ ions in these samples should have substituted the Ba²⁺ ions sites, because small quantities of Dy³⁺ can enter the crystal lattice and do not change the crystal structure of the matrix BaWO₄. A small right-shift of diffraction peak with the increasing Dy³⁺ concentration appeared in **Figure 1**. This may be due to the fact that the ionic radius of the Dy³⁺ (91.2 pm) is smaller than that of Ba²⁺ (135 pm) in the samples, causing smaller diffraction angle based on Bragg's law ($d\sin\theta = k\lambda$).

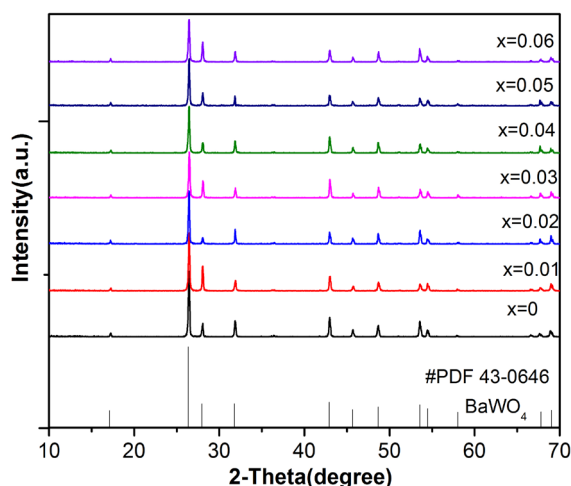


Figure 1. XRD patterns of Ba_{1-x}WO₄:xDy³⁺ (x = 0 ~ 0.06).

The luminescence properties were investigated by measuring the PL spectra of $\text{Ba}_{1-x}\text{WO}_4: x\text{Dy}^{3+}$ ($x = 0 \sim 0.06$) powders at room temperature. **Figure 2** shows the excitation and emission spectrum of the pure BaWO_4 without doping Dy^{3+} . When monitoring at 355 nm emission wavelength, the excitation spectrum of BaWO_4 contains the wide band from 250 nm to 300 nm with its maximum at 259 nm which belongs to the W-O charge transfer belt (CTB). No other peak was observed in the excitation spectra of un-doped BaWO_4 . According to the principle of atomic orbital, the strong absorption band of BaWO_4 in ultraviolet region comes from the high vibration energy transition that the electrons of group WO_4^{2-} jump from the ground state $^1\text{A}_1$ to $^1\text{B}(^1\text{T}_2)$. The wide peak will be emitted at around 350 nm if the electrons jump from $^1\text{B}(^1\text{T}_2)$, the minimum vibration energy level, back to $^1\text{A}_1$ ground state [3].

The excitation and emission spectrum of $\text{Ba}_{0.99}\text{WO}_4:0.01\text{Dy}^{3+}$ were recorded in **Figure 3**. Four main absorption peaks were presented in the 300 - 400 nm by monitoring the emission at 571 nm, which indicates that the near-UV LEDs based on the Dy^{3+} doped BaWO_4 phosphor can be used as pumping sources to obtain efficient emission. The spectrum presents four bands centered at 323 nm, 350 nm, 363 nm, 386 nm, corresponding to the $f \rightarrow f$ transitions within the Dy^{3+} ion: $^6\text{H}_{15/2} \rightarrow ^4\text{K}_{15/2}$, $^6\text{H}_{15/2} \rightarrow ^4\text{M}_{15/2}$, $^6\text{H}_{15/2} \rightarrow ^4\text{P}_{3/2}$, $^6\text{H}_{15/2} \rightarrow ^4\text{M}_{21/2}$, and the main peak is located at 350 nm [19]. The characteristics of the emission peaks at 483 nm (blue) and 571 nm (green) can be detected excited by 350 nm. The two peaks correspond to the electron transition of $^4\text{F}_{9/2} - ^6\text{H}_{15/2}$ and $^4\text{F}_{9/2} - ^6\text{H}_{13/2}$, respectively. Generally speaking, the phosphor luminescence performance can be affected by the matrix structure. The emitting light is mainly blue when Dy^{3+} ions occupy the high symmetry of inversion center lattices in the matrix, while the emitting light is mainly green when Dy^{3+} ions occupy the low symmetry of none inversion center lattices in the matrix. It can be shown from **Figure 3** that the green light part at 571 nm is much stronger than the blue one at 483 nm, which can be deduced that the Dy^{3+} ions occupy the low symmetry lattices in BaWO_4 [20]. Comparing the emission spectrum with the excitation spectrum in **Figure 2** and

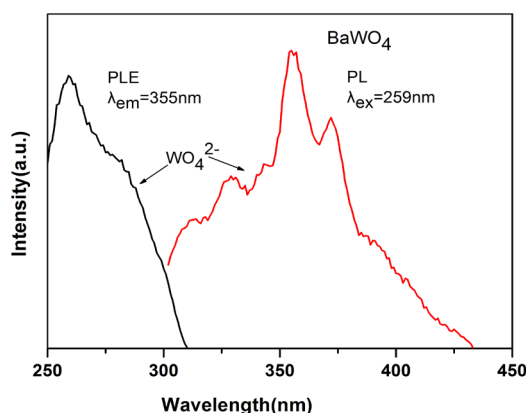


Figure 2. Excitation and emission spectra of the un-doped BaWO_4 samples.

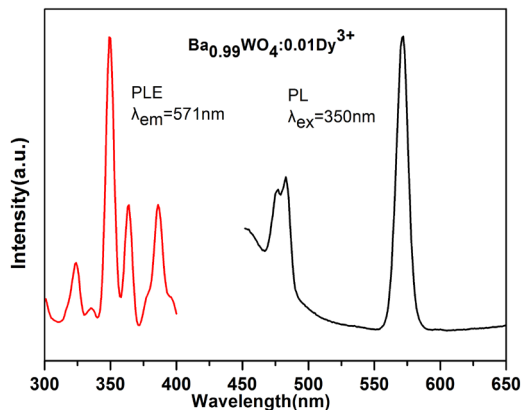


Figure 3. Excited and emission spectrum of $\text{Ba}_{0.99}\text{WO}_4:0.01\text{Dy}^{3+}$.

Figure 3, it can be found that the wide emission peak have much more overlap with the excitation peak of the Dy^{3+} ions at about 350 nm, when the electrons of WO_4^{2-} groups transfer from the minimum vibration energy level of ${}^1\text{B}({}^1\text{T}_2)$ state back to the ${}^1\text{A}_1$ ground state [21].

The emission spectrums of $\text{Ba}_{0.99}\text{WO}_4:0.01\text{Dy}^{3+}$ excited at 350 nm and 259 nm as a comparison were shown in **Figure 4**. The emission peaks of the sample excited at 259 nm include not only the characteristics of Dy^{3+} ions located at 483 nm (blue) and 571 nm (green), but also the wide ones of WO_4^{2-} groups whose electrons transfer from the minimum vibration energy level of ${}^1\text{B}({}^1\text{T}_2)$ state back to the ${}^1\text{A}_1$ ground state. The emission peak shapes of Dy^{3+} ions under the 259 nm excitation is the same as the ones under the 350 nm excitation. However, obviously, the emission peak intensity under 259 nm is stronger, which can be deduced that there exists energy transfer from WO_4^{2-} groups to Dy^{3+} ions and consequently strengthens the emission peaks of Dy^{3+} in $\text{Ba}_{0.99}\text{WO}_4:0.01\text{Dy}^{3+}$.

The emission spectra of $\text{Ba}_{1-x}\text{WO}_4:x\text{Dy}^{3+}$ ($x = 0 \sim 0.06$) phosphors excited at 350 nm were illustrated in **Figure 5**. The emission spectrum of all samples contains two characteristic peaks at 483 nm (blue) and 571 nm (green) which have no obvious changed with the increasing Dy^{3+} ions. The emission intensity of Dy^{3+} at 483 nm and 571 nm gradually increases with the increase of the Dy^{3+} content until the concentration of Dy^{3+} reaches to 0.05, and then the intensity decreases with the further increase of the Dy^{3+} content because of concentration quenching. Meanwhile, the emission peak intensity is relatively high, which shows that $\text{Ba}_{1-x}\text{WO}_4:x\text{Dy}^{3+}$ phosphors can obtain ideal emission light under 350 nm excitation.

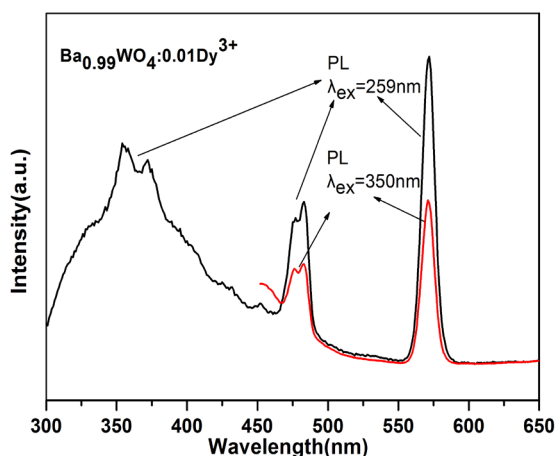


Figure 4. Emission spectrum of $\text{Ba}_{0.99}\text{WO}_4:0.01\text{Dy}^{3+}$ excited at 350 and 259 nm.

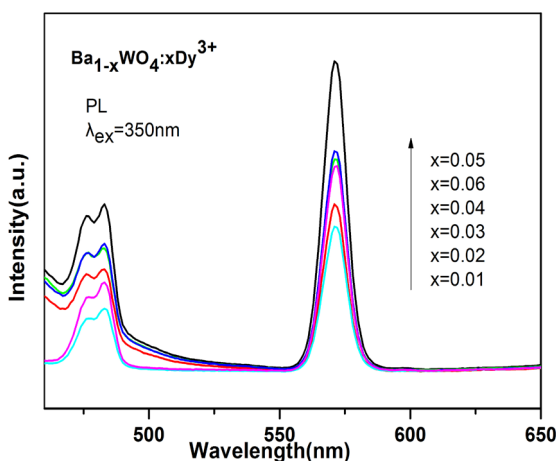


Figure 5. Emission spectrum of $\text{Ba}_{1-x}\text{WO}_4:x\text{Dy}^{3+}$ ($x = 0 \sim 0.06$) phosphors excited at 350 nm.

In order to investigate the difference in emission spectra resulted from various excitations, the PL emission spectra of $\text{Ba}_{1-x}\text{WO}_4:\text{xDy}^{3+}$ ($x = 0 \sim 0.06$) powders excited at 259 nm (host excitation) were shown in **Figure 6**. The emission spectra of the $\text{Ba}_{1-x}\text{WO}_4:\text{xDy}^{3+}$ phosphors consist of two main bands peaking at 483 and 571 nm which did not be changed as Dy^{3+} concentration changed. Within the emission transitions, the green band and the blue band are the predominant transitions. The emission intensity of Dy^{3+} at 483 nm and 571 nm gradually increases with the increasing of the Dy^{3+} content until the concentration of Dy^{3+} reaches to 0.05, and then it decreases with the further increase of the Dy^{3+} content because of concentration quenching. Comparing with the emission intensity difference at 483 and 571 nm, the emission peak of the characteristic peaks of Dy^{3+} is gradually strengthened with the increase of Dy^{3+} ions, indicating stronger energy transfer from the host to Dy^{3+} ions. The absorption rate of emission peak from the host is enhanced as the increase of Dy^{3+} ions, so that the emission peak intensity of the host is weakened while the characteristics emission peak of Dy^{3+} ion is relatively enhanced. Finally, the characteristics emission peak of Dy^{3+} ion is reduced as the increasing of Dy^{3+} ions, which is due to the shorter distance between different Dy^{3+} ions, and the stronger interact of ions. No radioactive transition is produced, leading to the concentration quenching effect.

The decay curves for the luminescence of $\text{BaWO}_4:\text{xDy}^{3+}$ ($x = 0.01, 0.03, 0.05$) phosphors as monitoring 575 nm under 259 nm excitation were shown in **Figure 7**. Based on the decay curves and loading time, the decay lifetimes of the samples were calculated to be $\tau = 13.693, 10.799$ and $9.514 \mu\text{s}$ corresponding with the content ($x = 0.01, 0.03, 0.05$). The decreasing of the host emission lifetime value of $\text{BaWO}_4:\text{xDy}^{3+}$ supports the presence of the energy transfer from WO_4^{2-} to Dy^{3+} in BaWO_4 host.

G. Blasse has pointed out that when a sensitizer or activator replaces the lattice in the matrix, the critical G. Blasse has pointed out that when a sensitizer or activator replaces the lattice in the matrix, the critical distance of the energy transfer meets the formula [22] [23]:

$$R_c \approx 2(3V/4\pi x_c N)^{\frac{1}{3}}$$

where V is the volume of the unit lattice, N is the atomic number of the unit lattice, x_c is the critical concentration. For the BaWO_4 host, $V = 0.4002 \text{ nm}^3$, and $N = 4$, the Dy^{3+} concentration x_c is 0.05, hence the average distance ($R_c = 0.576 \text{ nm}$) between Dy^{3+} can be calculated. The energy transfer mechanism is the very close to the distance between the ions. The electron cloud produces overlap, which leads to the exchanging electrons and the exchanging of energy. However, the critical distance of energy transfer is 0.5 nm, this value is less than the average distance $R_c = 0.576 \text{ nm}$ between Dy^{3+} ions. Hence the reason why concentration quenching of Dy^{3+} ions is not the energy transfer but the effect of the electric multipole moment-electric multipole moment effect.

According to the available CIE standard, we computed the CIE chromaticity coordinates (x, y) of the $\text{BaWO}_4:\text{xDy}^{3+}$ ($x = 0 \sim 0.06$) samples under the 259 nm excitation, which are marked as points 1 ~ 6 in **Figure 8**. The chromaticity coordinates are (0.3294, 0.3954), (0.3283, 0.3945), (0.3247, 0.3939), (0.3128, 0.3849), (0.3097, 0.3713) and (0.2946, 0.3657) corresponding with the increasing of Dy^{3+} concentration from $x = 0$ to 0.06. As

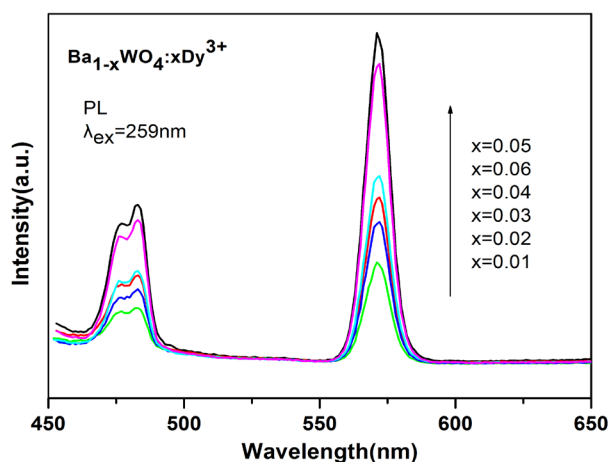


Figure 6. Emission peak of $\text{Ba}_{1-x}\text{WO}_4:\text{xDy}^{3+}$ ($x = 0.01 \sim 0.06$) between 450 and 650 nm excited at 259 nm.

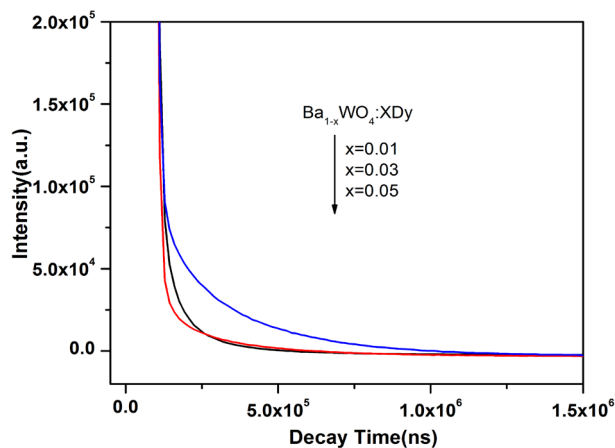


Figure 7. Photoluminescence decay curves of $\text{Ba}_{1-x}\text{WO}_4:\text{xDy}^{3+}$ ($x = 0.01, 0.03, 0.05$) monitored by 575 nm excited at 259 nm.

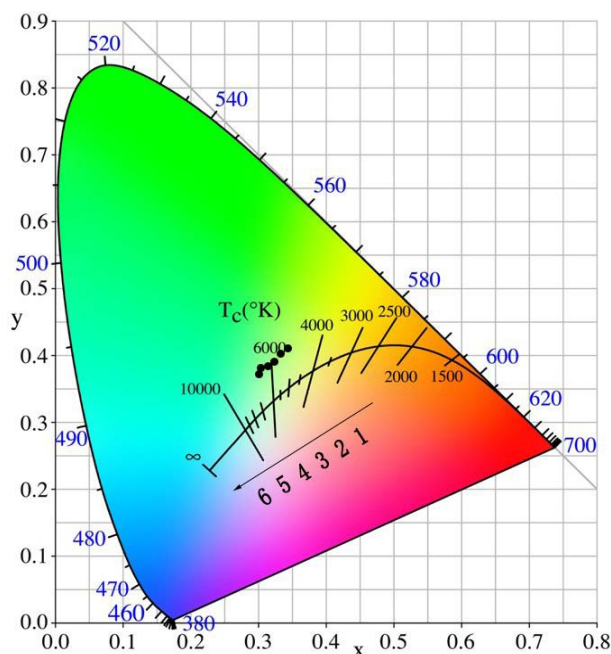


Figure 8. The chromaticity coordination (x, y) of $\text{BaWO}_4:\text{xDy}^{3+}$ ($x = 0 \sim 0.06$) phosphors excited under 259 nm in the CIE 1931 chromaticity diagram.

shown in **Figure 8**, the color tones slowly shift from green (represented by point 1, 2) to blue (represented by point 5, 6) with increasing concentration of Dy^{3+} ion. In summary, a fine tuning emission color can be easily realized by adjusting the emission proportion between green and blue light in the emission spectrum of Dy^{3+} , and the $\text{Ba}_{1-x}\text{WO}_4:\text{xDy}^{3+}$ can be applied in white LED as blue-green phosphor.

4. Conclusion

In summary, $\text{BaWO}_4:\text{xDy}^{3+}$ phosphors are synthesized by conventional solid state method. XRD studies confirm the formation of single phase in these samples, and there is no change in the structure by doping a small amount of Dy^{3+} . The efficient energy transfer takes place within the WO_4^{2-} groups to the Dy^{3+} ions, which is demonstrated by the photoluminescence excitation and photoluminescence decay times. The CIE coordinates show that the emission light color of $\text{BaWO}_4:\text{xDy}^{3+}$ samples changes from green to blue with the increasing of Dy^{3+} con-

centration. The PL emission intensity increases with Dy³⁺ content to its maximum at the optimum concentration of about 5 mol% Dy³⁺ in BaWO₄:xDy³⁺ phosphors, and then decreases due to the concentration quenching. Therefore, these phosphors can be efficiently excited by near-UV light, and be potential candidates for application in white LED.

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