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Synthesis and Color-tunable Luminescence of Ce^{3+} , Tb^{3+} Codoped $\text{Sr}_6\text{YSc}(\text{BO}_3)_6$ Phosphor

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Abstract

Multi-color emitting phosphors will be potential in the fabrication of solid state lighting devices. Color-tunable $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors were synthesized by a high temperature solid-state reaction, and the crystal structure and luminescence properties were investigated in detail. The photoluminescence excitation spectrum of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce}^{3+}$ show that the excitation peaks from 200 to 400 nm are attributed to the characteristic 4f-5d transitions of Ce^{3+} , and the broad-band blue emission can be also found from the as-synthesized $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce}^{3+}$. Under the excitation of near ultraviolet (n-UV) light, $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors exhibit not only a broad blue emission band originating from the f-d transition of Ce^{3+} ion but also a group of sharp characteristic green emission lines from the f-f transition of Tb^{3+} ion, respectively. The excitation spectra monitored at 544 nm emission of Tb^{3+} consists of the characteristic excitation bands originating from Ce^{3+} and Tb^{3+} ions, which proves the occurrence of the energy transfer between Ce^{3+} and Tb^{3+} . The energy transfer behaviors in $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors is also investigated by the lifetime measurement. The above results indicate that $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce},\text{Tb}$ can act as a potential candidate for near-UV-pumped light emitting diodes.

Keywords: Luminescence; Energy transfer; Phosphor

Background

In recent years, along with the energy shortage and global warming, the development of energy saving products in the lighting field, such as white light-emitting diode (WLED), has attracted great attention [1]. Due to the potential applications in indicators, green architectural lighting, automobile headlights, backlights, and general illuminations, WLEDs have become a kind of daily lighting source with excellent properties such as high efficiency, good light stability, long operational lifetime, and environmentally friendly characteristics [2-5]. Furthermore, many efforts have been made in the search of down-converting phosphors converting ultraviolet (UV) or blue light into a combination of red-green-blue light in order to obtain white light emission [6]. Commercial WLEDs lamp is commonly fabricated by using a blue InGaN LED chip and the yellow-emitting $\text{Y}_3\text{Al}_5\text{O}_{12}:\text{Ce}^{3+}$ (YAG:Ce) phosphor. However, such WLEDs have a poor color rendering index (CRI) and a high correlated color temperature (CCT) [7,8] because of lacking a red component. However, WLEDs can be also fabricated by pumping blue, green and red phosphors coated on a near-UV (n-UV) LED chip which has a high CRI [9]. For this reason, it is necessary to develop novel multi-color emission phosphors in the field of optical materials.

Recently, borates compounds attracted much attention due to their excellent optical properties, low synthesis temperature, less expensive raw materials, and high physical chemistry stability [10,11]. Among them, the new compounds with the general formula of $A_6MM(BO_3)_6$, ($A = Sr$; $M = Gd, Y$; $M = Al, Ga, In, Sc$ and Y) has been reported previously [12,13]. In this work, $Sr_6YSc(BO_3)_6$ was selected as the host material, Ce^{3+} , Tb^{3+} singly doped and co-doped $Sr_6YSc(BO_3)_6$ phosphors were firstly reported. It is well known that Ce^{3+} ions acts as a good sensitizer in co-doped materials, and transfers a part of energy to the activator ions, such as Eu^{2+} , Tb^{3+} and Mn^{2+} [14-16]. As for the Ce^{3+} - Tb^{3+} couples with fixed Ce^{3+} content, the Tb^{3+} emission from 5D_3 level will be quenched and 5D_4 emission will increase gradually with increasing Tb^{3+} concentration [17]. Herein, the novel color-tunable $Sr_6YSc(BO_3)_6:Ce^{3+}, Tb^{3+}$ phosphors have been obtained via the traditional solid-state method. Blue to blue-green emission can be realized in this series of phosphors by changing the Tb^{3+} concentration. Luminescent properties and energy transfer process between the sensitizer (Ce^{3+}) to activator (Tb^{3+}) in the $Sr_6YSc(BO_3)_6$ host have been studied, and the energy transfer mechanism is also investigated.

Methods

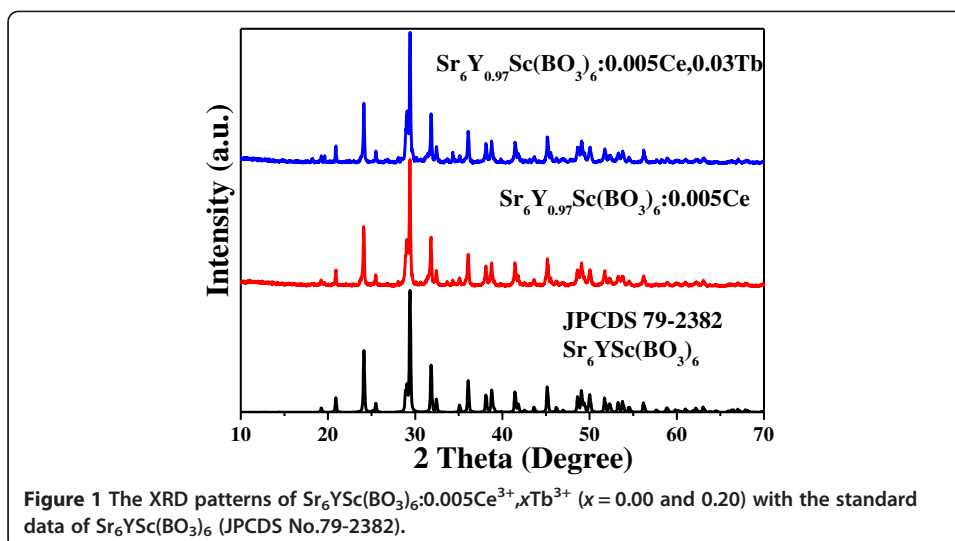
A series of $Sr_6YSc(BO_3)_6:Ce^{3+}, Tb^{3+}$ phosphors were prepared by using the conventional high temperature solid state reactions. $SrCO_3$ (99.5%), Y_2O_3 (99.99%), Sc_2O_3 (99.99%), H_3BO_3 (99.5%), CeO_2 (99.99%) and Tb_4O_7 (99.99%) were used as the starting materials. Stoichiometric amounts of the starting materials were thoroughly mixed and ground thoroughly in an agate mortar. Then, the mixture was transferred into an alumina crucible and calcined in a muffle furnace at 1100°C for 6 h under CO reducing atmosphere. The as-prepared samples were reground into powder for measurement at room temperature.

The crystal structures of the samples were checked by X-ray powder diffraction (SHIMADZU, XRD-6000, 40 kV and 30 mA, $Cu\ K\alpha\ \lambda = 0.15406\ nm$). Photoluminescence excitation (PLE) and emission (PL) spectra were performed by using a JOBIN YVON FluoroMax-3 fluorescence spectrophotometer with a photomultiplier tube operating at 400 V, and a 150 W Xe lamp used as the excitation lamp. The decay time were carried on using a spectro-fluorometer (HORIBA, JOBIN YVON FL3-21), and the 250 nm pulse laser radiation (nano-LED) was used as the excitation source. And the CIE coordinates were calculated based on the photoluminescence spectra between 375 and 650 nm.

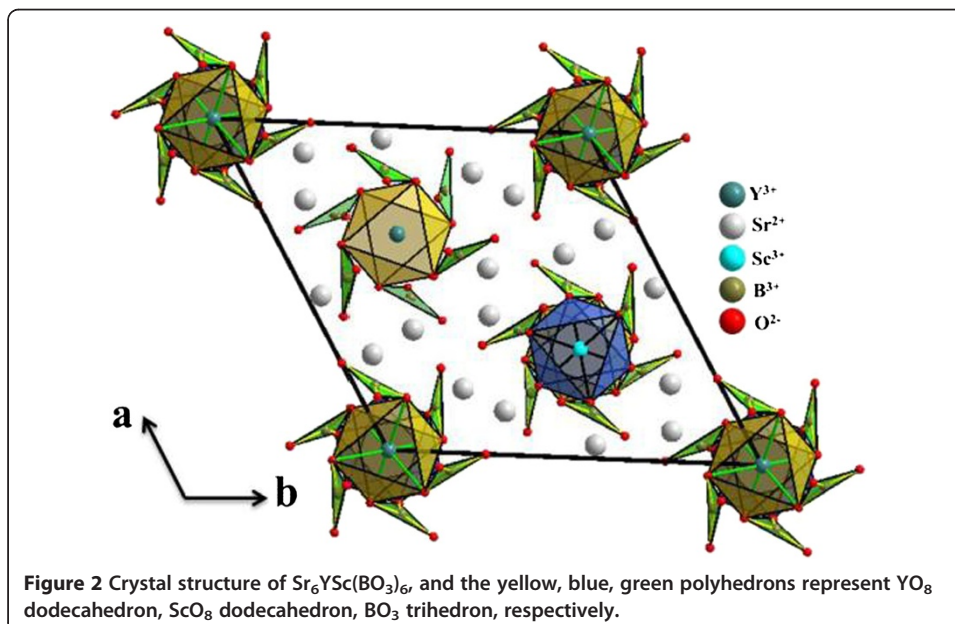
Results and discussions

Crystal structure

Figure 1 illuminates the XRD patterns of as-prepared $Sr_6YSc(BO_3)_6:0.005Ce^{3+}$, $Sr_6YSc(BO_3)_6:0.005Ce^{3+}, 0.03\ Tb^{3+}$ and $Sr_6YSc(BO_3)_6:0.005Ce^{3+}, 0.20\ Tb^{3+}$ phosphors. All the peaks match well with the standard data of JPCDS card no. 79-2382 ($Sr_6YSc(BO_3)_6$), and no other crystalline phase can be detected, indicating that all the as-synthesized samples are of single phase. These results illuminate that the doped Ce^{3+} and Tb^{3+} ions were completely dissolved in the $Sr_6YSc(BO_3)_6$ host lattice without any significant change in the crystal structure. Figure 2 shows the crystal structure of $Sr_6YSc(BO_3)_6$,



and yellow, blue, green polyhedrons represent YO_8 dodecahedron, ScO_8 dodecahedron, BO_3 trihedron, respectively, which belongs to the trigonal space group $R\bar{3}(148)$, and the lattice parameters are $a = 12.284(1) \text{ \AA}$, $c = 9.268(2) \text{ \AA}$ and $V = 1211.15(33) \text{ \AA}^3$. Sr^{2+} , Y^{3+} , Sc^{3+} and B^{3+} metal ions are surrounded by quantitative oxygens. Y^{3+} and Sc^{3+} ions are randomly located in eightfold dodecahedral $[\text{Y}/\text{ScO}_8]$ sites. The B^{3+} ions are in three fold trihedron $[\text{BO}_3]$ sites. The Sr^{2+} is connected to nine oxygen while four O (1) is only connected with B^{3+} ions with Sc is surrounded by three O (2) and the other oxygen namely O (3) relate to the Y^{3+} ions [12]. It is well known that an acceptable percentage difference in ion radii between the doped and substituted ions must not exceed 30%. Herein, it is known that the ionic radius of 8-coordinated Y^{3+} is 1.019 \AA , which is close to that of Ce^{3+} (1.143 \AA) or Tb^{3+} (1.04 \AA) while the ionic radius of 8-coordinated Sc^{3+} is 0.87 \AA , which is too small for Ce^{3+} or Tb^{3+} to be substituted. Consequently, it is



reasonable that the Ce^{3+} and the Tb^{3+} ions are completely incorporated into the host lattice by substituting for Y^{3+} sites in the $\text{Sr}_6\text{YSc}(\text{BO}_3)_6$ host.

Luminescence properties

Figure 3 demonstrates the photoluminescence excitation (PLE) and emission (PL) spectra of Ce^{3+} and Tb^{3+} single doped and $\text{Ce}^{3+}, \text{Tb}^{3+}$ codoped $\text{Sr}_6\text{YSc}(\text{BO}_3)_6$ phosphors. As shown in Figure 3(a), the excitation spectrum monitored at 544 nm for $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.30 \text{ Tb}$ sample exhibits a shoulder and a broad band centered at 241 nm and 280 nm, which correspond to the spin-allowed and spin-forbidden $4f^8-4f^75d$ transition of Tb^{3+} ions, respectively [18]. The PL spectrum consists of emission lines at 411, 489, 544, 583 and 623 nm, which should be ascribed to the transitions from the $^5\text{D}_4/^5\text{D}_3$ excited state to the $^7\text{F}_j$ ($J=6-3$) ground states of Tb^{3+} [19]. Due to a magnetic dipole allowed transition, the intensity of $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transition peaks at 544 nm is much higher than that of the other emission lines. The emission spectrum under the excitation of 365 nm of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}$ (Figure 3(b)) has an asymmetric blue band ranging from 375 to 600 nm with a maximum at 423 nm which is due to the characteristic $5d^1 \rightarrow 4f^1$ transition of Ce^{3+} . The PLE spectrum consists of three major peaks corresponding to the typical transition of Ce^{3+} . Moreover, it can be seen from Figure 3(c) that the emission spectrum monitored at 365 nm of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}, 0.20 \text{ Tb}^{3+}$ phosphor exhibits not only the obvious blue band from Ce^{3+} but also a group of sharp characteristic green line from Tb^{3+} . Compared with that of the $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}$ sample, one can find that their PLE spectra are similar monitored by the emission of 423 nm. By Monitoring the 544 nm emission of Tb^{3+} , the excitation spectrum consists of both the excitation bands of Ce^{3+} ions and that of the Tb^{3+} ions. The observed results show significant contribution for the excitation of emission of $\text{Ce}^{3+}, \text{Tb}^{3+}$ codoped samples originating from f-d transition of Ce^{3+} , which also provides an obvious evidence of the energy transfer from Ce^{3+} to Tb^{3+} . Furthermore, PLE spectrum monitored at 544 nm emission for the $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.30 \text{ Tb}^{3+}$ sample shows some line-type excitation lines in the range of 300 and 500 nm, which are ascribed to the $4f \rightarrow 4f$ electrons transition of Tb^{3+} ions

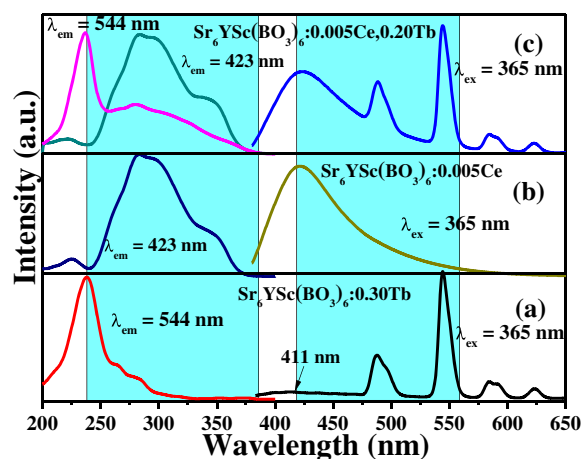
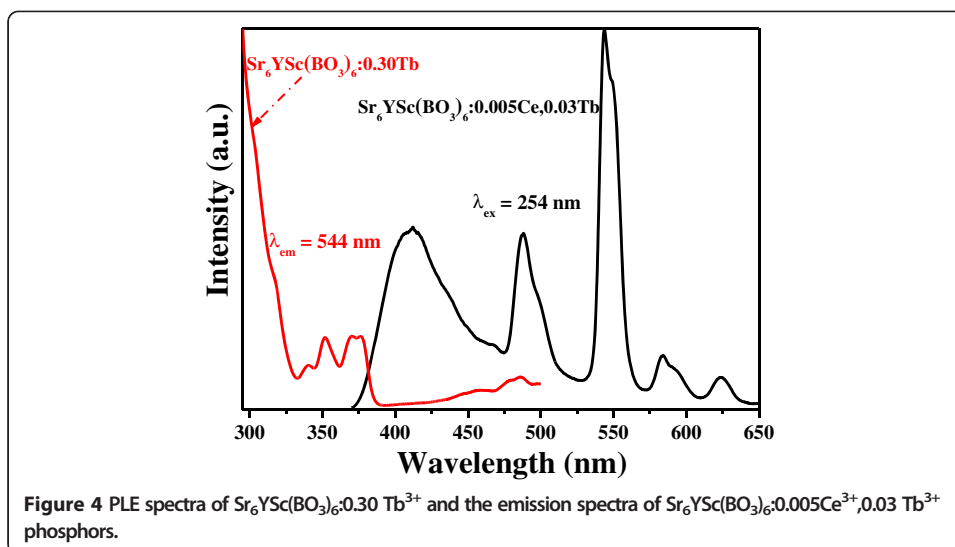
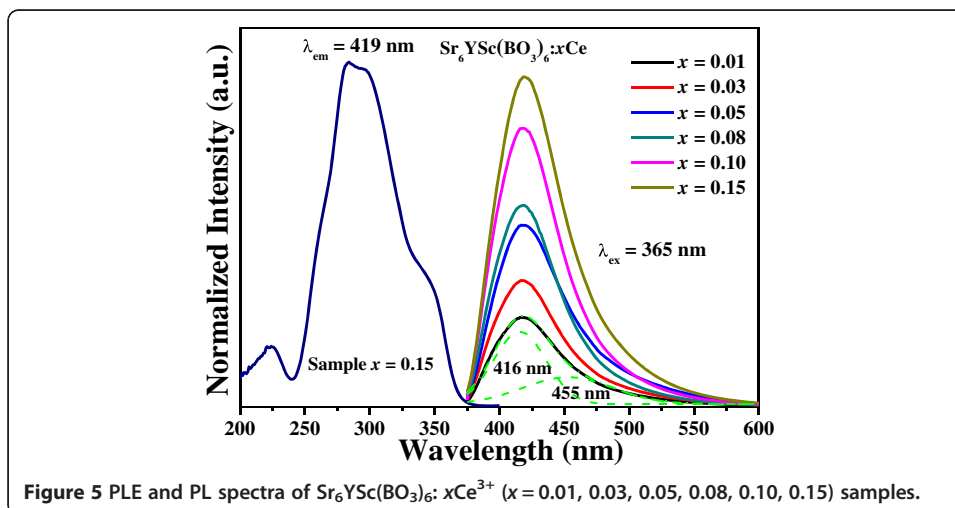


Figure 3 The PLE and PL spectra of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.30 \text{ Tb}^{3+}$ (a), $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}$ (b), $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}, 0.20 \text{ Tb}^{3+}$ (c) phosphors.



(Figure 4). Compared with Figure 3(b), one can find the spectral overlap between the emission spectrum of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}$ and the excitation spectrum of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.30\text{Tb}^{3+}$, which means the possible energy transfer process. Figure 4 also gives the emission spectrum of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+},0.03\text{Tb}^{3+}$ phosphor upon the excitation of 254 nm, which includes the broad band from Ce^{3+} and the narrow lines from Tb^{3+} . Moreover, the spectral overlap of the excitation spectrum of Tb^{3+} -doped samples and emission spectrum of $\text{Ce}^{3+},\text{Tb}^{3+}$ codoped samples also appears suggesting the possible energy transfer process.

PLE and PL spectra of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:x\text{Ce}^{3+}$ phosphors are shown in Figure 5. Under the excitation of 365 nm, all of the emission spectra exhibit the similar profile with different relative intensities. The emission band gradually becomes broader and red-shifts with increasing Ce^{3+} concentrations. After Gaussian deconvolution, the broad emission band of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.01\text{Ce}^{3+}$ can be well decomposed into two components centered at about 416 and 455 nm. The excitation spectrum monitored at 419 nm shows a typical broad band in the range of 200–400 nm, which is ascribed to the 4f-5d transition of Ce^{3+} .



The energy transfer process from Ce^{3+} to Tb^{3+} is also investigated by the decay curves of Ce^{3+} emission in codoped samples of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+},x\text{Tb}^{3+}$ ($x = 0.00, 0.01, 0.03, 0.05, 0.10, 0.20, 0.30$). Figure 6 exhibits the room temperature decay curves of Ce^{3+} emission at 419 nm under the excitation at 250 nm, and the lifetime values of Ce^{3+} in also given in Figure 6. These decay curves are analyzed and they can be fitted successfully based on the following second-order exponential equation:[20].

$$I(t) = I_0 + A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2) \quad (1)$$

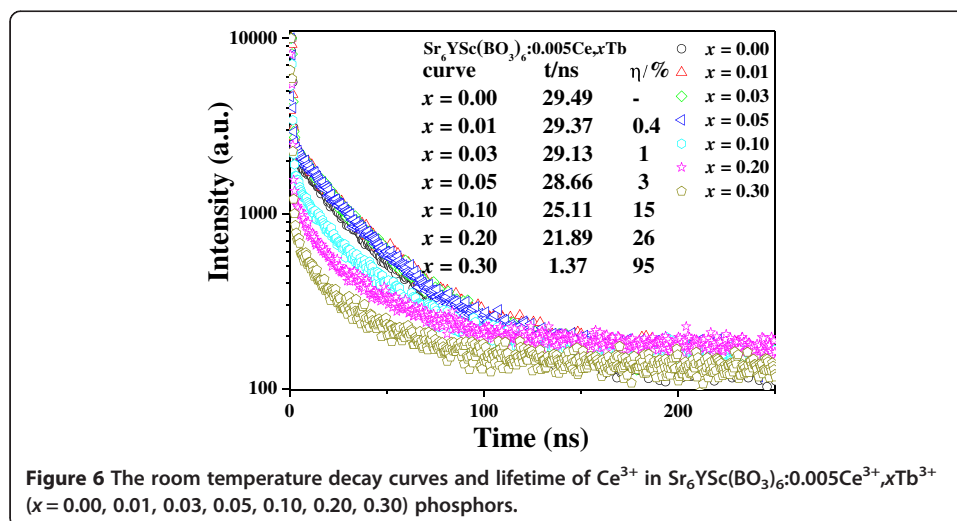
Here I and I_0 correspond to the luminescence intensity at time t and initially, respectively. A_1 and A_2 are two constants which are related with the initial intensity, τ_1 and τ_2 are the lifetimes for the exponential components. By using these parameters, the average lifetime constant (τ^*) can be calculated by the formula as follows,

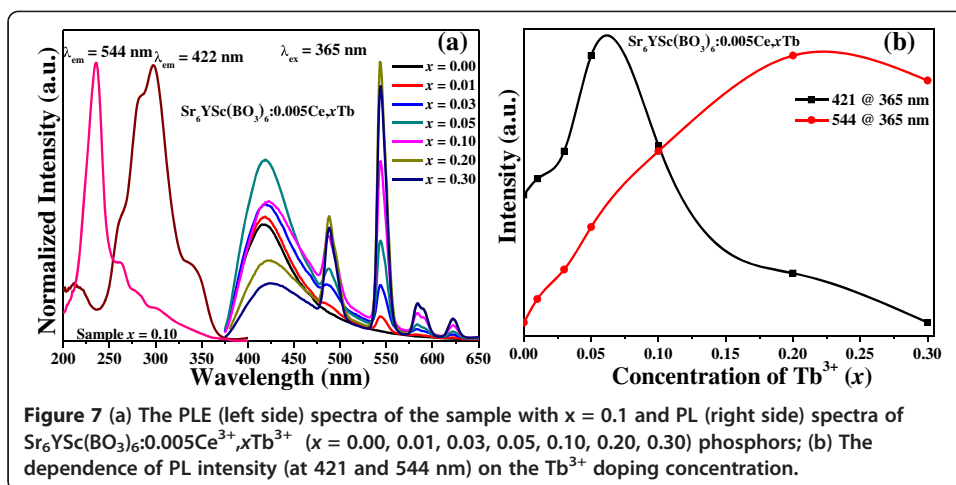
$$\tau^* = (A_1\tau_1^2 + A_2\tau_2^2)/(A_1\tau_1 + A_2\tau_2) \quad (2)$$

The measured fluorescence lifetimes (τ^*) were calculated to be 29.49, 29.37, 29.13, 28.66, 25.11, 21.89 and 1.37 ns for $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+},x\text{Tb}^{3+}$ with $x = 0.00, 0.01, 0.03, 0.05, 0.10, 0.20$ and 0.30 , respectively. With increasing Tb^{3+} concentration, the value of τ^* is observed to decrease gradually. Therefore, the energy transfer efficiency from Ce^{3+} to Tb^{3+} was calculated by the following formula:

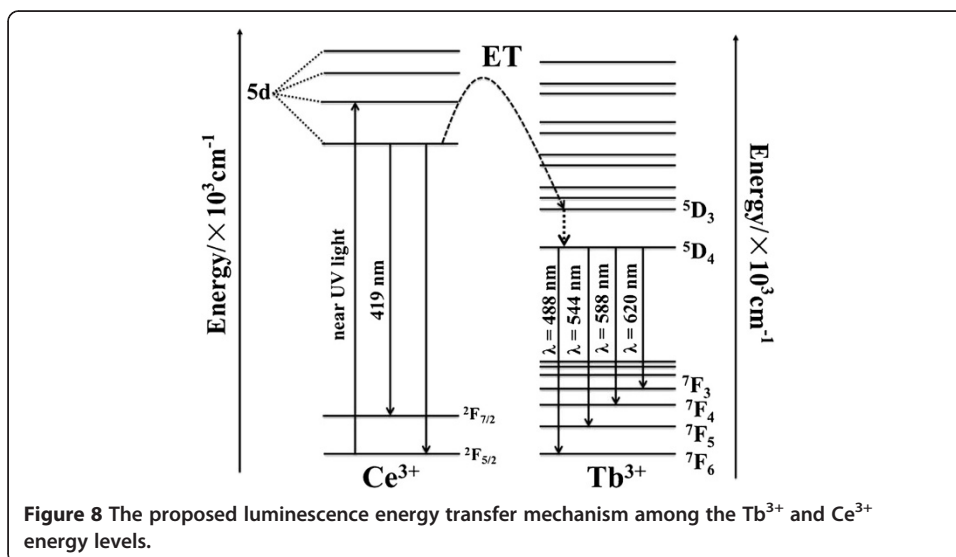
$$\eta_T = 1 - \tau_S/\tau_0 \quad (3)$$

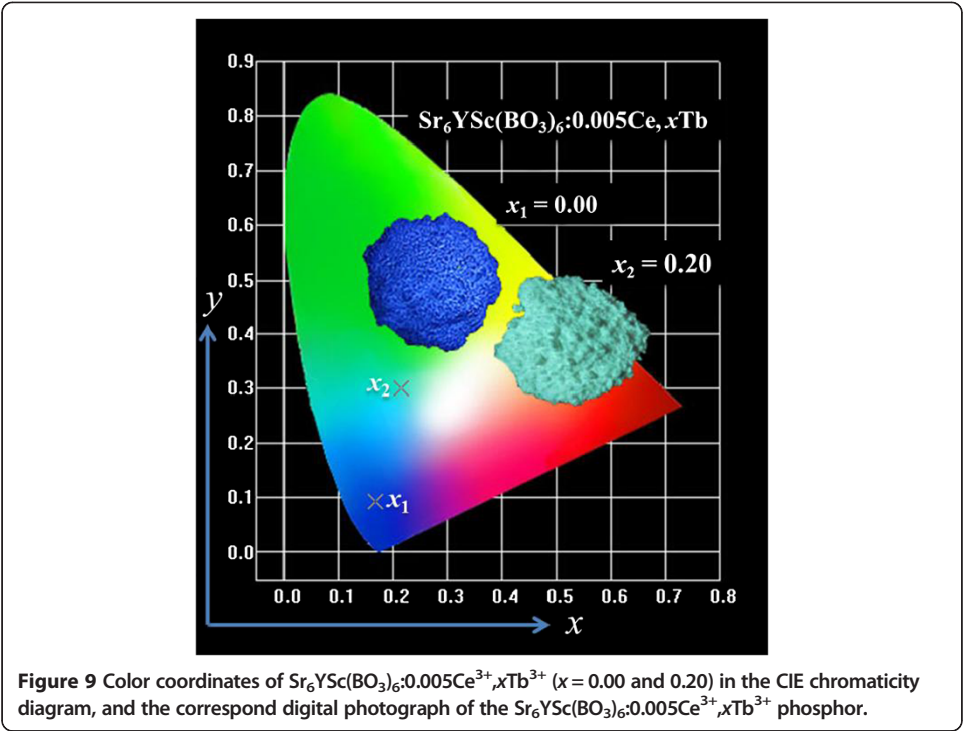
where τ_0 and τ_S stand for the lifetimes of Ce^{3+} in the absence and the presence of Tb^{3+} , respectively. As shown in the inset of Figure 6, the energy transfer efficiency (η_T) increases with increasing Tb^{3+} concentration. The energy-transfer efficiencies from Ce^{3+} to Tb^{3+} in $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+},x\text{Tb}^{3+}$ phosphors are calculated to be 0, 4%, 1%, 3%, 15%, 26% and 95% corresponding to $x = 0.00, 0.01, 0.03, 0.05, 0.10, 0.20$ and 0.30 . All the obtained results prove that the energy-transfer process from Ce^{3+} to Tb^{3+} in $\text{Sr}_6\text{YSc}(\text{BO}_3)_6$ is very efficient. In order to obtain the color-tunable emission and further optimize the green emission of Tb^{3+} ions, Tb^{3+} concentration dependent PL spectra of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+},x\text{Tb}^{3+}$ phosphors are studied. The excitation and emission





spectra of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+},x\text{Tb}^{3+}$ phosphors with different Tb^{3+} doping contents ($x = 0, 0.01, 0.02, 0.05, 0.10, 0.20, 0.30$) upon an excitation wavelength of 365 nm are given in the Figure 7. $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+},x\text{Tb}^{3+}$ phosphors show blue and green emission bands centering at about 419 nm and 544 nm due to the variation of the relative doping concentrations. As we all know that the emission spectrum of Tb^{3+} ions shows a characteristic group of sharp lines corresponding to the transitions of $^5\text{D}_J$ ($J = 3-4$) and $^7\text{F}_J$ ($J = 0-6$) states. As seen in Figure 7(a), an obvious blue emission band and some green emission peaks are observed in the emission spectra of all the $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce},x\text{Tb}$ samples under the 365 nm excitation. As also seen in Figure 7(b), for $x \leq 0.05$, the intensities for Ce^{3+} blue emission at 421 nm increased abnormally. Simultaneously, the emission intensity about 419 nm and the characteristic emission of Tb^{3+} increases. With the different radiation excitations, the same emission spectral profile also can be found in Figure 4. At the same time, with the increase in the Tb^{3+} concentration ($0.05 \leq x \leq 0.20$), the intensity of blue emission at 419 nm decreased while the emission intensity of Tb^{3+} due to transitions $^5\text{D}_4$ to $^7\text{F}_J$ ($J = 6-3$) increased. As for the high Tb^{3+} doping concentration ($x \geq 0.20$), all the emission intensities decrease because of the





concentration quenching effect suggesting that the possible energy transfer process could be existed between Ce^{3+} and Tb^{3+} . On the basis of the above spectral analysis, the excitation and emission mechanism of Ce^{3+} and Tb^{3+} , and the energy transfer processes ($\text{Ce}^{3+} \rightarrow \text{Tb}^{3+}$) in this host is schematically shown in Figure 8. The energy transfer process taking place in the system can be explained as follows. When Ce^{3+} and Tb^{3+} are co-doped in the same host, it is accepted that Ce^{3+} ions can absorb the near ultraviolet light of 365 nm. And the energy transfer take place from 5d energy level of Ce^{3+} to the high excitation levels $^5\text{D}_3$ of Tb^{3+} , which relaxes to $^5\text{D}_4$ later, which transitions to the low level of $^7\text{F}_J$ ($J = 6-3$).

Figure 9 exhibits the CIE chromaticity diagram and the corresponding digital photographs of the $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}$ and $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}, 0.20 \text{ Tb}^{3+}$ phosphor. The violet and blue light emission with CIE coordinates of (0.1682,0.0934) and (0.2156,0.3004) can be obtained, as shown in Figure 9. Table 1 gives a summary of the x and y values of CIE chromaticity coordinates for $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}, x\text{Tb}^{3+}$

Table 1 CIE chromaticity coordinates of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}, x\text{Tb}^{3+}$ ($x = 0.00, 0.01, 0.03, 0.05, 0.10, 0.20, 0.30$) phosphors under the 365 nm excitation

Sample composition (x) of $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+}, x\text{Tb}^{3+}$	CIE coordinates (x, y)
0	(0.1682,0.0934)
0.01	(0.1704,0.1073)
0.03	(0.1759,0.1385)
0.05	(0.1787,0.1414)
0.10	(0.1901,0.2024)
0.20	(0.2156,0.3004)
0.30	(0.2243,0.3261)

phosphors with different Tb^{3+} concentrations under the excitation of 365 nm. With increasing Tb^{3+} content, the chromaticity coordinates for $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:0.005\text{Ce}^{3+},x\text{Tb}^{3+}$ could be tuned from blue (0.1682,0.0934) to blue-green (0.2243,0.3261) position by adjusting the doping content of Tb^{3+} ions.

Conclusions

In summary, $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors was synthesized successfully by the conventional high temperature solid-state reactions. The luminescence spectra and decay curves demonstrated that Ce^{3+} ion can absorb UV photons via the allowed 4f-5d absorption and greatly enhance the green emission of Tb^{3+} ion under the 365 nm excitation. $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce}^{3+},\text{Tb}^{3+}$ shows obvious absorption peaks including the absorption lines of Ce^{3+} ions and Tb^{3+} ions. Due to existence of the energy transfer, the emission hue of the phosphor can be varied from blue (0.1682,0.0934) eventually to bluish green (0.2243,0.3261) with increasing Tb^{3+} concentration and a fixed Ce^{3+} content in $\text{Sr}_6\text{YSc}(\text{BO}_3)_6$. Thus, the obtained $\text{Sr}_6\text{YSc}(\text{BO}_3)_6:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphor is expected to be developed as a suitable phosphor candidate for the application in near-UV excited white LEDs.

Competing interests

The authors declare that they have no competing interests.

Authors' contribution

XC carried out the experimental details and wrote the draft paper. ZX designed the study and gave the discussion. All authors read and approved the final manuscript.

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