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Luminescence properties and energy transfer investigations of $Sr_2B_2O_5:Ce^{3+},Tb^{3+}$ phosphors

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Abstract

Ce^{3+} and Tb^{3+} co-doped $Sr_2B_2O_5$ phosphors were synthesized by the solid-state method. X-ray diffraction (XRD) was used to characterize the phase structure. The luminescent properties of Ce^{3+} and Tb^{3+} co-doped $Sr_2B_2O_5$ phosphors were investigated by using the photoluminescence emission, excitation spectra and reflectance spectra, respectively. The excitation spectra indicate that this phosphor can be effectively excited by near ultraviolet (n-UV) light of 317 nm. Under the excitation of 317 nm, $Sr_2B_2O_5:Ce^{3+},Tb^{3+}$ phosphors exhibited blue emission corresponding to the *f–d* transition of Ce^{3+} ions and green emission bands corresponding to the *f–f* transition of Tb^{3+} ions, respectively. The Reflectance spectra of the $Sr_2B_2O_5:Ce^{3+},Tb^{3+}$ phosphors are noted that combine with Ce^{3+} and Tb^{3+} ion absorptions. Effective energy transfer occurred from Ce^{3+} to Tb^{3+} in $Sr_2B_2O_5$ host due to the observed spectra overlap between the emission spectrum of Ce^{3+} ion and the excitation spectrum of Tb^{3+} ion. The energy transfer efficiency from Ce^{3+} ion to Tb^{3+} ion was also calculated to be 90%. The phosphor $Sr_2B_2O_5:Ce^{3+},Tb^{3+}$ could be considered as one of double emission phosphor for n-UV excited white light emitting diodes.

Keywords: Energy transfer; Luminescence; Phosphor; White LEDs

1. Introduction

In recent years, white-light emitting diodes (white LEDs) have become a very important lighting source due to their potential application in many fields. They have emerged as the fourth generation of illumination technology for its advantages such as low-cost, environmentally friendly, energy-saving and good electrochemical performance. Currently, most commercially available white LEDs are based on phosphor-converted (PC) emission method. In PC LEDs devices, a blue light InGaN LED chip loaded with a yellow phosphor YAG:Ce $((Y_{1-a}Gd_a)_3(Al_{1-b}Ga_b)_5O_{12}:Ce^{3+})$ to obtain white light, which is now widely used as a conventional white LEDs [1]. The white LED based on $YAG:Ce^{3+}$ phosphor exhibits a poor color rendering index [2]. White LEDs can also be fabricated by the combination of near ultraviolet (near-UV) chip and the red/green/blue light emitted from the phosphors, which provides superior color uniformity with a high CRI [3,4]. This type of white LED requires high-performance blue, green and red phosphors, which can be excited by UV light, so the research on the new optical materials becomes a hot issue. For this reason, it is necessarily for develop a series novel multi-color emission phosphor in the field of optical materials. At present, research on multicolor-emitting phosphor that usually designed according to the energy transfer between different activators (such as Eu^{2+}–Mn^{2+}, Ce^{3+}–Mn^{2+}, Ce^{3+}–Eu^{2+} and Ce^{3+}–Tb^{3+}) in some hosts is a hot topic [5–8]. To the best of our knowledge, both Ce^{3+} and Tb^{3+} ions are important activators and they are widely used in phosphors for fluorescent lamps. Ce^{3+} and Tb^{3+} co-doped materials have been applied in blue and green-light emitting phosphors, because the energy transfer from Ce^{3+} to Tb^{3+} is usually very effective for obtaining bright luminescence of Tb^{3+}.

Up to now, many phosphors have been reported, including silicates, borates, aluminates, sulfides, molybdates, oxynitrides/nitrides, and so on [9–13]. Recently, haloborates have attracted much attention due to their optical transparency, low melting point, easily-shaped and cost-effective properties [14]. These

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compounds are functionally versatile and can serve as excellent host matrices for efficient luminescence, because of their particular 4*f* electronic configuration of trivalent rare earth ions in numerous hosts. Meanwhile, borate-based materials are useful classes of hosts for luminescence due to their high UV transparency and exceptional optical damage threshold [15].

$Sr_2B_2O_5$ was selected as host materials in this paper, whose structure and linear optics have been reported by Lin et al. [16]. In addition, the photoluminescence properties of $Sr_2B_2O_5$:Tb^{3+},Li^+ have also been investigated by Wang [17]. However, the effect of the energy-transfer processes on the photoluminescence properties of $Sr_2B_2O_5$:Ce^{3+},Tb^{3+} under the near-UV excitation has not been investigated. In this paper, a novel intense dual-chromatic blue/green emitting phosphor, $Sr_2B_2O_5$:Ce^{3+},Tb^{3+}, was synthesized and reported. Ce^{3+} ion and Tb^{3+} ion co-doped $Sr_2B_2O_5$ phosphors exhibit two blue and green emission bands under the excitation of UV light. An asymmetric blue emission originates from Ce^{3+} ion, whereas the green emission originates from Tb^{3+} ion. Meanwhile, the energy transfer is thoroughly investigated by their emission spectra. After tuning the content of Tb^{3+} ion, the emission color variation attributed to effective energy transfer from Ce^{3+} ion to Tb^{3+} ion has been discussed. This result clearly demonstrates that the phosphor $Sr_2B_2O_5$:Ce^{3+},Tb^{3+} could be considered as one of double emission phosphor for n-UV excited white light emitting diodes.

2. Experimental

2.1. Sample preparation

All the compounds were synthesized by a conventional solid-state reaction. $SrCO_3$ (analytical reagent, A.R.), H_3BO_3 (A.R.), CeO_2 (99.99%) and Tb_4O_7 (99.99%) were used as the starting materials in this solid-state route. Stoichiometric amounts of the starting materials were thoroughly mixed in an agate mortar. An excess of 10 mol% of H_3BO_3 as a flux and the powder reactants were blended. The mole fraction of the H_3BO_3 flux was varied to determine the effects on light output. The well-mixed stoichiometric mixture was put into an alumina crucible. First, the mixture was heated up to 600 °C and kept at this temperature for 2 h in air, and then mixed again. The final reaction was fired at 800 °C for 4 h, and carbon powder was used as a reducing agent, by which the samples were covered during firing.

2.2. Sample characterization

The phase structure of the as-prepared phosphor was recorded by an X-ray powder diffraction spectroscopy (XRD, Shimadzu, XRD-6000) operating at Cu Kα radiation, 40 kV, 30 mA, and a scan speed of 2.0° (2θ)/min. All excitation and emission spectra were recorded by using HITACHI model F-7000 fluorescence spectrophotometer with a 400 V photomultiplier tube and a 150 W xenon lamp. The measurement of reflectance spectra was performed by using a Hitachi 3010 UV–vis spectrometer. All the measurements were performed at room temperature.

3. Results and discussion

3.1. Crystal structure

According to Lin, Cheng et al.'s work [16], the crystal structure of $Sr_2B_2O_5$ phase belongs to monoclinic crystal system with a space group of $P2_1/c$, the lattice constants of monoclinic $Sr_2B_2O_5$ are: $a = 7.719(4)$ Å, $b = 5.341(1)$ Å, $c = 11.873(2)$ Å and $Z = 4$. Fig. 1 illustrates the X-ray powder diffraction pattern for $Sr_2B_2O_5$:Ce^{3+},Tb^{3+} phosphors annealed with different mole fraction of the H_3BO_3. As shown in Fig. 1(a) and (b), some characteristic diffraction peaks corresponding to $Sr_2B_2O_5$ phase appear except for the low diffraction peaks intensities. When the excess concentration of H_3BO_3 reaches at 10 mol%, the observed characteristic diffraction peaks of $Sr_2B_2O_5$ become sharper and stronger, and the impure phases disappeared, as seen in Fig. 1(c). This result clearly demonstrates that 10 mol% is the appropriate excess concentration in the $Sr_2B_2O_5$:Ce^{3+},Tb^{3+} phosphors. All samples of $Sr_2B_2O_5$:Ce^{3+},Tb^{3+} match well with the XRD patterns 73–1930, which illuminates that these samples all crystallize in the monoclinic structure and no significant effect on their structures as introduce activators Ce^{3+} and Tb^{3+} in our experimental range.

3.2. Luminescence properties of $Sr_2B_2O_5$:Ce^{3+}

Fig. 2 presents the excitation and emission spectra of the $Sr_2B_2O_5$:Ce^{3+} phosphors. It can be seen that the excitation spectrum consists of an asymmetric broad band with a maximum at 317 nm at the observation wavelength $\lambda_{obs} = 392$ nm, which corresponds to the transition from the 4*f* state of Ce^{3+} ion to the excited Ce^{3+} ion 5*d* state. Meanwhile, the asymmetric broad excitation bands are assigned to the electronic transitions from the ground state to the different crystal field splitting bands of excited 5*d* states of Ce^{3+} [18]. Considering the emission spectra of Ce^{3+} doped $Sr_2B_2O_5$

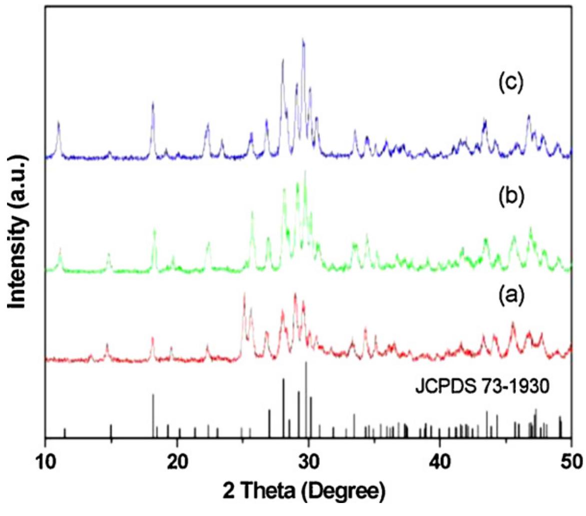

Fig. 1. The XRD patterns of $Sr_2B_2O_5$:$0.02Ce^{3+}$,$0.15Tb^{3+}$ phosphors for different excess concentrations of H_3BO_3 compared with JCPDS card no. 73-1930. (a) 3%, (b) 5%, and (c) 10%.

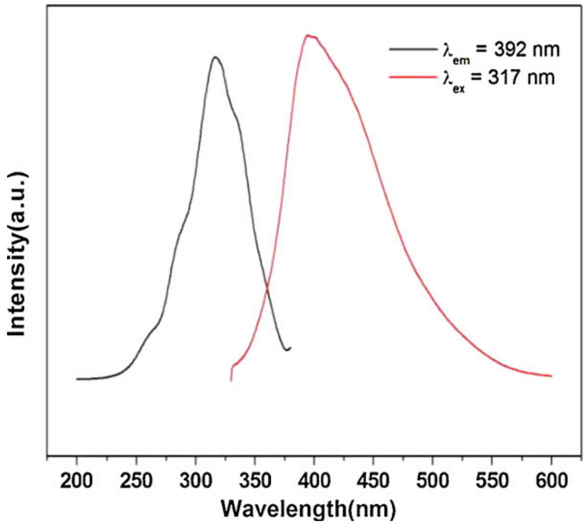

Fig. 2. The excitation and emission spectra of Ce^{3+}-doped $Sr_2B_2O_5$ phosphors. (λ_{ex} = 317 nm, λ_{em} = 392 nm.)

phosphors, an UV excitation at 317 nm leads to a broad asymmetric blue emission band at 392 nm, which is the result of parity-allowed transitions of the lowest component of the 5*d* state to $^2F_{7/2}$ and $^2F_{5/2}$ levels of Ce^{3+}.

3.3. Luminescence properties of $Sr_2B_2O_5$:Tb^{3+}

Fig. 3 shows the excitation and emission spectra of Tb^{3+}-doped $Sr_2B_2O_5$ phosphors. The excitation spectra of $Sr_2B_2O_5$:0.15Tb^{3+} is measured by monitoring the green emission of Tb^{3+} ion at 545 nm and shows two absorption bands at 220–290 nm and 300–400 nm, which is due to the spin-allowed $4f^8$–$4f^75d$ transition of Tb^{3+} and the 4*f*–4*f* transitions of Tb^{3+}, respectively. In order to produce stronger emission, the excitation wavelength of 245 nm is adopted in this study according to the excitation spectrum of Tb^{3+}-doped $Sr_2B_2O_5$ phosphors. The emission spectrum exhibits a set of characteristic optical transitions $^5D_4$–^7F_J of Tb^{3+} ion, which are situated at about 488, 545, 586 and 623 nm, due to $^5D_4$–^7F_J (J = 6, 5, 4 and 3) transitions [19]. The dominated emission peak is at 545 nm.

3.4. Luminescence properties of $Sr_2B_2O_5$:Ce^{3+},Tb^{3+}

Fig. 4 shows the excitation and emission spectra of Ce^{3+} and Tb^{3+} co-doped $Sr_2B_2O_5$ phosphors. The spectral overlap between the emission band of Ce^{3+} and the excitation band of Tb^{3+} supports the occurrence of the energy transfer from Ce^{3+} to Tb^{3+}, as shown in Figs. 2 and 3. Energy transfer process played an important role in the present blue and green double-emitting system $Sr_2B_2O_5$:Ce^{3+},Tb^{3+}. Therefore, the energy transfer phenomenon between the sensitizer (Ce^{3+}) and activator (Tb^{3+}) is investigated through 332 nm light excitation. The excitation of Ce^{3+} and Tb^{3+} co-doped $Sr_2B_2O_5$ shows a strong broad band absorption in n-UV region from the contribution of Ce^{3+}, which makes it suitable to be excited by n-UV LED chip. It is clearly observed that the excitation is consistent with that of Ce^{3+} singly doped $Sr_2B_2O_5$ phosphor. However, the excitation spectra monitored at 545 nm not only show the broad band at 245 nm owing to the $4f^8$–$4f^75d$ transition of Tb^{3+} but also show the three broad bands ranging from 300 to 380 nm due to the $2F_{5/2}$–5*d* transitions of Ce^{3+} ion. The emission spectrum presents a broad band centred at 400 nm except the peaks at 488, 545, 586 and 623 nm. The broad band centred at about 400 nm is attributed to superposition of 5*d*–4*f* transition of Ce^{3+} ion and the superposition of $^5D_4$–^7F_J (J = 6, 5, 4, 3) of Tb^{3+}. These results suggest that energy transfer from Ce^{3+} to Tb^{3+} ion take place. The phosphor $Sr_2B_2O_5$:Ce^{3+},Tb^{3+} could be considered as one of double emission phosphor for n-UV excited white light emitting diodes.

3.5. Diffuse reflection spectra

A good evidence of absorption in the near-UV region from activator ions was obtained through comparison of the

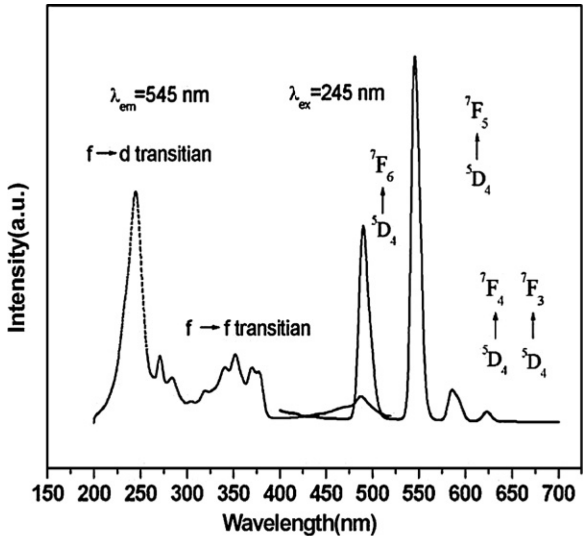

Fig. 3. The excitation and emission spectra of $Sr_2B_2O_5$:0.15Tb^{3+} phosphors. (λ_{ex} = 245 nm, λ_{em} = 545 nm.)

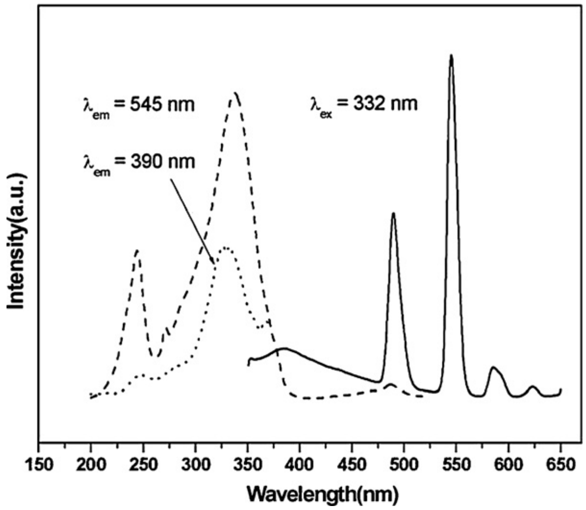

Fig. 4. The excitation and emission spectra of $Sr_2B_2O_5$:0.02Ce^{3+},0.15Tb^{3+} phosphors.

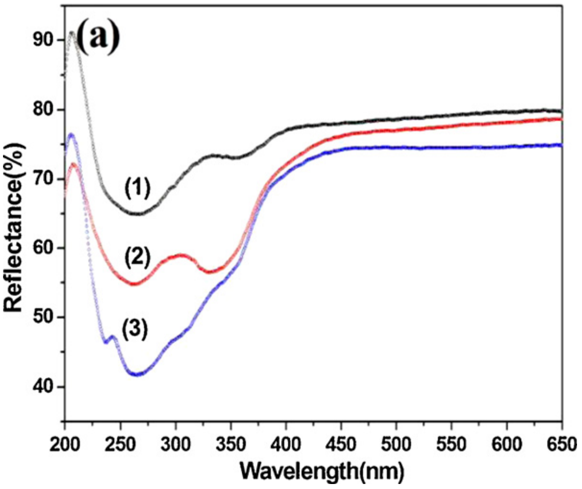

Fig. 5. Diffuse reflectance spectra of $Sr_2B_2O_5$ host, $Sr_2B_2O_5$:0.02Ce^{3+} and $Sr_2B_2O_5$:0.02Ce^{3+},0.15Tb^{3+}.

reflectance spectra (Fig. 5) of host and activator doped $Sr_2B_2O_5$. Fig. 5 shows the diffuse reflection spectra of $Sr_2B_2O_5$ host, $Sr_2B_2O_5$:0.02Ce^{3+} and $Sr_2B_2O_5$:0.02Ce^{3+},0.15Tb^{3+}. For $Sr_2B_2O_5$ host, a decrease in reflectance from 230 to 330 nm was noted. The middle points at about 270 nm may be used to estimate the approximate band gap of host material $Sr_2B_2O_5$ [19]. Reflectance spectra for the $Sr_2B_2O_5$:0.02Ce^{3+} exhibit two absorption bands, which may be attributed to the Ce^{3+} $4f$–$5d$ transition. When co-doping Ce^{3+} and Tb^{3+} ions, we can conclude that $Sr_2B_2O_5$:0.02Ce^{3+},0.15Tb^{3+} phosphor has the strongest absorption in the reflectance spectra which is found to be consistent with those observed in the excitation spectra.

3.6. Energy transfer mechanism

The energy level scheme with electronic transitions (Ce^{3+},Tb^{3+}) and energy transfer processes (Ce^{3+} to Tb^{3+}) is schematically shown in Fig. 6. When the Ce^{3+} ion is irradiated by the UV light, Ce^{3+} ion is excited to higher energy level and then relaxes to lower energy levels until it arrives at $5D_{3/2}$ energy level. One part of the absorption energy of Ce^{3+} has been released in the form of broad band emission at 392 nm light, other part is contributed to the energy transfer process from Ce^{3+} ion to Tb^{3+} ion. And then the energy transfer takes place from $^5D_{3/2}$ of Ce^{3+} to the $^5D_4$ level of Tb^{3+}. Finally, the level of $^5D_4$ gives the strong emission of Tb^{3+} $^5D_4$–^7F ($J = 6, 5, 4, 3$).

3.7. Energy transfer efficiency

The dependence of emission spectra for $Sr_2B_2O_5$:0.02-Ce^{3+},$x$$Tb^{3+}$ ($x = 0$, 0.01, 0.03, 0.08, 0.15, and 0.20) on the concentration of Tb^{3+} is shown in Fig. 7. Intense characteristic blue emission of Ce^{3+} ion and green emission peaks of Tb^{3+} ion are observed to in the emission spectra of all samples under the excitation of 332 nm UV light. When the Ce^{3+} doping concentration is fixed, the intensity of Ce^{3+} emission decreases monotonously with the increase of Tb^{3+}, while the intensity of Tb^{3+} emission improves rapidly, even reaches a maximum at $x = 0.15$, and then remarkably decreases due to concentration quenching. This effect is due to a higher Tb^{3+} mole concentration ($x = 0.15$). The distance between Tb^{3+} ions becomes closer and the energy migration to a second Tb^{3+} becomes higher, thus, concentration quenching of Tb^{3+} occurs. This phenomenon indicates that the quenching concentration of Tb^{3+} is 15% ($x = 0.15$). The result is also related to the energy transfer probability occurring from Ce^{3+} ion to Tb^{3+} ion.

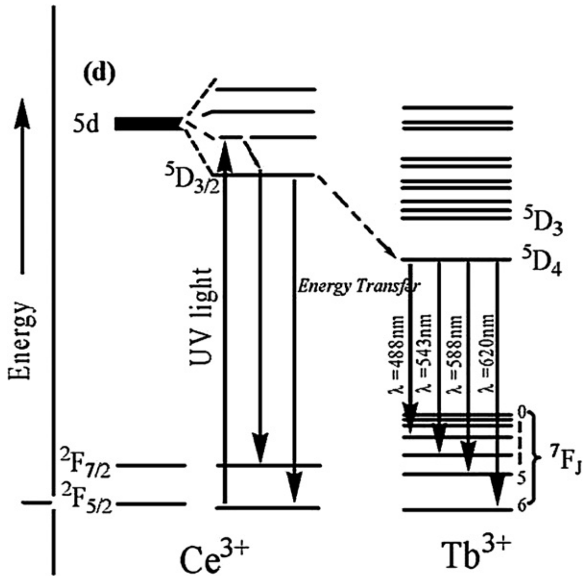

Fig. 6. The energy level diagram and visible emission transitions for Ce^{3+} ion and Tb^{3+} ion in the $Sr_2B_2O_5$:0.02Ce^{3+},0.15Tb^{3+} phosphors. The energy transfer process from Ce^{3+} to Tb^{3+} in the $Sr_2B_2O_5$:0.02Ce^{3+},0.15Tb^{3+} phosphors.

The energy-transfer efficiency (η_T) of Ce^{3+} to Tb^{3+} can be expressed by the following equation: [20]

$$\eta_T = 1 - \frac{I_S}{I_{S0}} \tag{1}$$

where I_{S0} and I_S are the luminescence intensities of the sensitizer in the absence (Ce^{3+}) and presence of the activator (Tb^{3+})

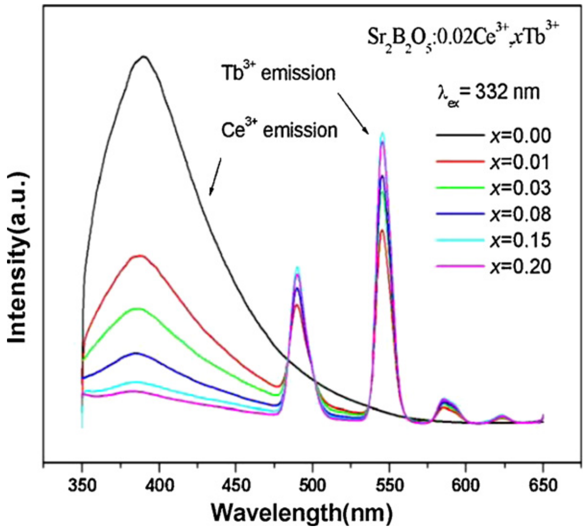

Fig. 7. The emission spectra of $Sr_2B_2O_5$:0.02Ce^{3+},$x$$Tb^{3+}$, with x equal to 0, 0.01, 0.03, 0.08, 0.15, and 0.20, respectively.

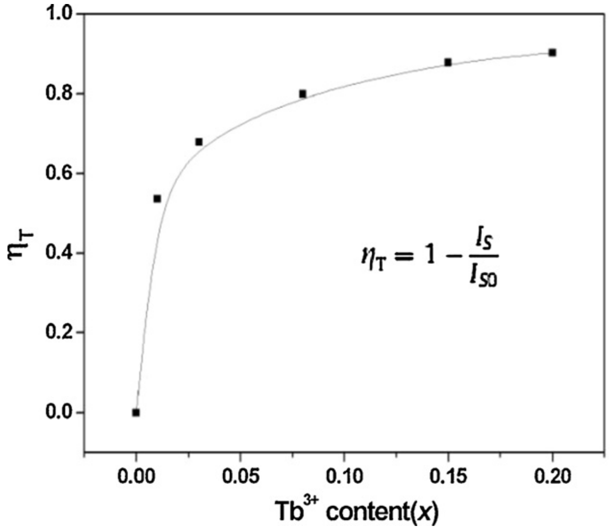

Fig. 8. Energy transfer efficiency (η_{ET}) of $Sr_2B_2O_5$:$0.02Ce^{3+}$,xTb^{3+} phosphors ($x = 0$, 0.01, 0.03, 0.08, 0.15, and 0.20).

respectively. The energy transfer efficiency from Ce^{3+} to Tb^{3+} in $Sr_2B_2O_5$:$0.02Ce^{3+}$,xTb^{3+} phosphors are calculated to be 0, 0.54, 0.68, 0.80, 0.87 and 0.90, respectively. Furthermore, it can be plotted as a function of Tb concentrations ($x = 0.00$, 0.01, 0.03, 0.08, 0.15, and 0.20) (Fig. 8), which clearly shows the relationship between energy transfer efficiency (η_T) and Tb^{3+} concentration. With increasing Tb^{3+} concentration, the value η_T is observed to increase gradually. The largest energy transfer efficiency can be found as 90%. All the phenomenon luminescent spectra and the energy transfer efficiency prove that the energy transfer process from Ce^{3+} ion to Tb^{3+} ion is very efficient.

4. Conclusions

A blue and green double-color emitting phosphor $Sr_2B_2O_5$:$0.02Ce^{3+}$,xTb^{3+} phosphors was successfully prepared by the solid-state reaction. The $Sr_2B_2O_5$:$0.02Ce^{3+}$,xTb^{3+} phosphors shows two emission bands located at blue (392 nm) and green band (545 nm) spectra region originated from the transition of Ce^{3+} and Tb^{3+}, respectively. The relative intensities of which change with the rare earth dopant concentrations Ce^{3+} ion and Tb^{3+} ion. The spectroscopic data indicate that the Ce^{3+} ion to Tb^{3+} ion energy-transfer process takes place in the host of $Sr_2B_2O_5$. With increasing Tb^{3+} ion concentration, the energy transfer efficiency (η_T) is observed to increase gradually, and even reach up to 90%. This result clearly demonstrates that the phosphor $Sr_2B_2O_5$:Ce^{3+},Tb^{3+} could be considered as one of double emission phosphor for n-UV excited white light emitting diodes.

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