Front. Chem. China 2007, 2(4): 393–397
DOI 10.1007/s11458-007-0074-9

RESEARCH ARTICLE

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Microwave synthesis and photoluminescent properties of Sr_2CeO_4/Ln^{3+} (Ln = Er, Ho, Tm)

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Abstract Sr_2CeO_4/Ln^{3+} (Ln = Er, Ho, Tm) phosphors were synthesized with the microwave radiation method for the first time. The luminescent properties of the samples were investigated and the up-conversion luminescence of Er^{3+}, Ho^{3+} and Tm^{3+} doped Sr_2CeO_4 phosphors was observed. The spectra indicate that the energy transfer takes place from the triplet excited state of MLCT (metal-to-ligand charge transfer) state for Sr_2CeO_4 (sensitizer) to the rare earth ions (activator).

Keywords Sr_2CeO_4, Er^{3+}, Ho^{3+}, Tm^{3+}, luminescence, up-conversion luminescence, microwave radiation

1 Introduction

In recent years, the research activities on the up-conversion about the doping of rare earth ions underwent a tremendous upsurge. Among them, Er^{3+}, $Ho^{3+}$and Tm^{3+} ions are of particular interest due to their abundant energy level and fluorescence emissions in the regions of visible lights and near infrared rays [1]. In 1998, Danielson et al. [2] reported a novel blue luminescent material, Sr_2CeO_4, obtained by use of a combinatorial technique. This material was found to exhibit efficient blue-white luminescence under the excitation with ultraviolet (UV) lights, X-rays or cathode rays. Several researchers also investigated the detailed emission mechanism of this phosphor. Sr_2CeO_4 is a phosphor with 100% active center concentration, so all CeO_6 octahedra may be considered as luminescence centers and the quantum efficiency is very high. It owns a broad emission band, and may also act as a sensitizer to transfer the absorbed energy to the dopants (activator), such as the rare earth ions [3]. The rare earth-doped Sr_2CeO_4 (Sr_2CeO_4: Ln^{3+}; Ln = Eu^{3+}, Sm^{3+}) was investigated, and the characteristic emissions corresponding to doped rare earth ions affected by energy transfer were observed in literatures [4−8]. However, only one paper has been published concerning the doping of Er^{3+}, Ho^{3+} and Tm^{3+}ions in Sr_2CeO_4. In that paper, Sr_2CeO_4/Ln^{3+} (Ln = Er, Ho, Tm) phosphor particles were prepared by using an emulsion liquid membrance (ELM) system [9] and the energy transfer from Ce^{4+} to Ln^{3+} was investigated, but the effect of the concentration of dopant ions (Ln^{3+}) on the emission of Sr_2CeO_4/Ln^{3+} was not discussed in detail, and the methods of preparing Sr_2CeO_4 are complicated by demanding a very high calcination temperature.

The method of microwave irradiation, which shows the characteristic of the rapid and homogeneous heating rate [10] and the capability of producing luminescent materials with remarkably improved performance, has become one of the most distinctive methods in the synthesis of new materials. In this paper, Sr_2CeO_4/Ln^{3+} (Ln = Er, Ho, Tm) phosphors were synthesized by the microwave irradiation method for the first time. The effect of the concentration of dopant ions (Ln^{3+}) on the luminescence of Sr_2CeO_4/Ln^{3+} was investigated and the up-conversion luminescence of Er^{3+}, Ho^{3+} and Tm^{3+} doped Sr_2CeO_4 phosphors was observed.

2 Experimental

2.1 Materials and methods

The starting materials are: $SrCO_3$, CeO_2, Er_2O_3, Ho_2O_3 (all of 99.99% purity). The experimental setup used for microwave heating is an LGMG-501371 domestic microwave oven from the electronic limited company of Lejin Electronic Ltd. in Tianjin. Excitation and emission spectra were recorded at the room temperature with a Hitachi F-4500 spectrofluorometer from the Rily Company of Japan.

2.2 Synthesis of Sr_2CeO_4/Ln^{3+} (Ln = Er, Ho, Tm)

Following Sr_2CeO_4/Ln^{3+} compounds can be prepared by using a mixture of Sr, Ln and Ce with a ratio of $(2-x):x:1$.

Translated from *Journal of Hebei Normal University* (*Natural Science Edition*), 2007, 31(2): 212–216 [译自: 河北师范大学学报 (自然科学版)]

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(1) Sr_2CeO_4/xEr^{3+} ($x = 0.01, 0.02, 0.04, 0.08$)
(2) Sr_2CeO_4/xHo^{3+} ($x = 0.01, 0.02, 0.05, 0.08, 0.10$)
(3) Sr_2CeO_4/xTm^{3+} ($x = 0.01, 0.02, 0.05, 0.08, 0.10$)

In the microwave synthesis experiments, the $SrCO_3$, CeO_2, Er_2O_3, Ho_2O_3 and Tm_2O_3 were weighed respectively, fully mixed and placed in a small crucible within a dual crucible system. The microwave irradiation was carried out with adjustable power and heating time in the presence of an appropriate amount of MnO_2 as microwave absorbent. The obtained sample was taken out and cooled. In a preliminary study, the best reaction condition for the microwave synthesis of Sr_2CeO_4/Ln^{3+} was determined to be 560 w for 60 min. Thus, the power of the microwave oven was set up to 560 w and the heating time 60 min for the synthesis.

2.3 Spectra measurements

The emission and excitation spectra of the samples were recorded by using a Hitachi F-4500 spectrofluorometer. The controlling slit, excitation and emission rasters are all 2.5 nm.

3 Result and discussion

3.1 Luminescent properties of Sr_2CeO_4/Er^{3+}

Figure 1 shows the emission spectra of Sr_2CeO_4/xEr^{3+} when excited with $\lambda = 283$ nm. As shown in Fig. 1, the emission spectra of Sr_2CeO_4/xEr^{3+} are composed of broadband peaks and line peaks. A broadband peak around 470 nm appears between 400 and 540 nm originating from the Ce^{4+}-O^{2-} MLCT (metal-to-ligand charge transfer). A number of line emission peaks are attached to the broadband peak originating from the 4f→4f excited state of Er^{3+}, which could be assigned to $^2H_{11/2}\rightarrow{}^4I_{15/2}$ (525 nm) and $^4S_{3/2}\rightarrow{}^4I_{15/2}$ (548 nm) transitions, respectively. Among them, the highest peak is located at 548 nm. In addition, a splitting occurs at the line emission peaks of Er^{3+} ions due to the crystal field interaction, and the splitting extents of emission peaks are determined by the symmetry of the crystal field around the host materials [12,13].

Sr_2CeO_4/xEr^{3+} shows various emission spectra by changing the concentration of Er^{3+} ions, as shown in Fig. 1. Both the intensities of the Ce^{4+}-O^{2-} MLCT and Er^{3+} characteristic emission at 525 nm diminish while the highest intensity of Er^{3+} emission lines at 548 nm starts to increase and then diminishes by increasing the concentration of Er^{3+} ions, x, from 0.01 to 0.08. Among them, the highest emission intensity appears at $x = 0.02$. For $x = 0.08$, the main emission is the line peaks of Er^{3+} ions, and the broadband peak around 470 nm originating from the Ce^{4+}-O^{2-} MLCT is basically quenched. The results indicate that the energy transfer takes place from the triplet excited state of MLCT (metal-to-ligand charge transfer) state for Sr_2CeO_4 (sensitizer) to Er^{3+} ions [9].

In the spectra, the characteristic emission band of Er^{3+} ions is quite narrow and the intensity is very low. This is because the electron configuration of Er^{3+} ions is $4f^{11}$ and the ground state is $^4I_{15/2}$. The core-polarization and orbital crossing in $5s^25p^6$ electron shell cause the energy of unsaturated 4f electron to be higher than that of $5s^25p^6$. Moreover, the orbital radius of $5s^25p^6$ electrons (0.176 nm) is wider than that of 4f (0.100 nm). Consequently, the 4f electrons are effectively shielded by the $5s^25p^6$ electron shell. The f-f transition is the transition of 4f electrons within the shell which makes the electron-phonon coupling between the rare earth ions and the crystal lattice very weak, even far lower than the spin-orbit interaction. The effect of the host crystal field on the 4f electrons shows a magnitude at about 10^1–10^2 cm^{-1}, and thus the characteristic emission of Er^{3+} ions is very narrow in band width and the intensity is very low [10].

Figure 2 shows the excitation spectra of $Sr_2CeO_4/0.02Er^{3+}$ when excited with $\lambda = 468$ and 548 nm respectively. As shown in Fig. 2, a broadband peak originating from the Ce^{4+}-O^{2-} MLCT is observed in both excitation spectra 1 and 2, in which the highest peak appears at 283 nm and a shoulder peak is observed at 336 nm. In addition, weak peaks are observed at 379 and 489 nm when excited with $\lambda = 548$ nm, which correspond to the $^4I_{15/2}\rightarrow{}^4G_{11/2}$ and $^4I_{15/2}\rightarrow{}^4F_{7/2}$ t transitions of Er^{3+} ions [11] respectively. Because Er^{3+} ions show the $^4S_{3/2}\rightarrow{}^4I_{15/2}$ characteristic transition with $\lambda = 548$ nm, the

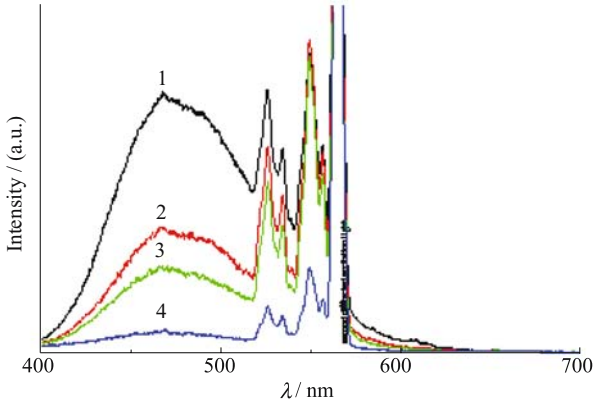

Fig. 1 Emission spectra of Sr_2CeO_4: xEr^{3+} ($\lambda_{ex} = 283$ nm)
1. $x = 0.01$; 2. $x = 0.02$; 3. $x = 0.04$; 4. $x = 0.08$

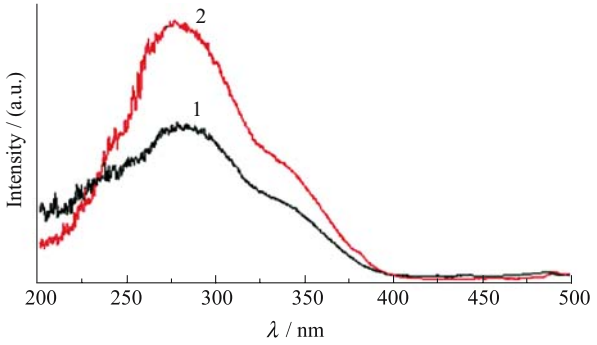

Fig. 2 Emission spectra of Sr_2CeO_4: $0.02Er^{3+}$
1. $\lambda_{em} = 468$ nm; 2. $\lambda_{em} = 548$ nm

luminescence of Er^{3+} ions is mainly attributed to the energy transfer from the triplet excited state of the MLCT state to the intra-4f excited state of Er^{3+} ions through Ce^{4+}-O^{2-} [9].

Figure 3 shows the emission spectra of Sr_2CeO_4/xEr^{3+} when excited with $\lambda = 658$ nm. As shown in Fig. 3, two obvious peaks appear at 525 and 548 nm, which are assigned to the green up-conversion luminescence of the $^2H_{11/2}\rightarrow{}^4I_{15/2}$ and $^4S_{3/2}\rightarrow{}^4I_{15/2}$ transitions respectively. And when Er^{3+} ions are doped at 4%, the green up-conversion luminescence is the best [1,14].

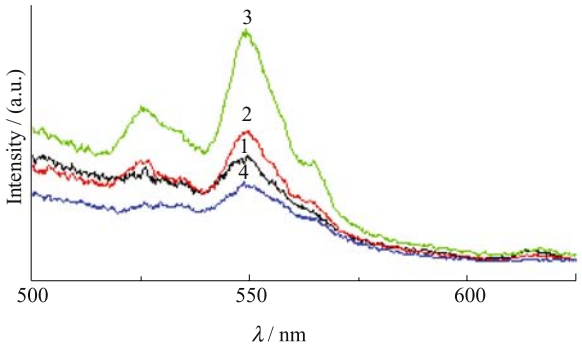

Fig. 3 Emission spectra of $Sr_2CeO_4:xEr^{3+}$ ($\lambda_{ex} = 658$ nm)
1. $x = 0.01$; 2. $x = 0.02$; 3. $x = 0.04$; 4. $x = 0.08$

3.2 Luminescent properties of Sr_2CeO_4/Ho^{3+}

Figure 4 shows the emission spectra of Sr_2CeO_4/xHo^{3+} when excited with $\lambda = 283$ nm. As shown in Fig. 4, the emission spectra of Sr_2CeO^4/xHo^{3+} are composed of broadband peaks and line peaks. A broadband peak around 470 nm appears between 400 and 540 nm originating from the Ce^{4+}-O^{2-} MLCT. A number of line emission peaks are attached to the broadband peak originating from the 4f→4f excited state of Ho^{3+}, which are assigned to the $^5F_3\rightarrow{}^5I_8$(491 nm), $^5S_2\rightarrow{}^5I_8$(542, 549 nm) and $^5G_6$, $^5F_1\rightarrow{}^5I_7$(614 nm) transitions [12,15−17], respectively. Among them, the strongest peak appears at 549 nm.

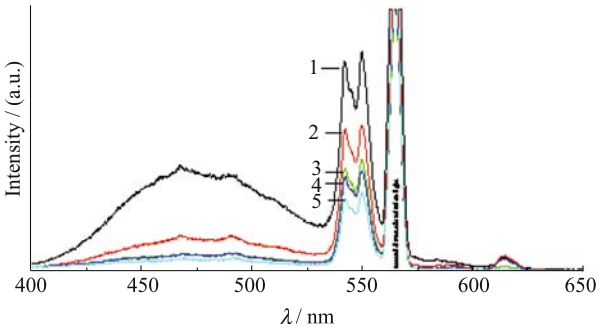

Fig. 4 Emission spectra of $Sr_2CeO_4:xHo^{3+}$ ($\lambda_{ex} = 283$ nm)
1. $x = 0.01$; 2. $x = 0.02$; 3. $x = 0.05$; 4. $x = 0.08$; 5. $x = 0.10$

Sr_2CeO_4/xHo^{3+} shows various emission spectra by changing the concentration of Ho^{3+} ions, as shown in Fig. 4. Both the emission intensities of the Ce^{4+}-O^{2-} MLCT and the $^5F_3\rightarrow{}^5I_8$ (491 nm), $^5S_2\rightarrow{}^5I_8$ (542, 549 nm) characteristic transitions of Ho^{3+} ions are gradually diminished by increasing the concentration of Ho^{3+} ions while that of $^5G_6$, $^5F_1\rightarrow{}^5I_7$ (614 nm) transition almost does not change. For $x \geqslant 0.05$, the main emission is the line peaks of Ho^{3+} ions, and the broadband peak around 470 nm originating from the Ce^{4+}-O^{2-} MLCT is very weak. The results indicate that the energy transfer takes place from the triplet excited state of MLCT (metal-to-ligand charge transfer) state for Sr_2CeO_4 (sensitizer) to Ho^{3+} ions [9].

When x changes from 0.01 to 0.10, the strongest emission peak of Ho^{3+} around 549 nm is gradually diminished, and the highest peak is at $x = 0.01$. It is likely that the distance diminishes and the interaction increases continuously between Ho^{3+} ions by increasing the concentration of Ho^{3+} ions. Thus, it is inevitable that the energy transfer of some non-radiative transitions between Ho^{3+} ions occurs. Besides the multiphonon decay, the life of the emission energy state is also decreased due to the process of the cross-relaxation between Ho^{3+} ions with the same properties, so the luminescence intensity diminishes [17].

Figure 5 shows the excitation spectra of $Sr_2CeO_4/0.01Ho^{3+}$ when excited with $\lambda = 468$ and 549 nm, respectively. As shown in Fig. 5, a broadband peak originating from the Ce^{4+}-O^{2-} MLCT is observed in both excitation spectra 1 and 2, in which the highest peak appears at 283 nm and a shoulder peak is observed at 336 nm. In addition, a weak line peak is observed at 421 nm when excited with $\lambda = 549$ nm, corresponding to the $^5I_8\rightarrow{}^5G_5$ transition of Ho^{3+} ions [16]. Because Ho^{3+} ions show the $^5S_2\rightarrow{}^5I_8$ characteristic transition with $\lambda = 549$ nm, the luminescence of Ho^{3+} ions is mainly attributed to the energy transfer from the triplet excited state of the MLCT state to the intra 4f excited state of Ho^{3+} ions through Ce^{4+}-O^{2-} [9].

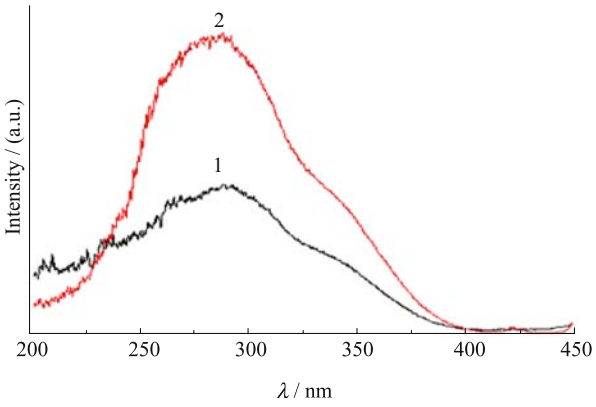

Fig. 5 Excitation spectra of $Sr_2CeO_4:0.01Ho^{3+}$
1. $\lambda_{em} = 468$ nm; 2. $\lambda_{em} = 549$ nm

Figure 6 shows the emission spectra of Sr_2CeO_4/xHo^{3+} when excited with $\lambda = 880$ nm. As shown in Fig. 6, three obvious peaks appear at 481, 550 and 587 nm, which are assigned to the blue up-conversion luminescence of the $^5F_3\rightarrow{}^5I_8$ transition, green of the $^5S_2\rightarrow{}^5I_8$ transition, and

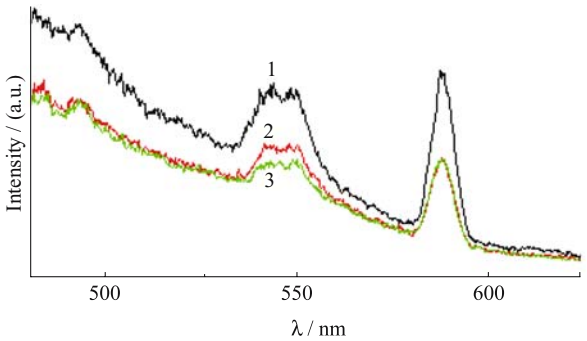

Fig. 6 Emission spectra of Sr_2CeO_4:xHo^{3+} (λ_{ex} = 880 nm)
1. x = 0.01; 2. x = 0.05; 3. x = 0.10

yellow of the $^5G_4 \rightarrow {}^5I_6$ transition [18,19], respectively. And when Ho^{3+} ions are doped at 1%, the results are the best. Also as shown in Fig. 6, the up-conversion fluorescence is a broadband emission around 550 nm between 540 and 557 nm. This emission energy corresponds to the transition from the $^5S_2$ state to the ground state $^5I_8$ or it may be caused by the transition from the $^5F_4$, $^5S_2 \rightarrow {}^5I_8$, since the energies of $^5S_2$ and $^5F_4$ are close to each other [20,21]. However, the up-conversion fluorescence mechanism of Ho^{3+} ions is a step absorption of up-conversion in a single ion. Therefore, the up-conversion broadband of fluorescence emission from 540 to 557 nm, corresponding to the $^5S_2 \rightarrow {}^5I_8$ transition, is observed when excited with λ = 880 nm. In addition, the ground state $^5I_8$ splits into 17 levels in theory, so the band of fluorescence emission is very wide [19].

3.3 Luminescent properties of Sr_2CeO_4/Tm^{3+}

Figure 7 shows the emission spectra of Sr_2CeO_4/xTm^{3+} when excited with λ = 283 nm. As shown in Fig. 7, the emission spectra of Sr_2CeO_4/xTm^{3+} are composed of broadband peaks and line peaks. A broadband peak around 470 nm appears between 400 and 540 nm originating from the Ce^{4+}-O^{2-} MLCT. A number of line emission peaks are attached to the broadband peak originating from the 4f→4f excited state of Tm^{3+}, which are assigned to the $^1G_4 \rightarrow {}^3H_6$ (476 nm) and $^1D_2 \rightarrow {}^3H_5$ (544 nm) transitions, respectively [9,12]. Among them, the strongest peak appears at 476 nm. In addition, a splitting occurs at the emission peaks of Tm^{3+} ions from the $^1G_4 \rightarrow {}^3H_6$ (476 nm) and $^1D_2 \rightarrow {}^3H_5$ (544 nm) transitions due to the crystal field interaction. And the splitting extents of emission peaks are determined by the symmetry of the crystal field around the host material. Sr_2CeO_4/xTm^{3+} shows various emission spectra by changing the concentration of Tm^{3+} ions, as shown in Fig. 7. Both the emission intensities of the Ce^{4+}-O^{2-} MLCT and the $^1G_4 \rightarrow {}^3H_6$ (476 nm) transition of Tm^{3+} ions are gradually decreased by increasing the concentration of Tm^{3+} ions, but the emission peak of the $^1D_2 \rightarrow {}^3H_5$ (544 nm) transition no longer occurs with $x \geqslant 0.02$. For $x \geqslant 0.05$, the main emission is the line peaks of Tm^{3+} ions, and the broadband peak around 470 nm originating from the Ce^{4+}-O^{2-} MLCT is basically quenched. The results indicate that the energy transfer takes place from the triplet excited state of MLCT (metal-to-ligand charge transfer) state for Sr_2CeO_4 (sensitizer) to Tm^{3+} ions [9].

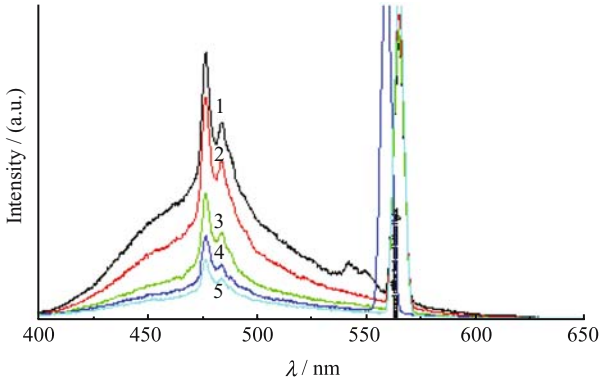

Fig. 7 Emission spectra of Sr_2CeO_4: Tm^{3+} (λ_{ex} = 283 nm)
1. x = 0.01; 2. x = 0.02; 3. x = 0.05; 4. x = 0.08; 5. x = 0.10

Figure 8 shows the excitation spectra of $Sr_2CeO_4/0.01Tm^{3+}$ and $Sr_2CeO_4/0.05Tm^{3+}$ when excited with λ = 476 nm. As shown in Fig. 8, a broadband peak originating from the Ce^{4+}-O^{2-} MLCT is observed in both excitation spectra 1 and 2. Because Tm^{3+} ions show the $^1G_4 \rightarrow {}^3H_6$ characteristic transition with λ = 476 nm, the luminescence of Tm^{3+} ions is mainly attributed to the energy transfer from the triplet excited state of the MLCT state to the intra 4f excited state of Tm^{3+} ions through Ce^{4+}-O^{2-} [9].

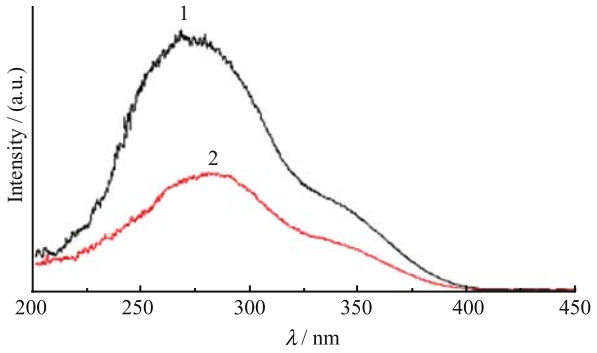

Fig. 8 Excitation spectra of (λ_{em} = 476 nm)
1. Sr_2CeO_4: 0.01 Tm^{3+}; 2. Sr_2CeO_4: 0.05 Tm^{3+}

Figure 9 shows the emission spectra of Sr_2CeO_4/xTm^{3+} when excited with λ = 650 nm. As shown in Fig. 9, the blue up-conversion luminescence appears at 476 nm, which is

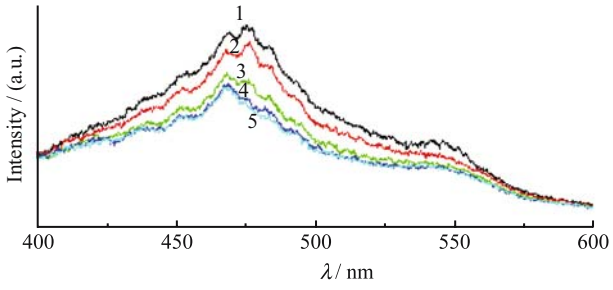

Fig. 9 Emission spectra of Sr_2CeO_4:xTm^{3+} (λ_{ex} = 650 nm)
1. x = 0.01; 2. x = 0.02; 3. x = 0.05; 4. x = 0.08; 5. x = 0.10

assigned to the $^1G_4 \rightarrow {}^3H_6$ transition. And when the doping amount of Tm^{3+} ions is 1%, the blue up-conversion luminescence is the best [14,22]. In addition, a group of weak broadband emissions appears at 544 nm, which are assigned to the $^1D_2 \rightarrow {}^3H_5$ transition [12].

4 Conclusions

(1) Sr_2CeO_4/Ln^{3+} (Ln = Er, Ho, Tm) phosphors are synthesized by the microwave irradiation method for the first time. Compared with the methods in literature, our method shows that the heating time is shortened to 60 min, easy to operate and no need for a high calcinating temperature.

(2) Sr_2CeO_4/Ln^{3+} (Ln = Er, Ho, Tm) shows various fluorescence spectra by changing the concentration of Ln^{3+} ions. The spectra indicate that the energy transfer takes place from the triplet excited state of MLCT (metal-to-ligand charge transfer) state for Sr_2CeO_4 (sensitizer) to the rare earth ions (activator).

(3) The spectra of Sr_2CeO_4/Er^{3+} show that the green up-conversion luminescence of Er^{3+} ions appear at 525 and 548 nm when excited with $\lambda = 658$ nm. And when Er^{3+} ions are doped at 4%, the green up-conversion luminescence is the best. The spectra of Sr_2CeO_4/Ho^{3+} show that a blue up-conversion luminescence of Ho^{3+} ions appears at 525 nm, green at 550 nm and yellow at 548 nm, respectively, when excited with $\lambda = 880$ nm. And when Ho^{3+} ions are doped at 1%, the green up-conversion luminescence is the best. The spectra of Sr_2CeO_4/Tm^{3+} show that a blue up-conversion luminescence of Tm^{3+} ions appears at 476 nm when excited with $\lambda = 650$ nm. And when Tm^{3+} ions are doped at 1%, the blue up-conversion luminescence is the best.

Acknowledgements The authors are grateful to Miss Rong He, Zhen Wei and Qing Liang of Hebei Normal University for assistance in translation. The authors are also grateful to the National Natural Science Foundation of China (Grant No. 50472020) and Doctor Foundation of Hebei Normal Univrsity (Grant No. 103261) for the financial support.

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