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# Photoluminescence and energy transfer studies on $Eu^{2+}$ and $Ce^{3+}$ co-doped $SrCaSiO_4$ for white light-emitting-diodes

Hong Yu[a], Yawen Lai[b], Guimei Gao[a], Li Kong[a], Guanghuan Li[a], Shucai Gan[a,*], Guangyan Hong[c]

[a] College of Chemistry, Jilin University, Changchun 130026, PR China
[b] College of Geoexploration Science and Technology, Jilin University, Changchun 130026, PR China
[c] State Key Laboratory of Rare Earth Resource Utilization, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, PR China

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ABSTRACT

The luminescence of $SrCaSiO_4:Eu^{2+}$, $Ce^{3+}$ is studied as a potential ultraviolet light-emitting diodes (UV-LEDs) phosphor that is capable of converting the ultraviolet emission of a UV-LED into green light with good luminosity. There are two emissions peaks peaking at 420 and 500 nm, respectively. The two emissions come from d–f transitions of $Ce^{3+}$ and $Eu^{2+}$, respectively. Effective energy transfer occurs in $Ce^{3+}/Eu^{2+}$ co-doped $SrCaSiO_4$ due to a part of spectral overlap between the emission of $Ce^{3+}$ and excitation of $Eu^{2+}$. Co-doping of $Ce^{3+}$ enhances the emission intensity of $Eu^{2+}$ greatly by transferring its excitation energy to $Eu^{2+}$. The $Ce^{3+}/Eu^{2+}$ energy transfer, thoroughly investigated by the diffuse reflection emission and excitation spectra, photoluminescence decay curves, is demonstrated to be in the mechanism of electric dipole–dipole interaction.

## 1. Introduction

White-light-emitting diodes (W-LEDs), the next-generation solid state lighting, have attracted much attention due to their superior features such as higher rendering index (CRI), higher luminosity efficiency, and lower energy consumption [1,2]. W-LEDs have been widely used as backlights for electronic devices and are anticipated to replace traditional fluorescent lamps for general illumination as their efficiency and color rendering properties are significantly improved [3]. The most common w-LEDs have been commendably realized using $YAG:Ce^{3+}$ as a broadband yellow phosphor coated on the blue InGaN chip [4–6], however, such a combination offers high luminescence efficiency but a poor color rendering index (<80) [7]. In order to overcome these disadvantages mentioned above, new class of phosphors, especially those can be effectively excited by ultraviolet or blue light and can emit strong blue, green, yellow or red light, gained importance [8–10]. In order to meet the optimum requirements of w-LEDs, tricolor phosphors with high stability and intense absorption in UV or near UV spectral region are in great demand [11].

Co-doping of the activators with f–d electron configuration, such as $Ce^{3+}$–$Eu^{2+}$, is an efficient method to obtain phosphors excited by UV or near UV [12,13]. $Eu^{2+}$ and $Ce^{3+}$ exhibit similar optical properties originating from the $4f^n \rightarrow 4f^{n-1}5d^1$ electronic transition, hosts with $Eu^{2+}$ as an activator also can be used for $Ce^{3+}$ doped phosphors, with the addition of $Ce^{3+}$, the luminous intensity of $Eu^{2+}$ emission enhanced. Silicates phosphors, such as: $Sr_2SiO_4:Ce^{3+}$, $Eu^{2+}$, $Li_2SrSiO_4:Ce^{3+}$, $Eu^{2+}$ [14,15] which show that co-doping of $Ce^{3+}$ could enhance the emission intensity of $Eu^{2+}$ greatly by transferring its excitation energy to $Eu^{2+}$ which due to a part of spectral overlap between the emission of $Ce^{3+}$ and excitation of $Eu^{2+}$. As far as we know, the luminescence property and the energy transfer of $SrCaSiO_4:Eu^{2+}$, $Ce^{3+}$ phosphors have not yet been investigated.

In the present work, we synthesized and studied the luminescence property and the energy transfer of $SrCaSiO_4:Eu^{2+}$, $Ce^{3+}$ phosphors. The photoluminescence properties were studied by measuring the diffuse reflection, emission and excitation spectra, and photoluminescence decay curves. The energy transfer from $Ce^{3+}$ to $Eu^{2+}$ exists in $SrCaSiO_4$ lattice, the emission intensity of $Eu^{2+}$ was enhanced by the energy transfer from $Ce^{3+}$ to $Eu^{2+}$. $SrCaSiO_4:Eu^{2+}$, $Ce^{3+}$ is a promising phosphor that can be effectively excited by UV and suitable for use in white LEDs.

## 2. Experimental

All the samples were prepared by a solid-state reaction technique. Analytical reagent grade (99.90%) $SrCO_3$, $CaCO_3$, $H_2SiO_3$, $Li_2CO_3$ were obtained from Beijing Chemical Reagent Research Institute of China and spectrographically pure (99.99%) $Eu_2O_3$, $Ce_2(C_2O_4)_3$ were supplied by Yuelong Rare Earth of China, were employed as reactants. $Li_2CO_3$ as the charge compensator, the charge compensation for the substitution of $Sr^{2+}$ by $Ce^{3+}$ is achieved by adding equimolar concentration of $Li^+$, the incorporation of $Li^+$ ions greatly enhance the luminescence intensity probably

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* Corresponding author. Tel.: +86 431 87652736.
*E-mail address:* gansc@jlu.edu.cn (S. Gan).

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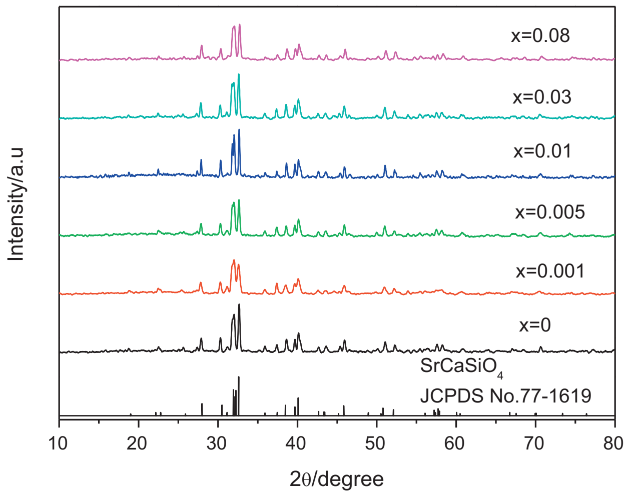

**Fig. 1.** XRD patterns of $SrCaSiO_4$:$x$$Eu^{2+}$ samples with different $Eu^{2+}$ concentration ($x$ = 0–0.08).

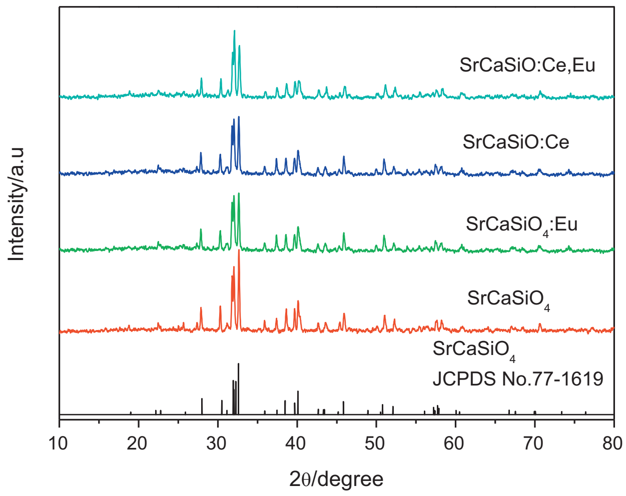

**Fig. 2.** XRD patterns of $SrCaSiO_4$, $SrCaSiO_4$:0.005 $Eu^{2+}$, $SrCaSiO_4$: 0.01 $Ce^{3+}$ and $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.01 $Ce^{3+}$.

due to the influence of charge compensation of alkali metal ions, and thus stabilize the structure and enhance the luminescence [16]. Stoichiometric mixtures were blended and milled thoroughly in an agate mortar and then transferred into the corundum crucible, subsequently calcined at successively at 1350 °C for 2 h in a reducing atmosphere.

All samples were characterized by X-ray powder diffraction (XRD). The XRD measurements were performed on a Bruke/D8-Advance with Cu Kα radiation ($\lambda$ = 1.518 Å). The operation voltage and current were maintained at 40 kV and 40 mA, respectively. A scan rate of 2°/min was applied to record the patterns in the range of $2\theta$ = 10–80°. The excitation and emission spectra were measured by a Hitachi F-7000 fluorescence spectrofluorometer equipped with a 150 W Xe lamp. The luminescence decay curves were obtained from a Lecroy Wave Runner 6100 Digital Oscilloscope (1 GHz). The diffuse reflectance spectra were obtained by a Hitachi U-4100 spectrophotometer with the reflection of black felt reflection (3%) and white $Al_2O_3$ (reflection 100%) in the wavelength region of 200–600 nm. All the experiments were performed at room temperature.

## 3. Results and discussion

The XRD patterns of $SrCaSiO_4$:$x$$Eu^{2+}$ ($x$ = 0–0.08) with different content of $Eu^{2+}$ as dopant are shown in Fig. 1. All of the diffraction peaks are accord with $SrCaSiO_4$ (JCPDS card No. 77–1619). All these samples are of single phase without any impurities. This indicates that doping of $Eu^{2+}$ in the $SrCaSiO_4$ with such a small concentration does not influence the crystallizing behavior of the host. The crystal structure of $SrCaSiO_4$:$Eu^{2+}$ has been refined to orthorhombic [17], space group *Pmnb* with $a$ = 0.564 nm, $b$ = 0.703 nm, $c$ = 0.964 nm, $V$ = 0.383 nm$^3$. In this work, the radius of $Eu^{2+}$ (0.117 nm) is similar to that of $Sr^{2+}$ (0.118 nm), possibly, the $Eu^{2+}$ ions preferably occupy the $Sr^{2+}$ ions [18].

The XRD patterns of $SrCaSiO_4$, $SrCaSiO_4$:0.005 $Eu^{2+}$, $SrCaSiO_4$:0.01 $Ce^{3+}$ and $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.01 $Ce^{3+}$ samples are shown in Fig. 2. All of the diffraction peaks are accord with $SrCaSiO_4$ (JCPDS card No. 77–1619). This indicates that doping of $Eu^{2+}$ and $Ce^{3+}$ both can adulterate the $SrCaSiO_4$ with such a small concentration and do not influence the crystallizing behavior of the host. The radius of $Ce^{3+}$ (0.101 nm) is similar to that of $Ca^{2+}$ (0.100 nm), possibly, the $Ce^{3+}$ ions preferably occupy the $Ca^{2+}$ ions [18].

The photoluminescence (PL) excitation and emission spectra of $Eu^{2+}$ doped $SrCaSiO_4$ phosphor samples with 0.5 mol% concentration are shown in Fig. 3. It can be seen clearly that the excitation spectrum covers the region from 250 to 400 nm with a maximum at 330 nm. The excitation band attributes to the transition of $4f^7 \rightarrow 4f^65d^1$ and the excitation from 250 to 400 nm matches well with UV LED emission. The emission spectrum excited by 330 nm covers the broad band from 400 to 650 nm with a maximum at 500 nm, which corresponds to the transition of $4f^65d^1 \rightarrow 4f^7$.

The doping concentration of luminescent centers is an important factor influencing the phosphor performance. Therefore, it is necessary to confirm the optimum doping concentration. The emission spectra of $SrCaSiO_4$:$x$$Eu^{2+}$ ($x$ = 0.001–0.08) phosphors with different $Eu^{2+}$ concentration under 330 nm excitation are shown in Fig. 4. It can be seen that no distinct diversifications about the shapes and positions of the emission spectra when the concentration of $Eu^{2+}$ varies in a wide range. The intensity of all of the emission are enhanced significantly with the $Eu^{2+}$ concentration increasing, and gradually decreases as the doping concentration becomes higher than $x$ = 0.005. This may occurs because of exchange interaction, radiation reabsorption, or multipole–multipole interaction [19]. In the present case, radiation reabsorption dues to spectral overlap alone cannot be fully responsible for nonradiative energy transfer between the $Eu^{2+}$ ions. Hence, the process of energy transfer should be electric multipole–multipole interaction. The probability of energy transfer between the $Eu^{2+}$ ions dues to multipole–multipole interaction is distance-dependent [20,21]. The distance between the $Eu^{2+}$ ions becomes smaller with the addition of $Eu^{2+}$ ions which leading to the high probability of energy transfer among the $Eu^{2+}$ ions, thereby increases the loss of energy [22,23], so the emission peak inten-

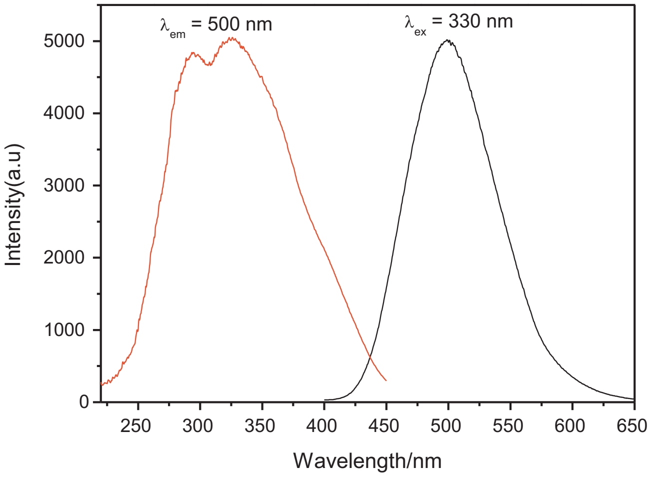

**Fig. 3.** Photoluminescence excitation and emission spectra of $SrCaSiO_4$:0.005 $Eu^{2+}$.

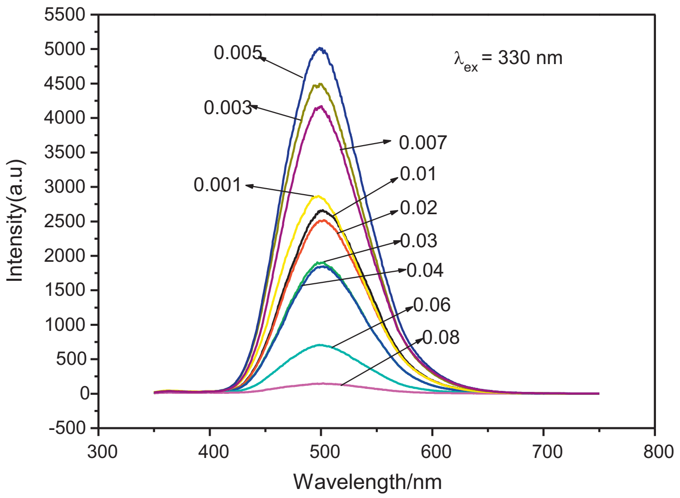

**Fig. 4.** Photoluminescence emission spectrum of SrCaSiO$_4$:$x$Eu$^{2+}$ with different Eu$^{2+}$ concentration under 330 nm excitation ($x$ = 0.001–0.08).

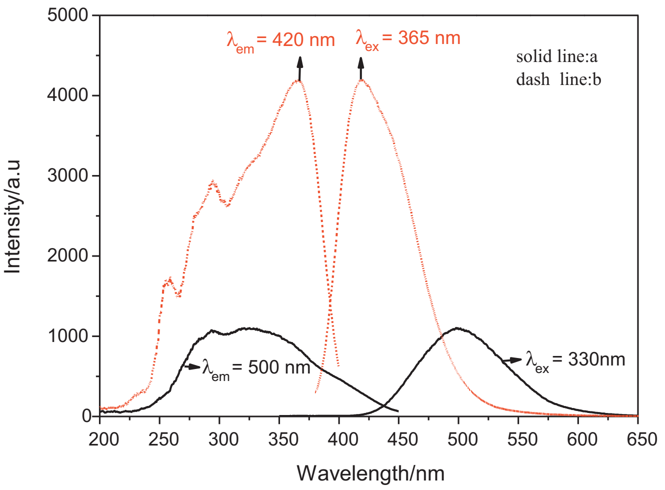

**Fig. 5.** Photoluminescence excitation and emission spectra of SrCaSiO$_4$:0.005 Eu$^{2+}$ (a) and SrCaSiO$_4$:0.01 Ce$^{3+}$ (b).

sity get lower. The Eu$^{2+}$ ions concentration is the main factor to influence the emission peak intensity.

The concentration quenching is due to energy transfer from one activator (donor) to another until the energy sink (acceptor) in the lattice is reached. Hence, the energy transfer will strongly depend on the distance ($R$) between the Eu$^{2+}$ ions, which can be obtained using the following equation [24]

$$R_c \approx 2\left[\frac{3V}{4\pi X_c Z}\right]^{1/3} \qquad (1)$$

where $X_c$ is the critical concentration, $Z$ is the number of trivalence of rare earth ions in the SrCaSiO$_4$ unit cell [$N$ = 4 in SrCaSiO$_4$], and $V$ is the volume of the unit cell ($V$ = 0.426 nm$^3$ in this case). The critical concentration is estimated to be about $x$ = 0.005, where the measured emission intensity and decay time begin to decrease. The critical distance ($R_c$) between the donor and acceptor can be calculated from the critical concentration, for which the nonradiative transfer rate equals the internal decay rate (radiative rate). Blasse assumed that, for the critical concentration, the average shortest distance between the nearest activator ions is equal to the critical distance. By taking the experimental and analytic values of $V$, $N$ and $X_c$ (0.383 nm$^3$, 4, 0.005, respectively), the critical distance $R_c$ is estimated by Eq. (1) to be about 3.3 nm.

The PL excitation and emission spectra of SrCaSiO$_4$:0.01 Ce$^{3+}$ phosphor sample and SrCaSiO$_4$:0.005 Eu$^{2+}$ phosphor sample are shown in Fig. 5, the concentration of Ce$^{3+}$ and Eu$^{2+}$ are the optimum concentration. It can be seen clearly that the intensity of the excitation and emission of Ce$^{3+}$ is about triple of Eu$^{2+}$. The excitation spectrum of SrCaSiO$_4$:0.01 Ce$^{3+}$ is monitored at 420 nm (emission of Ce$^{3+}$) with the peaks at 256, 290 and 365 nm, which are due to the transition of 4f$^1$ → 4f$^0$5d$^1$ of Ce$^{3+}$. The emission spectrum of SrCaSiO$_4$:0.01 Ce$^{3+}$ excited by 365 nm is a board band with a maximum at 420 nm. Compare the spectra of SrCaSiO$_4$:0.005 Eu$^{2+}$ and SrCaSiO$_4$:0.01 Ce$^{3+}$, it can be seen that there has a part of superposition between the excitation spectrum of SrCaSiO$_4$:0.005 Eu$^{2+}$ and the emission spectrum of SrCaSiO$_4$:0.01 Ce$^{3+}$. Hence, it may forecast that from Ce$^{3+}$ to Eu$^{2+}$ may occur effectively energy transfer.

The diffuse reflectance spectra of the SrCaSiO$_4$:0.005 Eu$^{2+}$, SrCaSiO$_4$:0.01 Ce$^{3+}$ and SrCaSiO$_4$:0.005 Eu$^{2+}$, 0.01 Ce$^{3+}$ samples are shown in Fig. 6. All the diffuse reflectance of the tests are 100% (take Al$_2$O$_3$ as the standard). As the result of the intense reflection of SrCaSiO$_4$:0.01 Ce$^{3+}$, the diffuse reflectance reaches to 180. Ce$^{3+}$ ions have three absorption bands are present at 233, 262 and 390 nm, respectively, all the bands are attribute to the electronic transition of 4f$^1$ → 4f$^0$5d$^1$ of Ce$^{3+}$. Eu$^{2+}$ ions have two intense absorption bands present at 249 and 350 nm, respectively, the absorption bands are attribute to the electronic transition of 4f$^7$ → 4f$^6$5d$^1$ of Eu$^{2+}$. From Fig. 6 and to compare the curves a and c, it can be seen that co-doped Ce$^{3+}$ in SrCaSiO$_4$:0.005 Eu$^{2+}$ phosphor can make the absorption bands increased, it is also a proof that co-doped Ce$^{3+}$ could enhance the luminous intensity of the SrCaSiO$_4$:0.005 Eu$^{2+}$ phosphors.

The PL excitation spectra of SrCaSiO$_4$:0.005 Eu$^{2+}$ (a: $\lambda_{em}$ = 500 nm) and SrCaSiO$_4$:0.005 Eu$^{2+}$, 0.01 Ce$^{3+}$ (b: $\lambda_{em}$ = 420 nm, c: $\lambda_{em}$ = 500 nm) samples are shown in Fig. 7. It shows that the luminous intensity of the excitation spectrum of SrCaSiO$_4$:0.005 Eu$^{2+}$, 0.01 Ce$^{3+}$ (Fig. 7c) is obviously stronger than that of SrCaSiO$_4$:0.005 Eu$^{2+}$ (Fig. 7a) under the same monitoring wavelength. It is a proof that co-doped Ce$^{3+}$ could enhance the luminous intensity of the SrCaSiO$_4$:0.005 Eu$^{2+}$ phosphors. In SrCaSiO$_4$:0.005 Eu$^{2+}$, 0.01 Ce$^{3+}$ excitation spectrum (Fig. 7c), we observed that the excitation peak of Ce$^{3+}$($\lambda_{ex}$ = 365 nm), it indicates that it exists the energy transfer from Ce$^{3+}$ to Eu$^{2+}$. In SrCaSiO$_4$:0.005 Eu$^{2+}$, 0.01 Ce$^{3+}$(Fig. 7b), we only observed the excitation spectrum of Ce$^{3+}$, while Ce$^{3+}$ co-doped SrCaSiO$_4$:0.005 Eu$^{2+}$ can make the excitation spectrum broader, it indicates that it is feasible to use the excitation wavelength of both Ce$^{3+}$ and Eu$^{2+}$. In order to optimizing the

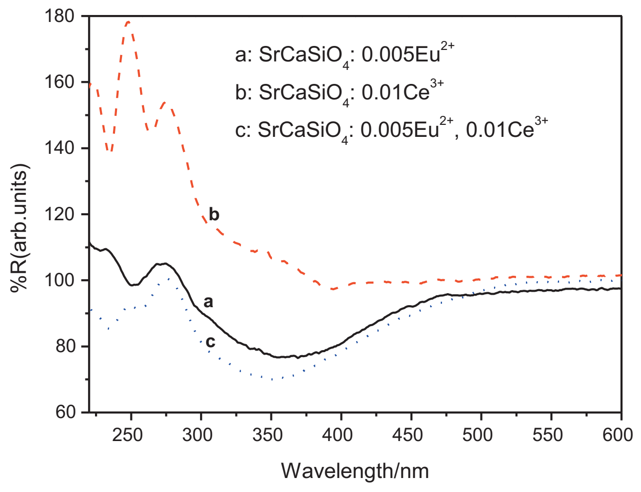

**Fig. 6.** The diffuse reflectance spectra of SrCaSiO$_4$:0.005 Eu$^{2+}$ (a), SrCaSiO$_4$:0.01 Ce$^{3+}$ (b), SrCaSiO$_4$:0.005 Eu$^{2+}$, 0.01 Ce$^{3+}$ (c).

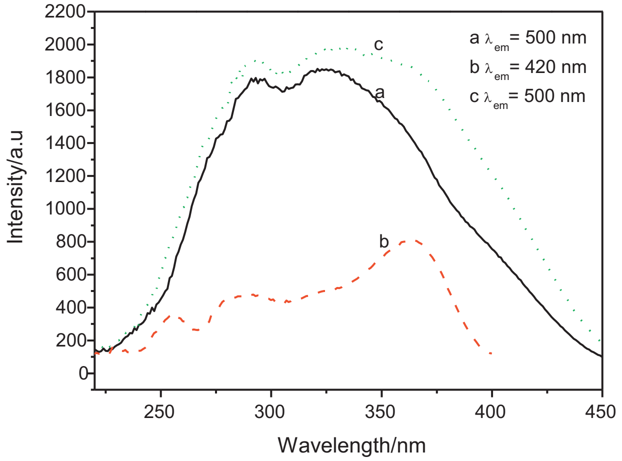

**Fig. 7.** The photoluminescence excitation spectrum of $SrCaSiO_4$:0.005 $Eu^{2+}$ (a: $\lambda_{em}$ = 500 nm) and $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.01 $Ce^{3+}$(b: $\lambda_{em}$ = 420 nm, c: $\lambda_{em}$ = 500 nm).

emission spectra, we fixed the $Eu^{2+}$ concentration to alter the $Ce^{3+}$ concentration.

The PL emission spectra ($\lambda_{ex}$ = 365 nm) of $SrCaSiO_4$:0.005 $Eu^{2+}$, $x$$Ce^{3+}$ and $SrCaSiO_4$:0.01 $Ce^{3+}$ phosphor samples are shown in Fig. 8. The emission spectrum of $SrCaSiO_4$:0.005 $Eu^{2+}$ (Fig. 8a) is a broad band with a maximum at 500 nm. The emission spectra of $SrCaSiO_4$:0.005 $Eu^{2+}$, $x$$Ce^{3+}$ (Fig. 8b–d) with two emission peaks at 420 and 500 nm, respectively. To compare the curves a–d, it shows that a few of $Ce^{3+}$ co-doped in $SrCaSiO_4$:0.005 $Eu^{2+}$ phosphors could not only increase the luminous intensity of $Eu^{2+}$ but also make the luminous color transfer from yellow-green to blue-green, when the optimum concentration of $Ce^{3+}$ at 0.01 mol, the luminous intensity of $SrCaSiO_4$:0.005 $Eu^{2+}$, $x$$Ce^{3+}$ phosphors is maximum, and then gradually decreases as the $Ce^{3+}$ concentration increasing ($x > 0.01$). It is a proof that co-doped $Ce^{3+}$ could enhance the luminous intensity of the $SrCaSiO_4$:0.005 $Eu^{2+}$ phosphors.

The fluorescence decay curves of $SrCaSiO_4$:0.005 $Eu^{2+}$ and $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.01 $Ce^{3+}$ samples are shown in Fig. 9. The typical decay curve of phosphors can be fitted using the single exponential method below, and the equation can be described by Blasse and Grabmarier [25], it is well-known that the decay behavior can be expressed as follows

$$I = I_0 \exp\left(\frac{-t}{\tau}\right) \qquad (2)$$

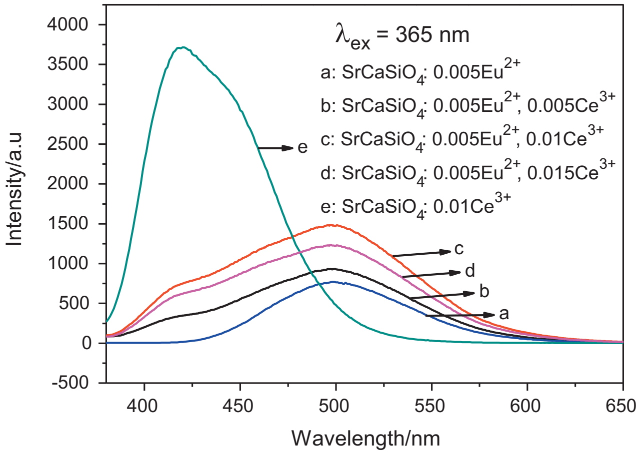

**Fig. 8.** Photoluminescence emission spectra of $SrCaSiO_4$:0.005 $Eu^{2+}$ (a), $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.005 $Ce^{3+}$ (b), $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.01 $Ce^{3+}$ (c), $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.015 $Ce^{3+}$ (d) and $SrCaSiO_4$:0.01 $Ce^{3+}$ (e).

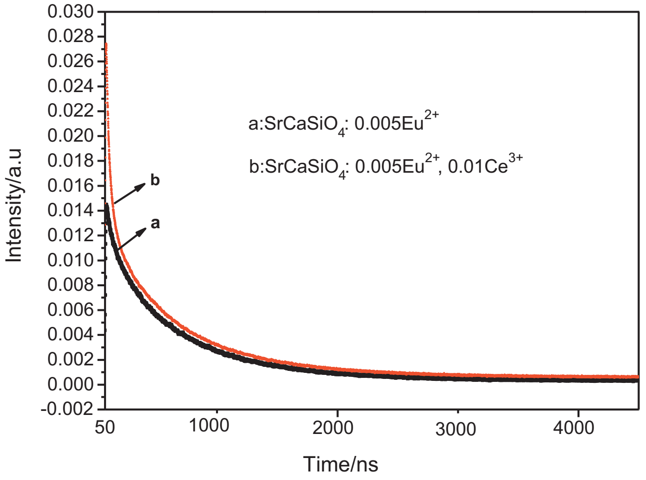

**Fig. 9.** The fluorescence decay curves of 500 nm emission of $Eu^{2+}$. $SrCaSiO_4$:0.005 $Eu^{2+}$ (a) and $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.01 $Ce^{3+}$ (b).

where $I_0$ and $I$ are the luminescence intensities at time 0 and $t$, respectively, and $\tau$ is the fluorescence lifetime. Based on the exponential Eq. (2) and fluorescence decay curves (Fig. 9), the fitting results of the fluorescence lifetime of $Eu^{2+}$ in $SrCaSiO_4$:0.005 $Eu^{2+}$ and $SrCaSiO_4$:0.005 $Eu^{2+}$, 0.01 $Ce^{3+}$ samples are 198 and 611 ns, respectively. According to Fig. 9, it can be seen clearly that after doping $Ce^{3+}$ content, the lifetime of $Eu^{2+}$ in $SrCaSiO_4$ phosphors is increased accordingly. This is a convincing proof that from $Ce^{3+}$ to $Eu^{2+}$ occurs effectively energy transfer.

## 4. Conclusions

In summary, the photoluminescence properties and the $Ce^{3+}$/$Eu^{2+}$ energy transfer of the SrCaSiO4:$Eu^{2+}$, $Ce^{3+}$ phosphors were investigated. The SrCaSiO4:$Eu^{2+}$, $Ce^{3+}$ phosphors show a green emission band with a maximum at 500 nm. Energy transfer from $Ce^{3+}$ to $Eu^{2+}$ is due to the superposition between the emission spectrum of $Ce^{3+}$ and the excitation spectrum of $Eu^{2+}$, which was confirmed by the evidences collected from the diffuse reflection, emission and excitation spectra, and photoluminescence decay curve. With $Ce^{3+}$ co-doping, the excitation of $Eu^{2+}$ in UV range is greatly enhanced and its corresponding emission intensity is increased by the reabsorption of the excitation energy from $Ce^{3+}$. The fluorescence lifetime of $Eu^{2+}$ is also greatly enhanced (from 198 to 611 ns), it provides the indirect evidence of the $Ce^{3+}$/$Eu^{2+}$ energy transfer. Therefore, the $SrCaSiO_4$:$Eu^{2+}$, $Ce^{3+}$ phosphor can act as a potential green light emitting phosphor for UV-LEDs.

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