Improving luminescent property of $SrIn_2O_4$:Eu^{3+} by co-doped A^+ (A = Li, Na, K) or Sm^{3+}*

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A series of $SrIn_2O_4$:Eu^{3+} phosphors are synthesized by a high temperature solid-state method, and their luminescent properties are investigated. They can be excited by 395-nm radiation, and produce red emission (619 nm); however, they have a low absorption of near-ultraviolet light with the wavelength of 400 nm–405 nm. When co-doped with A^+ (A = Li, Na, K), the emission intensity of $SrIn_2O_4$:Eu^{3+} is significantly enhanced, but its emission and excitation spectral profile is unchanged. With co-doping Sm^{3+}, not only is the emission intensity of $SrIn_2O_4$:Eu^{3+} enhanced, but also the absorption is broadened and strengthened in the range of 400 nm–405 nm. The effect of Sm^{3+}-doped content on the emission intensity of $SrIn_2O_4$:Eu^{3+}, Sm^{3+} is investigated, and the optimal Sm^{3+} content is 0.02 mol.

Keywords: luminescence, $SrIn_2O_4$:Eu^{3+}, A^+ (A = Li, Na, K), Sm^{3+}

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1. Introduction

In comparison with conventional illumination applications, the white light-emitting diode (w-LED) offers many advantages such as longer service lifetime, thermal resistance, and higher efficiency.[1–3] However, commercial white LEDs, which combine a blue LED chip with a yellow light emitting phosphor YAG:Ce, have a less satisfactory color rendering in the red region, leading to the fact that the white LED has a poor color rendering property.[4] Therefore, in order to achieve a new kind of white LED, other ways are suggested, for example, a white LED can be obtained by combining a tri-color (red, green, and blue) phosphor or a single-phase white emitting phosphor with an ultraviolet or near ultraviolet (n-UV) LED.[5–10] Therefore, more and more attention has been paid to the development of a new tri-color or white emitting phosphor that can be excited in the range of UV or n-UV light, due to the necessity of increasing the efficiency of white LEDs. Among the tri-color phosphors, some sulfide phosphors such as (Ca, Sr)S:Eu^{2+} and Y_2O_2S:Eu^{3+} are used as red phosphors for commercial white LEDs. However, their chemical stability and efficiency are not desirable. Therefore, it is necessary to seek the new red phosphors with high luminance and satisfactory chemical stability.

The trivalent europium ion, Eu^{3+}, is usually adopted as a red emitting center thanks to its intrinsic characteristic that Eu^{3+} ($4f^6$) ions can create red emission with a high luminescent efficiency and can be effectively excited by UV and blue light.[11–15] Therefore, in order to develop novel red emitting materials for serving as solid state lighting, Eu^{3+}-doped materials still attract much interest. Recently, Eu^{3+}-activated vanadate garnet, molybdate, tungstate scheelites, and Eu^{2+}-doped $M_2Si_5N_8$ (M = Ca, Sr, Ba) novel red emitting materials have been reported.[16–20] Eu^{3+}-doped MIn_2O_4 (M = Ca, Sr) can also create red emission, such as $SrIn_2O_4$:Eu^{3+}.[21] However, it is very important that its excitation lines via the characteristic f–f transition of Eu^{3+} in $SrIn_2O_4$ are located at n-UV. Therefore, the f–f transition of Eu^{3+} is too narrow to cover the whole wavelength range of the emission of the n-UV LED source, 395 nm–405 nm.[22] In addition, for MIn_2O_4 (M = Ca, Sr), Eu^{3+} may substitute for M^{2+} (M = Ca, Sr) or In^{3+}.[23] Generally, a kind of cation is replaced by another with different valence in the matrix, and substitution content is restricted because of charge imbalance. Otherwise, the replacement cation could not come into the matrix totally. It does not only result in the appearance of a new phase but also affects the luminescent property of materials. Therefore, charge compensation is needed on this condition.[24] If Eu^{3+} substitutes for In^{3+}, the radius of Eu^{3+} is 0.107 nm, which differs from that of In^{3+} (0.080 nm).[22,24] Thereby, some Eu^{3+} ions are assumed to substitute for Sr^{2+} (0.118 nm).[22,24] The different radii between Sr^{2+} and Eu^{3+} create a distortion in the crystal structure, which may lead to a negative influence on the luminescence of phosphor. Therefore, whether Eu^{3+} substitutes for M^{2+} (M = Ca, Sr) or In^{3+}, the charge compensation may be used. However, to our knowledge, there are few reports about the charge balance in the $SrIn_2O_4$:Eu^{3+} phosphor being used

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for a white LED.

In order to broaden absorption and improve luminescent property of $SrIn_2O_4$:Eu^{3+}, in this paper, $Sr_{1-x-x}In_2O_4$:xEu^{3+}, xA^+ (A = Li, Na, K), $Sr_{1-x-y}In_2O_4$:xEu^{3+}, and ySm^{3+} phosphors are synthesized by a high temperature solid-state method, and the effect of A^+ and Sm^{3+} on the luminescent property of $SrIn_2O_4$:Eu^{3+} is investigated.

2. Experiment

A series of $Sr_{1-x}In_2O_4$:xEu^{3+}, $Sr_{1-x-y}In_2O_4$:xEu^{3+}, ySm^{3+}, $Sr_{1-x-x}In_2O_4$:xEu^{3+}, and xA^+ (A = Li, Na, K) phosphors are synthesized by high temperature solid-state reactions in air. The raw materials $SrCO_3$, In_2O_3, Eu_2O_3, Sm_2O_3, Li_2CO_3, Na_2CO_3, and K_2CO_3 (all materials are of analytical grade) are weighed with an appropriate stoichiometric ratio, then blended and milled thoroughly in an agate mortar for 1 h. The mixed powders are put into an alumina crucible and calcined in a muffle furnace at 550 °C for 6 h, and then sintered at 1200 °C for 12 h. Finally, the samples are cooled down to room temperature and ground thoroughly again into powders. Moreover, the samples are sieved under the same conditions to ensure a similar particle size distribution.

The product is characterized by powder X-ray diffraction (XRD) (D/max-rA, CuKα, 40 kV, 100 mA, $\lambda = 0.15406$ nm). The excitation and emission spectra of the samples are recorded by using a Hitachi F-4600 fluorescence spectrophotometer equipped with a 450-W Xe lamp. The measurements are performed at room temperature.

3. Results and discussion

3.1. Crystal structure

The XRD patterns for all the samples are measured, and a similar diffraction pattern is observed for each sample. The unchanged diffraction pattern indicates that a small amount of Eu^{3+} ions has no influence on the crystal structure of $SrIn_2O_4$. As an example, figure 1 shows the XRD patterns of $0.02Sm^{3+}$, $0.1Eu^{3+}$, and $0.1Eu^{3+}$/$0.02\ Sm^{3+}$-doped $SrIn_2O_4$, respectively. The diffraction peak position of the sample is consistent with JCPDS (Joint Committee of Powder Diffraction Standard) card No. 33-1336 for $SrIn_2O_4$. This means that the crystalline structure of $SrIn_2O_4$ cannot be influenced by a small amount of impurities. $SrIn_2O_4$ has an orthorhombic crystal structure with a space group of *Pnam* (62), and the lattice parameters are $a = 0.9830$ nm, $b = 1.1496$ nm, and $c = 0.3269$ nm.

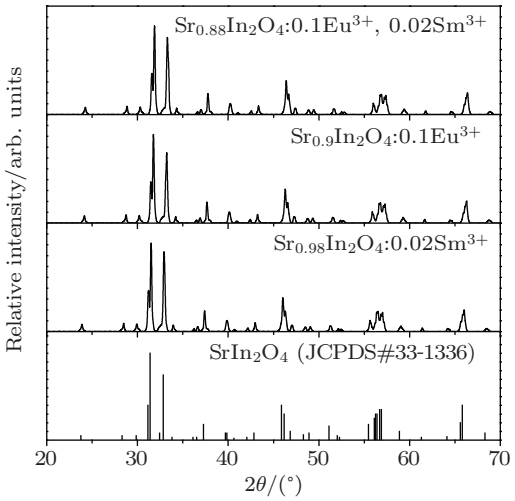


Fig. 1. XRD patterns of $Sr_{0.98}In_2O_4$:$0.02Sm^{3+}$, $Sr_{0.9}In_2O_4$:$0.1Eu^{3+}$, and $Sr_{0.88}In_2O_4$:$0.1Eu^{3+}$, $0.02Sm^{3+}$ with standard data of $SrIn_2O_4$ (JCPDS No. 33-1336).

3.2. Luminescent property of $SrIn_2O_4$:Eu^{3+}

The emission spectra of $SrIn_2O_4$:Eu^{3+} with different Eu^{3+}-doped content are measured upon 395-nm radiation excitation, and the excitation spectra of $SrIn_2O_4$:Eu^{3+} are measured by monitoring 619-nm emission. The results present the same spectral profile for all samples. As an example, figure 2 depicts the emission and excitation spectra of $SrIn_2O_4$:$0.1Eu^{3+}$. It shows many emission lines which correspond to the transition from the $^5D_{0,1,2}$ excited states to the ^7F_J ground states of Eu^{3+}, such as $^5D_2 \rightarrow {}^7F_3$ (515 nm), $^5D_1 \rightarrow {}^7F_1$ (540 nm), $^5D_1 \rightarrow {}^7F_2$ (559 nm), $^5D_0 \rightarrow {}^7F_1$ (590 nm), $^5D_0 \rightarrow {}^7F_2$ (619 nm), $^5D_0 \rightarrow {}^7F_3$ (656 nm), and $^5D_0 \rightarrow {}^7F_4$ (692 nm).[11] The strongest emission is located at 619 nm in respect that Eu^{3+} occupies the site without inversion symmetry. The excitation spectrum comprises a broad band in the range of 200 nm–300 nm which corresponds to the O^{2-}–Eu^{3+} charge transfer band, and two narrow lines, in the region of 300 nm–400 nm, are located at 341 nm and 395 nm, originating from the $^7F_0 \rightarrow {}^5D_4$ and $^7F_0 \rightarrow {}^5L_6$ transitions of Eu^{3+}, respectively.[11] However, $SrIn_2O_4$:Eu^{3+} has low absorption in the region of n-UV (400 nm–405 nm).

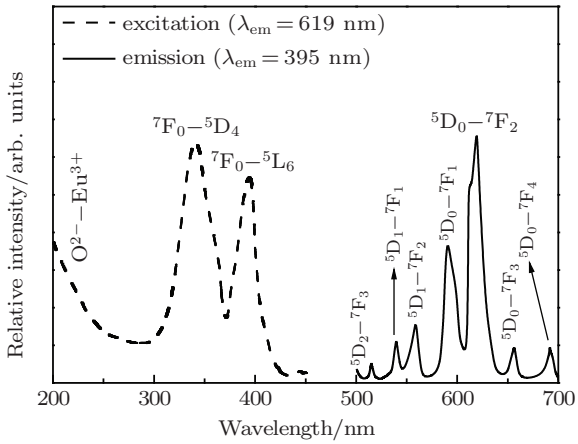


Fig. 2. Emission and excitation spectra of $SrIn_2O_4$:$0.1Eu^{3+}$ (λ_{ex} = 395 nm).

3.3. Effect of A^+ (A = Li, Na, K) on the luminescent property of $SrIn_2O_4:Eu^{3+}$

The radius of Eu^{3+} ($r_{Eu} = 0.107$ nm) is higher than that of In^{3+} ($r_{In} = 0.080$ nm) and is close to that of Sr^{2+} ($r_{Sr} = 0.118$ nm). For $SrIn_2O_4:Eu^{3+}$, if In^{3+} ion is replaced by Eu^{3+} ion, Sr^{2+} may be replaced by a part of Eu^{3+} ions due to the difference in the radii of Eu^{3+} and In^{3+}.[22,24] If an Eu^{3+} ion enters the host crystal lattice and preferentially substitutes for an Sr^{2+} ion, this may induce a charge imbalance and lattice distortion, then lead to point defects in the structure which would increase the non-radiative process that results in the reduction of luminescence intensity. Because the luminescent properties of phosphors arise from the complex interaction among host structure, activators, defects, and interfaces, charge compensation is needed. For $Sr_{1-x}In_2O_4:xEu^{3+}$, the incorporation of alkali metal ions A^+ (A = Li, Na, K) might offset the charge imbalance which is generated by Eu^{3+} substitution for Sr^{2+}, thus reducing the lattice distortion and enhancing the luminescent intensity.

Figure 3 presents the X-ray diffraction patterns of $Sr_{0.9}In_2O_4:0.1Eu^{3+}$ and $Sr_{0.8}In_2O_4:0.1Eu^{3+}$, $0.1A^+$ (A = Li, Na, K). For $SrIn_2O_4:Eu^{3+}$, A^+ (R = Li, Na, K), two Sr^{2+} ions are substituted by one Eu^{3+} ion and one A^+ ion, expressed as[23]

$$2Sr_{Sr}^{\times} \rightarrow Eu_{Sr}^{\bullet} + A_{Sr}'.$$

The results show that the peak positions and intensity of all samples agree well with the JCPDS standard card (No. 33-1336) of $SrIn_2O_4$ crystal. This means that co-doped A^+ does not cause significant change in the host lattice structure. However, when co-doped with A^+, the diffraction peak positions of the samples shift, which is attributable to the alkali metal ions substituting for Sr^{2+} ions in the host lattice.[25] The radii of K^+ ($r_K = 0.138$ nm) and Na^+ ($r_{Na} = 0.124$ nm) are larger than those of Sr^{2+}, which results in a smaller angle shift of the peaks in the XRD patterns. Also, the radius of Li^+ ($r_{Li} = 0.082$ nm) is smaller than that of Sr^{2+}, which leads to a larger angle shift of the diffraction peaks.[26]

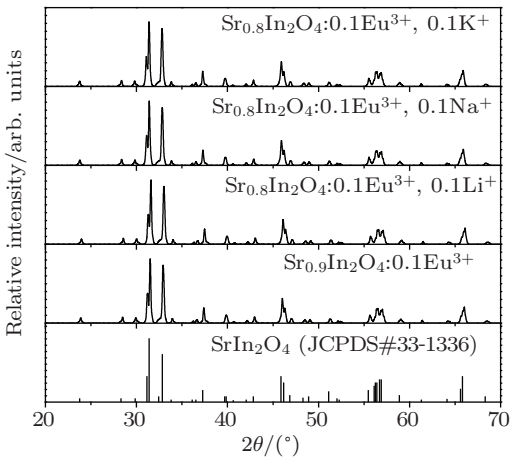


Fig. 3. XRD patterns of Eu^{3+}, A^+ (A = Li, Na, K) co-doped $SrIn_2O_4$, with standard data of $SrIn_2O_4$ (JCPDS No. 33-1336).

$Sr_{0.9}In_2O_4:0.1Eu^{3+}$ and $Sr_{0.8}In_2O_4:0.1Eu^{3+}$, $0.1A^+$ phosphors are excited by 395-nm radiation excitation and show the same spectral profile. As an example, figure 4 shows the emission spectra of $Sr_{0.8}In_2O_4:0.1Eu^{3+}$, $0.1A^+$. Its luminescent intensity is stronger than that of $Sr_{0.9}In_2O_4:0.1Eu^{3+}$ phosphor; in other words, the luminescent intensity of $Sr_{0.9}In_2O_4:0.1Eu^{3+}$ phosphor can be effectively enhanced by the charge compensation A^+, and co-doped Na^+ presents the optimal effect. The radius of Na^+ ion is close to that of Sr^{2+} ions. When $2Sr^{2+}$ is replaced by one Eu^{3+} ion and one Na^+ ion, the change in crystal structure and lattice parameters of $Sr_{0.8}In_2O_4:0.1Eu^{3+}$, $0.1Na^+$ would be very small. In contrast, with co-doped Li^+ or K^+ ions, larger lattice distortion would be induced in the host lattice. Therefore, Na^+ ion might be the optimal charge compensation for $Sr_{0.9}In_2O_4:0.1Eu^{3+}$ phosphor.

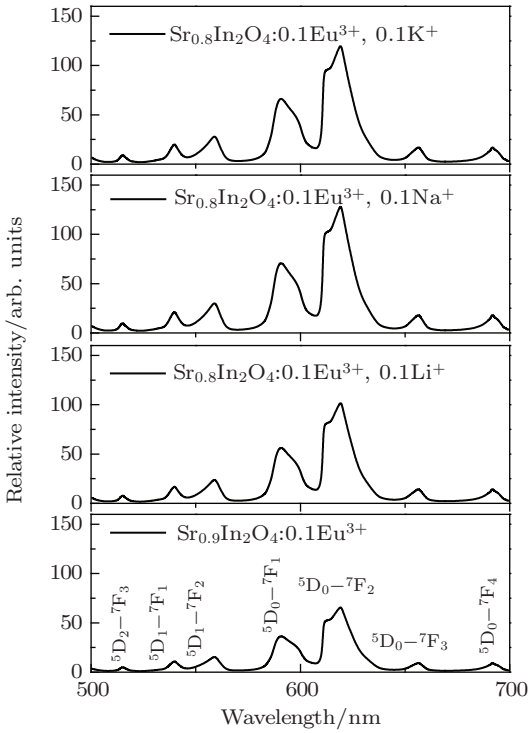


Fig. 4. The emission spectra of $Sr_{0.8}In_2O_4:0.1Eu^{3+}$, $0.1A^+$ (A = Li, Na, K) and $Sr_{0.9}In_2O_4:0.1Eu^{3+}$ ($\lambda_{ex} = 395$ nm).

The dependence of the red emission intensity is located at 619 nm on the Eu^{3+} concentration (x) in $Sr_{1-x}In_2O_4:xEu^{3+}$ and $Sr_{1-x-x}In_2O_4:xEu^{3+}$, xA^+ (A = Li, Na, K) is shown in Fig. 5. All three types of alkali metal ions enhance the luminescence of the phosphors, and the highest emission intensity can be obtained by co-doping Na^+ ion. Moreover, the enhancement of the emission intensity is very limited with a low concentration of Eu^{3+}, which can be assigned to the fact that less charge imbalance forms during the substitution of Sr^{2+} by Eu^{3+} when the concentration of Eu^{3+} is low, and the effect of the charge compensation is limited.[24]

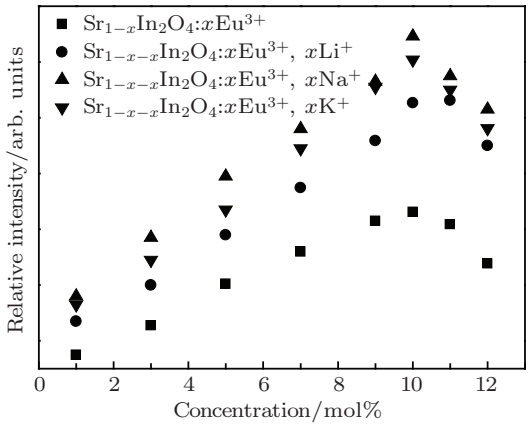


Fig. 5. The emission intensity of $Sr_{1-x-x}In_2O_4:xEu^{3+}$, xA^+ (A = Li, Na, K) and $Sr_{1-x}In_2O_4:xEu^{3+}$ as a function of the charge compensation content (λ_{ex} = 395 nm).

3.4. Effect of Sm^{3+} on the luminescent property of $SrIn_2O_4:Eu^{3+}$

The ionic radii of Eu^{3+} and Sm^{3+} (0.108 nm) are similar to that of Sr^{2+}, but considerably larger than that of In^{3+}. If only Eu^{3+} or Sm^{3+} is introduced to Sr_2InO_4, and substitutes for Sr^{2+}, the charge balance must be considered. However, in co-doped Eu^{3+}/Sm^{3+}, the charge loss in the lattice Sr_2InO_4 is probably compensated by Sr^{2+} vacancies (V_{Sr}) and the mode can be described as[25]

$$Eu_2O_3 \rightarrow 2Eu^{\bullet}_{Sr} + 3O^{\times}_{O} + V''_{Sr},$$
$$Sm_2O_3 \rightarrow 2Sm^{\bullet}_{Sr} + 3O^{\times}_{O} + V''_{Sr}.$$

Under the different radiation excitation (λ_{ex} = 395, 400, and 405 nm), $Sr_{0.98}In_2O_4:0.02Sm^{3+}$ and $Sr_{0.9}In_2O_4:0.1Eu^{3+}$ present the characteristic transition emission of Sm^{3+} and Eu^{3+}, respectively, but their luminescent intensities are different with the different radiation excitation. In order to compare the $Sr_{0.98}In_2O_4:0.02Sm^{3+}$ to $Sr_{0.9}In_2O_4:0.1Eu^{3+}$, the same spectral region of two phosphors is adopted (for emission, 500 nm–700 nm; for excitation, 200 nm–450 nm).

Figure 6(a) shows that $Sr_{0.9}In_2O_4:0.1Eu^{3+}$ has several emission peaks in the region of 500 nm–700 nm which originate from the typical $^5D_{0,1,2} \rightarrow ^7F_J$ transition of Eu^{3+}. It can be excited by 341 nm and 395 nm, but has a low absorption in the region of 400 nm–405 nm. Figure 6(b) shows that $Sr_{0.98}In_2O_4:0.02Sm^{3+}$ has three orange–red emission bands, which are attributed to transitions from $^4G_{5/2}$ excited state to $^6H_{J'/2(J'=5,7,9)}$ ground states of Sm^{3+}. The strongest emission is located at 607 nm, which corresponds to the $^4G_{5/2} \rightarrow ^6H_{7/2}$ typical transition of Sm^{3+}. The other emission peaks are at 561 nm and 650 nm and are attributed to the $^4G_{5/2} \rightarrow ^6H_{5/2}$ and $^4G_{5/2} \rightarrow ^6H_{9/2}$ transitions of Sm^{3+}, respectively. Its excitation spectra comprise a broad band in the range of 200 nm–300 nm corresponding to the O^{2-}–Sm^{3+} charge transfer band, and some narrow lines located in the region of 300 nm–450 nm, 322 nm, and 410 nm which can be ascribed to the $^6H_{5/2} \rightarrow ^4K_{17/2}$ and $^6H_{5/2} \rightarrow ^4K_{11/2}$ transitions of Sm^{3+}, respectively.[27] However, it cannot be effectively excited by the 395-nm–400-nm n-UV light.

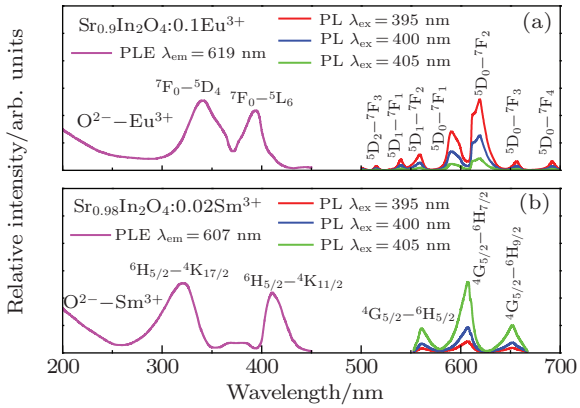


Fig. 6. (color online) Emission and excitation spectra of $Sr_{0.9}In_2O_4:0.1Eu^{3+}$ (a) and $Sr_{0.98}In_2O_4:0.02Sm^{3+}$ (b).

$SrIn_2O_4:Eu^{3+}$ has low absorption in the region of 400 nm–405 nm. However, as shown in Fig. 7, for Eu^{3+}/Sm^{3+} co-doped $SrIn_2O_4$, the characteristic transition emission of Eu^{3+} can be significantly observed under three different levels of radiation excitation. For 405-nm radiation, which is the strongest excitation of the $^6H_{5/2} \rightarrow ^4K_{11/2}$ transition of Sm^{3+}, the $^4G_{5/2} \rightarrow ^6H_{7/2}$ (607 nm) characteristic emission of Sm^{3+} can also be found.[28] However, $SrIn_2O_4:0.1Eu^{3+}$, $0.02Sm^{3+}$ presents nearly the same emission band as $SrIn_2O_4:0.1Eu^{3+}$ under the 395-nm and 400-nm radiation excitations, and does not have the characteristic emission of Sm^{3+}. This means some of the excited Sm^{3+} ions act as sensitizers to transfer energy to Eu^{3+} ions, and Eu^{3+} ions are sensitized to show the characteristic emission of Eu^{3+}.[29,30] As a result, the dominant emission in the spectrum is the emission of the $^5D_0 \rightarrow ^7F_2$ transition of Eu^{3+}. Monitored at 619-nm emission, the excitation band is strengthened and broadened in the region of 400 nm–405 nm. In other words, the excitation spectrum of $SrIn_2O_4:Eu^{3+}$ is adjusted by adding a small amount of Sm^{3+}. A new excitation band (400 nm–405 nm) can be observed in the excitation spectrum of $SrIn_2O_4:Eu^{3+}$, Sm^{3+}, which provides an additional excitation possibility in the region of 400 nm–405 nm. This is the same as the results with Sm^{3+}/Eu^{3+} co-doped TiO_2, and Sm^{3+} acts as a bridge to transfer energy from the host to Eu^{3+} ions.[31] The excitation band of Eu^{3+} ions is found to be dominant in the excitation spectrum of $SrIn_2O_4:Eu^{3+}$, Sm^{3+}. This means the energy transfer from Sm^{3+} to Eu^{3+} occurs in the lattice. More detailed information on the energy transfer mechanism between the different ions can be found in Refs. [31]–[34].

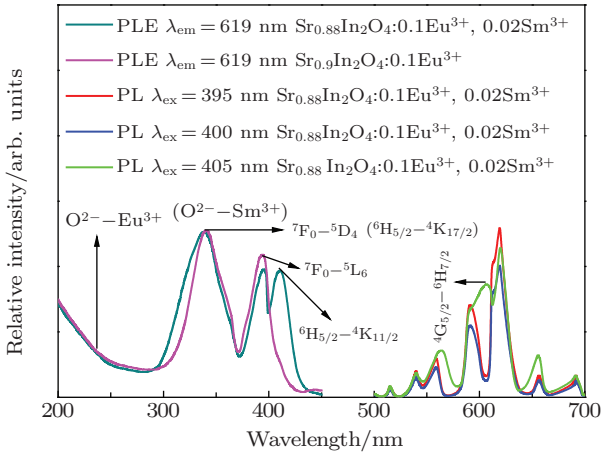


Fig. 7. (color online) Emission and excitation spectra of $Sr_{0.88}In_2O_4$:0.1Eu^{3+}, 0.02Sm^{3+}.

Figure 8 shows the dependence of the Eu^{3+} emission intensity (for 619 nm) of $Sr_{0.9-y}In_2O_4$:0.1Eu^{3+}, ySm^{3+} on the molar concentration of Sm^{3+} (y = 0–0.03), and Eu^{3+} molar concentration is 0.1. It shows that the emission intensity first increases with the increase of Sm^{3+} doping content, reaches a maximum at 0.02 mol Sm^{3+}, and then decreases. For Eu^{3+}/Sm^{3+} co-doping $SrIn_2O_4$, the Eu^{3+} emission intensity can be enhanced and the absorption band can be strengthened and broadened. This means that the energy transfer between Eu^{3+} and Sm^{3+} occurs, and leads to an obvious increase of Eu^{3+} emission intensity. However, co-doped Sm^{3+} may reduce the distance of the activators in the host lattice, which can encourage the non-radiative transition, and result in a slight decrease of Eu^{3+} emission intensity. Moreover, the probability that the optical excitation gets trapped in defects or impurity sites increases, which strengthens the process of non-radiative relaxation during the energy migration.[35,36] So there is an optimal Sm^{3+}-doped content: 0.02 mol.

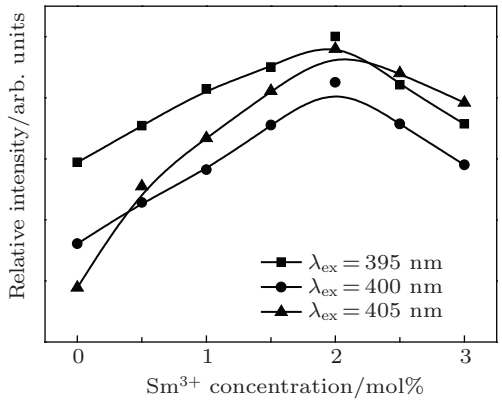


Fig. 8. Emission intensity of $Sr_{0.9-y}In_2O_4$:0.1Eu^{3+}, ySm^{3+} (y=0, 0.005, 0.01, 0.015, 0.02, 0.025, 0.3) (λ_{ex} = 395, 400, and 405 nm).

4. Conclusions

$SrIn_2O_4$:Eu^{3+}, $SrIn_2O_4$:Eu^{3+}, Sm^{3+}, and $SrIn_2O_4$:Eu^{3+}, A^+ (A = Li, Na, K) are synthesized by a high temperature solid-state reaction. The emission intensity of $SrIn_2O_4$:Eu^{3+} can be significantly enhanced by the charge compensation A^+ (A = Li, Na, K), and Na^+ is demonstrated to be the best one because its ion radius is similar to that of Sr^{2+}. The luminescent properties of Eu^{3+} in $SrIn_2O_4$ can be adjusted by co-doping Sm^{3+}. Its emission intensity can be enhanced and is maximized at 0.02-mol Sm^{3+}, and its absorption of n-UV can be extended (in the region of 400 nm–405 nm) and strengthened. These results indicate that $SrIn_2O_4$:Eu^{3+}, Sm^{3+} may be a promising red emitting phosphor for n-UV-based white LEDs.

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