

Research Article

Concentration Dependence of Luminescent Properties for $\text{Sr}_2\text{TiO}_4:\text{Eu}^{3+}$ Red Phosphor and Its Charge Compensation

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$\text{Sr}_2\text{TiO}_4:\text{Eu}^{3+}$ phosphors using M^+ ($\text{M} = \text{Li}^+$, Na^+ , and K^+) as charge compensators were prepared by the solid-state reaction. The powders were investigated by powder X-ray diffraction (XRD) and photoluminescence spectra (PL) to study the phase composition, structure, and luminescent properties. The results showed that Li^+ ion was the best charge compensator. The phase was Sr_2TiO_4 when the doping concentration was small ($x \leq 10.0\%$). When x reached 15.0%, the phase turned into $\text{Sr}_3\text{Ti}_3\text{O}_7$ because of the structure damage. The phosphor could be effectively excited by ultraviolet (365, 395 nm) and blue light (465 nm), and then it emitted intense red light that peaked at around 620 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_2$). In addition, the emission of 700 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_4$) enhanced the red light color purity. The CIE chromaticity coordinates of samples with the higher red emission were between (0.650, 0.344) and (0.635, 0.352). Doped layered titanate $\text{Sr}_2\text{TiO}_4:\text{Eu}^{3+}$ is a promising candidate red phosphor for white LEDs which can be suited for both near-UV LED chip and blue LED chip.

1. Introduction

White light-emitting diodes (LEDs) are considered to be next-generation lighting devices. They have many advantages such as energy saving, environment friendliness, and small size [1–3]. Phosphor conversion method is a principal method in all the technologies to achieve white light owing to its easy achievement, high efficiency, and low cost.

The combination of blue chip and yellow phosphors has already been developed and is commercially available, but the lack of red emitting makes the color rendering index (CRI) lower [4]. The other tricolor white LEDs consisting of red, green, and blue (RGB) phosphors excited with a UV chip emitting 400 nm also have challenges. The luminescence intensity of commercial red phosphors is much weaker than green and blue phosphors. For excellent color render index, both methods need efficient red phosphors that should have the excitation wavelength matching with the emission of the blue LEDs ($\lambda_{\text{em}} = 440\text{--}470\text{ nm}$) or the UV LEDs ($\lambda_{\text{em}} = 350\text{--}410\text{ nm}$). Therefore, the development of a red phosphor with high luminance and satisfactory chemical

stability is a key technology for achieving warm white LEDs.

As a result, a kind of red phosphor with perovskite structure attracts much attention [5–7]. Perovskite structure $\text{MTiO}_3:\text{Pr}^{3+}$ ($\text{M} = \text{Ca}$, Sr , and Ba) phosphors emitted intense red light at 610 nm when excited by UV-light [8, 9]. Its intensity was greatly enhanced by adding charge compensation agents such as Al^{3+} , Li^+ , and Na^+ [10–12]. The optimized excitation wavelength of $\text{CaTiO}_3:\text{Eu}^{3+}$ was 400 nm, suitable for near-ultraviolet (N-UV) LED chip and emitted red light at 618 nm [13, 14]. However, the intensity is not so high that more-efficient red phosphors are needed to achieve an acceptable efficiency for white LEDs. Layered perovskite compounds have longer distance between layers so that they have bigger doping concentration. Therefore, layered perovskite compounds are good host materials for phosphors. Sr_2TiO_4 is a typical layered perovskite compound. According to these, trivalent Europium ion-activated Sr_2TiO_4 phosphor is prepared by solid-state reaction, and its luminescence properties are investigated to see whether it has the potential to be a red phosphor for N-UV or blue light LED chip.

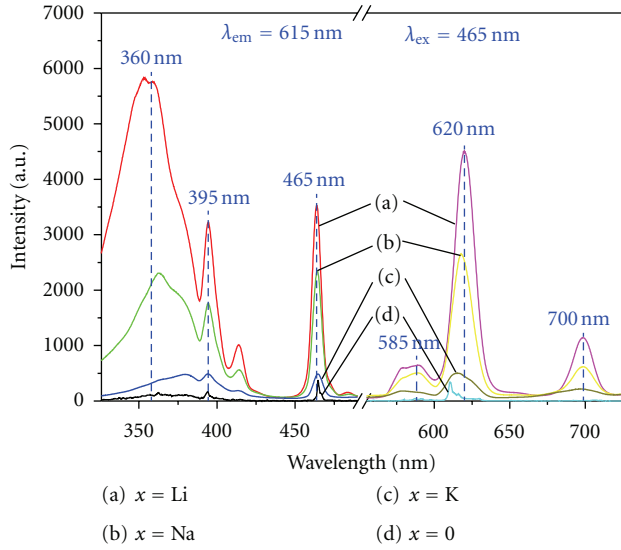


FIGURE 1: PL ($\lambda_{ex} = 465$ nm) and PLE ($\lambda_{em} = 615$ nm) spectra of $(\text{Sr}_{0.8}\text{Eu}_{0.1}\text{M}_{0.1})_2\text{TiO}_4$ phosphor with different charge compensation agents: (a) $\text{M} = \text{Li}$ (b) $\text{M} = \text{Na}$, (c) $\text{M} = \text{K}$, and (d) without agents.

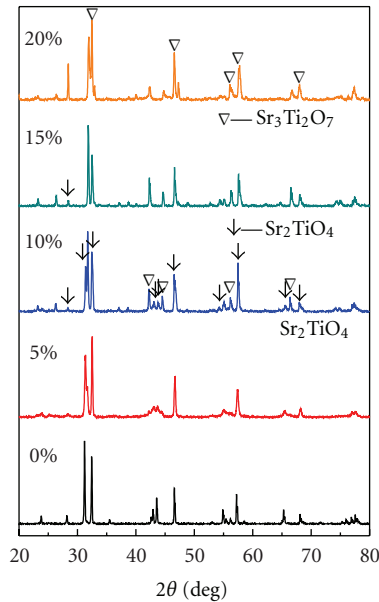


FIGURE 2: XRD patterns of $(\text{Sr}_{1-2x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ powders (x (mol%) = 0.0, 5.0, 10.0, 15.0, 20.0).

2. Experimental

2.1. Sample Preparation. All powder samples were synthesized through the solid-state reaction technique. High-purity SrCO_3 , TiO_2 (analytical grade) and Eu_2O_3 (>99.99%) were mixed thoroughly in alcohol by ball milling and then dried. Appropriate amounts of Li_2CO_3 , Na_2CO_3 , or K_2CO_3 (analytical grade) were added as the charge compensators. The synthesis was performed at 1100°C for 2 h under air atmosphere in electric tube furnace. Series of

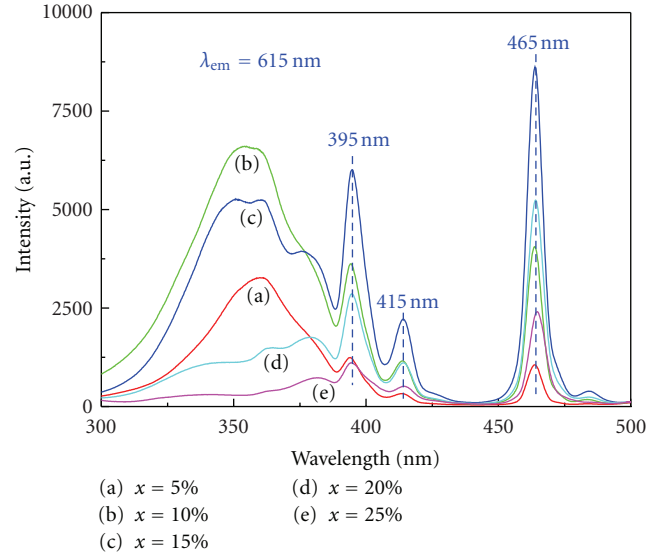


FIGURE 3: (a) Excitation spectra ($\lambda_{em} = 615$ nm) of $(\text{Sr}_{1-2x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$: (a) $x = 5.0\%$, (b) $x = 10.0\%$, (c) $x = 15.0\%$ (d) $x = 20.0\%$, (e) $x = 25.0\%$, and (b) Gaussian peak fitting of the $(\text{Sr}_{0.7}\text{Eu}_{0.15}\text{Li}_{0.15})_2\text{TiO}_4$ phosphor powder.

$(\text{Sr}_{1-2x}\text{Eu}_x\text{M}_x)_2\text{TiO}_4$ ($\text{M} = \text{Li}^+$, Na^+ , K^+ ; x (mol%) = 5.0, 10.0, 15.0, 20.0, 25.0) powders were prepared. SrCO_3 and Eu_2O_3 come from Sinopharm Chemical Reagent Co., Ltd., and Li_2CO_3 , Na_2CO_3 , K_2CO_3 and TiO_2 come from Shanghai Lingfeng Chemical Reagent CO., LTD.

2.2. Characterization. The crystalline phases of synthesized powders were determined by X-ray diffraction (XRD, D/Max2500, Rigaku, Japan) using $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) in the range of $5^\circ - 80^\circ$ with a step size of 0.02° . The photoluminescence spectra of the phosphors were measured using a fluorescent spectrophotometer (FL3-221, HORIBA, Jobin Yvon, France) at room temperature.

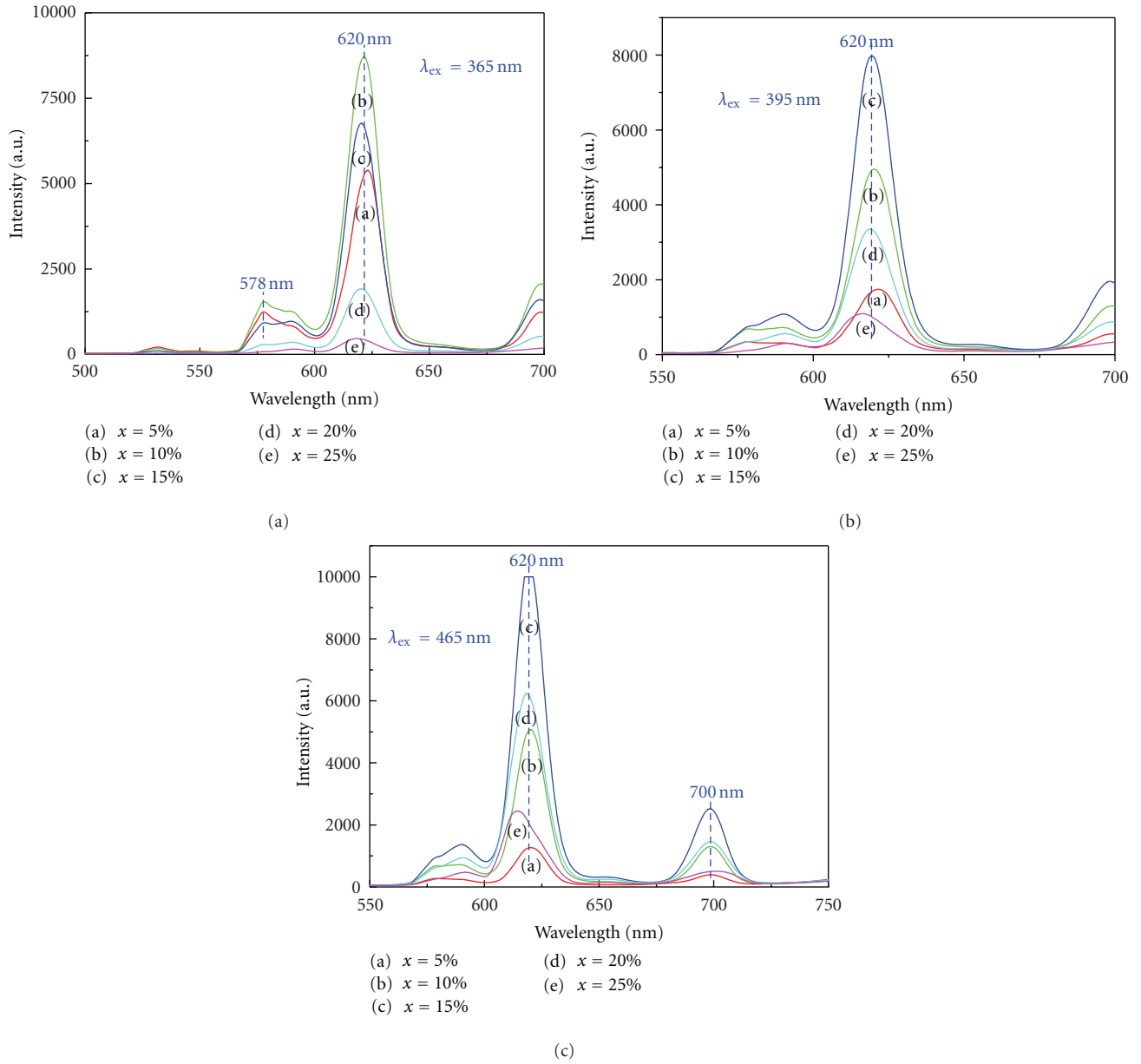


FIGURE 4: Emission spectra (a) $\lambda_{\text{ex}} = 365 \text{ nm}$, (b) $\lambda_{\text{ex}} = 395 \text{ nm}$, and (c) $\lambda_{\text{ex}} = 465 \text{ nm}$ of $(\text{Sr}_{1-x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ (a) $x = 5.0\%$, (b) $x = 10.0\%$, (c) $x = 15.0\%$, (d) $x = 20.0\%$, (e) $x = 25.0\%$.

3. Results and Discussion

3.1. The Choice of Charge Compensators. Eu^{3+} ions are expected to replace Sr^{2+} ions. It would be difficult to keep charge balance in the lattice. Therefore, Eu^{3+} ions may not be fully introduced into Sr^{2+} sites in order to keep charge balance. Eu^{3+} may exist in Eu_2O_3 state, and it would lead to the decrease of emission intensity. This problem can be solved by adding charge compensators. Li^+ , Na^+ , and K^+ ions are always chosen as charge compensators for phosphors [15, 16]. Because the radii of Li^+ , Na^+ , and K^+ are small which are easy to enter into lattice, and they are all of +1 valence which is convenient for charge compensation. Therefore, Li^+ , Na^+ , and K^+ were added in the same molar

weight as Eu^{3+} to act as the charge compensators to improve the luminescence intensity of $\text{Sr}_2\text{TiO}_4:\text{Eu}^{3+}$.

Figure 1 shows the excitation and emission spectra of $(\text{Sr}_{0.8}\text{Eu}_{0.1}\text{M}_{0.1})_2\text{TiO}_4$ phosphor with different charge compensators. The shape and positions were similar in the PL and PLE spectra for all the samples. Excitation and emission intensities were enhanced obviously after adding charge compensators. Before adding charge compensators, Eu^{3+} could not substitute Sr^{2+} sites totally in order to maintain chemically neutral $\text{Sr}_2\text{TiO}_4:\text{Eu}^{3+}$ phosphor. Some Eu^{3+} could not act as activated ions, and the impurity phase could interdict the energy transfer between matrix and activated ions and restrain the Sr_2TiO_4 grains growth during the

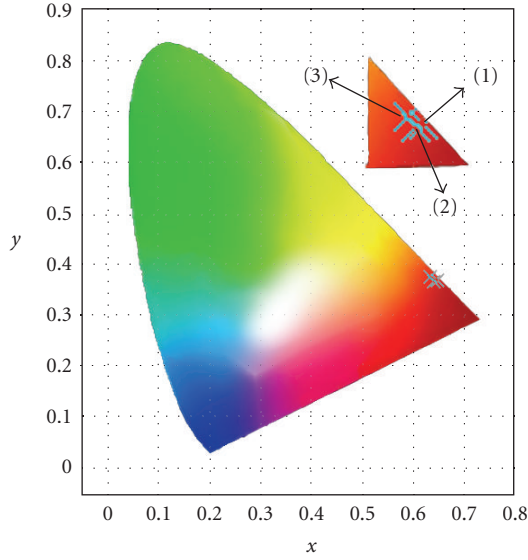


FIGURE 5: The CIE chromaticity coordinates of red phosphors: (1) $(\text{Sr}_{0.8}\text{Eu}_{0.1}\text{Li}_{0.1})_2\text{TiO}_4$ ($\lambda_{\text{ex}} = 365$ nm), (2) $(\text{Sr}_{0.7}\text{Eu}_{0.15}\text{Li}_{0.15})_2\text{TiO}_4$ ($\lambda_{\text{ex}} = 395$ nm), (3) $(\text{Sr}_{0.7}\text{Eu}_{0.15}\text{Li}_{0.15})_2\text{TiO}_4$ ($\lambda_{\text{ex}} = 465$ nm).

sintering process [17]. After adding charge compensators, Eu^{3+} ions were able to be fully introduced into Sr^{2+} sites, and charge compensators and vacancy could substitute Sr^{2+} sites so that SrO and Eu_2O_3 could react with TiO_2 to a great extent. In addition, Li_2CO_3 , Na_2CO_3 , and K_2CO_3 can act as flux agents to promote the formation of luminescence materials polycrystal. Compared with Na^+ and K^+ , the intensity was enhanced greatly when adding Li^+ as charge compensator. These phenomena are assigned to the fact that the ionic radius of Li^+ (0.92 Å) to the one of Sr^{2+} (1.31 Å) is the smallest, the one of Na^+ (1.24 Å) being second, and the one of K^+ (1.55 Å) is the biggest so that it develops the distortion grade different, which impacts on the luminous properties and crystal structures [18]. Thus, Li^+ is selected as charge compensator in the following experiments.

3.2. XRD of $(\text{Sr}_{1-2x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ (x (mol%) = 0.0, 5.0, 10.0, 15.0, 20.0) Powders. Figure 2 shows the XRD patterns of Eu^{3+} ions doped Sr_2TiO_4 phosphors with different Li^+ concentration as charge compensator. At first, when the doping concentration was small ($x = 5.0\%$), all the peaks were indexed by Sr_2TiO_4 without any impurity. After x reached 10.0%, there appeared some $\text{Sr}_3\text{Ti}_2\text{O}_7$ peaks, but Sr_2TiO_4 was the major phase. When $x = 15.0\%$, $\text{Sr}_3\text{Ti}_2\text{O}_7$ turned to be the major phase, because the structure of Sr_2TiO_4 was damaged with the increase of Eu^{3+} and Li^+ . Until x came to 20.0%, the phase composition consisted of $\text{Sr}_3\text{Ti}_2\text{O}_7$ and some impurities, because the content of Sr^{2+} was too little and the radius of Li^+ was too small to support the whole structure. After doping Eu^{3+} , peaks shifted to higher 2θ values relatively, which indicated that Eu^{3+} had occupied the sites of Sr^{2+} , because the radius of Eu^{3+} is 1.12 Å (CN = 9), smaller than that of Sr^{2+} (1.31 Å, CN = 9). When

Eu^{3+} occupied the site of Sr^{2+} , the lattice would shrink so that the diffraction peaks would shift to high diffraction angles compared to pure Sr_2TiO_4 and $\text{Sr}_3\text{Ti}_2\text{O}_7$ [19].

3.3. Luminescent Properties of $(\text{Sr}_{1-x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ (x (mol%) = 5.0, 10.0, 15.0, 20.0) Phosphors. Figure 3(a) presents the excitation spectra of $(\text{Sr}_{1-x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ under different Eu^{3+} concentrations ($\lambda_{\text{em}} = 615$ nm). The excitation spectra consisted of a wide excitation band from 325 to 425 nm and some sharp line peaks of characteristic transitions of Eu^{3+} . The broad band was a charge transfer band (CTB) which was caused by several charge transition. Figure 3(b) shows the results of Gaussian peak fitting of the PLE spectra of $(\text{Sr}_{0.7}\text{Eu}_{0.15}\text{Li}_{0.15})_2\text{TiO}_4$. Peak 1 (350 nm) was ascribed to the $\text{O} \rightarrow \text{Ti}$ of phase $\text{Sr}_3\text{Ti}_2\text{O}_7$, peak 2 (380 nm) was ascribed to the $\text{O} \rightarrow \text{Ti}$ of phase Sr_2TiO_4 , and peak 3 (362 nm) was ascribed to the $\text{O} \rightarrow \text{Eu}$. The sum of deconvoluted curves (red dash line) was almost fitted with observed line (black solid line). The other intense 395, 465 and weak 415 nm excitation peaks related the intra-4f transitions from ground state $^7\text{F}_0$ to $^5\text{L}_6$, $^5\text{D}_2$, and $^5\text{D}_3$, respectively. The shape and position were similar except that the intensity of absorption varied with the increase of Eu^{3+} concentration. What interests us is that the intensity of CTB is stronger than that of intra-4f transitions at lower Eu^{3+} concentration ($x \leq 10.0\%$), and it mainly performs wide band absorption of matrix. Then the intensity of CTB decreases with the increase of Eu^{3+} doping concentration. However, characteristic sharp line spectra increase continuously until $x = 15.0\%$, and it performs 395 and 465 nm intense linear excitation peaks. With the increase of Eu^{3+} and Li^+ , the phase composition changes from Sr_2TiO_4 to $\text{Sr}_3\text{Ti}_2\text{O}_7$ to some impurities, and impurity is bad for luminescence intensity. However, both intensity of CTB and intra-4f transitions are high when Eu^{3+} doped in layered titanate, and there is reason to believe that $\text{Sr}_2\text{TiO}_4:\text{Eu}^{3+}$ and even $\text{Sr}_3\text{Ti}_2\text{O}_7:\text{Eu}^{3+}$ are promising red phosphors for white LEDs.

Figures 4(a), 4(b), and 4(c) are the emission spectra of $(\text{Sr}_{1-x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ excited by 365, 395, and 465 nm, respectively. The shape and position of the emission spectra were similar, and they all emitted dominated intense 620 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_2$) red light and weak 578 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_1$), 700 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_4$) light. $^5\text{D}_0 \rightarrow ^7\text{F}_2$ and $^5\text{D}_0 \rightarrow ^7\text{F}_4$ are electric dipole transitions of Eu^{3+} , which are very sensitive to the local environment around the Eu^{3+} , and depend on the symmetry of the crystal field. As we know, the transitions are forbidden when Eu^{3+} occupies centrosymmetric sites. The structure of Sr_2TiO_4 is tetragonal K_2NiF_4 type, and perovskite layers are interleaved with SrO layers. In Sr_2TiO_4 , all Sr ions occupy positions between the perovskite layers (9-oxygen-ion-coordinated sites with C_{4v} symmetry) [20]. Therefore, it indicates that Eu^{3+} has occupied the noncentrosymmetrical sites of Sr^{2+} . Compared with usual luminescence of Eu^{3+} (594, 615 nm) [21], the luminescence of Eu^{3+} in layered perovskite has some red shift which performs better red light color purity and higher luminescent intensity. In addition, the emission of 700 nm in some degree enhances red light color purity, too. Those are

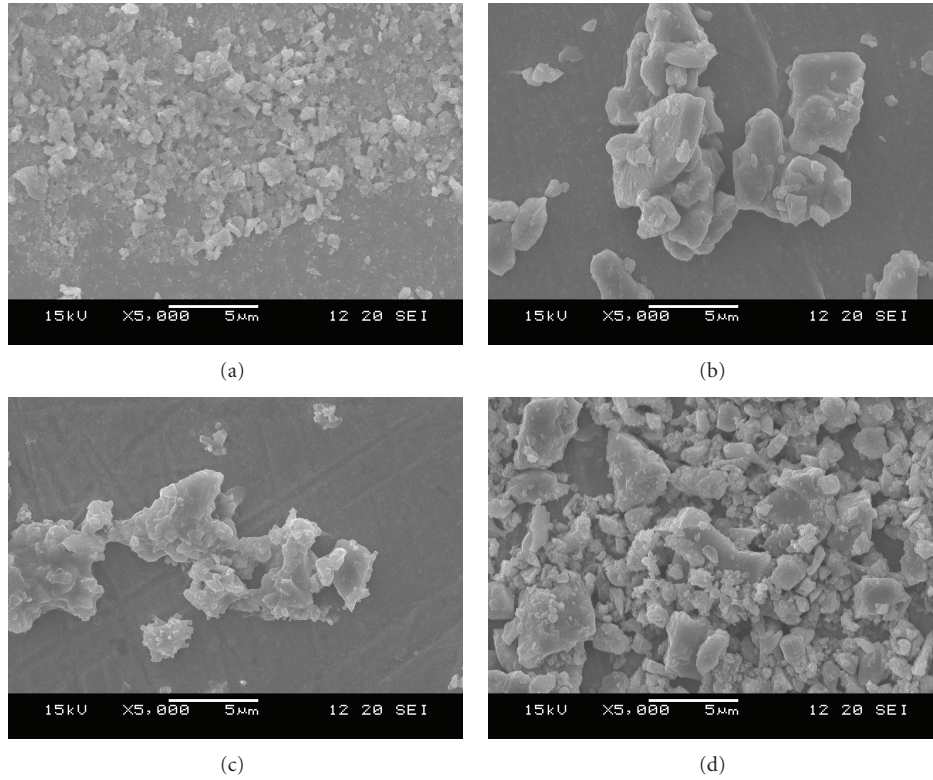


FIGURE 6: SEM micrographs of $(\text{Sr}_{1-x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ phosphor powders: (a) $x = 5.0\%$, (b) $x = 10.0\%$, (c) $x = 15.0\%$, and (d) $x = 20.0\%$.

more beneficial to make it become a red light compensation material for white LEDs.

The emission intensity was found to increase with the increase of Eu^{3+} concentration up to 10.0 mol% ($\lambda_{\text{ex}} = 365 \text{ nm}$). While excited at 395 and 465 nm, the best doping concentration was 15.0 mol%. Because, when $x = 10.0\%$, the phase is Sr_2TiO_4 and TiO_4^{4-} group has strong absorption at UV light region [22], it has the strongest luminescence intensity when excited at 365 nm. However, when $x = 15.0\%$, the phase is $\text{Sr}_3\text{Ti}_2\text{O}_7$. The crystal system of $\text{Sr}_3\text{Ti}_2\text{O}_7$ is tetragonal, and double perovskite layers are interleaved with SrO layers. In $\text{Sr}_3\text{Ti}_2\text{O}_7$, some Sr ions occupy positions between the perovskite layer (9-oxygen-ion-coordinated sites with C_{4v} symmetry), and the other Sr ions occupy positions in the center of the perovskite layer (12-oxygen-ion-coordinated sites with D_{4h} symmetry) [20]. In spite of the 12-oxygen-ion-coordinated sites in $\text{Sr}_3\text{Ti}_2\text{O}_7$, 9-oxygen-ion-coordinated sites are vast majority; therefore, it emits characteristic intense red light of Eu^{3+} which is sensitive to the surrounding symmetry of the crystal field. Usually, Eu^{3+} doping concentration is less than 5.0% in normal phosphor. However, the doping concentration of Eu^{3+} is up to or even over 10.0% in Sr_2TiO_4 and $\text{Sr}_3\text{Ti}_2\text{O}_7$ host. The main reason is that layered perovskite has SrO layers interleaved in perovskite layers, and it makes bigger space between layers so that they have bigger doping concentration which leads to the higher luminescence intensity. In conclusion, layered titanate is a good matrix for phosphors, and Eu^{3+} doped

layered titanate is a good red phosphor for white LEDs which can be suited for both near-UV LED chip and blue LED chip.

The samples that are emitting most intense red light respectively excited at 365, 395, and 465 nm are chosen to calculate their CIE chromaticity coordinates. The corresponding CIE chromaticity coordinates using symbol “ x ” to indicate their positions are shown in Figure 5, and they change between (0.650, 0.344) and (0.635, 0.352), which is due to the variability of the relative intensities of 620 nm and 700 nm mainly. They are all close to coordinates of the “ideal red” which is (0.67, 0.33) [23]. In addition, their CIE chromaticity coordinates are rather close the edge of CIE diagram, indicating that this kind of phosphors shows better color purity in solid-state lighting.

3.4. SEM of $(\text{Sr}_{1-x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ (x (mol%) = 5.0, 10.0, 15.0, 20.0) Phosphor Powders. It is well known that crystallinity and surface morphology of phosphors has a strong effect on the luminescence properties. Figure 6 shows the SEM morphology of $(\text{Sr}_{1-x}\text{Eu}_x\text{Li}_x)_2\text{TiO}_4$ with different Eu^{3+} and Li^+ concentration, and all presented irregular polygon. The particles enlarged with the increase of Eu^{3+} concentration. The particle size was about $1 \mu\text{m}$ when $x = 5.0\%$ and kept around $5 \mu\text{m}$ when x reached 10.0%, 15.0%, and 20.0% which corresponded with the requirements of particle size to phosphor. It has been established that Li^+ was conducive to the formation of crystalline phase, and it is mainly because of the function of Li_2CO_3 flux [24]. However, the special doping

concentration of active Eu^{3+} also has some influence on the particle size [25].

4. Conclusions

In this paper, $\text{Sr}_2\text{TiO}_4:\text{Eu}^{3+}$, M^+ ($\text{M} = \text{Li}^+$, Na^+ , and K^+) phosphors were synthesized through the solid-state reaction. M^+ as charge compensators led to the increase of emission intensity, and Li^+ was confirmed to be the best charge compensator. The excitation spectra consisted of a wide excitation band and some sharp line peaks of characteristic transitions of Eu^{3+} when Eu^{3+} doping concentration is low ($x \leq 10.0\%$), the phase is Sr_2TiO_4 , and the intensity excited by CTB (365 nm) is stronger than that of intra-4f transitions. After x reached 15.0%, the structure damaged and the phase became $\text{Sr}_3\text{Ti}_2\text{O}_7$. The intensity excited by CTB became weaker and characteristic emission peaks of Eu^{3+} stronger when excited at 395 and 465 nm. Because Sr_2TiO_4 has strong absorption at UV light region, while $\text{Sr}_3\text{Ti}_2\text{O}_7$ has two layers and there are more noncentersymmetrical Sr^{2+} sites for substituting. Those phosphors all exhibited intense 620 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_2$) red light and weak 578 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_1$), 700 nm ($^5\text{D}_0 \rightarrow ^7\text{F}_4$) light. The CIE chromaticity coordinates of three samples that are emitting most intense red light change between (0.650, 0.344) and (0.635, 0.352), close to coordinates of the “ideal red” which is (0.67, 0.33). The particle size meets the demands of phosphor. Doped layered titanate $\text{Sr}_2\text{TiO}_4:\text{Eu}^{3+}$ is a promising candidate red phosphor for white LEDs which can be suited for both near-UV LED chip and blue LED chip.

Acknowledgments

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