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Synthesis, Luminescent Properties and White LED Fabrication of Sm$^{3+}$ Doped Lu$_2$WMoO$_9$

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Abstract: In this paper, Sm$^{3+}$ doped Lu$_2$W$_{0.5}$Mo$_{0.5}$O$_9$, Lu$_2$WMoO$_9$, and Lu$_2$(W$_{0.5}$Mo$_{0.5}$O$_4$)$_3$ materials were synthesized by using a two-step solid-state reaction method. The synthesized materials were characterized by X-ray diffraction (XRD) patterns, field emission scanning electronic micrograph (FE-SEM) pictures, photoluminescence (PL) excitation and emission spectra, and temperature-dependent emission intensities. Orange-reddish light could be observed from the phosphors under ultraviolet (UV) 365 nm light. The Sm$^{3+}$ doped Lu$_2$WMoO$_9$ had enhanced PL intensities compared to the other two materials. The excitation, the energy transfer, the nonradiative relaxation, and the emission processes were illustrated by using schematic diagrams of Sm$^{3+}$ in Lu$_2$MoO$_9$. The optimal Sm$^{3+}$ doping concentration was explored in the enhancing luminescence of Lu$_2$WMoO$_9$. By combing the Sm$^{3+}$ doped Lu$_2$WMoO$_9$ to UV 365 nm chips, near white lighting emitting diode (W-LED) were obtained. The phosphor can be used in single phosphor-based UV W-LEDs.

Keywords: luminescent materials; photoluminescence; ultraviolet (UV); white lighting emitting diode (W-LED)

1. Introduction

As a new generation light source, phosphor-based converted white-light emitting diodes (W-LEDs) have been researched and used due to their advantages in energy saving, high luminous efficiency, reliability, and environmental friendliness, etc. [1,2]. The commonly used commercial phosphor based W-LEDs are usually based on the combination of blue LED chips with yellow Y$_3$Al$_5$O$_{12}$:Ce$^{3+}$ (YAG:Ce$^{3+}$) phosphors [3]. Due to the scarcity of red emissions, the W-LEDs have high correlated color temperature and other problems, such as low color-rendering index, low thermal quenching temperature, chromatic stabilities, narrow visible range, etc. [4,5]. Correspondingly, the combination of ultraviolet (UV) LED chips combined with tri-color RGB (red, green, blue) phosphors or blue LED chips combined with green and red phosphors have been proposed [6,7]. Therefore, the phosphors which have broad absorption band in the UV and/or blue wavelength band and red phosphors have been widely researched [8,9].

For the intense and broad charge transfer band (CTB) absorption in the UV wavelength band and excellent physical and chemical stability, molybdate and tungstate host phosphors have been widely studied [10,11]. The most widely investigated rare-earth ion is the Eu$^{3+}$ ion due to its characteristic red emissions from the $^5{\text{D}}_0$$\rightarrow$$^7{\text{F}}_2$ transition [12] or Eu$^{2+}$ [13]. As an alternative choice of Eu$^{3+}$, new red emitters need to be explored, too. Sm$^{3+}$ is a good choice as an orange-red activator ion in luminescent materials [14]. The Sm$^{3+}$ doped luminescent materials can be used in solid-state lighting devices [15,16]. The Sm$^{3+}$ characteristic emissions are $^4{\text{G}}_{5/2}$$\rightarrow$$^6{\text{H}}_{5/2}$ transitions. The yellow $^4{\text{G}}_{5/2}$$\rightarrow$$^6{\text{H}}_{5/2}$ transition is a magnetic dipole transition (MD), which is insensitive to the local environment.
The $^4G_{5/2} \rightarrow ^6H_{7/2}$ transition is an orange emission, which is a combined MD and electric-dipole (ED) transition. While the red emission $^4G_{5/2} \rightarrow ^6H_{9/2}$, is an ED transition, which is greatly influenced by the local symmetry of the Sm$^{3+}$ ion [16]. The ED/MD transition intensity ratio is usually used to judge the local environment around the Sm$^{3+}$; the ED transition is stronger, the asymmetry property is greater, and vice versa.

For the broad UV band absorption of tungstates and molybdates, the energy transfer process can take place in the RE-doped tungstates and molybdates considering the energy transfer theory [17,18]. Sm$^{3+}$, Eu$^{3+}$, Dy$^{3+}$, Ho$^{3+}$, etc., doped Y$_2$WO$_6$ and Lu$_2$WO$_6$ have been well reported and researched [15,17,19,20]. Unlike usually used YAG:Ce$^{3+}$, Lu$_3$Al$_5$O$_{12}$:Ce$^{3+}$/CaAlSiN$_3$:Eu$^{2+}$ phosphors [21,22], the CTB of WO$_6^{6-}$ locates at about 300 nm and cannot be directly used in UV LED devices. As a choice, Mo$^{6+}$ can be added to shift the excitation band to a longer wavelength of tungstates as well as enhancing the luminescence of Eu$^{3+}$ [23–25]. Based on the above consideration, some Sm$^{3+}$ doped lutetium tungsten molybdenum oxides Lu$_2$W$_{0.5}$Mo$_{0.5}$O$_6$, Lu$_2$(Mo$_2$O$_9$, and Lu$_2$(W$_{0.5}$Mo$_{0.5}$O$_{13}$ were synthesized. Enhancing luminescence of Sm$^{3+}$ and red-shifted excitation band compared Lu$_2$W$_{0.5}$Mo$_{0.5}$O$_6$ and Lu$_2$(W$_{0.5}$Mo$_{0.5}$O$_{13}$ were obtained in Lu$_2$WMoO$_9$ materials. The excitation, the energy transfer, and the emission processes in Lu$_2$WMoO$_9$ were elucidated, and the optimal doping concentration of Sm$^{3+}$ in Lu$_2$WMoO$_9$ was studied. W-LEDs were obtained by combining the Sm$^{3+}$ doped Lu$_2$WMoO$_9$ to 365 nm UV chips. The experimental results suggest that the enhancing Sm$^{3+}$ doped Lu$_2$WMoO$_9$ phosphors can be used in single-phosphor-based W-LEDs.

2. Materials and Methods

By using a two-step solid-state reaction method reported in reference [26], Sm$^{3+}$ doped Lu$_2$W$_{0.5}$Mo$_{0.5}$O$_6$, Lu$_2$WMoO$_9$, and Lu$_2$(W$_{0.5}$Mo$_{0.5}$O$_{13}$ were synthesized. Lutetium oxide (Lu$_2$O$_3$, 99.99%, Shanghai, China), Tungsten oxide (WO$_3$, 99.99%, Shanghai, China), Molybdenum trioxide (MoO$_3$, 99.95%, Shanghai, China), and Samarium oxide (Sm$_2$O$_3$, 99.99%, Shanghai, China) were used as raw materials. Stochiometric of mixed raw materials were pre-heated at 600 °C for 2 h then calcined at 1200 °C for 4 h, and the Sm$^{3+}$ doped phosphors were obtained. For comparison, the optical properties of Sm$^{3+}$ doped Lu$_2$W$_{0.5}$Mo$_{0.5}$O$_6$, Lu$_2$WMoO$_9$, and Lu$_2$(W$_{0.5}$Mo$_{0.5}$O$_{13}$, the Sm$^{3+}$ concentrations were kept Lu$_2$O$_3$:Sm$_2$O$_3$ = 98.2. To explore the optimal Sm$^{3+}$ doping concentration in Lu$_2$WMoO$_9$ phosphors, the Sm$^{3+}$ concentration (corresponding Lu$_2$O$_3$ 0.5, 1, 2, 3, 5, and 7 mol% were selected to synthesis the phosphors.

The obtained materials were characterized by X-ray diffraction (XRD, Rigaku, Tokyo, Japan) patterns, field emission scanning electron microscope (FE-SEM, Zeiss, Jena, Germany) images, and room temperature photoluminescence (PL, Thermo scientific, Waltham, USA) excitation and emission spectra. The data were recorded by the same apparatuses in reference [26]. The LED lamps were fabricated by coating the 3 mol.% Sm$^{3+}$ doped Lu$_2$WMoO$_9$ phosphors onto the 365 nm LED chips. The fabrication procedure and the measurements are similar to those used in [26].

3. Results and Discussion

3.1. Crystalline and Morphology

Figure 1 shows XRD patterns of materials synthesized at 1200 °C for 4 h with the raw materials Lu$_2$O$_3$(Sm$_2$O$_3$):WO$_3$:MoO$_3 = 1:0.5:0.5, 1:1:1, 1:1.5:1.5, and kept Lu$_2$O$_3$:Sm$_2$O$_3$ = 98.2. The raw materials synthesized with Lu$_2$O$_3$(Sm$_2$O$_3$):WO$_3$:MoO$_3 = 1:0.5:0.5$ can be referred to the reference data of monoclinic phase of Lu$_2$WO$_6$ with JCPDS no. 23-1211. As 50% percent of W$^{6+}$ was replaced by Mo$^{6+}$, the diffraction peaks became weaker, as marked by an asterisk in Figure 1. Similar phenomena have been observed [26]. With the raw materials Lu$_2$O$_3$(Sm$_2$O$_3$):WO$_3$:MoO$_3 = 1:1:1$, the XRD pattern could be referred to the reference data of Lu$_2$Mo$_2$O$_9$ with JCPDS No. 28-0613. The diffraction peak was marked as an inverted triangle which became stronger as 50% percent of Mo$^{6+}$ was replaced by W$^{6+}$. For the raw materials Lu$_2$O$_3$(Sm$_2$O$_3$):WO$_3$:MoO$_3 = 1:1.5:1.5$, the obtained XRD pattern
could be referred to the orthorhombic phase of Y$_2$W$_3$O$_{12}$ with the JCPDS No. 15-0447\cite{27}. Using different molar ratio of raw materials, the obtained phosphor crystalized in different crystalline phases, which will affect the luminescence greatly.

**Figure 1.** X-ray diffraction (XRD) patterns of phosphors synthesized at 1200 °C for 4 h and reference data.

Low magnification (scale bars are all 1 mm) and high magnification (scale bars are all 200 nm) FE-SEM images of 1 mol.% Sm$^{3+}$ doped Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$ (a, b), Lu$_2$MoWO$_9$ (c, d), and Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$ (e, f) are shown in Figure 2, respectively. The Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$ phosphor was composed of 300–500 nm size particles with smooth surfaces. The Lu$_2$MoWO$_9$ phosphor was composed of 1–2 mm microparticles, and some nanoparticles appeared on the surface of the microparticles. The Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$ phosphor was composed of the biggest microparticles with a size of ~3–5 mm. Similarly, some nanoparticles emerged, too. The FE-SEM pictures suggest that with an increasing molar ratio of Lu$_2$O$_5$(Sm$_2$O$_3$):WO$_2$:MoO$_3$, the size of the particles increased.

**Figure 2.** Field emission scanning electronic micrograph (FE-SEM) pictures of Sm$^{3+}$ doped Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$ (a,b), Lu$_2$MoWO$_9$ (c,d), and Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$ (e,f).
3.2. PL Spectra

Figure 3 presents the room temperature PL spectra of 2 mol.% Sm\(^{3+}\) doped Lu\(_2\)MoWO\(_9\) phosphor. By monitoring the Sm\(^{3+}\) 614 nm emission from \(^4\)G\(_{5/2}\)→\(^6\)H\(_{7/2}\), the PL excitation spectrum is shown in the left part in Figure 3. The spectrum was composed of two components, which include a broad charge transfer band (CTB) and several sharp Sm\(^{3+}\) 4f–4f peaks. The CTB was located in the wavelength range of 200–450 nm, with the strongest peak at about 368 nm, which was consistent with UV 365 nm chips. The CTB had two peaks located at 305 nm and 368 nm, which were ascribed to O\(^2−\)-W\(^6+\) and O\(^2−\)-Mo\(^6+\) CTBs, respectively. The sharp peaks located at 407 nm and 469 nm originated from the \(^6\)H\(_{5/2}\) ground state to the \(^4\)F\(_{7/2}\), \(^4\)I\(_{13/2}\) excited states, respectively [28]. With the CTB 368 nm excitation, the PL emission spectrum is illustrated in the right part of Figure 3. There were four emission peaks at 567, 603 (614), 650 (660), and 712 nm, which could be ascribed to the Sm\(^{3+}\)\(^4\)G\(_{5/2}\) to \(^6\)H\(_{5/2}\), \(^7/2\), \(^9/2\), \(^11/2\) transitions, respectively. The orange-red emission of \(^4\)G\(_{5/2}\)→\(^6\)H\(_{7/2}\) was the strongest one. The ED transition \(^4\)G\(_{5/2}\)→\(^6\)H\(_{9/2}\) was stronger than the MD transition \(^5\)G\(_{7/2}\)→\(^6\)H\(_{9/2}\), which indicated the asymmetrical nature of Sm\(^{3+}\) in the Lu\(_2\)MoWO\(_9\) lattice. The broad and strong excitation band locates in the UV wavelength range, which means that the phosphors could be efficiently excited by the UV chips and can be used in UV-based LEDs.

![Figure 3](image-url)

**Figure 3.** Photoluminescence (PL) excitation (left part) and emission (right part) spectra of 2 mol.% Sm\(^{3+}\) doped Lu\(_2\)MoWO\(_9\).

To illustrate the excitation, the energy transfer (ET), the nonradiative relaxation (NR), and the emission processes in the Sm\(^{3+}\) doped Lu\(_2\)MoWO\(_9\) phosphor were schematically plotted (Figure 4). The Lu\(_2\)MoWO\(_9\) host lattice absorbs the UV light in the 250–420 nm wavelength range due to the O\(^2−\)-W\(^6+\), O\(^2−\)-Mo\(^6+\), and O\(^2−\)-Sm\(^{3+}\) CTBs [29–31]. The absorption energy was transferred to the higher excited states of Sm\(^{3+}\). Then the NR processes occurred. The energy was relaxed to the excited state of \(^4\)G\(_{5/2}\), and the radiative electron transitions from the \(^4\)G\(_{5/2}\) state to \(^6\)H\(_{5/2}\), \(^7/2\), \(^9/2\), \(^11/2\) states occurred, giving the orange-red emissions. In addition, the \(^6\)H\(_{5/2}\) ground state of Sm\(^{3+}\) could also absorb the excitation energy to the higher excited states. After the NR processes to the excited state of \(^4\)G\(_{5/2}\), orange-red emissions could be recorded for the \(^4\)G\(_{5/2}\) to \(^6\)H\(_{5/2}\), \(^7/2\), \(^9/2\), \(^11/2\) transitions.
Figure 4. Schematic diagrams of Sm$^{3+}$ charge transfer band (CTB) in Lu$_2$MoWO$_9$.

The PL excitation (left part) and emission (right part) spectra of the three samples of Sm$^{3+}$ doped Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$, Lu$_2$MoWO$_9$, and Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$ are illustrated in Figure 5. The Lu$_2$MoWO$_9$ sample presented the strongest excitation and emission intensities. The CTB band was located at about 368 nm, and the strongest emission was located at about 614 nm. Compared to those of Lu$_2$MoWO$_9$, the Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$ illustrated weaker PL excitation and emission intensities, the excitation and emission intensities of Lu$_2$MoWO$_9$ were about 4.5 times of those of Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$. The Sm$^{3+}$ doped Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$ presented the weakest CTB intensity and the absorption peak located at about 288 nm. On the one hand, in the Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$ and Lu$_2$MoWO$_9$ phosphors, the CTB absorption intensities were much stronger than those of the 4f–4f excitation from Sm$^{3+}$. On the other hand, the situation was the opposite. In the Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$ material, the intensity of the PL excitation from Sm$^{3+}$ 4f–4f was stronger than that of CTB. With 405 nm excitation, the Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$ presented the strongest emission at about 600 nm and the different splits of those of Lu$_2$MoWO$_9$ and Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$, which illustrated that the Sm$^{3+}$ was located at different crystalline sites.

Figure 5. PL excitation (left part) and emission (right part) spectra of Sm$^{3+}$ doped Lu$_2$Mo$_{0.5}$W$_{0.5}$O$_6$, Lu$_2$MoWO$_9$, and Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$.

For the Sm$^{3+}$ doped Lu$_2$MoWO$_9$ presents the strongest PL intensities, the PL excitation and emission spectra of Sm$^{3+}$ doping concentration of Lu$_2$(1−x)Sm$_x$MoWO$_9$ were explored. Figure 6 shows the excitation spectra of Lu$_2$(1−x)Sm$_x$MoWO$_9$ by monitoring at Sm$^{3+}$ 614 nm emission. With the increase in Sm$^{3+}$ concentration, the intensities of the CTB firstly increased, achieved the maximum with the x = 0.03, and then decreased when the x values increased further. For clarity, the x value-dependent CTB intensities are shown in the inset (Figure 6). The results suggest that the optimal x value was 0.03 for CTB excitation.
The corresponding emission spectra of Lu$_{2(1-x)}$Sm$_{2x}$MoWO$_9$ are shown in Figure 7. With UV 368 nm excitation, the characteristic emissions of Sm$^{3+}$ were recorded. With the x values increasing, the PL emission intensity increased, reached the maximum with x equals 0.03, and decreased when the x value increased further. For the CTB excitation, the Sm$^{3+}$ optimal doping was 0.03.

3.3. W-LED Fabrication and Characterization

For the CTB locates at UV wavelength range, and orange-red emission can be observed under UV 365 nm light, the Sm$^{3+}$ doped Lu$_2$MoWO$_9$ phosphor was fabricated to UV LED by combining 365 nm chips. Figure 8 shows a representative PL emission spectrum of packaged LED with a 50 mA current pumping. The spectrum suggests that the Sm$^{3+}$ doped Lu$_2$MoWO$_9$ phosphor could be efficiently excited by 365 nm light from the chip and gave orange-redish light. Combined with the chip light, the fabricated LED gave near-white light, which is shown in the inset of Figure 8a. The corresponding recorded Commission International de l’Eclairage (CIE) diagram is presented in the insets as Figure 8b, and the chromaticity coordinates were 0.453 and 0.346, which deviated slightly from the white standard point, 0.333 and 0.333. The chromaticity coordinates were located in the white area and near to red area in the diagram. The measured color temperature was 2260 K, and the color purity was 0.397. The PL emission spectrum of the phosphor obtained from LED was
consistent with the spectrum recorded under the fluorescence spectrophotometer. In addition, the temperature-dependent emission intensities were measured and added as an inset (Figure 8c). With the temperature increasing from 50 °C to 250 °C, the emission intensity decreased continuously. At 150 °C, the emission intensity maintained 67% intensity of 50 °C. The data and results suggest that the obtained Sm$^{3+}$ doped Lu$_2$MoWO$_9$ phosphor has potential applications as a single phosphor in UV chip-based phosphor-converted W-LEDs.

Figure 8. The emission spectrum of a packaged LED with 3 mol.% Sm$^{3+}$ (x = 0.03) doped Lu$_2$MoWO$_9$ phosphor, insets show the lighted LED (a), corresponding chromaticity coordinates in CIE diagram (b), and temperature-dependent emission spectra of phosphor (c).

4. Conclusions

Through a solid-state reaction method, Sm$^{3+}$ doped Lu$_2$MoO$_{6.5}$W$_{0.5}$O$_6$, Lu$_2$MoWO$_9$, and Lu$_2$(Mo$_{0.5}$W$_{0.5}$O$_4$)$_3$ phosphors were obtained. The Sm$^{3+}$ doped Lu$_2$MoWO$_9$ illustrated the strongest PL intensities in the three samples. The host CTB absorption located at about 370 nm was much stronger than the 4f–4f excitations of Sm$^{3+}$ in the Lu$_2$MoO$_{6.5}$W$_{0.5}$O$_6$ and Lu$_2$MoWO$_9$. The ET process played an important role in the Sm$^{3+}$ emissions. The optimal doping concentration of Sm$^{3+}$ was 0.03 for CTB excitation in Lu$_2$(1−x)Sm$_x$MoWO$_9$ phosphors. The temperature-dependent PL emission intensity suggested that the intensity decreased continuously with the temperature increasing. At 150 °C, the intensity was maintained at 67% of that at 50 °C. By combining 365 nm chips with Sm$^{3+}$ doped Lu$_2$MoWO$_9$ phosphors, W-LEDs could be obtained. The results suggest that the obtained phosphors could be used in single phosphor-based UV W-LEDs.

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