Energy transfer and luminescent properties in Tb$^{3+}$ and Eu$^{3+}$ co-doped CaMoO$_4$/SrMoO$_4$ thin films

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Abstract

A series of Eu$^{3+}$ and Tb$^{3+}$ co-doped CaMoO$_4$/SrMoO$_4$ luminescent thin films were prepared by a facile solution method, and they were annealed at 550 °C for 2 h. The luminescent properties of the thin films were studied, which involve the energy transfer from Tb$^{3+}$ to Eu$^{3+}$. The emission color can be changed from green to red, with increasing Eu$^{3+}$ doping concentration in Tb$^{3+}$-doped CaMoO$_4$/SrMoO$_4$ thin films. In addition, it was observed that the PL intensity of Eu$^{3+}$ will enhance when Tb$^{3+}$ ions are incorporated into Eu$^{3+}$-doped CaMoO$_4$/SrMoO$_4$ thin films. The optical band gaps of the luminescent thin films are found to be in the range of 4.49 to 4.72 eV. These results revealed that Eu$^{3+}$ and Tb$^{3+}$ co-doped CaMoO$_4$/SrMoO$_4$ luminescent thin films have a significantly potential application in electroluminescent devices.

1. Introduction

In the recent years, rare earth ions-doped molybdates have been widely studied due to their excellent chemical, optical, and thermal properties. Rare earth ions doped luminescent materials have abundant emission spectra, because of their 4f orbits with rich energy levels [1–8]. Eu$^{3+}$ and Tb$^{3+}$ are red and green light activator ions, which have been well investigated in many luminescent materials [9–14]. The red and green luminescence of Eu$^{3+}$ and Tb$^{3+}$ ions are owing to the $^5D_0 \rightarrow ^7F_J$ transitions of Eu$^{3+}$ and Tb$^{3+}$ transitions of Tb$^{3+}$. In addition, the energy can be transferred from Tb$^{3+}$ ions to Eu$^{3+}$ ions in Tb$^{3+}$ and Eu$^{3+}$ co-doped CaMoO$_4$ phosphors, which can lead to the emission color change from green to red and the enhancement of luminescence intensity of Eu$^{3+}$ ions [14–25]. CaMoO$_4$ and SrMoO$_4$ crystals are two good host materials for Eu$^{3+}$ and Tb$^{3+}$ ions, owing to their scheelite structure and excellent chemical properties.

The optical band gap of Eu$^{3+}$ and Tb$^{3+}$ co-doped CaMoO$_4$/SrMoO$_4$ luminescent materials are significantly narrower than that of rare earth doped oxides, which means they may be used as the emitting layer in electroluminescent thin film devices. The luminescent thin films have a high potential application in plasma display panel [26], field emission display [27, 28], cathode ray tubes [29]. Therefore, developing an efficient and facile solution approach to deposit highly luminescent rare earth doped CaMoO$_4$/SrMoO$_4$ thin films is desirable. According to the previous reports, numerous methods were adopted to synthesize rare earth doped CaMoO$_4$/SrMoO$_4$ phosphors, such as solid-state reaction [30, 31], sol-gel method [32] and co-precipitation [33], but only few papers were focused on rare earth ions-doped CaMoO$_4$/SrMoO$_4$ thin films. In this work, we developed a facile solution method to fabricate Eu$^{3+}$ and Tb$^{3+}$ co-doped CaMoO$_4$/SrMoO$_4$ luminescent thin films. Their luminescent properties and energy transfer between Tb$^{3+}$ and Eu$^{3+}$ were investigated.
2. Materials and experimental

Ca(NO$_3$)$_2$·4H$_2$O (99%), CH$_3$(CH$_2$)$_2$COOH (99%) and CH$_3$OH (A.R.) were purchased from Beijing Chemical Works. MoO$_3$ (99.9%), Sr(CH$_3$COO)$_2$ (99%), CH$_3$(CH$_2$)$_2$NH$_2$ (99%), Eu(CH$_3$COO)$_3$·3H$_2$O (99.99%), and Tb(CH$_3$COO)$_3$·6H$_2$O (99.99%) were bought from Aladdin Inc.

First, 0.1 mmol MoO$_3$, 0.01 mmol Eu(AC)$_3$, 0.09-x mmol Ca(NO$_3$)$_2$, x mmol Tb(AC)$_3$ (0 ≤ x ≤ 0.01), 2.0 ml of CH$_3$(CH$_2$)$_2$COOH, and 2.0 ml of CH$_3$(CH$_2$)$_2$NH$_2$ were loaded into a glass vial and the mixture was stirred at 140 °C for 1 h. Afterwards, the solution was diluted with 6.0 ml of methanol to obtain Tb$^{3+}$ doped Ca$_{0.9-x}$MoO$_3$: Eu$^{3+}$ Tb$^{3+}$ precursor solution. Next, the thin film was deposited on a quartz substrate by spin-coating the precursor solution at 2000 rpm for 15 s and was sintered at 250 °C for 1 min. Then, the thin film was further annealed at 550 °C.
for 2 h in a furnace. The same procedures were used to fabricate Ca$_{0.97-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ (0 ≤ x ≤ 0.1), Sr$_{0.85-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ (0 ≤ x ≤ 0.1), and Sr$_{0.9}$MoO$_4$: Tb$_{0.15}$: Eu$^{3+}$ thin films.

3. Results and discussion

3.1. Photoluminescence (PL) properties

According to the previous reports, the Eu$^{3+}$ and Tb$^{3+}$ doping concentrations meaningfully affect the PL intensity of Eu$^{3+}$, Tb$^{3+}$ co-doped CaMoO$_4$ phosphors [14–25]. Figure 1(a) shows the PL spectra excited at 254 nm of Ca$_{0.97-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin films with different Tb$^{3+}$ doping concentrations from 0 to 10 mol%. It should be noted that the thin films were annealed at same conditions. In emission spectra of Ca$_{0.97-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin films, the emission peaks are attributed to the transition of Eu$^{3+}$ ions from $^5D_0$ to $^7F_x$, $x = 0, 1, 2, 3, 4$ levels, which locate at 545, 594, 616, 656, and 704 nm, respectively [34, 35]. But it was observed that the PL intensity at 616 nm (Eu$^{3+}$: from $^3D_0$ to $^7F_2$) firstly increases and then decreases with the increase of Tb$^{3+}$ doping concentration, indicating the existence of the energy-transfer from Tb$^{3+}$ to Eu$^{3+}$. We found that the optimal composition (the highest PL intensity) is Ca$_{0.97}$MoO$_4$: Eu$_{0.07}$, Tb$_{0.03}$. Under the irradiation of UV254 light, the emission colors of the thin films can be changed from red to orange with the increase of Tb$^{3+}$ doping concentration, as shown in figure 1(b).

For Ca$_{0.97-x}$MoO$_4$: Tb$_{0.03}$, Eu$^{3+}$ thin films, the energy transfer from Tb$^{3+}$ to Eu$^{3+}$ is more obvious. Figure 1(c) shows a series of PL spectra for Ca$_{0.97-x}$MoO$_4$: Tb$_{0.03}$, Eu$^{3+}$ thin films with different Eu$^{3+}$ doping concentrations (0 ≤ x ≤ 0.1). The Eu$^{3+}$ doped Ca$_{0.97-x}$MoO$_4$: Tb$_{0.03}$, Eu$^{3+}$ thin films were all annealed at 550 °C for 2 h. The PL characteristic peaks of Tb$^{3+}$ ions at 492, 546, 586, and 621 nm can be clearly observed for Eu-free Ca$_{0.97}$MoO$_4$: Tb$_{0.03}$ thin film, which are owing to $^7D_4$ to $^7F_{J}$ (J = 6, 5, 4, 3) f–f transitions of Tb$^{3+}$ ions [36, 37]. However, the PL characteristic peaks of Eu$^{3+}$ ions at 616, 656, and 704 nm appear in PL spectra when Eu$^{3+}$ ions were doped into Ca$_{0.97-x}$MoO$_4$: Tb$_{0.03}$, Eu$^{3+}$ thin films. Meanwhile, it was found that the PL intensities of Tb$^{3+}$ ions dramatically decrease when a tiny amount of Eu$^{3+}$ ions were doped into Ca$_{0.97-x}$MoO$_4$: Tb$_{0.03}$, Eu$^{3+}$ thin films, indicating that the energy transfer from Tb$^{3+}$ to Eu$^{3+}$ occurs. In addition, it was revealed that the PL intensities of Eu$^{3+}$ ions firstly increase and then decrease, with increasing Eu$^{3+}$ doping concentrations from 0 to 10 mol%. We found that the Ca$_{0.96}$MoO$_4$: Tb$_{0.03}$, Eu$^{3+}$ thin film has the
strongest PL intensity. Their emission colors can be tuned from green to red by changing the Eu$^{3+}$ doping concentration in Ca$_{0.97-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin films, as shown in figure 1(d). Apart from Tb$^{3+}$ and Eu$^{3+}$ characteristic emission peaks, a broad emission peak at around 400 nm was observed, which can be ascribed to the oxygen vacancy-related intrinsic defects.

Apart from CaMoO$_4$ host, the luminescent properties and energy transfer were also investigated for Tb$^{3+}$ and Eu$^{3+}$ ions co-doped SrMoO$_4$ thin films. The PL spectra of Sr$_{0.85-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ (0 ≤ x ≤ 0.1) thin films are showed in figure 2(a), which were excited at 254 nm. The emission spectra of Tb$^{3+}$ doped Sr$_{0.85-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin films are similar to those of Tb$^{3+}$ doped Ca$_{0.9-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin films. The changing trends of PL intensity and color of Sr$_{0.85-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin films are the same with those of Ca$_{0.9}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin films with increasing the Tb$^{3+}$ concentration. For Sr$_{0.85-x}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin films, the strongest luminescence was achieved for Sr$_{0.76}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$ thin film, as shown in figures 2(a) and (b).

Additionally, the energy transfer from Tb$^{3+}$ and Eu$^{3+}$ ions was studied by changing the Eu doping concentration in Sr$_{0.85-x}$MoO$_4$: Tb$^{3+}$, Eu$^{3+}$ (0 ≤ x ≤ 0.1) thin films. As shown in figure 2(c), the PL peaks of Tb$^{3+}$ ions gradually decrease with increasing Eu$^{3+}$ doping concentration in Sr$_{0.85-x}$MoO$_4$: Tb$^{3+}$, Eu$^{3+}$ thin films; meanwhile the PL characteristic peaks of Eu$^{3+}$ ions increase, confirming that the energy transfer from Tb$^{3+}$ and Eu$^{3+}$ ions is evident. The emission colors of Sr$_{0.85-x}$MoO$_4$: Tb$^{3+}$, Eu$^{3+}$ thin films can be adjusted from green to red by changing the doping concentrations of Tb$^{3+}$ and Eu$^{3+}$ ions, as shown in figures 2(c) and (d). We found that the optimal chemical composition is Sr$_{0.78}$MoO$_4$: Tb$^{3+}$, Eu$^{3+}$.

### 3.2. UV–vis absorption properties

Figures 3(a)–(d) present the UV–vis absorption spectra of Ca$_{0.87}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$, Ca$_{0.9}$MoO$_4$: Tb$^{3+}$, Eu$^{3+}$, Sr$_{0.76}$MoO$_4$: Eu$^{3+}$, Tb$^{3+}$, and Sr$_{0.78}$MoO$_4$: Tb$^{3+}$, Eu$^{3+}$ thin films, respectively. The broad absorption band from 200 nm to 300 nm result from the charge transfer transitions of O-Mo in MoO$_4^2-$ groups. The strongest absorption peaks locate at around 230 nm for the four samples. In addition, the prominent absorption peaks should be due to the band-to-band transition rather than the transition of impurity levels.
The surface morphology of CaMoO$_4$: Tb$^{3+}$, Eu$^{3+}$ and SrMoO$_4$: Tb$^{3+}$, Eu$^{3+}$ thin films was observed by scanning electron microscope (SEM), as shown in figure 6(a). The thin film in figure 6(a) was fabricated by spin-coating precursor solution on a quartz substrate for once. The thickness of the thin film can be increased by repeating spin-coating/sintering process. After five spin-coating/sintering cycles, smooth and crack-free CaMoO$_4$: Tb$^{3+}$, Eu$^{3+}$ thin film with a thickness of 110 nm was fabricated, as shown in figures 6(b) and (c). In this work, all of the characterizations were conducted for the thin films spin-coated once except for the cross-sectional SEM image. It can be found that the thin film is consisted of dense nanoparticles with a mean particle size of 45 nm.
4. Conclusions

In conclusion, the luminescent properties of Eu$^{3+}$ and Tb$^{3+}$ co-doped CaMoO$_4$/SrMoO$_4$ thin films and the energy transfer from Eu$^{3+}$ and Tb$^{3+}$ ions were investigated. It was observed that the PL intensity of Eu$^{3+}$ can be enhanced when Tb$^{3+}$ ions are incorporated into Eu$^{3+}$-doped CaMoO$_4$/SrMoO$_4$ thin films. Fully coverage and crack-free luminescent thin films were fabricated by a facile solution approach. The band gaps ($E_g$) of luminescent thin films were found to be 4.71, 4.72, 4.55, and 4.49 eV for Ca$_{0.67}$Mo$_2$O$_7$: Eu$^{3+}_{0.15}$, Tb$^{3+}_{0.03}$, Ca$_{0.9}$Mo$_2$O$_7$: Tb$^{3+}_{0.03}$, Eu$^{3+}_{0.07}$Sr$_{0.78}$Mo$_2$O$_7$: Eu$^{3+}_{0.15}$, Tb$^{3+}_{0.07}$ and Sr$_{0.75}$Mo$_4$: Tb$^{3+}_{0.15}$, Eu$^{3+}_{0.07}$ thin films. These rare-earth ions co-doped luminescent thin films have a huge potential application in light-emitting diodes as the emitting layer or the photo-conversion layer.

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