

Design, synthesis and characterization of an orange-yellow long persistent phosphor: $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}, \text{Tm}^{3+}$

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Abstract: A novel orange-yellow emitting $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}, \text{Tm}^{3+}$ phosphor with bright and long persistent luminescence (LPL) has been newly developed. The incorporation of Tm^{3+} into the $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}$ as an auxiliary activator dominates its long persistent luminescence and thermoluminescence characteristics to a large extent. The emissions in $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}, \text{Tm}^{3+}$ for both fluorescence and LPL are due to the $5d \rightarrow 4f$ transitions of Eu^{2+} . The orange-yellow long persistent luminescence with the chromaticity coordination of (0.53, 0.46) can persist for nearly 220 min at recognizable intensity level ($\geq 0.32 \text{ mcd/m}^2$). This investigation provides a new and efficient long persistent phosphor which enriches the color of the existing LPL.

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OCIS codes: (160.4670) Optical materials; (160.4760) Optical properties; (250.5230) Photoluminescence.

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1. Introduction

Long persistent phosphors have attracted considerable attention for various displays and signing applications as friendly environmental and energy economized materials [1–5]. Now the long persistent phosphors and their relevant applications are challenged by the generation of multicolor long persistent luminescence (LPL). In principle, we can get any color-emitting LPL by mixing the three primary color-emitting long persistent phosphors. Until now, long persistent phosphors for two of the tricolor, blue (CaAl_2O_4 : Eu^{2+} , Nd^{3+}) and green (SrAl_2O_4 : Eu^{2+} , Dy^{3+}), have been commercially available. But long persistent phosphors for the red color, the third of tricolor, are still in search. On the other hand, this above method is hard to work in practice because we can hardly guarantee the very consistent LPL decay process for different components to ensure the uniformity of LPL color during the fade of the LPL. In addition, most of the tricolor long persistent phosphors currently available cannot be efficiently excited by the same excitation source [6]. So the exploration of any color-emitting long persistent phosphor is promising and thus has attracted intensive research interests in the past decades.

Nowadays, a great amount of novel long persistent phosphors based on different hosts have been reported in the literature, here we will give some typical examples, e.g. $\text{Ca}_2\text{Al}_2\text{SiO}_7$: Ce^{3+} [417 nm, >1 h] [7], $\text{CaMgSi}_2\text{O}_6$: Eu^{2+} , Dy^{3+} [447 nm, >4 h] [8], $\text{Sr}(\text{Ca})_2\text{MgSi}_2\text{O}_7$: Eu^{2+} , Dy^{3+} [470 (516) nm, 20 h] [9], $\text{Sr}_4\text{Al}_{14}\text{O}_{15}$: Eu^{2+} , Dy^{3+} [486 nm, 15 h] [10], Lu_2O_3 : Tb^{3+} , Ca^{2+} [green, 15 h] [11], $\text{Y}_2\text{O}_2\text{S}$: Ti , Mg [565 nm, 5 h] [2], Sr_3SiO_5 : Eu^{2+} , Dy^{3+} [570 nm, 6 h] [5], $\text{Y}_2\text{O}_2\text{S}$: Sm^{3+} [606 nm, 1.5 h] [12], CaSiO_3 : Sm^{3+} [pink, 5 h] [13], $\text{Sr}_2\text{Si}_3\text{N}_8$: Eu^{2+} , Tm^{3+} [612 nm, 10 min] [14], $\text{Y}_2\text{O}_2\text{S}$: Eu^{3+} , Mg^{2+} , Ti^{3+} , [orange to red, 1 h] [15] and Sr_2SnO_4 : Sm^{3+} [reddish-orange, 1 h] [16]. It can be noted that the performances of blue and green emitting long persistent phosphors have meet the requirement for practical applications, however, long wavelength emitting (> 600 nm) long persistent phosphor, whose persistent time last longer than 2 h, is still in great scarcity. Therefore, there is a strong desire for the development of long persistent phosphors with long wavelength emissions in recent years.

The Eu^{2+} -doped $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2$ produces a broadband emission peaked at 620 nm, which is a potential orange-yellow phosphor for white light emitting diodes [17]. This long wavelength emitting phosphor attracts our intense interest. Until now, it has been recognized that Eu^{2+} are good activators for long persistent phosphors because its 5d electron state is usually close to the conduction band of the host, which makes trapping of electron become possible [4,18,19]. In addition, by our observation, Eu^{2+} activated compounds containing alkaline-earth and aluminum ions as cations usually show LPL, for example, MAl_2O_4 : Eu^{2+} , Dy^{3+} ($\text{M} = \text{Ca}$, Sr , Ba) [1,20,21], SrAl_4O_7 : Eu^{2+} , Dy^{3+} [22], $\text{Sr}_4\text{Al}_{14}\text{O}_{25}$: Eu^{2+} , Dy^{3+} [10], $\text{Sr}_3\text{Al}_2\text{O}_6$: Eu^{2+} , Dy^{3+} [23]. Another interesting feature is that Eu^{2+} activated compounds containing sulfur ions as anions

also demonstrate LPL phenomenon, e.g. $\text{Y}_2\text{O}_3\text{S}:\text{Mg}^{2+},\text{Ti}^{3+}$ [8], $\text{CaS}:\text{Eu}^{2+},\text{Tm}^{3+}$ [21]. The present investigated compound $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2$ contains strontium and aluminum ions as cations, and oxygen as well as chlorine ions as anions. Considering that chlorine and sulfur are of similar chemical properties owing to they are adjacent elements in the periodic table, hence, we can predict that $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2$ may have the potential to serve as a new host material for Eu^{2+} containing long persistent phosphors. Further, the codoping of trivalent rare earth ions is generally capable of greatly improving the LPL of Eu^{2+} doped compounds. Usually with proper codopants, the persistent time can be increased by a factor of ten, for example, Dy^{3+} in $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+},\text{Dy}^{3+}$ [1]. Tm^{3+} as codopants in sulfides and nitrides based long persistent phosphors have been reported in literatures [14,18]. An interesting point is that in these sulfides and nitrides Eu^{2+} emissions are in the long wavelength range. And it seems that for long wavelength emitting long persistent phosphors containing Eu^{2+} , the best codopants is Tm^{3+} in order to produce excellent LPL properties. Based on the above clues, there could be a great possibility to generate efficient LPL from Eu^{2+} , Tm^{3+} -codoped $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2$. This motivated us to prepare $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+},\text{Tm}^{3+}$. As expected, strong orange-yellow LPL is observed in it. Next, we mainly report on its synthesis and LPL properties.

2. Experimental

The investigated phosphors in this work were synthesized through the solid-state reaction with SrCO_3 (A. R.), Al_2O_3 (A. R.), $\text{SrCl}_2\cdot 6\text{H}_2\text{O}$ (A. R.), Eu_2O_3 (99.99%) and Tm_2O_3 (99.99%) as raw materials. Stoichiometric mixtures of raw materials were homogeneously mixed and ground, and subsequently the mixture were placed in alumina crucibles with covers and sintered at 1100 °C for 4 h under ambient atmosphere in an electric tube furnace, and the sintered products were ground again in an agate mortar. Then the powder products were sintered at 900 °C for 2 h in a reducing atmosphere ($\text{N}_2:\text{H}_2=95:5$) to reduce the Eu from its trivalent state to its divalent state. After firing, the samples were cooled to room temperature in the furnace, and ground again into powder for subsequent use.

The phase identification of samples was carried out by a Rigaku D/Max-2400 X-ray diffractometer with Ni-filter Cu K α radiation. The excitation and emission spectra were measured by a FLS-920T fluorescence spectrophotometer with Xe 900 (450W xenon arc lamp) as the light source. The decay curves were recorded by a PR305 Phosphorophotometer. The thermoluminescence (TL) curves were measured with a FJ-427 TL meter (Beijing Nuclear Instrument Factory) with a heating rate of 1°C/s in the temperature range from 20 to 400 °C. Prior to the TL measurement, 0.0080 g samples were exposed to radiation for 10 min by 365 nm UV light from a low pressure Hg lamp with a power of 30 W. All measurements were carried out at room temperature except the TL curves.

3. Results and discussion

In our work, the effect of Eu^{2+} and Tm^{3+} doping concentration on LPL properties were investigated. The optimum phosphor is $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015},\text{Tm}^{3+}_{0.03}$ in which the persistent time of LPL achieves maximum. Figure 1 shows the XRD patterns of $\text{Sr}_{2.985}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$ and $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015},\text{Tm}^{3+}_{0.03}$. All the observed peaks can be indexed to the pure phase of $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2$ and match well with JCPDS card 80-0564, indicating the high purity and crystalline of the samples in this work. The doping of Eu^{2+} and Tm^{3+} does not make any noticeable variation of the XRD patterns.

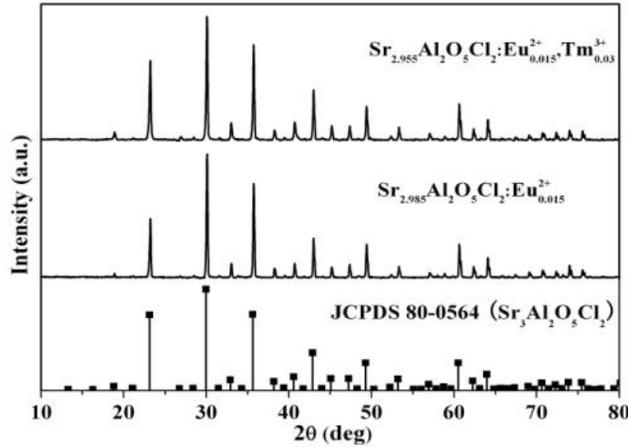


Fig. 1. XRD patterns of $\text{Sr}_{2.985}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Tm}^{3+}_{0.03}$ and JCPDS Card No.34-0379

Both $\text{Sr}_{2.985}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$ and $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Tm}^{3+}_{0.03}$ produce bright and efficient orange-yellow emission under UV excitation. The excitation and emission spectra of Eu^{2+} doped $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2$ have been reported by Tang *et al* [17]. Our photoluminescence results about $\text{Sr}_{2.985}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$ agree well with theirs and thus it will not be discussed here. Figure 2 displays the excitation and emission spectra of $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Tm}^{3+}_{0.03}$. The excitation spectrum is a broadband consisting of unresolved bands from 200 to 450 nm, which is due to the $4f^7-4f^65d^1$ of Eu^{2+} . The emission spectrum exhibits a broad emission band peaked at 620 nm, originating from the typical $4f^65d^1-4f^7$ transition of Eu^{2+} . The typical Tm^{3+} emission are not observed, indicating that it may not serve as luminescence centers in $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Tm}^{3+}_{0.03}$ and may play a role of trapping centers as that of in $\text{CaS}:\text{Eu}^{2+}$, Tm^{3+} [18].

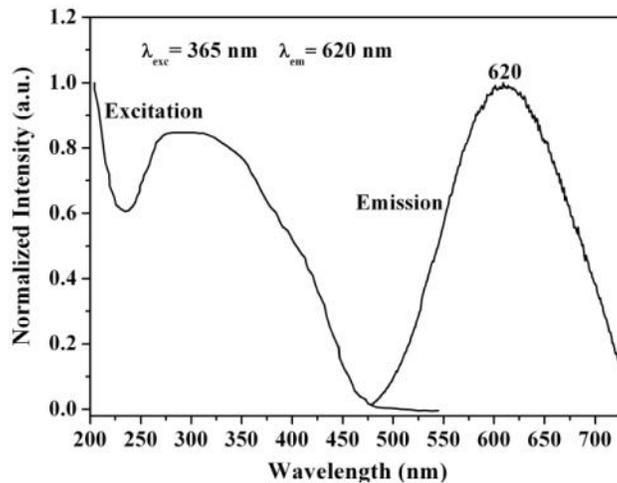


Fig. 2. Excitation and Emission spectra of $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Tm}^{3+}_{0.03}$.

As indicated above, when exposed to a 365 nm UV lamp, both $\text{Sr}_{2.985}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$ and $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Tm}^{3+}_{0.03}$ emit intense orange-yellow light, however, after the excitation source is switched off, only $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Tm}^{3+}_{0.03}$ yields orange-yellow LPL. An encouraging result of the present work is that the orange-yellow LPL in $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, $\text{Tm}^{3+}_{0.03}$ can be clearly observed with the naked eye in the dark, as seen in the inset A of Fig. 3 which was taken at 1 min after the exposure to the 365 nm UV

light. The LPL decay curve of the $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ is shown in Fig. 3. It can be seen that the initial LPL intensity of $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ can reach about 5000 mcd/m^2 and its LPL can last about 220 min at recognizable intensity level ($\geq 0.32 \text{ mcd/m}^2$). The inset B in Fig. 3 shows the LPL spectra of $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ measured at different times ($t = 1, 8 \text{ min}$) after removal of the excitation source. For comparison, its UV-excited emission spectrum is also shown in the inset B of Fig. 3. It is obvious that the shape and bandwidth of the UV-excited emission spectrum and the LPL spectra is similar, suggesting the LPL is due to the $4f^65d^1 \rightarrow 4f^7$ transitions of Eu^{2+} as well.

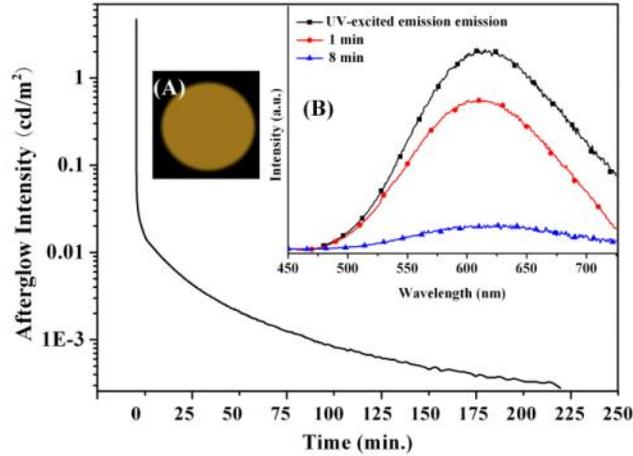


Fig. 3. The LPL decay curve of $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$; Inset A: Photos of prepared $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ sample was taken at 1 min after the removal of the 365 nm UV light. Inset B: the LPL spectra of $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ measured at different times (1 and 8 min) after removal of the excitation source ($\lambda_{\text{exc}} = 365 \text{ nm}$).

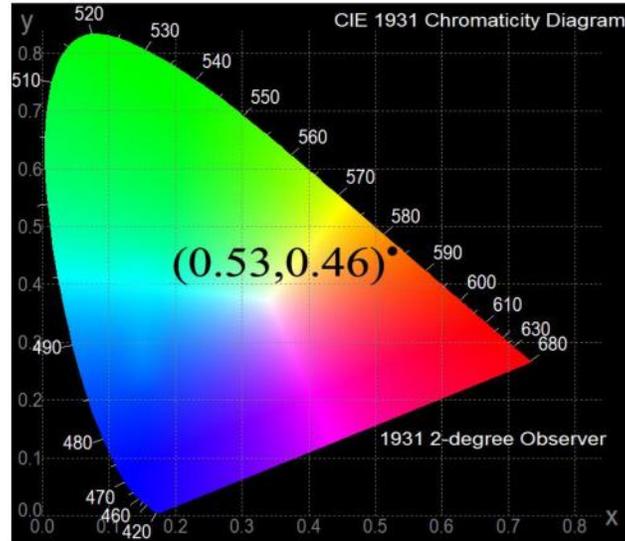


Fig. 4. CIE chromaticity diagram of the $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ at 1 min after the removal of the excitation source.

Figure 4 is the corresponding CIE chromaticity diagram of the $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ at 1 min after the removal of the excitation source. Point with chromaticity

coordination of (0.53, 0.46) in Fig. 4 locates in the region of yellowish-orange color, indicating that the LPL in $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ is orange-yellow.

The significant role of traps has been recognized in the field of LPL. In order to characterize the traps, TL measurements are performed on $\text{Sr}_{2.985}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$ and $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ and their TL curves are illustrated in Fig. 5. For $\text{Sr}_{2.985}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$, clearly, there is almost no TL phenomenon. For the codoped sample, one peak predominates at 61 °C and another two weak bands exist at 128 and 305 °C, respectively, suggesting that Tm^{3+} may serve as trapping centers. For long persistent phosphors, one critical factor is the suitable trap depth correlated to the TL peaks [1,24]. The optimized TL peak is situated slightly above room temperature (50-120 °C) for better LPL properties [3,5]. Therefore, the predominating peak at 61 °C in $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$ may be responsible for its orange-yellow LPL. However, the detailed mechanism of LPL in $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}, \text{Tm}^{3+}$ is needed to be further investigated.

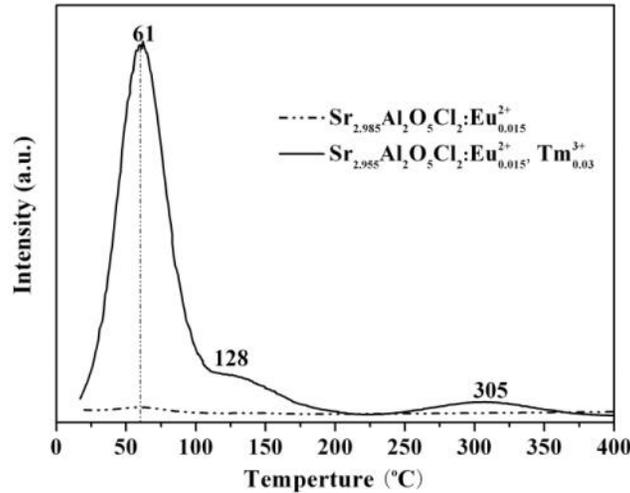


Fig. 5. Thermoluminescence curves of $\text{Sr}_{2.985}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}$ and $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}_{0.015}, \text{Tm}^{3+}_{0.03}$

4. Conclusion

Orange-yellow LPL was obtained from $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}, \text{Tm}^{3+}$ prepared via a solid state reaction. The intense orange-yellow LPL predominates at 620 nm, due to the $4f^{65}d^1 \rightarrow 4f^7$ transition of Eu^{2+} . The initial intensity of the orange-yellow LPL can reach nearly 5000 mcd/m² and its LPL can last about 220 min at recognizable intensity level (≥ 0.32 mcd/m²). The incorporation of Tm^{3+} into the matrix is essential to achieve the trapping centers associated with the dominant TL peak at 61 °C, which is responsible for the intense orange-yellow LPL. The present $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}, \text{Tm}^{3+}$ phosphor enriches the color of LPL and is a new member in the family of long persistent phosphors.

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