



## Recent progress in multicolor long persistent phosphors

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### ARTICLE INFO

Available online 17 December 2011

#### Keywords:

$\text{Ba}_4(\text{Si}_3\text{O}_8)_2:\text{Eu}^{2+}$

$\text{Dy}^{3+}$

$\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}$

$\text{Tm}^{3+}$

$\text{Sr}_2\text{SnO}_4:\text{Sm}^{3+}$

$\text{BaMgSiO}_4:\text{Eu}^{2+}$

### ABSTRACT

This paper highlights work from our laboratory on the recent progress in green, orange-yellow, and red long persistent phosphors. In addition, the phosphorescence mechanism in  $\text{Eu}^{2+}$ -containing phosphors is also discussed.

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### 1. Introduction

Long persistent phosphors have attracted considerable attention for various displays and signing applications as environmentally friendly and energy efficient materials [1–5], especially after the persistent luminescent property of  $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}$  was greatly improved by doping  $\text{Dy}^{3+}$  [1].

Now the commercial long persistent phosphors are aluminates phosphor ( $\text{CaAl}_2\text{O}_4:\text{Eu}^{2+}$ ,  $\text{Dy}^{3+}$  or  $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}$ ,  $\text{Dy}^{3+}$ ). However, the aluminates host has a bad water resistance. New applications for long persistent phosphors have been presented lately, such as radiation detection [6] as well as sensors for structural damage, fracture of materials [7–9], and temperature [10], which requires longer duration time and steadier host materials compared to the aluminates host. Moreover, the color of long persistent phosphors is very important. In principle, we can get any color-emitting afterglow by mixing the three primary color-emitting long persistent phosphors. Until now, long persistent phosphors for two of the tricolor, blue ( $\text{CaAl}_2\text{O}_4:\text{Eu}^{2+}$ ,  $\text{Nd}^{3+}$ ) and green ( $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}$ ,  $\text{Dy}^{3+}$ ), have been commercially available. But materials for the red color, the third of tricolor, are still in search. On the other hand, the present method is hard to work in practice because we can hardly guarantee the very consistent phosphorescence decay process for different components to ensure the uniformity of afterglow color during the fade of the long persistent phosphors. In addition, most of the tricolor long persistent phosphors currently available cannot be efficiently excited by the same excitation source [11]. So the exploration of any color-emitting long persistent phosphor is promising and thus has attracted intensive research interests in the past decades.

Although the long persistent phosphors are currently available commercially, only schematic mechanisms of the afterglow were proposed [12], and, since those mechanisms were derived from special phosphors, they could not be used therefore to explain all the factors, which affect the luminescence properties of persistent materials. Without the guidance of correct mechanisms, general techniques that allow the synthesis of the persistent materials with designated coloration and/or lifetimes certainly could not be obtained.

Based on the above research situation, our research work mainly focused on the following two aspects: (1) the development of novel long persistent phosphors. (2) the study on mechanism of long persistent phosphors.

### 2. Results and discussion

#### 2.1. Novel long afterglow phosphor

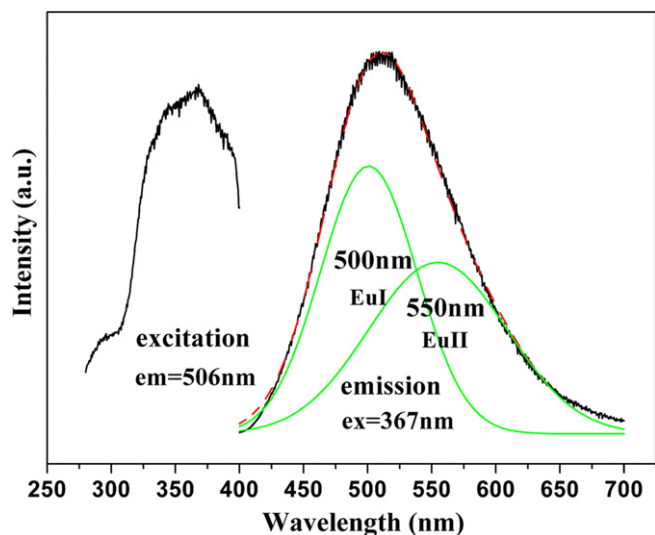
##### 2.1.1. Novel bluish-green long persistent phosphor $\text{Ba}_4(\text{Si}_3\text{O}_8)_2:\text{Eu}^{2+}$ , $\text{Dy}^{3+}$

In the recent years, long-lasting alkaline earth silicate phosphors prepared through solid reaction method were reported in some literatures. [13–15] It reveals that these phosphors are suitable hosts with high chemical stability and water-resistant property compared to sulfide phosphors and aluminates phosphors.

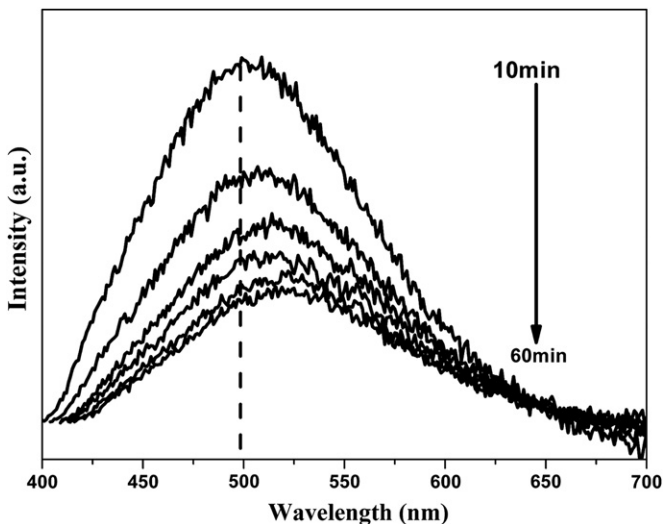
Recently, we obtained a novel bluish-green long persistent phosphor  $\text{Ba}_4(\text{Si}_3\text{O}_8)_2:\text{Eu}^{2+}$ ,  $\text{Dy}^{3+}$ . Two emission centers (Eu (I) and Eu (II)) were found in  $\text{Ba}_4(\text{Si}_3\text{O}_8)_2$  due to the substituted of  $\text{Eu}^{2+}$  in different  $\text{Ba}^{2+}$  sites, as shown in Fig. 1.

Fig. 2 reveals the phosphorescence spectra of  $\text{Ba}_{3.982}(\text{Si}_3\text{O}_8)_2:0.008\text{Eu}^{2+}\cdot0.01\text{Dy}^{3+}$  measure at different times after removal of the excitation source. As shown in Fig. 2, the emission peak of

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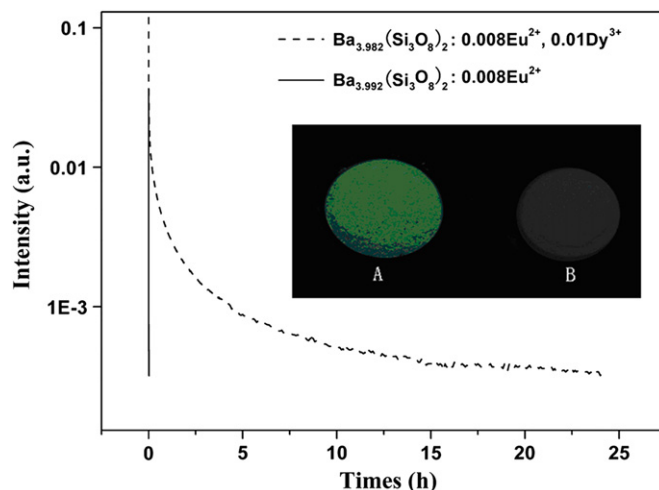
**Fig. 1.** Excitation and emission spectra of  $\text{Ba}_{3.982}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}, 0.01\text{Dy}^{3+}$  (the green lines are the Gaussian profiles). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



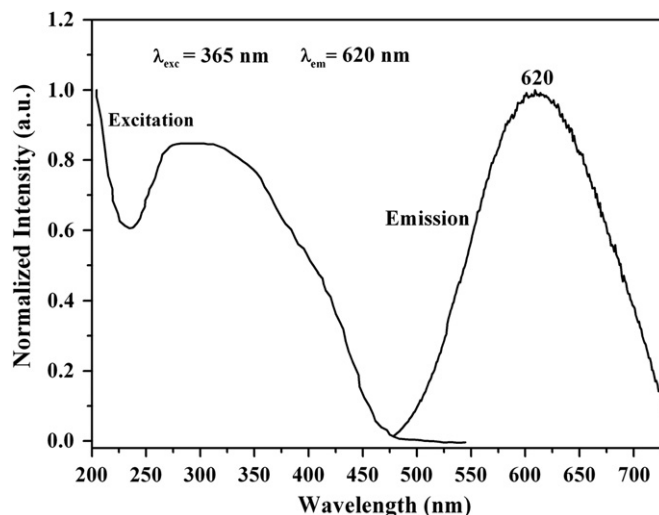
**Fig. 2.** Phosphorescence spectra of  $\text{Ba}_{3.982}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}, 0.01\text{Dy}^{3+}$  measure at different times after removal of the excitation source ( $\lambda_{\text{ex}}=367\text{ nm}$ ).

$\text{Eu}^{2+}$  moves from 506 nm to 522 nm as the delay time increases, which suggest that both Eu (I) and Eu (II) have contribution to the afterglow, however, the decay rate of Eu (I) is faster than that of Eu (II).

Fig. 3 exhibits the afterglow decay curves of  $\text{Ba}_{3.992}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}$  and  $\text{Ba}_{3.982}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}, 0.01\text{Dy}^{3+}$ . The afterglow time of  $\text{Ba}_{3.992}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}$  is only 2 min, which seems like a vertical line in Fig. 3. However, a very significant result of our present work is that the afterglow time of  $\text{Ba}_{3.982}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}, 0.01\text{Dy}^{3+}$  is more than 24 h at recognizable intensity level ( $\geq 0.32\text{ mcd/m}^2$ ), which is comparable for the commercial aluminates phosphors. The afterglow color photographs of the samples for 1 min after the removal of the 365 nm UV lamp are also shown in the inset of Fig. 3. It is intuitionistic that the bluish-green afterglow of  $\text{Eu}^{2+}, \text{Dy}^{3+}$  codoped sample (inset of Fig. 3A) is much stronger than that of  $\text{Eu}^{2+}$  single doped sample (inset of Fig. 3B), indicating the  $\text{Dy}^{3+}$  plays an important role in the afterglow process.



**Fig. 3.** Afterglow decay curves of  $\text{Ba}_{3.992}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}$  and  $\text{Ba}_{3.982}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}, 0.01\text{Dy}^{3+}$ . Inset A: long afterglow photographs of  $\text{Ba}_{3.982}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}, 0.01\text{Dy}^{3+}$ . Inset B: long afterglow photographs of  $\text{Ba}_{3.992}(\text{Si}_3\text{O}_8)_2: 0.008\text{Eu}^{2+}$ . The photographs were taken in the darkroom for 1 min after the removal of the 365 nm ultraviolet lamp. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)



**Fig. 4.** Excitation and Emission spectra of  $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}_{0.015}, \text{Tm}_{0.03}$ .

### 2.1.2. Novel Orange-yellow long persistent phosphor $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}^{2+}, \text{Tm}^{3+}$

$\text{Eu}^{2+}, \text{Tm}^{3+}$ -codoped  $\text{Sr}_3\text{Al}_2\text{O}_5\text{Cl}_2$  was prepared by solid state reaction with  $\text{SrCO}_3$  (A. R.),  $\text{Al}_2\text{O}_3$  (A. R.),  $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$  (A. R.),  $\text{Eu}_2\text{O}_3$  (99.99%) and  $\text{Tm}_2\text{O}_3$  (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  $1100^\circ\text{C}$  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  $900^\circ\text{C}$  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.

In our work, the effect of  $\text{Eu}^{2+}$  and  $\text{Tm}^{3+}$  doping concentration on afterglow properties were investigated. The optimum phosphor is  $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}_{0.015}, \text{Tm}_{0.03}$  in which the persistent time of phosphor achieves maximum. Fig. 4 displays the excitation and emission spectra of  $\text{Sr}_{2.955}\text{Al}_2\text{O}_5\text{Cl}_2:\text{Eu}_{0.015}, \text{Tm}_{0.03}$ . The excitation spectrum is a broadband consisting of unresolved bands from 200

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