Research Article

Luminescence Performance of Yellow Phosphor $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3\text{:Eu}^{3+}, \text{Gd}^{3+}$ for Blue Chip

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The $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3\text{:Eu}^{3+}, \text{Gd}^{3+}$ phosphors are synthesized by high temperature solid-phase method. Multiple techniques including X-ray diffraction (XRD), and scanning electron microscopy (SEM) are used to examine the surface morphology and structural properties of $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3\text{:Eu}^{3+}, \text{Gd}^{3+}$ phosphors. The optical properties are presented and discussed in terms of photoluminescence (PL) and photoluminescence excitation (PLE) spectra. The as-obtained $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3\text{:Eu}^{3+}, \text{Gd}^{3+}$ phosphors show higher PL emission intensity (at 591, 611 nm). The peaks at 591 and 611 nm are attributed to $\text{Eu}^{3+} {}^5\text{D}_0\text{--}{}^7\text{F}_1$, ${}^5\text{D}_0\text{--}{}^7\text{F}_2$. Gd^{3+} has a strong sensitization on Eu^{3+} . A certain amount of Sr^{2+} and Ba^{2+} is contributed to the intensity of light emission. After being irradiated with blue light, the phosphor samples emit yellow light. This suggests its potential applications in many fields.

1. Introduction

White light-emitting diode (WLED) has been developed rapidly over the past decade, since its advanced properties such as long life time, high efficiency, and being environmentally friendly without use of mercury [1, 2]. Currently, there are three main methods for WLED technology: blue chip and yellow phosphor approach; tricolor LED chip direct mixing method; ultraviolet conversion method [3, 4]. Generally, white LEDs are composed of a blue LED chip GaN and yellow phosphor such as $\text{Y}_3\text{Al}_5\text{O}_{12}\text{:Ce}^{3+}$. This system has a low color rendering index because of its deficiency in the red region of the visible spectrum [5, 6]. The other approach to overcome this drawback is to add a red-emitting phosphor to compensate the deficiency [7–10].

Titanate system has excellent properties such as very good physical and chemical stability, its stability in epoxy resin or other silicone encapsulating materials; meanwhile rich resources of titanium lead to the clear price advantage compared with other molybdates and tungstate. It provides favorable conditions for the development of rare earth titanate functional materials. Partial titanate MTiO_3 (M is

generally alkaline earth metal or alkali metal and rare earth ion composition) basic phosphor is perovskite compound [11], which is the stoichiometric chemical formula ABO_3 . Typically, a cation ionic radius is larger, mainly Ca, Sr, Ba, and alkaline earth metal ions Na, Ce; B ion radius is smaller mainly Ti, Nb, and so forth [12, 13]. Perovskite structure is mainly composed of B site cations and oxygen ions consisting of $[\text{BO}_6]$. Figure 1 shows the crystal structure of MTiO_3 . Partial titanate is a face-centered cubic which close-packed with cation of larger radius and O^{2-} . The transition metal Ti^{4+} of small radius is surrounded by six O^{2-} , and locate at the center of oxide octahedral $[\text{TiO}_6]$ [14, 15]. Since the research of Diallo et al. for $\text{CaTiO}_3\text{:Pr}^{3+}$ titanate phosphor, its excellent performance has attracted wide attention [11]. Fu et al. got the $\text{CaTiO}_3\text{:Eu}^{3+}$ red phosphor by doping Eu^{3+} [16]. In recent years, Yang et al. prepared $\text{Ba}_{0.77}\text{Ca}_{0.23}\text{TiO}_3\text{:Eu}^{3+}$ phosphors [17]. Due to the small concentrations of Eu^{3+} , it was surrounded by matrix like Ti^{4+} which formed a cluster. Other than the energy transfer for direct excitation, there are also some transfers between matrix and Eu^{3+} . The spectral characteristics of different Eu^{3+} doped position are also studied.

FIGURE 1: The crystal structure of MTiO_3 .

In this study, the $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}, \text{Gd}^{3+}$ phosphors are synthesized by high temperature solid-phase method, with Eu^{3+} as activator and Gd^{3+} as sensitizer. The CRI (Color Rendering Index) of the material is improved by adding some Ba. Compared with the traditional YAG: Ce in the missing part of red light, the light of this phosphor is more pure on yellow which solved YAG: Ce partial yellow-green issues. YAG: Ce main emission peak located at 560 nm which belongs to yellow-green band. The optimum excitation wavelength of this phosphor locates at 466 nm position which accords with the excitation wavelength of blue chip. Adjusting the ratio of blue and yellow light can get more pure white light, and the $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}, \text{Gd}^{3+}$ phosphor is expected to be widely used in the WLED field [18, 19].

2. Materials and Methods

2.1. Materials Preparation. $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}, \text{Gd}^{3+}$ was synthesized successfully via a conventional high temperature solid state reaction method. The reactants used in the preparation were $\text{Sr}(\text{NO}_3)_2$ (analytical grade, Tianjin Guangfu Fine Chemical Research Institute), $\text{Ba}(\text{NO}_3)_2$ (analytical grade, Tianjin Guangfu Fine Chemical Research Institute), TiO_2 (analytical grade, Tianjin Guangfu Fine Chemical Research Institute), Eu_2O_3 (analytical grade, Baotou Research Institute of Rare Earths), and Gd_2O_3 (analytical grade, Baotou Research Institute of Rare Earths). The stoichiometric amount of starting materials was weighed out and then mixed and milled thoroughly for 1 h in an agate mortar. Afterwards, the mixtures were transferred into corundum crucibles and pre-fired at 500°C for 1 h in a furnace. After cooling down naturally to room temperature, they were ground again for 30 min and finally calcined at 1100°C for 3 h in furnace.

FIGURE 2: The XRD patterns of the $\text{SrTiO}_3: \text{Eu}^{3+}$.

2.2. Analysis Methods. The structures of the phosphor were established by X-ray diffractometer (XRD) (Shimadzu, XRD-6000, Cu K α target) and the morphology of the particles was observed by field emission scanning electron microscope (FE-SEM) (Sirion 200, Philip). The photoluminescence properties of the phosphors were studied on fluorescence spectrophotometer (Shimadzu, model RF-5301 PC).

3. Result and Discussion

3.1. Structural Analysis. Structurally, SrTiO_3 crystallizes in a cubic structure, space group Pm-3m (no. 227), with a lattice parameter $a = 3.905 \text{ \AA}$. Figure 2 presents the XRD patterns of $\text{SrTiO}_3: \text{Eu}^{3+}, \text{Gd}^{3+}$ phosphors. It is in good agreement with JCPDS Card number 84-0433. The lattice constants of the phosphors are calculated as $a = 3.900 \text{ \AA}$ for $\text{SrTiO}_3: 0.05\text{Eu}^{3+}$, consistent with the unit-cell parameters reported in the literature. The ionic radii (r) of Sr^{2+} (CN = 12) are 112 pm. The ions radii of dopant elements, Eu^{3+} (CN = 12, $r = 107 \text{ pm}$) and Gd^{3+} (CN = 12, $r = 93.8 \text{ pm}$), are expected to occupy the Sr^{2+} sites in the SrTiO_3 host due to the close radii and identical valence of the ions [20]. There are only tiny TiO_2 impurity phases seen at $28\text{--}32$ degrees which does not have apparent effect on the performance and can be ignored.

3.2. Morphology Analysis. Figure 3 shows the SEM images of $\text{SrTiO}_3: \text{Eu}^{3+}, \text{Gd}^{3+}$ calcined at 1100°C for 3 h. From the sample global views and local features in Figure 3, we can see that the morphology of material is close to the sphere, with good homogeneity and smooth surface, and its size is about $0.25 \mu\text{m}$. In the LED material encapsulation process, morphology of the fluorescent powder has a greater impact on luminescent properties. It is generally believed that when the morphology of material is nearly sphere and up to the grade of close packing, we can improve the luminous intensity per unit volume [21]. In the experiment, the prepared samples can reach such requirements.

3.3. Luminescent Properties Analysis. Figure 4 presents the excitation and emission spectra of the $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}$,

FIGURE 3: SEM images of $\text{SrTiO}_3: \text{Eu}^{3+}, \text{Gd}^{3+}$ calcined at 1100°C for 3 h.

FIGURE 4: The excitation ($\lambda_{\text{em}} = 611 \text{ nm}$) and emission ($\lambda_{\text{ex}} = 466 \text{ nm}$) spectra of the $\text{SrTiO}_3: \text{Eu}^{3+}$.

Gd^{3+} sample. The excitation spectrum was measured at 611 nm monitored. Emission spectra were measured at 466 nm excited. The excitation spectrum (curve a) extends at 394 nm and 466 nm, due to ${}^7\text{F}_0-{}^5\text{L}_6$ and ${}^7\text{F}_0-{}^5\text{D}_2$ transitions of the Eu^{3+} . The $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}, \text{Gd}^{3+}$ phosphor shows a yellow emission band peaking at 591 nm and 611 nm under 466 nm excitation (curve b), which are attributed to $\text{Eu}^{3+} {}^5\text{D}_0-{}^7\text{F}_1, {}^5\text{D}_0-{}^7\text{F}_2$ [22].

In titanate phosphor, Eu^{3+} replaces the sites of Sr^{2+} in matrix. When the amount of incorporation of Eu^{3+} is changed, the number of Eu^{3+} replaces Sr^{2+} as luminescent center is changing. As Eu^{3+} concentration increases, the emission intensity increased with the adding of emission center, especially particular evident of ${}^5\text{D}_0-{}^7\text{F}_1$ transition. However, when the doping concentration is increased and the distance of luminescent center is decreasing, nonradiative energy transfer between luminescent centers has become increasingly evident, result of concentration quenching [23, 24], thereby reducing the emission intensity. Figure 5(a) shows the emission spectra of different concentrations of Eu^{3+} doping. The insets in Figure 5(a) are the change of intensity with the concentrations of Eu^{3+} . The spectra indicate

that the optimum doping concentration is 5% corresponding to the strongest emission intensity.

Figure 5(b) presents the emission spectra of different Gd^{3+} doping amount and the intensity trend. It can be seen that under 466 nm excited, all characteristic peaks in emission spectra are attributed to Eu^{3+} , and the strongest peak locates at 611 nm. Compared to the single doped Eu^{3+} , the enhancement on ${}^5\text{D}_0-{}^7\text{F}_2$ is significantly higher than ${}^5\text{D}_0-{}^7\text{F}_1$, which is due to the addition of the symmetry of Gd^{3+} in crystal structure. With the changing of concentration, the luminous intensity was reduced after the first enhancement process. Since the concentration increases to a certain value, interaction between the dopant ions within the host lattice, the energy released in the form of heat and energy passed to the luminous center of Eu^{3+} reduction, resulting in nonradiative transitions greater than radiative transitions, reducing the emission intensity of the product [25]. From Figure 5(b) it can be found that the optimum doping concentration is 3% corresponding to the strongest emission intensity.

Figure 6 shows the energy level diagram of Eu^{3+} and Gd^{3+} and the energy transfer process within or between rare earth ion energy levels when codoping Eu^{3+} and Gd^{3+} . The energy transfer process of Gd^{3+} to Eu^{3+} includes cross relaxation and direct transmitting. Under the excitation of ultraviolet to blue light, Gd^{3+} is excited and jumped from the ground state ${}^8\text{S}_{7/2}$ to ${}^6\text{G}_J$ excited state and then reradiation jumps back to ${}^6\text{P}_J$, through the cross relaxation process that transfers the excitation energy to neighboring Eu. The obtained energy of Eu^{3+} exactly conforms to the ${}^7\text{F}_J \rightarrow {}^5\text{D}_J$ transition energy. Excited Eu^{3+} returns to the ground state and emits orange light near 600 nm. On the other hand, the Gd^{3+} returning to the ${}^6\text{P}_J$ state directly transmits remaining energy to another nearby Gd, and when facing Eu^{3+} , Eu^{3+} absorbs energy and was excited to a higher excited state, then quickly radiated to ${}^5\text{D}_J(1,2,3)$ as nonradiative relaxation. Finally it was radiated to ${}^7\text{F}_J$ state. As a result, the energy absorbed by Gd^{3+} passes through two stages to excite Eu^{3+} [26–28].

3.4. Impact of Alkaline Earth Metal Doping. Crystal structure of the matrix material has a huge impact on luminescent properties. The experiment adding some Ba^{2+} to substitute Sr^{2+} is made to study its impact on the phase. Figure 7 shows the $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}$ ($\text{Sr}:\text{Ba} = 1:0, 4:1, 3:2, 2:3, 1:4$) XRD patterns and the standard BaTiO_3 (75-2120) and SrTiO_3 (74-1296) comparison chart. As can be seen, with the change in amount of Ba^{2+} added, the diffraction peak intensity was changed. Since Ba^{2+} (1.35 Å) and Sr^{2+} (1.18 Å) atomic radius have some differences, the substitution of Sr will produce changes in the crystal structure, and the corresponding XRD patterns are inevitably changing. With the increasing of proportion of Ba^{2+} , the original representative SrTiO_3 diffraction peak becomes low, and there were many miscellaneous peaks. When $\text{Sr}^{2+}:\text{Ba}^{2+}$ were 2:3 and 3:2, the two phases of BaTiO_3 and SrTiO_3 coexist. When $\text{Ba}^{2+}:\text{Sr}^{2+} = 4:1$, some low peaks again become acute. It can be found through XRD pattern that with the changing of doping percentage XRD patterns

FIGURE 5: Emission spectra of different doping concentrations: (a) Different doping concentrations of Eu^{3+} and (b) different doping concentrations of Gd^{3+} under 5% Eu^{3+} doped.

FIGURE 6: Energy level diagram of Eu^{3+} and Gd^{3+} .

FIGURE 8: The emission spectrum of $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}$.

FIGURE 7: The $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}$ (Sr: Ba = 1:0, 4:1, 3:2, 2:3, 1:4) XRD patterns.

are in good agreement with BaTiO_3 (75-2120), indicating that the majority of Ba^{2+} replaces the sites of Sr^{2+} ; the situation becomes crystal structure of BaTiO_3 structure majority [29, 30].

The emission spectrum of $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3: \text{Eu}^{3+}, \text{Gd}^{3+}$ is shown in Figure 8. From the figure we can see that the peaks which located at 591 and 611 nm did not change dramatically with the different proportions of Sr^{2+} and Ba^{2+} ; with the increasing of Ba, the luminescent intensity of phosphor powder shows a downward trend after the first rising. Since the different radii of Sr^{2+} (1.18 Å) and Ba^{2+} (1.35 Å), the incorporation of Ba^{2+} not only affects the lattice constant but also affects the symmetry of the crystal structure, thereby reducing the crystal field strength. With Ba^{2+} ions being incorporated, the outermost d-orbital is affected by the crystal field greatly; when Ba^{2+} incorporation amount exceeds a certain limit, the performance of the spectral emission is

intensely reduced. When the proportion of Sr^{2+} and Ba^{2+} is 4 : 1 the luminous intensity increased, from which we can see that a certain amount of Ba^{2+} is contributed to the intensity of light emission [31, 32].

The spectra at 591 nm and 611 nm were observed; we can found that the two corresponding relative strengths have some changes. The change of Eu^{3+} lattice site results in the different emission spectrum. The luminescence emission peak at 591 nm is corresponding to $\text{Eu}^{3+} {}^5\text{D}_0\text{-}^7\text{F}_1$ which is magnetic dipole transitions and 611 nm of ${}^5\text{D}_0\text{-}^7\text{F}_2$, which is electrical dipole transition. It is associated with the symmetry of the crystal. When there is no Ba^{2+} substituted Sr^{2+} , the transition of ${}^5\text{D}_0\text{-}^7\text{F}_1$ is dominant as the symmetrical sites. But with the increasing of Ba^{2+} , symmetry has a great change; Eu^{3+} substituted Ba^{2+} or Sr^{2+} which are in asymmetric sites; the transition of ${}^5\text{D}_0\text{-}^7\text{F}_2$ is prominent. Adjusting the ratio of blue and yellow can receive desired white light.

4. Conclusions

A series of $\text{Sr}_x\text{Ba}_{1-x}\text{TiO}_3\text{:Eu}^{3+}, \text{Gd}^{3+}$ phosphors were synthesized via the conventional high temperature solid state reaction method. The morphology of material is close to the sphere, and its size is about $0.25\text{ }\mu\text{m}$. The emission color of them was yellow to red, which was achieved with Eu^{3+} doped. The adding of Gd^{3+} could increase the intensity of Eu^{3+} . The energy transfer process of Gd^{3+} to Eu^{3+} includes cross relaxation and direct transmitting of Gd^{3+} and Eu^{3+} . It can be found corresponding to the strongest emission intensity the optimum doping concentration of Eu^{3+} and Gd^{3+} is 5% and 3%, respectively. The doping of Eu^{3+} and Gd^{3+} did not change the crystal structures but influence on the crystallinity. However after doping with Ba^{2+} , the crystal symmetry and ionic environment are changed; thus the spectral intensity and the spectral structure are transformed. By changing the proportion of the magnetic dipole transitions and electrical dipole transitions controllable switched the spectral structure of the optimum ratio of Sr^{2+} and Ba^{2+} is 4 : 1. According to the configuration coordinates, with blue chips, the sample can be well applied to WLED.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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