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Properties of a novel Ba₅Si₈O₂₁:Eu²⁺, Nd³⁺ phosphor: Bulk and 1D nanostructure with PVP synthesized by sol-gel and electrospinning

Yuanlin Yao, Zhufa Zhou^{*}, Feng Ye

College of Chemistry, Chemical Engineering and Materials Science, Soochow University, Suzhou 215123, PR China

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ABSTRACT

In this study, a sunlight-activated $Ba_5Si_8O_{21}$: Eu^{2+} , Nd^{3+} (BSEN) persistent luminescent particles were firstly synthesized by sol-gel method, and then combined with polyvinyl pyrrolidone (PVP) to fabricate one-dimensional functional fiber by electrospinning. XRD, photoluminescence, fluorescence microscope, SEM and TEM were used to investigate the crystal structure, the morphology, the luminescent properties and water resistance of BSEN particles and functional fibers. The results show that BSEN owns a monoclinic crystalline and can be effectively and repeatedly excited by both ultraviolet and sunlight. The BSEN particles were water-resistant and uniformly dispersed in the PVP fibers. Both BSEN particles and functional fibers possessed broad excitation spectra from 250 nm to 450 nm with maximum at 341 nm and exhibited a fluorescent and phosphorescent emission band from 365 nm to 650 nm with maximum at around 489 nm with long afterglow up to 240 min. The new functional fiber has potential to be used in flexible coating, textile and optical display.

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1. Introduction

Persistent luminescent material, which can emit visible or nearinfrared light for a long time after the excitation source been cut off, is a potential solution to the energy issue [1,2]. Persistent luminescent material has various applications in in-vivo imaging [3], nano probe [4], night vision [5] owing to its long afterglow. Many persistent luminescent materials are in application. For example, ZnS:Co, Cu [6] is used for watch dials but sensitive to moisture. SrAl₂O₄:Eu²⁺, Dy³⁺ has super long afterglow and high brightness but bad hydrolysis resistance [7]. M₂MgSi₂O₇:Eu²⁺,Dy³⁺ (M: Sr, Ca, Ba) possesses excellent afterglow and stable chemical property but lack of color diversity [8,9]. However, very few studies are reported in the sunlight-activated persistent luminescent material. It is economic and environmental friendly to use sunlight to activate persistent luminescent material. So far, the outstanding sunlightactivated material is Zn₃Ga₂Ge₂O₁₀:Cr³⁺, which emits nearinfrared light beyond 360 h with excitation spectrum from 300 nm to 650 nm [10].

Sol-gel preparation [11,12] is an effective method to synthesize persistent luminescent material compared to solid-state method

* Corresponding author. E-mail address: zhouzhufa@suda.edu.cn (Z. Zhou).

http://dx.doi.org/10.1016/j.jallcom.2017.04.102 0925-8388/© 2017 Elsevier B.V. All rights reserved. [13]. Usually, the latter demands high synthesis temperature, and particles prepared by this method are not small enough. Electrospinning is a facile method to fabricate one-dimensional fibers which draw considerable efforts in research and have various applications in optics, sensors and lasers due to their superior physical and chemical property [14,15].

The secret of sunlight-activated persistent luminescence is to broaden the width of excitation spectra so that the material can be directly activated by solar irradiation. Take Ba₅Si₈O₂₁:Eu²⁺, Dy³⁺ as an example, even if the persistent luminescence can last 8 h after solar irradiation, the width of excitation spectra of Eu^{2+} , Dy^{3+} codoped Ba₅Si₈O₂₁ do not get any broader than Eu²⁺ single doped Ba₅Si₈O₂₁ [16]. One effective method to tune the photoluminescence spectra is change the co-doping ions. Therefore, a persistent luminescent material of Ba₅Si₈O₂₁:Eu²⁺, Nd³⁺ (BSEN) has been synthesized by sol-gel method, which have broader excitation spectra and low cost due to the rare earth price and act as precursor for electrospinning. The precursors were used to synthesize onedimensional functional fibers by electrospinning [17,18]. The problem is that, the pure inorganic non-metallic fibers are instable and brittle, thus not suitable for application. To solve this issue, persistent luminescent material were mixed with polymer, and made into fibers by electrospinning [19-21]. Here, polyvinyl pyrrolidone (PVP) and Ba₅Si₈O₂₁:Eu²⁺, Nd³⁺ (BSEN) were mixed to synthesize functional fiber through electrospinning. The fiber owns





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2. Experimental

2.1. Synthesis and preparation of PVP/Ba₅Si₈O₂₁:Eu²⁺, Nd³⁺ fiber

A series of persistent luminescent materials $Ba_5Si_8O_{21}:0.02Eu^{2+}$, xNd^{3+} (x = 0, 0.02, 0.04, 0.06, 0.08) were synthesized by sol-gel method. The selection of molar ratio of lanthanides in this study is based on the past experience and random [16]. The raw materials were analytical grade $Ba(NO_3)_2$ (Sinopharm Chemical Reagent Co., Ltd, abbreviated as SCR), Si(OC₂H₅)₄ (TEOS, SCR), Eu₂O₃ (4N, SCR) and Nd₂O₃ (4N, Aladdin). Firstly, Eu₂O₃ and Nd₂O₃ were dissolved in nitric acid and then stirred with the distilled water. Secondly, $Ba(NO_3)_2$, TEOS and ethanol were added into the solution, and the pH was adjusted to 2 by adding nitric acid dropwise. The solution was stirred at 60 °C until formation of transparent gel. Thirdly, the gel was dried at 110 °C for 12 h. The resulting white gel was then sintered at 1250 °C for 3 h in a graphite reducing atmosphere.

In the following, PVP was dissolved in distilled water with a concentration of 0.2 g/mL and the solution was stirred at room temperature for 2 h. The $Ba_5Si_8O_{21}$:0.02Eu²⁺, 0.04Nd³⁺ particles (10 wt% of the functional fiber) were added in the solution and dispersed in ultrasonic vibrator for 1.5 h. The suspension was loaded in an injector and the injector was fixed on electrospinning equipment. The flow rate was set at 1.5 mL/h and the voltage 15 KV, the distance between collector and syringe needle was 15 cm. The fibers ejected on the collector were the target product. Then the fibers were dried in vacuum oven at 30 °C for 12 h.

2.2. Characterizations

XRD patterns were taken on a Rigaku S/Mx diffractometer (PAN analytical, The Netherlands) using Cu Kα radiation ($\lambda = 1.5406$ Å). Excitation and emission spectra and afterglow decay curves were collected by a fluorescence spectrophotometer (Hitachi F-2500, Japan). Absorption spectra were collected by a UV–Vis–NIR spectrophotometer equipped with an integrating sphere (Shimadzu UV-3600, Japan). Morphology images were taken on SEM (Hitachi S-4700, Japan) and TEM (FEI, America). Fluorescence microscopic images were taken on an inverted fluorescence microscope (EVOS, USA) at room temperature.

3. Results and discussion

3.1. XRD analysis of $Ba_5Si_8O_{21}$: Eu^{2+} , Nd^{3+} particles

We synthesize a new persistent luminescent material $Ba_5Si_8O_{21}$:Eu²⁺, Nd³⁺ (BSEN) using sol-gel method. Fig. 1 shows the XRD patterns of $Ba_5Si_8O_{21}$:0.02Eu²⁺ and $Ba_5Si_8O_{21}$:0.02Eu²⁺, 0.04Nd³⁺. The main diffraction peaks are consistent with the monoclinic $Ba_5Si_8O_{21}$ (JCPDS Card No.83–1443, a = 32.675, b = 4.695, c = 13.894, β = 98.10°). There are two conceivable sites that Eu²⁺ ions (1.25 Å) and Nd³⁺ ions (1.109 Å) to be incorporated, one is Ba^{2+} (1.42 Å) sites and the other is Si⁴⁺ (0.42 Å) sites. Considering the effective ionic radii of cations with different coordination numbers [22], Eu²⁺ ions and Nd³⁺ ions prefer to occupy one of the [BaO₈] site rather than [Si₃O₈] sites. Moreover, no impurities are found in the range of the scanning scope, which reveals that Eu²⁺ ions and Nd³⁺ ions had been incorporated in the monoclinic lattice without distorting the matrix remarkably [16].

3.2. Morphologies of PVP/Ba₅Si₈O₂₁: Eu^{2+} , Nd³⁺ functional fiber

Fig. 2 shows the SEM and TEM images of BSEN particles and PVP/



Fig. 1. XRD patterns of $Ba_5Si_8O_{21}{:}0.02Eu^{2+}$ and $Ba_5Si_8O_{21}{:}0.02Eu^{2+}, 0.04Nd^{3+}$ particles. JCPDS Card No. 83–1443.

BSEN functional fibers. Fig. 2a shows the fine size distribution of BSEN particles after being sintered at 1250 °C with an average size of 0.45 μ m. Fig. 2b shows that the functional fibers are randomly oriented and possess relatively uniform diameter. Because the process of electrospinning is complicated, every factor can affects the result more or less, including the orifice size, collector distance, flow rate and voltage. Therefore, it is hard to control the diameter of fibers precisely. The functional fibers would have evaporated liquid solvent since synthesized and shrinked a little which can be seen in Fig. 2c that some tiny wrinkles appeared on its appearance. Fig. 2d shows various shapes and sizes of BSEN particles are randomly distributed in the PVP fibers successfully and the functional fibers' diameter is approximately 2 μ m.

3.3. Photoluminescence of $Ba_5Si_8O_{21}$: Eu^{2+} , Nd^{3+} particles and PVP/ $Ba_5Si_8O_{21}$: Eu^{2+} , Nd^{3+} fiber

In this work, a series of persistent luminescent material $Ba_5Si_8O_{21}$: Eu²⁺, Nd³⁺ were synthesized by sol-gel method. All the samples show single monoclinic crystalline. For the purpose of confirming the best doping ratio of Eu/Nd, the univariate analysis of doping concentration of Nd³⁺ was conducted. As shown in Fig. 3a, the excitation spectra of BSEN particles cover a broad band from 250 nm to 450 nm with a maximum at 341 nm which correspond to the absorption spectra in Fig. 3c. Under the excitation of 341 nm, the BSEN particles exhibit an asymmetric broad band from 365 nm to 650 nm with a maximum at 489 nm which can be ascribed to the typical transition of the excited state $({}^{4}F_{6}^{5}D_{1})$ to the ground state $({}^{4}F_{7})$ configurations of Eu²⁺ ions [23]. The PL spectra reveal that the Eu³⁺ ions are reduced to Eu²⁺ because the typical emission peaks of Eu³⁺ around 580 nm, 590 nm, 612 nm, 650 nm and 698 nm due to the transition ${}^5D_0 \rightarrow {}^7F_{J (j=0, 1, 2, 3, 4)}$ are not found [24]. It is indicated that the emitter center is Eu²⁺ ions, and there are three nonequivalent Ba^{2+} sites in the matrix which can be substituted by the Eu^{2+} ions [16], therefore the asymmetric emission spectra are well-fitted by three Gaussian profiles (dark yellow dashed lined) in Fig. 3b.

Moreover, compared with the PL intensity of $Ba_5Si_8O_{21}$: Eu^{2+} , the PL intensity of $Ba_5Si_8O_{21}$: Eu^{2+} , Nd^{3+} is enhanced along with the increase of the concentration of Nd^{3+} at the beginning and reaches the highest intensity when the concentration of Nd^{3+} at 0.04 and then starts to decrease. The reason can be ascribed to the



Fig. 2. SEM images of BSEN particles (a) and PVP/BSEN fibers (b, c) and TEM image of PVP/BSEN functional fiber (d).



Fig. 3. Excitation and emission spectra of BSEN particles and PVP/BSEN fibers (a). Excitation and emission spectra of BSE and BSEN particles (b). Absorption spectra of BSEN particles (c). Fluorescence and persistent luminescence emission spectra (1 min after removing excitation) of PVP/BSEN fiber (d).



Fig. 4. Normalized excitation spectrum of BSEN particles. The red dashed line represents the solar irradiation at Earth's surface at AM 1.5G (a). Persistent luminescence spectra of BSEN particles after activation by solar irradiation for 30 min, the inset is the emission spectra of BSEN particles irradiated by sunlight and with 280 nm, 310 nm and 400 nm cutoff filters for 30 min (b). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 5. Afterglow images of BSEN particles taken from 0 s to 4 h after exposure to sunlight for 30 min.

concentration quenching effect [25,26] that Eu²⁺ and Nd³⁺ are induced in the matrix as activator to create defects to enable the matrix the property of photoluminescence, the defects and PL intensity are in a competitive relation and there always exist a suitable status that balance the structure and PL intensity. Therefore, we take Ba₅Si₈O₂₁:0.02Eu²⁺, 0.04Nd³⁺ as an example to show its other excellent properties in the following.

The PL spectra of PVP/BSEN fiber is shown in the Fig. 3a. The fiber can emit 489 nm light under 341 nm excitation. However, the PVP may decrease the PL intensity of fiber. The reasons are: (i) PVP polymer has reflection, scattering, absorption and other effects on light, when PVP/BSEN fibers excited by UV or sunlight, the energy can not be efficiently transmitted to the BSEN luminescent materials embedded in the fibers, and thus, the emission energy is weakened by PVP polymer. (ii) the physical properties of PVP polymer such as transparency and UV-resistant property may also

Fig. 6. Emission and persistent luminescence spectrum of BSEN water solution under 365 nm and after stopping activation for 1 h (a). Freshly prepared BSEN water solution activated by UV lamp at 365 nm (b) and aged solution for 1 h with UV lamp turn off (c) and turn on (d) at 365 nm.

lower the intensity [27]. Besides, Fig. 3d shows the fluorescence and persistent luminescence emission of PVP/BSEN fibers. The two curves both cover from 365 nm to 650 nm under 341 nm excitation. Moreover, the trend of fluorescence and persistent luminescence emission spectra is similar except different magnitude.

In addition, the BSEN particles own a good property of sunlightactivation and persistent luminescence. Fig. 4a shows a broad band excitation spectrum of BSEN and solar irradiation spectrum from 250 nm to 650 nm at Earth's surface under an air mass of 1.5 global (AM 1.5G). Generally speaking, the broader the excitation spectra is, the more energy the material can store and transfer into luminescence from solar irradiation. Obviously, the excitation spectra of BSEN(FWHM = 104.47 nm) is broader than the BSE's (FWHM = 93.64 nm) in Fig. 3b, and the excitation spectrum of BSEN overlaps with the solar irradiation spectrum from UV to visible light, which suggests that BSEN can be effectively activated by sunlight (Fig. 5). Fig. 4b shows the persistent luminescence spectra of Ba₅Si₈O₂₁:0.02Eu²⁺, 0.04Nd³⁺ particles after activation by solar irradiation for 30 min. The inset shows the emission spectra of Ba₅Si₈O₂₁:0.02Eu²⁺, 0.04Nd³⁺ particles irradiated by sunlight and with 280 nm, 310 nm and 400 nm cutoff filters for 30 min. It is safe to say that the BSEN particles still exhibit persistent luminescence even if the UV is filtered from sunlight.

In addition, BSEN owns an excellent property of water resistance, which ensures the material suitable for outdoor decoration and illumination. In order to prove this property, we performed water resistance experiments by preparing a BSEN water solution. Fig. 6a shows the PL intensity of BSEN water solution under 365 nm and after stopping activation for 1 h. Fig. 6b is the picture of the freshly prepared solution activated by UV lamp at 365 nm, Fig. 6c and d are pictures of aged solution for 1 h with UV lamp turn off and on at 365 nm respectively. Clearly, we can see that the solution still emit green visible light after 1 h with a lower brightness. Also the

Fig. 7. Fluorescence microscope image (a, $\lambda = 400$ nm) and phosphorescence microscope images (b) of PVP/BSEN functional fibers after removing excitation for 5min.

Fig. 8. Decay curves of BSEN particles excited by UV and sunlight, $Ba_5Si_8O_{21}$:0.02Eu²⁺ and PVP/BSEN functional fiber. Dynamic data were collected under 341 nm excitation at fixed time interval.

Table 1The fitting results of samples decay curves.

Sample	τ_1 (min)	τ_2 (min)
BSEN(UV activated) BSEN(Sunlight activated)	2.66 0.54	42.74 6.44
BSE	12.62	2.35
PVP/BSEN	0.69	8.67

aged solution is as bright as the freshly prepared solution once been activated again.

The fluorescent properties of PVP/BSEN functional fibers were investigated by fluorescence microscope. Fig. 7a shows the fluorescence image of PVP/BSEN fiber excited by blue light ($\lambda = 400$ nm). The fibers emit green visible light. Fig. 7b are phosphorescence images of PVP/BSEN functional fiber after stoppage of excitation for 5min. The fibers also emit green visible light although the brightness is getting weak with time lapse.

Here, the decay curves of different samples were shown in Fig. 8. The decay curves can be divided into two parts, one is the rapid attenuation whose slope is very steep, and the other is the slow decay whose slope is very gentle. The decay curves can be well-fitted with a double exponential equation which reflects the trend of the decay. The form of the equation is as follows [9]:

$$I = I_{01} \exp\left(-\frac{t}{\tau_1}\right) + I_{02} \exp\left(-\frac{t}{\tau_2}\right)$$
(1)

where I represents the photoluminescence intensity. I_{01} and I_{02} are constants. τ_1 and τ_2 are decay constants, deciding rates for the rapid and slow exponentially decay components respectively. The fitting results of constants. τ_1 and τ_2 are shown in Table 1. BSEN can be excited by both UV lamp at 365 nm as well as sunlight. The two green lines display the same decay trend and both have an afterglow more than 45 min, but the intensity of sunlight-activated BSEN particles is lower. Meanwhile, the PVP/BSEN functional fiber show the similar trend to that of BSEN particles. The afterglow was more than 45min and the intensity was also lower. However, compared with the other three samples, the slow decay process of $Ba_5Si_8O_{21}$: 0.02Eu²⁺ is rather weak, which can be seen more evident in the fitting results in Table 1. These results indicate that the dope of Nd³⁺ greatly improve the afterglow time. Actually, it is not enough for persistent luminescent materials to meet the commercial utilization demand without doping [28]. Many researches have conducted studies on the persistent luminescence, however, the defects caused by doping is very hard to confirm experimentally [29]. The probable mechanism may be that the luminescence originates from the transition of the excited state $({}^{4}F_{6}^{5}D_{1})$ to the ground state $({}^{4}F_{7})$ of Eu²⁺ ions [30], the afterglow comes from the slow release of electrons captured in trap energy level which produced in the matrix by doping with co-activator Nd³⁺ ions. The energy level lies between excitation state and ground state of Eu²⁺ ions [31]. When the material is excited, a part of the electrons return to the lower energy level and produce luminescence. The other electrons are captured in the trap energy level. When the electrons in the trap energy level absorb energy, they return to the excitation state again, and then transfer to the ground state and produce luminescence. The release of captured electrons is a continuous process; thus the material exhibits afterglow and the decay time is determined by the depth and concentration of trap energy level. Although the doping of Nd^{3+} is unlikely to deepen the trap energy level, the concentration of trap energy level is increased than the original state [32], which still prolong the decay time.

4. Conclusions

Persistent luminescent material $Ba_5Si_8O_{21}$:Eu²⁺, Nd³⁺ was synthesized by sol-gel method successfully and a new functional fiber PVP/BSEN was prepared by electrospinning. The photoluminescence spectra and persistent luminescence spectra of the BSEN and PVP/BSEN fiber exhibit a broad band emission spectra from 365 nm to 650 nm with a maximum at 489 nm, which can be ascribed to the transition of ${}^{4}F_{6}^{5}D_{1} \rightarrow {}^{4}F_{7}$ of Eu^{2+} . The afterglow is prolonged due to the co-doping of Nd³⁺. The BSEN particles own a good water resistance and can be excited by sunlight directly which endows the BSEN particles wider application. The PVP/BSEN fiber owns the properties of luminescence from BSEN and flexibility from PVP, and can emit visible green light by excitation of sunlight. For these properties above, the new functional fiber may be potentially commercial useful in display, textile and flexible coating.

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