

The effect of acid & alkali treatment on the luminous properties of red luminous material: $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent

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$\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} / light conversion agent is a new luminous material that can emit red light in the darkness after being excited was fabricated by combining light conversion agent on to $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} particles through Y_2SiO_5 . In order to investigate the effect of acid treatment & alkali treatment on the luminous properties of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} / light conversion agent, several kinds of samples were fabricated. The morphology of the luminous materials was analyzed by scan electron microscopy (SEM), the excitation and emission behavior were evaluated by Fluorescence Spectrophotometric analysis, the afterglow property was tested through PR-305 afterglow brightness tester. Results showed that part of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} was destroyed and some of the light conversion agent drop down from the surface of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} in the process of being treated. The shape and the peak of excitation and emission spectra showed little change, but the intensity became weaker as the treatment time increased. The afterglow brightness and the afterglow time dropped down gradually.

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1. Introduction

Rare earth aluminates luminescence material activated by divalent europium has many remarkable characteristics, such as high brightness and long afterglow [1-3], which serve a wide range of applications in many fields. In recent years, luminescence properties of rare earths persistent luminescence materials and rare earth complex luminescence materials have been widely studied [4-7]. Rare earth aluminates luminescence materials have generated much research interest in many fields such as materials science, chemistry, bio-technology and information technology. The applications of luminescence materials as textile materials are currently among the most popular and promising topics of research in textile science and technology. In 1998, a German company developed a novel fiber by adding Zn sulfide and Cd, Zn sulfide, which emitted green light in the ultraviolet radiation [8]. In 2002, two American companies developed luminous fiber on the admixture of a radioisotope ^{147}La , but the involvement of radioisotopes limited its uses. In 2004, E. Shim studied on the luminescence and mechanical properties of photoluminescent core bicomponent fibers which were produced with a sheath-core morphology [9]. Since 2000, Japanese researchers developed a kind of luminous fiber with skin-core structure [10-13]. In 2004, Jiangnan University, Wuxi, China, successively developed a kind of colorful rare earth luminous fiber [14], which could be

widely used in the development of all kinds of textiles having excellent properties of high luminescence intensity, long afterglow time, no radiation etc.

Rare earth luminous fiber (hereafter called the luminous fiber) is produced with polyethylene terephthalate, polyamide or polypropylene added rare-earth luminous materials. The luminous fiber exhibits superior brightness characteristics and afterglow decay properties. It absorbs visible light for 5 minutes and then can emit light for more than 10 hours in the darkness. The luminous fiber is a new kind of functional textile material which has no radioactivity and it is safe for the skin. This fiber has many practical applications in ergonomically designed safety protective clothing, as a decorative fabric, as a soft nap for toys, in embroidery works etc. Luminous fiber warning clothes, which are made for mine workers, could certainly help rescuers in locating trapped miners in times of crisis exactly, and is also convenient to save themselves if a dangerous situation arises during the rescue operations [15-16].

Phosphor, which is the cure material, used in the luminous fiber is mainly $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}$, Dy^{3+} currently [17]. Alkaline earth aluminates $x\text{MO}\cdot y\text{Al}_2\text{O}_3:\text{Eu}^{2+}$, RE^{3+} ($\text{M} = \text{Ca}, \text{Sr}, \text{Ba}$) are functional inorganic materials with strong luminescent intensity at the blue/green regions [18-20]. They show high quantum efficiency, long afterglow life, and good chemical stability. Recently, great progresses have been made in aluminate rare earth luminescent

materials due to its good water resistance and excellent chemical stability. However, the color of fiber's emitting light is rather monotonous at present, which limits the development of rare earth strontium aluminate luminous fibers. In our previous study [21], a new composite luminescent material $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent, which can emit red light in the darkness after being excited, was prepared through combining light conversion agent on to $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}, \text{Dy}^{3+}$ particles. But the energy transfer efficiency is $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}, \text{Dy}^{3+}$ to light conversion agent is lower and need to be improved, which can boost its luminous properties. The property of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ is stable compared to other phosphors, and the emission peak is located around 470 nm, the excitation peak of light conversion agent is located around 465 nm [22], which is helpful to the energy transfer, finally the red phosphor $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent has been successfully manufactured, but the stable of this red phosphor for application in the luminous fiber such as acid or alkali treatment has not been understood.

In order to investigate the stability of luminous properties of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent. In this study, $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent was prepared and treated with acid or alkali, the luminous properties were studied.

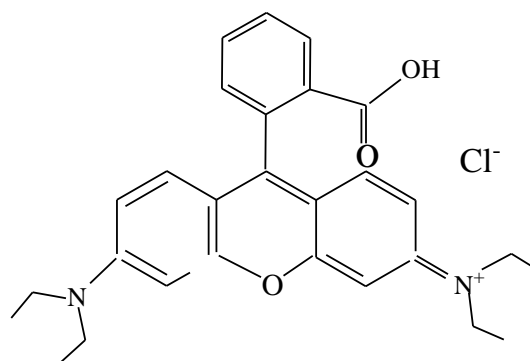
2. Experimental

2.1. Experimental

Materials: SrCO_3 (A.R.grade), MgO (A.R.grade), SiO_2 (A.R.grade), Eu_2O_3 and Dy_2O_3 (99.99% purity), H_3BO_3 (A.R.grade), ethanol (A.R.grade), light conversion agent were purchased from Sinopharm Chemical Reagent Co., Ltd China.

Preparation of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$: SrCO_3 , MgO , SiO_2 , Eu_2O_3 and Dy_2O_3 , H_3BO_3 were employed as raw materials for the solid-state reaction. The raw materials were ground together in a ball mill to give a homogeneous mixture, and the resulting powder was annealed at 1300 °C for 4 h in the carbon reducing atmosphere to give the complex $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$.

Preparation of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent: $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ and ethanol were put into an evaporating dish, then adding nitrate into it. After that the YsiX3 was added half an hour later. Afterwards, light conversion agent was added into compound above in the case of stirring and heating constantly ($\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ and light conversion agent were combined. Finally the samples were dried and the products were milled and sieved to get the desired samples. The structure of light conversion agent used in this study is shown as follow:



2.2. Sample preparation

Samples were placed in the solution of hydrochloric acid (0.5wt%) and sodium hydroxide solution(0.5 wt %) for 3, 6, 9, 12 min, respectively.

2.3. Characterization

Scanning electron microscopy : In order to investigate the distribution of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent with different light conversion agent concentration, the longitudinal structure of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent being treated were observed using scanning electron microscopy (SEM) on an SEM microscope (Quanta 200, the Netherlands), at an accelerating voltage of 20 kV. All samples were dried and coated with gold before scanning.

Luminous properties: The excitation and emission spectra of all the samples were measured at room temperature with an excitation wavelength of 360 nm using a fluorescence spectrophotometer (HITACHI 650-60, Japan) with a Xe flash lamp as an excitation source; the slit was 1 nm-5 nm in width; the excitation wavelength was from 200 nm to 800 nm and the scan speed was 120 nm/min. Afterglow decay curves were tested using PR-305 afterglow brightness tester (excitation illumination: 1000 lx, excitation time: 15 min). All measurements were carried out at room temperature. Before the testing, samples must be placed in the darkness for more than 15 h to be certain that afterglow illumination has been attenuated completely.

3. Results and discussion

3.1. Acid and alkali treatment

3.1.1. SEM analysis

The SEM images of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent being treated by acid and alkali are shown in Fig. 1. From figure it can be found that $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ was destroyed in a certain degree

than that of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ without being treated by acid or alkali. At the same time light conversion agent

can be completely coated on phosphor surface.

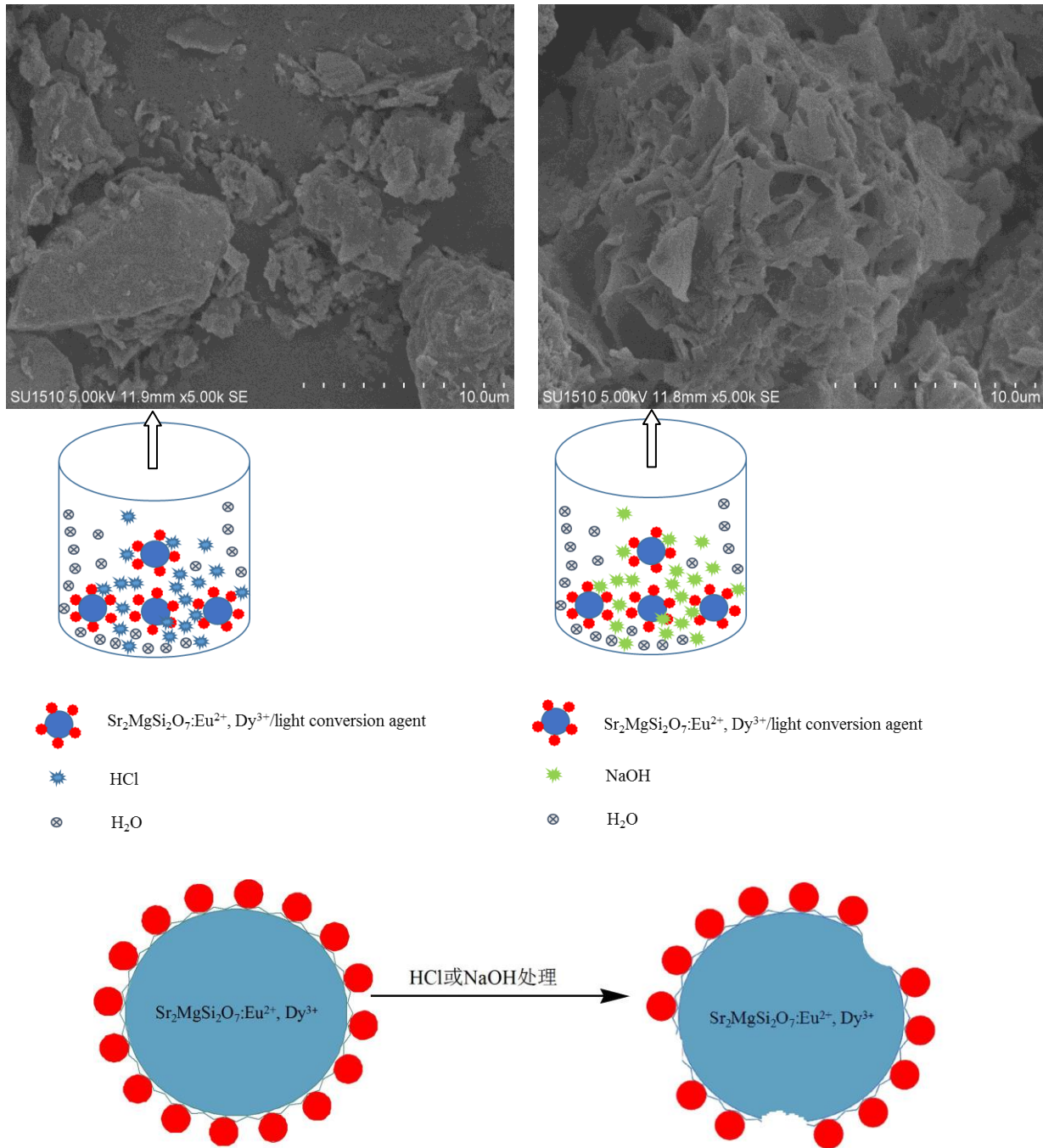


Fig. 1. Molecular structure of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent after being treated by acid and alkali (color online)

Irregularly shaped particles with sharp edges are clearly visible in the Fig. 1, and part of them were smooth on the surface of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ as shown in Fig. 1, From Fig. 1, It indicated that light conversion agent was completely coated on phosphor surface, however, chemical bond between $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ and light conversion agent may be broken, some of the light

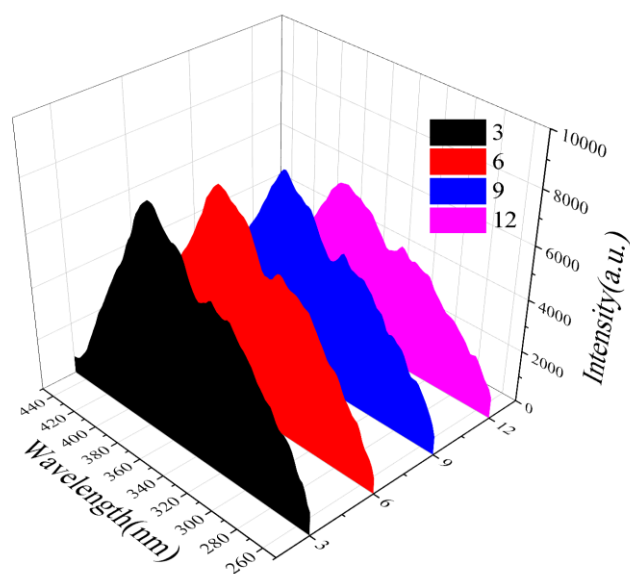
conversion agent drop down from the surface of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ in the process of being treated. Which would affect the luminous property of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ /light conversion agent. It is because that light conversion agent would absorb the light emit by $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$, and the electrons inner from could transit to the excited state from the

ground state. Finally because of the unstable energy, electronics in the excited would be back to the ground state, and released energy in the form of light. Forming continuous process of light energy (emit by $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+}) to the light conversion agent (absorption), and continuous energy transferred from $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} to light conversion agent.

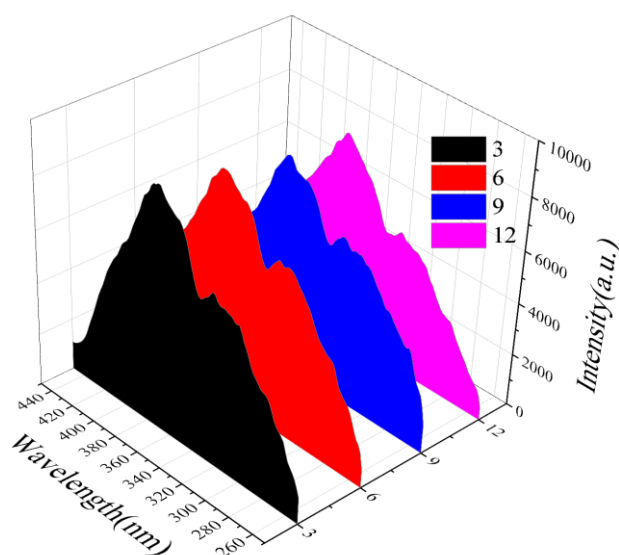
3.2. Excitation spectra of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent after being treated by acid and alkali

Excitation spectra of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent with acid-alkali treatment for different time are shown in Fig. 2. From Fig. 2 and the reference [23] it can be seen that the excitation spectrum of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} luminous fiber in the visible region had a wide excitation band, which was located at the wavelength range of 250 ~ 450 nm, and indicated a very good visible light excitation advantage. Also the excitation intensity of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent being treated was lower than that of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent without being treated, whatever, the excitation intensity of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent decreased as the treatment time increased, and the excitation peak was located around 360 nm, which belonged to the $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} [24], that is to say the main phase of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} was not destroyed in the treatment process. For the reason of excitation intensity decrease, it is thought that the very broad bands excitation spectrum extending from 300 to 450 nm, the peak center at 365 nm is due to the 4f-5d transition of Eu^{2+} ion and the specific lattice structure [25]. When it was treated by acid or alkali, part of the phase of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} was destroyed, losing its luminous properties, which can be seen from the Molecular structure of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent.

The results show that the compounds could be excited by ultraviolet and visible light. For the 5d electron lies in external layer in a naked and unshielded state, the splitting of energy level is strongly affected by the crystal field, and it makes the 4f5d - 4f transition of Eu^{2+} ion broad, it is closely related to the lattice structure. Thus the excitation intensity of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent would differ from each other when it was being treated by acid or alkali with different time.



(1) acid treatment



(2) alkali treatment

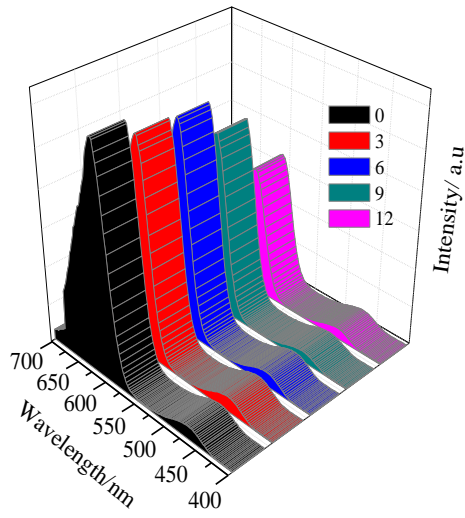
Fig. 2. Excitation spectra of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent with acid-alkali treatment for different time (color online)

3.3. Emission spectra of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent after being treated by acid and alkali

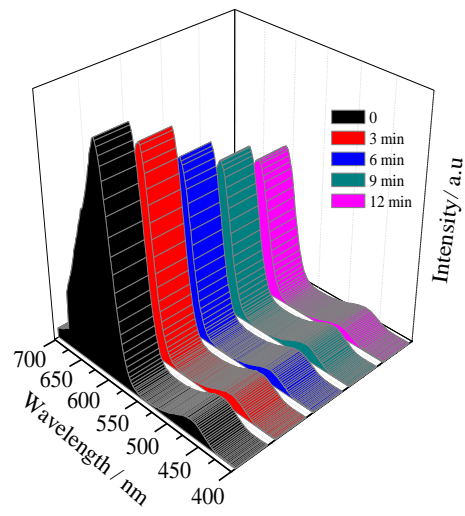
In order to investigate the acid-alkali resistance of emission properties. $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} /light conversion agent samples were put into the acid and alkali solution for several minutes as shown in Fig. 3. From Fig. 3 it is indeed that the principle of emission spectra were similar with each other containing two emission peaks around 470 nm and 600 nm, which accorded to the characteristic emission of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} and light

conversion agent we used in this article. However, the emission intensity was different from each other as the acid and alkali treatment time changed. The intensity of emission peak around 470 nm and 600 nm decreased gradually along with the treatment time increased.

properties, the emission intensity at 470 nm decreased gradually, and the energy transfer from $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ to light conversion agent. The light conversion agent absorbed by light conversion agent was less than that without being original. Thus the emission intensity at 600 nm decreased gradually



(1) acid treatment



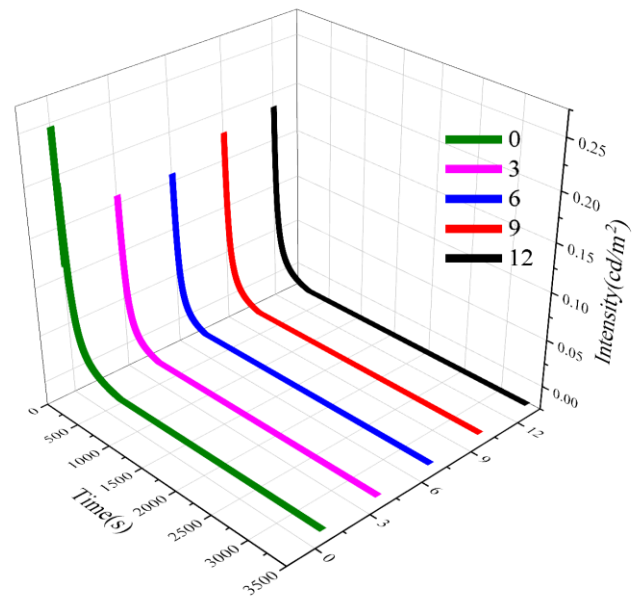
(2) alkali treatment

Fig. 3. Emission spectra of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent with acid-alkali treatment for different time (color online)

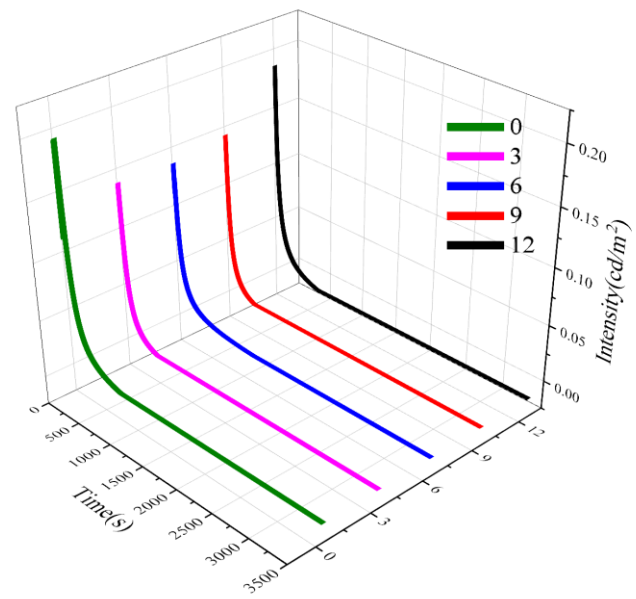
At the same time, Except for the emission peaks at 470 nm and 600 nm, no other emission could be seen in the band; this indicates that the Eu^{2+} is the only luminescence center [26], the light conversion agent just can absorb the light that $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ emit then form its characteristic emission [27].

From the analysis above, it is known that when $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent being treated with acid or alkali, part of the $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ would be destroyed, which would lower the luminous

3.4. Afterglow property of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent after being treated by acid and alkali



(1) acid treatment



(2) alkali treatment

Fig. 4. Afterglow of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent with acid-alkali treatment for different time (color online)

Fig. 4 shows the afterglow decay characteristics of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent with acid-alkali treatment for different time. The samples were been irradiated by UV light for 15 min and then been moved into the brightness meter. Collect brightness value at different point-in-times and then a graph of the persistent luminescence intensity versus time had been obtained by means of nonlinear fitting. From Fig. 4 it can be observed that afterglow brightness of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent treated with acid-alkali is lower to that of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent without being treated.

It is well known that light conversion agent was in favor of red emission promotion, however, the intensity of afterglow brightness and the afterglow time by the naked eye in the dark decreased gradually as the treatment time increased. It is because that the light conversion agent covered on the surface of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ in the process of fabricating this red phosphor: $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent, and part of the exciting light energy was absorbed and reflected by this light conversion agent. On the other hand, part of the light energy emitting by phosphors was absorbed and reflected by light conversion agent to some extent. As the light conversion agent covered on the $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ increased, more and more excited light was reflected and refracted, which reduced the emission intensity of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ and also the red light emitted by light conversion agent was reflected by light conversion agent. the excess light conversion agent can not get effective excitation and more of the light emitted by the light conversion agent was reflected by comparison.

From the analysis above we can refer to the conclusion that light conversion agent has an important influence on the afterglow properties of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent and it is generally believed that the afterglow intensity obtained are mainly due to the luminous properties of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ and light conversion agent. If the $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ was destroyed, the light energy transfer from $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ to light conversion agent became lower and the emission intensity of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$, which affected the afterglow properties of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent.

From Fig. 4, it can be found that the decay process of samples include rapid, moderate and slow decay processes. Afterglow illumination can be expressed by equation (1).

$$I = I_0 + I_1 e^{-t/\tau_1} + I_2 e^{-t/\tau_2} + I_3 e^{-t/\tau_3} \quad (1)$$

In the equation, I_0 and t_0 are modified constants, I_1 , I_2 , and I_3 are the starts of luminescence intensity of fast, moderate and slow decay stage respectively. τ_1 , τ_2 , and τ_3 represents the decay time of fast, moderate and slow decay. It is considered that three decay processes are caused by the different depth of trap levels in materials and the influence of light conversion agent, the shallower the depth of trap level is, the easier for electronics in traps to

escape, which brings about the short life of the fast decay process. The deeper the depth of trap level forms a slow decay process as well. Therefore different decay processes correspond to different depth of the trap level, which also influence the energy transfer from $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ and light conversion agent.

4. Conclusions

In order to investigate the luminous properties stability of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent. $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ was prepared by means of solid-state reaction, and $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent was fabricated by combining light conversion agent on to $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ particles through YSiX_3 , and it was treated with acid and alkali in different times. The luminous properties were studied through testing their excitation and emission spectra as well as afterglow property. The conclusions are summed up as follows:

- (1) From SEM analysis, it is showed that part of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ was destroyed and some of the light conversion agent drop down from the surface of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}$ in the process of being treated. Which would affect the luminous property of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent.
- (2) The shape and the peak of excitation and emission spectra showed little change, but the intensity became weaker as the treatment time increased.
- (3) The afterglow brightness of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent treated with acid-alkali is lower to that of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}, \text{Dy}^{3+}/\text{light}$ conversion agent without being treated. And the intensity of afterglow brightness and the afterglow time by the naked eye in the dark decreased gradually as the treatment time increased.

Acknowledgments

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