

The preparation and functional studies of the porous long afterglow luminescent materials

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ABSTRACT

As a kind of energy storage materials, the long afterglow luminescent material is used in many application fields. In this paper, the pore-forming agent of ammonium bicarbonate is added in the raw material, and the simple high temperature solid state method is adopted to prepare the long persistence luminescent materials with different pore structures. All the long afterglow materials are the series of Sr₂MgSi₂O₇: Eu²⁺, Dy³⁺, where the photoluminescence spectrum is the characteristic curve of the Eu²⁺ with the emission peak located around 465 nm. The pore diameter increases with the increase of the amount of pore-forming agent, which reaches the maximum with the size of 10–200 μm when the mass content of ammonium bicarbonate is 50%, accompanied by the result that the afterglow performance is improved by more than 72%. All the hole wall is composed of grains. The epoxy resin was used as the filling agent, provided light channels in the porous long persistence luminescent materials, and the afterglow performance could be improved by more than 40%. The initial intensity increases from 3.56 cd/m² (without pore-forming agent) to 9.51 cd/m². It provides an effective way for the application of long afterglow luminescence materials.

1. Introduction

With the energy depletion and environmental pollution continuously deteriorating, developing new energy conservation and environmental protection materials and products, enhancing and improving the existing such material performance become the focus of the development of science and technology. Long afterglow luminescent material can store energy when absorption natural light or lighting light source, continue to glow, and realize luminescence without electricity [1–3]. It is considered to be a kind of important energy-saving environmental protection material. The new generation of long persistence materials include aluminate and silicate long persistence luminescent materials [4–7]. Since reported firstly, they have aroused considerable attention all around the world. Especially the silicate long persistence luminescent materials Sr₂MgSi₂O₇: Eu²⁺, Dy³⁺ has a vast application prospect, it has excellent luminescent property, good chemical stability, strong resistance to water, no radiation, etc [8–10].

Despite the long afterglow luminescence materials have great application prospect in security signs and emergency signalization [1], persistent pigments [11], optical storage media and solar cell [12], photocatalysis [10,13], sensors [14], fingerprint detection [15], vivo imaging [16], and drug carriers [17]. The most researches on the long

afterglow luminescence materials still in the early stage, there are many challenges need to be explored now. At present, the commonly used preparation methods of long afterglow luminescence materials are high temperature solid phase method, but the grain diameter is relatively large in this way, and during the process of the preparation of the powders, the higher energy grain surface are easy to be damaged, which will lower the luminescence performance greatly [7]. At the same time, due to light scattering at grain boundaries, this material has poor transparency, and the inside material cannot be excited effectively, causing the great waste of the materials and performance of luminescence. There are many scholars use different chemical methods to obtain the long persistence luminescent materials with different morphology [18–23], but the obtained luminescence performance can still not match or surpass that prepared using solid phase method in the same size of the luminescent powder. Therefore, improving brightness of long afterglow material especially the initial brightness is a necessary way for the development of long afterglow materials. How to make the inside block exhibit afterglow performance without destroying the defects and crystal is a key issue to solve this problem and has great practical significance.

In view of the above-mentioned problem, in this paper, the proportion of pore-forming agent is studied to obtain porous

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$\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Dy}^{3+}$ long persistence luminescent materials with different pore distribution and sizes. On the premise of the completeness of matrix, the transparent filling material provide light channel to increase the amount of absorption of light. The implementation of the research has important scientific significances on the application and promotion of long afterglow luminescence materials and luminescence mechanism investigation.

2. Experiments

2.1. Synthesis

The $\text{Sr}_{1.96}\text{MgSi}_2\text{O}_7:0.02\text{Eu}^{2+},0.02\text{Dy}^{3+}$ materials were synthesized by a high-temperature solid state reaction method. Analytical reagent SrCO_3 , MgO , SiO_2 , H_3BO_3 , Eu_2O_3 (99.99%) and Dy_2O_3 (99.99%) which purchased from Sinopharm Chemical Reagent Co., Ltd. (China) were used. Then the raw materials were mixed for 1 h, followed by pre-sintering for 2 h under 1100°C , and subsequently mixed with the NH_4HCO_3 which was used as the pore-forming agent for 1 h. 2 g powder was pressed into one pellet with the diameter of 1 cm which was subsequently sintered at 1290°C in reducing gas flow (10 at.% $\text{H}_2 + 90$ at.% Ar) for 2 h. The samples were obtained after cooling down to room temperature in the furnace, and then filled by epoxy resin (from Wuxi Qianguang Chemical Materials Company (Jiangshu, China)) using the method of ultrasonic oscillation.

2.2. Measurements

The structures and phases of the samples were analyzed using an X-ray powder diffraction (PANalytical X'pert Pro type, Netherlands) with $\text{Cu } K_\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). The surface morphologies of the samples were characterized using field emission scanning electron microscope (FESEM, Carl zeiss, Germany). A Hitachi F4600 fluorescence spectrometer was employed to record the photoluminescence (PL) spectrum of the samples. The afterglow curves of samples were obtained by a long afterglow material optical test system with artificial light ($1000 \pm 5\text{lx}$). A ROSB TL/OSL3DP thermoluminescent dosimeter was used to get the thermoluminescence (TL) curves with the heating rate of 1 K/s . The measurements above were implemented at room temperature.

3. Results and discussion

3.1. XRD

Fig. 1 shows the XRD of porous long persistence luminescent materials $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Dy}^{3+}$ added pore-forming agent with different mass percent. All the peaks are indexed to the $\text{Sr}_2\text{MgSi}_2\text{O}_7$ tetragonal structure as the compound crystallizes (No. 113, JCPDS card: 75–1736, cell parameters: $a = 7.995 \text{ \AA}$, $c = 5.152 \text{ \AA}$, space group: P-421 m [24,25]). The results indicate that the lattice of all the samples keep good crystallinity, and the pore-forming agent and rare earth elements have no obvious impacts on the lattice structure of $\text{Sr}_2\text{MgSi}_2\text{O}_7$. Ammonium bicarbonate breaks down into ammonia, water and carbon dioxide at lower temperatures. The addition of pore-making agent did not introduce new impurities and have no significant influence on the chemical composition of long afterglow luminescent materials but the morphology and luminescence performance. Furthermore, as can be seen in Fig. 1, the peaks of the samples were sharper and stronger with the addition of pore-making agent, and the crystallinity of the long afterglow luminescent materials was improved with increase in pore-making agent. More hole structure allows raw materials to have a larger specific surface area that contact with firing environment, and the solid-state reaction proceeds more fully, so the crystallinity was improved.

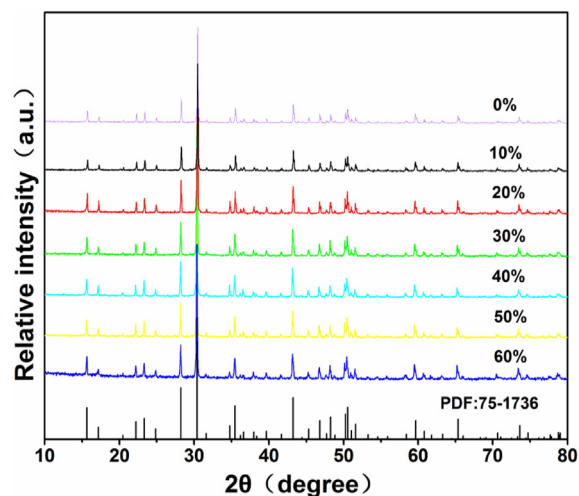


Fig. 1. The XRD of porous long persistence luminescent materials $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Dy}^{3+}$ added pore-forming agent with different mass percent (wt.% = 0%, 10%, 20%, 30%, 40%, 50%, 60%).

3.2. SEM analysis

Fig. 2 shows the SEM of porous long persistence luminescent materials $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Dy}^{3+}$ added pore-forming agent with different mass percent compared with that without pore-forming agent. As can be seen from the SEM, the addition of Ammonium bicarbonate have a great influence on the morphology of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Dy}^{3+}$ which has an obvious porous structure. All the porous long persistence luminescent materials have different pore structure and size, the size ranges from 10 to $100 \mu\text{m}$. With the increase of pore-forming agent, the samples generate more pores, and meanwhile the pores become bigger, leading to more exposed grain surface for absorbing light. It is can be seen that all the wall of hole is composed of grains. With the increase of pore-forming agent, the wall of hole is gradually becoming thinner. When the pore-forming agent content is 10%, the wall of hole is the thickest and contains many grains. As more pore-forming agent is added, the pore size becomes bigger and bigger. When the pore-forming agent content is 50%, the thickness of the wall of hole is only a few grains' size and consists of monolayer grains. When the dosage of pore-forming agent is more than 50%, the wall of hole collapsed in the sintering process. The volume density changed as the pore-forming agent was added. The volume density of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Dy}^{3+}$ with different pore-forming agent added is list in Table 1, and it ranges from 1.698 g/cm^3 with no pore-forming agent contained to the minimum 1.122 g/cm^3 .

3.3. Photoluminescence performance

Fig. 3 shows the excitation and emission spectra of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Dy}^{3+}$ phosphor with different porous structure, in which all the excitation wavelength are located around 397 nm. As is anticipated, the porous structure has no obvious effects on the position of the peak. All the peaks of the emission spectra are located around the wavelength of 465 nm with the range spanning from 430 nm to 550 nm, and it belongs to the $4f^5 d^1 \rightarrow 4f^7$ transition of Eu^{2+} [26,27], No other emission band is observed. But the intensities of the emission bands are different, the photoluminescence performances degrade gradually when the wall of the porous structure becomes thin. When the content of pore-forming agent is more than 50%, the collapse of pore structure happened, whereas the emission intensity increase, both of which are caused by the changes in the number of defects in the surface of grains [28,29].

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