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Influence of Eu^{3+} doping concentration on the luminescence properties of Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanoarrays via sol–gel template method

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ABSTRACT

Firstly, $Y_{(2-x-0.04)}O_3$: xEu^{3+}, $Mg^{2+}_{0.02}$, $Ti^{4+}_{0.02}$ (x = 5.0 mol%, 5.5 mol%, 6.0 mol%, 6.5 mol% and 7.0 mol%) nanotube arrays were synthesized by sol–gel template method to serve as the precursors. Then the precursors were calcined in CS_2 condition at 850 °C for 2 h to obtain the red long afterglow phosphor Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays with different Eu^{3+} doping concentration. The samples of theY_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays were examined by scanning electron microscopy (SEM), X-ray diffraction (XRD), photoluminescence spectroscopy and long-lasting phosphorescence. SEM proves that the Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays are highly ordered and have uniform size. XRD testified that the main phase constitution of the phosphor is Y_2O_2S. From the spectrum, the main emission peaks at 616 nm and 626 nm are excited by the 339 nm UV excitation, which are ascribed to the Eu^{3+} ions transition from $^5D_0$ to $^7F_2$. After irradiation by 365 nm UV radiation for 10 min, the decay time of the Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays with 6.5 mol% Eu^{3+} doping concentration could last for over 280 s ($\geqslant$1 mcd/m^2).
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1. Introduction

In recent years, more and more attention has been paid to the research on the single dimensional long-lasting nanocrystal phosphor which has the comprehensive applications in fuel cell, electronics, optics, biochemistry and medical devices [1–5]. The luminescence properties of this phosphor can be greatly affected by the dimension and morphology. Now, there are some methods of preparation for the fine nano-sized powders, such as homogenous precipitation method, sol–gel method, hydrothermal synthesis, sol–gel template method and so on [6–9]. The sol–gel template method which shows some advantages of the nanotube arrays with uniform diameters and highly ordered structure has become a promising method for the preparation of well-crystallized nanomaterials [9]. A great many nanocrystal phosphors with controlled morphology have been synthesized, for instance, some rare earth oxides and hydroxides,: Y_2O_3, ZrO_2, ZnO, $Y(OH)_3$, $Eu(OH)_3$, $La(OH)_3$ and $Gd(OH)_3$ [10–15]. As for rare earth oxysulfide, such as Y_2O_2S, Gd_2O_2S, Eu_2O_2S and La_2O_2S are known as nanorods [16], La_2O_2S and Nd_2O_2S as nanowires [17] and Y_2O_2S as nanotubes [18].

The Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} which has the advantages of stable chemical property, strong luminous intensity and long afterglow time is one of the best red long afterglow phosphors. The Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays, which have been synthesized via sol–gel template method, will be probably applied to biochemistry and medical devices on account of the highly ordered and integrated structure [9]. However, the luminescence properties of the Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays can be improved. Following the Zhang's notation, Eu^{3+}-doped tellurate garnet $Li_3Y_3Te_2O_{12}$, the luminescence spectrum will change significantly with the increasing of Eu^{3+} doping concentration [19]. Consequently, it is a significant work to improve the luminescence properties of the long-lasting nanocrystal phosphors.

In this paper, the Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays with different Eu^{3+} doping concentration have been synthesized by sol–gel template method. The effects of the disparate Eu^{3+} doping concentration on the spectral characteristic and luminescence properties of the Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays are also discussed systematically.

2. Experimental

2.1. Preparation of AAO templates

Firstly, the high purity aluminum sheet (99.999%, 7.5 cm × 1.5 cm × 0.5 mm) was annealed at 500 °C for 3 h in muffle and then decreased in acetone for 10 min. The aluminum plate (as anode) was electropolished to a mirror in a 1:4 (v/v) perchloric acid/ethanol solution under a constant current of 1 A at 0–3 °C for 10 min. Then polished aluminum plate (as anode) was processed

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by two steps anodizing which were weak oxidation and strong oxidation process. The polished aluminum plate (as anode) was oxidized at 40 V in 0.3 mol/L oxalic acid ($C_2H_2O_4$) at 0–3 °C for 6 h. In order to remove the alumina layer, the anode was subsequently immersed in an aqueous solution of H_3PO_4 6 wt% and H_2CrO_3 1.8 wt% for 8 h at room temperature. The anode was anodized again at 40 V for 10 min, then the voltage was slowly increased to 100 V with 1 V/min and the anode was anodized at 100 V for 2 h. Next the voltage was decreased to 10 V with 2.5 V/min, then the anode and the cathode were exchanged, and at last the electric current was kept around 1 A for 3 min to separate AAO template from the Al substrate. And the multihole AAO template was corroded by 5 wt% H_3PO_4 at room temperature (25 °C) for 5 h. Finally, the AAO template with highly ordered nanoporous arrays was obtained. The chemicals include oxalic acid, perchloric acid, ethanol solution, H_3PO_4, CrO_3 and deionized water. The oxalic acid, perchloric acid, H_3PO_4 and CrO_3 were purchased from the third Chemical Reagent Factory of Tianjin in China. The ethanol solution was purchased from the Tianda Chemical Experiment Factory of Tianjin in China. The deionized water was prepared ourselves.

2.2. Preparation of Y_2O_3: Eu^{3+}, Mg^{2+}, Ti^{4+} sol with different Eu^{3+} concentration

Firstly, Y_2O_3 (99.9%, 0.04 mol), Eu_2O_3 (99.99%, 0.0010 mol, 0.0011 mol, 0.0012 mol, 0.0013 mol, 0.0014 mol) and $Mg(OH)_2{\cdot}4MgCO_3{\cdot}2H_2O$ (99.3%, 0.00016 mol) were dissolved in 17.35 ml HNO_3 (65%) and then stirred the mixture at 80 °C for 1 h to form five kinds of Eu^{3+} doping concentration nitrate solutions. Secondly, these nitrate solutions were added into 400 ml ethanol and stirred for 20 min. Thirdly, the butyl titanate solutions modified by the acetyl acetone for 2 days were added into the nitrate solutions and stirred for 10 min. After the pH of these solutions were regulated to 1 with HNO_3 (65%), the solutions were evaporated at 80 °C in the process of stirring to form five kinds of Y_2O_3: Eu^{3+}, Mg^{2+}, Ti^{4+} sol (3.0 mol/L) with different Eu^{3+} doping concentration. Then these five kinds of Y_2O_3: Eu^{3+}, Mg^{2+}, Ti^{4+} sol (3.0 mol/L) were put into five jars which were marked A1, A2, A3, A4 and A5 respectively.

2.3. Preparation of Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanoarrays with different Eu^{3+} concentration

Firstly, the AAO templates were divided into five sections which were marked A1, A2, A3, A4 and A5. Then the A1, A2, A3, A4 and A5 were dipped into the above five jars of Y_2O_3: Eu^{3+}, Mg^{2+}, Ti^{4+} sol with different Eu^{3+} doping concentration accordingly and the jars were pumped by the rotary-vane vacuum pump. After 3 min, the five AAO templates were removed and allowed to dry in the oven at 70 °C for 2 h. The five AAO templates which were taken out and immersed into the corresponding Y_2O_3: Eu^{3+}, Mg^{2+}, Ti^{4+} sol with different Eu^{3+} doping concentration were reprocessed by the above steps 10 times. Secondly, the AAO templates were put in a muffle furnace and calcined at 600 °C for 2 h with the velocity of 6 °C/min. After the temperature of the templates cooling down to the room temperature, the adhesive substance on the surface of the five templates was cleaned out by abrasive paper. Thirdly, the templates were etched by 2.0 mol/L aqueous NaOH for 8 min to obtain the samples of Y_2O_3: Eu^{3+}, Mg^{2+}, Ti^{4+} nanoarrays with different Eu^{3+} doping concentration. Finally, the templates A1, A2, A3, A4 and A5 were calcined in CS_2 condition at 850 °C for 2 h to form the long afterglow phosphors Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} with different Eu^{3+} concentration.

The morphologies of the AAO template and Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+}/AAO nanoarrays were characterized by a scanning electron microscopy (SEM, Hitachi S-4800FESEM). The PL spectra of the samples were measured by an F-280 spectrophotometer. The XRD pattern of the sample was examined by an X-ray diffractometer (XRD, DX-2500). The afterglow decay curves were measured by the brightness meter (ST-86LA) with a stopwatch. All the measurements were performed under the room temperature.

3. Results and discussions

3.1. SEM analysis

Fig. 1 indicates the SEM images of the AAO template. Fig. 1(a) is the top-view which shows that the surface of the AAO template is smooth and the average diameter of the pore is about 100 nm. In Fig. 1(b), the image proves that the nanoporous arrays are highly ordered.

Fig. 2 shows the SEM images of Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays with Eu^{3+} doping concentration (6.5 mol%). In Fig. 2(a), the image indicates that the AAO template is filled with the Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} which formed the nanorods with orderly arrangement, and the Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays are in a uniform diameter of about 100 nm. In Fig. 2(b), the image demonstrates that the cross-section, which is etched by NaOH solution (2 mol/L) and calcined in the CS_2 condition at 850 °C for 2 h, inherits the morphology of the nanotube arrays with the highly ordered and regularly arranged structure.

3.2. XRD spectra

Fig. 3 shows the X-ray diffraction pattern of Y_2O_2S: Eu^{3+}, Mg^{2+}, Ti^{4+} nanotube arrays with Eu^{3+} doping concentration at 6.5 mol%, which indicates that the sample matches well with the no. 24-1424 JCPDS Card. For the ionic radius of Eu^{3+} (0.112 nm) is close to that of Y^{3+} (0.106 nm), the Eu^{3+} doping does not upset the crystal structure and lattice parameters of Y_2O_2S, but the Eu^{3+} ion

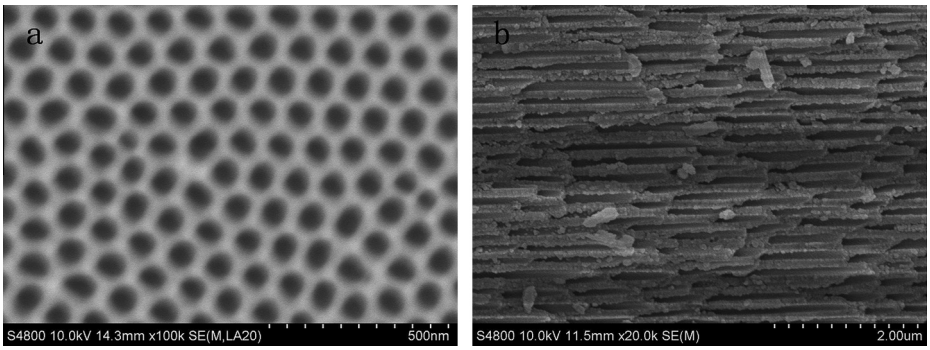

Fig. 1. SEM images of the AAO template: (a) the top-view, (b) the cross-section.

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