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Synthesis and properties of $SrAl_2O_4:Eu^{2+},Dy^{3+}$ nanowires

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ABSTRACT

The $SrAl_2O_4:Eu^{2+},Dy^{3+}$ nanowire was successfully synthesized by means of catalyst-assisted thermal chemical vapor deposition method. Their morphology, structure, composition, luminescent properties are explored in way of SEM, TEM, XRD and PL analysis. The nanowires diameter is uniform distributed in 50∼80 nm, but orientation distribution is irregular, with the length varying from 4 μm to 20 μm. When heated up to the temperature of 1200 °C for three hours, the optimum synthesis is achieved with the alumina substrate covered by Al nanoparticles. The emission peak reaches to 517 nm with 365 nm light excitation and the luminous intensity was down to 1/10 of the initial brightness in 20 mins. The dielectric property was investigated at the room temperature, which show stronger dielectric loss ability.

1. Introduction

The value of $SrAl_2O_4:Eu^{2+},Dy^{3+}$ is rapidly recognized by the public since its birth [1–4] and consequently, has been widely used in many fields [5–8]. Its excellent properties accorded it great potential applications in optical equipment [9] and electronic equipment [10–13]. At present, the synthesis of $SrAl_2O_4:Eu^{2+},Dy^{3+}$ mostly relies on high temperature solid state method, and the product is particles with irregular surfaces and nonuniform size which can reach micron [14,15], which can not meet the needs of super clear display and imaging in vivo [16,17], thus, great importance should be attached to nanostructure long afterglow phosphor [18]. At present, there is more $SrAl_2O_4:Eu^{2+},Dy^{3+}$ nanopowder preparation but fewer $SrAl_2O_4:Eu^{2+},Dy^{3+}$ nanowires preparation, and the nanowires with diameter ratio longer than 20 are seldom reported [19–22]. The long afterglow and luminous performance of $SrAl_2O_4:Eu^{2+},Dy^{3+}$ nanopowder are not as better as a micron level decline. Therefore, in order to maintain the long afterglow, the Luminous intensity and nanoscale [23], it is necessary and meaningful to study $SrAl_2O_4:Eu^{2+},Dy^{3+}$ nanowires. Among the synthesis of nano materials, CVD is used frequently [23–28] and most likely to be integrated into the microelectronic technology and micro-fabrication technology. This paper focuses on the synthesis of $SrAl_2O_4:Eu^{2+},Dy^{3+}$ nanowires by Catalyst-assisted thermal chemical vapor deposition method, the luminescence, dielectric loss of phosphors $SrAl_2O_4:Eu^{2+},Dy^{3+}$ nanowires were also investigated in detail as well [29,30].

2. Experiment

The samples were prepared in horizontal vacuum tube furnace through a catalyst-assisted thermal chemical vapor deposition process. With analytically pure $SrCO_3$, Al_2O_3, Eu_2O_3 and Dy_2O_3 as raw materials, Al nanopowders (60∼90 nm) as catalysts, the raw material $Sr_{0.963}Al_2O_4:Eu_{0.013},Dy_{0.024}$ is calcined at 1400 °C for 3 h in the central heating zone of tubular vacuum sintering furnace with gas mixture (5% H_2, 95% Ar) kept at 10^5 Pa pressure and 30 sccm flow. Alumina substrate with Al nanopowders is used to collect resultant from the place 20 cm away from the central reaction zone and thermocouple is employed to test the corresponding temperature. And then, the resultants were collected.

With an X-ray diffractometer (XRD-7000), a scanning electron microscope (JSM-7800F) and a transmission electron microscope (JEM-2010(HR)). The crystal structure and morphology of the products were investigated. In order to study the optical, dielectric properties of samples, a Photo luminescence (F-4500) and vector network analyzer (Agilent E5071C) were employed.

3. Results and discussion

Fig. 1 shows X-ray diffraction patterns of resultant at 1200 °C for 3 h. The result of XRD reveals that the product is monoclinic

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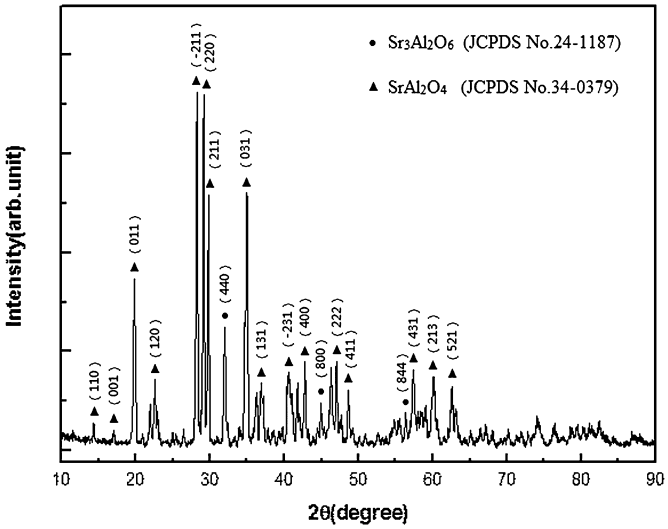

Fig. 1. XRD patterns of resultant.

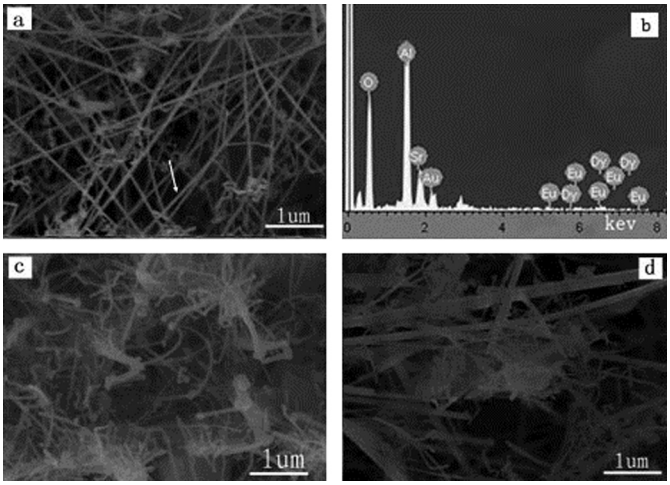

Fig. 2. SEM of resultant at 1200 °C, for 3 h (a), for 1 h (c) and for 5 h (d); (b) EDS image of arrow points in the image (a).

$SrAl_2O_4$ with little cubic structure of $Sr_3Al_2O_6$, and $Sr_3Al_2O_6$ is the intermediate of the reaction.

Fig. 2 shows SEM and EDS of the position where the arrow points, (a), (c), (d) corresponds to resultant with different calcination time. The deposition temperature of (a) resultant is about 1200 °C for 3 h, as shown in Fig. 2(a), the nanowires diameter is uniformly distributed between 50∼80 nm, but its orientation distribution is irregular, with the length varying from 4 μm to 20 μm. Its diameter size is proportional to Al nanoparticles size in the substrate, and its length is affected by the quantity of $SrAl_2O_4$:Eu^{2+}, Dy^{3+} generated in the atmosphere. Fig. 2(b) is the EDS analysis of the position the arrow points in the Fig. 2(a), the atomic ratios of the elements of the nanowires are in agreement with the stoichiometric formula $SrAl_2O_4$:Eu^{2+}, Dy^{3+}, the Au is sprayed when electron microscope samples are prepared. Fig. 2(c) shows SEM of product at 1200 °C for 1 h, nanowires are short, bent and diameter is uneven, the head of nanocone has a particulate matter, the size and the length of which is about 100 nm and 1.5 μm, this accords with VLS growth pattern. Fig. 2(d) shows SEM of product at 1200 °C for 5 h, its main products are nanorods, as can be seen from Fig. 2(d), the diameter becomes thicker and distributed unevenly, its head does not have particulate matter.

Fig. 3 is the TEM, SAED, HRTEM of a nanowire in Fig. 2(a), Fig. 3(a) for a single $SrAl_2O_4$:Eu^{2+}, Dy^{3+} nanowire low magnification TEM, from which we can see that the surface of the nanowire is smooth, and uniform diameter is about 50 nm, and the length is not less than 2 μm. Fig. 3(b) is SAED in Fig. 3(a) for the square area, the zone axis direction is [100], it is confirmed with a monocline, in which (010) facet interplanar spacing is about 0.88 nm, (001) facet interplanar spacing is about 0.50 nm.

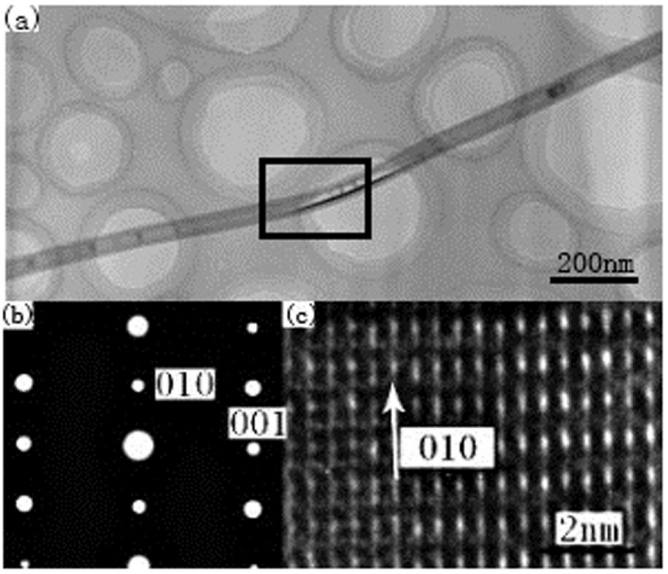

Fig. 3. TEM image (a), SAED pattern (b) and HRTEM image (c) of $SrAl_2O_4$:Eu^{2+}, Dy^{3+}.

Fig. 3(c) is HRTEM image of nanowire. The interplanar spacing is consistent with Fig. 3(b). Atomic arrangement has no obvious defects and dislocations, which is a good single crystal. The direction of the white arrow in the figure is the growth direction of the nanowire, that is, the [010] direction. The SAED and HRTEM show that the growth direction of the nanowire is mainly [010].

Our preparation $SrAl_2O_4$:Eu^{2+}, Dy^{3+} nanowires has overcome the insufficiency of literature [18–21]. The key technology of this experiment is a gas mixture (5% H_2, 95% Ar) and flow velocity. Under the condition of 1200 °C, Al will evaporate (its melting point is 660 °C), the residual oxygen in the furnace keeps Al surface generating Al_2O_3 membrane, it cannot evaporate and play a weaker catalytic role. The H_2 in the mixed gases restore the membrane into Al and Al nanoparticles plays a catalytic role, the two steps repeat alternately which is the forming conditions of nanowires, the ideal nanowires forming condition depends greatly on the length of preparing time. If the time is not long enough, due to the little $SrAl_2O_4$:Eu^{2+}, Dy^{3+} in the atmosphere, the nanowires would shape up in the form of bean sprout. Whereas the long preparing time would result to the instability of nanowires, at the same time, the nanowires would become longer horizontally and thicker.

The fluorescence spectrometer is used to test the resultants of three different deposition time and particles at room temperature. Comparing four corresponding emission spectrum and the afterglow curve in the same conditions, it can be seen, from Fig. 4(1), (2), that the emission peak is located at 517 nm (Block emission peak is 520 nm) with 365 nm light excitation, and the luminous intensity was down to 1/10 of the initial brightness in 20 mins. Performance of the sample with the synthesis time of 3 hours is best. Its light intensity compared with the powder's ($D_{50} = 750$ nm) increased by 13.0%, 4.8% higher at 20 mins. We believes the blue shifts may be associated with the quantum size effect of the nanowires, which increased the kinetic energy of the electrons and resulted in 4f to 5d level spacing increases with the decrease of particle size, and at the same time, the emission peak slight blue shifts [19].