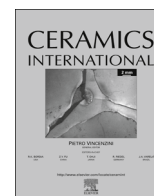




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Fabrication and properties of transparent Tb:YAG fluorescent ceramics with different doping concentrations



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ABSTRACT

Terbium doped yttrium aluminum garnet (Tb:YAG) transparent ceramics with different doping concentrations were fabricated by the solid-state reaction method using commercial Y_2O_3 , $\alpha-Al_2O_3$ and Tb_4O_7 powders as raw materials. Samples sintered at 1750 °C for 20 h were utilized to observe the optical transmittance, microstructure and fluorescence characteristics. It is found that all the Tb:YAG ceramics with different doping concentrations exhibit homogeneous structures with grain size distributions around 22–29 μm . For the 5 at% Tb:YAG transparent ceramics, the grain boundaries are clean with no secondary phases. The photoluminescence spectra show that Tb:YAG ceramics emit predominantly at 544 nm originated from the energy levels transition of $^5D_4 \rightarrow ^7F_5$ of Tb^{3+} ions, and the intensity of the emission peak reaches a maximum value when the Tb^{3+} concentration is 5 at%. The in-line transmittance of the 5 at% Tb:YAG ceramics is 73.4% at the wavelength of 544 nm, which needs to be further enhanced by optimizing the fabrication process. We think that Tb:YAG transparent ceramics may have potential applications in the high-power white LEDs.

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1. Introduction

Yttrium aluminum garnet ($Y_3Al_5O_{12}$) has been widely used as a host material in fluorescence applications and solid-state lasers owing to its stable physical and chemical properties [1–5]. Rare-earth-doped YAG materials are promising phosphor candidates in cathode-ray tubes (CRT) [6], field emission display (FED) [7], scintillation [8], vacuum fluorescent displays (VFD) [9] and white light emitting diodes (W-LED) [10–12]. The introduction of lanthanide activated phosphors into fluorescent lamps and emissive displays has greatly improved the light output and the color rendering during the last two decades.

The W-LED can be fabricated by employing blue, green, and red emitting multiphase phosphors excited by an ultraviolet InGaN chip [13]. Among the rare-earth ions, Tb^{3+} ion exhibits a strong green emission line attributed to $^5D_4 \rightarrow ^7F_5$ transition under the excitation of ultraviolet light [14,15]. Therefore, Tb^{3+} ion is often used in green phosphors for W-LED. The Tb-activated YAG

phosphor is insensitive to temperature variation and shows little tendency to saturate at high current excitations [16–19], so it has received widespread concern due to its excellent luminescent properties. However, phosphors have some disadvantages in packaging. For example, organic polymer used as a packaging material has poor heat dispersion so that it often brings about the problems of discolor and aging leading to the devices failure [20]. In addition, the refractive indexes between phosphors and encapsulation resin are different, which will cause light scattering and degenerate the luminous efficiency [21,22]. In order to overcome the above drawbacks, researchers have proposed counterpart fluorescent ceramics to replace the phosphors [23–26]. Compared with powder phosphors, transparent polycrystalline ceramics have the advantages of higher thermal conductivity, homogeneity, optical transparency, better chemical stability and mechanical properties [27–30].

In this work, transparent Tb:YAG fluorescent ceramics were fabricated by the solid-state reaction method using highly pure commercial Y_2O_3 , $\alpha-Al_2O_3$ and Tb_4O_7 powders as raw materials with tetraethyl orthosilicate (TEOS) and magnesium oxide (MgO) as sintering aids. The influences of Tb^{3+} concentration on optical transmittance, microstructure and fluorescence characteristics of Tb:YAG transparent ceramics were mainly investigated.

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2. Experimental procedures

Commercial powders of α - Al_2O_3 (99.99%, Fenghe Ceramics Co., Ltd., Shanghai, China), Y_2O_3 (99.999%, Yuelong New Materials Co., Ltd., Shanghai, China) and Tb_4O_7 (99.99%, Yuelong New Materials Co., Ltd., Shanghai, China) were used as raw materials to fabricate $(\text{Tb}_x\text{Y}_{1-x})_3\text{Al}_5\text{O}_{12}$ ($x=0, 0.01, 0.03, 0.05,$ and 0.1) ceramics. MgO powder (99.998%, Alfa Aesar, Tianjin, China) and tetraethoxysilane (TEOS, > 99.999%, Alfa Aesar, Tianjin, China) were used as sintering aids in all the samples. Then the powders were mixed in ethanol and ball-milled with high-purity corundum balls for 12 h. After ball milling, the slurry was dried at 70°C for 2 h in an oven and then sieved through 200-mesh screen. The powder mixtures were calcined at 600°C for 4 h to remove the organic components. The calcined powders were uniaxially pressed into 20 mm diameter pellets at low pressure and then the green bodies were cold isostatically pressed at 250 MPa. The pellets were sintered at 1750°C for 20 h in a tungsten mesh-heated vacuum furnace under vacuum (10^{-3} Pa) during holding. The heating and the cooling rates were 2°C min^{-1} and $10^\circ\text{C min}^{-1}$, respectively. Finally, the ceramics were mirror-polished on both surfaces to 5 mm to test the in-line transmittance. The polished specimens were thermally etched at 1450°C for 3 h to expose the grain boundaries.

Micrographs of the starting powders and the powder mixture were observed with a field emission scanning electron microscopy (FESEM, S-4800, Hitachi, Japan). The specific surface areas of the powders were measured using a gas sorption analyzer (Model Micromeritics ASAP 2010, Norcross, USA) based on the Brunauer-Emmett-Teller (BET) method. Phase compositions of the specimens were identified by X-ray diffraction (XRD, Model D/max2200PC, Rigaku, Japan). XRD analysis was performed on a

Huber Imaging Plate Guinier Camera G670 (Cu $\text{K}\alpha_1$ radiation, $k=1.54056 \text{ \AA}$, 40 kV/30 mA, Ge monochromator). The 2θ for all data ranged from 10° to 80° with 0.02° step size. The in-line transmittance was measured with a UV-VIS-NIR spectrophotometer (Model Cary-5000, Varian, USA). Microstructures of the thermally etched surfaces of the ceramics were observed by a field emission scanning electron microscopy (FESEM, SU8220, Hitachi, Japan). Grain sizes of the sintered samples were measured by the linear intercept method (more than 400 grains counted) and the average grain size was calculated by multiplying the average linear intercept distance by 1.56 [31]. The fluorescence spectrum and fluorescence lifetime were measured at room temperature using an FLS920 Edinburgh Analytical Instruments Fluorescence Spectroscopy LifeSpec PS Spectrophotometer with Xenon lamp as a light source.

3. Results and discussion

Fig. 1 shows FESEM micrographs of the starting powders and the powder mixture after ball milling. The average particle sizes of Y_2O_3 , α - Al_2O_3 and Tb_4O_7 powders are about $3 \mu\text{m}$, $0.3 \mu\text{m}$ and $2 \mu\text{m}$, respectively. The specific surface areas (S_{BET}) of the starting powders (Y_2O_3 , α - Al_2O_3 , Tb_4O_7) and the powder mixture after ball milling are 3.12, 8.59, 2.87 and $7.65 \text{ m}^2/\text{g}$, respectively. It can be seen that there are some aggregations in the Y_2O_3 and Tb_4O_7 powders. The aggregated Y_2O_3 particles contain many primary particles with the diameter of about 100 nm, as is shown in the inset of Fig. 1(a). However, they can be readily ground to finer particles during ball milling, which is confirmed in Fig. 1(d).

The XRD patterns of the ceramics with different Tb^{3+}

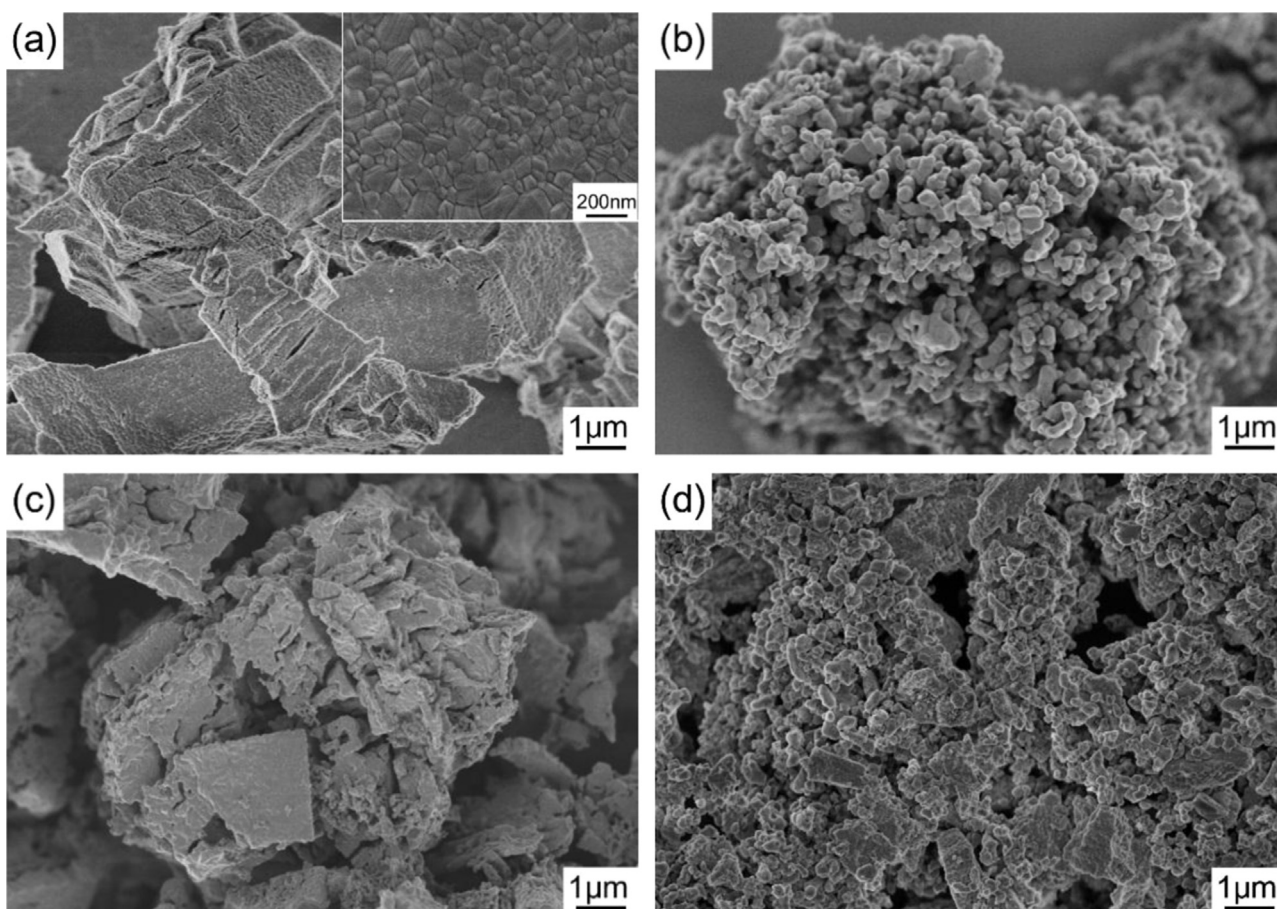


Fig. 1. FESEM micrographs of the starting powders (a) Y_2O_3 , (b) α - Al_2O_3 , (c) Tb_4O_7 and (d) powder mixture after ball milling.

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