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Fabrication and luminescent properties of highly transparent Er₃Al₅O₁₂ ceramics

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ABSTRACT

Highly transparent $\rm Er_3Al_5O_{12}$ (ErAG) ceramic was fabricated by a solid-state reactive sintering method under vacuum. The optical property, microstructure and up-conversion luminescence of the ErAG ceramic were investigated. For the 3 mm thick sample, the in-line transmittance at the wavelength of 3000 nm and 425 nm were about 84% and 81%, respectively, which was very close to the theoretical transmittance of the $\rm Er_3Al_5O_{12}$ single crystal. Micrograph of the ErAG transparent ceramic exhibited a pore-free structure and the density of the ceramic was measured to be 6.38 g/cm³. Average grain size of the ceramic was about 9 μ m. When pumped by a 980 nm laser diodes (LD), strong green and red emission in the ErAG ceramic was observed from the photoluminescence (PL) spectrum. The luminescent properties of the ceramic under the excitation of LD with various pumping power were investigated.

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1. Introduction

Rare earths (REs) doped materials have been attracting increasing attentions in recent years due to that the REs possess abundant energy levels and luxuriant spectral properties [1–3], which can be applied in many fields, such as solid-state laser, thermal neutron dosimetry and solid-state lighting [4–6]. Among the REs, ${\rm Er}^{3+}$ was extensively studied, especially in the application of up-conversion luminescent materials because ${\rm Er}^{3+}$ ions are sensitive to the infrared light, and can efficiently transfer the infrared light to visible and violet light [7,8]. Therefore, the ${\rm Er}^{3+}$ -doped materials are generally used to improve the utilization of infrared light.

As is well acknowledged, for luminescent applications, host

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materials with low cut-off phonon energy and wide band gap are expected [9,10]. Especially, for laser or high power lighting applications, the phosphors should be provided with high thermal conductivity, as well as excellent thermal and chemical stability [11,12]. Transparent ceramics with garnet structure have been attracting great attentions not only due to that the garnet ceramics meet the requirement of physical and chemical stability, but also because of their excellent optical qualities [13]. Among which, Er³⁺doped YAG transparent ceramics have been widely studied [14–17]. Different concentrations of Er³⁺ ions were incorporated into the YAG matrix to study the effect of doping concentration on the upconversion luminescent properties. They found that the ratios of green and red emission intensities varied with the Er³⁺ concentrations from 0 to 90% [17,18]. However, the transparent ErAG ceramics and their luminescent properties have been paid few attentions previously. In the present research work, a highly transparent ErAG ceramic was fabricated. The optical, microstructure and luminescent properties were investigated.

2. Experimental procedures

The starting materials were high-purity powders of Er₂O₃

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(99.99%, Rare-chem. Hi-Tech. Co., Ltd., 99.99%, Guangdong, China), and Al₂O₃ (99.99%, Aladdin Industrial Inc., Shanghai, China). They were weighed out in stoichiometric proportions with the ratio of Er:Al at 3:5. TEOS (>99.9%) and MgO (>99.99%) were added as sintering aids at the level of 0.5 wt% and 0.05 wt%, respectively. The powders were mixed in anhydrous alcohol for 12 h by ball milling to make the ceramic slurry. After that, the slurry was dried and sieved through 200-mesh screen. The as prepared powders were then uniaxially pressed to pellets (Φ 20 mm) at ~15 MPa followed by a cold-isostatically press at ~200 MPa. The compacted pellets were sintered in a tungsten mesh-heated furnace at 1780 °C for 12 h under a vacuum of around 10^{-3} Pa, and then annealed at 1400 °C for 10 h in air to remove oxygen vacancy. Finally, the specimens were polished on both surfaces to a thickness of 3 mm for optical and luminescence property characterization.

Phase of the ceramic sample was identified by X-ray diffraction (XRD) using a Japan Rigaku D/MAX 2200 PC diffractometer with Cu K α radiation. Density was measured by the Archimedes method. The in-line transmittances were measured on a UV-VIS spectrometer (Carry 5000 Spectrophotometer, Varian, U.S.A.). The polished and then thermal-etched surface of the ceramic was observed using a scanning electron microscopy (SEM, JSM-6390, JEOL, Japan). The sample was excited with a 980 nm laser diode, and the upconversion property was characterized using the Fluorolog-3 spectrometer (Jobin Yvon, France).

3. Results and discussion

XRD pattern of the ceramic sample sintered at 1780 °C for 12 h is shown in Fig. 1. All of the observed peaks match well with the characteristic of ErAG phase (JCPDS: 78–1451), and no other impurity phases are detected, indicating that single-phased ceramic was obtained, which will probably provide the ceramic with good optical quality and excellent luminescence properties.

For the sample with 3 mm in thickness, the in-line transmittance was shown in Fig. 2. Although compared with single crystals, polycrystalline ceramics usually have more complicated microstructures containing numerous grains and grain boundaries, which might greatly degrade the optical properties of the ceramic, the sample fabricated presently shows excellent optical quality. The transmittances at wavelengths of 3000 nm and 425 nm were about 84% and 81%, respectively. The results were very close to that of the transparent Er³⁺-doped YAG ceramics with a relative low doping

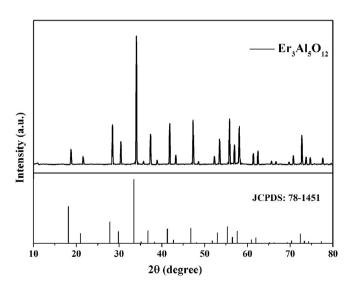


Fig. 1. X-ray diffraction pattern of the transparent ErAG ceramic.

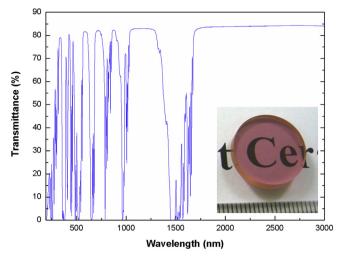


Fig. 2. In-line transmittance of the ErAG ceramic with 3 mm thickness. The inset shows the photograph of the ErAG ceramic sample.

content in our previous work [19]. Meanwhile, from the inset in Fig. 2, one can see that words can be clearly read through the asfabricated ceramic, showing uniformity of optical quality of the sample.

The good optical quality benefits from the property of the ceramic's microstructure. It can be seen in Fig. 3 that the average grain size of the ErAG ceramic is about 9 μ m, and there are no pores or impurities in the grains or on the grain boundaries. The density of the sample was 6.38 g/cm³, calculated by the Archimedes method. These results indicate that a dense ceramic was obtained, which is beneficial to the luminescence of the ceramic.

From the transmittance spectrum in Fig. 2, it shows strong absorption at wavelength of about 980 nm, which is due to the $^4I_{15/2}$ \rightarrow $^4I_{11/2}$ transition of Er³+. A 980 nm LD was used to pump the ceramic, strong visible light emission was intuitively observed, photograph of the excited ErAG ceramic sample was displayed in the inset of Fig. 4. The corresponding room temperature emission spectrum was recorded from 500 to 700 nm, also shown in the inset of Fig. 4. Green (centered at 560 nm) and red (centered at 680 nm) emission bands are observed, which originate from the radiative transition of $^2H_{11/2}/^4S_{3/2}$ \rightarrow $^4I_{15/2}$ and $^4F_{9/2}$ \rightarrow $^4I_{15/2}$, respectively. Zhou et al. [18] has studied the luminescence properties of high Er

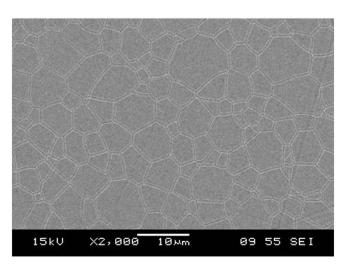


Fig. 3. SEM photographs of the polished and thermal etched surface of the transparent ErAG ceramic.

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