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Yb:Y_2O_3 transparent ceramics processed with hot isostatic pressing

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ABSTRACT

Highly transparent 5 at.% Yb:Y_2O_3 ceramics were fabricated by using a combination method of vacuum sintering and hot isostatic pressing (HIP). Co-precipitated Yb:Y_2O_3 powders, with 1 at.% ZrO_2 as the sintering aid, were used as the starting material. The Yb:Y_2O_3 ceramics, vacuum sintered at 1700 °C for 2 h and HIPed at 1775 °C for 4 h, exhibited small grain size of 1.9 μm and highly dense microstructure. In-line optical transmittance of the ceramics reached 83.4% and 78.9% at 2000 and 600 nm, respectively. As the ceramic slab was pumped by a fiber-coupled laser diode at about 940 nm, a maximum output power of 0.77 W at 1076 nm was achieved, with a corresponding slope efficiency of 10.6%.

1. Introduction

Since the first CW laser oscillation was achieved by using a Nd^{3+}-doped yttrium aluminium garnet (Nd:YAG) ceramic in 1995 [1], transparent ceramics acting as solid state laser gain medium have been widely studied. With the development of ceramic processing, it is possible to fabricate ceramics with high transparency. Transparent ceramics are very promising host materials for high power solid state laser applications, due to their better mechanical properties as compared with glasses and single crystals. For example, by using high quality Nd:YAG ceramics, solid state lasers with an output power of >100 kW have been achieved [2]. In recent years, much attention has also been paid to the fabrication of sesquioxides transparent ceramics, such as Y_2O_3, Sc_2O_3, and Lu_2O_3, because they are considered to be better laser host materials as compared with the garnet ceramics for the high power applications, due to their higher thermal conductivity and lower thermal expansion [3,4] Ytterbium doped yttria (Yb:Y_2O_3) transparent ceramics have attracted great attention in recent years, due to their relatively long fluorescence lifetime, high quantum efficiency and broad absorption and emission band [5,6].

In order to obtain transparent ceramics suitable for high power laser application, microstructure without residual pores must be achieved. Meanwhile, smaller grain size is more favorable in order to ensure higher thermal shock resistance and higher mechanical strength [7]. Hot isostatic press (HIP) sintering has been verified to be an effective sintering technique to achieve full densification of transparent ceramics without much grain growth, as the applied high pressure during the final-stage sintering can provide strong driving force for densification [8]. Accordingly, the sintering temperature can be reduced, so that the grain growth can be restricted.

In the present study, Yb:Y_2O_3 transparent ceramics, with 1 at.% ZrO_2 as the sintering aid, were fabricated by using vacuum sintering followed by HIP. Chemical co-precipitated powders were used as the starting material. Microstructure, optical property, thermal conductivity and laser performance of the HIPed Yb:Y_2O_3 ceramics were investigated.

2. Experiments

2.1. Ceramic fabrication

First, yttrium and ytterbium nitrate solutions were prepared by dissolving the corresponding oxide (purity > 99.99%) in hot nitric acid solution. Then, the yttrium nitrate, ytterbium nitrate and zirconium oxychloride octahydrate solutions were stoichiometrically mixed according to the composition of $(Y_{0.94}Yb_{0.05}Zr_{0.01})_2O_3$. The

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mother solution was diluted by using de-ionized water until the concentration of Y^{3+} reached 0.5 mol/L. The mixture of NH_4OH and NH_4HCO_3, with molar ratio 1:0.9, was used as the precipitant. Yb:Y_2O_3 precursor was produced by dripping the precipitant solution into the mother solution at a rate of 6 ml/min under mild stirring at room temperature until the PH value reached 8–9. The resultant precursor, after being aged for 1 day, was filtered by using a suction filter and then washed six times with de-ionized water and two times with alcohol. The wet precursor was completely dried at 75 °C for 48 h and then calcined at 1300 °C for 5 h. The calcined powders were first uniaxially pressed into pellets of 22 mm in diameter at 15 MPa and the pellets were cold-isostatic pressed at a pressure of 200 MPa. After that, the pellets were vacuum sintered at 1700 °C for 2 h at vacuum of $\leq 10^{-3}$ Pa, followed by hot isostatic pressing (HIPing) at 1700–1775 °C for 4 h at 198 MPa in argon (Ar). Finally, the sintered samples were annealed at 1400 °C for 10 h in air to remove the color centers.

2.2. Characterization

Morphologies of the precursors and calcined powders were observed by using a Leo 1550 field emission scanning electron microscope (SEM, Leo 1550, Cambridge, Cambridgeshire, UK). In-line optical transmittance of the ceramics was measured by using a UV-VIS-NIR spectrometer (Carry 5000, Agilent, Santa Clara, CA, USA). Relative density of the ceramics was determined using the Archimedes method. Microstructure of ceramics was examined by using a scanning electron microscope (SEM, JSM-6360A, JEOL, Tokyo, Japan). Average grain size of the ceramics was estimated by using the line intercept method [9], with over 300 grains for each sample. Thermal conductivity was measured by using a physical property measurement system (Quantum Design, USA).

3. Results and discussion

Fig. 1 shows SEM images of the precursor and the calcined powders. Both the precursor and the calcined powder showed good dispersity. Primary particle size of the precursor was estimated to be below 60 nm, while that of the 1300 °C calcined powders was about 170 nm.

Fig. 2 shows SEM images of the 5 at.% Yb:Y_2O_3 ceramics after vacuum sintering and HIPing. After vacuum sintering at 1700 °C for 2 h, relative density of the sample reached 97.5% without the presence of any open pores. Average grain size of the ceramic was 700 nm, with a narrow grain size distribution. After HIPing at 1700 °C for 4 h, porosity of the ceramic was decreased drastically, with an average grain size of only 1.1 μm, as shown in Fig. 2(b). However, there were still a small number of grain boundary pores, as highlighted by the red circles in Fig. 2(b). When the HIPing temperature was increased to 1740 °C and above, Yb:Y_2O_3 ceramics with fully dense microstructure could be obtained (Fig. 2(c) and (d)). Average grain size of the 1775 °C HIPed sample was increased to 1.9 μm, with no residual pores observed in the SEM image.

Fig. 3 illustrates in-line optical transmittance and photographs of the HIPed Yb:Y_2O_3 transparent ceramics. The absorption peak located at between 800 and 1100 nm is attributed to the $^2F_{7/2}$-$^2F_{5/2}$ transition. The as-vacuum-sintered sample (Sample A) was totally opaque, because there were still many residual grain boundary pores inside the sample. However, it can be seen that transparency of the ceramics was improved significantly after the HIPing process. As the HIPing temperature was increased from 1700 to 1775 °C, the transmittance of the ceramics was improved gradually. The enhancement in the transmittance should be attributed to the elimination of the residual pores. So far, the best HIPing temperature was 1775 °C, leading to the ceramic with in-line optical transmittances of 83.4% and 78.9% at 2000 and 600 nm, respectively.

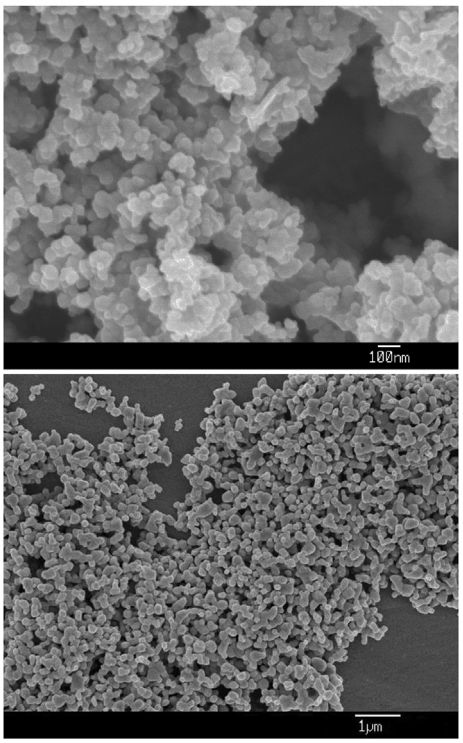

Fig. 1. FESEM images of the precursor (a) and the 1300 °C calcined powders (b).

Thermal conductivities of the HIPed Yb:Y_2O_3 ceramics are listed in Table 1. For comparison, the values of Yb:Y_2O_3 transparent ceramics with the same atom percentage of Yb^{3+} (5 at.%) reported in the open literature are also included. Because the thermal conductivity is usually decreased with the presence of foreign ions [10,11], it is not surprising that the room temperature thermal conductivity of our 5 at.%Yb:Y_2O_3 ceramics (8.84 W/m·K) was much lower than that of pure Y_2O_3 ceramics (13 W/m K) [11].

Fig. 4 shows a setup of the Yb:Y_2O_3 ceramic laser experiment. The pump source used in our experiment is a fiber-coupled laser diode with maximum power of about 25 W at the wavelength of about 940 nm. Collimated by a convex lens (F1) with focus length of 35 mm, the pump beam was then focused into the Yb:Y_2O_3 ceramics with a spot radius of about 140 μm by a spherical convex lens (F2) with focus length of 100 mm. In the experiment, a polished and uncoated Yb:Y_2O_3 ceramic slab was used as the laser gain medium. It was cut into a size of 3.18 mm in length and 4 mm × 4 mm in cross-sectional dimension. To remove the generated heat, the ceramic was wrapped with indium foil and tightly mounted in a copper block whose temperature was water cooled to 18.0 °C. A simple two-mirror cavity with total cavity length of about 12 mm was employed in the experiment. The input plano–plano mirror M1 was dichroic coated with high reflectivity (R>99.7%) for 1000–1100 nm and high transmission for pump wavelength. The M2 mirror was a plano–plano output couple. Two different output couples (M2) with transmission of 5% and 7.5% were utilized in the experiment to investigate the output performances of the laser.

In the experiment, the laser output power was measured with a laser power/energy meter (NOVAII, OPHIR). The CW laser output power as a function of the absorbed pump power with different

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