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Densification and microstructural evolution of yttria transparent ceramics: The effect of ball milling conditions

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Abstract

Ho:Y_2O_3 ceramics were prepared by using solid-state reaction method with commercial oxide powders. The effect of ball milling condition, specifically slurry concentration (C_S), on densification, microstructure evolution and transmittance of the Ho:Y_2O_3 ceramics was studied. Ho:Y_2O_3 powders were ball milled with slurry concentrations of 33.2–17.4 vol%. The samples were vacuum sintered between 1400 and 1850 °C. Over the C_S range of 33.2–18.4 vol%, decreasing slurry concentration improved the densification, normal grain growth and optical transmittance of the Ho:Y_2O_3 ceramics. The 18.4 vol% Ho:Y_2O_3 powders showed the lowest agglomeration and highest densification rate, from which the ceramics prepared have the best transmittance. The grain growth exponents of the Ho:Y_2O_3 ceramics during final-stage of sintering were also studied.
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1. Introduction

Over the last 20 years, holmium-doped solid-state lasers have been proven as an excellent two micro eye-safe light sources, for medical applications, remote sensing of atmospheric CO_2 and H_2O, laser ranging and time-resolved spectroscopy.[1–3] Since Ikesue et al.[4,5] successfully fabricated transparent neodymium-doped yttrium aluminum garnet (Nd:YAG) ceramics and first achieved their laser oscillation in 1995, YAG ceramics have become one of the important laser host materials. Nowadays, it is not unrealistic to fabricate highly transparent YAG-based ceramics that have optical transmittance comparable to YAG single crystals. Moreover, ceramics have advantages such as high yield, short fabrication period and ease of making large sizes.[3,6] Recent studies indicated that transparent Y_2O_3 ceramics possess higher thermal conductivity and lower thermal expansion than YAG, and thus are considered as a better host material for solid-state lasers. [2,7]

In order to fabricate Y_2O_3 transparent ceramics suitable for laser applications, their density should be very close to 100%. It is well known that porosity is the most common residual defect in transparent ceramics.[8,9] It can lead to severe degradation of optical quality, because of the huge difference in refractive index between air and the host materials. Many efforts have been made during the past 40 years to eliminate pores in Y_2O_3 ceramics. One effective method is to dope Y_2O_3 with sintering aids, such as La_2O_3,[10] HfO_2,[11] ThO_2[12] and ZrO_2[13], to speed up densification and control grain growth. It is also feasible to decrease porosity and improve optical quality by using hot isostatic pressing (HIP). High quality Er:Y_2O_3 and Nd:YAG ceramics for solid-state lasers have been fabricated successfully by using HIP.[7,11] No matter what fabrication methods are adopted, there is a consensus among researchers that to completely remove porosity, highly dispersive and fine raw powders should be available, mostly by using either ball milling[4,14] or chemical co-precipitation methods.[15–20] According to Ciftcioglu,[21] even a small amount of agglomeration of powders would produce coarse pores that were difficult to remove during the sintering. In

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1988, Sordelet et al.[22] compared the sintering behaviors of well dispersed monosized Y_2O_3 powder and agglomerated Y_2O_3 raw powder. They concluded that densification rate of the former was higher than that of the latter. Gong et al.[23] also demonstrated that the agglomeration degree of co-precipitated YAG powders decreased significantly with the assistance of freeze-drying process, as compared with those dried in the oven. The subsequent YAG ceramics from the freeze-dried powder showed much less porosity, larger grain size and better transmittance. All these studies indicated that fabrication of transparent ceramics is very sensitive to the quality of raw powders, especially the degree of agglomeration.

Solid-state reaction is the most widely used route to fabricate transparent ceramics, due to its short fabrication time and feasibility of mass production. However, commercial Y_2O_3 raw powders have large particle size and severe agglomeration, which are hard to be fully sintered. Therefore, an effective ball milling process should be conducted to break the particles and the agglomeration. It has been proved that ball milling parameters, such as size and density of milling balls,[24] ball and powder filling, slurry concentration[25] and viscosity of liquid medium,[26] greatly affect ball milling efficiency. Under an appropriate ball milling condition, fine powders with relatively low degree of agglomeration and good sinterability can be produced. For example, Zou et al. demonstrated that an effective ball milling process could help reduce the agglomeration of Lu_2O_3 particles. By using such powders, they succeeded in fabricating Lu_2O_3 transparent ceramics with excellent transmittance (70% at 500 nm wavelength).[14] However, up to now, there have been few reports discussing how specific ball milling parameters would affect densification, grain growth and transmittance of transparent ceramics.

In this work, we report the fabrication of Ho:Y_2O_3 transparent ceramics by using solid-state reaction process with commercial Y_2O_3 raw powders. We found that slurry concentration has a strong effect on morphology of the powders, and densification, grain growth and transmittance of the final Ho:Y_2O_3 ceramics. As far as we know, this phenomenon has not been discovered and discussed yet, as it is easy to ignore the importance of slurry concentration during ball milling.

2. Experimental procedures

2.1. Ceramic fabrication

Commercial Y_2O_3 (purity >99.99%), and Ho_2O_3 (purity >99.99%) powders were mixed, according to the stoichiometric composition $(Ho_{0.005}Y_{0.995})_2O_3$ with 3 wt% ZrO_2 (purity >99%) as sintering additive. The mixture of Y_2O_3, Ho_2O_3 and ZrO_2 powders was then ball milled by using a planetary milling machine (PM400; Retsch, Haan, Germany) for 15 h at a speed of 140 rpm (rotation per minute), with agate balls (5 mm in diameter) and ethanol (99.99% purity; Merck, Darmstadt, Germany) as ball milling media. Powder and ball filling was fixed (the weight ratio of powder:balls was 1:2). The solid content (C_S) of the slurry (slurry concentration) was adjusted between 33.2 and 17.4 vol% (C_S = 33.2, 23.7, 20.7, 19.5, 18.4 and 17.4 vol%) by controlling the amount of ethanol. The slurry was then dried at 80 °C for 24 h in an oven and sieved through a 140-mesh screen. After removing organic components by calcining at 800 °C for 3 h, the powders were uniaxially pressed into 15 mm diameter pellets at 15 MPa. Then the green body pellets were further cold isostatically pressed (CIPed) at 200 MPa. After CIP, the green bodies were sintered using a vacuum furnace under vacuum ($P \leq 10^{-3}$ Pa) for various times at temperatures ranging between 1400 and 1850 °C.

2.2. Characterization of the powders and sintered samples

Morphologies of the raw powders and ball milled powders were observed by using a Leo 1550 field emission scanning electron microscope (SEM, Leo 1550, Cambridge, Cambridgeshire, UK). Ball milled slurry viscosity was examined using a Haake Viscometer (model: VT550). The Brunauer–Emmett–Teller (BET) surface area of the powders was determined by using a Micromeritics Tristar II-3020 nitrogen adsorption apparatus. Particle size analysis was carried out on a Particle Size Analyzer (Zeta Plus, Brookhaven). After the vacuum-sintering process, the ceramics were annealed in air at 1400 °C for 10 h (Nabertherm LHT 04/17) to eliminate the oxygen vacancies. Then they were fine polished on both surfaces by using a Hyprez precision lapping machine (Model: EJW400-IN-D, Engis, Japan). Bulk densities of the sintered samples were measured by using the Archimedes method. Room temperature transmittance spectra of the ceramics were recorded with a UV–VIS–NIR spectrometer (Carry 5000 Spectrophotometer, Varian, USA). Before microstructural observations, the ceramics were thermally etched in air for 1 h at temperatures lower than sintering temperatures by 100 °C. A scanning electron microscope (SEM, JSM-6360A, JEOL, Tokyo, Japan) was then used to exam the sample's surface microstructure. Average grain size of the sintered samples was calculated by the following equation[27]:

$$G = 1.56L, \quad (1)$$

where, L is the average linear intercept distance (200 grains counted).

3. Results and discussion

3.1. Morphology of ball milled powders

Figs. 1(a) and (b) shows FESEM images of the Y_2O_3 raw powder. It can be seen that the commercial Y_2O_3 raw powders have particle size of 1–4 μm, which is consistent with the result measured by using the Particle Size Analyzer (Fig. 1(c), average 2.3 μm). Fig. 1(b) exhibits a high magnification image of the raw powder. It shows that the particles are strongly agglomerated, as it is difficult to distinguish individual particle unit from the large agglomerates. The corresponding BET surface area (S_{BET}) of the Y_2O_3 raw powders is 2.4 m^2/g.

Fig. 2(a) and (b) exhibits FESEM images of the Ho_2O_3 and ZrO_2 raw powders. It is clearly that the Ho_2O_3 powders are also

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