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Synthesis of nanocrystalline yttria powder and fabrication of Cr,Nd: YAG transparent ceramics

Jiang Li*, Wenbin Liu, Benxue Jiang, Jun Zhou, Wenxin Zhang, Liang Wang, Yiqiang Shen, Yubai Pan, Jingkun Guo

Key Laboratory of Transparent Opto-functional Inorganic Materials, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200050, PR China

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ABSTRACT

Nanocrystalline yttria powders were synthesized by a precipitation method from yttrium nitrate solution using ammonia water as a precipitant. It was found that the addition of small amount of ammonia sulfate in yttrium nitrate solution can reduce the agglomeration of the produced yttria powders. Y_2O_3 powder with an average particle size of 67 nm was obtained by calcining the precursor at 1100 °C for 2 h. Transparent 0.1 at.%Cr,1.0 at.%Nd:YAG ceramics were fabricated by a solid-state reaction and vacuum sintering with CaO as a charge compensator and tetraethyl orthosilicate (TEOS) as a sintering aid using the prepared Y_2O_3 , and the high-purity commercial powders of α -Al₂O₃, Nd₂O₃ and Cr₂O₃ as raw materials. Fully dense Cr,Nd:YAG ceramic with some secondary phase of alumina was obtained by sintering at 1750 °C for 10 h. The average grain size of the sample was about 10 µm and the in-line transmittance was ~52% at 1064 nm, which is lower than the transmission (~82%) of the sample from all-commercial oxide powders. The low transmittance of Cr,Nd:YAG ceramic is mainly caused by the formation of secondary phase due to the non-stoichiometry of the starting powders. In order to overcome the current limitations of the study, weight loss cause by the decomposition of SO_4^{2-} ions and the absorption of water or organic materials in yttria nanopowder should be taken into account.

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1. Introduction

Highly transparent Nd:YAG polycrystalline ceramics have gained more and more attention as novel solid-state laser materials due to their several remarkable advantages, such as high doping concentration, easy fabrication of large scale, low cost, multilayer and multifunctional ceramics lasing components, and mass production, compared with Nd:YAG single crystals [1–8]. Laser-diode pumped passively *Q*-switched microchip solid-state lasers with high peak power have been shown to be useful sources for many applications, which has been demonstrated in Cr⁴⁺,Nd³⁺:YAG single crystals [9–11]. So by modern ceramic sintering technology, Cr⁴⁺,Nd³⁺:YAG transparent ceramics can be fabricated, which may be a more potential self-*Q*-switched laser material used for generating sub-nanosecond laser pulses relative to single crystals with the same composition.

Usually there are two typical methods to fabricate rare earth doped YAG (RE:YAG) transparent. One is solid-state reactive sintering of oxide powder mixture [12–22]. The other way is vacuum sintering of RE:YAG powders synthesize by wet-chemical method

[23–29]. In our previous work, transparent Cr⁴⁺,Nd³⁺:YAG ceramics were successfully fabricated by solid-state reaction and vacuum sintering [30–32]. The commercial α -Al₂O₃ powders were homogeneous and with average particle size of about 300 nm. The γ -Al₂O₃ powders used were even finer compared with α -Al₂O₃. However, the yttria powders with large agglomerates were of micrometer-size, which may inevitably affect the sinterability and the microstructure of the sample. In the present work, yttria nanopowders with homogeneous particle size were prepared by a wet chemical method, which was similar with the way reported by Wen et al. [33] By using the synthesized yttria powders and the commercial α -Al₂O₃, Cr₂O₃ and Nd₂O₃ powders as raw materials, fabrication of transparent Cr⁴⁺,Nd³⁺:YAG ceramics by solid-state reaction and vacuum sintering was investigated. For comparison, Cr⁴⁺,Nd³⁺:YAG transparent ceramics have also been fabricated with the same process using large-scaled Y₂O₃ commercial powder and other same starting powders.

2. Experimental

Commercial yttria powder (Shanghai Yulong New Materials Co., Ltd., 99.99%) was dissolved in a high-purity nitric acid (Shanghai Lingfeng Chemical Reagent Co., Ltd., super purity), and then was diluted with deionized water to the concentration of 0.3 M. 2.0 M ammonia water (Shanghai Lingfeng Chemical Reagent Co., Ltd., reagent grade) was used as precipitant and 2.6 g ammonia sulfate was added to a 1250 ml yttrium nitrate solution. The precursor precipitate was synthesized by dripping the

^{*} Corresponding author. Tel.: +86 21 52412816; fax: +86 21 52413903. *E-mail address:* lijiang@mail.sic.ac.cn (J. Li).

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Fig. 1. Solid-state reactive fabrication process of Cr,Nd:YAG transparent ceramics.

ammonia water solution into the mother salt solution at a speed of 3 ml/min under mild agitation. The resultant suspension was aged for 2 h with mild agitation by a magnetic stirrer and the final pH value of the precipitate slurry was 8.0. Then the precipitate slurry was filtered using a suction filter, washed three times with deionized water, rinsed two times with the ethyl alcohol and dried at 90 °C for 24 h. The dried cake was crushed with a corundum pestle and mortar and calcined at different temperatures for 2 h to obtain nanocrystalline yttria powder.

The obtained nanocrystalline yttria powder (or commercial large-sized Y2O3 powder) and other commercial high-purity powders of α-Al₂O₃ (Shanghai Wusong Chemical Co., Ltd., 99.99%), Nd₂O₃ (Shanghai Yuelong New Materials Co., Ltd., 99.99%) and Cr₂O₃ (Sinopharm Chemical Reagent Co., Ltd., spectral purity) were used as starting materials to result in a chemical composition of 0.1 at.%Cr,1.0 at.%Nd:YAG. The commercial CaO (Sinopharm Chemical Reagent Co., Ltd., spectral purity) was used as a charge compensator, because Ca²⁺ can balance the charge when Cr³⁻ changes into Cr4+. The commercial tetraethyl orthosilicate (TEOS, Shanghai Lingfeng Chemical Reagent Co., Ltd., spectral purity) was used as a sintering aid. All the composites were mixed by ball-milling with high-purity (99.7%) alumina balls in anhydrous alcohol for 10 h. The mixtures were dried at 90 °C, sieved 200-mesh screen, dry-pressed under 100 MPa into ω 20 mm disks and finally cold isostatically pressed under 250 MPa. The compacted disks were sintered at the temperature range of 1600-1780 °C for 10 h in a tungsten mesh-heated vacuum furnace (KZG-110F, Shanghai Chenrong Electrical Furnace Co., Ltd., Shanghai, China) under $3\times 10^{-3}\,\text{Pa}$ vacuum during holding. The specimens were placed in a molybdenum crucible. The heating rate was 5 °C/min and the cooling rate was 10 °C/min. The sintered specimens were annealed at 1450 °C for 20 h in air. The solid-state reactive fabrication process of Cr,Nd:YAG transparent ceramics is schematically illustrated in Fig. 1, which is similar with that reported before [34–37]. For cell parameter comparison, 0.1 at.%Cr:YAG and 1.0 at.%Nd:YAG transparent ceramics were fabricated by sintering at 1750 °C for 10 h.

TG–DTA analysis was recorded on a Netzsch STA 449C instrument. Measurements were taken under a continuous flow of air (20 ml min⁻¹). Samples were heated at $10 \,^{\circ}$ C min⁻¹ to $1200 \,^{\circ}$ C and then cooled to ambient in air.

Phase identification of powders was performed by a Rigaku D/max2200PC X-ray diffractometer (XRD) using nickel filtered CuK_{α} radiation (1.5406 Å) in the range of 2θ = 10–80°. The tube current and voltage were 40 mA and 40 kV, respectively. The scanning speed was 4° min⁻¹ and the step size was 0.02. The XRD data of powders are refined by Rietveld method using the Maud program (Version 2.30).

The XRD data of ceramics for phase identification were collected at ambient temperature with a HUBER Imaging Plate Guinier Camera G670 [S] (Cu K_{\alpha1} radiation, $\lambda = 1.54056$ Å, 40 kV/30 mA, Ge monochromator). The 2θ for all data ranged from 10° to 80° with 0.005° step size. The acquired data are refined by Rietveld method using the Maud program (Version 2.30).

FTIR of the as-prepared precursor and the calcined powders were measured on a Nicolet NEXUS 7000C spectrophotometer in the 400–4000 cm⁻¹ range using the KBr pellet (~1 wt% sample) method. Each analysis consisted of a minimum 32 scans and the resolution was ± 2 cm⁻¹.

Microstructures of the powders were observed on a JEOL JEM 2100F FETEM instrument. Samples were prepared using a carbon-coated copper grid (150 meshes). Powders were dispersed in ethanol using an ultrasonic horn, and then a drop of the dispersed powder/ethanol mixture was deposited on the grid. The grid was then dried in air. The FETEM was used with an accelerating voltage of 200 kV. The particle size from FETEM micrograph is the average value by statistic method.

Specific surface area analyses were conducted at 77 K using a Norcross ASAP 2010 micromeritics, with N₂ as the absorbate gas. Samples were degassed at 150 °C until the air pressure was below 5 μ m Hg. The specific surface areas were calculated using the BET multipoint method with 8 data points. The average particle size of the calcined powders is calculated from specific surface area.

Densities of the sintered specimens were measured by the Archimedes method, using deionized water as the immersion medium. Microstructures of the fractured surfaces and the natural surfaces were observed by EPMA (Model JXA-8100, JEOL, Japan). Mirror-polished samples (1 mm thick) on both surfaces were used to measure the in-line transmittance (Model U-2800 Spectrophotometer, Hitachi, Japan).

3. Results and discussion

Fig. 2 shows the TG/DTA curves of the synthesized precursor. The TG curve showed that complete thermal decomposition of the precursor into oxides was achieved at about 1100 °C with a total mass loss of 35.6% which is higher than the value (19.3%) expected for a precursor of pure hydroxide. Chemical analysis was not performed on the precursor. However, previous work [38,39] revealed that Y³⁺ usually precipitated as basic salt of approximate formula $Y_2(OH)_5 NO_3 \cdot nH_2 O$ instead of pure hydroxide when ammonia water was used as precipitant. In fact, the mass loss of the present precursor is very close to the theoretical value (34.1%) calculated for Y₂(OH)₅NO₃·H₂O. At the temperature around 111 °C, the endothermic peak with a mass loss of 5.8% appears which is associated to the vaporization of physically bound absorbed water and crystal water. At the temperatures around 310 °C and 543 °C, the two endothermic peaks with a total mass loss of 27.8% were caused by the decomposition of $Y_2(OH)_5NO_3$. The mass loss at the temperature of 1000–1100 °C is mainly due to desulfurization [40].



Fig. 2. TG–DTA curves showing the decomposition process of the synthesized precursor.

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