



Fabrication of transparent neodymium-doped yttrium aluminum garnet ceramics by high solid loading suspensions

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Abstract

Dense neodymium-doped yttrium aluminum garnet (Nd:YAG) transparent ceramic was obtained by slip casting and solid-state reaction. The colloidal behavior of the aqueous suspensions of neodymia, yttria, and alumina mixed powders using Displex A as dispersant was investigated. The variation in zeta potential due to pH alteration was studied. The isoelectric point (IEP) was at pH 4.5 and 4 for the specimens with and without Displex A, respectively. The optimal dispersion conditions were achieved for the suspensions at pH 9.6 with 0.4 wt% Displex A. The green body prepared by slip casting was vacuum sintered from 1200 °C to 1750 °C. The grain size of the sintered body increased, and the pore size decreased with increasing sintering temperature. Pore-free Nd:YAG transparent ceramic with a grain size of 5–10 μm was obtained by sintering at 1750 °C for 10 h. The in-line transmittance of the annealed specimen reached 80.8% at 1064 nm.

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1. Introduction

Since the first polycrystalline Nd:YAG ceramic laser pumped by the diode laser excitation system was first reported by Ikesue et al. [1,2], laser ceramics have attracted considerable attention because of their greatly improved optical quality, which have produced highly efficient laser oscillations [3–7]. Compared with single crystals, Nd:YAG laser ceramic has superior properties, such as the ease of fabrication into complicated structures and the possibility for mass production [8–10]. A high density and a uniform green body are key factors to obtaining high-quality Nd:YAG laser ceramics. Two previously reported methods are used to obtain Nd:YAG ceramic green bodies [11–13]. Dry pressing is the most common technique for creating transparent Nd:YAG ceramics. High-purity Y₂O₃, Al₂O₃, and Nd₂O₃ powders are blended, ball milled, and pressed under low pressure into required shapes, and then cold isostatically pressed under a relatively high pressure [14,15]. However, a pore-free ceramic is difficult to obtain with vacuum sintering using the press formation

method because some air is always trapped in the closed micro-cavities formed by the tightly compacted particles in the green body during high-pressure formation. Nanopowder technology combined with the slip casting method is a proven effective process in the fabrication of transparent YAG ceramics because defects such as aggregates and agglomerates can be more readily managed [16,17]. However, the stoichiometry ratio is difficult to control due to the complicated process of nanopowder fabrication.

The methods of Nd:YAG ceramic fabrication reported in most papers are dry pressing and solid-state reaction sintering [5,18–20]. However, only a few papers reported the slip casting of aqueous metal oxide mixtures to produce transparent Nd:YAG [21–23]. A key point in the successful use of slip casting is achieving a well-dispersed suspension with high solid loading and low viscosity [24]. Modifying the particle surface with surfactants is necessary to achieve adequate distance between the particles in ceramic suspensions [25,26]. Both poly(acrylic acid) and ammonium polyacrylate are proven efficient dispersants in the aqueous solution of Y₂O₃, Al₂O₃, and Nd₂O₃ powders. However, the reported rheology varied with the molecular weights of the dispersants used and the statuses of the oxide powders [21,22].

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In the present study, the aqueous slurry of commercial neodymia, yttria, and alumina mixed powders with commercial dispersant was prepared by ball milling. The effect of pH, dispersant, and solid loading on the properties of the slurry was investigated to determine a suitable stabilization method. The microstructure of the Nd:YAG ceramic prepared by slip casting with high solid loading and solid-state reaction sintering was investigated.

2. Materials and experimental procedures

High purity alumina powder (99.99%; Aladdin, China), yttria powder (99.99%; Aladdin, China), neodymia (99.95%; Aladdin, China), and Dispex A40 (40% in water; Ciba, UK) were used. The oxide powders were always weighed out in stoichiometric proportions at the (Y+Nd):Al ratio of 3:5. Neodymia was doped into the YAG at 1.0 at%. These powders were mixed in deionized water with different dispersant and solid loading concentrations. The prepared aqueous suspensions were ball milled in polyurethane jars filled with corundum milling balls for 24 h at a speed of 180 r/min.

The viscosity of the suspension with different dispersant contents was measured at room temperature in a small sample adapter at a shear rate of 13.2 s^{-1} using a Brookfield DV-II +Pro viscometer with spindle #18 (USA). The viscosity measurement of the aqueous suspensions with different solid loading and optimum dispersant concentrations was performed at shear rates ranging from 1.32 s^{-1} to 132 s^{-1} .

The zeta potentials of the diluted neodymia, yttria, and alumina mixed powder suspensions (0.01 wt%) with or without dispersant were evaluated using a JS94J potential analyzer (Powereach, China). A 0.001 M NaCl solution was prepared to maintain the ionic strength of the powder suspensions. The solution pH was adjusted by adding diluted NaOH and HCl aqueous solutions.

Solid loading suspension (70 wt%) with optimum dispersant content and 0.5 wt% tetraethoxysilane (TEOS, 99.99%; Alfa Aesar) was subsequently slip cast in plaster mold to form the green body. After the complete removal of organics by calcination at $900 \text{ }^\circ\text{C}$, the green body was sintered in a vacuum furnace from $1200 \text{ }^\circ\text{C}$ to $1750 \text{ }^\circ\text{C}$ under a $1.0 \times 10^{-3} \text{ Pa}$ vacuum.

The Fourier transform infrared (FTIR) spectra and molecular weight of Dispex A40 were obtained using a Nicolet 6700 FTIR spectrometer (Nicolet, USA) and an Agilent gel permeation chromatograph (GPC; Agilent, USA). Morphological observations were performed with an EVO 18 scanning electron microscope (SEM; Carl Zeiss, German). Transmission measurement on polished ceramic was performed using a UV-vis and FTIR spectrometer (Lambda 35; PerkinElmer, USA).

3. Results and discussion

Fig. 1 shows the SEM micrographs of the starting alumina, yttria, and neodymia powders and the mixed powder after ball milling for 24 h. The primary alumina particles with a mean diameter of $0.4 \text{ }\mu\text{m}$ were less agglomerated compared with the other powders. No clearly agglomerated particles were observed

in the SEM micrographs of the yttria, but some particles about $5 \text{ }\mu\text{m}$ in size were found. The neodymia powders mainly contained micrometer-sized aggregates of nanosized primary particles. The sizes of particles after ball milling decreased obviously. However, clear yttria particles with a diameter more than $1 \text{ }\mu\text{m}$ were observed.

Fig. 2 shows the molecular weight distribution of the Dispex A determined by GPC using deionized water as solvent. The polyelectrolyte was quite polydisperse with an average $M_w=7320$. Fig. 3 shows the FTIR spectra of the Dispex A aqueous solution. The two peaks caused by the hydrogen bonding of the O–H and N–H bonds in the molecule were overlapped with a wide band from 3600 cm^{-1} to 3000 cm^{-1} . The band at 1453 cm^{-1} is attributed to the overlapping of the —COO— stretching vibration with the C–C asymmetric deformation [27]. In addition, the bands at 1324 and 1043 cm^{-1} are attributed to the skeletal C–C vibrations, and the absorption band at about 1644 cm^{-1} is attributed to the bending vibration of N–H, thereby confirming the presence of NH_4^+ [28]. Thus, Dispex A was a polycarboxylic acid ammonium with an average molecular weight of 7320.

Obtaining homogeneously dispersed suspensions with high solid loading is important to prepare high-quality ceramic parts by slip casting. In ceramic suspensions, a close correlation exists between the rheological property and the suspension structure, which depends on the zeta potential of the powders [29]. The effect of pH on the zeta potential of the powder in the slurry is shown in Fig. 4. The addition of Dispex A produced a pronounced effect on the electrokinetic behaviors of the mixed powders. The isoelectric point (IEP) is the value at which the net surface charge of a particle is zero [30]. The IEP of the mixed powder suspensions changed from pH 4.5 to 4. The particle surface is positively charged at $\text{pH} < \text{IEP}$ and negatively charged at $\text{pH} > \text{IEP}$. The curve shapes of the zeta potentials for the powder with or without dispersant remained almost the same. The zeta potential absolute value increased with increasing pH at $\text{pH} > \text{IEP}$ because of the increasing double-layer thickness and the total surface charge of the particle. The particles with dispersant exhibited a higher negative zeta potential than that of the particles without dispersant at the same pH ranging from 4.5 to 10 because of the absorption of dispersant on the powder. In this case, thermodynamic theories explain adsorption as a process that lowers the free energy of the system [31]. The Dispex A molecules contain the functional group $-\text{COONH}_4$, which dissociated into $-\text{COO}^{-1}$ when Dispex A was dissolved in the aqueous solution. The fraction dissociated or the percentage of the number of $-\text{COO}^{-1}$ to the total number of functional groups is affected by the pH value of the solution. With increasing pH, the additional $[-\text{COO}^{-1}]$ resulted in well-dispersed, highly stable suspensions [32]. The absolute value of zeta potential of the powders with dispersant reached 61 at $\text{pH}=9.6$. The effect of the dispersant on the zeta potential was negative at $\text{pH} > 10$ because of the surface dissolution and hydrolysis of yttria.

Fig. 5 shows the relationship between the apparent viscosity of the alumina suspension at pH 9.6 and the additive concentration

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