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Dispersion of nano-sized yttria powder using triammonium citrate dispersant for the fabrication of transparent ceramics

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ABSTRACT

In the present work, transparent Y_2O_3 ceramics were prepared via colloidal processing method from nano-sized Y_2O_3 powders. The effects of triammonium citrate (TAC) on the colloid stability of aqueous suspensions of nano-sized Y_2O_3 powders were studied. The surface properties of yttria powders were notably affected by the addition of TAC dispersant. The adsorption of TAC on the particle surface shifts the IEP to lower pH values and increases the absolute zeta potential in alkaline region. Rheological characterization of the investigated system revealed an optimal dispersant concentration of 1 wt%, which correlated well with the saturation adsorption of TAC on Y_2O_3 powder surfaces. The suspensions with solid loadings up to 35 vol% were achieved with further addition of Tetramethylammonium hydroxide (TMAH) into the dispersing system. The consolidated green bodies were treated by cold isostatic pressing to further increase the green density. Transparent Y_2O_3 ceramics were prepared after vacuum-sintering at 1700 °C for 5 h. The transmittances of the sample were 74.5% at 800 nm and 79.8% at 2000 nm, respectively.

1. Introduction

Transparent yttria ceramics have attracted great research interest in the past decades, mainly because of its promise for a wide range of applications as optical materials, such as luminous pipes for high-intensity-discharge lamps, heat-resistive windows, missile domes, host materials for scintillators and solid lasers, etc [1–3]. To date, it has been recognized that fabrication of optical ceramics with high transmittance requires state of the art processing control over every key steps of ceramics production, namely synthesis of sinterable starting powders, consolidation of a uniform green compact with high packing density and sintering to ceramics with a nearly pore-free microstructure [4–6]. For consolidation, it has been well accepted that colloidal processing method offers significant advantages over dry pressing method in terms of improved microstructure uniformity, increased packing density, and geometry versatilities of the consolidated green compacts [7–9]. Due to the greater ease with which the particles can slide over one another and rearrange in the wet state, colloidal processing method offers the potential to obtain more uniform and higher density green compacts [10]. Besides, the colloidal processing method also provides a variety of possible geometries, such as curved windows and domes, which are difficult to form using dry-pressing method [11,12]. Thus, the employment of colloidal processing routes to fabricate Y_2O_3 transparent ceramics has been quite fascinating [13–16].

After fully survey in the published literature, however, it has been found that research work addressing the colloidal processing of Y_2O_3 ceramics had been quite limited in comparison with other ceramics of similarly scientific and industrial importance, e.g., Al_2O_3, $BaTiO_3$, etc [10,12]. Uckikoshi firstly made an attempt to use PEI as a dispersant to stabilize the nano-sized Y_2O_3 suspensions [16]. Takai et al. prepared high solid content yttria slurries for gel casting using relatively coarse powders with particle sizes in the order of ~1 μm for high temperature structural applications [17]. Mouzon produced the Y_2O_3 suspensions with 23 vol% solid concentration using yttria powder with specific surface area of 17.2 m^2/g [14]. Optimization of the rheological properties of yttria suspensions has been investigated by Jin et al. by using three different commercially available polyelectrolyte dispersants [15]. The authors claimed that Dolapix CE64 was the most effective dispersant compared with Dispex A40 and Darvan C-N. More recently, colloidal processing has been studied by Santos et al. for fabricating yttria nettings [18]. Suspensions with 30 vol% of nano-sized powders (d_{50}=131.8 nm) were prepared with the addition of 1 wt% PAA dispersant at pH 10.5. Nevertheless, although

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fabrication of transparent ceramics using colloidal processing method has been endeavored, the Y_2O_3 ceramics prepared were translucent [14]. A novel aqueous gelcasting processing route using a water-soluble copolymer as both dispersing and gelling agent has been proposed recently [19]. The cast bodies were sintered at 1850°C for 6 h in vacuum to get highly transparent Y_2O_3 ceramics.

In colloidal processing, the quality of the final products is highly dependent on the stability and rheological properties of the ceramic suspensions. The preparation of a concentrated, well-dispersed slurry is a critical step for achieving unifrom, dense, dimensionally accurate ceramic green parts [9]. In fact, compared with other ceramic powder systems which have already been intensively investigated, the dispersion of yttria powders could be more difficult due to the highly reactive nature of Y_2O_3 powders in aqueous medium, which leads to additional difficulties in preparing stable suspensions with high solid contents [20,21]. It is known that Y_2O_3 powders undergo a reaction with water and release trivalent yttrium cations and their hydroxy complexes into solution, which strongly affect the stability of the suspension [22]. The phenomena has been found both in single ytria powder system as well as the powder systems involving yttrium species [14–24].

For these highly reactive systems, finding a suitable dispersant is quite important for the suspension preparation [14,16]. The dispersion of fine Y_2O_3 powder in aqueous medium using TAC as the dispersant was studied in this work. TAC was selected based on two considerations. Firstly, as a low-molecular-weight dispersant, TAC has been successfully applied to disperse many ceramic powders in aqueous solutions; and it has been indicated that the dispersant with small molecules can move freely around nanoparticles, which increases the effectiveness of adsorption [25–30]. Secondly, recent works on dispersion of nano-sized ZrO_2 powders verified that concentrated suspensions made with TAC exhibited lower viscosities than those dispersant based on polyelectrolyte, providing up the potential for achieving much higher solid content suspensions after optimization [31]. The aim of the present work was to prepare stable Y_2O_3 aqueous suspensions for fabricating Y_2O_3 transparent ceramics. The first part identifies the optimum conditions for suspension stability. Several critical factors such as pH, concentration of dispersant, solid loading, and surface chemistry of powders were studied. The most promising suspensions were cast into green bodies that were subsequently sintered for the fabrication of transparent Y_2O_3 ceramics.

2. Experimental procedure

2.1. Starting materials

Commercially available Y_2O_3 powder (99.99% pure; Huizhou Ruier Rare Chemical Hi-Tech Co., Ltd, Huizhou, China) was used as the starting material [32]. The specific surface area of the powder, measured by N_2 adsorption (Model TriStar II 3020, Micrometritics Instrument Corp., Norcross, GA) is 13.02 m^2/g. The analysis of particle morphology (Fig. 1) carried out by transmission electron microscope shows that the average diameter of the powder is ~86 nm, in good agreement with the particle diameter derived from specific surface area measurement.

Triammonium citrate (TAC) (Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) was used as an anionic dispersant. Deionized water was used as the dispersing medium. The needed pH of suspension was adjusted through adding reagent-grade HCl, KOH or Tetramethylammonium hydroxide (TMAH) aqueous solution.

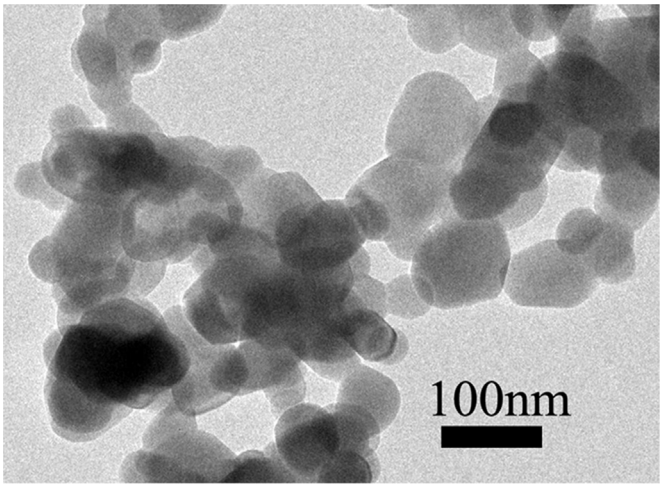

Fig. 1. TEM micrograph showing particle morphologies of the starting Y_2O_3 powder.

2.2. Suspension preparation

The suspensions used for all measurements were prepared by adding Y_2O_3 powder to deionized water with the desired TAC content. The added amount of TAC was referred to the total mass of Y_2O_3 powder in the suspension. The suspensions were then ultrasonically deagglomerated for 10 min by using a high-energy ultrasonic horn (JY92-IIN, Ningbo Scientz Biotechnology Co. Ltd, China) with an output power of 80 W. Subsequently, the suspensions were continuously dispersed by magnetic stirring for 15 min. The suspensions were dispersed again under the same conditions prior to measurements. In all the dispersing involving ultrasonication treatment, suspensions were cooled in an ice-water bath to avoid excessive heating.

2.3. Characterization techniques

The rheological behaviors of the suspensions were recorded by measuring the viscosity as a function of shear rate with a cone-plate viscometer (Brookfield DV-II+Pro, Brookfield Engineering Laboratories, USA). The measurements were performed immediately after suspension dispersing at room temperature environment.

Zeta-potential measurements for the Y_2O_3 suspensions with various amount of TAC as a function of pH were performed using an acoustic and electroacoustic spectrometer (DT-1202, Dispersion Technology Inc., Bedford Hills, USA). A dilute suspension of 5 wt% Y_2O_3 solid loading was used for this measurement due to the difficulties finding in performing a continuous titration toward acidic environment at higher solid loading in the equipment. The titration was automatically performed using a built-in autotitrator with 1 mol/L HCl and 1 mol/L KOH aqueous solution, as these titrants have been shown to have a relative small effect on electroacoustic measurements.

Y_2O_3 suspensions with 1 vol% solid loading were used to study the adsorption behavior of TAC. The fully dispersed suspensions were centrifuged at 3000 rpm for 40 min. The supernatant and resultant solid powders were then separated. For the suspensions with TAC concentration less than 1.0 wt%, the adsorbed amount of TAC was analyzed by measuring the concentration of TAC in the clear supernatant by using an Ultraviolet/visible/Near Infrared spectrophotometer (Model Lambda 750 S, Perkin Elmer, CT, USA). In the meantime, the adsorbed amount of TAC was determined using a DTA/TG analyzer (Model SETSYS Evolution-16, Setaram, Lyons, France) by measuring the weight loss of the dried powders