



A comparative approach to synthesis and sintering of alumina/yttria nanocomposite powders using different precipitants



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HIGHLIGHTS

- Urea proved to be an appropriate precipitant for obtaining a core-shell alumina/yttria nanocomposite.
- Alumina/yttria nanocomposite powders with more appropriate morphology and highly sinterability.
- A fine-grained YAG ceramic was obtained by SPS of alumina-yttria nanocomposite.

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ABSTRACT

Alumina/yttria nanocomposite powder as an yttrium aluminum garnet (YAG) precursor was synthesized via partial wet route using urea and ammonium hydrogen carbonate (AHC) as precipitants, respectively. The products were characterized using X-ray diffraction, field-emission scanning electron microscopy, transmission electron microscopy, Fourier transform infrared spectroscopy and energy dispersive spectroscopy. The use of urea produced very tiny spherical Y-compounds with chemical composition of $Y_2(CO_3)_3 \cdot nH_2O$, which were attracted to the surface of alumina nanoparticles and consequently, a core-shell structure was obtained. The use of ammonium hydrogen carbonate produced sheets of Y-compounds with chemical composition of $Y(OH)CO_3$ covering the alumina nanoparticles. A fine-grained YAG ceramic (about 500 nm), presenting a non-negligible transparency (45% RIT at IR range) was obtained by the spark plasma sintering (SPS) of alumina-yttria nanocomposite synthesized in the urea system. This amount of transmission was obtained by only the sintering of the powder specimen without any colloidal forming process before sintering or adding any sintering aids or dopant elements. However, by spark plasma sintering of alumina-yttria nanocomposite powder synthesized in AHC system, an opaque YAG ceramic with an average grain size of 1.2 μm was obtained.

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1. Introduction

Yttrium aluminum garnet (YAG) is an oxide ceramic with outstanding mechanical properties such as high-temperature strength and low-creep rate. YAG crystallizes with cubic symmetry so it does not exhibit any birefringence effects at the grain boundaries which lead to its high in-line transparency [1]. It is well known as an excellent IR transparent window material and is widely used as a photoluminescence material [2]. Properties such as high-purity, fine particle size and narrow size distribution, low-agglomeration and high-sinterability are essential for fabricating of transparent polycrystalline YAG ceramic [3]. Traditional solid-state

method for preparing YAG powders requires prolonged mechanical mixing as well as calcination at high-temperatures and also obtained powders have an uncontrollable morphology problem and introduced impurities are unavoidable during ball milling [4]. Hence, many kinds of wet chemical routes such as sol-gel [5], co-precipitation with ammonium hydrogen carbonate (AHC), urea or ammonia water as precipitants [6–8], microwave irradiation [9,10], solvothermal [11], supercritical water synthesis [12], spray pyrolysis [13], combustion synthesis [14] and homogeneous precipitation [15] have been used for synthesis of YAG powders. Unfortunately, most of the chemical methods suffer from complex and time-consuming procedures and possible mismatch in the solution behavior of the constituents. Although, sol-gel processing and co-precipitation methods were widely used for powder synthesis, one main disadvantage of these two methods is that

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ultrafine particles of the gel-like precursors underwent severe agglomeration during drying, causing poor-sinterability of the resultant YAG powders [16].

Recently, partial wet routes have been used for synthesis of YAG powders via alumina/Y-compound core-shell structure, in which a layer of Y-compound nanoparticles smoothly covers the alumina core. Sang et al. [17–19] by calcining of the alumina/Y-compound core-shell at 1250 °C could achieve the single-phase of YAG powders and by vacuum sintering of them at 1780 °C for 4 h, transparent YAG ceramic was obtained. It should be considered that most techniques used to produce YAG powders, whether they are solid state reactions or chemical synthesis, are conducted at high-temperature (>1000 °C). However, it is known that high-temperature processing is harmful to microstructure and in-turn the properties due to the non-uniform grain growth [20]. Therefore, low-temperature synthesis and calcination process of nanocrystalline powders of alumina/yttria nanocomposites should be investigated in detail.

In this circumstance, the synthesis of alumina/yttria nanocomposites with the stoichiometric ratio of YAG composition seems to have the capacity of creating a new way for the fabrication of YAG precursors. On the other hand, the uniform distribution of yttria in the alumina matrix could lead to higher transparency of the obtained YAG ceramic, even without adding any sintering aids, dopants or colloidal forming processes before sintering. Hence, the first objective of this study was to synthesize of alumina/yttria nanocomposites via a partial wet route as the YAG precursors. This work describes the influence of urea and AHC as the precipitating agents on the morphology and the sinterability of the alumina/yttria nanocomposites. The second objective was to use the spark plasma sintering (SPS) as an effective sintering technique for obtaining transparent YAG ceramic with high-density in a very short-cycle.

2. Experimental procedure

2.1. Synthesis method

Yttria powder (Merck, 99.9%) was dissolved in a high-purity nitric acid up to a transparent solution of $Y(NO_3)_3$ was obtained. Briefly, $Y(NO_3)_3$ and urea (Merck, 99.5%) were dissolved in distilled water to make a solution. Urea was applied as a precipitant agent at a ratio of 10:1, 20:1 and 33:1 urea: Y^{3+} . The α -alumina (US-Nano research, 99.9%, 50 nm) was added into the mixed solution, and dispersed using ultra-sonication. The mixed turbid liquid with a ratio of $Y/Al = 3/5$, was homogenized using a hot-plate magnetic stirrer at 90 °C for 3 h. After cooling down naturally, the resulting precursor material was collected via suction filtration and byproducts were removed by washing the particles with deionized water three times via suction filtration. After rinsing with anhydrous ethanol, the particles were dried in an oven at 100 °C for 24 h and then calcined at 550 °C for 3 h. To evaluate a slow-reaction rate during precipitation, NH_4HCO_3 solution with concentration of 2 mol/L was obtained by dissolving NH_4HCO_3 in deionized water and (Sigma Aldrich, 99%) at a ratio of 3:1 and 13:1 AHC to Y^{3+} was used as a precipitant by adding it to the above alumina suspension in the droplet form. Separate Y-compound particles were also synthesized by homogeneous precipitation with urea and AHC, in order to study the nature of these particles. The specimen No. and different synthesis conditions of the samples are listed in Table 1. The terminology of U for urea and A for AHC was used as a precipitating agent in the specimen No.

2.2. Preparation of YAG ceramic

The alumina-yttria nanocomposite powders were poured in a

Table 1

The name and conditions of different samples.

Specimen no.	[Precipitant]/[Y^{3+}]	Calcination (temperature-time)	Sintering process
U ₁	33	N/A	N/A
A ₁	13	N/A	N/A
U ₂	10	550°C-3 h	N/A
U ₃	20	550°C-3 h	N/A
U ₄	33	550°C-3 h	N/A
A ₂	3	550°C-3 h	N/A
A ₃	13	550°C-3 h	N/A
U ₅	33	550°C-3 h	SPS
A ₄	13	550°C-3 h	SPS

graphite die of the SPS apparatus (SPS 60-10). The temperature was measured by a pyrometer focused on the upper graphite punch. The main process parameters used for heating and holding at the sintering temperature, are shown in Fig. 1. The sintering temperature is 1450 °C and the pressure loading starts from 1 kN and increases before heating to 22 kN, corresponding to a maximal external pressure of 80 MPa. The soaking time for achieving transparency is about 10 min.

2.3. Characterization methods

X-ray diffraction (XRD) patterns were recorded on the Bruker D8 Advance X-ray diffractometer with Cu K α ($\lambda = 0.15406$ nm). The average crystallite size was calculated from Rietveld method using Maud software (version: 2.49). The composition and impurity contents of the synthesized powders were determined using an inductively coupled plasma (ICP- Model OES 730) chemical analysis. A Tescan Mira (III) field emission scanning electron microscopy (FESEM) was used to characterize the morphology of the samples. The chemical composition of the synthesized powders was evaluated via the energy-dispersive X-ray spectroscopy (EDX). A Philips cm30 transmission electron microscopy (TEM) was employed to study the core-shell structure. For powder preparation, the powder particles were dispersed in an ethanol using an ultrasonic stirrer and an adequate amount of dispersed solution was applied to a carbon-coated copper grid.

The Fourier transform infrared (FTIR) spectroscopic studies were performed on a JASCO FTIR-6300 spectrometer. After sintering process, the relative density of the ceramic was determined by Archimedes's method (the theoretical density of YAG was taken as 4.545 g/cm³) [21]. The sintered specimen was polished using a diamond powder and the transparency of it was determined by in-line transmission measurements using a FTIR spectroscopy (Shimadzu- 3500S) within the IR range.

3. Results and discussion

3.1. Phase evaluations and crystallite sizes measurement

The phase evaluations of the different nanocomposite powders (U₂, U₃, U₄, A₂ and A₃) are shown in Fig. 2. The diffraction peaks of the powders can be identified as corundum (α -Al₂O₃, JCPDS card No. 01-071-1128) with hexagonal symmetry and cubic Y₂O₃ (JCPDS card No. 41-1105) phases which indicate that the powders are the mixture of these two phases. No peaks of impurities were observed, confirming the formation of pure products. Based on the results, Alumina/Yttria nanocomposite with both urea and AHC precipitating agents is obtained.

The average crystallite size of the yttria phase in the alumina/yttria nanocomposite in both urea and AHC systems has been calculated from the results of XRD measurements by the Rietveld

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