

Mechanical and optical properties of spark plasma sintered transparent Y_2O_3 ceramics



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ABSTRACT

Commercial Y_2O_3 nanopowder was used to fabricate transparent Y_2O_3 ceramics by spark plasma sintering under the pressure of 100 MPa for 20 min with the heating rate of 100 °C/min. The microstructures, mechanical and optical properties of the Y_2O_3 ceramics sintered at different temperatures were investigated in detail. Densification occurred up to a sintering temperature of 1500 °C, and above 1500 °C, rapid grain growth and pore growth occurred. The highest relative density of 99.58% and the minimum average grain size of $0.58 \pm 0.11 \mu\text{m}$ were obtained at 1500 °C. The flexural strength, hardness and fracture toughness of the optimal spark plasma sintered Y_2O_3 ceramic were 122 MPa, 7.60 GPa and $2.06 \text{ MPa}\cdot\text{m}^{1/2}$, respectively. The Y_2O_3 ceramic sintered at 1500 °C had the in-line transmission of about 11–54% and 80% in the wavelength range of 400–800 nm and 3–5 μm , respectively.

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1. Introduction

There is increasing interest to develop transparent polycrystalline ceramics in recent decades because of the strong demand for many different applications, such as windows and domes, solid-state laser materials, scintillators, and transparent armors [1–7]. Polycrystalline ceramics have several advantages over single crystals. They can be formed into a variety of shapes, they can usually be produced in larger sizes cheaply, and their mechanical and thermo-mechanical properties are enhanced [4].

Many studies have focused on the transparent ceramics for applications as infrared (IR) windows. Optically transparent over a broad spectral transmission range, a low optical emissivity at elevated temperatures and good mechanical properties are required for these materials. The most durable materials for IR window, AlON ($9\text{Al}_2\text{O}_3 \cdot 5\text{AlN}$), sapphire (Al_2O_3), and Spinel (MgAl_2O_4), emit too much light at elevated temperatures. Unfortunately, the transmission of this material decreased rapidly when the wavelength was increased to approach five micrometers. Compared to this material, polycrystalline yttria (Y_2O_3) has a much lower emissivity at high temperatures, but its mechanical properties, such as hardness, are lower than those of sapphire, Spinel, and AlON [8]. The hardness and strength of ceramics are inversely

proportional to the square root of the grain size. Thus, it is expected that the hardness and strength of Y_2O_3 ceramics are increased with decreasing grain size.

The most important approaches to reduce or eliminate grain size of Y_2O_3 ceramics are the use of nanocrystalline powders, sintering aids and advanced sintering methods to limit the overall grain growth. Several studies have been carried out on the sinterability of nanocrystalline Y_2O_3 [9–11]. Willingham et al. investigated densification of nano- Y_2O_3 powders for IR window applications. They confirmed the superior sinterability of nanocrystalline Y_2O_3 as compared to microcrystalline Y_2O_3 powder [10].

Sintering aids can be used to assist densification of Y_2O_3 ceramics by changing sintering mechanisms. In order to obtain dense Y_2O_3 ceramics, a significant number of sintering additives have been used, such as ThO_2 [12], ZrO_2 [13], La_2O_3 [14], LiF [15], HfO_2 [16], as well as their combinations [17,18]. However, some of them (such as La_2O_3) can negatively affect the specific heat capacity, and consequently the resistance to thermal shock of this material [4].

Spark plasma sintering (SPS) is an advanced sintering technology that combines uniaxial pressure with heating by an electrical current passing through the die that contains the powder or green body. On the other hand, SPS is a new field-activated sintering technique that provides a way to quickly sinter ceramic powder, as well as nanostructured materials to full density at relatively low temperatures and very short times as compared with conventional sintering methods. Therefore, grain growth is controlled mainly by the higher heating rate (i.e. limited surface

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diffusion) and shorter processing times (i.e. no impediment from the porosity). Several researchers have investigated the densification of Y_2O_3 nano-powders by SPS in order to achieve fully dense and transparent Y_2O_3 ceramics [19–28]. In 2008, a Y_2O_3 body with a relative density of 97% was sintered by SPS at 850 °C for 1 h [19]. Chaim et al. reported on the fabrication of Y_2O_3 ceramics using SPS with a relative density of greater than 98% at 1400 °C for 5 min [20]. Nevertheless, it is demonstrated that relative densities of Y_2O_3 bodies should be greater than 99% in order to reach transparent Y_2O_3 bodies.

SPS was used for the fabrication of transparent Y_2O_3 ceramics with an average grain size of 40 μm , dense microstructures and a good optical properties for the first time by Mingsheng et al. [26]. These researchers used commercial Y_2O_3 powder with a mean particle size of 1 μm and SPS was followed by hot isostatic pressing (HIP); without any additive.

Yoshida et al. have reported on the fabrication of the translucent polycrystalline Y_2O_3 ceramics at low temperature using high-purity Y_2O_3 powder [28]. They were able to reach almost 99% of the relative density and in-line transmission of 6–46% in a wavelength range of 400–800 nm at 950 °C or 1050 °C with a heating rate of 2 °C/min by SPS.

Zhang et al., reported on the fabrication of transparent Y_2O_3 by high-pressure spark plasma sintering [25]. Uniaxial high pressure of 300 MPa at a temperature of 1050 °C for 1 h resulted in full density and nanometric grains of about 400 nm, which lead to transparent ceramics (in-line transmission of 68% at the wavelength of 700 nm). Recently, Ito et al. reported on the fabrication of transparent Y_2O_3 ceramics by SPS at moderate temperature [24]. Depending on how the authors applied sintering and annealing temperatures, they were able to measure transmission and Vickers hardness up to 81.6% (for wavelength of 2000 nm) and 9 GPa, respectively. According to these authors, reports the most important processing parameters are the sintering temperature, the heating rate, the applied pressure and the use of additives material.

Since the main use of Y_2O_3 transparent ceramics is in infrared windows and domes, they must enjoy high transmission and also proper mechanical properties. Although numerous studies have been carried out on the fabrication of transparent Y_2O_3 ceramics by SPS, most of them focused on densification, microstructure and the mid- infrared transmission spectra behavior of Y_2O_3 ceramics using SPS processes consists of low heating rate and high sintering time (Table 1). In the present study, transparent Y_2O_3 ceramic was fabricated using high heating rate and low sintering time (20 min). Besides microstructure and optical properties of SPS-sintered Y_2O_3 ceramics, the effects of sintering temperature on mechanical properties (hardness and “flexural strength for the first time”) were also studied. It should be mentioned that numerous studies have reported higher transmission for below 2.0 mm thickness (often 1.0 mm), while the specimen thickness in this paper is 2.5 mm.

2. Experimental

2.1. Starting materials

Nanocrystalline Y_2O_3 powder (Beijing Dk Nano Technology, China) with a high-purity (99.99%), particle size of 30–50 nm and a BET multi-point specific surface area of 30–50 m^2/g was used in the experiments. Fig. 1 shows the FESEM photographs of the as-received nanopowder. The ultra-fine nanopowder with almost spherical grains was weakly agglomerated.

Table 1

In-line transmission (at the wavelength of 700 nm) and grain size of transparent Y_2O_3 ceramics fabricated by SPS.

Condition of SPS	Grain size and in-line transmission	References
100 °C min ⁻¹ /1200 °C/45 min/vacuum/ 100 MPa	Specimen thickness: 1 mm 0.3 μm 63%	[24]
2 °C min ⁻¹ /1050 °C/1 h/ vacuum/ 300 MPa	Specimen thickness: 1.5 mm 0.4 μm 38%	[25]
2 °C min ⁻¹ /1050 °C/1 h/vacuum/ 80 MPa	Specimen thickness: 1.5 mm 0.4 μm 38%	[28]

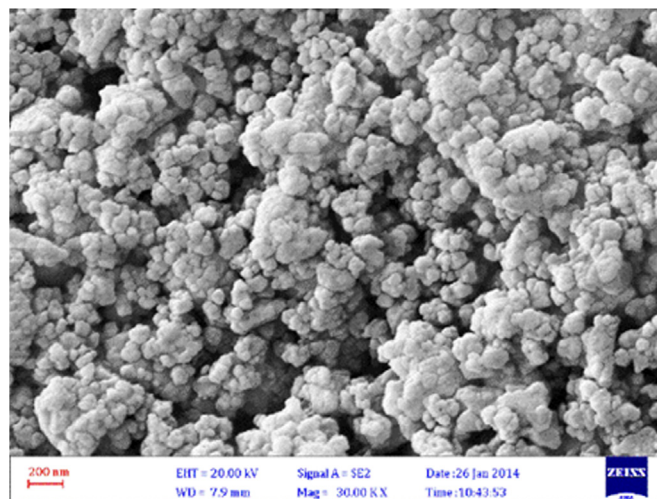


Fig. 1. FESEM photographs of the as-received nanopowder.

2.2. Experimental set-up

The Nanocrystalline Y_2O_3 powder was directly poured into a graphite die without any special treatment or additives, and was pressed by a 20 mm diameter graphite punch in vacuum (2–3 Pa). In order to facilitate specimen extraction after sintering and prevent possible reaction between punch and powder, a 0.2 mm thick graphitic foil was inserted between the powder and the die/punch. The graphite die and punch were heated to 850 °C in 3 min, followed by high heating to the sintering temperature (1200–1600 °C) and a 20-min hold at the sintering temperature (Fig. 2) using a spark plasma sintering machine (SPS 60–10, Malek Ashtar University of Technology, Iran) with a pulse duration of 3.3 ms. The power was then turned off and the specimen was cooled at a rate of 50 °C/min to room temperature in the SPS chamber. A preload of about 2 MPa was applied in the beginning to insure good electrical conduction between the plunger, specimen and the mold. Then, the target pressure of 100 MPa was uniformly loaded in the temperature range of 1100 °C to sintering temperature during heating. After the dwell step, all the specimens were cooled to the room temperature, during which the pressure was gradually released. The temperature was measured by an optical pyrometer focused on the non-through hole (1 mm diameter and 2 mm depth) in the graphite die. Finally, a sintered disk was obtained with a diameter of 20 mm and a thickness of 3 mm. The sintered body was then annealed at 1200 °C for 10 h in the air. Eventually, acquired bodies were ground to a thickness of 2.5 mm and carefully mirror-polished on both sides using diamond slurries (to

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