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Enhancement of the optical transmittance of hot-pressed transparent yttria ceramics by a multi-step sintering process

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ABSTRACT

In this work, we report the enhancement of the optical transmittance of yttria ceramics via a 'pre-sintering/hot-pressing/hot-isostatic pressing' multi-step process. The in-line transmittance reaches 78.0% at 400 nm and 83.2% at 1100 nm with an average grain size of approximately 1 μm. This sintering route is derived and upgraded from our previously reported one-step hot-pressing method for the fabrication of transparent yttria ceramics. Through the enhanced strength of the green body by pre-sintering, the formation of cracks and the breaking up of the as-hot-pressed samples can be effectively suppressed, leading to sound or crack-free but less transparent samples. After a post-HIPing treatment, the optical transmittance of the samples with a pre-sintering step was substantially improved to higher levels in the visible and the IR wavelengths compared to that of a sample which does not undergo pre-sintering. The corresponding mechanism was revealed by microstructural analyses.

1. Introduction

By means of easier fabrication routes, better mechanical properties, and comparable optical transmittance, transparent polycrystalline yttria ceramics are widely known as very important optical materials which can replace single-crystal yttria. Given their broad transparency range (0.2–8 μm), the high optical transmittance (> 80% in the visible region), as well as their outstanding corrosion resistance, polycrystalline transparent yttria ceramics have been successfully used in various applications, including transparent windows, domes, and laser hosts [1–3].

Owing to the high melting point (2430 °C) of yttria, it is not easy to fabricate transparent yttria ceramics with excellent optical transparency in which residual pores must be eliminated as completely as possible. In recent years, a number of high-performance transparent and laser yttria ceramics have been exploited by virtue of rapidly developing ceramic technologies [4–6]. However, according to the literature, yttrium aluminum garnet (YAG)-based transparent and laser ceramics are currently much more popular than yttria-based ceramics in spite of a few disadvantages of YAG. That is, yttria possesses higher thermal conductivity and less thermal expansion than YAG, which is critical for the thermal management of high-temperature windows and high-power laser media materials [7]. It is interesting to find that while the theoretical transmittance of yttria is comparable to that of YAG, the reported transmittance of yttria ceramics is always lower than that of YAG ceramics, especially in the visible region [1,7–9]. This implies that it is extremely difficult to achieve the theoretical transparency in yttria ceramics compared to YAG ceramics, which should be mainly attributed to the high melting temperature of yttria, as mentioned above. Additionally, yttria shows relatively poor mechanical properties in nature compared to many other transparent materials, including YAG, spinel, sapphire, and AlON [10]. However, mechanical strength is quite important for bulk ceramic materials, especially for optical windows or domes when used under extreme conditions, such as at high pressures or elevated temperatures. Therefore, it is crucial to enhance the mechanical properties of transparent yttria ceramics before they are used. Generally, polycrystalline ceramic materials can be effectively strengthened by decreasing their grain sizes. Nevertheless, it is very difficult to obtain transparent yttria ceramics with fine microstructures by conventional sintering routes, as the samples need to be sintered at high temperatures (e.g., ≥ 1800 °C). With some newly developed unconventional sintering methods, fine-grained transparent yttria ceramics have been successfully created. Huang et al. fabricated transparent yttria ceramics with a grain size of about 1–2 μm by means of oxygen-atmosphere sintering [3]. Serivalsatit et al. reported submicron-grained transparent yttria ceramics fabricated by a two-step sintering process combined

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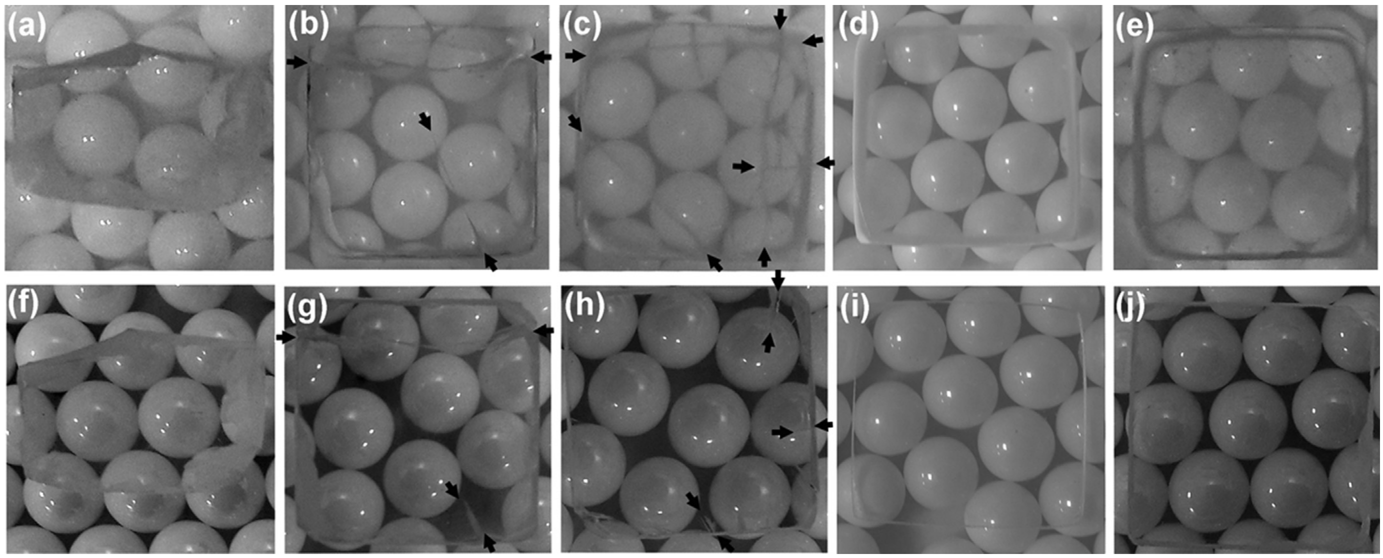

Fig. 1. Photographs of the as-hot-pressed samples pre-sintered at 1250 (a), 1300 (b), 1350 (c), 1400 (d), and 1450 °C (e); and photographs of the post-HIPed samples pre-sintered at 1250 (f), 1300 (g), 1350 (h), 1400 (i), and 1450 °C (j) (The start points and end points of the cracks in the samples are marked by the black arrows).

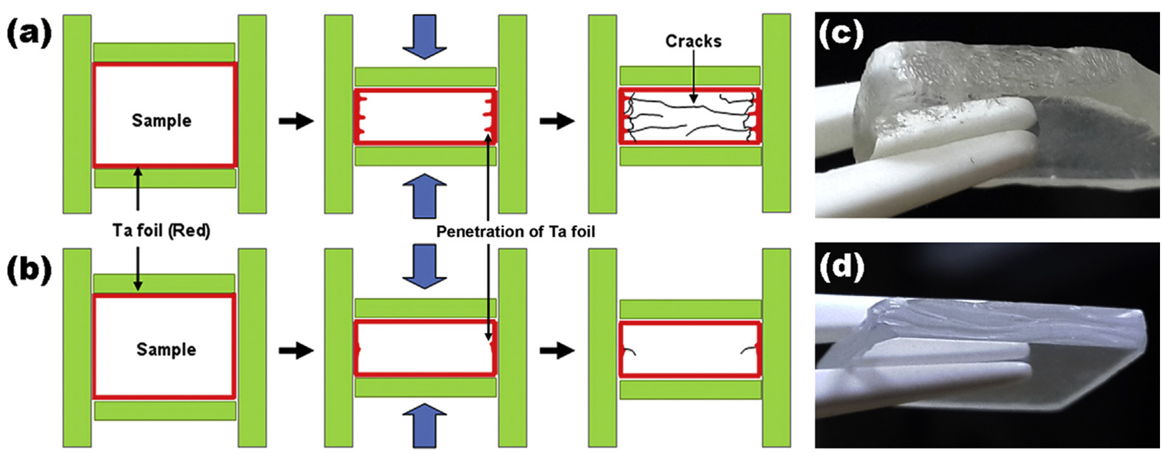

Fig. 2. (a) and (b) are the schematics of a possible formation mechanism of cracks in hot-pressed samples without pre-sintering and pre-sintered at proper temperatures, respectively; (c) and (d) show the side edges of the transparent ceramics (after HIPing) without pre-sintering and after being pre-sintered at 1450 °C, respectively.

Table 1
The relative densities of the samples before and after pre-sintering at various temperatures.

Pre-sintering temperature (°C)	Relative density (%)
Before pre-sintering	~44
1250 °C	45.9
1300 °C	46.7
1350 °C	47.8
1400 °C	49.5

with a post-HIPing treatment [7].

Very recently, our group developed a new practicable approach simultaneously to achieve fine microstructures and excellent transmittance in yttria ceramics by hot-pressing at a relatively low temperature of 1600 °C, which is enabled simply by wrapping the samples with tantalum foil for perfect prevention against carbon contamination from the graphite mold [11]. Although the sample is already highly transparent, its transmittance (i.e., 74.4% at 400 nm and 81.1% at 1100 nm) is still not high enough for use as a laser host material. Therefore, further improvements are necessary. Additionally, there remains another significant problem to be solved in that the hot-pressed samples usually have some cracks, resulting in the breaking up of some of the samples. A similar phenomenon was also claimed to occur in the hot-pressed transparent yttria ceramics reported by Majima et al. [12]. In order to improve the optical transmittance of the hot-pressed yttria ceramics further and find a solution for the aforementioned cracking problem, a multi-step sintering process consisting of pre-sintering, hot-pressing, and post-HIPing was exploited. The fabrication of crack-free yttria ceramics with improved optical transmittance was successfully demonstrated by the current multi-step process.

2. Experimental procedure

Commercially available Y_2O_3 (99.99%, Rare Metallic Co Ltd., Japan) and $ZrO(CH_3COO)_2$ (98%, High-Purity Chemicals, Japan) powders were used as raw materials. The raw powders were weighed according to the ZrO_2 doping concentration of 1 at% and were milled with ZrO_2 balls in anhydrous alcohol (99.9%, Samchun, Korea) for 24 h. After ball-milling, the slurry was dried by a rotary evaporator at 80 °C. The dried powder mixture was ground and sieved though a 150-mesh sieve and then calcined at 800 °C for 4 h to remove any organic components completely. The calcined powder mixture was dry-pressed at 5 MPa into 15 mm square