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Bimodal transparent alumina ceramics prepared with micro/nano-particles under high pressure

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ABSTRACT

Alumina nano-powder was used as the cement in bonding coarse spherical particles to fabricate transparent alumina ceramics under high pressure and modest temperature. Observations on the microstructure, density and microhardness of Al_2O_3 specimens prepared at 500–1200 °C under 5.0 GPa provided significant effects of additive nano-particles on the sintering of samples. The high pressure initiated plastic deformation of spherical particles, rearranged nano-particles filling the clearance among spherical particles and bonding each other to densify the products. The additive particles also contribute to grain boundary strengthening to obtain samples with enhanced transparency and microhardness.

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Transparent alumina ceramics can be achieved through a variety of approaches. Such as hot isostatic pressing sintering (HIP) [1,2], microwave sintering [3], vacuum sintering [4,5], high temperature sintering in hydrogen gas [6], spark plasma sintering (SPS) [7–10] and ultra high pressure sintering [11–13]. Due to the large grains increase light scattering in birefringent materials such as alumina [5], submicron (<0.5 μm) alumina powder was preferred as the starting material to obtain fine grains, high density and high transparency alumina ceramics. High temperature was indispensable during most alumina sintering for the sake of reducing the source of light scattering and absorption at grain boundaries and pores. While, the sintering temperature decreased to about 1000 °C with the simultaneous pressure application which also suppress grain growth during SPS sintering [9]. Differs from those technicals, ultra-high pressure (>1 GPa) sintering was valid to suppress the grain growth of alumina and further decreased the sintering temperature to 800 °C [12]. It has also shown the feasibility on the high pressure sintering of other nano-materials such as $MgAl_2O_4$ [14]and YAG [15].

Previous work has demonstrated that the spherical alumina powder sintered at lower temperature (<900 °C) was opaque due to the increased light scattering and absorption at the poor-bonded particle boundaries and large amount of closure pores. The recrystallized crystals and growth-pores at grain boundaries were responsible for the high optical dispersion and lack of transparency when the sintering temperature was above 900 °C [13]. Hellmig [16] and Ferkel [17] reported that the high sinter activity alumina nano-particles were a favorable cement in bonding coarse-grained corundum ceramics. So, it was supposed to bond micro-size spherical alumina particles and has been placed great expectations on improving the boundary situation to achieve transparent bulks with enhanced transparency and microhardness.

In this work, we used mixed size Al_2O_3 powder (85 wt.% micro-size spherical particles and 15 wt.% nano-particles) as the starting material to achieve transparent alumina ceramics under high pressure and modest temperature. Through analyzing the microstructure, density and Vickers microhardness of Al_2O_3 compacts prepared at 500–1200 °C under 5.0 GPa, the obvious effect of additive nano-particles on the sintering of samples was demonstrated.

The spherical powder (Bengbu China Triumph New Materials Co., Ltd., China) was composed of 99.8% Al_2O_3 (~90% α-Al_2O_3 and small amount of δ phase). The nano-size powder (Xuan Cheng Jing Rui New Material Co., Ltd., China) with a purity of 99.99%, contains 90% α-Al_2O_3 and a little θ phase. The scanning electron microscopy (SEM) image and X-ray diffraction (XRD) patterns of the starting material after acid treatment are shown in Fig. 1a, b and c respectively. Micro and nano-size Al_2O_3 mixture in a weight ratio of 17:3 was first treated by ultrasonic for 1 h to disperse the nano-particles, and then mechanical mixed in tumbling mixer for 12 h. The grain diameter of spherical granulated particles ranges between 1 and 38 μm as is presented in Fig. 1(a-1). After mechanical mixed, the smooth surfaces of spherical particles were covered by nano-size particles as is shown in Fig. 1(a-2). The average grain size of nano-powder is estimated to be 80 nm from Scherer's formula [18], which in consistent with the appearance in SEM (Fig. 1(a-3)).

The powder was loaded in molybdenum (Mo) capsules every 1.2 g, and then dried in vacuum furnace (~1.5×10^{-3} Pa and 800 °C) for 2 h to get rid of impurity gases attached to the grain surface. After vacuum heat treatment, the sample was pre-compressed (about 300 MPa) into thick discs (11.0 mm in diameter and 4.5 mm in height) with a relative density of about 80%, and followed by high-pressure and temperature

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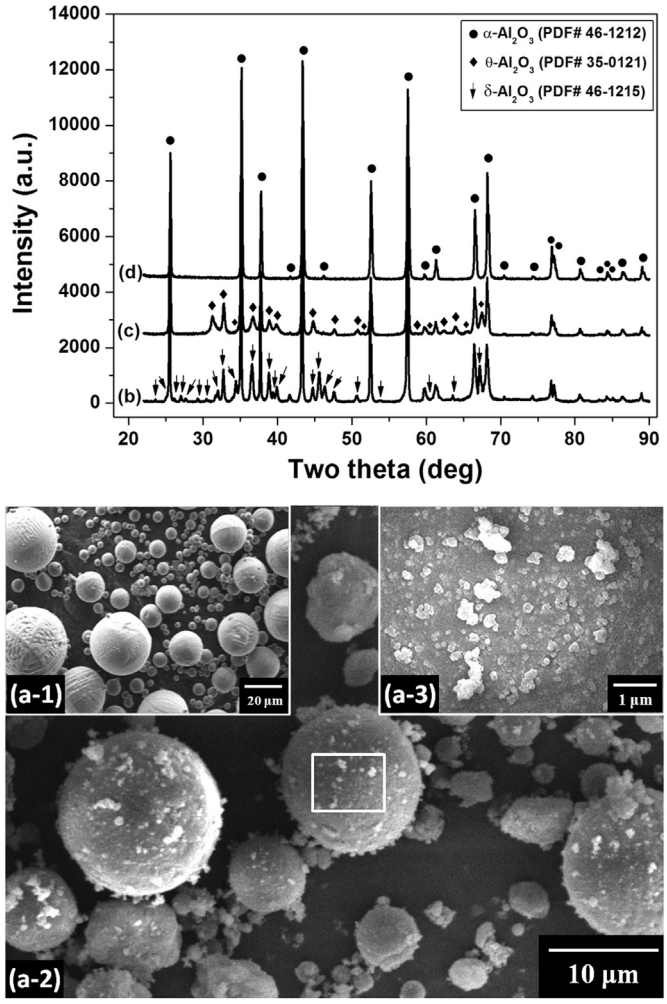

Fig. 1. SEM images of starting material and its XRD patterns after acid treatment, and the XRD pattern of sintered sample. (a-1) Spherical particles, (a-2) mixed powder, (a-3) enlarged part of rectangle in (a-2); (b) XRD of spherical powder; (c) XRD of nano-powder; (d) XRD of mixed powder sintered at 5.0 GPa/600 °C, metastable Al_2O_3 completely convert to α-Al_2O_3.

treatment which performed on a cubic press (DS6 × 14 MN, China). The cell temperature was measured directly using a PtRh6%–PtRh30% thermocouple, and the pressure was estimated from the oil pressure reading calibrated by the silver melting-point method at high pressures [19]. Detailed experimental procedures can be found elsewhere [13]. All the samples were sawed to a thickness of 0.6 mm and polished carefully on both sides to eliminate surface scattering.

One group of mixture powder was compressed to 5.0 GPa, following by high temperature treatment from 500 to 1200 °C. Another set of samples were just micro-size spherical particles and treated with the same *P-T* conditions for comparison. X-ray diffraction experiments were carried out to determine the phase composition of the sintered samples. The densities of the sintered bodies were obtained by the Archimedes method and grain boundary situation was investigated using metallo microscope and scanning election microscopy (SEM). Microhardness was determined on the polished surfaces using an applied load of 0.5 Kgf by the Vickers hardness tester.

Pure α-Al_2O_3 bulks without crack can be achieved when temperature increased above 600 °C under 5.0 GPa as is conveyed in Fig. 1d. The metallographic photos of the polished samples sintered under various conditions are shown in Fig. 2. The samples without adding nano-particles (Fig. 2a to c) show the similar grain boundary situation as the previous study [13], i.e., ultra-high pressure can initiate the plastic deformation of spherical particles to form "Y" shape grain boundary (Fig. 2a) and eliminate pores at modest temperature, promote bonding among particles with increasing temperature (Fig. 2b) but failed to inhibit growth-pores and non-uniform crystallites at grain boundaries at higher temperature (Fig. 2c). So, the transparency of sintered samples first increases and then shows an obvious decrease with increasing temperature.

With the additive nano-particles, the samples show obvious disparate microstructure as is presented in Fig. 2d–f. When sample sintered at 5.0 GPa/700 °C (Fig. 2d), the original boundaries among adjacent particles became partially continuous, in apparent contrast with the one which shows visible separation at particle interfaces (Fig. 2a), and indicating that significant welding occurred at the interfaces of spherical particles. Thus, the reduction of light scattering and absorption at grain boundaries promoted to fabricate transparent ceramics. With the temperature increased to 900 °C (Fig. 2e), the metallographic photo reveals the similar microstructure as the one sintered at 5.0 GPa/700 °C. Even though the sintering temperature increased to 1100 °C, the sample seems retained the similar characters as those sintered at 700 °C and 900 °C as is illustrated in Fig. 2f, resulting in no obvious decrease of transparency of sample. The boundary among severely deformed particles can still be distinguished. However, without adding nano-particle, the sample shows obvious decrease of transparency at the same *P-T* condition. The metallographic photo also implies that the original grain boundaries disappeared completely as is shown in Fig. 2c.

Accordingly, Fig. 2 implies that the additive nano-particles seems plays significant role in improving the transparency of samples, which might be focus on promoting bonding of spherical particles at modest temperature under high pressure and decreasing light scattering and absorption at grain boundaries. So, SEM was carried out to further examine the boundary situation.

Fig. 3 shows the SEM of fracture surfaces of samples sintered at 700–1100 °C under 5.0 GPa. As is elucidated in Fig. 3a that the sample without adding nano-particle after treated at 5.0 GPa/700 °C presents clear irregular hemispheric pits, which retained the contour of deformed spherical particles. The impression that particle boundaries are not well bonded is confirmed by the clean separation at particle interfaces and apparent intergranular fracture. So the sample is opaque due to the increasing light scattering and absorption at grain boundaries. While, at the same *P-T* condition, the sample sintered with the additive nano-particles shows large amount of obvious transgranular fracture (arrows in Fig. 3d) which was the evidence of well bonding among spherical particles as is shown in Fig. 3d. The size of the nano-size crystals is estimated to be around 200 nm in Fig. 3(d-2), which is a prefer size in transparent alumina sintering [5]. The small amount of metastable phase might transformed to nano-scale α-Al_2O_3 as the γ-Al_2O_3 did under high pressure and low temperature [11,20]. Those submicron particles were like cement to bond spherical particles, which is responsible for reducing light scattering and absorption at grain boundaries in comparison of the one without adding nano-particle. So, the obvious improved boundary situation contributes to apparent increase of transparency of samples.

With the temperature increasing to 900 °C (Fig. 3b), bonding at the interfaces of particles occurred in the pure micro-size powder sintered sample, but simultaneously with abnormal grain growth and growth-pores (rectangle in Fig. 3b). This phenomenon also can be found elsewhere [11,21,22]. While, in the mixture powder sintering, no obvious growth-pores occurred at the same *P-T* condition (Fig. 3e) even when the temperature further increased to 1100 °C (Fig. 3f). This might be the reason why no obvious decrease of transparency of samples sintered with additive nano-particles. Accordingly, from Fig. 3, we could conclude that the adding of nano-particles has provided visible insight into improving transparency of samples. The characters observed in Fig. 3 also in agreement with the metallographic observations in Fig. 2.

The relative densities of sintered samples under 5.0 GPa at various temperatures are shown in Fig. 4a. The samples sintered with nano-

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