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Original article

Effect of crystallographic orientation on transparency of alumina prepared using magnetic alignment and SPS

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ARTICLE INFO	A B S T R A C T
Keywords: Index differences Scattering coefficient Birefringence Strong magnetic field	Transparent polycrystalline alumina was developed over many years because its attractive properties are expected to find applications in many fields. Crystallographic orientation is one of the effective ways to improve transparency in birefringent ceramics such as alumina, because birefringence at grain boundaries can be suppressed by the alignment of optical axis. Fabrication of high-transparency alumina with an oriented c-axis and fine microstructure can be attained by slip casting in a strong magnetic field, followed by spark plasma sintering at 1150 °C for 20 min. The real in-line transmittance of the textured alumina with a thickness of 0.80 mm was 70% at $\lambda = 640$ nm, which was higher than that of randomly-oriented alumina. The c-axis orientation reduced the actual difference of the refractive index and suppressed remarkably the birefringence.

1. Introduction

Transparent polycrystalline alumina has been studied by numerous researchers since its initial development by Coble [1]. Extremely low porosities (< 0.05%) and small grain sizes ($< 1 \mu m$) are critical for obtaining highly transparent alumina with good mechanical properties including high strength and wear resistance. A residual porosity of only 0.1% can deteriorate transparency, and large grains can increase light scattering in birefringent materials such as alumina [2]. In existing transparent alumina, the aforementioned microstructural features have been obtained by sintering at low temperatures and under pressure using hot isostatic pressing (HIP) [3] or spark plasma sintering (SPS) [4]. Kim et al. pointed out that slow heating is more effective in obtaining high density and a fine microstructure, although rapid heating has been recognized as one of the most prominent advantages of SPS [5-7]. According to Roussel et al., highly dense transparent Al₂O₃ ceramics have been created using discrete, ultrafine, high-purity Al₂O₃ nanoparticles sintered by the standard SPS technique [8]. Nanko et al. reported that fully-densified Al₂O₃ ceramics with fine grains were obtained by SPS through a two-step heating profile [9]. The use of high pressure during SPS has also been reported to be effective in improving the transparency of alumina [10,11]. Alumina has an asymmetric crystal structure with one optical axis, but if its crystal orientation can be aligned parallel to the optical c-axis, incident light can penetrate without birefringence. Therefore, crystallographic orientation in ceramics with anisotropic polycrystalline alumina may reduce light scattering and improve light transmittance.

We have previously reported that a strong magnetic field can control crystallographic orientation, even for diamagnetic ceramics, through colloidal processing for particles dispersed in a slurry. This technique can be applied to a wide variety of ceramics with asymmetric crystal structure such as Al_2O_3 , TiO₂, ZnO, AlN, SiC, and CaBi₄Ti₄O₁₅ [12–18]. The alignment of the c-axis in alumina can be kept parallel to the direction of the magnetic field [12]. Mao et al. reported that alumina prepared using a strong magnetic field exhibited higher optical transparency than a sample without a magnetic field [19]. In addition, transparent crystalline-oriented polycrystalline alumina has been fabricated using a magnetic field followed by HIP, and its transmittance perpendicular to c-axis was higher than that of a randomly-oriented sample [20].

In this research, we attempted to improve the transparency parallel to the c-axis in alumina, and investigate the effect on birefringence of the c-axis orientation due to an applied magnetic field at its grain boundary when densified by SPS with a slow heating rate.

2. Experimental procedure

The starting material was high-purity (99.9%), spherical α -Al₂O₃ powder (AA-03 Sumicorundum grades, Sumitomo Chemical Co. Ltd., Japan) with an average particle size of 0.39 µm and a Brunauer-Emmett-Teller (BET) specific surface area of 4.5 m²/g. The Al₂O₃ powder was dispersed in distilled water with an appropriate amount of

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Fig. 1. Photos of alumina sintered by SPS at 1150 °C for 10 min at a heating rate of 5 °C/ min. Samples were prepared (a) without a magnetic field (R5), (b) in a magnetic field (T5), (c) without colloidal processing (P5).



Fig. 2. Total forward transmission of the alumina sintered at 1150 $^\circ C$ for 10 min at a heating rate of 5 $^\circ C/min.$ (t = 0.8 mm).

dispersant to ensure dispersion by mutual electrosteric repulsion. The suspensions were mixed with a magnetic stirrer, and ultrasonicated for 10 min to break the agglomerated powder. Suspensions with 30 vol% solid were consolidated by slip casting. A strong 12 T magnetic field was applied to the suspension during the consolidation process at room temperature. The green compacts before sintering were further densified without disturbing the particle orientation by cold isostatic



Fig. 3. Real in-line transmission of alumina sintered at 1150 $^\circ C$ for 10 min at a heating rate of 5 $^\circ C/min.~(t=0.8~mm).$



Fig. 4. Photos of alumina sintered by SPS at 1150 °C for 20 min at a heating rate of 2 °C/ min. Samples were prepared (a) without a magnetic field (R2), (b) in a magnetic field (T2), (c) without colloidal processing (P2).

pressing at 392 MPa for 10 min and calcined at 500 °C for 1 h in air in order to burn off the dispersant. Final sintering was carried out at 1150 °C under a uniaxial pressure of 100 MPa using a Spark Plasma Sintering machine (SPS-1050, Sumitomo Coal Mining Co., Ltd, Japan). The pressing direction during SPS was parallel to the orientation direction of the *c*-axis by the magnetic field. The temperature was

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