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Fabrication of Textured Ceramics by Slip Casting in a High Magnetic Field and Heating

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The control of texture in ceramics is one of the ways for effectively improving their properties. Ceramics with asymmetric unit cells shows anisotropic susceptibility, but it is very small in diamagnetic ceramics such as Al_2O_3 and TiO_2, therefore, the effects of magnetic fields on these ceramics had been generally neglected. Recently, superconducting magnet technologies have developed and have been used for various applications. The dispersion of powders in a suspension is important when effectively applying a magnetic field to the powders. We demonstrate in this paper that the high texture of TiO_2 can be controlled by a high magnetic field applied to these powders in a suspension followed by heating.
Key words: high magnetic field, colloidal processing, textured ceramics, titania

1. INTRODUCTION

The controlled development of texture in polycrystalline materials is effective in improving physical and mechanical properties. Previous studied have reported the production of textured ceramics using the Templated Grain Growth method [1], tape casting, high-temperature deformation [2] and the RBAO (reaction bonding of aluminum oxide) technique [3, 4].

A magnetic field has been rarely applied to paramagnetic and diamagnetic ceramics such as alumina because their susceptibility is very small, but the development of superconducting magnet technologies has enhanced the application of a high magnetic field to various materials [5, 6]. A large interaction between the agglomerated particles in a suspension is also one of the factors that prevent the powders in a suspension from rotating by applying a magnetic field. The dispersion of powders in a suspension is necessary for effective utilization of the magnetic field. Colloidal processing was used in this study because the processing is very effective in developing consolidated fine particles to avoid heterogeneous agglomerates by using repulsive surface forces [7, 8]. We have demonstrated in previous studies that the highly textured microstructure of pure dense alumina can be controlled by a high magnetic field applied to alumina powder in a suspension followed by heating [9, 10].

Titania (TiO_2) is an important electronic ceramic for use in diverse applications. Titania also has several polymorphic phases, commonly anatase (tetragonal) or rutile (tetragonal). Anatase converts to rutile at temperatures of 400-1200°C. This transformation temperature depends on several parameters, such as impurities and grain size [11-13]. Heating is an important process for developing textured microstructures in the material prepared by slip casting in a high magnetic field [9]. If a phase transformation occurs during the heat treatment, this transformation could have an effect on the forming a textured ceramic because crystals may rotate during the transformation. We demonstrate in this study that this method (slip casting in a high magnetic field followed by heating) can be applied to titania which exhibits anatase-to-rutile transformation during heating.

2. EXPERIMENTAL PROCEDURE

The starting material was fine spherical TiO_2 powder (C.I.Kasei Co., Ltd., Japan) with particles averaging 30 nm. This powder purity is greater than 99.8%. This powder was dispersed in distilled water with an added polyelectrolyte (poly (ammonium acrylate) A-6114, Toaghosei Co., Japan) to ensure dispersion by electrosteric repulsion between the particles [14]. The suspensions were prepared with 20 vol% solids. The suspensions were mixed with a magnetic stirrer, and an ultrasonic horn (UPS-600, Shimazu Inc., Japan) was operated for 10 minutes to disperse the powder [15, 16]. The suspensions were degassed in a vacuum, then poured into a gypsum mold, and left to consolidate. A high magnetic field of 10T was applied to the suspensions during the slip casting at room temperature. The green compacts were further densified by cold isostatic pressing (CIP) at 400MPa for 10 minutes without disturbing the particle orientations. The samples were isothermally heated at a temperature between

1273K and 2073K for 2 h in air without a magnetic field.

The degree of crystalline texture was determined using equation (1) from the intensities of the X-ray diffraction measurement.

$$P = I_{002}/(I_{002} + I_{110}) \qquad (1)$$

Where I_{002} and I_{110} are the intensities from the 002 and the 110 reflections on the surface perpendicular to the direction of the magnetic field, respectively. The number of grains with the c-axis parallel to the magnetic field increases as the degree of crystalline texture approaches 1. The samples were polished and then thermally etched by heating at temperatures 200 K lower than the heat-treatment temperature for 1 h. SEM was used for observation of microstructures.

3. RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction patterns of the starting powder and a body sintered at 1273K. The starting TiO_2 powder consists of mainly anatase and small amounts of rutile. The XRD analysis showed that heating at 1273K fully transforms the anatase into rutile crystals.

Figure 2 illustrates the XRD profiles of the specimen which was compacted by slip casting in 10 T, followed by heating at 1573K for 2 h, together with the rutile standard. The direction of the magnetic field was perpendicular to the bottom surface (parallel to the fluid flow during slip casting). In the surface perpendicular to the magnetic field (T-plane in Fig. 2), the intensities of the (002) peaks are very large. By contrast, in the surface parallel to the magnetic field (S-plane in Fig. 2), the intensities of the (*hk*0) peaks are very large. Therefore, it was demonstrated that the crystalline texture with the c-axis parallel to the magnetic field was developed by slip casting in a high magnetic field and heating. The texture can be explained by the anisotropic susceptibility of anatase (tetragonal) with an asymmetric unit cell.

Figure 3 shows the degree of crystalline texture calculated from equation (1) together with the shrinkage as a function of the heating temperature for the specimens prepared by slip casting in 10 T (B ⊥ the bottom surface) and the specimens without a magnetic field. For the specimens without a magnetic field, the degree of crystalline texture is approximated to be 0.09 in agreement with that calculated from the JCPDS value. The specimen prepared without a magnetic field was confirmed to be a crystalline untextured material. By comparison, for the specimens prepared by slip casting in 10 T, the degree of crystalline texture at 1273 K is small, but the value is larger than that of the untextured material. The degree of crystalline texture in the specimens prepared in a high magnetic field increased with the increasing heating temperature of more than 1273 K where the shrinkage was a maximum at 18% where the relative density is 94% of theoretical. The crystallographic texture development occurs after densification. The anatase-to-rutile transformation occurred during

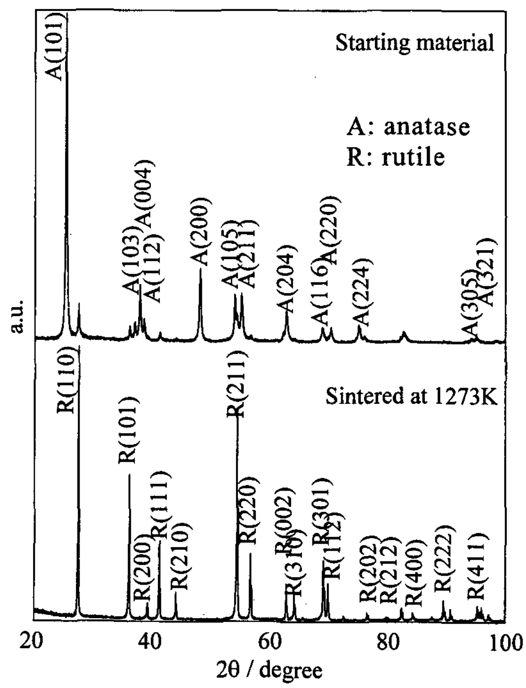

Fig.1 X-ray diffraction patterns of starting material and a specimen sintered at 1273K for 2h.

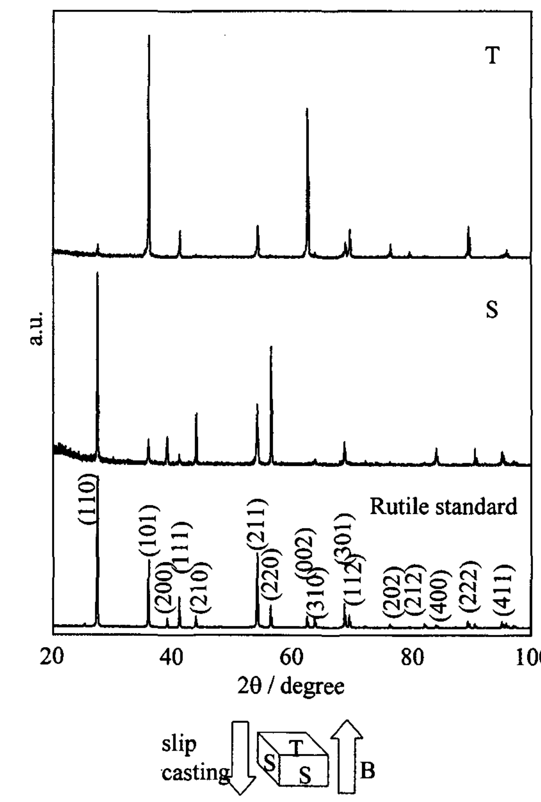

Fig.2 X-ray diffraction patterns of titania sintered at 1573 K in the planes perpendicular and parallel to the magnetic field, which is parallel to the fluid flow. The bottom profile shows a diffraction pattern of the rutile standard.

heating, whereas the degree of orientation increased after transformation.

Figure 4 shows SEM images of the polished sections of the specimen without a magnetic field and the specimen prepared by slip casting in 10 T (H ⊥ the bottom surface), followed by sintering at 1873K for 2 h. It can be seen that the equiaxed grains appear randomly distributed in the untextured material without a magnetic field (Fig. 4 (a), (b)). The grain size in the textured material is larger than that in the specimen without a magnetic field (Fig. 4 (c)). It seems in Fig. 4 (d) that the slightly elongating grains are oriented parallel to the magnetic field.

4. SUMMARY

By using colloidal processing, the fine titania powder is well dispersed in the suspension. The suspension was consolidated by slip casting in a high magnetic field (10T) followed by heating. Highly textured rutile can be obtained after densification, though the anatase-to-rutile transformation occurs during heating. Elongated grains parallel to the magnetic field are obtained in the textured titania.

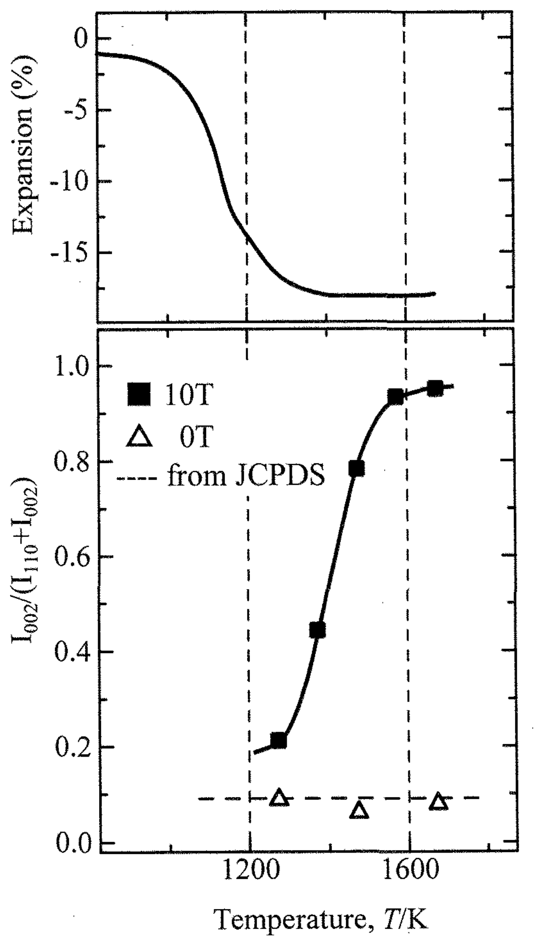

Fig.3 Effect of temperature on the degree of crystalline texture and the expansion during heating.

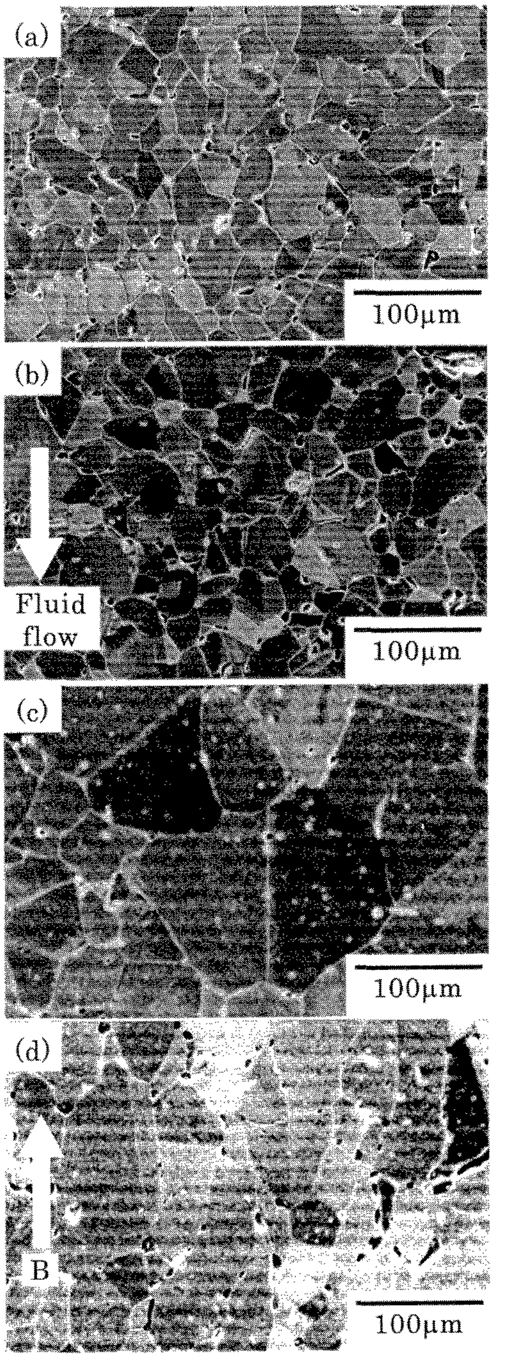

Fig.4 Microstructures of titania sintered at 1673 K. (a) and (b) are prepared by slip casting without a magnetic field. (a) is the surface perpendicular to the fluid flow and (b) is the surface parallel to the fluid flow. (c) and (d) are prepared by slip casting in a magnetic field (10T). The applied magnetic field was parallel to the fluid flow. (c) is the surface perpendicular to the magnetic field and (d) is the surface parallel to the magnetic field.

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