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Microstructure of alumina compact body made by slip casting

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Abstract

An attempt was made to understand the structural changes and particle-packing behavior of compacted alumina (α-Al_2O_3) made by slip casting. Packing structure of powder particles was examined in alumina compact made by slip casting with the crossed polarized light microscope in the transmission mode. Anisotropic structure was present in the specimen. A repeated change of color was noted with the rotation of the specimen at every 45° increments in the cross-section of compact cut parallel to the flow direction of water in the casting process. The alumina particles were aligned with the longest axis normal to the flow direction. The center of the cross-section showed a rather disordered packing structure. Isotropic optical property was noted in the specimen cut perpendicular to the flow direction. Isotropic microstructure was found in all directions of the compact made by the spontaneous drying process, in which less apt water flow occurred during drying. The development of anisotropic structure was insensitive to the dispersing state of particles in the slurry.

Keywords: Al_2O_3; Failure analysis; Slip casting; Optical microscopy; Water suction; Anisotropy

1. Introduction

This paper went for an explanation of the structural changes and the process-related particle-packing behavior of compacted alumina via slip casting route, by means of an optical polarization technique. Slip casting is one of the most promising forming methods and is widely applied for the commercial production of ceramics.[1,2] However, the method needs to be improved.[3] Deformation during sintering is one of the most serious problems. A possible origin of this problem is clearly the anisotropic packing structure of powder particles in green compact and must be inherent of this forming method. Particles of elongated shape, which is typical in raw powders of industrial grade and of non-cubic system, should develop this kind of structure under shear stress field in the forming operation. However, there is very limited understanding on the relevance among the dispersion of particle in slurry, the consolidation process of compact and anisotropic structures in green and sintered bodies in slip casting.[4–10] Although the conventional slip casting was done with the slurry of water dispersed raw powder, the process-related particle-packing behavior is essential subject to regardless of aqueous (water) or nonaqueous solvent system (ethanol, epoxy resin etc.).[11–16] It is very interesting to examine the packing structure of powder particles and its relevance to the characteristics of slurry and forming conditions in slip cast compact.

The new characterization technique is very promising for directly characterizing the anisotropic structures in green compacts as demonstrated in many papers.[17–28] Interesting characteristics of packing structure were revealed for the first time with the technique, such as the orientations of elongated Al_2O_3 particles in an injection-molded compact body,[21] in granules prepared by spray drying[22,24] and in the sintered alumina prepared by slip cast processing.[25] Also in nonaqueous system' green compacts, the method visualized the filler packing-structure which was contained in the epoxy composite system filled with ceramic particles.[26–28]

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The present study examines the structural changes and the process-related particle-packing structure in Al_2O_3 compact body made by slip casting. Particular focus is placed on the anisotropic packing features of elongated Al_2O_3 particles in compact, and its relevance to the dispersing state in slurry and the condition of water removal in forming process.

2. Experimental procedure

Details of the procedure for specimen preparation have been described in a previous paper.[25] In brief, a commercial low-soda Al_2O_3 powder was used as the raw material. The particle has an elongated shape as is typical of this type of powder. The powder was dispersed in an ion-exchanged water with a commercial dispersant containing ammonium polyacrylate (0–2 mass%) to form a slurry of 50 vol.% solids loading. The slurry was ball-milled for 24 h in an Al_2O_3 medium and vacuum-treated for 10 min for de-airing. A plaster mold was used to produce a compact (65×65×7 mm) by slip casting as shown in Fig. 1. The water was sucked from both sides of the largest faces of the compact. The figure also shows the expected packing structure of particles developed by this water flow schematically. There should be a neutral plane at the middle of the compact where the packing structure is isotropic. A specimen of the same size was also prepared by spontaneous drying on an aluminum foil. No rigorous flow of water occurred in this drying method. Compact was detached from the plaster or foil and dried at 110 °C for 24 h, then heated at 500 °C for 5 h, to remove the dispersant. The dried compacts then were calcined at 1000 °C to reinforce its strength for microscopic observations.

After made transparent by thinning and application of an immersion liquid, the internal structure of compact was examined with an optical microscope (Model POL-TP21, Nikon, Inc., Tokyo, Japan) in the transmission mode, using both normal and crossed polarized lights. A typical thickness of samples for observation was around 30 μm. Two cross-sections were examined; one was parallel and the other perpendicular to the direction of water flow in the casting process as shown in Fig. 1. A sensitive tint plate ($n = 530$ nm) was used to examine the optical anisotropy in the compact under the crossed polarized light transmission microscope with white light. Methylene iodide (RI = 1.74) was used as an immersion liquid.

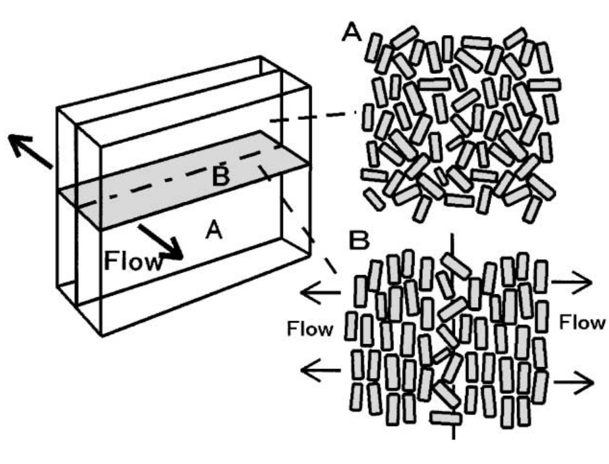

Fig. 1. Schematic diagram illustrating the shape of the body, the planes of the examination (A and B), the direction of water flow during casting process, and the packing structures of powder particles in planes (A and B) of the green body made by slip casting.

3. Results and discussion

Fig. 2 shows SEM micrograph of the powder applied in this study. The particles have a sub-micron size and an elongated shape. This non-equiaxed shape of the particle is very important for developing the anisotropic packing structure of particles in the compact under a water flow.

Fig. 3 shows the photomicrographs of Al_2O_3 compacts made by the slip casting of the dispersed slurry (dispersant 0.2 mass%). These photographs were taken on the parallel cross-section, i.e. the specimens for observation were cut parallel to the flow direction of water in slip casting. The compact showed a repeated change of color between yellow/pink/blue with its rotation for every 45° increments, except the narrow pink line at the center. This line was always noted in the

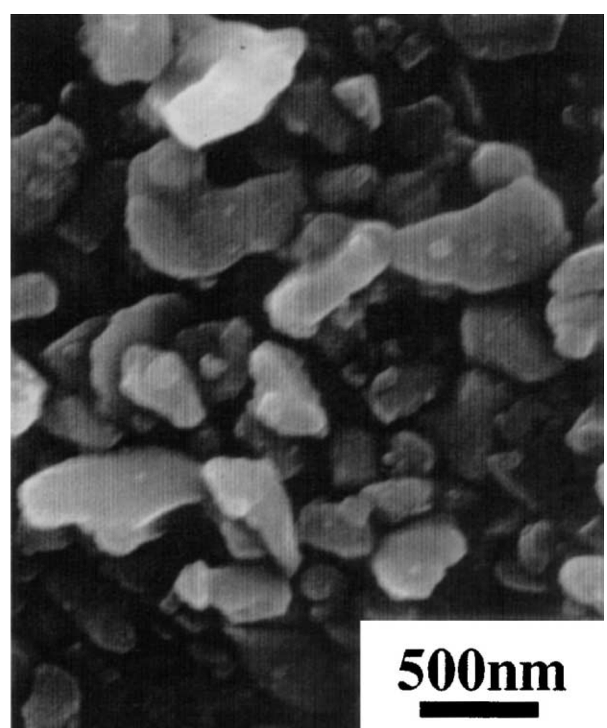

Fig. 2. Scanning electron micrograph of Al_2O_3 raw powder.

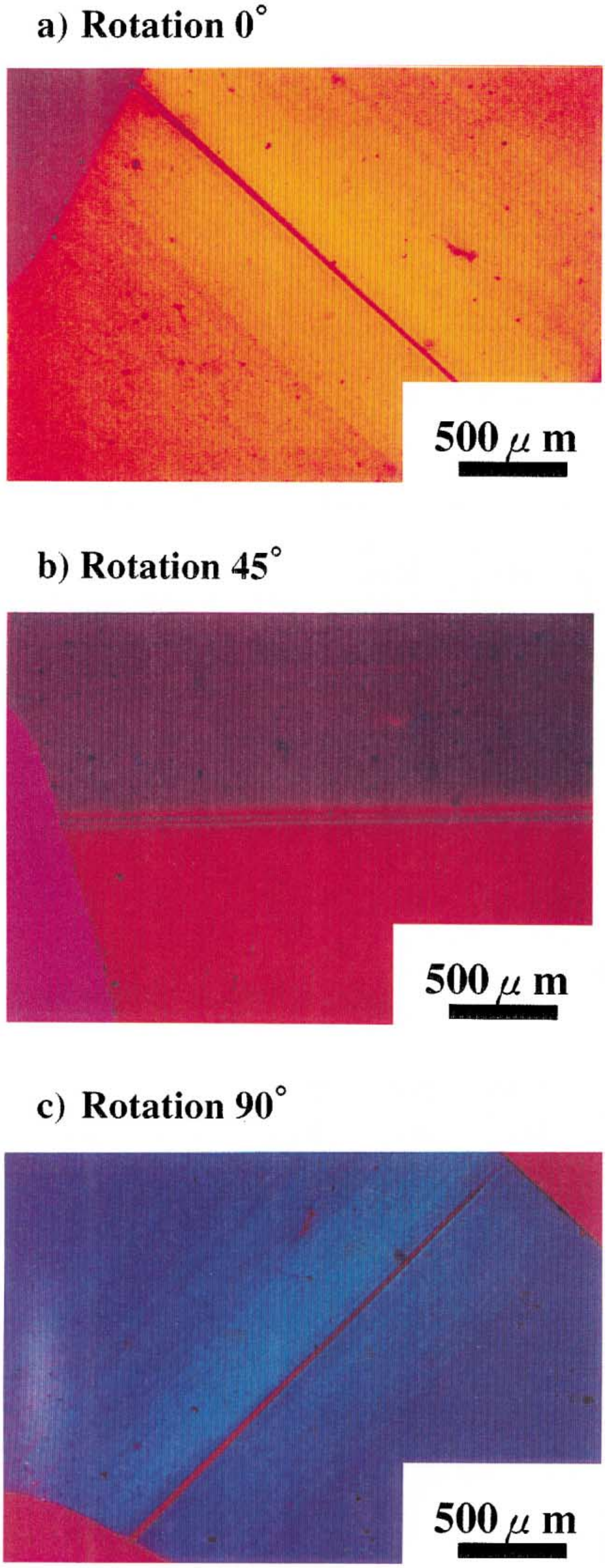

Fig. 3. Optical photomicrographs of Al_2O_3 compact bodies made by the slip casting of the dispersed slurry (dispersant 0.2 mass%). Photographs were taken at parallel cross-section, i.e. specimens were cut parallel to staple dehydrating direction. Photographs were taken in transparent crossed polarization with tint plate.

micrograph for all angles of rotation. The color change of the matrix corresponds to optical anisotropy; the subtraction and addition retardation. This anisotropy of optical property is clearly developed by the anisotropy in the packing structure of the matrix, i.e. aligned particles of elongated particle shape. The pink line at the center shows the optical isotropy of this region. The region of the mid-plane of the compact clearly has a disordered packing structure of powder particles, as expected. The presence of a few lines at both sides the pink line showed that the structure near the mid-plane is slightly different from other region of the matrix.

Fig. 4 shows the detailed observation for the central region in the parallel cross-section. Features marked as (A) and (B) correspond to large alumina particles of elongated shape. They show directly the direction of particle orientation in the region. The particle (A) located outside of the central line lays parallel to the central line. The particle (B) located inside of the central line is oriented not parallel to the central line. Additional observation confirmed that those results be general for the present compact. Clearly a majority of particles is oriented with their longest side parallel to the central line in the matrix of the compact. Particles located in the central line are randomly oriented.

It was also noted that the dispersing condition affected the structure only little. The packing structure was similarly anisotropic in the specimens made from the slurries with no dispersant and/or 2 mass% except the central region. This observation shows that the development of the anisotropic structure is inherent to slip casting.

Fig. 5 shows the similar photomicrograph of Al_2O_3 compact made with the dispersed slurry (dispersant 0.2 mass%) taken on the perpendicular cross-section; i.e. specimens were cut perpendicular to the flow direction of water in casting. The color remains pink for all angles

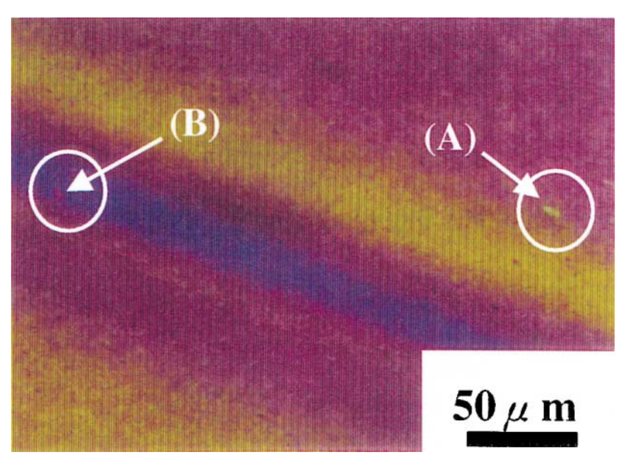

Fig. 4. Optical photomicrograph of Al_2O_3 compact bodies made by the slip casting of the dispersed slurry (dispersant 0.2 mass%). The photograph was taken at parallel cross-section, i.e. specimens were cut parallel to staple dehydrating direction. The photograph was taken in transparent crossed polarization with tint plate.

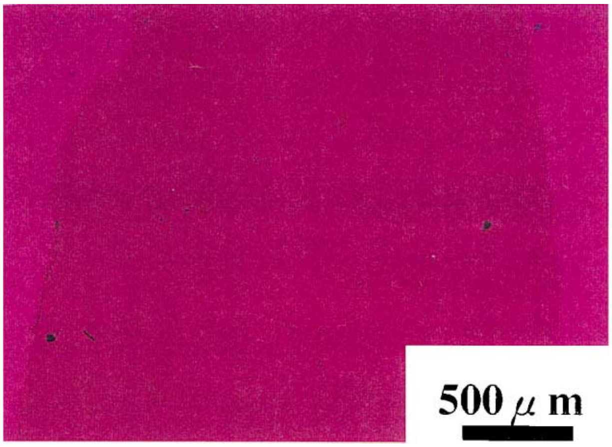

Fig. 5. Optical photomicrograph of Al_2O_3 compact bodies made by the slip casting of the dispersed slurry (dispersant 0.2 mass%). The photograph was taken at perpendicular cross-section, i.e. specimens were cut perpendicular to staple dehydrating direction. The photograph was taken in transparent crossed polarization with tint plate.

of rotation in this specimen. Viewed from this direction, the structure of the specimen is apparently isotropic. Alumina particles are randomly oriented in this cutting direction of specimen as expected.

Fig. 6 shows the photomicrograph of Al_2O_3 compact made by the spontaneous drying with the dispersed slurry (dispersant 0.2 mass%), in which the development of anisotropic structure is the easiest among all slurries examined in this study. The micrograph was taken on the cross section parallel to the flow direction of water in drying. The color remains bluish, and showed subtle changes of color with sample rotation. However, the intensity is pale rather than the slip cast, and the optical anisotropy was identical in all cut cross-sections. It is

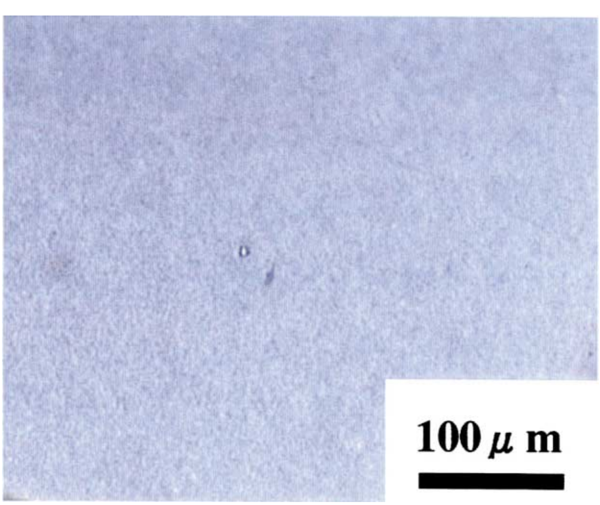

Fig. 6. Optical photomicrograph of Al_2O_3 compact bodies made by the spontaneous drying process of the dispersed slurry (dispersant 0.2 mass%). The photograph was taken at parallel cross-section, i.e. specimens were cut parallel to staple dehydrating direction. The photograph was taken in transparent crossed polarization with tint plate.

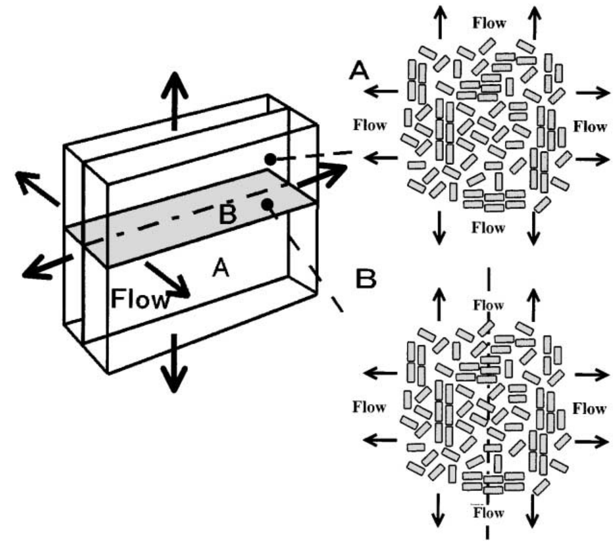

Fig. 7. Schematic diagram illustrating the shape of the body, the planes of the examination (A and B), the direction of water flow during drying process, and the packing structures of powder particles in planes (A and B) of the green body made by the spontaneous drying.

clear on isotropic microstructure. The flow of water in spontaneous drying is clearly too gentle to orient particles in slurry.

The present observation confirms the packing structure of powder particles in the slip cast compact shown schematically in Fig. 1. The rigorous flow of water in casting process is clearly responsible for the formation of the unique packing structure in the present specimens. Another important role should be assigned to the elongated plate-like morphology of corundum α-Al_2O_3 particles. The particles are carried with their longest side perpendicular to the flow direction of water in casting and sits on the powder bed previously deposited on the plaster mold. The powder bed thus deposited should have developed the anisotropic structure in this study.

Very little anisotropy in the spontaneously dried specimen (Fig. 6) shows that the gentle flow water in drying does not exert enough force on particle for orientation. Certainly, some orientations remain in the specimen, but it is not strong and identical in all cut cross-sections, as shown schematically in Fig. 7. There also may enough time for relaxation of particle alignment in this specimen. This orientation of particles appears to be inherent to the slip casting with elongated particle, and to be insensitive to the dispersant constituents or the dispersing state of the slurry.

4. Conclusions

An Al_2O_3 compact body made by slip casting inherently contained a characteristic packing structure of powder particles. Cross-section of the compact body cut parallel to the flow direction of water showed a repeated change of color between bright/dark and yellow/blue under rotating specimens at every 45° increments, except the central region. Particles of elongated shape align with their longest axis perpendicular to the flow direction in the matrix. Those in the central region align randomly in the structure. The packing structure of particles was apparently isotropic in the cross-section of compact cut perpendicular to the flow direction. The compact made by a spontaneous drying process showed weak anisotropic optical properties, but the anisotropy was identical in all cut cross-sections. The compact has isotropic packing structure. The rigorous water flow during slip casting is clearly responsible for the development of the anisotropic packing structure of particles. The development of anisotropic structure was insensitive to the dispersing state of particles in the slurry.

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