Characterization of Large Defects in Alumina Green Compacts with Liquid Immersion – Polarized Light Microscopy

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Abstract

We have confirmed that coarse particles and pores in alumina green compacts can be precisely detected with liquid immersion- polarized light microscopy. The experiments were performed on the green compacts containing artificially induced coarse particles and pores. An optimum calcination temperature was chosen to be compatible for adequate strength of specimen for handling and with no significant microstructural change by densification. The sample fixture was developed in order to prepare the thin specimen with consistent thickness to provide consistent images. The technique successfully reveals the coarse particles for both single- and poly-crystalline compacts with right shapes and sizes. Where coarse particles and pores co-existed, their images were clearly discriminated due to their anisotropic and isotropic behavior under polarized light.

KEY WORDS: Alumina, Coarse particles, Large Pores, Liquid immersion - Polarized light microscopy

1. Introduction

Large defects are virtually always present in the ceramic compacts and cause unpredictable variations of strength and other properties of the ceramics.[1)] They are formed as resultants of inadequate processing. Inhomogeneous structures in green compacts survive subsequent densification processes and persist in the final products.[2)] Characterization of these large defects in green compact is necessary to overcome the problems in ceramics processing before firing.

Previously, Uematsu *et al.* developed a direct characterization technique with transmission optical microscopy for the large defects in the green and the sintered ceramics.[2)- 6)] They reported very informative results for improving ceramics processing. They succeeded the qualitative as well as the quantitative evaluation of large pores in ceramics. Recently, they applied a cross polarized light microscopy in the transmission mode for determining particle orientation in alumina ceramics.[7), 8)] They found that the coarse particles can be observed as the large bright features in green compacts using this method. Coarse particles are one of the most detrimental defects in ceramics. It is well known that they strongly affect the sintering behavior[9), 10)] and the properties of ceramics.[11), 12)] However, for a better understanding of the effects in ceramics, applicability of the characterization method for various kinds of coarse particles should be further improved. Since there are various types of coarse particles in ceramics raw powders,[13)] additional discrimination is needed in order to clarify problems in processing.

In this study, applicability of the liquid immersion-polarized light microscopy for determination of various types of large defects in green compacts was examined. Artificial defects were intentionally employed for the examination. Attention was focused especially on sample preparation techniques for better observation of defects in green compacts. Reproducibility of sample preparation is necessary to obtain consistent images. For this objective, a novel sample fixture was developed in this study and its performance was examined by round robin experiment.

2. Experimental procedure

Alumina granules were prepared according to the procedure reported elsewhere.[6)] Two types of coarse particles, fused alumina and Bayer processed alumina, with the size ranging from 32 to 38 μm and polystyrene spheres of 40 μm are employed to make the artificial defects in green compacts. Mixture of granules and artificial defect sources were uniaxially pressed at 9.8 MPa and then cold isostatically pressed at 196 MPa to obtain green compacts. They were heated to form artificial pores by burning out the spheres and to provide adequate strength of specimen for handling in subsequent machining processes.

A novel sample fixture as illustrated in Fig. 1 was developed to prepare thin specimens with consistent thicknesses (150 μm in this experiment). The consistent

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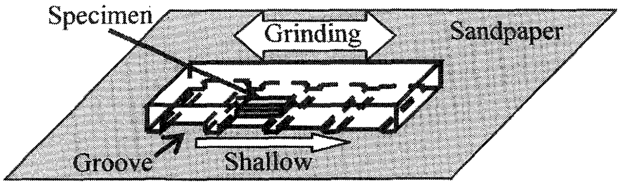

Fig. 1 Schematic illustration of the sample fixture and procedure of sample thinning

thickness is necessary to obtain consistent images. The fixture was made from dense and fine grained alumina ceramic to maintain groove depth by improving abrasive wear resistance.[15), 16)] Some grooves were formed on them by abrasive grinding with a conventional surface grinding machine and diamond wheel.

Specimens with the size about 5 x 5 x 5 mm^3 were taken from calcined compacts and were ground by sandpaper to 5 x 5x 1 mm^3. Then it was put between the sandpaper and the deepest groove of the fixture. The fixture was rubbed against the sandpaper with direction as shown in Fig. 1. Grit size of the sandpaper was decreased, step by step, from 400 mesh to 2000 mesh. After one side of the specimen was finished, the opposite side was ground again with the sandpaper of 400 mesh. The specimen was transferred into shallower grooves and ground again. The procedure was repeated to the target thickness. After the specimen was transferred to the groove with target depth, it was ground with decreasing grit size of sandpaper from 400 mesh to 2000 mesh.

The appearance of the coarse particles was examined with a SEM and a cross polarized microscopy. Characterization of large defects artificially induced in green compact was conducted with liquid immersion-polarized light microscopy. Methylene iodide was used as the immersion liquid.

3. Results

Figure 2 (a) shows SEM micrographs of the coarse particles used in this study. These appeared to be single particles for fused alumina and aggregates for Bayer processed alumina. Fig 2 (b) shows liquid immersion-cross polarized light micrographs of coarse particles. Essentially uniform brightness was noted for single fused alumina. Bright to dark, or inverse, transitions of these spots were noted as a result of sample rotation. The result indicates that each particle of fused alumina is essentially a single crystal. On the other hand, complicated structures consisting of bright and dark spots were noted for a particle by the Bayer process. Brightness transitions of these regions were also noted with sample rotation. These features indicate that the coarse particles are consisted of aggregated or agglomerated particles aligned to various directions. These characteristics must be noted in the green compact as abnormal contrast.

Shrinkage of green compacts with heating started from approximately 900 °C. 1100 °C was selected as the optimum calcination temperature due to following considerations. Specimens with adequate strength for

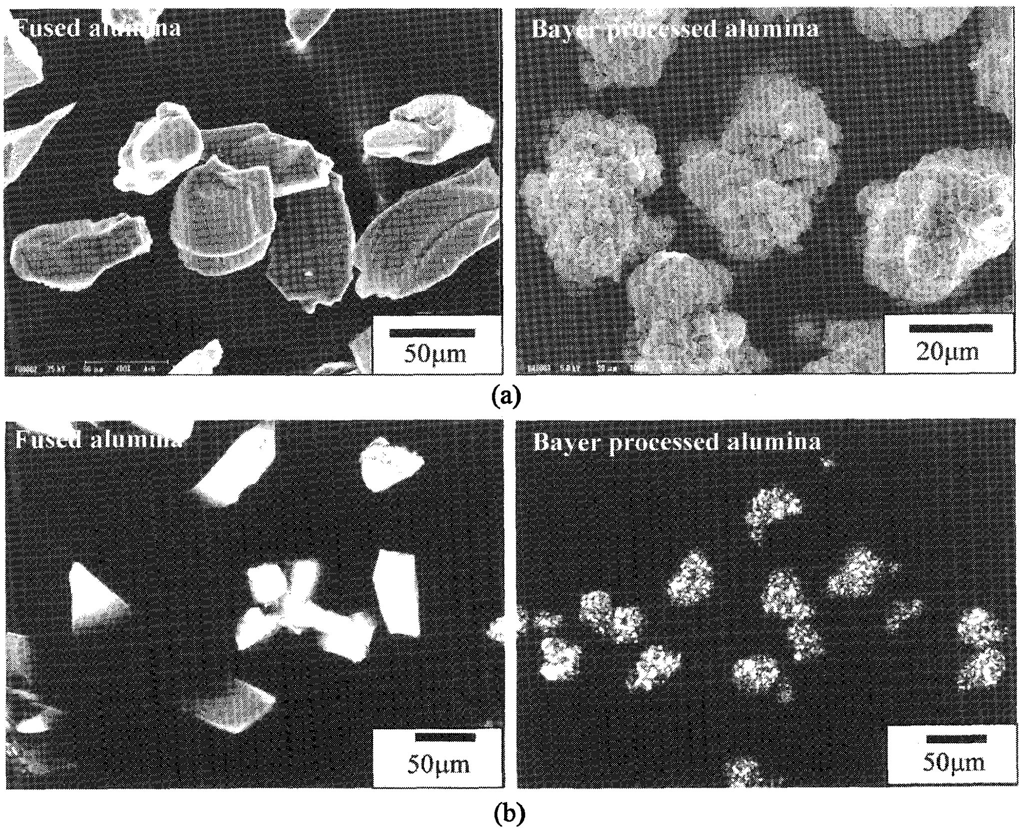

Fig. 2 (a) SEM micrographs of coarse particles prepared by different procedures
(b) Liquid immersion - cross polarized light micrographs of coarse particles

machining were achieved above 1100 °C and those calcined below this temperature broke during machining. The linear shrinkage of this specimen at 1100 °C was less than 1 %. On the other hand, that calcined above 1300 °C is hard for machining. Additionally, significant microstructural change is likely at this temperature, since linear shrinkage of the specimen exceeded 6 %.

Table 1 shows sample thickness prepared by three different investigators using the sample fixture shown in Fig 1. The groove depth of the fixture was set to 150 μm. Each value is the average and standard deviations of seven specimens. The thickness was very consistent between all investigators and was close to the targeting one. Reproducible preparation of better specimen for characterization was very difficult without the fixture.

Figure 3 (a) shows the internal structures of compacts containing the coarse fused alumina particles observed with the liquid immersion - polarized light microscopy. Many bright spots are noted. These spots repeated appearance and disappearance with sample rotation. The appearance is the same as for fused alumina coarse particles shown in Fig. 2 (b). Fig. 3 (b) shows the internal structure of compact containing the coarse aggregated particles. Abnormal contrast, similar to that shown in Fig. 2 (b) is observed. Their brightness also changed with sample rotation. These results suggest that the method is applicable for characterizing various types of coarse particles in green compacts.

Figure 4 shows the internal structure of a compact containing the coarse fused alumina particles and artificial pores. Some bright elongated spots and dark circular features are noted. The bright spots repeated appearance and disappearance with the sample rotation. The behavior is the same as fused alumina as shown in Fig. 3 (a). On the other hand, no brightness change was observed for circular features. The result indicates that the circular features have no optical anisotropy. Additionally, their size was exactly 40 μm in diameter, which is the same as the polystyrene spheres added to make the artificial pores. It can be concluded from the above results that the circular features corresponds to artificial pores.

It can be concluded that the method enables us to identify nondestructively the pores and coarse particles in green compacts, which are the major large defects in ceramics. The number of pores and coarse particles actually observed with the method agreed in order of magnitude with induced ones. The method is also applicable for quantitative evaluation of large defects in ceramics before firing.

4. Discussion

The liquid immersion - polarized light microscopy was successively applied for evaluation of both single crystalline and aggregated alumina coarse particles introduced into powder compacts. The method also enables us to discriminate nondestructively between those coarse particles and other defects such as pores because of their different optical behavior under polarized light.

Table 1 Thickness of the specimens prepared using the sample fixture

Investigator	Groove depth / μm	Thickness / μm
1		146±6
2	150	147±3
3		147±5

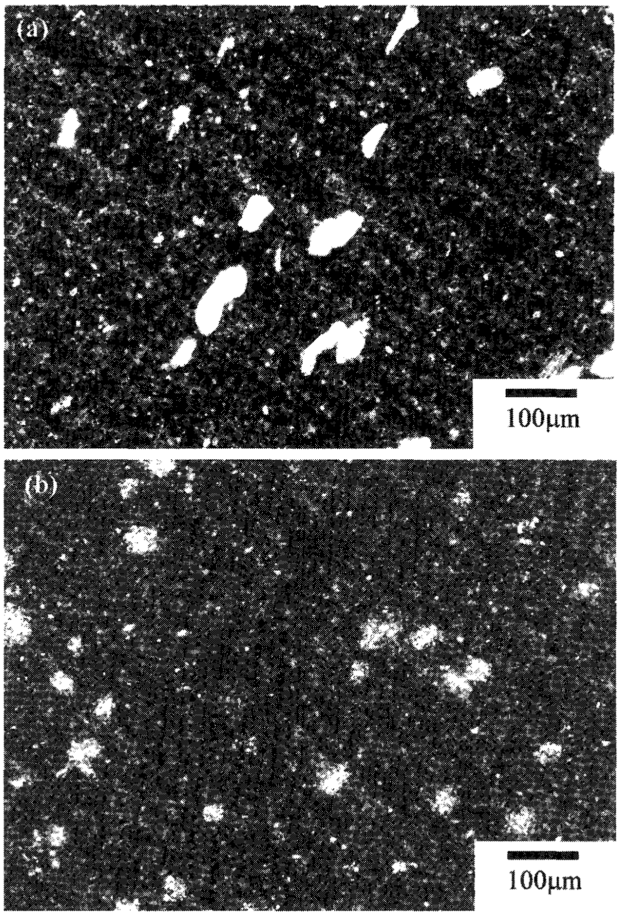

Fig. 3 Liquid immersion – cross polarized light micrographs of the compact containing (a) fused alumina and (b) aggregated alumina coarse particles

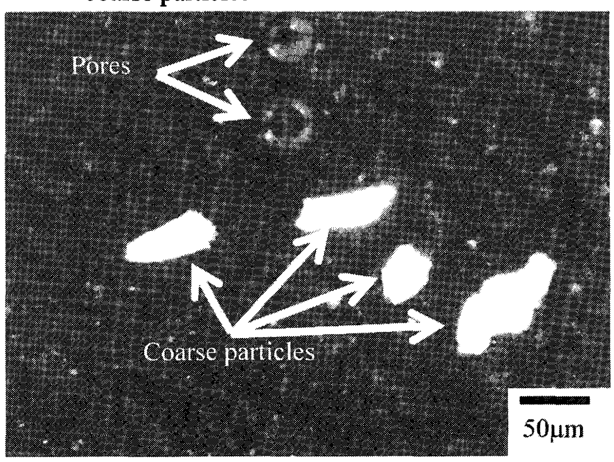

Fig. 4 Liquid immersion – cross polarized light micrograph of the compact containing fused alumina coarse particles and artificial pores

These constitute are the major defects in ceramics.

Preparation of samples with consistent thickness is crucial to achieve more qualitative as well as quantitative evaluation of large defects in green compacts. The sample fixture developed in this study enables not only experienced but also inexperienced researchers to prepare the thin specimen reproducibly.

It is very important that the method is applicable for characterizing various types of coarse particles in green compact as shown in Fig. 3 (b). Since there are various types of coarse particles in industrial grade low-soda alumina ranging from several hundreds to thousands of ppm.[13] Appearances of the coarse particles under the polarized light microscopy were very different as shown above. It may be due to their production route. Fused alumina is commonly prepared by crushing large alumina crystals. Hence they are essentially consisting of single crystals. While aggregated coarse particles were made through Bayer process.[14] Formation of microcracks on aluminum trihydroxide occurs during their thermal decomposition. Then, large aggregated alumina is formed from large mother salt. The crystallographic direction of alumina particles in the aggregate strongly depends on that in the mother salt. This fact indicated that the crystallographic direction of the aggregated alumina particles aligned in some direction. Therefore, the aggregated particles were found with complex abnormal contrast as shown in Fig. 3 (b).

5. Conclusions

The large defects such as coarse particles and pores in green compacts were successfully evaluated and discriminated with the liquid immersion - polarized light microscopy. The number of the large defects actually observed well agreed with that of added ones.

The sample fixture developed in this study enables ones to prepare the thin specimen with consistent thickness which is crucial for qualitative as well as quantitative evaluation of the large defects in green compacts.

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