Enhancing the contrast of low-density packing regions in images of ceramic powder compacts using a contrast agent for micro-X-ray computed tomography

Tsuyoshi HONDO, Zenji KATO and Satoshi TANAKA†

Department of Materials Science and Technology, Nagaoka University of Technology, 1603–1 Kamitomioka-machi, Nagaoka, Niigata 940–2188, Japan

In this study, a method for observing the internal pore structure of a ceramic powder compact using micro-X-ray computed tomography (micro-CT) was examined. Powder compacts have low densities, which makes it difficult to distinguish the low-density structures and large pores. In this study, the effect of a contrast agent (potassium iodide solution) on micro CT images was examined. First, the powder compact was made by die-pressing using alumina granules. Then, the powder compact was immersed into the contrast agent in a vacuum. As a result, the large pores in the powder compact were emphasized as white areas in the images.

Key-words : Alumina, Powder compact, Granule, Computed tomography, Contrast agent, Potassium iodide

In particular, optical microscope studies have revealed that coarse defects are formed in ceramics during the sintering or densification process because of nonuniformity of alumina powder compacts.18) Furthermore, for dry-pressed alumina ceramics, traces of the granules that remain after pressing are often transformed into coarser defects in the ceramics.9,13) Thus, an understanding of the effects of inhomogeneity in the powder compact is essential for improving the fabrication processes for engineering ceramics. But generalization of the results was difficult due to the limitation of characterization technique. Recently, significant advances have been made in micro-focus X-ray computed tomography (micro-CT).14,15) Accordingly, micro-CT has begun to be used in the ceramics field to analyze the internal structure in dental materials, etc.17)–21) Micro-CT is also a useful tool for nondestructive and three dimensional visualization of bone and tooth structures.19) In micro-CT images, the contrast reflects differences in the X-ray absorption. However, in powder compacts, the large pores and loose packing structure are difficult to distinguish because of their similar degrees of X-ray absorptivity.22,23) To enhance the contrast, various agents such as isopropyl or osmium solutions are used to provide radiopacity for observations in living bodies.24,25) Inspired by this technique, the objective of this study is to examine potassium iodide as a contrast agent to make the internal coarse defects in alumina powder compacts more clear in micro-CT images. Iodine is among the most common contrast agent for enhancing X-ray images.26) In particular, potassium iodide aqueous solutions have low viscosity, high absolute iodine content, and a lower price.

Commercial alumina granules (AKS20 Sumitomo Chemical, Japan) were used as the raw material. Median size D50 of granules is 65 μm. First, the granules are poured in a metal die of 10 mm in diameter and uniaxially pressed at 40 MPa. The powder compact was then densified by cold isostatic pressing at 190 MPa. The de-binder process was carried out by heating the compact at 800°C with a heating rate of 3°C/min in air. The bulk density of the de-binderd powder compact was measured from its size and weight. The relative density of the samples after the de-binder treatment was 57%. This level of densification of the powder compact in the forming process is typical for these materials.

The compact was then cut into a small piece of ~ 1 mm × 1 mm × 2 mm in size and observed using a scanning electron microscope (JEOL5310, JEOL, Japan). Besides, the compact was observed by an optical microscope. Sample was thinned to 100 μm, and made transparent by an immersion liquid, methylene iodide, whose refractive index is 1.74 and nearly same with that of alumina 1.76.

As mentioned above, micro-CT distinguishes the components of different materials based on their differences in X-ray absorption. At the most basic level, a tomographic reconstruction of a projection image provides us with a 3D map of the X-ray absorption in which each voxel (volume element, or pixel, of the 3D map) represents the X-ray absorption at that point. Because different material features and phases often have different X-ray absorption properties, we can easily identify the different features from these images. For X-ray micro-tomography, the spatial resolution of these 3D maps or images can approach less than 1 μm.

For this study, the sample was immersed in 1 M potassium iodide aqueous solution (KI solution) in a vacuum desiccator for 10 min and dried for an hour. The same sample was also imaged without KI solution as a reference. The sample was then set in a high-resolution micro-CT instrument (Skyscan 1172 Bruker AXS, USA). The voltage and current of the X-rays were 100 kV and 100 μA, respectively, and the focus size of the X-rays was 0.7 μm according to the catalog data. The cross-sectional image was reconstructed from the data using commercial software.

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Corresponding author: S. Tanaka; E-mail: stanaka@vos.nagaokaut.ac.jp

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The 3D image was also constructed using commercial software (CTvox, Bruker AXS, USA). The 3D image was also constructed using commercial software (CTvox, Bruker AXS, USA).

Figure 1 shows an SEM micrograph of the fracture surface of the powder compact. The granules were deformed by the pressing, and interstices among granules were observed clearly. The sintering of primary particles does not proceed at 800°C, but the particles remained well packed in the granules.

Figure 2 shows optical image of the sample taken by the optical microscope with transmitted light. For the whole, traces of the granules with diameters of several tens of micrometers were observed well. The image reveals that the granules remain after pressing as mentioned in past studies. However, packing structure at the boundary between granules are ambiguous.

Figure 3 shows cross-sectional images of the sample prepared without KI taken using micro-CT. First, the concentric circles with the max radius of 1000μm are artifacts with ring shape, which are caused by the micro-CT apparatus. These artifacts appear on the axis of the rotation axis of sample stand. And these are able to be ignored. In this image, pores and low-density regions are observed as darker than dense areas, because pores do not absorb X-rays. A few small pores can be observed as black areas. In addition, ring-shaped pores with diameters of several tens of micrometers were also observed. These ring-shaped pores were due to interstices among granules. As for the low-density regions, they can be explained as resulting from de-binder treatment, as observed in previous studies. In the spray drying process, it is well known that the binder is segregated to the surfaces of the granules, and after heating at 800°C, the areas containing the binder become low-density packing structures.

Figure 4 shows cross-sectional images of the sample prepared with KI solution. The position observed is almost same as that in Fig. 3. Here, pores and low-density regions are observed as brighter than the dense areas, because the KI in the pores or low-density packing structures absorbs X-rays well. In contrast, the alumina particles appear relatively dark. Thus, in this figure, the gray areas are alumina granules. These areas appear among white regions resembling dendrite crystals. As shown Fig. 4, the interstices in the low-density packing structures were emphasized by the KI solution. In particular, the shapes of granules with sizes of several tens of micrometers could be observed in detail in the micro-CT images.
several locations. In addition, ring shape artifacts as shown in Fig. 3 did not appear by enhancement of contrast by KI here.

Figure 5 shows an image adjusted to emphasize the contrast in Fig. 4. In this figure, it can be confirmed that KI is present throughout the entire cross-section of a sample. Figure 6 shows the 3D pore structure in the powder compact. As was apparent in Fig. 5, the low-density structures are spread not only two-dimensionally but also three-dimensionally.

Thus, we have demonstrated that in X-ray computed tomography, the use of contrast media is effective for visualization of low-density regions in porous materials such as the powder compact. The low-density structure could be observed to be spread three-dimensionally in the compact. Compared to SEM or optical microscopy, the novel information as mentioned above is obtained with non-destructively by micro-CT with enhancement technique. Here, we used potassium iodide solution as the contrast agent. However, the appropriate contrast agent for a different material could be selected by considering the properties of the matrix. For example, potassium iodide may be useful for materials with light elements as alumina, carbon, boron carbide, silicon nitride, etc. Further studies on powder processing of ceramics using this method will be conducted in the near future.

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