Prediction of strength based on defect analysis in Al₂O₃ ceramics via non-destructive and three-dimensional observation using optical coherence tomography

Fumika SAKAMOTO¹, Takuma TAKAHASHI², Junichi TATAMI¹,²,³ and Motoyuki IIJIMA¹,²

¹Graduate School of Environment and Information Sciences, Yokohama National University, 79–7 Tokiwadai, Hodogaya-ku, Yokohama 240–8501, Japan
²Kanagawa Institute of Industrial Science and Technology, 705–1 Shimoizumi, Ebina, Kanagawa 243–0435, Japan

Non-destructive analysis is important in terms of improving the reliability of ceramics. In the study, the internal structure of Al₂O₃ ceramics with artificial pores was observed via optical coherence tomography (OCT). The OCT observation was performed on a surface perpendicular to the tensile surface of the specimen. The spherical pores were successfully observed via OCT. The strength and fracture origin were predicted from the size and position of the observed pores. The lowest predicted strength (which is realized as the strength of the specimen) was in agreement with the bending strength obtained via three-point bending tests. The actual fracture origin observed by scanning electron microscope was also the same as that predicted via the OCT observation. Thus, the results indicated that the nondestructive testing via OCT was useful in terms of predicting the strength and fracture origin of ceramics.

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1. Introduction

Ceramics are indispensable materials in many applications because of excellent thermal, chemical, electrical properties.¹⁻³ However, further improvement in mechanical reliability, such as higher strength and less variation in strength, are required because ceramics are more brittle than metals and polymers.⁴⁻⁵ Based on fracture mechanics, the strength of a brittle solid is derived as a function of the fracture toughness, defect size, and shape factors based on the shape of the defect, and this is termed as the Griffith equation as follows:⁶

\[
\sigma_f = \frac{K_{IC}}{Y\sqrt{a}}
\]  

(1)

where, \(\sigma_f\) denotes the strength, \(K_{IC}\) denotes the fracture toughness, \(a\) denotes the crack length, and \(Y\) denotes the shape parameter. Based on this equation, the strength decreases if the size of defect in the ceramics increases. Furthermore, the strength depends on the size of the defect and also on the existing position because the stress in the component is not always uniform.⁷⁻⁸ Therefore, in order to improve the mechanical reliability of the ceramic components, it is necessary to quantitatively evaluate the size and position of the defects as fracture origins.

In previous studies, observation of fracture origins was conducted via various techniques. The most common technique corresponds to fractography in which the fracture origin is identified from the appearance of the crack propagation and its size and position are estimated.⁹ Although it is possible to determine the type, size, and position of the fracture origin via fractography, it is impossible to nondestructively identify the fracture origins. Defects in ceramics are also observed via optical microscopy. Uematsu et al. proposed an observation technique for the internal structure of ceramics via a transmission optical microscope in which a specimen was processed to a thin piece of approximately 200 μm and observed under a microscope with transmitted light to observe defects in the ceramics.¹⁰⁻¹¹ They have reported that variation in the bending strength of ceramics was successfully explained via the defect size distribution obtained by the method. Specifically, infra-red light exhibited better transmission than visible light in terms of observing the interior. An ultrasonic microscope was used to observe defects in ceramics.¹² In the technique, although information in the depth direction was obtained and it was possible to specify cracks, a liquid was required to observe the internal structure of ceramics with high accuracy. Recently, X-ray CT is
also applied for the internal observation of ceramics. It is possible to three-dimensionally observe the internal structure, and the size and position of the defect are estimated via nondestructive inspection. However, estimation of an internal structure with higher accuracy requires more time and a smaller specimen. Therefore, in either method, it is impossible to observe the material in a normally existed state with higher accuracy and non-destruction. However, it is necessary to devise an inspection technique for non-destructive monitoring of the defects in the ceramics, which has a high resolution to detect defects as fracture origin and facilitates three-dimensional observation to identify the size and position of the defects and high-speed scanning for better productivity.

Optical coherence tomography (OCT) is a visualization technique that uses coherent light even in opaque materials with micrometer-resolution and three dimensions. It is developed in the medical field for examination of fundus eye and periodontal disease. The recently-developed swept source OCT (SS-OCT) (which used in this study) exhibits faster imaging time delay than the other OCT systems. Initially, a single depth profile termed as A-scan is performed. Subsequently, two-dimensional cross-sectional imaging (termed as B-scan) is obtained by laterally scanning the light and correcting the sequential A-scan. Finally, a three-dimensional image (termed C-scan) is also obtained by two-dimensional scanning of the light nondestructively. The depth at which information can be obtained with A-scan is determined by the refractive index of the material and the laser’s ability. When Al₂O₃ is observed with commercially available OCT, the typical coherence length, namely observable depth, is 2.3 mm. If B-scan and C-scan are performed 1 mm each, and the observation interval is 3 mm, the observation ends at 5.5 s. In previous researches, microchannels embedded in Al₂O₃ and the interface of the Al₂O₃/ZrO₂ laminate using OCT were observed. It is also reported that spherical artificial pores with dimensions of 50 μm at a depth of 700 μm were observed from the surface in Al₂O₃. The prediction of strength based on defect analysis in ceramics using OCT is not performed to date. However, OCT is a useful tool to observe defects in ceramics because OCT can obtain high-speed, high-resolution, non-destructive, and three-dimensional observations.

The objective of the study involves clarifying the usefulness of OCT in observation of defects as fracture origins in ceramics. Hence, Al₂O₃ ceramics with artificially introduced spherical pores as typical examples of defects in ceramics were prepared, and their internal structure was observed via SS-OCT. The results indicated the size and position of the pores, and thus the fracture origins and strength were predicted and compared with those actually estimated via three-point bending tests.

2. Experimental procedure

In the study, 1 vol% of spherical carbon particles (BEAPS-C180, Asahi Organic Materials Co., Tokyo, Japan, Average particle diameter 180 μm) were dry mixed with Al₂O₃ granules (AKS20, Sumitomo chemical Co., Osaka, Japan) in a mixer (Turbula Shaker Mixer, Shinnmaru Enterprises Co., Osaka, Japan) for 30 min. The powder mixture was subsequently compacted using a WC/Co die at a pressure of 50 MPa, and this was followed by cold isostatic pressing at a pressure of 200 MPa. The Al₂O₃ ceramics were fabricated by heating the green body at 1600°C for 2 h in air. Specimens with dimensions corresponding to 18 mm × 1.5 mm × 2 mm were obtained via grinding. The edge of the specimens were chamfered.

SS-OCT (IVS-2000-WR, Santec Co., Aichi, Japan) with a center wavelength of 1.3 μm and a wavelength sweep width of 170 nm was used to observe the internal structure of the sintered body. As shown in Fig. 1, the width, length, and thickness of the specimen were defined as the x, y, and z axes, respectively. The position of the origin was on the tensile surface and the side of the specimen, and under the loading point in the three-point bending test. B-scan in the xz plane via OCT was carried out in the range of 2.4 mm in the x-axis direction, and 1.4 mm in the z-axis direction, respectively. The center of C-scan to y-axis direction was below the loading point of three-point bending test. The rage of C-scan was 1.7 mm in the y-axis direction. Additionally, the sampling space of B-scan and C-scan corresponded to 2.9 μm/pixel. The time required for three-dimensional observation in the volume of 2.4 × 1.7 × 1.4 mm was approximately 30 s. Pores affecting the strength were observed on a plane perpendicular to the tensile plane in the three-point bending test, and thus the strength was predicted from size and position of the pores in the OCT.
image on the $xz$ plane from the calculation method described later. In order to detect the pore, the brightness and contrast of the obtained OCT images were adjusted, and the portion where reflection occurred was highlighted using image processing software [Premier pro CC (Adobe Systems Inc., CA, U.S.A)]. Three specimens (specimen A, B, C, hereafter) were used in this study.

A three-point bending test was performed on a specimen after the OCT observation. The three-point bending set-up included a span length of 15 mm and crosshead speed of 0.25 mm/min. The maximum tensile stress is calculated from the measured maximum load and the size of the specimen based on Eq. (2) as follows:

$$\sigma_t = \frac{3PL}{2wt^2}$$

where $\sigma_t$ denotes the bending strength, $P$ denotes the maximum load at failure, $L$ denotes the distance between the lower supports, $w$ denotes the width of the specimen, and $t$ denotes the thickness of the specimen. The bending test of the specimen was conducted to measure the maximum tensile stress. The fracture surface of the specimen after the three-point bending test was observed via scanning electron microscope (SEM, JSM-6390LV, JEOL Co., Tokyo, Japan) and confocal laser scanning microscope (VK-X200, Keyence Co., Osaka, Japan), and the fracture origin was identified.

The fracture toughness was measured via the surface crack in flexure (SCF, hereafter) method. Specifically, Al2O3 ceramics which are prepared by the same process as the stated above without any carbon particles were cut into $3\, \text{mm} \times 4\, \text{mm} \times 30\, \text{mm}$ samples, and the surfaces were mechanically polished. A crack was induced via a Knoop indenter under loads corresponding to 40 N. The specimen was ground using diamond slurry until the indentation and associated residual stress field were removed. The fracture stress was measured via a three-point bending test with a span of 30 mm and a crosshead speed of 0.5 mm/min. The fracture toughness is calculated using Eq. (1) and the following equations:  

$$K_{IC} = Y\sigma_t\sqrt{a}$$

$$Y = \frac{\sqrt{\pi MH_2}}{\sqrt{Q}}$$

$$M = \left[1.13 - 0.09\left(\frac{a}{W}\right)\right]$$

$$+ \left[0.54 + \frac{0.89}{\left[0.2 + (a/c)\right]} + 14(1 - a/c)^{24}\right] \cdot (a/W)^4$$

$$H_2 = 1 - \left[1.22 + 0.12\left(\frac{a}{c}\right)\right] \cdot \left(\frac{a}{W}\right)$$

$$+ \left[0.55 - 1.05(a/c)^{0.75} + 0.47(a/c)^{1.5}\right] \cdot (a/W)^2$$

$$Q = 1 + 1.464(a/c)^{1.65}$$

where $a$ denotes the crack depth, $2c$ denotes the crack width, $W$ denotes the specimen height, and $\sigma_t$ denotes the fracture stress. The values of $a$ and $c$ were measured via optical tomography observation.

### 3. Results and discussion

Figure 2(a) shows the original OCT image on the $xz$ plane of the specimen A where pores affecting strength perpendicular to the tensile direction are observed. In the study, a section of the specimen with a volume corresponding to $2.4 \times 1.7 \times 1.4 \, \text{mm}$ was observed via OCT. The top of the image is the tensile surface of the specimen. The scale of $z$-direction is displayed in consideration of the refractive index of Al2O3 (1.77). In the OCT image, the region wherein the higher reflection occurred is displayed more brightly. A pair of upper and lower white contours was observed in the enclosed part as shown in the dashed line of Fig. 2(a). The added spherical pore formers disappeared during firing, and thus spherical pores are formed in the Al2O3 ceramics. In the OCT observation, laser light is reflected on the surface of the spherical pore, and it passes through the pore and only a low amount of reflection occurs on the side surface, and this is followed by reflection again on the bottom surface. Therefore, the results indicate that a pair of brighter contours observed in the OCT image corresponds to a pore. Figure 2(b) shows the image processing applied to the original image. Hereinafter, the image subjected to the processing is shown as an OCT image. The pore in the processed OCT image [Fig. 2(d)] is observed more clearly than that in the original one [Fig. 2(c)]. The observed pore looks not exactly circular but slightly elliptical. The length in A-scan direction in an OCT image is inverse proportion to the refractive index and air having smaller refractive index than that of Al2O3 exists in the pore. In consideration of the difference in the refractive index, Fig. 2(e) indicates the image multiplied by 1.77 to the depth direction. As a result, aspect ratio of the pore in the OCT image became 0.98 and the shape of the observed...
pore is regarded as a circle, which means that the pore can be assumed as a sphere.

Typical six pores with larger sizes (when compared to those observed in the measurement range) in the specimen A are shown in Fig. 3. The size and the position of the pores are summarized in Table 1. The diameter of the pore was measured when its geometry was assumed as a circle. Additionally, the white reflected part was measured by determining the center of the reflection area. The average pore size as listed in Table 1 is 132 µm. Given that the green density is 60%, the shrinkage of the spherical pores corresponded to approximately 20% after densification. This implies that the size of the pore formed using pore formers of 180 µm was estimated as 146 µm, and this was in good agreement with the pore size measured by OCT. Based on Evans et al., penny shaped cracks are generated in the circumferential direction around the spherical pore.²²) Figure 4 shows the SEM image of an enlarged view of the surface of an artificial pore observed on the fracture surface in the study. Thermal grooving like a crack was clearly found on the surface of the spherical pore. Confocal laser scanning microscopy image of the surface of an artificial pore on the fracture surface and the surface profile measured from the image are shown in Fig. 5. The
surface profile was corrected in consideration that the pore surface was sphere. As reported in the previous study, the thermal grooving was confirmed to be sharp like crack. In consideration that the pore is spherical, it is reasonable to consider the pore as a penny shape crack though it is not exactly a crack. Therefore, in the study, it was assumed that the spherical pore observed via OCT corresponded to penny shape cracks with the same diameter as the pore. Hence, the strength (wherein it was assumed that the specimen was broken from each pore assumed as a penny shape crack) was predicted based on fracture mechanics.

Initially, we estimated the maximum tensile stress $\sigma_{\text{max}}$, which was applied to a pore assumed as a penny shape crack to ensure failure. In the study, the pores were divided into two types. The first type corresponds to a pore that is present inside the sample. We assumed that all of the pore of this type corresponds to penny shape cracks in the finite field. However, the stress intensity factor in this case has not been reported. Conversely, the stress intensity factor, $K_1$, of the penny shape crack in the infinite body is expressed in Eq. (8) as follows:

$$K_1 = 2\sigma \sqrt{\frac{a}{\pi}} \quad (8)$$

where, $a$ denotes the radius of the crack and $\sigma$ denotes the stress applied to the crack. On the other hand, the stress intensity factors to a penetrate crack in semi-infinite bodies are reported. [Eqs. (9) and (10)] The expressions are as follows:

$$K_1 = F_1 \sigma \sqrt{\pi a} \quad (9)$$

where, $a$ denotes the half of the length of the penetrate crack, $b$ denotes the distance from the center of the crack to the surface, and $C_n$ denotes coefficients of $(a/b)^n$ depending on $n$. In the case of an infinite body, the shape factor of the penny shape crack is $2\sqrt{\pi a}/\pi$ as per Eq. (8). With respect to penetrating cracks, $b$ corresponds to $\infty$ in Eq. (10) and $F_1$ corresponds to 1, and thus the coefficient of $\sigma$ is $\sqrt{\pi a}$ based on Eq. (9). A comparison of both the coefficients indicated that the stress intensity factor of the penny shape crack in the infinite body was $2/\pi$ times as much as those of the penetrant crack in the infinite body. We assumed that the relationship also holds in semi-infinite body, and thus the stress intensity factor of penny shape cracks in semi-infinite body was expressed in Eq. (11) as follows:

$$K_1 = \frac{2}{\pi} F_1 \sigma \sqrt{\pi a} \quad (11)$$

In the study, the stress intensity factor of the penny shape crack present in the ceramics was calculated by using the above equation.

The stress applied to the pore to be broken was estimated using the fracture toughness measured by the SCF method. The crack grows at the point where the stress intensity factor equal to the fracture toughness. When the stress intensity factor decrease after the crack grows, crack propagation should arrest. At this time, the increase in the applied stress is needed for more crack propagation. The maximum stress, $\sigma_{\text{max}}$, applied to the pore was estimated as the stress when the crack unstably propagated.

The other type corresponded to a pore exposed on the surface. This was considered as a semi-elliptic crack in a manner similar to that when the spherical pore present inside was considered as a penny shape crack. The stress intensity factor is estimated using the Newman-Raju equation. The maximum stress applied to the pore before unstable crack growth was estimated in a manner similar to the penny shape crack inside the specimen.

The distribution of tensile stress existed in the three-point bending test specimen. In this case, the maximum stress applied to the specimen (i.e., the bending strength of the specimen) did not always correspond to the maximum stress applied to the pore. The stress in the specimen was expressed as a function of the maximum stress of the specimen and position. Based on beam theory, tensile stress of a position in the three-point bending test specimen was calculated. In the study, based on the results, the bending strength, $\sigma_{\text{c}}$, is estimated as follows:

$$\sigma_{\text{c}} = \sigma_{\text{max}} \times \frac{L}{\sqrt{\frac{L}{2} - |y|}} \times \frac{t}{\sqrt{\frac{t}{2} - |z|}} \quad (12)$$

where, $y$ and $z$ denote the position of the pore, $\sigma_{\text{max}}$ denotes the maximum tensile stress applied to the pore.
before unstable crack propagation, $L$ denotes the length of the lower span, and $t$ denotes the thickness of the specimen.

The predicted strength calculated based on the shape and position of the pores in specimen A is listed in Table 1. The expected strength of each pore depended on the size and position. Based on the weakest-link assumption, the pore with the lowest strength should correspond to fracture origin, and the strength from the pore should correspond to the actual strength of the specimen in the bending test. Thus, the results indicated that pore No. 2 should be the fracture origin among the pores shown in Fig. 3, and the bending strength of the specimen should be 300 MPa. In this case, stable crack growth did not need before unstable crack propagation. In the actual three-point bending test, the bending strength of specimen A calculated from the maximum fracture load and the size of specimen corresponded to 299 MPa. This was in agreement with the predicted value. **Figure 6** shows a comparison between the OCT image showing the pore of No. 2 in the specimen A and SEM image of the fracture surface. The results indicated that the pores in OCT and SEM images exhibited the same size and existed at the same position.

The strength was also predicted as listed in Table 2 for the other samples. The predicted strength was in agreement with the actually measured strength. In sample C, the fracture origin was not exposed on the surface of the specimen. In this case, the strength and fracture origin were successfully predicted from the OCT images. The fracture origin as observed via the OCT and SEM images is in good agreement as shown in **Fig. 7(a)**. This indicated that the OCT observation predicts the bending strength, and it was confirmed that the pores predicted as fracture origins from the OCT observation corresponded to the fracture origins in an actual fracture test. Two pores were observed on the fracture surface. The reason for the difference between the OCT and SEM images is that a crack did not propagate straight due to stress concentration at the other pore, namely the actual fracture surface was not even as shown in **Fig. 7(b)**.

### 4. Conclusions

In the study, artificially induced spherical pores in Al$_2$O$_3$ ceramics were observed via OCT. The strength of ceramics with pores predicted from the size and position estimated by OCT was in agreement with the experimentally measured strength. The OCT observation also identified the fracture origin. The results indicated that 3D OCT observation of the internal structure of ceramics is evidently useful.

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