Estimation of Weibull modulus from coarser defect distribution in dry-pressed alumina ceramics

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Strength variation of alumina ceramics formed by dry-pressing of granules was examined by the number density of coarser defects. The Weibull modulus \( m \) was related to the exponent \( n \) of the power function of the number density of coarser defects by strength simulation; \( m = 1.82n - 1.92 \). The Weibull modulus was determined by a series of strength data, which were calculated by the simulated bending tests of the samples. The number density of coarser defects was given by the assumed power function. The various Weibull moduli were obtained for some kinds of series. The experimental results of the number density of coarser defects by direct observation of alumina ceramics and the Weibull modulus from strength tests were plotted well on the calculated line except a series of sample containing coarser defects with ambiguous boundaries. The relationship between the Weibull modulus and the parameter of the power function concurred with past studies; \( m = 2n - 2 \).

Key-word : Weibull modulus, Strength, Coarser defects, Alumina, Dry-pressing

1. Introduction

Unpredictable variation of strength is one of the most serious problems for application in structural ceramics. Coarser defects with the size typically over 50 \( \mu \)m are formed in ceramics during the manufacturing process and are fatal in the reduction and variation of the strength. The number of coarser defects and their positions varies for each sample in ceramics. This causes the scattering of strength.1,2 The Weibull modulus is given by the slope of the proposed equation. Matsuo et al. also presented a statistical approach for estimating the Weibull parameters from the crack-size distribution function and the fracture toughness without fracture stress data.3,4 The Weibull modulus \( m \) is given by the slope of the proposed equation.

In our recent study, we have reported explicitly that coarser defects with the size over 50 \( \mu \)m governed the variation of both flexural strength and the distribution through theoretical and experimental approaches in an alumina ceramic system.5–8 The size distribution of coarser defects was obtained experimentally by observation of the internal structure on thin samples with a transmission optical microscope. The distribution was found to follow a power function. The morphology of coarser defects was spherical and crack-like with several branches, and has been related to the manufacturing process, i.e., their origin is the granules of size about 50 \( \mu \)m and their morphological changes during dry-pressing and sintering.9–13 The strength and their variation (the Weibull modulus \( m = 12.7 \)) were simulated precisely based on the linear fracture mechanics using coarser defects size distribution.14 The results of the simulation agreed extremely well with the experimental strength and its variation (\( m = 12.4 \)).

In this study, the objective is to relate the coarser defect size distribution to the flexural strength variation (the Weibull modulus \( m \)) by simulation and experimental approaches. First, the number density of coarser defects is assumed as a power function and the range of coarser defect size are above 50 \( \mu \)m. The flexural stresses for hundreds of samples are calculated with this number density of coarser defects. Second, the parameter of the coarser defects functions are related to the Weibull moduli for each material. Finally, the validity of the simulated results is compared with the experimental results of the Weibull modulus in the flexural test and the parameter of distribution function of the
observed coarser defects.

2. Calculation of strength variation derived from the coarser defect size distribution

2.1 Calculation procedure

The flexural strength data is calculated from the simulated four-point bending tests based on the fracture mechanics using several hundred samples with coarser defects. The procedure of simulation is the same as our past study. Figure 1 illustrates the model specimen for the four-point bending tests. All defects are assumed as penny shape because coarser defects are composed of a group of pores and cracks rather than a simple pore in ceramics produced through the dry-pressing process using the granules. They are assumed to orient perpendicular to the direction of tensile stress in the flexural test in Fig. 1. Tensile stressed region between supported pins in the bottom half of the samples is considered for simulated flexural test.

The number density of the coarser defects is given as a power function as shown in past studies. The coarser defects are introduced into the region of the simulated samples based on the number density of the coarser defects $f$ (the number of defects per unit volume). $f$ is expressed as the following equation,

$$f(c) = Ac^{-n}$$  \hspace{1cm} (4)

where, $c$ is defect size, and $A$ and $n$ are the coefficients of a power function. The function has been found in many results in the observation of coarser defects for past studies. In the region of coarser defect size, for which the number of defect is much less than one within this volume, a random number is generated to determine the presence of the defect in this volume. Once the numbers of the defects for each size are determined for this particular model specimen, the locations of these defects are determined by generating the random number.

The critical stresses are calculated for all defects in a specimen according to the linear fracture mechanics. The $i$th critical stress $\sigma_i$ is calculated from size and location of the $i$th defect by the following equation,

$$\sigma_i = \frac{K_{ic}}{Y \sqrt{a}}$$  \hspace{1cm} (5)

where $K_{ic}$ is the critical stress intensity factor which is a constant 3.5 MPa m$^{1/2}$ for alumina ceramics, $a$ is the defect radius which means a half of defect size $c$ in this calculation, and $Y$ is the shape factor which depends on the location of the defect. Stress distribution is considered to account for the location of each defect in the case of the flexural tests.

Two kinds of shape factors $Y$ are needed for stress calculations of internal and surface defects separately. For an internal defect, the crack propagates from the nearest edge to tensile surface as indicated in Fig. 1(A). The shape factor $Y$ of the nearest edge of crack is calculated for stress intensity factor at the point.

$$Y = 0.637 \left[ 0.9998 + 0.0955 \left( \frac{a}{d} \right) - 0.4696 \left( \frac{a}{d} \right)^2 + 0.6690 \left( \frac{a}{d} \right)^3 \right]$$  \hspace{1cm} (6)

where, $d$ is the depth of defect, and $a$ the defect radius in the model. The crack reaches the tensile surface and spreads all over the cross section of the specimen, and the specimen is fractured.

In the case of the surface defect, two types of defect shapes are considered with calculation of shape factor $Y$; one type of defect is deeper than the defect radius and the other type less deep. The former is considered as a semi-circular defect with an area equivalent to the defect, and the latter as a semi elliptical defect, as indicated in Fig. 1(B). In both cases, the comparison of the shape factor is required between two points which have the possibility to yield the maximum stress concentration. The shape factors $Y$ at different points are obtained by utilizing Newman–Raju equations. At the point $1$ in Fig. 1(B);

$$Y_1 = \frac{\sqrt{\pi} MH_2}{Q}$$  \hspace{1cm} (7)

where

$$Q = 1 + 1.464 \left( \frac{a}{b} \right)^{0.65}$$  \hspace{1cm} (8)

and

$$M = \left[ 1.13 - 0.09 \left( \frac{a}{b} \right) - 0.54 - \frac{0.89}{0.2 + (a/b)} \right] \left( \frac{a}{2h} \right)^{24}$$

$$+ \left[ 0.5 - \frac{1}{0.65 + (a/b)} + 14 \left( \frac{a}{b} \right)^{24} \right] \left( \frac{a}{2h} \right)^{24}$$  \hspace{1cm} (9)

$$H_2 = 1 - \left[ 1.22 + 0.12 \left( \frac{a}{b} \right) \left( \frac{a}{2h} \right)^{0.75} \right] + \left[ 0.55 - 1.05 \left( \frac{a}{b} \right) + 0.47 \left( \frac{a}{b} \right)^{0.75} \right] \left( \frac{a}{2h} \right)^{0.75}$$  \hspace{1cm} (10)

In these equations, $a$ is the depth of semiellipse crack, $b$ the radius of semiellipse on tensile surface, and $h$ the half height of specimen, as indicated in Fig. 1. At the point $2$;
where

\[ H_i = 1 - \left[ 0.34 + 0.11 \left( \frac{a}{b} \right) \left( \frac{a}{2h} \right) \right] \]

\[ S = \left[ 1.1 + 0.35 \left( \frac{a}{2h} \right)^3 \right] \sqrt{\frac{a}{b}} \] (13)

Maximum \( Y \) (\( Y_1 \) or \( Y_2 \)) must be used to calculate the fracture strength. These equations have been applied in past studies to measure the fracture toughness of specimen with a semielliptic precrack.\(^{(1)}\)

The strength of the specimen corresponds to the smallest value among all critical stresses for each defect. The simulation is applied to some series of samples with various number densities of the coarser defects. The number of samples in each series is 500. The flexural strength \( \sigma \) is plotted, when \( \sigma_u \) is assumed to be zero, as the following equation,

\[ \ln (1 - F)^\frac{1}{m} = m \ln \sigma - m \ln \xi \] (14)

where \( F \) is the probability of failure at stress \( \sigma \), \( m \) the Weibull modulus, and \( \xi \) the scale parameter. The Weibull moduli \( m \) of each series is estimated using maximum likelihood method standardized by the Japanese Industrial Standard (JIS R1625).

### 2.2 Calculated results

**Figure 2** shows the logarithmic plots of the probability density of the coarser defect indicated in Eq. (4). The exponent \( n \) of the power function is assumed as 6 – 9, and \( A = 10^{10} \). The densities of the coarser defects shift to the finer side with increasing \( n \). The parameter \( n \) is the rate at which the defects density approaches zero.

**Figure 3** shows the Weibull plots of simulated strengths for various samples which includes coarser defects based on the power functions with \( A = 10^{10} \) and \( n = 6 – 9 \), respectively. The average strengths and their Weibull moduli \( m \) are 330–930 MPa and 8.9–14.3 for the samples with \( n = 6 – 9 \), respectively.

**Figure 4** shows the logarithmic plots of the number density of the coarser defect indicated in Eq. (4) for the case of constant \( n \). The coefficient \( A \) is assumed to vary from \( 10^9 – 10^{12} \) and \( n = 8 \). The densities shift to the finer side with decreasing \( A \), and are parallel in this case.

**Figure 5** shows the Weibull plots of simulated strengths for various samples for the case of constant \( n \) and \( A = 10^9 – 10^{12} \). The mean strengths range 512–897 MPa for \( A = 10^{12} – 10^9 \). The range of Weibull moduli \( m \) is narrow, 12.3–13.2.

**Figure 6** shows the relationship between the Weibull modulus \( m \) and the exponent in the power function of the coarser defects distribution \( n \). The parameters \( A \) and \( n \) are in the ranges of \( A = 10^9 – 10^{13} \) and \( n = 5 – 11 \), respectively. The Weibull modulus \( m \) is proportional to \( n \) for all values of coefficient \( A \). The approximated formula with a least square method is as follows,

\[ m = 1.82n - 1.92 \] (15)

and the determination coefficient \( R^2 \) is 0.9773. This result agreed well to the analytical results of Eq. (3) in past studies.
3. Experimental procedures

Commercial granules (TM–DS–6, Taimei Chemicals Co., Ltd., Japan) and spray-dried granules from commercial alumina powder (AL160SG–1, Showa Denko Co., Ltd., Japan) were used for the starting material. The green compacts were prepared by a uniaxial pressing at 20 MPa followed by a cold isostatic pressing at 200 MPa. The green compacts were sintered at 1350–1600 °C for 2–27 h for TM–DS–6 and at 1580 °C for 2–27 h for AL160SG–1. The sintered ceramics were machined to thin samples of 150 μm thick for observation of internal defects, and also to the rectangular specimen 4 mm × 3 mm × 40 mm for the 4-points bending test. The coarser defects were observed with an optical microscope (BX–51, Olympus Optical Co., Ltd., Japan) in the transmission light mode with the objective lens of magnification ×4. In this study, we counted the number only for defects with the size equal to and over 50 μm. The flexural strength was measured by the four points bending test. The inner and outer spans were 10 and 30 mm, respectively. The loading rate was 0.5 mm/min. The number of specimens is 15–25.

Table 1. Characteristics of Alumina Ceramic Samples

<table>
<thead>
<tr>
<th>Granule</th>
<th>Sintering condition</th>
<th>Number of sample</th>
<th>Relative density [%]</th>
<th>Grain size [μm]</th>
<th>Average strength [MPa]</th>
<th>Fracture toughness [MPa m0.5]</th>
</tr>
</thead>
<tbody>
<tr>
<td>TM–DS–6</td>
<td>1350:2 h</td>
<td>25</td>
<td>99.5</td>
<td>1.2</td>
<td>512</td>
<td>3.5</td>
</tr>
<tr>
<td></td>
<td>1400:2 h</td>
<td>15</td>
<td>99.6</td>
<td>1.9</td>
<td>458</td>
<td>3.5</td>
</tr>
<tr>
<td></td>
<td>1500:2 h</td>
<td>15</td>
<td>99.5</td>
<td>3.9</td>
<td>404</td>
<td>3.4</td>
</tr>
<tr>
<td></td>
<td>1600:2 h</td>
<td>15</td>
<td>99.4</td>
<td>5.5</td>
<td>401</td>
<td>3.5</td>
</tr>
<tr>
<td>AL–160SG–1</td>
<td>1580:2 h</td>
<td>15</td>
<td>98.3</td>
<td>2.5</td>
<td>460</td>
<td>3.6</td>
</tr>
<tr>
<td></td>
<td>1580:8 h</td>
<td>15</td>
<td>98.7</td>
<td>4.1</td>
<td>437</td>
<td>3.8</td>
</tr>
<tr>
<td></td>
<td>1580:27 h</td>
<td>15</td>
<td>98.6</td>
<td>5.8</td>
<td>394</td>
<td>3.9</td>
</tr>
</tbody>
</table>

Fig. 7. Example of defects observed in specimens sintered at various temperatures with optical microscopy (TM–DS–6). Sintered at (a) 1350, (b) 1400, (c) 1500 and (d) 1600°C.

4. Results

Table 1 shows characteristics of alumina samples. The average strength is 394–512 MPa. The fracture toughness is 3.5–3.9 MPa m0.5. The characteristics corresponded to industrial alumina grade.

Figure 7 shows a photomicrograph of coarser defects found in sintered ceramics made with TM–DS–6 granules. The sintering temperatures are (a) 1350, (b) 1400, (c) 1500, and (d) 1600°C, respectively. The coarser defects in sintered ceramics at 1350°C has clear boundary between defect and surrounding ceramics. Whereas, they are very unclear in the samples sintered at 1600°C. Detailed examination with scanning electron microscope (SEM) showed the coarser defects with clear boundary are derived from the contour of granules or dimples in granules. Unclear defects were frequently found in the sample sintered at high temperature and the frequency increased with sintering temperature. The unclear defect consists of a coarse defect and many small defects surrounding it.

Figure 8 shows the logarithmic plots of the measured probability distribution of the coarser defects in alumina ceramics made from TM–DS–6 granules. The exponent n of fitting lines was determined using the least square method. They decreased from 8.0 to 4.5 with increasing sintering temperature at 1350–1600°C. The distribution shifts to the coarser side and widen...
with increasing sintering temperature.

**Figure 9** shows the Weibull plots of experimental strengths for specimens made from TM–DS–6 granules. The average strengths slightly decreased from 429 to 401 MPa with increasing sintering temperature from 1350 to 1600°C. Their Weibull moduli $m$ is about 9.6 to 14.8.

**Figure 10** compares experimental and calculated Weibull modulus $m$ to the exponent $n$ in the number density of coarser defect. The points show experimental results at the sintering temperature from 1350 to 1600°C for TM–DS–6 samples and the sintering time from 2 to 27 h for AL160SG–1. Solid line in the figure is the calculated relationship between $n$ and $m$ shown in Fig. 4. Results are on the calculated line except samples made from TM–DS–6 granules at 1600°C and from AL160SG granules sintered for 27 h. In these samples, coarser defects had unclear boundary.

5. Discussion

The Weibull modulus $m$ for the flexural strength is found to be simply related to the exponent $n$ in the power function of the number density of the defect size in both the simulation based on the fracture mechanics and the experimental results. The
6. Conclusions

Strength variation was estimated by the number density of coarser defects. The Weibull modulus $m$ was related to the parameter $n$ of the power function of the probability function of coarser defects; $m = 1.82n - 1.92$. The Weibull modulus was determined by a series of strength data, which were calculated by the simulated bending test. The number density of coarser defects was assumed as a power function. The Weibull moduli were proportion to the parameter $n$ of the power function of the number density of coarser defects. The experimental results concurred well with the calculated line. The result of this study has a similar tendency of the relationship between the Weibull modulus and the function of coarser defects. The number density of coarser defects could be expressed by the power function, because the coarser defects were developed during the manufacturing processing using granules.

References