General paper

DEPENDENCE OF STRENGTH ON SIZE OF FLAW DOMINATING FRACTURE IN CERAMICS

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Abstract: Bending tests using a silicon nitride and an alumina were carried out under 4-point bending to clarify the relation between the strength and the size of flaw observed at a fracture origin. When the fracture originated from a smaller flaw, the strength was confirmed to be lower than that expected from the fracture toughness criterion. A convenient procedure using the mean strength and the fracture toughness was proposed to estimate the strength degradation depending on flaw size. A new model was also developed assuming the stable cracking caused by the separation per one grain ahead of a flaw prior to the final unstable fracture. A Monte Carlo simulation based on the proposed model was made to explain the anomalous behavior of small flaw. The relation of strength vs. crack length simulated by taking account of grain-size distribution showed good agreement with the experimental result. The lower bound in scatter of strength depending on flaw-size was suggested to be estimated by using the size of the largest grain expected in a material.

Key words: *Ceramics, Small flaw, Grain fracture model, Bending strength, Probabilistic method, Grain-size, Monte Carlo simulation, Silicon nitride, Alumina*

1 INTRODUCTION

Most of engineering ceramics is well known to be broken at an inherent flaw, such as pore, inclusion, extremely large grain or grinding-induced crack. It has been reported, however, that the fracture originating from a smaller flaw in ceramics occurs at a lower stress than that predicted on the basis of the plane-strain fracture toughness criterion[1-4]. This suggests that it is dangerous to estimate the strength of a ceramic component by using fracture mechanics parameters obtained for large cracks.

Some models[3-7] have been proposed to explain the anomalous behavior of small flaw in ceramics. The simplest model in an engineering treatment is proposed that the constant a_0 depending on a material microstructure is added to the initial crack length a in evaluating the stress intensity factor[3-5]. The size of a few grains is often adopted for the constant a_0 . Mathematical treatments of these models may be easy in an engineering design, though no model explains the formation process of such a fictitious crack or the dispersion in the strength depending on flaw-size distribution.

In this paper, bending test and fractographic observation were carried out using a gas pressure sintered silicon nitride and a pressureless sintered alumina to clarify the relation between the bending strength and the size of flaw identified as a fracture origin. A convenient method of estimation was proposed to represent the strength degradation depending on flaw size. A modeling of cracking process[8] was also developed for the stable fracture of a series of grains with distinct sizes just ahead of a flaw during loading. The flaw-size dependent strength as mentioned above and observed

scatter in the strength were simulated from a microstructural aspect by using a Monte Carlo method.

2 BENDING TESTS

2.1 Materials and Testing Procedure

The materials used in this study were a gas pressure sintered silicon nitride EC-141 produced by NTK Ceramic Division, NGK Spark Plug Co., and a pressureless sintered alumina AL-1 produced by Japan Fine Ceramic Center. Mechanical properties of materials[9] are listed in Table 1. The fracture toughness K_{IC} shown in Table 1 was obtained by the bending test using a single-edge pre-cracked specimen, which satisfied the recommendation in JIS(Japanese Industrial Standard) R1607. The mean grain size d_0 was $1.6\mu\text{m}$ for the silicon nitride and $5.3\mu\text{m}$ for the alumina. Table 2 shows parameters presenting the Weibull distribution $F(d)$ of grain size d ;

$$F(d) = 1 - \exp\left[-\left(\frac{d - d_l}{d_s}\right)^{m_d}\right], \quad (1)$$

where d is in μm , and d_l , d_s and m_d are the location, scale and shape parameters, respectively. The distribution of Eq.(1) measured on a cross section will be used as the distribution of grain size in bulk. Specimens with dimensions of $3\text{mm} \times 4\text{mm} \times 50\text{mm}$ were cut from plates of $100\text{mm} \times 100\text{mm}$ with 10mm thickness. Two types of specimen for each material were prepared under two different conditions of final-grinding; ones were ground in their longitudinal directions with #400-mesh diamond wheel and others with #800-mesh wheel. The corners of specimen surface subjected to tensile stress were chamfered by 0.2mm with #800-mesh wheel.

Bending tests were conducted under 4-point bending with the inner and outer span length of 10mm and 30mm, respectively. The stress rate was controlled to be 100MPa/s at the position subjected to the maximum stress in a specimen. All tests were carried out in an ambient atmosphere; at temperature of $290 \pm 1K$ and relative humidity of $65 \pm 5\%$.

Table 1. Mechanical properties of materials.

Material	Si ₃ N ₄	Al ₂ O ₃
Density (Mg/m ³)	3.92	3.23
Fracture toughness K_{IC} (MPa√m)	6.0	4.4
Young's modulus E (GPa)	320	380
Poisson's ratio ν	0.27	0.24

Table 2. Parameters in Weibull distribution of grain-size.

Material	Si ₃ N ₄	Al ₂ O ₃
Shape parameter m_d	1.23	1.04
Location parameter d_l (μm)	0.129	0.704
Scale parameter d_s (μm)	2.85	7.28

Table 3. Strength statistics of specimens ground under different condition.

Material	Si ₃ N ₄ #400	Si ₃ N ₄ #800	Al ₂ O ₃ #400	Al ₂ O ₃ #800
Mean Value (MPa)	1035	1085	385	376
Coefficient of variation	0.067	0.067	0.073	0.037

Table 4. Strength statistics of pooled data.

Material	Si ₃ N ₄	Al ₂ O ₃
Mean value σ_{fo} (MPa)	1060	381
Shape parameter m_σ	16.2	19.7
Scale parameter σ_s (MPa)	1094	391

2.2 Results

Fifteen specimens ground under each condition were tested for both materials. The result of bending strength σ_f is summarized in Table 3. The difference in the mean strength due to the grinding condition is not remarkable in both materials, though the coefficient of variation of strength is smaller for the alumina specimens ground with #800-mesh wheel than the others. The proportion of the number of surface flaws to that of embedded flaws, which were identified as fracture origins by a scanning electron microscope, was not affected by the grinding condition in

this work. Consequently, the strength characteristics of specimens ground under two distinct conditions will not be distinguished in the later analysis.

The mean value σ_{fo} of pooled strength data was 1060MPa for the silicon nitride and 381MPa for the alumina. Figure 1 shows distributions of pooled strength data plotted in Weibull probabilistic papers. The cumulative probability was calculated by the median rank method. For both materials, the distribution can be presented by a straight line, i.e. the following 2-parameter Weibull distribution $F(\sigma_f)$ is adequate to represent the strength distribution.

$$F(\sigma_f) = 1 - \exp[-(\frac{\sigma_f}{\sigma_s})^{m_\sigma}], \quad (2)$$

where σ_f is in MPa, and σ_s and m_σ are the scale and shape parameters, respectively. The calculated parameters with the mean value σ_{fo} are shown in Table 4. Since the shape parameter is 16.2 for the silicon nitride and 19.7 for the alumina, the scatter of strength in the silicon nitride is a little bit larger than that in the alumina.

Fig.1. Distribution of bending strength.

3 RELATION BETWEEN STRENGTH AND FLAW-SIZE

The equivalent crack length l [2] is introduced to discuss the problem on a flaw-size dependent strength. Using the stress intensity factor K and the applied stress σ_a , the length l is defined as

$$l = aM_o^2 = \frac{1}{\pi} \left(\frac{K}{\sigma_a} \right)^2, \quad (3)$$

where M_o is the multiplication factor for K -estimation determined by a crack geometry and/or a loading mode[10-13]. The equivalent crack length means the length of a virtual crack giving the identical stress intensity factor in an infinite plate subjected to the applied stress σ_a . The introduction of l enables us to discuss the relation between the flaw size and the strength, independently of the shape and location of a flaw, or a loading mode.

Fig.2. Relation between equivalent crack length and fracture strength.

Figure 2 presents the relation between the equivalent crack length and the fracture stress. The solid straight line in Fig.2 represents the K_{IC} criterion in the relation between the crack length and the strength as follows;

$$K_{IC} = \sigma_f(\pi l)^{1/2}. \quad (4)$$

Compared with the relation of Eq.(4), the strength shifts toward a lower strength region with decreasing

flaw size. This implies that the use of fracture mechanics parameters obtained for large cracks will result in an overestimate for the strength of ceramics. Employing the concept[5] adding the specific length l_o to the initial crack length l in Eq.(4), the relation of strength vs. crack length is reduced to

$$K_{IC} = \sigma_f[\pi(l + l_o)]^{1/2}. \quad (5)$$

The broken curves in Fig.2 indicate the relation obtained by setting l_o $1.5\mu\text{m}$ for the silicon nitride and $34\mu\text{m}$ for the alumina in Eq.(5). Although the relation of Eq.(5) presents the central trend in a dispersed experimental result, it can not explain the scatter of data itself. Equation (5) is convenient for the engineering application, though l_o must be determined experimentally through a tedious fractographic observation.

Now, a new parameter l^* using the mean strength σ_{fo} and the fracture toughness K_{IC} , both of which are expected to be available in the catalog of a material, is proposed in estimating l_o as follows;

$$l^* = \frac{1}{\pi} \left(\frac{K_{IC}}{\sigma_{fo}} \right)^2. \quad (6)$$

A similar parameter has been proposed in analyzing the propagation of short fatigue cracks in metallic materials[14]. The relation between the length l_o obtained experimentally and the parameter l^* defined as above is shown in Fig.3. Reference data[4,15], which were obtained in 3-point bending tests of a partially stabilized zirconia(PSZ), silicon nitride, and alumina with 92% and 96% purity, and ring compression tests of a silicon nitride and sialon, are also plotted with solid marks in Fig.3. A good correlation is found between the two length parameters for different materials and loading modes; the specific length l_o is almost half of l^* . Since the parameter l^* is calculated by using the mean strength and the fracture toughness without a tedious fractographic observation, l^* will be very useful in the strength design using fracture mechanics parameters for large cracks.

Fig.3. Estimation of specific equivalent crack length.

4 MODELING OF FRACTURE PROCESS AND SIMULATION

In this chapter, a possible model of fracture process is discussed to explain the flaw-size dependent strength, including the scatter of strength, observed in ceramic materials.

4.1 Grain Fracture Model

In most engineering ceramics, the surface free energy in a single crystal is found to be lower than the effective surface energy required for the unstable crack propagation through a polycrystalline material[16-18]. The higher energy for a polycrystalline is ascribed to much rougher fracture surface than that of a single crystal, because the area of fracture surface is larger in a rougher crack-path. When the crack length is large enough compared to the grain size, the effective surface energy in cracking may correspond to $K_{IC}^2(1 - \nu^2)/E$ for the plane strain state, where E is the Young's modulus and ν the Poisson's ratio of a material.

(a) Averaged stress in the grain ahead of the initial crack-tip.

(b) Averaged stress in the grain ahead of the tip of growing crack.

Fig.4. Grain fracture model and averaged stress per grain.

The above empirical fact enables us to presume a possible modeling of fracture process as follows. Even prior to the final unstable fracture, the separation of one grain just ahead of the crack-tip may occur when the elastic strain energy stored in the grain during

loading exceeds the surface energy of a single crystal. It is noted that the surface energy of a single crystal is much less than the energy to propagate a large crack. The final fracture is also assumed to occur when the stress intensity factor at the tip of crack formed by the grain fracture process exceeds the plane-strain fracture toughness. In contrast with metallic materials, a localized slip, i.e. a steep gradient of strain or stress in one grain is not allowable even at relatively high temperature in ceramic materials, because of difficulty in dislocation movement[19]. This validates the above assumption for the fracture in every one grain ahead of the tip of cracking zone. If the fracture of a grain proceeds during loading, the stable crack growth and the crack growth resistance curve are expected to appear.

Assuming the grain fracture model as above, the elastic strain energy stored in one grain at the time t should be estimated by using the stress $\sigma_d(t)$ averaged per the grain with the size of d as shown in Fig.4(a). If the stress distribution at the distance r from the tip of crack at the time t is designated $\sigma(r, t)$, the averaged stress per grain $\sigma_d(t)$ is calculated as

$$\sigma_d(t) = \frac{1}{d} \int_0^d \sigma(r, t) dr. \quad (7)$$

Consider a crack with half length of a in an infinite plate subjected to uniform applied stress $\sigma_\infty(t)$ at the time t . The strict solution[16] for the stress distribution ahead of the crack tip is given by

$$\sigma(r, t) = \sigma_\infty(t) \frac{(a + r)}{[r(2a + r)]^{1/2}}. \quad (8)$$

Substitution of Eq.(8) into Eq.(7) yields

$$\sigma_d(t) = \sigma_\infty(t) \left(1 + \frac{2a}{d}\right)^{1/2}. \quad (9)$$

It is postulated that the crack grows by one grain-size d when the strain energy stored in one grain during loading exceeds the surface energy γ_s of a single crystal. Since the strain energy in the crack opening mode is supposed to be released for such a grain fracture, the above criterion for the assumed grain geometry of a cube with side length of d will be represented by using the averaged stress $\sigma_d(t)$ perpendicular to a crack face as follows.

$$\frac{\sigma_d(t)^2}{2E} d^3 = 2\gamma_s d^2. \quad (10)$$

Note that γ_s is the surface energy of a single crystal, but not that of a polycrystalline. In the subsequent loading process, the averaged stress per grain should be calculated by using re-distribution of stress corresponding to a crack growth and a stress increment at the time t' , as shown in Fig.4(b). After the calculation, it is checked whether the grain fracture criterion of Eq.(10) is satisfied or not in the grain just ahead of newly cracked zone. In the actual state, the size of a grain ahead of the crack-tip is not always constant, and it shows scatter under a certain distribution

depending on a material microstructure, as schematically illustrated in Fig.4. The stress intensity factor K at the new crack-tip is simultaneously evaluated at every calculating stage. The fracture strength σ_f can be determined as the applied stress $\sigma_\infty(t)$ at the time when the value of K becomes equal to the fracture toughness K_{IC} .

4.2 Simulation and Discussion

Suppose an alumina with the following parameters as a model material in the simulation. The mean grain-size, the Young's modulus and the fracture toughness of the model material are $d_0=10\mu\text{m}$, $E=349\text{GPa}$ and $K_{IC}=3.6\text{MPa}\sqrt{\text{m}}$, respectively[4].

Employing the sublimation-energy data, the surface energy of an alumina single crystal was theoretically calculated at 293K, resulting in the average of 1.13J/m^2 and the minimum of 1.06J/m^2 [19]. On the other hand, the maximum of 7.3J/m^2 and the minimum of 6.0J/m^2 were measured by using a double-cantilever beam specimen[19]. In this simulation, the latter minimum value of 6.0J/m^2 is adopted as γ_s because the value is considered as the effective energy in an actual fracture and gives a conservative estimation.

crack-tip in one trial. Grain-sizes were given by using an observed grain-size distribution; uniform random-numbers were used so that the grain-size distribution agrees with that shown in Table 2. In this case, a simulation gives a certain fracture stress and another a different one, because a series of grains located ahead of the crack-tip in a trial is different from that in another trial. One hundred trials for each crack length were made in this simulation.

Figure 5 shows the simulated result with the experimental result as indicated by square or circle marks. In Fig.5, the crack length a is normalized by the mean grain-size d_0 and the fracture strength σ_f by $K_{IC}/(\pi d_0)^{1/2}$. Since the crack is supposed to exist in an infinite plate, the crack length a is identical to the equivalent crack length l as defined by Eq.(3). An error bar in Fig.5 presents the scatter band connecting the maximum and minimum strength obtained by the simulation for a fixed normalized crack length. The straight line presents the K_{IC} criterion given by Eq.(4). As seen in Fig.5, larger scatter of strength is found in the region of smaller crack. This implies that larger effect of the microstructural variation on the fracture process appears for smaller flaw. Compared at the same crack length, the scatter is a little larger in the alumina than the silicon nitride.

The following relation given by modifying Eq.(5) is proposed to predict the lowest bound of strength scatter.

$$K_{IC} = \sigma_f [\pi(a + d_{99.99})]^{1/2}, \quad (11)$$

where $d_{99.99}$ is defined as the grain-size calculated for the cumulative probability of 99.99% in the grain-size distribution. The value of $d_{99.99}$ is an estimate of the size of the largest grain in a material under consideration. The calculated size by using parameters in Table 2 was $d_{99.99} = 11d_0$ for the silicon nitride and $d_{99.99} = 12d_0$ for the alumina. The broken curve in Fig.5 indicates the relation of Eq.(11) with $d_{99.99} = 12d_0$, which gives a more conservative estimation. The observed strength is found to disperse above the broken curve. This suggests that the lowest bound of strength scatter may be predicted by using the expected maximum size of grain in a material.

5 SUMMARY

In the present study, 4-point bending tests were carried out using a gas pressure sintered silicon nitride and a pressureless sintered alumina. When the fracture originated from a smaller flaw, the strength was found to become lower than that expected from the fracture toughness criterion. A convenient procedure using the mean strength and the fracture toughness was proposed to estimate the strength degradation depending on flaw size. A new model of cracking process in ceramics was also proposed to explain the flaw-size effect on the strength. The model was established assuming the stable cracking due to separation per one grain prior to the final unstable fracture. The crack growth by the size of one grain was postulated to occur when the strain energy stored in

Fig.5. Comparison of simulated relation on flaw-size vs. strength with experimental result.

Based on the proposed grain fracture model, a Monte Carlo simulation was carried out by locating a series of grains with distinct sizes ahead of the

the grain just ahead of cracking zone during loading exceeds the surface energy of single crystal. Based on the proposed grain fracture model, the simulation was made to describe the anomalous behavior of small flaw. Through a Monte Carlo simulation by changing the sizes of grains ahead of the crack-tip, the scatter in strength of smooth specimen was found to become larger for smaller flaw. The lower bound of strength scatter depending on flaw size was also suggested to be determined by using the size of the largest grain expected in a material.

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