Fracture Mechanics Study of Ring Crack Initiation in Ceramics by Sphere Indentation*

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In our previous papers, we showed that the ring crack initiation strength (microfracture strength) of gas-pressure-sintered silicon nitride, evaluated by the sphere indentation method, is not related to the conventional flexural strength of the same material, using the concept of effective area based on the Weibull distribution function. In the present study, to clarify the reason for this, a model for initiation of a ring crack emanating from a surface microcrack was introduced. Using this model, the microfracture strength was discussed from the viewpoint of fracture mechanics. The dependence of the ring crack initiation load on the indenter radius was also examined. As a result, the essential difference between the microfracture and flexural strengths was well interpreted using this model.

Key Words: Ceramics, Fracture Mechanics, Stress Intensity Factor, Fracture Toughness, Ring Crack, Indentation, Spherical Indenter, Fracture Resistance Curve

1. Introduction

Applications of structural ceramics to components requiring high strength and high resistance to heat, wear and corrosion are expected. A major problem associated with ceramics, however, is the occurrence of local surface damage, which results in events such as failure of ceramic turbine blades due to collisions with particles included in high-speed combustion gas⁽¹⁾, and flaking damage caused by contact between the balls and races in ceramic bearings⁽²⁾. In brittle materials such as ceramics, high stress caused by contact or impact with other bodies is only slightly relieved by plastic deformation. Thus local surface damage due to high stress has in danger of catastrophic fracture of the system. Therefore, investigation of surface failure is important.

Although a guideline is required to indicate the strength levels in the surface layers of ceramics, an effective measure for evaluating the surface damage has not yet been established. We previously investigated the sphere indentation method utilizing the phenomenon of ring crack initiation, as shown in Fig. 1, for the purpose of evaluating the surface damage in ceramics^{(3),(4)}.

Fig. 1 Schematic of the contact between sphere and plate, and ring crack initiation in the plate

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It is necessary to analyze the fracture phenomena associated with spherical indentation in order to establish a strength evaluation method. Various papers, in which many kinds of glasses were used to examine ring crack initiation, have been published in the last five decades^{(5)–(11)}. Recently, investigations have been carried out on polycrystalline ceramics, such as carbides⁽¹²⁾, thorium dioxide⁽¹³⁾, alumina⁽¹⁴⁾ and silicon nitride^{(15),(16)}. However, no theory which can be used to explain consistently the load at ring crack initiation, the position of ring crack, and the influence of the indenter radius on ring crack initiation has yet been developed.

We have revealed the relationship between the ring crack initiation strength (defined as the microfracture strength) and the conventional flexural strength of silicon nitride^{(3),(4)}. The results showed that the relationship could not be explained by the concept of effective area based on the Weibull distribution function. In the present study, a ring crack initiation model which was proposed by Frank and Lawn⁽⁹⁾ and Wilshaw⁽¹⁰⁾ is introduced to determine the reason why the above relationship could not be elucidated. Using this model, the microfracture strength was discussed from the viewpoint of fracture mechanics.

2. Results of Microfracture Strength Estimation Using the Concept of Effective Area

In this section we outline the experimental results and the estimated microfracture strengths reported in previous papers^{(3),(4)}.

2.1 Experimental results

Both indentation and flexural tests were carried out using gas-pressure-sintered silicon nitride (EC 141; NGK Spark Plug Co., Ltd.). Young's modulus (E_i) and Poisson's ratio (ν_i) for this material are 320 GPa and 0.27, respectively. Figures 2(a) and 2(b) show Weibull plots of the microfracture and flexural strengths. Figure 2(a) shows the results for specimens finished using a #800 diamond wheel (IGSN and BGSN specimens; "I", "B" and "GSN" denote indentation, flexural (bending) and gas-pressure-sintered silicon nitride, respectively). Figure 2(b) shows the results for specimens, the surface layer of which was removed in a series of rapping/polishing operations (IGSNP and BGSNP; "P" denotes polishing). As spherical indenters, hot isostatically pressed silicon nitride balls with diameters of 5/32, 5/16 and 15/32 inches, corresponding to $\phi 4$ mmSN, $\phi 8$ mmSN and $\phi 12$ mmSN in Fig. 2 were used. The elastic constants of the balls are $E_2=304$ GPa and $\nu_2=0.28$. The microfracture strengths $(\sigma_r)_{p,max}$ explicitly differ from the flexural strengths $\sigma_{b,max}$, as

(a) IGSN and BGSN specimens

(b) IGSNP and BGSNP specimens

Fig. 2 Weibull plots of the microfracture and flexural strengths

(a) Average values (b) Standard deviations

Fig. 3 Estimated and measured values of the microfracture strength of IGSN

shown in Fig. 2. It is also found that median cracks, which are relatively large flaws remaining in the surface layers of specimens after the grinding process, are not fracture origins, since there were no changes in differences between $(\sigma_r)_{p,max}$ and $\sigma_{b,max}$ after the removal of the surface layers.

2.2 Estimated results

The disagreement between the strength distributions might be due to the difference in the effective areas. Therefore, both the average values and the standard deviations of $(\sigma_r)_{p,max}$ from the experimental

data for $\sigma_{b,\max}$ were estimated in terms of the effective area, where a 2-parameter Weibull distribution was assumed. Representative estimated values for IGSN are shown in Fig. 3. Although the estimated average values agreed well with the measured values (Fig. 3 (a)), disagreements between the standard deviations of the estimated and measured values were evident (Fig. 3(b)). A similar tendency was found for IGSNP. Therefore, it was concluded that the relationship between the microfracture and flexural strengths could not be interpreted using the concept of effective area.

3. Discussion

3.1 Impracticability of the concept of effective area

The reason for the difference between the microfracture and flexural strengths is as follows. It is assumed that an incipient microscopic crack remains in the surface of a plate as shown in Fig. 4. In the Hertz-Huber stress field⁽¹⁷⁾, high tensile stress in the radial direction is concentrated locally in the surface layer in the case of spherical indentation. The tensile stress rapidly attenuates in the subsurface direction and finally becomes compressive stress. Therefore a deep surface crack has a different fracture origin from a shallow one, and ring crack initiation may not occur at the relatively deep flaw in the case of spherical indentation, in contrast to the case of flexural loading. The stress variation in such a microscopic region is not considered when using the conventional concept of effective area, and so the problem above cannot be solved. This may be the reason for the impracticability of the effective area concept.

3.2 Ring crack initiation model

The model in which a ring crack is initiated at an incipient microcrack in the surface, as shown on the left side of Fig. 4, was proposed by Frank and Lawn⁽⁹⁾ and Wilshaw⁽¹⁰⁾. Wilshaw presented a simple model, assuming that the crack is shallow and normal to the

Fig. 4 Model of ring crack initiation at a surface microcrack

surface, and that the surface tensile stress outside the contact circle is uniformly distributed perpendicular to the crack surface. Even within a microscopic region, however, the stress is not distributed uniformly but changes in the subsurface direction. Thus, we propose a modification of Wilshaw's model, and discuss the relationship between the stress intensity factor at the crack tip and the microfracture strength.

It was assumed that ring crack initiation occurred when the stress intensity factor K_I at the tip of an incipient crack of depth c reached a certain critical value. K_I for this model can be expressed using the solution for the edge crack problem⁽¹⁸⁾ given below, without considering the influence of the free surface.

$$K_I = 2 \left(\frac{c}{\pi} \right)^{1/2} \int_0^c \frac{(\sigma_r)_p(r_c, z)}{(c^2 - z^2)^{1/2}} dz, \quad (1)$$

where $(\sigma_r)_p$ is the radial stress distribution along the crack. Using Eq. (1), the variation in K_I with indentation load P was calculated, as a function of the crack position on the surface r_c and the crack depth c . Figure 5 shows the results for an example with indentation using a $\phi 4$ mm SN sphere ($\phi 3.969$ mm silicon nitride ball), with $r_c = 290 \mu\text{m}$ and $c = 10 \mu\text{m}$. The value $r_c = 290 \mu\text{m}$ was determined by referring to experimental results given in previous papers (see Table 1)^{(3),(4)}. A general explanation of the variation in K_I is given as follows, for the case in which the contact boundary approaches the incipient crack with increasing P . (A) K_I increases almost linearly with increasing P when the boundary is far from the crack ($1.5 \leq r_c/a < \infty$). (B) K_I reaches a peak in the P - K_I curve when the boundary is near the crack ($1.0 < r_c/a \leq 1.5$). After that, it decreases quickly as the boundary becomes closer to the crack. (C) K_I becomes negative when the boundary reaches the crack ($r_c/a = 1.0$). Ring crack initiation never occurs at this position unless K_I exceeds a critical value before it reaches the maximum. These results suggest that ring crack

Fig. 5 Example of relationship between stress intensity factor and applied load

initiation does not occur at the contact boundary where the maximum tensile stress is generated in the surface, but slightly outside the boundary, as shown in the experiments^{(3),(4)}.

An example of the influence of c on K_I is shown in Fig. 6, where K_I was computed for $\phi 4$ mmSN sphere indentation with $r_c = 290 \mu\text{m}$. In the case of spherical indentation, K_I decreases rather than increases when c is greater than a certain value, in contrast to the case of simple tension or flexure.

3.3 Flaw size in relation to fracture origin

The average values of both the ring crack initiation load P_f and the ring crack radius $r_f (=r_c)$ obtained for IGSN and IGSNP are shown in Table 1^{(3),(4)}. Using these values, K_I at ring crack initiation was calculated as a function of c . The results are shown for IGSN in Fig. 7(a) and for IGSNP in Fig. 7(b). It is found that K_I has a maximum with respect to c and has a tendency to decrease when c exceeds a certain value. Therefore, deep cracks are almost unrelated to ring crack initiation.

Horikawa et al.⁽¹⁹⁾ observed the surface properties of alumina specimens ground with #325 and #800 diamond wheels, using a transmission electron microscope. It was revealed that cracks with depths similar to length of a crystal grain were distributed with high density in the surface, primarily along grain boundaries. Thus, the surfaces of the specimens used in previous experiments, i.e., IGSN, BGSN, IGSNP and BGSNP, are assumed to have also the above surface properties. In the case of spherical indenta-

Table 1 Average values of ring crack initiation load P_f and ring crack radius r_f (for IGSN and IGSNP)

Specimen	Indenter	Sample size	P_f , N	r_f , μm
IGSN	4mmSN	21	1889	288
	8mmSN	26	4330	478
	12mmSN	28	7191	645
IGSNP	4mmSN	21	1235	251
	8mmSN	24	2890	410

Fig. 6 Influence of incipient microcrack depth on the variation in stress intensity factor

tion, because the radial stress $(\sigma_r)_p$ decays rapidly from the surface to the subsurface, ring crack initiation probably occurs not at a relatively large flaw but at a microcrack in the surface. EC-141 grains are rodlike with lengths of up to about $10 \mu\text{m}$. With reference to region of this size, it is considered that microcracks with depths of $c \leq 10 \mu\text{m}$ are distributed over the surfaces of specimens. When spherical indentation is carried out on such a surface, ring crack initiation may occur as K_I for one of the surface microcracks reaches the fracture toughness K_{Ic} . For the indenters with radii of about $2 \sim 6 \text{ mm}$ used in these experiments, the maximum values of K_I in IGSN and IGSNP for $c \leq 10 \mu\text{m}$ are $5.0 \sim 5.5 \text{ MPa} \cdot \text{m}^{1/2}$ and $4.0 \sim 4.5 \text{ MPa} \cdot \text{m}^{1/2}$, respectively (see Fig. 7).

There is often crack growth resistance in ceramics due to the stress shielding effect of the process zone wake generated along the crack surface during crack growth, and the resistance parameter K_R increases with stable crack growth⁽²⁰⁾. The plot of K_R against crack extension is known as a crack growth resistance curve (R -curve). This curve is generally followed for longer cracks. On the other hand, the wake does not exist in shorter cracks with lengths of the order of one grain, so K_R does not increase. Shorter cracks grow in an unstable manner when K_I reaches the threshold stress intensity factor K_{th} , corresponding to the lower boundary of K_R ^{(21),(22)}. A value of $K_{th} \approx 4.0 \text{ MPa} \cdot \text{m}^{1/2}$ for EC-141 has been obtained^{(22),(23)}. Therefore, cracks with depths of up to $10 \mu\text{m}$ should be able to develop at $K_I = K_{th} \approx 4.0 \text{ MPa} \cdot \text{m}^{1/2}$. This value of K_{th} is in fairly good agreement with the maximum value of K_I at ring crack initiation computed from the experimental data, as shown in Fig. 7(b). For IGSN, the maximum value of K_I is slightly higher than K_{th} , probably due to overestimation of K_{th} because of the residual compressive stress in the surface layer after grinding⁽⁴⁾.

It is concluded that ring crack initiation occurs at

Fig. 7 Relationship between incipient microcrack depth and the stress intensity factor at ring crack initiation

one of incipient microcracks with depths of up to one grain when the condition $K_I = K_{th}$ is satisfied. The actual microcrack at which the ring crack initiation occurs is determined probabilistically because of the position and depth distributions of the microcracks. Therefore values of P_f and r_f are essentially scattered. However, microcracks are distributed densely at small intervals of the order of one grain length, so it is likely that the influence of the probability factor on these values is small. This is why amounts of scatter of P_f and r_f was very small in previous experiment^{(3),(4)}.

3.4 Difference between indentation and flexural fracture

It is speculated that surface microcracks which are origins of microfracture (ring crack initiation) are also origins of flexural fracture corresponding to high strength data in Fig. 2. On the other hand, comparatively large fabrication flaws near the surface would become origins of flexural fracture corresponding to low strength data. The difference in the sizes of fracture origins between high flexural strengths and microfracture strengths is probably very small. The tensile stress at a subsurface during flexure loading, however, is sufficiently high (in contrast to sphere indentation loading), so that a large flaw would be a fracture origin if it existed there. Since rupturing at such large flaws occurs at low strengths, the flexural strength data obtained experimentally would be widely scattered. Therefore, it can be said that the essential differences in the magnitudes and amounts of scatter for the microfracture and flexural strengths result from the differences in the sizes and distributions of the incipient flaws governing fractures.

4. Dependence of Ring Crack Initiation Load on Indenter Radius

We have also investigated the dependence of ring crack initiation load P_f on indenter radius R , and found that this relationship could also not be explained using the concept of effective area^{(3),(4)}. Thus, the relationship between P_f and R is interpreted from the viewpoint of fracture mechanics.

The relationships were $P_f \propto R^{1.22}$ for IGSN and $P_f \propto R^{1.34}$ for IGSNP in the experiments. The following is generally known to hold for many brittle materials: P_f is nearly proportional to R (Auerbach's law⁽²⁴⁾) when R is small. However, Tillet⁽⁶⁾ found experimentally that P_f is proportional to R^2 when R is large. Hence it is expected that the P_f - R relationship can be expressed using a curve, the slope of which varies gradually from 1 to 2 with increasing R . An explanation of the above results including our results is as follows.

The radial stress $(\sigma_r)_p$ in the elastic half-space expressed in terms of cylindrical coordinates in Fig. 1 is given by⁽¹⁷⁾

$$\frac{(\sigma_r)_p}{p_0} = \frac{1-2\nu_1}{3r'^2} \left\{ 1 - \left(\frac{z'}{u} \right)^3 \right\} + \left(\frac{z'}{u} \right)^3 \frac{u^2}{u^4 + z'^2} + \frac{z'}{u} \left[\frac{(1-\nu_1)u^2}{1+u^2} + (1+\nu_1)u \tan^{-1} \left(\frac{1}{u} \right) - 2 \right], \quad (2)$$

where p_0 is the maximum contact pressure and u is given by

$$u^2 = \frac{1}{2} [r'^2 + z'^2 - 1 + \sqrt{(r'^2 + z'^2 - 1)^2 + 4z'^2}]. \quad (3)$$

$r' = r/a$, $z' = z/a$ and a is the contact radius. Substituting $r'_c (= r_c/a)$ for r' , then

$$(\sigma_r)_p = p_0 \phi(r'_c, z', \nu_1). \quad (4)$$

ϕ is a function of r' , z' and ν_1 . From Eq. (4) and Eq. (1),

$$K_I = 2p_0 \sqrt{\frac{c}{\pi}} \int_0^c \frac{\phi(r'_c, z', \nu_1)}{\sqrt{c'^2 - z'^2}} dz' \quad (5)$$

is obtained. Equation (5) can be alternatively expressed as

$$K_I = \frac{3P}{\pi a^{3/2}} \sqrt{\frac{c'}{\pi}} \int_0^{c'} \frac{\phi(r'_c, z', \nu_1)}{\sqrt{c'^2 - z'^2}} dz', \quad (6)$$

where $c' = c/a$. From the elastic contact theory, a is

$$a = \left[\frac{3}{4} RP \left\{ \frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2} \right\} \right]^{1/3} = \left[\frac{3}{4} RPY \right]^{1/3}. \quad (7)$$

Thus, Eq. (6) can be expressed as

$$K_I = \left[\frac{12}{\pi^3 Y} \right]^{1/2} \left(\frac{P}{R} \right)^{1/2} c'^{1/2} \Phi(c'), \quad (8)$$

where $\Phi(c')$ is given by

$$\Phi(c') = \int_0^{c'} \frac{\phi(r'_c, z', \nu_1)}{\sqrt{c'^2 - z'^2}} dz'. \quad (9)$$

Substituting P_f for P , Eq. (8) becomes

$$K_{th} = \left[\frac{12}{\pi^3 Y} \right]^{1/2} \left(\frac{P_f}{R} \right)^{1/2} c'^{1/2} \Phi(c'). \quad (10)$$

Arranging Eq. (10) to given P_f , we obtain

$$P_f = \frac{\pi^3 Y}{12} K_{th}^2 c'^{-1} \Phi(c')^{-2} R = \alpha R. \quad (11)$$

Equation (11) expresses the criterion for ring crack initiation. Auerbach's law is approved unless the coefficient α is related to R . However α is not regarded as a constant because $c' = c/a$ is variable: increasing P or R results in an increase in a . Therefore α is actually a function of P_f and R .

The P_f - R relationship was examined using Eq. (11). The results obtained are shown in Fig. 8. In Eq. (11), K_{th} was $4.0 \text{ MPa} \cdot \text{m}^{1/2}$, r'_c was taken as 1.13 in accordance with the experimental results and $\Phi(c')$ was obtained by numerical computation. The experimental values for IGSNP are also shown in Fig. 8 for comparison. The slopes of the three curves for $c = 1 \mu\text{m}$, $5 \mu\text{m}$ and $10 \mu\text{m}$ are all approach 1 with decreasing R and 2 with increasing R . However, the value of P_f increases with decreasing R when R is smaller than about 0.5 mm for $c = 5 \mu\text{m}$ and about 2.0 mm for $c = 10 \mu\text{m}$.

Fig. 8 Relationship between indenter radius and ring crack initiation load

μm . The reason for this is as follows. a decreases as R decreases. This gives rise to an increase in c' and a decrease in K_I . Therefore, P_f apparently increases in Eq.(11).

The dependence of ring crack initiation load on indenter radius in a broad range can be well expressed as stated above, provided that microcracks are distributed densely on the surface of the ceramic specimen, and that one of these cracks with maximum K_I gives rise to fracture when $K_I = K_{th}$. The theory based on the K_{th} criterion is accurate, since experimental values for IGSNP are consistent with the envelope of a series of P_f - R curves for $c = 1 \sim 10 \mu\text{m}$, as shown in Fig. 8.

It is known that many brittle materials exhibit elastic-plastic behavior resulting in irreversible deformation beneath a spherical indenter when the indenter radius R is small^{(8),(11),(15),(16)}. In preliminary experiments, in which indentation tests using a $\phi 1.6 \text{ mm}$ SN sphere were carried out, impressions before ring crack initiation in both IGSN and IGSNP were also observed. Therefore, application of the above theory, which is based on elastic fracture mechanics, is limited.

5. Conclusions

The ring crack initiation strength (microfracture strength) in gas-pressure-sintered silicon nitride subjected to sphere indentation tests has been investigated from the viewpoint of fracture mechanics. The following conclusions were drawn.

(1) To clarify the reason why the relationship between the microfracture and flexural strengths cannot be explained using the concept of effective area, a model in which ring crack initiation occurs at a surface microcrack has been introduced. Using this model, the difference between the microfracture and flexural strengths could be interpreted qualitatively.

(2) The stress intensity factor K_I at a surface

microcrack tip in the spherical indentation was calculated. The results indicated that there is a certain crack depth at which K_I is a maximum.

(3) The reason why ring crack initiation does not occur at the contact boundary, where the maximum tensile stress is generated, but slightly outside the boundary is that surface microcracks at the boundary cannot grow towards the subsurface, where compressive stress is dominant.

(4) The ring crack initiation load P_f obtained experimentally can be explained quantitatively, provided that surface microcracks with depths of the order of one grain are distributed densely in the surface of the specimen, and that one of these cracks with maximum K_I gives rise to fracture when K_I reaches K_{th} , the lower boundary of the crack growth resistance parameter K_R .

(5) Experimental results have shown that P_f is proportional to R or R^2 for small or large R , respectively. These results can also be interpreted using the ring crack initiation model and the theory of the K_{th} criterion.

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