Relationship between the cone crack and fracture mode in ceramics under high-velocity-projectile impact

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Ceramics will be widely used for enhancing the efficiency of power generating systems, particularly gas turbines. However, ceramics used in these systems suffer damage owing to impact by foreign objects. In this study, various ceramic plates are impacted by a spherical projectile with an impact velocity of 320 m/s. The volume of the cone cracks formed on the plates decreases with an increase in the fracture toughness of the ceramic material. No cone cracks are formed on the zirconia (3Y-TZP) plate because crack formation is prevented by stress-induced phase transformation outside the impacted region. The volume of the cone crack is higher and the energy consumed by surface formation is smaller in transgranular-fractured ceramics than in intergranular-fractured ones. The fracture process can be controlled by controlling the characteristics of the grain boundaries, as well as by stress-induced phase transformation.

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1. Introduction

Enhancing the efficiency of power generating systems is a global challenge. Ceramics are promising candidates that can be used to increase the efficiency of power generation systems, particularly gas turbines, because they are lightweight materials with high heat and abrasion resistance. However, ceramics used in gas turbines are susceptible to impact by foreign objects.^{1)–7)} Because of their brittle nature, ceramics suffer extensive damage upon high-speed impact even by small particles. Therefore, it is important to have a thorough understanding of the relation between the damage/fracture behavior of ceramics and physical properties such as strength, modulus and fracture toughness so that they can be used for various applications.

Several researchers^{8),9)} have conducted damage/fracture tests on ceramic materials under quasi-static conditions. The damage/fracture behaviors of different materials can be compared since the methods for evaluating such behaviors have been standardized. However, there are few comprehensive studies on the impact behavior of ceramics; in these studies, impact tests have been carried out on a few ceramic materials.^{4),5),7),10)–23)} Impact tests have been conducted under different conditions on one or two ceramics, as has been described below.

The fracture behavior of three commercial gas-turbine-grade silicon nitrides—AS800, SN282, and NC132—has been reported.⁵⁾ AS800 and SN282 are toughened silicon nitrides, whose microstructures have been tailored so that they contain elongated grains. NC132 is a fine-grained silicon nitride. The specimens were impacted at ambient temperature by a steel ball projectile (diameter: 1.59 mm) with a velocity of 220–440 m/s. AS800 showed the highest impact resistance, mainly because of its high fracture toughness (K_{IC}) and elongated grain structure, while NC132 shows the lowest impact resistance.

The relationship between the critical impact velocity (V_C) for crack initiation and the material properties of silicon nitrides with different microstructures was investigated.¹³⁾ The specimens were impacted by a zirconia ball projectile (diameter: 1 mm) with a velocity of 620 m/s. Upon impact, Hertzian cone crack formation occurred in all the specimens at the critical impact velocity (V_C^H). In accordance with the Hertzian cone crack theory, a clear relationship could be observed between V_C^H and K_{IC} ; however, no clear relationship could be established between V_C^H and Young's modulus.

According to previous reports, the damage/fracture behavior of a ceramic is affected by its fracture toughness, which can be controlled by suitably altering the microstructure. Therefore, the microstructure of a ceramic material is an important factor that affects its impact fracture behavior. Hence, it is necessary to understand the relationship between the microstructures and fracture behaviors of ceramic materials. However, there is no clear information on the strength characteristics of the grain boundary against impact damage/fracture properties. Stress-induced phase transformation helps enhance the K_{IC} value of ceramic materials such as zirconia. Till date, there is no precise report on impact damage/fracture behavior in ceramics with stress-induced phase transformation.

In this study, we conducted a series of impact tests on ceramics with various characteristics to investigate the influence of the microstructures and properties of the ceramics, such as toughening and characteristics of grain boundary, on the fracture behavior.

2. Experimental procedure

2.1 Materials

The materials used in this study were boron carbide (B_4C), zirconia (3Y-TZP), alumina–zirconia, mullite, and silicon nitride. Further, three types of alumina ceramics were used: A99.99, A99 and A96 (alumina with 99.99% purity, 99% purity and 96% purity, respectively). Ceramic green bodies were pressed uniaxially at 98 MPa. Alumina (A96, A99), zirconia, and mullite

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Table 1. Mechanical properties of specimens

	Density [g/cm ³]	Flexural strength [MPa]	Fracture toughness [MPa·m ^{0.5}]	Young's modulus [GPa]	Poisson ratio	Hv
Boron carbide	2.376	271	3.4	402	0.163	2408
Alumina (A96)	3.651	373	3.1	302	0.224	1158
Alumina (A99)	3.930	284	4.7	388	0.233	1624
Alumina (A99.99 1300°C)	3.934	591	2.9	397	0.235	1951
Alumina (A99.99 1450°C)	3.964	444	4.1	399	0.237	1729
Alumina (A99.99 1600°C)	3.933	334	5.8	394	0.236	1456
Zirconia (3Y-TZP)	6.055	890	4.8	214	0.314	1292
Alumina–zirconia	4.257	673	3.9	366	0.241	1756
Mullite	3.054	277	1.6	208	0.271	1035
Silicon nitride	3.216	876	6.9	302	0.275	1506

were sintered at 1515–1670°C in an ambient atmosphere. Sintering temperature of Alumina (A99.99) was adjusted at 1300–1600°C. Silicon nitride and B₄C were sintered at 1900–2100°C in an Ar atmosphere. The sintered powders were machined to obtain specimens with dimensions of 70 mm (width) × 4 mm (thickness). The properties (strength, grain-boundary conditions, and grain size) of the sintered ceramics were varied by selecting the appropriate material and process conditions. In this study, the mechanical properties, particularly fracture toughness, were varied extensively because it has been reported^{5),13)} that the fracture toughness of a ceramic material affects its damage/fracture behavior. Grain-boundary properties were also given attention, because they affect fracture toughness.

The density, flexural strength, fracture toughness, Young's modulus, Poisson ratio and Vickers hardness (Hv) of the specimens were measured in accordance with the JIS-R1634(1998), JIS-R1601(1995), JIS-R1602(1995), JIS-R1607(1995), JIS-R1601(1995) and JIS-R1610(2003) standards, respectively. The fracture toughness was measured using the single-edge pre-cracked beam (SEPB) method. These mechanical properties are listed in **Table 1**.

2.2 Impact test equipment

A spherical SUJ-2 projectile (diameter: 4 mm) was accelerated using a light gas accelerator (using He gas) such that it would impact the test specimens with a velocity of approximately 320 m/s. In this type of accelerator, the projectile is accelerated by He gas with the help of a sabot. The test specimen was mounted on a holder fixed perpendicular to the direction of motion of the projectile and set in the test chamber having observation windows. The fracture process was observed using a high-speed camera (Shimadzu, HPV-1). A back plate was set behind the ceramic specimen for recovering the debris collected during the impact. The appropriate size ratio of the projectile and the specimen was selected for a rough estimate of the behavior of the ceramic blade upon impact by metallic debris in a gas turbine.⁵⁾ **Figure 1** shows the experimental setup.

2.3 Raman microprobe measurement

Raman microprobe measurements were conducted to evaluate the phase transformation in zirconia (3Y-TZP). The measurement conditions are shown in **Table 2**. Measurements were conducted at the center (at this point, the projectile was impacted) of the impact region and outside the impact region.

2.4 Critical energy release rate

According to the Griffith theory, cracks propagate when

Fig. 1. Experimental setup.

Table 2. Measurement conditions

Ar ion laser	514.5 nm	Exposure time	30 s
Laser power	400 mW	Cumulated number	2
Laser diameter	2 μm	Field lens diameter	50 times the pupil diameter
Eyesight	40 μm		

released elastic strain energy in specimen is higher than the surface formation energy. The critical energy release rate for crack propagation is defined as below.

$$G_{IC} = \frac{K_{IC}^2(1 - \nu^2)}{E} \quad [\text{J/m}^2] \quad (1)$$

where K_{IC} is the fracture toughness, ν is the Poisson ratio and E is the Young's modulus.

Yasuda et al. have noted that the critical energy release rate was that of transgranular fracture (G_{IC}^{trans}) plus that of intergranular fracture (G_{IC}^{inter}) weighted by the area ratio of transgranular fracture (f).²⁵⁾ Thus critical energy release rate is defined as below.

$$G_{IC} = (1 - f)G_{IC}^{\text{inter}} + fG_{IC}^{\text{trans}} \quad (2)$$

$$= (G_{IC}^{\text{trans}} - G_{IC}^{\text{inter}})f + G_{IC}^{\text{inter}} \quad (3)$$

In this study, regression analysis was conducted through experimental results of alumina (A99.99), then G_{IC}^{trans} and G_{IC}^{inter} were calculated.

3. Experimental results

3.1 Specimens after the impact

Figure 2 shows the front-face image of the impact fracture in the alumina–zirconia specimen. As shown in Fig. 2, all the projectiles shattered after the impact on the front surface of all the ceramic specimens. **Figure 3** shows the formation of a cone

Fig. 2. Front-face image of the impact fracture of alumina-zirconia specimen.

Fig. 3. Rear face of the alumina (A99) specimen after projectile impact.

Fig. 4. Rear face of the zirconia (3Y-TZP) specimen after projectile impact.

crack and radial cracks on the rear face of alumina (A99) specimen after the impact. Cone crack formation is induced by the shear force generated from the interaction between shock waves and reflected waves. However, no cone crack formation was observed in the zirconia (3Y-TZP) specimen (Fig. 4).

3.2 Volume of the cone crack

The volume of the cone crack was estimated from the thicknesses and areas of the holes formed on the front and rear faces of the specimens. The relationship between the volume of the cone crack and mechanical properties of the specimen (density, flexural strength, fracture toughness, Young's modulus, and Hv) was examined. The results showed that the volume of the cone crack was strongly correlated with the fracture toughness, as shown in Fig. 5. In all the specimens except zirconia

Fig. 5. Relation between volume of cone crack, fracture toughness and main fracture mode.

Fig. 6. SEM image of cone crack (mullite).

Fig. 7. SEM image of cone crack (alumina (A99)).

(3Y-TZP), the volume of the cone crack decreased with an increase in the fracture toughness. Thus, we concluded that the fracture toughness of a ceramic affects cone crack formation.

Figures 6–8 show the scanning electron microscope (SEM) images of the surface of the cone cracks formed in mullite, alumina (A99) and silicon nitride. These images reveal that the main fracture mode in mullite was transgranular fracture, while both transgranular and intergranular fractures occurred in alumina (A99); in silicon nitride, intergranular fracture was mainly observed. The fracture mode and the fracture toughness of each specimen also were shown in Fig. 5. As shown in

Fig. 8. SEM image of cone crack (silicon nitride).

Table 3. Range of fracture toughness and main fracture mode

	Main fracture mode
$1.6 < K_{IC} < 3.4$	Transgranular fracture
$3.1 < K_{IC} < 4.7$	Both transgranular and intergranular fractures
$5.8 < K_{IC} < 6.9$	Intergranular fractures

Table 3, the relationship between the main fracture mode and the fracture toughness were determined. The crack volumes were strongly affected by the fracture mode.

Cone crack formation did not occur in zirconia (3Y-TZP), probably because of stress-induced phase transformation.^{16),22),24)} To confirm this, microprobe Raman measurements were conducted. Raman shifts were observed near 370 and 570 cm^{-1} in the spectrum of the outside the impact region, indicating the presence of monoclinic crystals in the microstructures of Zirconia (3Y-TZP);²⁴⁾ however, no such peak was observed in the spectrum of the center of the impact region, as shown in **Fig. 9**.

Fig. 9. Raman spectra of Zirconia (3Y-TZP) surface.

Fig. 10. Optical microscope image of debris collected from boron carbide.

Fig. 11. Optical microscope image of debris collected from alumina-zirconia.

This observation indicated that tetragonal crystal→monoclinic crystals ($t \rightarrow m$) transformation was induced outside the impact region. However, at the center of the impact region, impact-pressure-induced $t \rightarrow m$ transformation with concurrent volume expansion did not occur.²²⁾

The volume of the cone crack in zirconia (3Y-TZP) was smaller than that of the other ceramics because of stress release and/or prevention of crack extension by stress-induced phase transformation outside the impact point.

Stress-induced phase transformation prevents the formation of cone cracks under the experimental conditions employed in this study. The abovementioned results reveal that the fracture mode controls the volume of the cone crack formed in the ceramic specimens. Thus, grain-boundaries surely affect the volumes. The amount of debris obtained from a specimen depends on the volume of the cone crack formed in the specimen; hence, the amount of debris can be controlled by controlling the cohesive strength of the grain boundaries as well as by stress-induced phase transformation.

3.3 Debris generated from the rear face of the specimen

The debris formed in a given impact experiment was collected using the back plate with a double-face tape. That can catch the debris. **Figures 10** and **11** show the optical microscope images of the debris collected from B_4C and alumina-zirconia, respectively. From these figures, it could be seen that some of the debris particles were smaller than 50 μm in size, and detailed observations indicated that the main fracture mode in mullite, alumina (A99.99 1300°C) and B_4C was transgranular fracture. The size of the debris particles obtained from alumina (A96, A99, A99.99 1450°C) and alumina-zirconia was approximately

Table 4. Area ratio of transgranular fracture and critical energy release rate

	Area ratio of transgranular fracture	Critical energy release rate [J/m ²]
Alumina (A99.99 1300°C)	0.91	20
Alumina (A99.99 1450°C)	0.26	40
Alumina (A99.99 1600°C)	0.11	81

Table 5. Critical energy release rate of intergranular fracture and transgranular fracture

	Critical energy release rate [J/m ²]
G_{IC}^{inter}	73
G_{IC}^{trans}	11

Table 6. Calculation results of the percentage of the energy consumed by surface formation to kinetic energy of the projectile

	Crack surface areas [mm ²]	Area ratio of transgranular fracture	Critical energy release rate [J/m ²]	Energy consumed by surface formation [J]	Percentage of the energy consumed by surface formation to kinetic energy of the projectile [%]
Mullite	7740	0.97	11	0.09	0.63
Alumina (A99.99 1300°C)	8346	0.88	20	0.17	1.11
Alumina (A99.99 1450°C)	7112	0.25	40	0.28	1.72
Alumina (A99.99 1600°C)	4201	0.10	81	0.34	2.17
Silicon nitride	3358	0.02	145	0.49	3.21

100 μ m, while that of the debris particles obtained from alumina (A99.99 1600°C) and silicon nitride was approximately 200 μ m. The fracture mode in the debris particles was similar to that observed on the cone crack surfaces. Therefore, the observed fracture mode was concluded to be the same on the cracks and in debris, and the debris formed during transgranular fracture comprised small-sized particles.

3.4 Energy consumed by surface formation

The area ratio of transgranular fracture and the critical energy release rate are shown in **Table 4**. The area ratio of transgranular fracture was measured in the SEPB test specimen. As shown in 2.4, the critical energy release rate of transgranular fracture and that of intergranular fracture were calculated by using the regression analysis through Eq. (1) and the area ratio of transgranular fracture of alumina (A99.99). Calculated results are shown in **Table 5**. The critical energy release rate of intergranular fracture was higher than that of transgranular fracture, thus cracks can propagate easier in transgranular fracture than in intergranular fracture. Consequently, Fig. 8 indicated that the volume of the cone crack of ceramics have the low fracture toughness was large, because the critical energy release rate of transgranular was low.

The estimated energy consumed by surface formation of alumina (A99.99), mullite and silicon nitride were calculated. The energy was the products of the total surface area and the critical energy release rate. The surface area consist of the radial crack surface area, the cone crack surface area and the debris surface area. The debris surface area was measured by analyzing image of the back plate. Because debris were flat, the debris surface area was assumed to be the twice of projected area. More than 98% of debris were caught by the back plate. Calculated results of the percentage of the energy consumed by surface formation to kinetic energy of the projectile were shown in **Table 6**. Compared with Table 4, the area ratio of transgranular fracture of the cone crack was similar with that of the SEPB test specimen. The crack surface area of mullite and alumina (A99.99 1300°C) were large, however, the energy consumed by surface formation was low because of the low critical energy release rate. The relationship between the percentage of the energy consumed by surface formation to the kinetic energy of the projectile and the area ratio of transgranular fracture and the fracture toughness were shown in **Fig. 12** and **Fig. 13**. As shown in Fig. 12 and

Fig. 12. The relationship between the percentage of the energy consumed by surface formation to kinetic energy of the projectile and the area ratio of transgranular fracture. **Fig. 13.** The relationship between the percentage of the energy consumed by surface formation to kinetic energy of the projectile and fracture toughness.

Fig. 13, the percentage of the energy consumed by surface formation to kinetic energy of the projectile was about from 0.5 to 3.5%. The percentage of energy consumed by surface formation to kinetic energy of the projectile was large with fracture toughness and was small with the area ratio of transgranular fracture. Consequently, energy consumed by surface formation was related to fracture mode and the cone crack volume.

4. Conclusion

The characteristics of fracture caused by a high-velocity impact were investigated in ten types of ceramics. Projectiles (diameter: 4 mm; velocity: 320 m/s) from a one-stage light gas accelerator were used for the impact experiments. In the case of all the ceramics, the projectiles shattered after the impact on the front surface of the specimens, and cone crack formation was observed excepting zirconia (3Y-TZP). The volume of the cone crack was small with fracture toughness.

SEM observations of the cone crack surface revealed that the volume of the cone crack was probably dependent on the fracture mode. The volume of the cone crack formed on the surfaces of mullite, B_4C , and alumina (A99.99 1300°C) which underwent transgranular fracture, was high; however, the size of debris formed from these specimens was very small. On the other hand, in the case of silicon nitride and alumina (A99.99 1600°C), which underwent intergranular fracture, the volume of the cone crack was small and a large size of debris was formed. Cone cracks with moderate volumes were formed in the other ceramics, in which both transgranular and intergranular fractures occurred. The fracture mode strongly affected the volume of the cone crack and the size of debris formed. The relationship between the main fracture mode and fracture toughness were determined. The critical energy release rate of transgranular fracture and that of intergranular fracture were calculated by using the regression analysis through Eq. (1) and the area ratio of transgranular fracture of alumina (A99.99). Calculated results revealed that cracks can propagate easier in transgranular than in intergranular, thus the volume of the cone crack of ceramic specimen have the low fracture toughness was large because of the transgranular fracture. Because of stress-induced phase transformation outside the impact region, cone crack formation did not occur in the zirconia (3Y-TZP) specimen.

In addition, the percentage of the energy consumed by surface formation to kinetic energy of the projectile was about from 0.5 to 3.5%. The percentage was not so large, but was smaller in transgranular fracture than in intergranular fracture, which clearly indicated impacted fracture of ceramics was controlled by grain boundary.

The abovementioned results reveal that the fracture mode controls the volume of the cone cracks formed and that stress-induced phase transformation prevents the formation of cone cracks under the experimental conditions used in this study. Since the amount of debris depends on the volume of the cone crack, it can be controlled by controlling the cohesive strength of the grain boundaries in the microstructures of the ceramics, as well as by stress-induced phase transformation.

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