Effect of Crack Size on the Tensile Strength of Ceramics in a High-Temperature Corrosive Environment*

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A study was conducted on the effect of crack size or surface roughness on the tensile strength of ceramics used to the lower-quality fueled gas turbine or diesel engine. In general, removal of the surface defects and/or forming of the correct shape of the ceramic materials requires machining of the part at enormous cost and a large expenditure of time. Even if the materials were carefully machined, the fact that their surfaces would still be extensively attacked by corrosive ashes causing considerable damage, makes such an effort seemingly useless. The experimental results show that in the case of partially stabilized zirconia at 650°C, the existence of artificial corrosive ash containing vanadium pentoxide and sodium sulphate on flaws significantly enhances the tensile strength of the ceramic. However, in the case of silicon carbide at 900°C, no remarkable differences could be found between the results with or without corrosive ash. In this experiment, the hardness indentation method was used as a quantitative tool to simulate mechanical surface defects.

Key Words: Material Testing, Tensile Strength, Ceramics, Crack, Hardness Indentation Method, Corrosive Ash

1. Introduction

Certain ceramic materials are carefully ground at enormous cost and with a considerable time expenditure in order to remove machining marks or flaws, since the rupture strength of ceramics is severely governed by microdamage. Even if the materials are carefully machined, their surfaces would still be attacked by corrosive ash in a lower-quality fueled gas turbine or diesel engine. With the continuation of such service conditions, the surface quality of an affected zone may fall below that of the machine-finished surface, hence negating the advantages of the elaborate machining.

Under the corrosive environment at elevated temperature it seems the presence of flaws or cracks is independent of the life and strength of ceramics. Furthermore, even if the sizes of the flaws are approximately the same in dimensions, their lives seem to vary with the material properties, temperature, envi-

ronment and load pattern. Therefore, at the design or manufacturing stage of ceramic materials, it is important to evaluate the correlation between the parameters affecting the life of the materials. This report deals with an experimental investigation on the fracture mechanism of ceramics considering the above-mentioned points.

2. Experimental Procedure

2.1 Materials and testing apparatus

Silicon carbide, SiC, with surface roughness of 0.8 μm and the yttrium partially stabilized zirconia PSZ, with surface roughnesses of 3.2 μm and 0.8 μm which are in the process of being developed for gas turbine and diesel engine applications, were used for the present research. Figure 1 shows the configuration of the specimen and its holder. A creep rupture testing apparatus, lever ratio 1:10, was used for uniaxial tensile testing. The load was applied by individual weights.

To prevent or minimize the bending stress due to eccentric support with a stiff holder, the test specimen was supported with four pins of 304 stainless steel which were placed at each shoulder in anticipation of

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plastic deformation. This support method always introduced failure at the shoulder where the stress concentration factor α was 1.35. For this reason, a crack pinning by Vickers indentation and a corrosive ash painting, at a width of about 5 mm (20 mg/cm²), were applied only to the middle portion of the specimen.

2.2 Testing conditions

The surface was indented by a Vickers indenter having loads ranging from 1 to 50 kgf (9.8 N to 490 N). Three surface conditions were prepared as follows:

- 1) Painting corrosive ash to an indentation.
- 2) Painting corrosive ash to a plane surface (without indentation).
- 3) No corrosive ash on an indentation.

The plane specimen without an indentation was not examined in air since the specimen always fails at the shoulder.

Table 1 lists the size of microcracks introduced by the Vickers indentation together with the indenter load. The radial crack length was measured by optical microscopy after the rupture of specimens. The cross-sectional views around the Vickers indentation after the test are shown in Figs. 2, 3, 4, 6 and 7. As shown in

these figures, the contours of the cross-sectional view of PSZ are clearer than those of SiC.

The artificial corrosive ash, which corresponds to molten salt, was prescribed by the method of the 123rd Committee on Heat Resisting Metals and Alloys of the Japan Society for the Promotion of Science⁽¹⁾. This powder is composed of two compounds: 15%wt. Na₂SO₄ and 85%wt. V₂O₅. The compounds were found to undergo different reactions at the following two temperature ranges⁽²⁾.

The tests were carried out on two kinds of engineering ceramics in air and in a corrosive environment at a temperature of 650°C for PSZ and 900°C for SiC.

These test temperatures are relatively low for engineering ceramics, but in the case of PSZ, are almost comparable to the surface temperature around the exhaust valve of the diesel engine used in our laboratory. Although there still remains some practical problems that these experiments involved an unrealistic procedure as the process of concentration of ash was not gradual but instant, using a rich powder adhesion, it does not seem that there is a qualitative difference as would influence the results.

3. Results and Discussion

In tensile tests on ceramic materials, it is difficult to avoid the bending stress in a gripping system. In this experiment, the specimens were supported by four stainless steel pins, as shown in Fig. 1, and prior to the test, a specimen was heated to its test temperature for 25 to 100 hours with a load of about 10% of the fracture strength. This, in effect, improved the fitting of the contact surface by plastic deformation. The contacts were thus kept smooth and the bending moment or bending stress by the deflection was minimized.

In the case of PSZ, the plastic deformation of the pins was insufficient due to the relatively low testing temperature, while in the case of SiC, sufficient deformation was achieved after a holding time of 100 hours at 900°C.

To verify the tight contact between a specimen and four pins after heating with some load, the following experiment was done. First, four strain gauges were adhered to each side face of a virgin specimen and loaded at room temperature where, in most cases, the gauges indicated unbalanced loading. After the measurement, the specimen was heated for 25 to 100 hours with about 10% loading of the fracture strength.

Table 1 Vickers indentation dimensions (μm)

Indenter load (kgf)		1	10	30	50
PSZ	2a	40	120	204	260
	1	—	100	215	340
	c	—	160	317	470
	d	—	100	150	352
SiC	2a	50	90	150	206
	1	50	130	340	600
	c	75	175	415	703
	d	—	—	—	—

(in μm)

* Refer to Fig.2 for the symbols used.

Fig. 1 Schematic drawing of specimen and holder

Following this, new gauges were put into place while holding the weight, and the strain was measured. By this procedure, the loading balance was improved and sufficient results were obtained. Bending tests were also performed for a few specimens to compare the correlation between the bending strength and the tensile strength.

As a result of the tests, the proposed support method appears to be adequate, although in general, the choice of materials to obtain the desired deformation is a difficult problem.

3.1 Cross-sectional views and their geometries

The Vickers hardness indentation method was used as a quantitative tool to simulate mechanical surface damage. The crack length from the corner of the indentation which depends on the degree of load was found, as shown in Figs. 2, 3 and 4 and in Table 1. Table 1 lists the quantitative features of these microstructures as judged by optical microscopy. After the fracture, Palmqvist cracks were clearly observed on the cross section of PSZ. In the case of SiC, the cross section was examined for cracks and was recognized to have distinct median cracks at the low load, while at the high load, the microscopic images were not as

clear⁽³⁾.

Vaguely semicircular section was hemmed beyond the crack area in PSZ as shown in Fig. 2. This pattern, the post-indentation marker, seems to be closely connected with the fracture mechanism but it has not been analyzed until now.

The contours of the SiC specimen were different from those of PSZ, where the whole section was covered by a radially expanding smooth plastic zone. This zone shown in Figs. 3 and 4 was not as smooth as the PSZ specimen. Consequently, it seems to have affected the feature of the fractures.

Figure 5 shows the indented surface after heating for 25 hours in corrosive ash. In this case, no cracks at the corner of the indentation were found while considerable swelling at the board of indentation was found. As shown in Figs. 6 and 7, after the heating of PSZ and placement of the corrosive ash on the indented surface, the contours became vague, but in the case of SiC, it did not differ from the previous case.

3.2 Vickers hardness

The Vickers hardness, measured at room temperature on the PSZ and SiC specimens is shown in Figs. 8 and 9, respectively. To verify the effect of the holding time in corrosive ash, the specimen was heated for 25, 100 and 250 hours. The penetrating layer of PSZ became heavier as time increased. The

Fig. 2 Fracture surface of PSZ heated in air (Indentation load, 30 kgf)

Fig. 3 Fracture surface of SiC heated in air (Indentation load, 10 kgf)

Fig. 4 Typical view of fracture surface of SiC (Indentation load, 10 kgf on SiC heated in air)

Fig. 5 Surface outline of PSZ Indentation after heating in corrosive ash

Fig. 6 Fracture surface of PSZ heated in corrosive ash for 100 h

Fig. 7 Fracture surface of SiC heated in corrosive ash for 250 h

Fig. 8 Radial crack length as a function of indenter load for PSZ at room temperature

Fig. 9 Radial crack length as a function of indenter load for SiC at room temperature

change of the attacked layer of SiC was not as clear as that with PSZ. In any case, no difference in hardness as a result of the holding period could be found.

3.3 Tensile strength

Variations in tensile strength with an indented load or in flaw size for various heating periods are shown in Figs. 10 and 11 for PSZ and SiC, respectively. As mentioned before, satisfactory results of tensile tests were obtained after considerable preheating. In this case, however, the increased hold time does not always provide better results; it was found that a hold time of 100 hours was a necessary and sufficient condition.

Applying corrosive ash on the flaws of PSZ can lead to significant improvements in the tensile fracture stress, as shown in Fig. 10. The role of the ash in causing such strengthening seems to result in the improvement of brittleness or blunting of the crack tips. Although it is unknown whether or not a specimen was supported without bending stress, the positive effect of applying the ash was recognized.

Figure 12 shows the bending strength of PSZ

Fig. 10 Tensile strength vs Vickers indenter load as a parameter of exposure time before loading for PSZ

Fig. 11 Tensile strength vs Vickers indenter load as a parameter of exposure time before loading for SiC

Fig. 12 Bending strength vs exposure time without load as a parameter of Vickers indenter load for PSZ

specimens as a function of exposure time in corrosive ash. This data indicates that the strength of noncracked specimens declines as time increases, while the cracked specimens have comparable strength regardless of the crack size owing to the improvement of brittleness or blunting of the crack tips.

It was concluded that under a corrosive environment where the material deteriorated gradually, the elaborate machining required to remove the surface defect was almost a meaningless procedure. The cause of this weakening in corrosive ash as time passed is not due to a crack propagation but seems to be linked to the deterioration or reduction of area. On the other hand, the SiC ceramics show a completely different behavior. For these materials, the fracture strength as a function of indentation load exhibits no significant difference between having and lacking ash.

The surface of the SiC specimen under the corrosive ash was covered with small-scale pits of the order of 20 μm depth and in some places, pits of 100 μm depth were found. These defects were far too small in comparison with those of Vickers indentation cracks. Generally speaking, these small defects decrease the strength of the ceramics. However, while corrosive ash forms small pits, it decreases the sensitivity of crack propagation or blunts the crack tips. That is, applying corrosive ash has both the merit of crack-arresting ability and the demerit of causing defects. In any case, it was found that in corrosive ash, the 10-micron order surface defect in SiC does not

affect the tensile or bending strength.

4. Conclusions

Using a creep rupture testing apparatus in tensile loading, ceramics having considerable defects in corrosive ash were tested at temperatures of 650°C for PSZ and 900°C for SiC. Corrosive ash containing vanadium pentoxide and sodium sulphate was applied to an artificial flaw produced by Vickers indentation on the specimen. Bending tests were also performed for comparison with the tensile data. The following results were obtained:

(1) The surface damage of PSZ in a high-temperature corrosive environment, regardless of the size of the cracks, does not affect the tensile strength significantly. This means that the elaborate machining of the surface is useless for ceramic materials under the service conditions of a corrosive environment. It also suggests that some suitable chemical processing of the surface is useful to eliminate or minimize the crack propagation.

(2) The corrosive ash on SiC ceramics with surface flaws contributes little to the strength as compared to that on PSZ ceramics. The tensile strength depends only on the size of the flaw but it seems that small-sized damage by the corrosive attack in the order of 20 to 100 microns in depth did not affect the strength.

Under ordinary circumstances, these scale defects must be the initiation of a crack and result in fracture. But in a corrosive environment, a small-sized defect, i.e., less than 100 microns in depth, does not result in damage, as in the case of PSZ.

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