Tensile Test of Sintered Silicon Nitride Ceramic at Elevated Temperatures*

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The test system was newly developed for precisely evaluating the tensile strength of ceramics at elevated temperatures. Four-point bending and tensile tests were performed on the sintered silicon nitride ceramic in the temperature range from room temperature to 1300°C. The track of ductile fracture was observed on the fracture surface of a tensile specimen tested at temperatures above 1100°C, suggesting the occurrence of inelastic deformation. The fracture stress of bending specimens was about 1.18~1.43 times as large as that of the tensile specimen. The test temperature dependency, however, was very similar in both the fracture stresses in the whole test temperature range. The fracture toughness from tensile and bending specimens were almost the same in the test temperature range from R.T. to about 800°C. The fracture toughness obtained from the chevron-notch-type specimen was almost coincident with that from the Knoop-indented specimen in the temperature range from R.T. to 900°C. However, they manifested some differences in temperatures above this, suggesting the occurrence of adhesion at the crack tip in the Knoop-indented specimen.

Key Words: Silicon Nitride Ceramic, Test System, Elevated Temperatures, Tensile Strength, Flexural Strength, Fracture Toughness, Knoop-Indented Specimen, Chevron-Notch-Type Specimen

1. Introduction

Recently, the scope of usage of engineering ceramics with excellent heat-resistance has extended to machine components operating at elevated temperatures. The application of these materials to machines operating at elevated temperatures will be promoted even further by identifying their precise and reliable mechanical properties as well as by clarifying mechanisms of their deformation and fracture at elevated temperatures. In addition to this, we should attempt to accumulate extensive reliable data on the deformation and the strength of ceramics at elevated temperatures in order to design reliable high-temperature machine components.

Brittle materials such as ceramics are quite difficult to test under tensile loading because of the difficulty encountered in aligning the specimen and load axes. Therefore, extensive three- and four-point

bending tests have been performed on the ceramics⁽¹⁾. The precise stress-strain response of a ceramic, however, can be obtained only through performing a careful tensile test.

It is extremely difficult to measure tensile displacement of ceramics at test temperatures over 1000°C; a common contact-type extensometer is problematic because of the lack of the rigidity and the heat expansion of the rod which is pressed against the specimen at such temperatures. This, together with the above-mentioned difficulties involved in the tensile test, seems to be the main reason why a reliable stress-strain response for ceramics has not yet been ascertained at elevated temperatures.

In the present paper, the laser-beam-type extensometer which was presented in the earlier paper⁽²⁾ is applied to the measurement of tensile deformation at room temperature, and some preparations are made for measuring tensile displacement at elevated temperatures in the near future. Another purpose of this paper is to examine the strength under tensile⁻⁽³⁾⁻⁽⁸⁾ and bend⁻⁽¹⁾ loading and fracture toughness⁽⁹⁾⁻⁽¹²⁾ at elevated temperatures; the tensile and bending tests are performed on the specimen indented with the

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Knoop hardness tester, the chevron-notched specimen and the smooth specimen which were machined from the sintered silicon nitride ceramic, and the tensile and flexural strength, and the fracture toughness at elevated temperatures were examined.

2. Test Procedures

2.1 Material and test specimens

A sintered silicon nitride ceramic was used in the present study. The specimens are classified into those for bending and those for tensile tests. Figures 1 and 2 show the smooth and chevron-notched specimens for the former, and the smooth specimen and the specimen specifically designed for measuring the tensile displacement, which are used for the latter, are presented in Figs. 3(a) and (b). The shape of the smooth specimen for the tensile test was determined through the stress analysis using the elastic finite element method so that the fracture occurred around the central portion within the gauge length⁽⁶⁾⁽⁸⁾. Figure 4 shows the distribution of the stress at the side edge of the specimen along its axis. The elastic stress concentration factor is about 1.05 at the center of the specimen shown in Fig. 3(a). The tensile displacement was measured between the two projections by means of the laser-beam-type extensometer in the

Fig. 1 Shape and dimensions of bending specimen

Fig. 2 Shape and dimensions of chevron-notched specimen, where Y^* is the shape factor included in Eq. (2)

specimen shown in Fig. 3(b).

The fracture toughness was also examined using the following specimens; the indenter of the Knoop hardness tester was indented at the load, $P=490$ N, and the semielliptically-shaped precracks were introduced at the center on the surface of the specimens shown in Figs. 1 and 3(a).

(a) Specimen for measuring the strength

(b) Specimen for measuring the elongation

Fig. 3 Shapes and dimensions of tensile specimens

Fig. 4 Stress distribution on side edge of the tensile specimen, which was calculated through the elastic finite element method

2.2 Test system

Figure 5 shows the setup of the specimen holders machined from the heat-resisting super alloy and the tensile specimen. The specimen holders were connected with the loading rods attached to self-adjustable bearings with the glassy surface of a globe. The four sections of the small pipes made of the heat-resisting super alloy were put between the specimen holder and the shoulders of the tensile specimen, as shown in this figure, to ensure a good alignment between the specimen and load axes during tensile loading⁽⁸⁾.

The tensile specimen was heated in an electrical resistance-type-heating furnace having two windows through which the laser beam passed. The temperature variation was kept within one percent of the setup test temperature in the range of 1.0 cm above and below the center of the mounted specimen, and within a radius of 1.5 cm around the center of the furnace.

The closed-loop-type hydraulic test system was employed for loading. The displacement associated with loading was measured by means of the laser-beam-type extensometer on which we reported in the earlier paper⁽²⁾.

2.3 Test procedures

Before tensile testing, four gauges were affixed to the four side surfaces of all the test specimens, which were schematically shown in Fig. 6, and the bending component of the strain accompanied with tensile loading was checked. A load corresponding to about 40% of the expected tensile strength was imposed on

Fig. 5 Setup of the specimen holders for tensile loading and tensile specimen

the specimen, and then the difference between the strains detected by the four gauges was examined. When the maximum of the differences in the strains was over 3% of the average of the four strains, the alignment was adjusted by inserting a ceramic fiber between the specimen shoulder and the small pipe so that the maximum amount of the strain difference became below 3% of the average strain. After finishing such a preliminary test for every specimen, the tensile test was started. Figure 6 represents a result of the preliminary test showing that good alignment was attained: the strains measured at the four sites are almost coincident with one another at a given stress, and the maximum amount of the differences in strain is below 3% of the average strain.

The specimen was heated up to the targeted test temperature at a heating rate of 5°C/min., being preloaded at about 15 to 20% of the expected fracture stress in air. The tensile test was performed at the cross head speed, 0.1 mm/min. after the specimen was maintained for 30 min. at the test temperature. Incidentally, the ceramic fiber employed for adjusting the alignment did not apparently change in quality by being heated up to the test temperatures targeted in the present study.

The displacement associated with the tensile loading at elevated temperatures was measured by means of the laser-beam-type extensometer. Figure 7 is a schema showing the principle of the proposed measurement. The laser beam emitted from the irradiator (A) passes through the interval between the upper and lower projections of the specimen, which is shown in Fig. 3(b), and then reaches the receiver (B). The receiver (B) continuously detects the increase in the amount of the laser beam passing through the interval,

Fig. 6 Stress-strain responses of silicon nitride ceramics at room temperature, which were obtained for examining the bending component of the strain generated under tensile loading, using four strain gauges

during the tensile loading, and transforms this into the displacement.

The strain gauge was affixed at the center between the two projections of the specimen, and the strain at this position and the displacement produced between the two projections were measured simultaneously during tensile loading.

The stress-strain relationship was examined through the following three methods in the specimen with two projections shown in Fig. 3(b). Firstly, we obtained the stress-strain relationship from the strain measured with the strain gauge, which was considered to be the correct one. Secondly, the displacement measured with the laser-beam-type extensometer was divided by the distance between the two projections, and the stress was plotted against this strain. Thirdly, the elastic finite-element-method calculation was made for the displacement between the two projections under tensile loading, where the relationship between stress and strain measured with the strain gauge was adopted as the basic material property, and then the stress-strain relationship was estimated in the same way as in the second one. These three stress-strain relationships are presented in Fig. 8.

The two stress-strain relationships which were obtained from the experiment and the F.E.M. analysis between the two projections are in quite good agreement in Fig. 8. Moreover, these strains which are averaged between the two projections are found to be about 10% larger than the strain measured at the center of the specimen at a given stress. The following is expected from these considerations. The stress-strain relationship at elevated temperatures can be determined from combining the displacement measured between the two projections by means of the laser-beam-type extensometer at elevated tempera-

Fig. 7 Schema of measurement of tensile deformation by means of laser-beam-type extensometer

tures and the F.E.M. calculation. This will be dealt with in a subsequent paper.

3. Analyses

3.1 Evaluation of fracture toughness from the Knoop-indented specimen

The precrack introduced with the Knoop hardness tester was assumed to be of semielliptical shape, and then the fracture toughness, K_{Ic} , was calculated through Eq. (1) which is the analytical solution by Raju and Newman⁽¹³⁾,

$$K_{Ic} = \frac{Y(\sigma_T/H + \sigma_B)}{\Phi(\lambda)} \sqrt{a}, \quad (1)$$

where $\Phi(\lambda)$ is a complete elliptical integral of the second kind involving $\lambda = a/c$ and is written as $\int_0^{\pi/2} [1 - (1 - a^2/c^2) \sin^2 \theta]^{1/2} d\theta$, where a is the crack depth and c is half the crack length on the specimen surface; σ_T and σ_B are a uniform tensile stress and an antiplane-bending stress acting perpendicularly to the crack surface, respectively, and Y and H are the coefficients determined from the shape and dimensions of the test specimen and precrack. Incidentally, this equation is reasonable only under the condition of $a < c$ which is fulfilled by all the precracks introduced in the present study.

3.2 Evaluation of fracture toughness from the chevron-notched specimen

The shape and dimensions of the chevron-notched specimen, and the stationing of the specimen and the loading and supporting pins for the four-point bending test are presented in Fig. 2.

According to the Bluhm's slice model⁽¹⁴⁾⁻⁽¹⁶⁾, the

Fig. 8 Comparison between the tested and calculated stress-strain relationships, where the strains measured through the laser-beam-type extensometer and calculated using F.E.M. are the ones averaged between the two projections of the specimen, while the strain gauge measured the strain at the central portion of the specimen

specimen is divided into n thin slices of ΔZ in thickness, m and $(n-m)$ of which are situated in the straight-through portion of the trapezoid and in the tapered portion of the chevron notch, respectively, as shown in Fig. 2. Then the compliance of each thin slice is estimated. The whole compliance of the chevron-notched specimen, C_{tr} , is calculated by totalling the compliance of the respective thin slices. The fracture toughness, K_{Ic} , is obtained from the following equation including C_{tr} ,

$$K_{Ic} = \frac{P}{B\sqrt{W}} \cdot \frac{1}{2} \cdot \frac{dC_{tr}}{da} \cdot \frac{a_1 - a_0}{a - a_0} \\ = \frac{P}{B\sqrt{W}} \cdot Y^*, \quad (2)$$

where P is the load, B is specimen thickness, W is specimen height, and a , a_0 and a_1 are the ratios of A , A_0 and A_1 to W ; $a = A/W$, $a_0 = A_0/W$ and $a_1 = A_1/W$. The quantities of B , W , A , A_0 and A_1 are given in Fig. 2.

Equation (2) means that the parameter Y^* which represents $(1/2)(dC_{tr}/da)((a_1 - a_0)/(a - a_0))$ assumes the minimum at the maximum of P since K_{Ic} is constant value. Thus, K_{Ic} of the chevron-notched specimen can be calculated by measuring only the fracture load in the four-point bending test in addition to estimating the minimum value of Y^* in the Y^*-a relationship.

4. Test Results and Discussion

4.1 Test temperature dependency of fracture stress

Figure 9 shows the small pipes which were used in the tensile test at 1 000° and 1 200°C being put between the specimen shoulders and the specimen holders, as shown in Fig. 5. It seems from this figure that the pipes made of the heat-resisting super alloy effectively collapsed to decrease the bending component of stress acting on the specimen during tensile loading⁽⁸⁾.

Figure 10 shows the tensile and flexural fracture stresses plotted against the test temperature in the smooth and the Knoop-indented specimens, where the tensile stress is obtained by multiplying the nominal stress by the stress concentration factor, 1.05, and the flexural stress is elastically calculated through the elementary mechanics. The inelastic deformation occurs in the silicon nitride ceramic at test temperatures above around 1 200°C⁽²⁾. This effect, however, is neglected on the estimation of the flexural fracture stress in the present study. According to this figure, the tensile strength is about 70 to 85 percent of the flexural strength in the smooth specimen at test temperatures ranging from room temperature to 1 000°C, and the difference between the two remains unchanged in this temperature range. Moreover, both

Fig. 9 Deformed state of small pipes put between specimen shoulder and specimen holder after high temperature tests

Fig. 10 Test temperature dependency of the fracture stress

the tensile and flexural strengths lower at 800°C, compared with those at room temperature. Then the strength decreases gradually with increasing test temperature at temperatures below around 1 000°C in the tensile test and below around 1 200°C in the bending test. Finally, it was found that the tensile and flexural strengths fall off quickly at test temperatures over about 1 000°C and over about 1 200°C, respectively.

In general, ceramics materials are composed of polycrystals and contain the glass layer in the grain boundary, which mainly consists of impurity ingredients. The viscosity of the glass layer decreases, and therefore a grain boundary slips more easily at elevated temperatures. Such a situation induces the crack originated at a defect self-contained in ceramic materials to grow slowly along the grain boundary at elevated temperatures, which is referred to as a slow crack growth (S.C.G.)⁽⁴⁾⁽¹⁷⁾. The drop of the strength at temperatures over around 1 000°C might be due to the slow crack growth phenomenon. Such a temperature range is almost coincident with the test temperature range in which the fracture stress drops dramatically in the tensile and bending tests, as shown in Fig. 10. Thus, the marked decrease in fracture stress at elevated temperatures seems to be due to slow crack growth as well as an inelastic deformation which occurs in silicon nitride ceramic at elevated temperatures.

The fracture stress is much larger under bend loading than under tensile loading at all test temperatures, as shown in Fig. 10. This may be understood through the concept of the effective volume⁽⁶⁾, a familiar factor in strength evaluation at room temperature. We do not have sufficient data to discuss the test mode dependency of the strength of the silicon nitride ceramic from the statistical viewpoint at elevated temperatures in the present study, and so this remains to be clarified in the near future.

The fractographs which will be presented later show the following. The track of the S.C.G. is detected on the surface fractured under bend loading at test temperatures over 1 200°C, but the fracture surface is apparently even and plain. Meanwhile, a protuberance evidencing occurrence of a ductilelike fracture is produced on the surface fractured under tensile loading at the same test temperatures. Thus, some differences in the process of the S.C.G. seem to exist under the two loading modes, and this may also cause the difference in the fracture stress under tensile and bend loading.

As stated before, the inelastic deformation effect is not considered on the estimation of the surface stress under bend loading at elevated temperatures. Therefore, it is noted here that the actual difference in

the fracture stress between the two loading modes is reduced at test temperatures where inelastic deformation occurs.

The specimens indented with the Knoop hardness tester at load 490 N also show larger fracture stress in the bending test than in the tensile test. The fracture stress in the latter is about 75 to 80% of that in the former at test temperatures from room temperature to 900°C, and the difference between the two tends to increase slightly at test temperatures over 900°C. Additionally, the tensile and flexural fracture stresses of the Knoop-indented specimens increase slightly with a rise in test temperature in the temperature range from about 900°C to 1 200°C, and then decrease with further temperature rise through their maximum values. After all, the fracture stress of the Knoop-indented specimens almost coincides with that of the smooth specimen under tensile loading at around 1 200 °C and under bend loading at around 1 300°C, respectively. These may be caused by the following; an adhesion arises around the precrack tip at these temperatures, in addition to the relief of the tensile residual stress around the precrack tip at about 1 000°C⁽¹⁸⁾.

4.2 Test temperature dependency of fracture toughness

Figure 11 shows the fracture toughnesses, K_{Ic} , plotted against test temperature, T , which are obtained from the Knoop-indented and chevron-notched specimens tested under bend loading and from the Knoop-indented specimen tested under tensile loading. The fracture toughness from the Knoop-indented specimens remains unchanged in the test temperature range from room temperature to about 800°C, and then increases, being followed by the decrease through the maximum, with a rise in the test temperature over 800°C in a way similar to the σ_f -vs- T relationship presented in Fig. 10. The fracture

Fig. 11 Test temperature dependency of the fracture toughness

toughnesses obtained from testing the Knoop-indented specimens under tensile and bend loading almost coincide with each other at test temperatures from room temperature to 800°C. The latter, however, becomes larger than the former at test temperatures over 800°C.

The strength of ceramics is significantly influenced by the strain rate at elevated temperatures⁽¹⁰⁾⁽¹⁹⁾. The reason why the bending test yields fracture toughness larger than the tensile test should be examined from the viewpoint of the difference in the strain rate produced between the two tests.

The fracture toughness determined from the chevron-notched specimen remains unchanged at test temperatures from room temperature to around 900°C. Then, this slightly decreases and reaches the minimum at about 1100°C, around which the fracture toughness of the Knoop-indented specimen reaches maximum, being followed by its increase with further rise of the test temperature. Thus, the temperature dependency of the K_{IC} obtained from the chevron-notched specimen is in contrast to that of the K_{IC} from the Knoop-indented specimens at test temperatures over around 1000°C. Maximizing of the K_{IC} in the Knoop-indented specimens might be due to the effect of the adhesion occurring around the precrack tip at around 1100°C. Additionally, the increase in the K_{IC} of the chevron-notched specimens might be caused by an inelastic deformation occurring at test temperatures over 1100°C.

The single specimen was tested at each elevated temperature, and such test data were presented in Figs. 10 and 11. The strength of ceramics at elevated temperatures has less scatter than that at room temperature, as inferred from Figs. 10 and 11. This is probably caused by the following; the stress concentration at defects weakens due to the inelastic deformation occurring in the ceramics at elevated temperatures. Thus, Figs. 10 and 11 seem to give the reliable data on the test temperature dependence of σ_f and K_{IC} .

4.3 Observations of fracture surfaces

Figures 12(a) through (d) show the scanning-electron-microscope photographs taken on the surface fractured at test temperatures, room temperature, 800°C, 900°C and 1000°C, respectively. According to these observations, all the fracture surfaces are rugged, regardless of test temperature, exhibiting a characteristic of a brittle fracture.

Figures 13 and 14 show the observations of the fracture surfaces obtained from the tests at 1100°C and 1200°C. These are the macroscopic observations of the fracture surface and the side view of the fractured portion, their schematic representations,

Fig. 12 Scanning electron microscope observations of the surface fractured at test temperatures from room temperature to 1000°C

and the scanning electron microscope observations of the positions, A and B marked in the schema of the fracture surface. It should be noted that a protuberance is formed at location A on the fracture surface.

(a) Optical microscope observation of the fracture surface and its schematic illustration

(b) Side view of the fracture surface

(c) Scanning electron microscope observation of region A marked in (a)

(d) Scanning electron microscope observation of region B marked in (a)

Fig. 13 Optical and scanning electron microscope observations of the surface fractured at 1 100°C and its side view

The protruding area extends much more on the surface fractured at 1 100°C than on the surface fractured at 1 200°C; the protruding portion is much more localized on the latter than on the former. Meanwhile, the

(a) Optical microscope observation of the fracture surface and its schematic illustration

(b) Side view of the fracture surface

(c) Scanning electron microscope observation of region A marked in (a)

(d) Scanning electron microscope observation of region B marked in (a)

Fig. 14 Optical and scanning electron microscope observations of the surface fractured at 1 200°C and its side view

height of the protruding portions is about 0.15 mm and 0.35 mm on the surfaces fractured at 1 100°C and 1 200 °C, respectively. Thus, the protuberance is much more developed at 1 200°C than at 1 100°C. Such protruding portions were found on both the fracture surfaces of the specimen.

According to the scanning electron microscope observations, lumps appear in the region B as well as in the region A, and cracks are observed around the lumps on the surfaces fractured at 1 100°C and 1 200°C, as shown in Figs. 13(c) and (d), and in Figs. 14(c) and (d).

The silicon nitride ceramic changes their deformation mode from an elastic to an inelastic mode at around 1 100°C, judging from the measurements of the load-displacement response⁽²⁾. Then, the protuberance seems to be created due to the inelastic deformation which causes the formation of the lumps at test temperatures over 1 100°C. Incidentally, no protuberance formed in the specimens fractured under bend loading at test temperatures from 1 100°C to 1 300°C.

5. Conclusions

A new test system which was composed of the closed-loop-type loading system, the electrical resistance-type heating system and the laser-beam-type displacement measuring system was developed for precisely evaluating the deformation and strength of engineering ceramic at elevated temperatures. Then, tensile and four-point bending tests were performed for the silicon nitride ceramics at elevated temperatures. The main results obtained are summarized as follows.

(1) The laser-beam-type extensometer proved useful for measuring the tensile displacement ranging from 100 μ m to 500 μ m with sufficient precision at room temperature.

(2) The fracture stress of the smooth specimen decreases with increase in test temperature, and the rate of its decrease increases greatly at test temperatures over 1 000°C in the tensile test and over 1 200°C in the bending test. Meanwhile, the fracture stress of the specimen indented with the Knoop hardness tester remains almost unchanged at test temperatures ranging from room temperature to 900°C. This increases and then decreases with the test temperature rise, through the maximum at the test temperature around 1 100°C~1 200°C. Moreover, the fracture stress of the Knoop-indented specimen almost coincides with that of the smooth specimen at test temperatures over 1 200°C under tensile loading and over 1 300°C under bend loading.

(3) The fracture toughness obtained from the chevron-notched specimen slightly decreased with

increasing test temperature and reached the minimum around 1 100°C. Meanwhile, the fracture toughness from the Knoop-indented specimens remains almost unchanged in the test temperature range from room temperature to 800°C, being nearly coincident with that from the chevron-notched specimen. Then, these increased with further temperature rise, and reached the maximum at around 1 100°C. The difference in the fracture toughness between both the specimens might be caused by an adhesion occurring around the pre-crack of the Knoop-indented specimen at elevated temperatures.

(4) Obvious marks of an inelastic deformation-induced fracture were left on the surface fractured under tensile loading at test temperatures over 1 100°C. It was proven from this and the measurements of the load-displacement relationship reported in the earlier paper that the silicon nitride ceramic undergoes an inelastic deformation at test temperatures over around 1 100°C.

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