Behavior of Mirror-like Region of Sintered Si_3N_4 under Rotary Bending and Static Fatigue

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常圧焼結窒化ケイ素の回転曲げ及び静疲労におけるミラー状領域の挙動

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Rotary bending and static fatigue tests were carried out on sintered Si_3N_4 at room temperature to study the effect of loading conditions on the fatigue behavior. It is confirmed that the fatigue behavior under rotary bending was different from that under static fatigue. The value of n under rotary bending was much smaller than that under static fatigue. The assumed fatigue limit under rotary bending was also lower than that under static fatigue. However, the characters of mirror-like regions observed on the fractured surfaces after both fatigue tests were almost the same as those after the static test. If the fracture initiation part was near the specimen surface, the mirror-like region was roughly semicircular, and the fracture propagated radially from the region. The size of each mirror-like region, determined from optical micrographs at a magnification of 25, was correlated with the applied stress regardless of the loading condition. Stress intensity factor at the deepest point in the mirror-like region was also almost constant. It is considered that the fatigue failure occurred when the size of mirror-like region reached a critical value which was dependent on the magnitude of applied stress, and that the difference of fatigue behavior under rotary bending and static fatigue was due to the subcritical crack growth behavior in the mirror-like region.

Key-words : *Cyclic fatigue, Static fatigue, Rotary bending, Sintered Si_3N_4 , Mirror, Mirror-like region*

1. Introduction

It is important to know the fatigue behavior of ceramics when using them as structural components, since the fatigue strength is generally lower than the static strength. Although it is known for some ceramics that the fatigue strength depends on the loading condition such as the applied stress state, basic data are few. More information seems to be necessary to clarify the fracture mechanism of ceramics and their reliability as structural materials.

Rotary bending and static fatigue tests were carried out on sintered Si_3N_4 at room temperature to study the effect of loading conditions on the fatigue behavior. The fatigue behavior of sintered Si_3N_4 under rotary bending was examined in the previous

study.¹⁾⁻³⁾ In the present study, a rotary bending test was performed on sintered Si_3N_4 whose strength was much higher than the previous sintered Si_3N_4 . A static fatigue test was performed using the non-rotating fatigue machine. The fatigue tests were performed for a fairly long period to examine the existence of a knee. The fractured surfaces after both tests had mirror-like regions which were similar to mirrors⁴⁾ observed on the fractured surfaces after a static test. Mirror-like region of fractured surfaces after the fatigue test has not been so clarified well as mirror in the static test. It seems necessary for the understanding of the fatigue fracture mechanism to clarify the character of mirror-like region. Therefore, the basic character of mirror-like region was examined, in the same manner as the previous study.¹⁾ Based on the results obtained, the difference and the similarity of the fatigue behavior under rotary bending and static fatigue were discussed.

2. Experimental procedure

The material used was sintered Si_3N_4 obtained from Kyocera. It was fabricated with Y_2O_3 and Al_2O_3 as additives.⁵⁾ The material had rod-shaped grains with various aspect ratios; average grain size was 1–2 μm . The specimen had a cylindrical shape. The diameter of the middle part was 8 mm and each end of the specimen had a larger diameter of 12 mm for chucking. The straight length of the middle part was 15.4 mm, and the two parts of different diameters were connected by a smooth curved surface without generating stress concentration. The specimen was ground perpendicularly to its axis so as to make the finished surface smooth; the maximum roughness of the middle part of the specimen was about 1 μm .

The machine used was an Ono's rotary bending fatigue testing machine operating 3420 cycles per minute. The loading type of the machine was four-point bending; the stress state of the specimen under rotary bending was reversed bending. The static and static fatigue tests were performed using the non-rotating fatigue machine. The static test was performed by increasing the applied stress gradually until the specimen was fractured. The static fatigue

test was performed by holding a constant load, and the held time was measured with a hand made apparatus capable of digital display for time.

Fractured surfaces of specimens were observed by both the optical microscope and the scanning electron microscope (SEM). The size of mirror-like region was determined by the optical microscope with a magnification of 25.

3. Results and discussion

3.1 Rotary bending fatigue strength

Rotary bending fatigue test was carried out generally within the range 10^4 to 10^8 stress-cycles. Some specimens were tested at cyclic numbers more than 10^8 to examine the existence of a knee; 10^8 stress-cycles is equivalent to about 20 days under the present test condition. Test points were plotted on a logarithmic graph shown in Fig. 1. In the figure, the arrowed points indicate those stopped testing. The straight line was obtained by the least squares method from the data except the arrowed points. As seen in the figure, the S - N curve can be approximately represented by a straight line up to about 10^8 stress-cycles and can be expressed by the formula,

$$\sigma^n N = \text{const.} \quad (1)$$

where σ is the stress amplitude and N is number of cycles to failure. The exponent n was 49.7, which was obtained from the slope of the straight line. The value of n for the present material was larger than the value for the previous sintered Si_3N_4 , as described later in Section 3.3.

As seen in the figure, the material has the character that its life increases remarkably as the stress amplitude decreases. In addition, it does not have a distinct knee at cyclic numbers less than 10^7 ; two specimens are fractured at 3.75×10^7 and 1.23×10^8 stress-cycles which are equivalent to about 8 and 25 days, respectively. However, from the figure, the knee seems to exist at cyclic numbers more than 10^8 ; the fatigue limit seems to be about 40 kg/mm². A similar S - N curve, indicating the existence of a knee, was obtained in the previous study³⁾ on sintered Si_3N_4 whose assumed fatigue limit was about half that of the present material. These rotary bending test results suggest that fatigue testing should be carried out at least for more than several tens of days for examining the basic fatigue strength of sintered

Fig. 1. S - N curve.

Si_3N_4 . Although it was reported⁶⁾ that sintered Si_3N_4 was fractured at cyclic numbers even more than 10^9 , this test was performed at very high frequency, 3 kHz; it took only about 4 days to obtain the data at 10^9 stress-cycles. As it can be considered that there is influence of frequency on number of cycles to failure, it seems that the specimens in the present test, which are not fractured under near assumed fatigue limit, will not be always fractured at cyclic numbers more than 10^9 .

3.2 Static fatigue strength

Static fatigue test was carried out generally for less than 10^6 seconds. Some specimens were tested more than 10^6 seconds to examine the existence of a knee. Test points were plotted on a logarithmic graph shown in Fig. 2. In the figure, the arrowed points indicate those stopped testing. The straight line was obtained by the least squares method from the data except the arrowed points. As seen in the figure, the S - t curve can be approximately represented by a straight line up to about 10^6 seconds and can be expressed by the formula,

$$\sigma^n t = \text{const.} \quad (2)$$

where σ is the applied stress and t is the time to failure. The exponent n was 179.1, which was obtained from the slope of the straight line. This value was much larger than the value of n obtained under rotary bending.

It is seen from the figure that the static fatigue limit, defined as the stress level below which prolonged service life expected, seems to exist at the time more than 10^6 seconds; the static fatigue limit seems to be 55 kg/mm². A similar S - t curve, indicating the existence of a knee, was obtained on the previous sintered Si_3N_4 ;⁷⁾ the test was performed on rectangular rods, using a four-point apparatus and a servo-hydraulic fatigue testing machine.

3.3 Comparison of fatigue behavior under rotary bending with that under static fatigue

It was confirmed that the fatigue strength under rotary bending decreased more remarkably than that under static fatigue. Each value of n for present and previous materials is listed in Table 1. It is clear that the value obtained under rotary bending whose stress state is reversed bending is small in comparison with that obtained under static fatigue. The similar tendency is understood, comparing with the values of n for sintered Si_3N_4 under tension-

Fig. 2. S - t curve.

Table 1. n for sintered Si_3N_4 at room temperature.

n	Experimental method	Reference
50	cyclic fatigue(reversed)	present study
179	static fatigue	present study
30	cyclic fatigue(reversed)	previous material, 1
75	static fatigue, four-point bending	previous material, 7
58	D.T.	previous material, 5

compression⁸⁾ and dynamic fatigue.⁹⁾ It is further seen that the value of n in reversed bending is smaller than that in repeated unidirectional bending, comparing with the reported data¹⁰⁾ on sintered Si_3N_4 in a four-point bending fatigue test. It is considered²⁾ that n is different because of the applied stress state. Although the mechanism is not clear, the applied stress state seems to have direct effect upon the crack growth behavior, comparing with the reported data¹¹⁾ on compact tension sintered Si_3N_4 specimens. It is also seen from the table that the value of n for the present material is larger than that for the previous material. The value of n may be different because of the material microstructure such as grain size, grain morphology and the character of grain boundary; the character of grain boundary such as glassy phase seems to have strong influence on the crack growth behavior.⁹⁾

It was confirmed that the fatigue strength under rotary bending was much lower than that under static fatigue. The assumed fatigue limits were 40 kg/mm² and 55 kg/mm², and were 63% and 86% of the mean static strength, respectively; a static bending test was performed on bar specimens using the non-rotating fatigue machine, and the mean static strength for 7 specimens was 64 kg/mm². By comparison of the fatigue limit under rotary bending $\sigma_{\text{R.B.}}$ with the static fatigue limit $\sigma_{\text{S.F.}}$, the ratio was obtained:

$$\sigma_{\text{R.B.}}/\sigma_{\text{S.F.}}=0.73 \quad (3)$$

The effective volume V_E of the specimen for rotary bending is different from that for static fatigue although the specimen geometry is the same. Taking into account of the size effect,¹²⁾ $\sigma_{\text{R.B.}}$ is related to $\sigma_{\text{S.F.}}$ by the following equation

$$\sigma_{\text{R.B.}}/\sigma_{\text{S.F.}}=(V_{\text{E,S.F.}}/V_{\text{E,R.B.}})^{1/m} \quad (4)$$

where m is the Weibull modulus. The value of m was 11.9, which was obtained from the distribution of static strengths for 40 specimens by a four-point bending test; the test specimens were rectangular rods with 3 mm thick and 4 mm wide, which were cut from the bar specimens made for the present study, and were ground perpendicularly to the longitudinal axis. The effective volume V_E is defined:

$$V_E=\int_V (\sigma/\sigma_{\text{max}})^m dV \quad (5)$$

The effective volumes $V_{\text{E,R.B.}}$ under rotary bending and $V_{\text{E,S.F.}}$ under static fatigue are obtained as follows:

$$V_{\text{E,R.B.}}=2V/(m+2) \quad (6)$$

$$V_{\text{E,S.F.}}=(2V/\pi)\int_0^{\pi/2}\sin^m\theta\cos^2\theta d\theta \quad (7)$$

where V is the volume of the middle part of the specimen. Taking $m=11.9$ and applying Eqs. (6) and (7), the ratio of $\sigma_{\text{R.B.}}$ to $\sigma_{\text{S.F.}}$ in Eq. (4) was 0.83. As this value is larger than that in Eq. (3), it is seen that the fatigue limit under rotary bending is lower than that under static fatigue, even taking into account of the size effect. It is considered that the initiation stress for the crack growth from the initial flaw under rotary bending is lower than that under static fatigue.

3.4 Mirror-like region of fractured surface

It was known by macroscopic observation that each fractured surface after both fatigue tests had a flat region and a rough region, as pointed out on previous sintered Si_3N_4 .^{1,7)} the flat region was referred to as mirror-like region. Each macrophotograph indicating the feature is shown in Figs. 3(a) and (b), respectively; mirror-like region is located to the highest part of each figure. The overall fractured surface after rotary bending test was relatively smooth compared with that after static fatigue test. However, it seemed that this difference was simply due to the magnitude of applied stress, since the fractured surface seemed to be rougher as the fracture stress was high; the rotary bending fractured surface of the present material was much rougher than that of the previous material.

Typical microphotographs of the fractured surface after rotary bending test are shown in Figs. 4(a) and (b). In each figure, Fig. 4(a) shows the microphoto-

Fig. 3. Overall fractured surfaces after fatigue tests. (a) After rotary bending test ($\sigma=42$ kg/mm², $N=2.94 \times 10^6$), (b) After static fatigue test ($\sigma=58$ kg/mm², $t=2.49 \times 10^5$ s).

Fig. 4. Typical microphotographs of fractured surface after rotary bending test ($\sigma=44$ kg/mm², $N=6.96 \times 10^5$), (a) of mirror-like region, (b) of the area surrounding mirror-like region.

graph of mirror-like region and Fig. 4(b) shows the area surrounding the region. The fracture occurred mainly as an intergranular process and the fracture features were not different in both regions. There was, moreover, no distinct boundary line of both regions under microscopic observation. It seemed⁷⁾ that the difference between the mirror-like region and the rough region was simply due to the irregularities in the fractured surface. Microscopic observations on the fractured surface after rotary bending test were similar to those after static fatigue test and to those after static test; no characteristic sign of cyclic fatigue fracture could be found out.

3.5 Character of mirror-like region

In many cases of the present material, the mirror-like region had the fracture origin such as a large pore or a porous region. Some specimens had mirror-like region which was located to the interior of the specimen, as shown in Fig. 5. This kind of mirror-like region was left out of consideration in this study.

In the case that the fracture initiation part was near the specimen surface, the mirror-like region was roughly semicircular and fracture propagated radially from this area, as shown in Fig. 6. The size of mirror-like region for various applied stresses of rotary bending, static fatigue and further static test was examined by the optical microscopic photo-

Fig. 5. Mirror-like region in the interior of fractured surface ($\sigma=43 \text{ kg/mm}^2$, $N=1.02 \times 10^7$).

Fig. 6. Typical mirror-like region of fractured surface ($\sigma=42 \text{ kg/mm}^2$, $N=2.94 \times 10^6$).

graphs. The relation between the depth of mirror-like region d and the applied stress σ is shown in Fig. 7. The straight line shown in the figure was obtained by the least squares method, considering three kinds of data as the same thing. As seen in the figure, the relation can be represented by the same straight line regardless of the loading condition, and can be expressed by the formula,

$$\sigma d^s = \text{const.} \quad (8)$$

The exponent s was 0.59, and the value was about the same as the literature value⁴⁾ which was obtained from the relation between mirror radius and the applied stress under static test.

The stress intensity factor K_I at the deepest point in the mirror-like was calculated, assuming mirror-like region as the semicircular surface crack. In the calculation, the expression¹³⁾ reported on a surface crack in a shaft was utilized. The formula is as follows:

$$K_I = F\sigma\sqrt{\pi b} \quad (9)$$

where σ is the bending stress, b is the crack depth, and F is the dimensionless quantity dependent on the shape of the crack, the ratio of the crack depth to the specimen size. The relation between the depth of mirror-like region d and stress intensity factor K_I obtained under each loading condition is almost constant, independent of the depth of mirror-like region. The mean and standard deviation are shown in the right side of the figure. The mean value was

$$K_I = 9.5 \text{ MN/m}^{3/2} \quad (10)$$

It was considered that the failure occurred when the

Fig. 7. Relation between depth of mirror-like region and applied stress.

Fig. 8. Relation between depth of mirror-like region and stress intensity factor.

size of mirror-like region reached a critical value dependent on the applied stress regardless of the loading condition. Although the critical value seemed to relate to fracture toughness K_{IC} of the material, the value obtained was higher than K_{IC} ; K_{IC} of the present material was $6.7 \text{ MN/m}^{3/2}$ which was measured by the Vickers indentation method.⁵⁾ The difference can not be explained fully.

The life of ceramics seemed to be controlled by the subcritical crack growth from the initial flaw. The subcritical crack growth behavior seemed to be different because of the applied stress state, as discussed in Section 3.3. However, it was known that the size of mirror-like region was correlated with the applied stress regardless of the loading condition, and that the failure occurred when its size reached a critical value dependent on the applied stress. It is considered that the difference of the fatigue behavior under rotary bending and static fatigue is due to the subcritical crack growth behavior in the mirror-like region. It is supposed that the K_I - V diagrams assumed in mirror-like regions are similar to those¹⁴⁾ measured on compact tension sintered Si_3N_4 specimens under reversed stress and constant stress.

4. Conclusions

The fatigue behavior of sintered Si_3N_4 under rotary bending and static fatigue was examined. The character of mirror-like region observed on each fractured surface was also examined. The results obtained are summarized as follows:

(1) The life of the material increased as the stress amplitude or the applied stress decreased. The S - N curve and the S - t curve could be represented by the straight line up to about 10^8 stress-cycles and 10^6 seconds, and could be expressed by the formulas, $\sigma^n N = \text{constant}$ and $\sigma^n t = \text{constant}$, respectively. The exponent n under rotary bending was much smaller than that under static fatigue.

(2) The knees seemed to exist at cyclic numbers more than 10^8 and at the time more than 10^6 seconds, respectively. The assumed fatigue limit under rotary

bending was lower than that under static fatigue, even taking into account of the size effect.

(3) The fracture features of fractured surfaces after both fatigue tests could not be distinguished, and the features were similar to those after the static test.

(4) The size of mirror-like region was correlated with the magnitude of applied stress regardless of the loading condition, and stress intensity factor at the deepest point in the region was almost constant.

(5) It was considered that the difference of fatigue behavior under rotary bending and static fatigue was due to the subcritical crack growth behavior in the mirror-like region.

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