

**Influence of Wave Form and Compressive Load  
on the Crack Propagation Behavior of a Sintered Silicon Nitride under  
Cyclic Load**

by

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and

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Abstract

In order to know influences of load wave form and compressive loads on the crack propagation behavior of sintered  $\text{Si}_3\text{N}_4$  under cyclic loads, the crack propagation rate was measured using compact tension specimens at room temperature. The main results obtained are as follows.

(1) Influence of stress ratio and test frequency on the crack propagation behavior under sinusoidal wave form were equal to those under square wave form .

(2) In the range of high  $K_{I\max}$ , the crack propagated nearly time-dependently. In the range of low  $K_{I\max}$ , the crack propagated cycle-dependently.

(3) Crack closure phenomenon was observed using the unloading elastic compliance method.

(4) The effective stress intensity factor range  $\Delta K_{\text{eff}}$ , together with  $K_{I\max}$ , can be a reasonable parameter to describe the crack propagation rate.

(5) The compressive load crushed the debris, lowered the crack opening load and resulted in a large  $\Delta K_{\text{eff}}$  which promote the increase of the crack propagation rate.

(6) In the crack propagation rate of this material under cyclic loads,  $K_{I\max}$  is the most important and  $\Delta K_{\text{eff}}$  is secondarily important.

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## 1. Introduction

Sintered silicon nitride is one of the most promising candidate as high temperature structural ceramics. Therefore, it is important to study its strength characteristics under cyclic load. We studied on the crack propagation behavior of two kinds of sintered silicon nitride under the cyclic load using compact tension (CT) specimen<sup>1-4</sup>, and many important characteristics such as the effects of stress ratio, the test frequency and fracture toughness on the crack propagation rate were made clear. Furthermore, these behaviors were compared with those of metals under corrosive environment. It was concluded that the crack propagation behaviors of sintered silicon nitride under cyclic load in ambient environment was much the same those of metals under cyclic stress corrosion cracking (SCC) condition<sup>2,3</sup>.

In this study, the crack propagation rate of CT specimen under the cyclic load was measured, and influence of compressive load and stress wave form were investigated. The crack closure load was also measured using unloading elastic compliance method, and importance of  $K_{I\max}$  and  $\Delta K_{\text{eff}} (= K_{I\max} - K_{\text{open}})$  on the crack propagation behavior was discussed.

## 2. Experimental

Material used in this study was a sintered silicon nitride produced by Toyota Motor Corp. being doped with  $Y_2O_3$  and  $Al_2O_3$ . The mechanical properties of the material are shown in Table 1. Fracture toughness of each specimen was estimated with IF method and denoted by  $K_C$ . Size and form of the test specimen is shown in Fig.1. The crack

Table 1 Mechanical properties of material tested.

Bending strength $\sigma_{3b}$ (MPa)	Young's modulus $E$ (GPa)	Fracture toughness $K_C$ (MPa $\sqrt{m}$ )	Hardness $H_v$
740	310	5.55	1257

length was measured with a metallurgical microscope. Sinusoidal stress was applied to the specimen in the controlled environment at 298K and 50% relative humidity. The crack closure behavior was detected through strain gages using unloading elastic compliance method<sup>5</sup>.

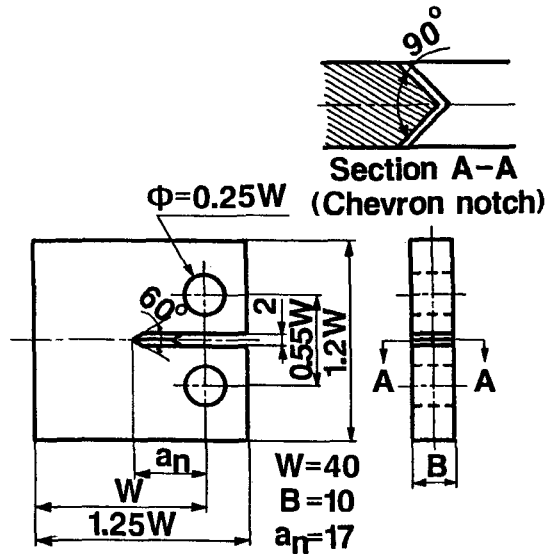


Fig.1. Configuration and dimensions of CT specimen.

### 3. Results and discussion

#### 3.1. Influence of test frequency on the crack propagation rate

Crack propagation rate under cyclic load with various test frequency  $f$  is shown in Fig.2. Stress ratio  $R$ , which was defined by  $\sigma_{\min}/\sigma_{\max}$ , was 0.1. The crack propagation rate  $da/dt$  was related to the crack propagation rate  $da/dN$  by formula (1).

$$da/dt = da/dN \times f \quad \dots \quad (1)$$

As the frequency increased, crack propagation rate increased in all test range. In order to distinguish the cycle-dependent crack propagation and the time-dependent crack propagation<sup>6</sup>), Fig.3 was reproduced from Fig.2. The scale of each coordinate system is the same in this figure. As inclination of the solid line is at 45 in the range of  $K_{I\max}$  equal or smaller than  $5.25\text{MPa}\sqrt{\text{m}}$ , the crack propagation is believed to depend fully on the

number of cycles. Inclination of the solid line approached to the horizontal line as  $K_{I\max}$  increased from  $5.25\text{MPa}\sqrt{\text{m}}$ . It is evident that time-dependent crack propagation prevailed increasingly as  $K_{I\max}$  increased. These crack propagation behaviors are about the same as those of our previous study<sup>4</sup> under cyclic stress with square wave form.

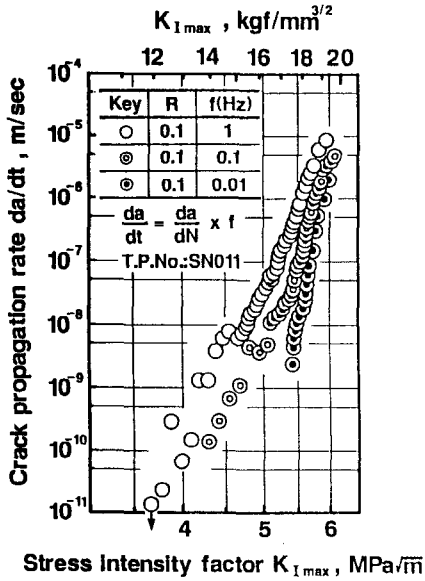


Fig.2. Relationship between crack propagation rate  $da/dt$  and maximum stress intensity factor  $K_{I\max}$  ( $R=0.1$ ).

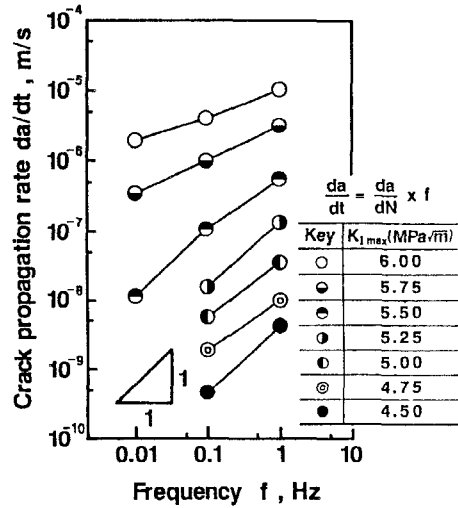


Fig.3. Relationship between crack propagation rate  $da/dt$  and test frequency  $f$ .

### 3.2. Influence of stress ratio on the crack propagation rate

The crack propagation rate under various stress ratios is shown in Fig.4. In this figure, normalized stress intensity factor  $K_{I\max}/K_C$  was used instead of  $K_{I\max}$ , in order to show the stress ratio effect clearly<sup>4</sup>. It is evident that, as the stress ratio decreased, the crack propagation rate increased. The relationship between the stress ratio  $R$  and  $K_{I\max}/K_C$  is shown in Fig.5. Although the decrease in the stress ratio resulted in increase in the crack propagation rate, degree of the stress ratio effect on the crack propagation rate differs largely whether the stress ratio is plus or minus. Further discussion is made afterwards along with the crack closure phenomenon.

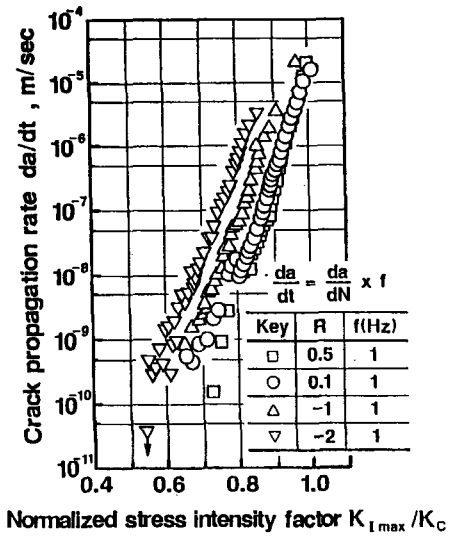


Fig.4. Relationship between crack propagation rate  $da/dt$  and normalized stress intensity factor  $K_{I_{max}}/K_C$  ( $f=1\text{Hz}$ ).

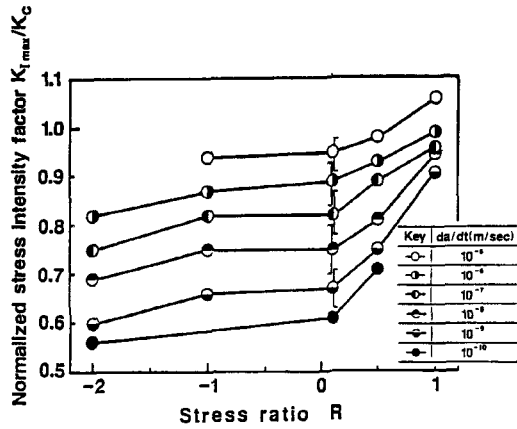


Fig.5. Relationship between stress ratio  $R$  and normalized stress intensity factor  $K_{I_{max}}/K_C$ .

3.3. Influence of stress wave form on the crack propagation rate

Figure 6 shows an example of the comparison of the crack propagation rate under sinusoidal wave form and square wave form. It is concluded that the crack propagation rate under square wave form was 7.5 times as much as that under sinusoidal wave form. As time-dependent crack propagation prevails in case of a large  $K_{I_{max}}$  as shown in Fig.3, the ratio of the crack propagation rate under square wave form to that under sinusoidal wave form was calculated on

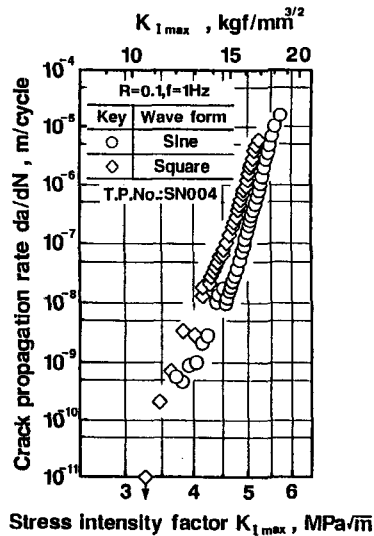


Fig.6. Comparison of crack propagation rate under sine wave and square wave ( $R=0.1, f=1\text{Hz}$ ).

the assumption that only time-dependent crack propagation occurred. This calculation was carried out by using the formula developed by A.G.Evans<sup>7</sup> and an exponent  $n(=83)$  which was obtained through  $K_{I\max}-da/dt$  relationship of a straight line under static load ( $da/dt = CK_{I\max}^n$ ). The calculated result was 7.7. This is very close to the above mentioned experimental result 7.5. However, the reason of the difference in the crack propagation rate in the range of small  $K_{I\max}$  as seen in Fig.6 cannot be made clear so far.

### 3.4. Crack closure phenomenon

Figure 7 is a scanning electron micrograph. It shows debris which is thought to be the grain crushed between the crack surfaces under cyclic load, and it also shows wear mark. As these were seen only on the fracture surface under cyclic loads and could not be seen on the fracture surface under monotonic load, it can be said that such debris and wear mark characterize the fracture surface under cyclic loads.

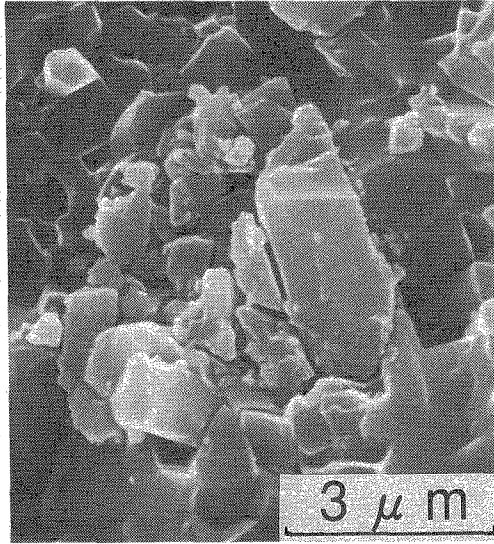


Fig.7. Scanning electron micrograph of fracture surface under cyclic load.

Load-displacement relationships obtained by using unloading elastic compliance method<sup>5</sup> are shown in Fig.8. From these figures, crack opening loads were determined as shown with bars in Fig.8. Relationship between  $K_{I\max}$  and  $K_{open}/K_{I\max}$  is shown in Fig.9. It can be seen that as stress ratio decreased from 0.5 to -2,  $K_{open}/K_{I\max}$  decreased consistently and the effective stress intensity factor range  $\Delta K_{eff}(=K_{I\max}-K_{open})$  increased. From above discussion, it is concluded that the compressive load crushed the debris more severely, and caused the crack opening load decrease,  $\Delta K_{eff}$  increase, and finally the crack propagation rate increased. Relationship between  $\Delta K_{eff}/K_{I\max}$  and  $K_{I\max}/K_C$  is summarized in Fig.10.  $K_{I\max}/K_C$  decreased linearly as  $\Delta K_{eff}/K_C$  increased.  $\Delta K_{eff}$  is superior to  $\Delta K$  in order to describe the crack propagation rate of the sintered

silicon nitride. Tanaka et al.<sup>8</sup> used  $\Delta K_{eff}$  successfully to describe the crack propagation rate of a sintered silicon nitride.

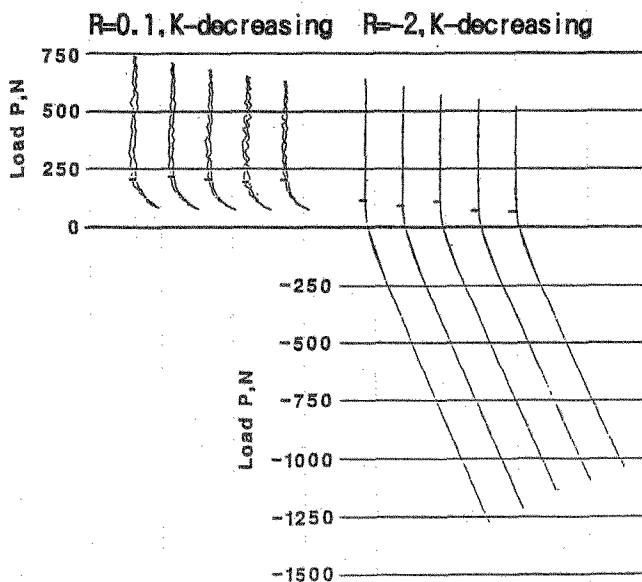


Fig.8. Examples of load-displacement curve.

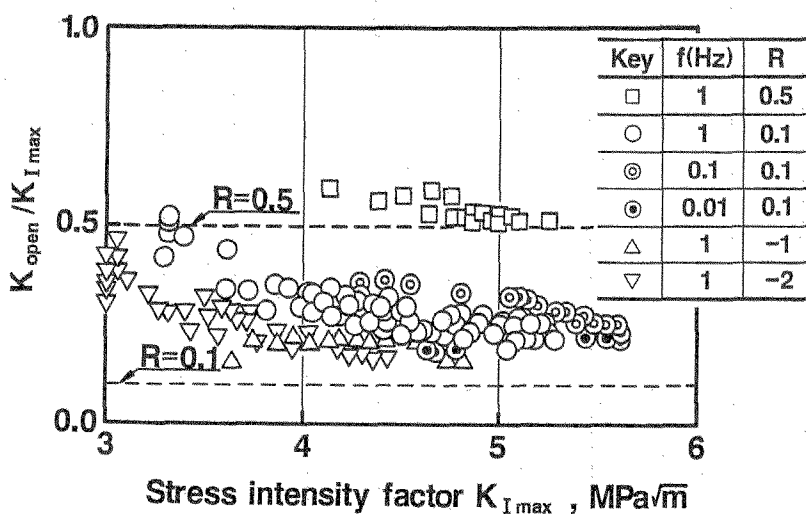


Fig.9. Relationship between  $K_{open}/K_C$  and maximum stress intensity factor  $K_{I_{max}}$ .

Next, an attempt was made to introduce the equivalent stress intensity factor range  $\Delta K_{eq}$ <sup>9</sup> defined by formula (2).

$$\Delta K_{eq} = \Delta K(1 - R)^{-\gamma} = \Delta K^{1-\gamma} K_{I_{max}}^{\gamma} \quad \dots \quad (2)$$

Replacing  $\Delta K$  with  $\Delta K_{eff}$ , the equivalent effective stress intensity factor range  $\Delta K_{eff,eq}$  can be obtained as follows.

$$\Delta K_{eff,eq} = \Delta K_{eff}(1 - R_{eff})^{-\gamma'} = \Delta K_{eff}^{1-\gamma'} K_{I_{max}}^{\gamma'} \quad \dots \quad (3)$$

Exponent  $\gamma$  or  $\gamma'$  relate to the contribution toward  $K_{I_{max}}$  to the crack propagation rate, and  $1-\gamma$  or  $1-\gamma'$  relate to the contribution to  $\Delta K$  or  $\Delta K_{eff}$  to the crack propagation rate.  $R_{eff}$  is the actual stress ratio determined considering the crack closure.  $\gamma'$  calculated on the basis of experimental results at stress ratio of 0.5 and 0.1 is shown in Fig.11.  $\gamma'$  is nearly unity, and  $\gamma$  decreases slightly as the crack propagation rate decreases. It is concluded that in case of high crack propagation rate only  $K_{I_{max}}$  is important for the crack to propagate, and in case of low crack propagation rate  $\Delta K_{eff}$  affects the crack propagation rate in greater or less degree. In conclusion, the most important factor is  $K_{I_{max}}$  and  $\Delta K_{eff}$  is important in secondary meaning for the crack propagation of sintered silicon nitride.

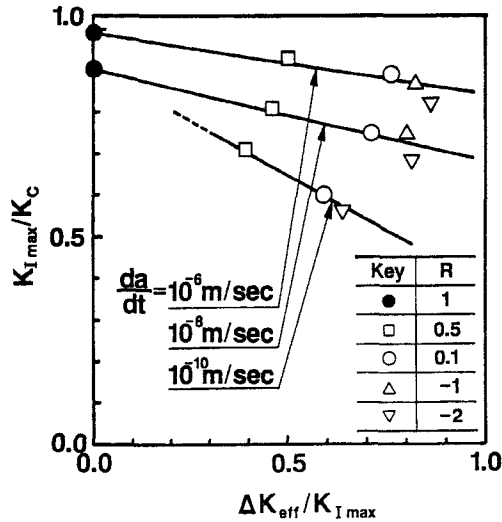


Fig.10. Relationship between normalized maximum stress intensity factor  $K_{I_{max}}/K_C$  and normalized effective stress intensity factor  $\Delta K_{eff}/K_{I_{max}}$ .



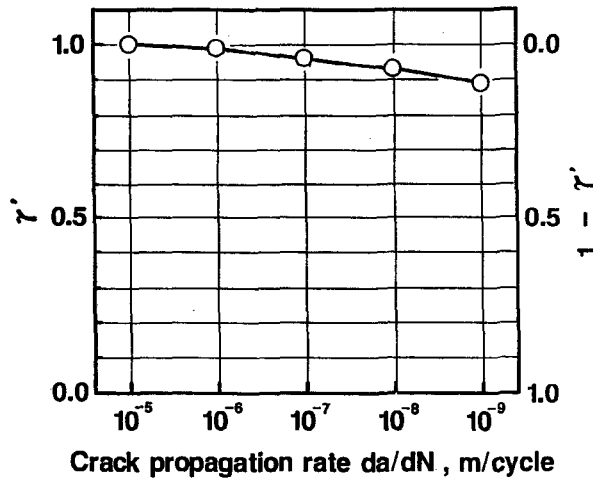


Fig.11. Relationship between  $\gamma$  and crack propagation rate  $da/dN$ .

#### 4. Conclusion

Using CT specimen made by sintered silicon nitride, the crack propagation rate under sinusoidal cyclic loads was measured and analyzed. The results obtained are as follows.

(1) Stress ratio and test frequency effects on the crack propagation rate under sinusoidal cyclic wave form is much the same that under square cyclic wave form. As the stress ratio decreased or test frequency increased, crack propagation rate increased. Only cycle-dependent crack propagation occurred in case of small  $K_{I\max}$ , and time-dependent crack propagation prevailed in case of large  $K_{I\max}$ .

(2) The crack propagation rate under square wave form was 7.5 times as much as that under sinusoidal wave form. The ratio of the crack propagation rate, which is calculated under the assumption that time-dependent crack propagation occurred, is 7.7, which is very close to the average experimental result of 7.5.

(3) Crack closure phenomenon was observed which came from debris getting stuck between the fracture surfaces.

(4) The crack closure load was measured under several stress ratio, involving compressive load. It was made clear that compressive load lowered the crack closure load and then increased  $\Delta K_{\text{eff}}$  and finally increased crack propagation rate.

(5) In the crack propagation rate of this material under cyclic loads,  $K_{\text{Imax}}$  is the most important and  $\Delta K_{\text{eff}}$  is secondarily important.

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