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The environmental effect on cyclic fatigue behaviour in ceramic materials

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In order to understand the environmental effects on cyclic fatigue, static and cyclic fatigue behaviour was investigated in air and in vacuum for normally sintered silicon nitride and alumina. The cyclic fatigue lifetime in vacuum is considerably longer than that in air, indicating a remarkable stress corrosion cracking effect in the latter, especially in alumina. In addition, the cyclic loading effect in vacuum is almost the same in silicon nitride relatively insensitive to environmental effects and alumina susceptible environmental effects. From such results, it has been found that cyclic fatigue in air is approximately expressed as the superposition of pure cyclic loading effect, which is defined as cyclic loading effect in vacuum, and environmental effect. This relation was applied to some kinds of ceramics with different values of fracture toughness or different microstructures and the results obtained were discussed.

1. Introduction

In recent years, the cyclic fatigue behaviour of ceramics has been actively investigated. It has been shown that crack growth occurs under cyclic loading condition in some ceramics like alumina [1–5], silicon nitride [6–11] and Mg–PSZ [12, 13], which is not explained by the subcritical cracking mechanism due to static fatigue alone. That is, the cyclic fatigue life is less than that predicted using the static data by integrating over the fatigue cycle and also the cyclic fatigue crack growth rate, as the function of the maximum applied stress intensity, is faster than the predicted one. While the precise micro-mechanisms for such cyclic fatigue behaviour are still unclear, it has been confirmed by many experimental results that such cyclic loading effect depends upon stress ratio [7, 10], crack size [9, 14–16], microstructure [8], etc., as is generally observed in metallic materials.

Since most of the studies on cyclic fatigue of ceramics have been investigated in air and most ceramics are susceptible to stress corrosion cracking, it is supposed that cyclic fatigue damage in air contains a considerable environmental damage component. In fact, direct evidence of environmental effects on cyclic fatigue has been found in studies in an inert atmosphere or vacuum and in air for Mg–PSZ [14] and silicon nitride [17, 18]. Noticeably, the cyclic loading effects in these materials are demonstrated in inert atmosphere or vacuum, but the cyclic fatigue lives are considerably longer in an inert atmosphere or vacuum than in air. These results imply that a large part of strength deterioration by cyclic loading in air is attributed to environmental effects. Despite such an environmental effect on cyclic behaviour in air, this influence has not been taken into consideration in most of the studies on cyclic fatigue. Strength deterioration due to cyclic loading in air, $\Delta\sigma_c$(air) consists of the environmental damage component, $\Delta\sigma_s$(air) and the pure cyclic damage component $\Delta\sigma_c$(pure). However, the relation among these is not well known so that it is important to split it into two components in order to understand cyclic fatigue behaviour.

Assuming that $\Delta\sigma_c$(air) is expressed as the superposition of $\Delta\sigma_s$(air) and $\Delta\sigma_c$(pure), it is supposed that $\Delta\sigma_c$(pure) is equal to the strength deterioration due to cyclic loading in vacuum. In this work, in order to investigate whether or not such a relation is valid, static and cyclic fatigue behaviour in air as well as in vacuum has been investigated for silicon nitride insusceptible to static fatigue in air and alumina susceptible to static fatigue in air.

2. Experimental procedure

Silicon nitride (Si_3N_4) and alumina (Al_2O_3) were prepared for the present experiment. Silicon nitride (SSN–1) was normally sintered at 1750 °C with Y_2O_3–$MgAl_2O_4$ additives. It has a rod-like grain structure. The grain size was considerably inhomogeneous and it was thus, difficult to determine average grain size. Alumina was normally sintered using the high purity powder (99.99 wt % Al_2O_3) at

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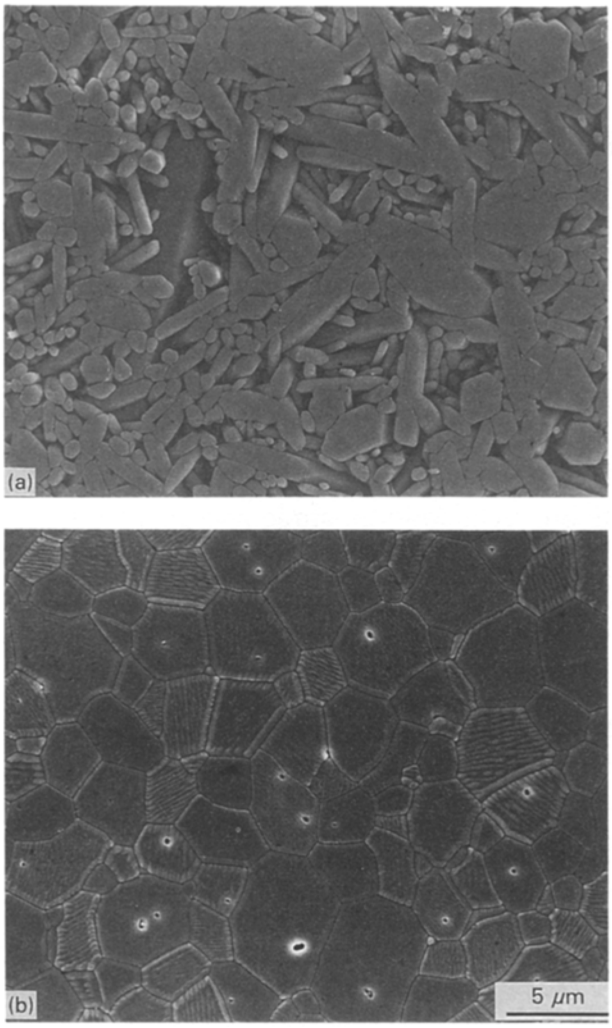

Figure 1 Scanning electron micrographs of polished and etched surfaces; (a) silicon nitride, (b) alumina.

1600 °C without additives and subsequently HIPed at 1400 °C. Its average grain size was about 4 μm. Microstructures of these materials are shown in Fig. 1; (a) silicon nitride and (b) alumina. Fracture toughness was measured by using the indentation fracture method. The fracture toughness values calculated from the equation of Anstis *et al.* [19] were 5.6 MPa $m^{1/2}$ for silicon nitride and 3.0 MPa $m^{1/2}$ for alumina. These materials, with dimensions of about $5 \times 5 \times 45$ mm, were ground and lapping-polished to produce the specimens for fatigue testing, with dimensions $4 \times 4 \times 40$ mm. Before fatigue testing, a precrack of length about 350 μm was introduced at the centre of the specimen by a Vickers indenter, using loads of 490 N for silicon nitride and 196 N for alumina.

Static and cyclic fatigue tests were conducted in four point bending (outer span 30 mm, inner span 10 mm). In cyclic fatigue a sign-wave loading was used at a stress ratio of $R = 0.1$ and a frequency of 20 Hz, using an electrohydraulic testing system. Experiments were carried out at room temperature in air and in vacuum (2×10^{-5} torr).

3. Results and discussion

3.1. Fatigue behaviour

Static and cyclic fatigue lifetime in air, as a function of the applied stress (or maximum stress), for SSN–1 and alumina are presented in Fig. 2(a) and (b). Arrows designate long-term survivors. The static fatigue effect in alumina is prominent, compared to that in SSN–1. Cyclic fatigue lifetimes are much less than static fatigue lifetimes in both materials and the existence of a cyclic loading effect in these materials is thus obvious. Cyclic fatigue lifetimes in vacuum in these materials are shown in Fig. 3. Cyclic fatigue damage is recognized even in vacuum. A similar tendency has been also found in other studies [14, 17, 18]. To clarify the extents of static fatigue damage and cyclic fatigue damage in both materials, the fatigue data were replotted by using the strength deterioration rate, σ/σ_f, defined as the stress values normalized by the flexural strength, σ_f, for each material, as shown in Fig. 4(a) for in air and 4(b) for in vacuum. From Fig. 4(a), it is indicated that σ/σ_f in both static and cyclic fatigue in air are less in SSN–1 than in alumina. Contrary to this, σ/σ_f in cyclic fatigue in vacuum is almost same in both materials (Fig. 4(b)), and further, σ/σ_f in vacuum is much larger than that in air. Such results indicate that since the cyclic fatigue in air is subjected to substantial environmental damage, σ/σ_f under cyclic loading is smaller in alumina susceptible to environmental effects as compared to SSN–1 which is relatively insusceptible to such effects, while the pure cyclic loading effect, which means the cyclic loading effect without environmental effect, is approximately equal in both materials.

3.2. Superposition of pure cyclic loading effect and environmental effect

Assuming that cyclic fatigue effect in air, $\Delta\sigma_c$(air) is defined by superposition of pure cyclic loading effect, $\Delta\sigma_c$(pure) and static fatigue effect under cyclic load, $\Delta\sigma_s$(air), as shown schematically in Fig. 5, the relation among those is given by Equation 1

$$\Delta\sigma_c(\text{air}) = \Delta\sigma_c(\text{pure}) + \Delta\sigma_s(\text{air}). \qquad (1)$$

If the relation between stress and lifetime under cyclic and static loads in air are known, the value of $\Delta\sigma_c$(pure) can be obtained. The static fatigue lifetime is generally given as

$$t_s = A\sigma_s^{-n} \qquad (2)$$

where t_s is time to failure under static load (the applied stress), σ_s and A and n are constants. According to Evans *et al.* [20], under the assumption that there is no enhanced cyclic loading effect, the time to failure under cyclic load, t_p, is expressed as

$$t_p = B\sigma_p^{-n} \qquad (3)$$

where σ_p is cyclic fatigue stress predicted from the data of static load, $B = Ah^{-1}$. For sinusoidal with

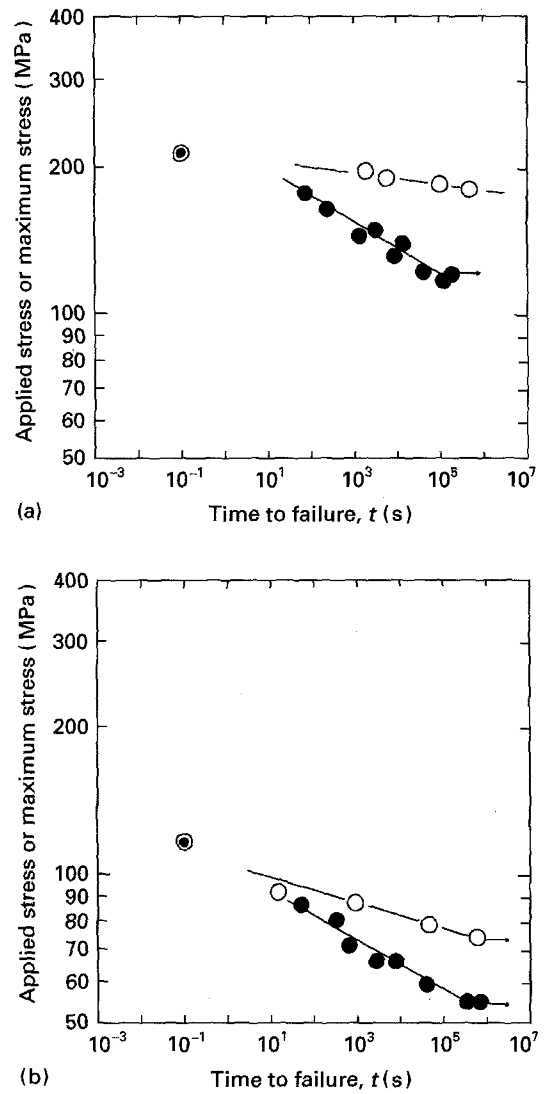

Figure 2 Static (○) and cyclic (●) fatigue lifetimes in air as a function of the applied stress or maximum stress; (a) silicon nitride, (b) alumina.

stress ratio R, h is given as following

$$h = \sum_{l=0}^{(n/2)T}\left[\frac{n!}{(n-2l)!(l!)^2}\right]\left[\frac{1-R}{2(1+R)}\right]^{2l}\left(\frac{1+R}{2}\right)^n \tag{4}$$

Also time to failure under cyclic load t_c is approximately expressed as

$$t_c = C\sigma_c^{-m} \tag{5}$$

where σ_c is the maximum applied stress. For $t_c = t_p = t$

$$\Delta\sigma_c(\text{pure}) = \log\frac{\sigma_c}{\sigma_p} = \left(\frac{1}{n} - \frac{1}{m}\right)\log t - D \tag{6}$$

where D is constant. If Equation 6 is true, the extent of pure cyclic loading effect in air is identical with that of cyclic loading effect in vacuum.

Fig. 6 shows the comparison of pure cyclic loading effect in air calculated from Equation 6 with cyclic loading effect in vacuum for (a) silicon nitride and (b) alumina. From these figures, it is inclined that cyclic

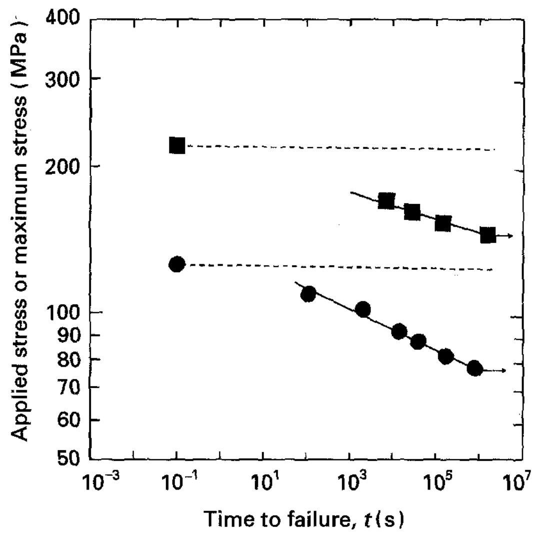

Figure 3 Cyclic fatigue lifetimes in vacuum as a function of the maximum stress in silicon nitride (■) and alumina (●).

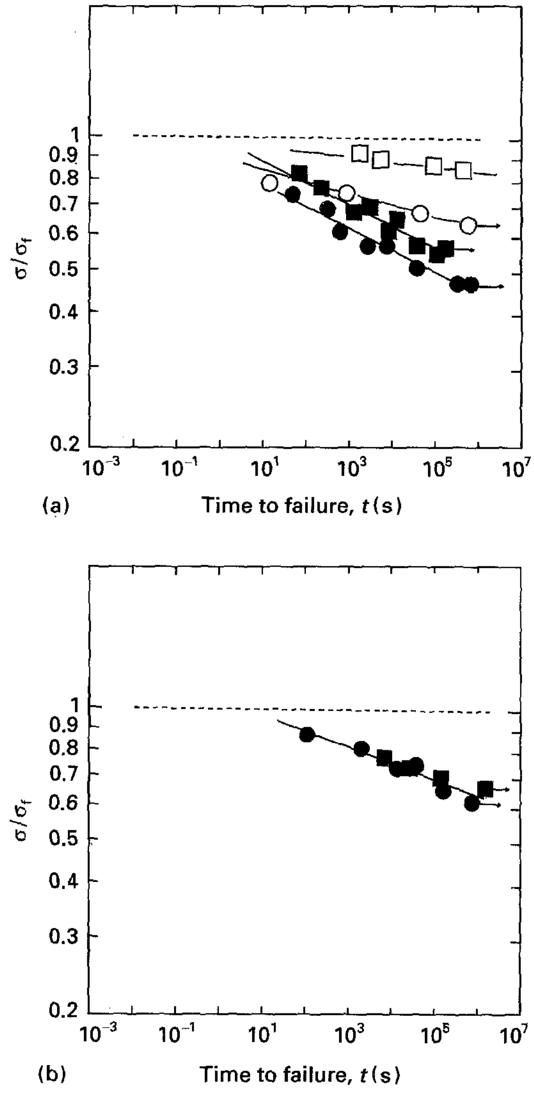

Figure 4 Replots of data (a) in air of Fig. 2 and (b) in vacuum of Fig. 3 using the stress values normalized by the flexural strength for each material. ○, Al_2O_3 (static); ●, Al_2O_3 (cyclic); □, Si_3N_4 (static); ■, Si_3N_4 (cyclic).

loading effect in vacuum is a little less than that in air. However, such differences can be ignored, considering the change of mechanical constant such as friction

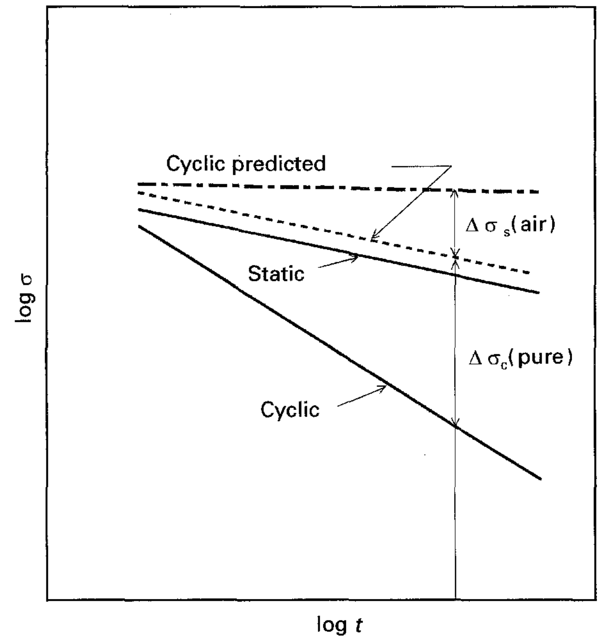

Figure 5 Schematic illustration of static and cyclic fatigue lifetimes of ceramics in air.

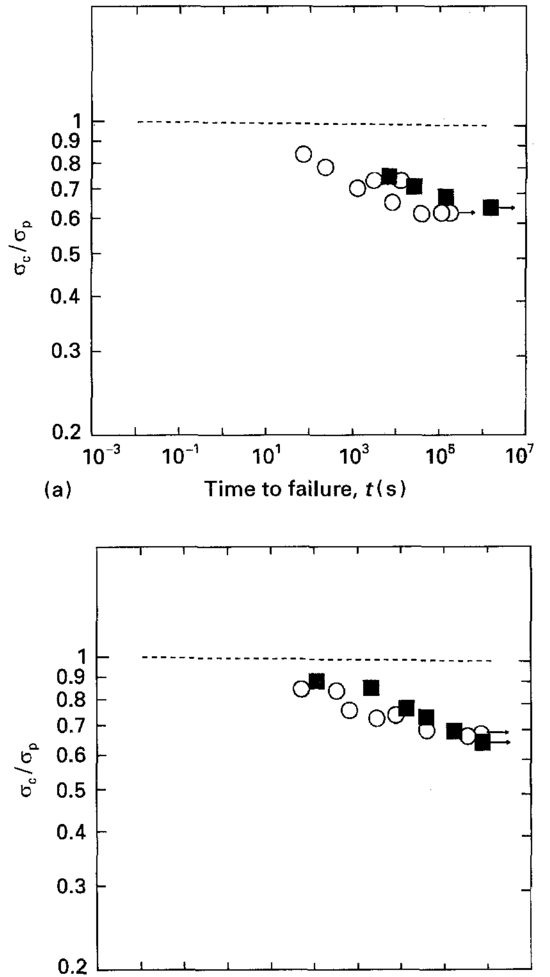

Figure 6 Comparison of pure cyclic loading effect in air calculated and cyclic loading effect in vacuum; (a) silicon nitride, (b) alumina. ○, air; ■, vacuum.

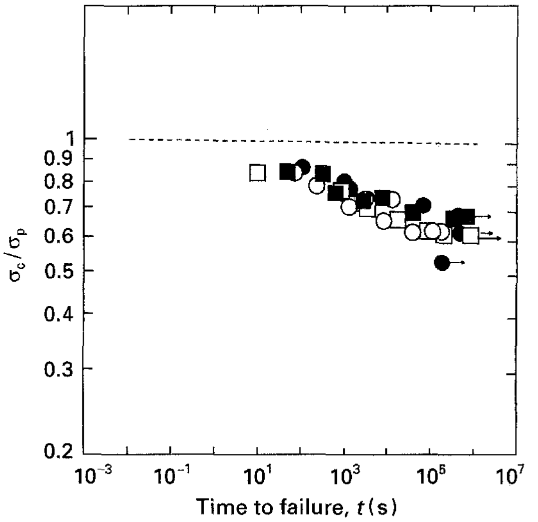

Figure 7 Pure cyclic loading effect in air calculated in relation to bonded silicon nitride (RBSN) and fine grained silicon nitride (SSN–2). The data of alumina and SSN–1 in Fig. 6 are included for comparison. ■, Al_2O_3; ○, Si_3N_4 (coarse grain); □, Si_3N_4 (fine grain); ●, RBSN.

coefficient due to environmental change, i.e. friction coefficients in most ceramic materials are smaller in air than in vacuum [21]. Since true cyclic loading effect obtained from fatigue data in air is approximately equal to cyclic loading effect in vacuum, it is supposed that the assumption of Equation 1 is valid. Hence, from Equation 6, in a ceramic material a pure cyclic loading effect is able to be obtained, if the cyclic and static loading effects in air are known.

3.3. The role of fracture toughness and glassy phase in fatigue behaviour

Fig. 7 shows $\log(\sigma_c/\sigma_p)$ versus $\log t$ relation for two kinds of silicon nitride with different microstructures, obtained from data of references [22–24]. One is reaction bonded silicon nitride (RBSN), and another is fine grained silicon nitride (SSN–2) as compared with the one (SSN–1) used in this work. The data of alumina and SSN–1 in Fig. 6 are also included for comparison. From this figure, the significant difference in the extent of pure cyclic loading effect in these materials used was not recognized. Hence, the pure cyclic loading effect in the materials characterized by intergranular fracture is approximately equal, irrespective of the kind of substance, and accordingly the slope of curves in this figure can be written as following

$$\frac{1}{n} - \frac{1}{m} = \frac{1}{l} \sim \text{const.} \qquad (7)$$

From Equation 7, it is understandable that the value of m in cyclic fatigue in air for various kinds of ceramic materials depends on the value of n alone. Such a result implies that if the static fatigue behaviour is known, the cyclic fatigue behaviour in air is roughly predictable.

The authors have shown that static fatigue in ceramics is closely related to fracture toughness and the presence of glassy phase. Fig. 8 shows the relation

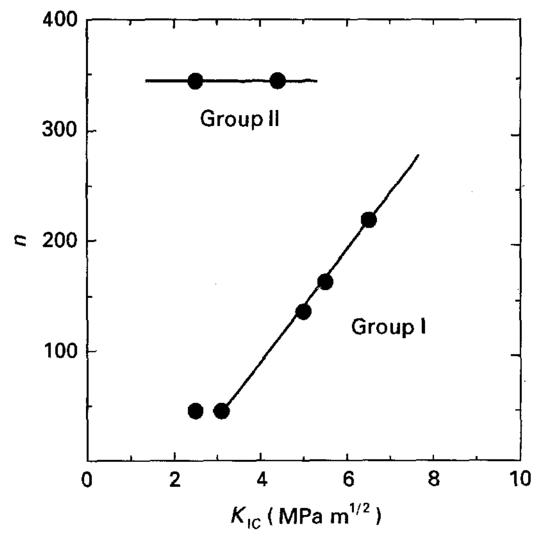

Figure 8 The relation between crack growth resistance parameter, n and fracture toughness, K_{IC} in various kinds of ceramic materials.

between n and fracture toughness, K_{IC} for various kinds of ceramic materials [22]. From this figure, $n = FK_{IC}$ for group I, oxide like alumina or non-oxide ceramics containing glassy phase like silicon nitride, where F is a constant. Contrary to this, for group II, non-glassy phase ceramics like reaction bonded silicon nitride, $1/n$ is approximately zero. Thus equation 7 is given as following.

For group I

$$\frac{1}{m} = \frac{1}{FK_{IC}} - \frac{1}{l} \tag{8a}$$

For group II

$$m = l = \text{const.} \tag{8b}$$

Since for group I F and l are constants, regardless of the kinds of materials, m depends upon K_{IC} only. Therefore, it is thought that the difference of cyclic loading effect in these materials, as shown in Fig. 4(a), is attributed to that of K_{IC}. Such a result indicates that the environmental effect is considerably sensitive to the change of microstructure or fracture toughness, while the pure cyclic loading effect is not sensitive to such changes. In addition, from Equation 8 static and cyclic fatigue behaviour is able to be predicted approximately, if data on fracture toughness, fracture strength and the presence of glassy phase of ceramic are known.

4. Conclusion

As a result of this study, the following conclusions are obtained.

1. The cyclic fatigue life in vacuum is considerably longer than that in air, indicating a remarkable environmental effect in the latter, but cyclic loading effect is obviously observed, even in vacuum, to a similar degree.
2. Cyclic fatigue in air can be expressed as the superposition of pure cyclic loading effect and environmental effect.
3. Applying this relation to various other kinds of ceramics, the difference of cyclic behaviour among the materials is mainly attributed to environmental effects. In the present materials of intergranular fracture type being used widely there have been insignificant differences of pure cyclic loading effect observed. This result indicates that the change of microstructure or fracture toughness considerably influences environmental damage, while such factors are insensitive to pure cyclic effect.

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