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几种生物陶瓷材料的裂纹扩展特性

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摘 要 采用破坏力学中的双扭矩实验法,研究了玻璃陶瓷、云母陶瓷、氧化铝陶瓷、氧化铝陶瓷材料分别在大气、水环境中的静负荷和循环负荷下的裂纹扩展特性,阐明了水环境和循环负荷对材料裂纹扩展特性 $(K_{\rm I}-V$ 特性)的影响。所研究的材料在水环境下的裂纹扩展速度均加快,但玻璃陶瓷和氧化铝陶瓷材料更为明显。在静负荷下这几种材料的裂纹扩展阻力由小到大的次序为。玻璃陶瓷 (N-0),玻璃陶瓷 (N-11),云母陶瓷,氧化锆陶瓷和氧化铝陶瓷,对于氧化铝和氧化锆陶瓷材料在循环负荷下的裂纹扩展速度均明显加快。

关键词 陶瓷材料 裂纹扩展 双扭矩 水环境 循环负荷

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BEHAVIOR OF CRACK PROPAGATION IN SOME BIO-CERAMICS

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ABSTRACT Most of bio–ceramics are oxide ceramics, and they undergo fatigue by stress corrosion cracking. In this paper, the effects of water environment and cyclic loading on the $K_{\rm I}$ –V characteristics were studied by the crack growth tests using double torsion ($D_{\rm T}$) method under static and cyclic loading in both environments of air and water for the bio–ceramics of glass ceramics, mica glass ceramics, alumina and zirconia. The increase in the crack velocity of all materials in water environment is observed, but the degree is more remarkable for glass ceramics and zirconia. The order of the crack propagation resistance under static loading is shown hereunder: Neoceram N–0 < Neoceram N–11 < Mica Glass < Zirconia < Alumina. The increase in the crack velocity under the condition of cyclic loading is clearly observed in alumina and zirconia.

KEY WORDS ceramics, crack growth, double torsion, water environment, cyclic loading

The applications of ceramics as biomaterial have been expected. Glass ceramics, alumina and zirconia are most useful materials as bio-ceramics. Most of bio-ceramics are oxide ceramics, when they are used for implant materials such as artificial joints, they undergo cyclic loading for a fairly long period in corrosive environment. The relation between the stress intensity factor $K_{\rm I}$ and the crack velocity V can be used to characterize the subcritical crack growth in ceramics, and the $K_{\rm I}$ -V characteristics can be used for their lifetime predictions based on fracture mechanics.

Thus, for practical use and investigation of the fracture mechanism, it is important to know what crack propagation behavior is observed under static and cyclic loading in both environments of air and water, in particular the effects of both cyclic load and water environment on the $K_{\rm I}$ -V characteristics. Although many studies of fracture behavior for bio-ceramics have been carried out^[1~7], the effects of both cyclic loading and water environment on the $K_{\rm I}$ -V characteristics almost have not been elucidated in particular zirconia^[8,9].

The crack growth tests were carried out under static and cyclic loading in both environments of air and water in order to examine the effects of both cyclic loading and water environment on the relationship between the stress intensity factor $K_{\rm I}$ and the crack velocity V for glass ceramics, mica glass ceramics, alumina and partially stabilized zirconia in the present study.

1. Experimental Procedure

The materials chosen in this investigation are two kinds of commercial glass ceramics with different grain sizes of 0.1 μ m and 1 μ m and different crystallization of 76% and 95%, respectively, mica glass ceramics, alumina and partially stabilized zirconia, and the chemical compositions of those ceramics are listed in Table 1.

Material	${ m SiO_2}$	Al ₂ O ₃	ZrO_2	${ m TiO_2}$	MgO	Na ₂ O	Fe_2O_3	Others
Glass								
N-0, N-11	67.0	23.0	3.0	2.0				5.0
Mica glass	46.0	16.0			17.0			21.0
Alumina	0.062	99.7		0.003	0.087	0.032	0.012	0.042
Zirconia	0.021	0.25	94.3			0.018	0.003	5.40

Table 1 Chemical composition (mass fraction, %)

The double torsion (DT) method utilizing the constant displacement and the constant load techniques was employed in the determination of $K_{\rm I}$ –V characteristics. The data of crack velocity were obtained from precracked double torsion specimens that contained a side groove (1 mm wide and 1.5 mm deep) to control the direction of the crack propagation as shown in Fig.1.

The specimens were placed on the test fixture so that the side groove was on the tensile surface of the specimen. The torsional loading is used to propagate the crack. The load is applied by four-point bending at the cracked end of the specimen as shown in Fig.1. In the cyclic loading, the specimens were subjected to a sinusoidal cyclic stress with a stress ratio (minimum stress/maximum stress) of 0.1 and a frequency of 3 Hz. The crack growth tests were performed

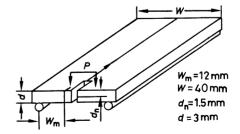


Fig.1 Configuration and dimension of specimen

in both atmospheres of air with relative humidity from 40% to 60% and ion exchanged water. The crack growth law in Eq.(1) is determined by the crack growth test^[10].

$$V = \frac{\mathrm{d}a}{\mathrm{d}t} = AK_l^n = V_c \left(\frac{K_\mathrm{I}}{K_\mathrm{IC}}\right)^n \tag{1}$$

where V-crack velocity,

 $K_{\rm I}$ -stress intensity factor,

A, n-crack propagation parameters,

 $V_{\rm c}$ -crack velocity at the critical stress intensity factor $K_{\rm IC}$.

The relationship between the stress intensity factor $K_{\rm I}$ and the applied load P is [11]

$$K_{\rm I} = PW_m \left[\frac{3(1+\nu)}{W d^3 d_m} \right]^{1/2} \tag{2}$$

where ν -Poisson's ratio and the other symbols are given in Fig.1. When the magnitude of the load is constant, the velocity is calculated as follows from the time $t_{\rm s}$ necessary for the crack to move a measured length a.

$$V_{\rm s} = \frac{a}{t_{\rm o}} \times \phi \tag{3}$$

where ϕ -Crack-profile correction factor proposed by Evans^[10].

The measured average crack velocity $V_c(=\phi a/t_c)$ for the cyclic loading should be converted into equivalent crack velocity V_{eq} .

$$V_{\rm eq} = g^{-1}(n,R) \times \frac{a}{t_c} \times \phi \tag{4}$$

$$g(n,R) = \sum_{r=0}^{[n/2]T} \frac{n!}{(n-2r)!(r!)^2} \left\{ \frac{1-R}{2(1+R)} \right\}^{2r} \left(\frac{1+R}{2} \right)^n$$
 (5)

where g(n, R)-the Evans's converting factor^[12],

R-stress ratio $(\sigma_{\min}/\sigma_{\max})$,

[n/2]T-truncated value of n/2.

When the stress ratio R=0.1, g(n,R) can be expressed as following equation

$$a(n,R) = \exp[-0.588 - 0.48\ln(n)] \tag{6}$$

2. Results and Discussions

Fig.2 shows the $\log V - \log K_{\rm I}$ relations of two kinds of glass ceramics (Neoceram N-0 and Neoceram N-11) and mica glass ceramics (Macor) resulting from a constant displacement method (a relaxation method). In any glass ceramics, the velocity in water is somewhat higher than that in air. Further, the curves in air exhibit two distinct regions, region I, where the rate of the chemical reaction near the crack tip controls crack growth, and region II, where diffusion of the corrosive species to the crack tip is rate controlling. On the other hand, in water only the straight lines

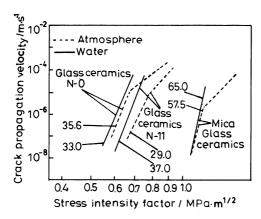
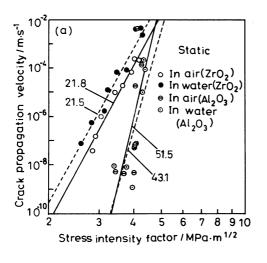


Fig.2 Effect of environment on K_{I} -V curves of various glass ceramics

corresponding to region I were obtained. So, it is considered that the crack velocity in region II, controlled by diffusion of corrosive species, depended on the amount of water vapor (H_2O) in air and water.

Figs.3 show the $\log K_{\rm I}$ - $\log V$ relationship of alumia and zirconia obtained by static and cyclic loading in both air and water environments, respectively, of which curves result from a constant load method. The region II is not observed distinctly in any case of these diagrams. The crack propagation parameter n, determined from the slopes of these curves, are described in these figures. The crack propagation parameter n, of



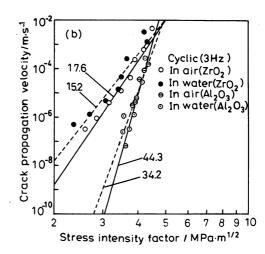


Fig.3 Effect of environment on $K_{\rm I}$ -V curves of alumina and zirconia (a) static loading, (b) cyclic loading

zirconia is clearly lower than that of alumina, and the crack velocity of zirconia is higher than that of alumina in both environments of air and water. The increase in the crack velocity in water environment is observed in both materials, but the degree is more remarkable for zirconia. Figs.4 show the effect of cyclic loading on the $\log K_{\rm I}$ - $\log V$ relationship in the case of respective environments of air and water. In any environments cyclic loading enhanced the crack propagation rate, that is to say, the decrease in the crack propagation parameter n, and the increase in the crack velocity due to cyclic loading are clearly observed in both materials in air and water environments. Fig.5a presents much more intergranular cracking in alumina and a mixture of intergranular and transgranular cracking are presented in Fig.5b in zirconia. From these photographs, it is considered

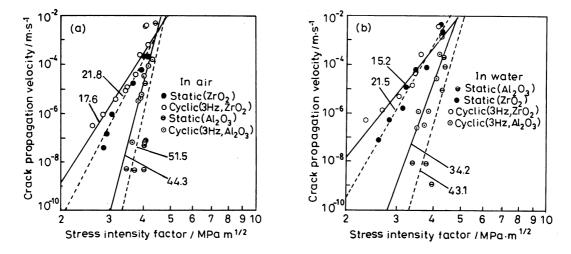


Fig.4 Effect of cyclic loading on $K_{\rm I}$ -V curves of alumina and zirconia (a) in air, (b) in water

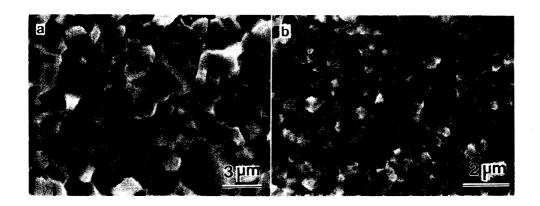


Fig.5 Fracture surfaces of alumina and zirconia observed by SEM (a) alumina, (b) zirconia

that the fracture in alumina occurred mainly at grain boundaries where SiO₂ bonds exist, while the fracture in zirconia occurred at both grain boundaries and the interior of the grain due to stress corrosion cracking with principal components of ZrO₂ and SiO₂. So, the crack in alumina will propagate in zigzags by intergranular cracking and the crack propagates more straight in zirconia than in alumina. It is known that intermittent crack growth due to grain bridging more often occurs at the tip of main crack in intergranular cracking. This means that propagation resistance in alumina becomes larger than in zirconia.

3. Conclusion

The increase of the crack velocity in water environment is observed in all materials, but the degree is more remarkable for glass ceramics and zirconia. The decrease of the crack propagation parameter n and the increase of the crack velocity under the condition of cyclic loading are clearly observed in alumina and zirconia in air and water environments.

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