Cyclic fatigue-crack propagation in sapphire in air and simulated physiological environments

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Abstract: Single-crystal aluminas are being considered for use in the manufacture of prosthetic heart valves. To characterize such materials for biomedical application, subcritical crack growth by stress corrosion (static fatigue) and by cyclic fatigue has been examined in sapphire along $(1\bar{1}00)$ planes in 24°C humid air and 37°C Ringer's solution (the latter as a simulated physiological environment). The relationships between crack-propagation rates and the linear-elastic stress intensity have been determined for the first time in sapphire for both modes of subcritical cracking. It was found that growth rates were significantly faster at a given stress intensity in the Ringer's solution compared to the humid air environment. Mechanistically, a true cyclic fatigue effect was not found in sapphire as experimentally measured cyclic fatigue-crack growth rates could be closely predicted simply by integrating the static fatigue-crack growth data over the cyclic loading cycle. © 2000 John Wiley & Sons, Inc. J Biomed Mater Res, 52, 488–491, 2000.

Key words: heart valve prostheses; sapphire; static fatigue; cyclic fatigue; crack growth

INTRODUCTION

Alumina ceramics such as sapphire have been widely considered for biomedical use owing to their chemical stability and superior tribological characteristics. In particular, the biocompatibility of sapphire has motivated its consideration for use in prosthetic heart valves: for example, as a tilting disk in a ceramic mechanical valve.[1] Because durability and reliability are vital requirements for such biomedical use, it is critical that the mechanical properties and anisotropy of sapphire be well documented and understood. Previous studies on single-crystal aluminas have considered principally fracture morphology and toughness.[1–4] However, because the human heart beats some 40 million times per year and cyclic fatigue resistance is considered to be a limiting property for many replacement cardiac valve devices,[5] the present note is focused on the subcritical crack-growth properties of sapphire.

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In general, crack growth in many polycrystalline ceramics such as alumina and silicon nitride is accompanied by crack bridging by interlocking grains behind the crack tip; this effectively shields the crack tip from the applied far-field loading and results in toughening in the form of marked resistance-curve (R-curve) behavior.[6,7] Under cyclic loading, the fracture process ahead of the crack tip is largely unaffected; however, crack growth is associated with the degradation of such bridging and a concomitant reduction in crack-tip shielding.[8–10] Corresponding subcritical crack growth under monotonic loading, often termed stress corrosion cracking or static fatigue, occurs primarily by environmentally assisted mechanisms at or very near the crack tip, e.g., the Si-O dissociative chemisorption reaction which occurs during the static fatigue of silica glasses in moist environments.[11,12] In untoughened ceramics where there are no shielding mechanisms in the wake of the crack tip to degrade, static fatigue can also provide the mechanism of failure under cyclic loads; here, cyclic fatigue-crack growth rates can be simply considered to result from static fatigue or overload fracture during the loading cycle and the ceramic shows no susceptibility to premature failure under cyclic loads.

This article describes an investigation of subcritical crack growth in sapphire under both monotonic and cyclic loading where the mechanisms of cyclic fatigue failure were unknown. Behavior was examined in environments of humid air and 37°C Ringer's solution; the latter was employed as a simulated physiological environment for the potential use of sapphire in biomedical implant devices.

MATERIALS AND EXPERIMENTAL PROCEDURES

Single crystals of alumina (Kyocera sapphire) were made by the edge-defined, film-fed growth method. The specimens were cut from a large single crystal by a diamond disc cutter and were machined into compact-tension C(T) specimens (of width $W = 25.4$ mm and thickness $B = 6.4$ mm) [Fig. 1(a)], with an orientation such that the crack was lying on the $(1\bar{1}00)$ plane [Fig. 1(b)]. This plane has been considered to be the probable fracture plane of the sapphire disks used in a ceramic heart valve.[1] After machining, samples were polished to better than 1 μm such that any microcracks on the surface of these dimensions were removed; however, because the C(T) samples were subsequently fatigue precracked, the presence of any smaller microcracks on the specimen surfaces was not a concern.

Specimens were tested on computer-controlled servohydraulic machines (Model 810; MTS Corporation, Minneapolis, MN) in environments of humid room air (~85% relative humidity at ~24°C) and 37°C Ringer's solution (unbuffered, 1 L contained 600 mg NaCl, 310 mg sodium lactate, 30 mg KCl, 20 mg CaCl). The pH of the solution was not measured. Because of the highly uniform nature of the single crystal material, a total of five specimens were used for testing.

Testing was conducted under both monotonic and cyclic loads to characterize static and cyclic fatigue-crack propagation behavior, respectively. Cyclic tests were performed at a sinusoidal frequency of 10 Hz and a load ratio (defined as the ratio of the minimum to maximum applied load) of $R = 0.1$, in general accordance with American Society for Testing and Materials standard fatigue-crack growth test practices,[13] modified for ceramics.[14] Corresponding static fatigue-crack growth tests were conducted by application of constant loads at levels sufficient to propagate the crack. In both cases, testing was conducted under closed-loop load control. Crack lengths were monitored *in situ* using back-face strain unloading compliance (resolution ± 30 μm), with values periodically checked using an optical microscope. Growth rates, *da/dN* and *da/dt*, were correlated to the alternating and mean stress intensities, ΔK and K, respectively; K values were computed from handbook solutions.[13]

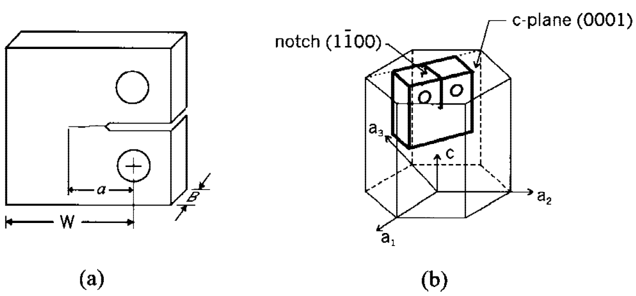

Figure 1. Schematic of (a) compact-tension geometry and (b) orientation of crack-plane with respect to crystallographic directions.

RESULTS AND DISCUSSION

Static fatigue-crack growth behavior

The variation in static-fatigue crack velocities, *da/dt*, with the applied stress intensity, K, for the sapphire is shown in Figure 2 for environments of 24°C humid air and 37°C Ringer's solution. Stable crack growth occurred over only a relatively narrow range of stress intensities, between $K \sim 1.5$ and 1.9 MPa√m. Crack velocities were comparable in the two environments, although somewhat faster in the Ringer's solution. Static fatigue threshold K_{TH} values, representing the applied stress intensity below which crack velocities become vanishingly small (<1 nm/s), were found to be 1.64 and 1.55 MPa√m for the humid air and Ringer's solution tests, respectively. The *da/dt-K* relationship could be described by the simple Paris-type power law:

$$\frac{da}{dt} = CK^p \quad (1)$$

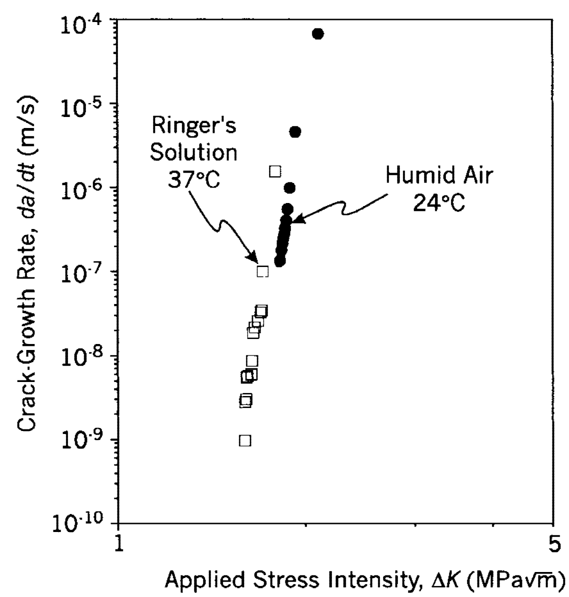

Figure 2. Static fatigue-crack growth rates versus applied stress intensity in both air and Ringer's solution.

where C and p are scaling constants. The crack-growth data were fit using a linear least-squares model to the transformed equation $\ln(da/dt) = p \cdot \ln(K) + \ln(C)$. Results of these fits are presented in Table I.

Cyclic fatigue-crack growth behavior

Corresponding crack-growth rates measured at $R = 0.1$ under cyclic loading, da/dN, are plotted for the two environments in Figure 3 as a function of the applied stress-intensity range, ΔK. We believe these data, which span 5 decades of growth rates, to be the first reported cyclic fatigue-crack propagation rate data for sapphire in the literature. Stable crack growth is again possible only over only a narrow range of stress intensity, specifically $1.45 < \Delta K < 1.83$ MPa√m; threshold ΔK_{TH} values were 1.60 and 1.45 MPa√m for cracks grown in humid air and Ringer's solution, respectively. The da/dN-ΔK relationship can be described by the relationship:

$$\frac{da}{dN} = C' \; \Delta K^{m} \tag{2}$$

where C' and m are scaling constants. Growth data were fit using a linear least-squares model to the transformed equation $\ln(da/dn) = m \cdot \ln(\Delta K) + \ln(C')$ with results presented in Table I.

At a given applied ΔK, growth rates were significantly faster in Ringer's solution (by roughly an order of magnitude) than in humid air. However, in addition to the increased damage associated with such environmentally assisted crack growth, crack-growth rates can be slowed down to some degree owing to the predominantly wedging effect of a fluid inside the crack. However, in the present work no evidence of such crack-tip shielding from the hydrodynamic pressure of the fluid inside the crack[15,16] was detected.

Mechanistic aspects

To assess whether cyclic fatigue-crack propagation is a true cyclic phenomenon or merely the result of static fatigue during the loading cycle, the growth rates under cyclic loading were predicted by integrating the static fatigue-crack growth data over the cyclic loading cycle, using the relationship of Evans and Fuller[17]:

$$\frac{da}{dN} = C \int_{0}^{1/f} \left[\frac{1}{2}(K_{max} + K_{min}) + \frac{\Delta K}{2}(\sin(2\pi f t)) \right]^{p} dt \tag{3}$$

where f is the test frequency and K_{max} and K_{min} are the maximum and minimum applied stress intensities in the fatigue cycle. This relationship is based on the notion that there is no cyclic fatigue effect; growth rates under cyclic loads are merely static fatigue-crack growth associated with the instantaneous magnitude of the stress intensity. Using Equation (3), we found the cyclic crack-growth rates predicted from the static fatigue data to be in excellent agreement with those measured experimentally (Fig. 3). This indicates that, to within experimental error, there is no true cyclic fatigue effect in sapphire, and hence no premature failure under cyclic loads over and above that associated with static fatigue. Such behavior is typical of untoughened ceramics and glasses, where crack-tip shielding mechanisms such grain bridging or stress-induced phase transformations, are absent or inactive.

Mechanistically, it is apparent that the 37°C Ringer's solution is the more aggressive environment during subcritical crack growth in sapphire. Such behavior is

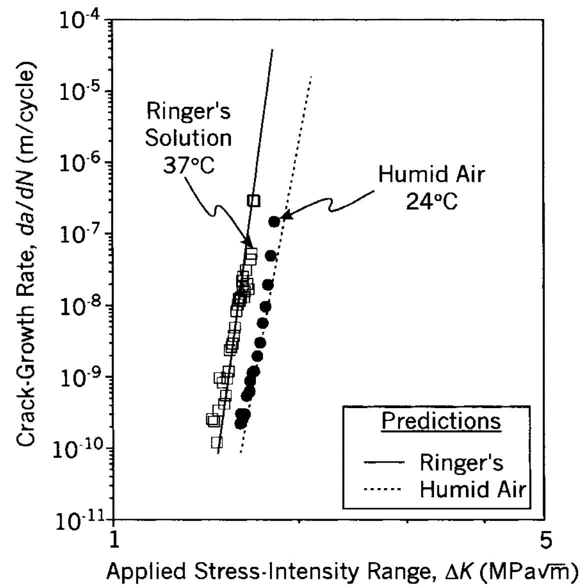

Figure 3. Crack growth in sapphire under cyclic loading conditions. Lines represent predictions from the integration of static (fatigue) crack-growth data over the applied loading cycle.

TABLE I
Fit Results for Equations (1) and (2)

Test Condition	C, C′	p,m	95% Confidence Interval on p,m	Correlation Coefficient
Static, humid air	1.8×10^{-20}	45.6	[41.8,49.5]	.99
Static, Ringer's	2.0×10^{-20}	54.2	[45.8,62.5]	.97
Cyclic, humid air	5.0×10^{-20}	46.1	[40.7,51.5]	.98
Cyclic, Ringer's	1.5×10^{-17}	43.0	[38.8,47.2]	.97

consistent with the studies of Kimura and coworkers,[18,19] who found that although single-crystal alumina was essentially immune to corrosion in deionized water, it did corrode somewhat in isotonic NaCl solution. Thus, although alumina is extremely stable and inert in the human body, the presence of a crack does promote environmentally assisted mechanisms at the crack tip which accelerate the rate of cracking. However, the corrosion resistance of sapphire does appear to be significantly better than that of polycrystalline alumina.[18]

CONCLUSIONS

Based on an experimental study of subcritical crack growth in sapphire along the $(1\bar{1}00)$ plane under static and cyclic loading conditions in 24°C humid (85% relative humidity) air and 37°C Ringer's solution, the following conclusions may be made.

1. Subcritical crack growth was observed under monotonic loading above threshold K_{TH} values of 1.55 and 1.64 MPa$\sqrt{m}$ in the Ringer's and air environments, respectively. Similarly, fatigue-crack growth was observed under cyclic loading conditions above ΔK_{TH} thresholds of 1.45 and 1.60 MPa$\sqrt{m}$ in the same environments. For both modes of cracking, growth rates were a marked function of the stress-intensity level.
2. Despite the presumed inertness of alumina in physiological environments, crack-growth rates in 37°C Ringer's solution were significantly higher at the same applied driving force than those observed in the air environment.
3. Cyclic fatigue-crack growth data could be well predicted from the static crack-growth results by integrating the latter data over the cyclic loading cycle. This implies that there is no (true) cyclic fatigue effect in sapphire.

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