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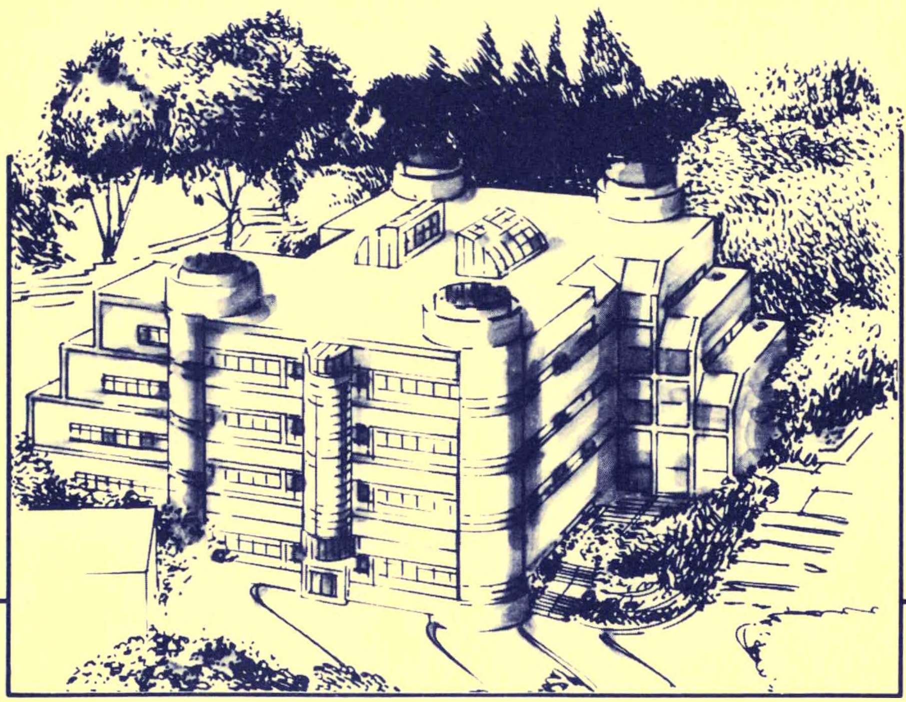

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CYCLIC FATIGUE-CRACK PROPAGATION IN CERAMICS: BEHAVIOR IN OVERAGED AND PARTIALLY-STABILIZED MgO-ZIRCONIA

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ABSTRACT

The growth of fatigue cracks under (tension-tension) cyclic loading is unequivocally demonstrated for ceramic materials, based on experiments using compact-tension specimens of a MgO partially-stabilized zirconia (PSZ), heat treated to vary the fracture toughness K_c from ~3 MPa$\sqrt{m}$ (overaged condition) to 16 MPa$\sqrt{m}$ (peak-toughness condition) and tested in inert and moist environments. Analogous to behavior in metals, cyclic fatigue-crack growth rates (over the range 10^{-11} to 10^{-5} m/cycle) are found to be a function of the stress-intensity range, environment, fracture toughness and load ratio, and to show evidence of crack closure. Similarly under variable-amplitude cyclic loading conditions, crack-growth rates show transient accelerations following low-high block overloads and transient retardations following high-low block overloads or single tensile overloads, again analogous to behavior commonly observed in ductile metals. Cyclic crack-growth rates are observed at stress intensities as low as 50% of K_c, and are typically some 7 orders of magnitude faster than corresponding stress-corrosion crack-growth rates under sustained-loading conditions.

INTRODUCTION

The projected use of ceramics rather than metallic materials for structural applications, such as in gas-turbine engines, has been motivated by their low density, far superior elevated-temperature strength and presumed insensitivity to degradation from cyclic fatigue [1]. However, several investigations [2-7] utilizing smooth specimens, sometimes containing identation flaws, tested under rotating bending, four-point bending or by repeated thermal stressing, have shown reduced lifetimes for alumina, zirconia-alumina, TZP and silicon nitride under cyclic, as opposed to static, loading conditions. Moreover, subcritical crack growth has been reported for several monolithic and composite ceramics tested under far-field cyclic compression loads [8,9]. Although several explanations have been suggested for such apparent fatigue behavior, including deformation and lateral cracking of crack-surface asperities on unloading [1,6], tensile opening from the wedging action of such asperities [1] or corrosion debris [10], friction-induced heating at the crack tip [11], and environmentally-assisted (stress-corrosion) cracking processes [6,10,11], there are few examples of unequivocal demonstrations of a true cyclic fatigue phenomenon in ceramics [1].

The refuted existence of a true fatigue effect in ceramics has been based primarily on their very limited crack-tip plasticity. Recently, however, based on the premise that other inelastic deformation mechanisms may prevail in these materials, such as microcracking, transformation "plasticity" or frictional sliding between a reinforcement phase and the ceramic matrix, Dauskardt et al. [11] provided the first persuasive evidence of true cyclic fatigue-crack growth in a MgO partially-stabilized zirconia (MgO-PSZ) ceramic, which has been marginally transformation toughened to a K_c of 5.5 MPa$\sqrt{m}$. Their results, which have been subsequently confirmed by Swain et al. [7] in a commercial MS-Grade MgO-PSZ, indicated that crack-growth rates were a power-law function of the stress-intensity range ΔK, with an exponent m of ~24, were sensitive to frequency and load ratio ($R = K_{min}/K_{max}$), and showed evidence of crack closure [12], analogous to behavior in metals.

In the present study, a more extensive examination of cyclic fatigue-crack growth in MgO-PSZ is undertaken to investigate: i) how fatigue resistance varies with microstructures of differing fracture toughness, ii) the role of environment on crack-growth rates, and iii) the nature of transient fatigue behavior following specific variable-amplitude cyclic loading sequences (i.e., post-overload behavior).

EXPERIMENTAL PROCEDURES

Tests were performed on precipitated partially-stabilized zirconia, containing 9 mol% magnesia (MgO-PSZ), chosen for its transformation-toughening behavior [13-16]. The microstructure consists of cubic ZrO_2 grains, ~50 μm in diameter, with lens-shaped tetragonal precipitates of maximum size 300 nm; up to 40% of the latter phase can undergo a stress-induced martensitic transformation due to the high stresses near the crack tip [16]. The MgO-PSZ was examined in four microstructural conditions, selected to vary the fracture toughness from a K_c of 2.9 MPa$\sqrt{m}$ in the overaged (non-transformation-toughened) condition to 16 MPa$\sqrt{m}$ in the peak-toughened condition. The heat treatments, together with respective ambient-temperature mechanical properties, are listed in Table I. Further details of the microstructural and mechanical-property characteristics of this material are described elsewhere [13-16].

Table I. Heat Treatments and Tensile Properties of MgO-PSZ

Condition	Heat Treatment	Young's Modulus, E (GPa)	Approx. Tensile Strength (MPa)	Fracture Toughness K_i (MPa$\sqrt{m}$)*	Fracture Toughness K_c (MPa$\sqrt{m}$)*
overaged	24 h at 1100°C	<200	300	2.5	2.9
low toughness	as received	208	300	~3	5.5
mid toughness	3 h at 1100°C	208	600	~3	11.5
peak toughness	7 h at 1100°C	208	400	~4	16.0

*K_i and K_c are the initiation and plateau toughness values from the R-curve.

Fatigue-crack propagation studies were performed on 3-mm-thick compact tension C(T) specimens, containing long (> 3 mm) through-thickness cracks, which were cyclically loaded at a load ratio of 0.1 and frequency of 50 Hz (sine wave) in high-resolution, computer-controlled electro-servohydraulic testing machines. Crack initiation was facilitated by a wedge shaped starter notch. Testing was performed in controlled room air (22°C, 45% relative humidity), dehumidified gaseous nitrogen and distilled water environments. Electrical-potential measurements across ~0.1-μm NiCr foils, evaporated onto the specimen surface, were used *in situ* to monitor crack lengths to a resolution better than 5 μm; unloading compliance measurements using back-face strain gauges were similarly used to assess the extent of fatigue crack closure in terms of the stress intensity K_{cl} at first contact of the fracture surfaces during the unloading cycle [17]. Crack-growth rates, da/dN, were determined over the range 10^{-11} to 10^{-5} m/cycle under both manual and computer-controlled K-decreasing and K-increasing conditions, with a normalized K-gradient of 0.80 mm^{-1} [18]; data are presented in terms of the applied stress-intensity range ($\Delta K = K_{max} - K_{min}$, where K_{max} and K_{min} are, respectively, the maximum and minimum stress intensities in the fatigue cycle). Fracture toughness values were measured from $K_R(\Delta a)$ resistance curves, determined by monotonically loading the fatigue-cracked C(T) specimens. Further experimental details are given elsewhere [11].

RESULTS AND DISCUSSION

Role of Fracture Toughness: Resistance curves for the four microstructures are illustrated in Fig. 1a. Whereas the overaged microstructure is fully transformed by heat treatment and shows only a shallow R-curve with a K_c of 2.9 MPa$\sqrt{m}$, the other PSZ microstructures undergo transformation toughening resulting in (plateau) toughnesses of 5.5 to 16 MPa$\sqrt{m}$. Note that as the R-curves were obtained in each specimen after the fatigue test, the full R-curves are not observed but begin at the final K_{max} of the ΔK range employed in the previous fatigue cycling. Corresponding cyclic fatigue-crack propagation data are plotted in Fig. 1b as a function of the stress-intensity range ΔK for a controlled room-air environment. It is apparent that growth rates show a conventional Paris law relationship [19]:

$$da/dN = C\,(\Delta K)^m \quad , \tag{1}$$

where the exponent m is in the range 21 to 42, and the constant C scales with the fracture toughness. Surprisingly, the overaged material in which the nonlinear deformation behavior associated with transformation plasticity has been removed, displays extensive cyclic fatigue-crack propagation behavior, with a similar power-law dependency on the stress-intensity range exhibited by the toughened materials. Moreover, the present data indicate that the overaged microstructure exhibits fatigue-crack growth at stress intensities below that required for crack initiation under monotonic loading on the R-curve. In fact, each set of data shows an apparent threshold, ΔK_{TH}, below which crack growth is presumed dormant; the value of ΔK_{TH} is seen to be approximately $\frac{1}{2}K_c$. Values of C, m and ΔK_{TH} for each microstructure are listed in Table II. It is apparent that resistance to cyclic fatigue-crack growth in MgO-PSZ is enhanced with increasing fracture toughness.

Table II. Values of C and m (in Eq. 1) and the Threshold ΔK_{TH} in MgO-PSZ

Condition	K_c (MPa$\sqrt{m}$)	C (MPa$\sqrt{m}$·m/cycle)	m	ΔK_{TH} (MPa$\sqrt{m}$)*
overaged	2.9	2.00×10^{-14}	21	1.6
low toughness	5.5	4.89×10^{-22}	24	3.0
mid toughness	11.5	5.70×10^{-28}	24	5.2
peak toughness	16.0	1.70×10^{-48}	42	7.7

*ΔK_{TH} defined at a maximum growth rate less than 10^{-10} m/cycle [18].

As described elsewhere [11], such fatigue-crack growth behavior in PSZ is *cyclically* induced and not simply subcritical cracking at maximum load. Tests on the low-toughness microstructure at constant K_{max}, where growth-rate behavior was monitored i) when the load was cycled between K_{max} and K_{min} ($R = 0.1$) compared to being held constant at K_{max} (Fig. 2), and ii) when the value of K_{min} was varied, clearly indicated a true cyclic fatigue phenomenon with growth rates proportional to the range of stress intensity. In fact as shown in Fig. 5a, under constant ΔK conditions, fatigue-crack growth rates in MgO-PSZ remain essentially constant.

Role of Environment: It has been suggested that fatigue in ceramics may alternatively be the result of stress-corrosion cracking [6,10,11]. To examine this hypothesis, cyclic crack-growth rates in the low-toughness microstructure were additionally measured in inert (dehumidified nitrogen gas) and corrosive (distilled water) environments; results are plotted as a function of ΔK in Fig. 3. As expected, growth rates are progressively faster in moist room air and water compared to inert nitrogen gas, indicating a marked corrosion-fatigue effect presumably involving the weakening of atomic bonds at the crack tip by adsorbing water molecules; however, crack growth is still observed in the inert atmosphere implying that, analogous to behavior in metals, *cyclic fatigue in the* *ceramic is a mechanically-induced cyclic process which may be accelerated by the environment.*

Mechanisms: Crack-Tip Shielding: As described in ref. 11, fatigue-crack growth in PSZ fractographically resembles monotonic overload failures with a predominantly transgranular fracture morphology and no evidence of fatigue striations. Crack-path morphologies, however, do show evidence of crack-tip shielding from frequent crack deflection and crack bridging from uncracked ligaments just behind the crack tip. In addition, analogous to behavior in metals, back-face strain compliance measurements gave clear indications of fatigue crack closure, with K_{cl}/K_{max} ratios varying from almost 0.4 close to ΔK_{TH} to ~0.1 close to K_c [11]. In view of the deflected nature of the crack paths, it is presumed that such closure results primarily from the wedging action of fracture-surface asperities (roughness-induced crack closure [12,20]). Evidence for shielding by transformation toughening, conversely, can be seen by examining the inelastic transformed zones that surround the crack using spatially-resolved Raman spectroscopy or Normaski techniques [21]; a three-dimensional Raman plot of such a zone in peak-toughness MgO-PSZ, both ahead and in the wake of the crack tip, is shown in Fig. 4. However, to date we have been unable to detect whether such zones are different in size or morphology for cyclically, as opposed to monotonically, loaded cracks at the same stress-intensity level.

Role of Variable-Amplitude Loading: The results described above pertain solely to constant-amplitude cyclic loading; to examine the influence of variable-amplitude loading, single and block overload sequences were applied during steady-state fatigue-crack growth in the mid- and peak-toughness MgO-PSZ materials. Results for high-low and low-high block overloads in mid-toughness material are shown in Fig. 5a. Over the first ~2.5 mm of crack advance, the crack-growth rate remains approximately constant under constant ΔK (= 5.48 MPa$\sqrt{m}$) conditions. On reducing the cyclic loads to a ΔK corresponding to 5.30 MPa$\sqrt{m}$ (high-low block overload), however, a marked transient retardation is seen followed by a gradual increase in growth rates until the (new) steady-state velocity is achieved. Similarly, by subsequently increasing the cyclic loads to a ΔK corresponding to 5.60 MPa$\sqrt{m}$ (low-high block overload), growth rates show an immediate transient acceleration before a decay to the steady-state velocity. Such behavior is exactly analogous to that widely observed in metals [22], where to the first order the crack-growth increment affected by the overload is comparable with the extent of the overload plastic zone. In the present experiments, the affected crack-growth increments are approximately 1 to 1.2 mm, slightly larger than five times the measured [21] transformed-zone width of 150 μm.

Similar crack growth retardation following a high-low block overload (ΔK = 9.5 to 8.5 MPa$\sqrt{m}$) is shown for peak-toughness MgO-PSZ in Fig. 5b; in addition, significant *delayed* retardation can be seen following a single tensile overload to a K_{max} of 12.3 MPa$\sqrt{m}$. Such results can be rationalized in terms of measured changes in crack closure [23], and are again analogous to behavior in metals [22]. Further details are described in ref. 23.

Comparison with Stress-Corrosion Cracking Data: It is clear from the present results that cyclic fatigue-crack growth in ceramics can occur at stress intensities as low as $\frac{1}{2}K_c$. Moreover, compared to behavior under monotonic loading, cyclic crack-growth rates in MgO-PSZ can be many orders of magnitude faster, and can occur at stress intensities far lower, than that required for stress-corrosion cracking [11]. This is illustrated in Fig. 6 for the mid-toughness microstructure tested in moist air, where cyclic crack velocities, plotted as a function of time (da/dt), are compared with corresponding stress-corrosion crack velocities measured under sustained loads; at equivalent K levels cyclic crack-growth rates can be seen to be up to 7 orders of magnitude faster.

It would thus appear that nonconservative estimates of the subcritical advance of incipient cracks, and serious overestimates of life, may result in PSZ ceramics if defect-tolerant predictions are based solely on sustained-load (stress-corrosion) and fracture-toughness data and do not consider a cyclic fatigue effect.

CONCLUSIONS

Based on a study of the growth of fatigue cracks in Mgo-PSZ ceramics under tension-tension cyclic loads, the following conclusions can be made:

1. Fatigue-crack growth in overaged (microcrack toughened) and partially-stabilized (transformation toughened) zirconia is unequivocally demonstrated to be a mechanically-induced cyclic process, which is accelerated in moist air and distilled water environments. Growth rates (da/dN) are found to be primarily a power-law function of the stress-intensity range (ΔK), with an exponent m in the range of 21 to

42.

2. Superior cyclic crack-growth resistance is found when the fracture toughness (K_c) of MgO-PSZ is increased; the apparent threshold for fatigue-crack growth (ΔK_{TH}), below which cracks are dormant, is approximately $\frac{1}{2}K_c$.

3. Cyclic crack growth in ceramics shows evidence of crack closure. Moreover, when subjected to variable-amplitude cyclic loading, fatigue cracks in MgO-PSZ experience a transient crack-growth retardation immediately following high-low block overloads, a transient acceleration immediately following low-high block overloads, and a delayed retardation following single tensile overloads; such behavior is exactly analogous to that commonly reported for metallic materials.

4. Cyclic crack velocities in MgO-PSZ are found to be up to 7 orders of magnitude faster, and threshold stress intensities almost 50% lower, than stress-corrosion crack velocities measured in identical environments under sustained-loading conditions. Such observations may have serious implications for defect-tolerant life predictions in zirconia ceramics.

Acknowledgments

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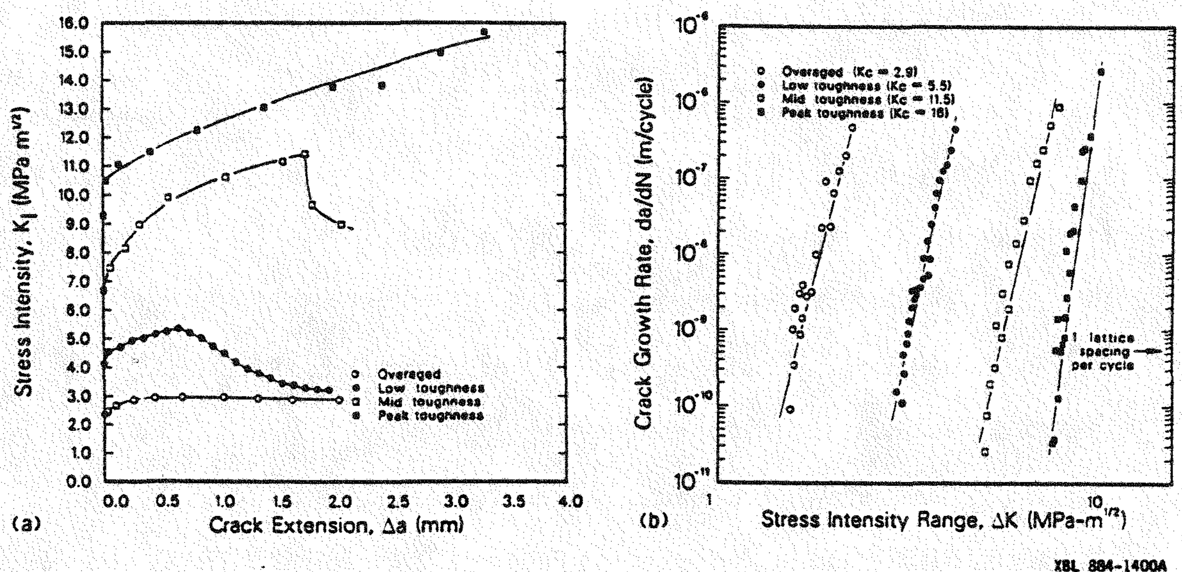

Fig. 1. Fracture-toughness and cyclic fatigue-crack growth behavior of MgO-PSZ heat treated to a range of toughnesses, showing a) $K_R(\Delta a)$ resistance curves, and b) crack-growth rates da/dN as a function of the stress-intensity range ΔK (room air environment at R = 0.1).

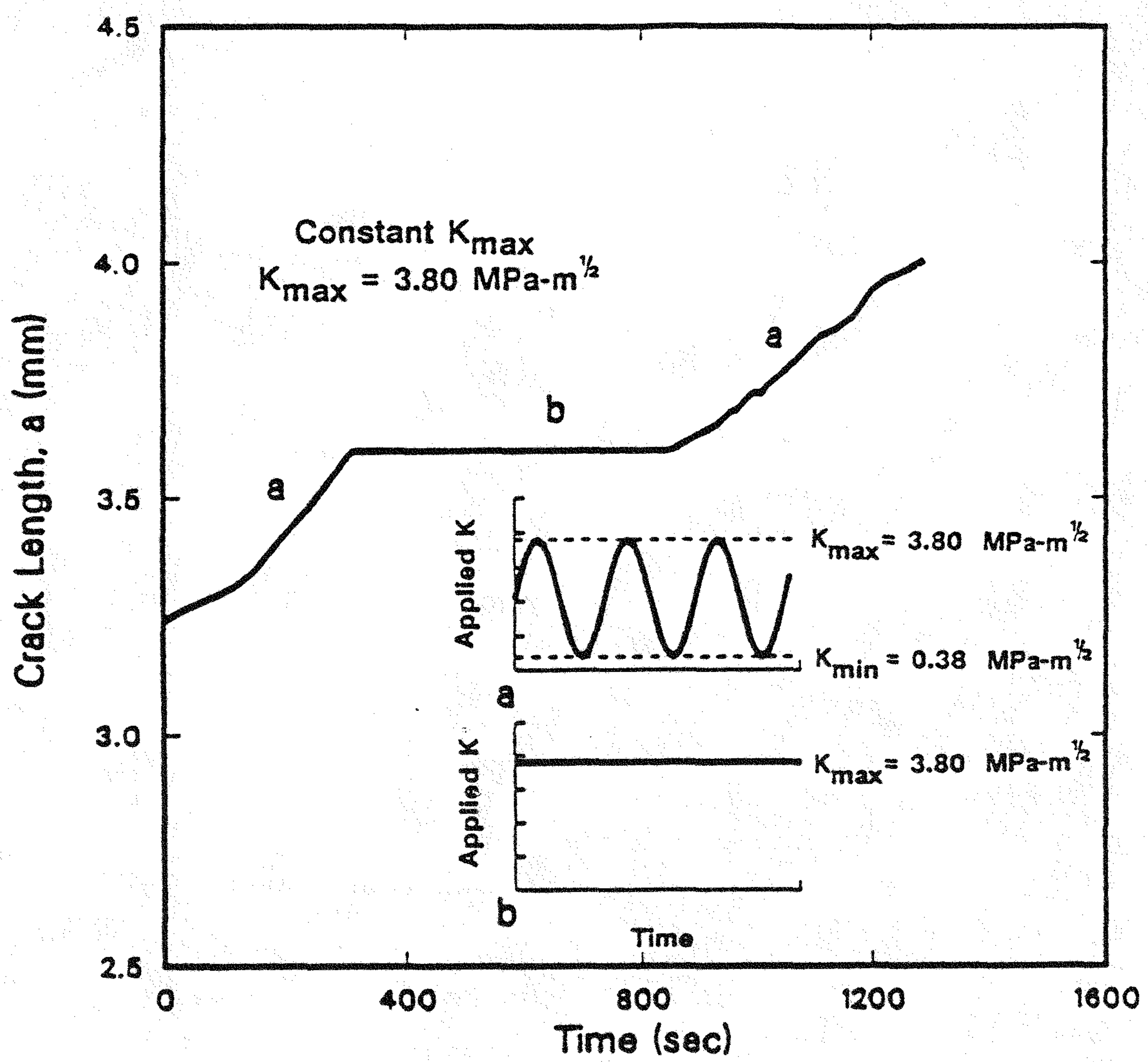

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Fig. 2. Effect in low-toughness MgO-PSZ of sustained and cyclic loading conditions on subcritical crack-growth rates at a constant K_{max} of 3.8 MPa$\sqrt{m}$ [11].

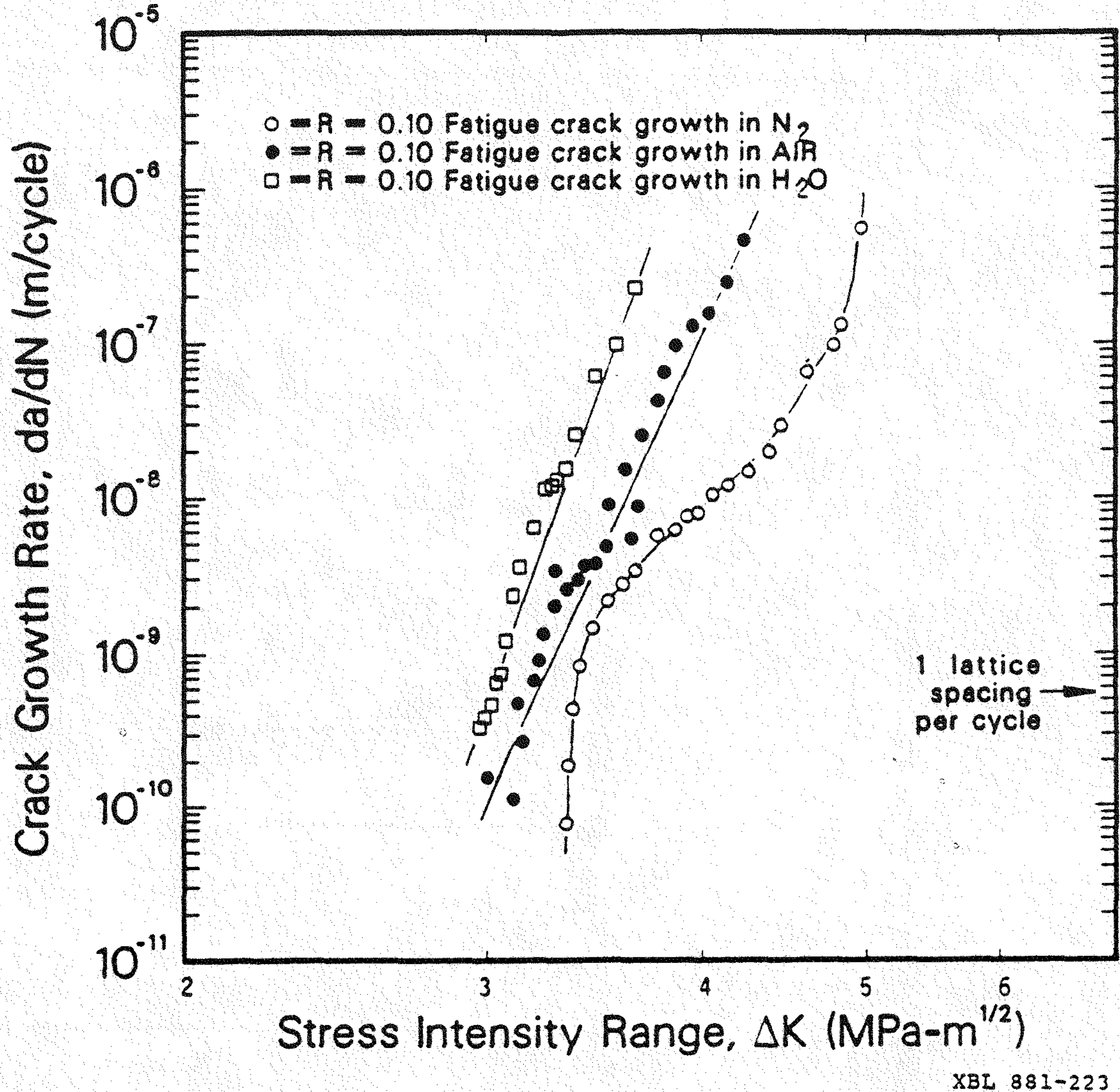

Fig. 3. Cyclic fatigue-crack growth rates da/dN as a function of ΔK in low-toughness MgO-PSZ in dry nitrogen gas, room air and distilled water, showing an acceleration in growth rates due to water vapor.

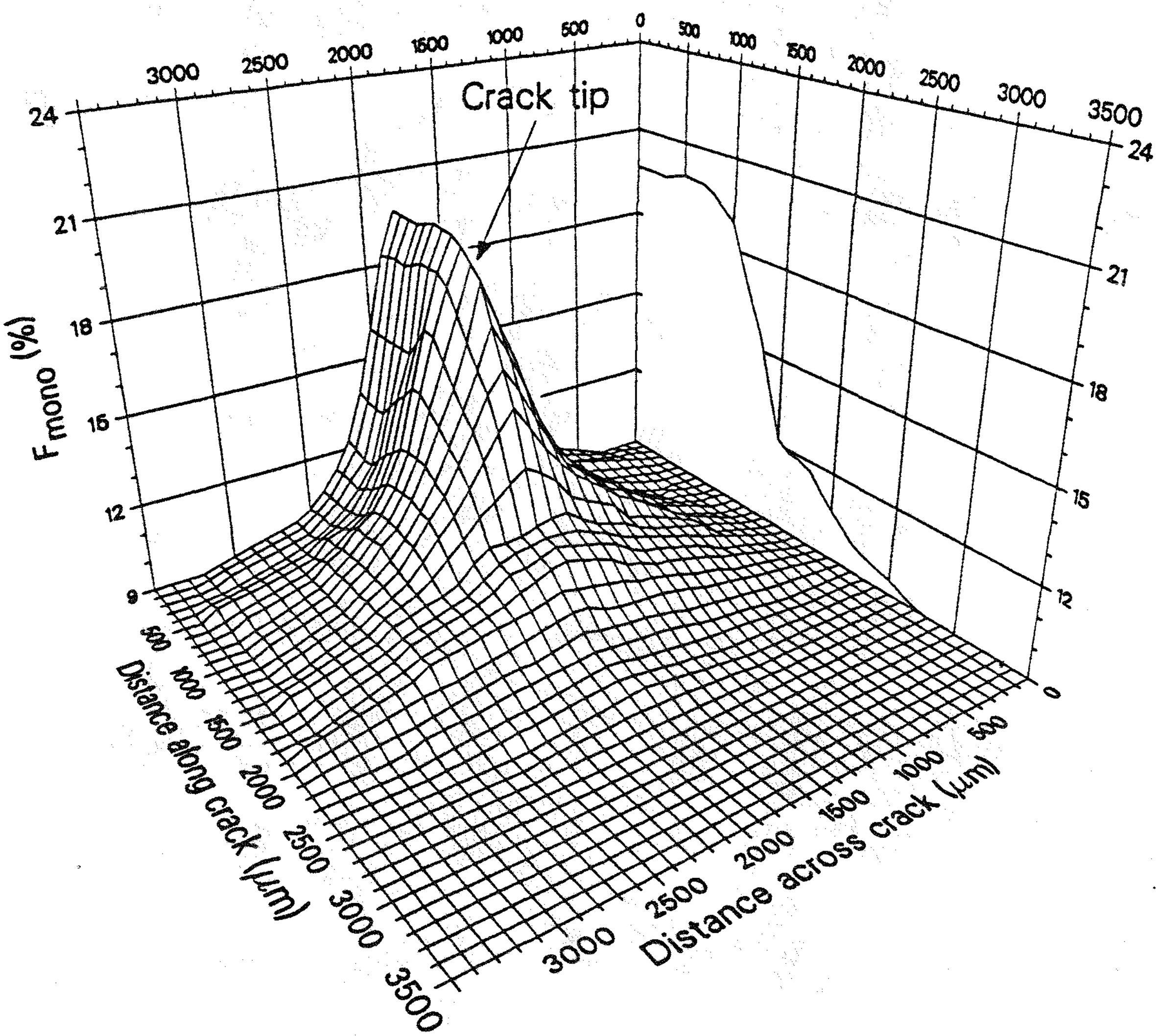

Fig. 4. Three-dimensional Raman spectroscopy plot of the morphology of the transformed zone both ahead, and in the wake of, the crack tip in peak-toughened MgO-PSZ. Plotted along the ordinate is the relative proportional of transformed (monoclinic) phase [21].

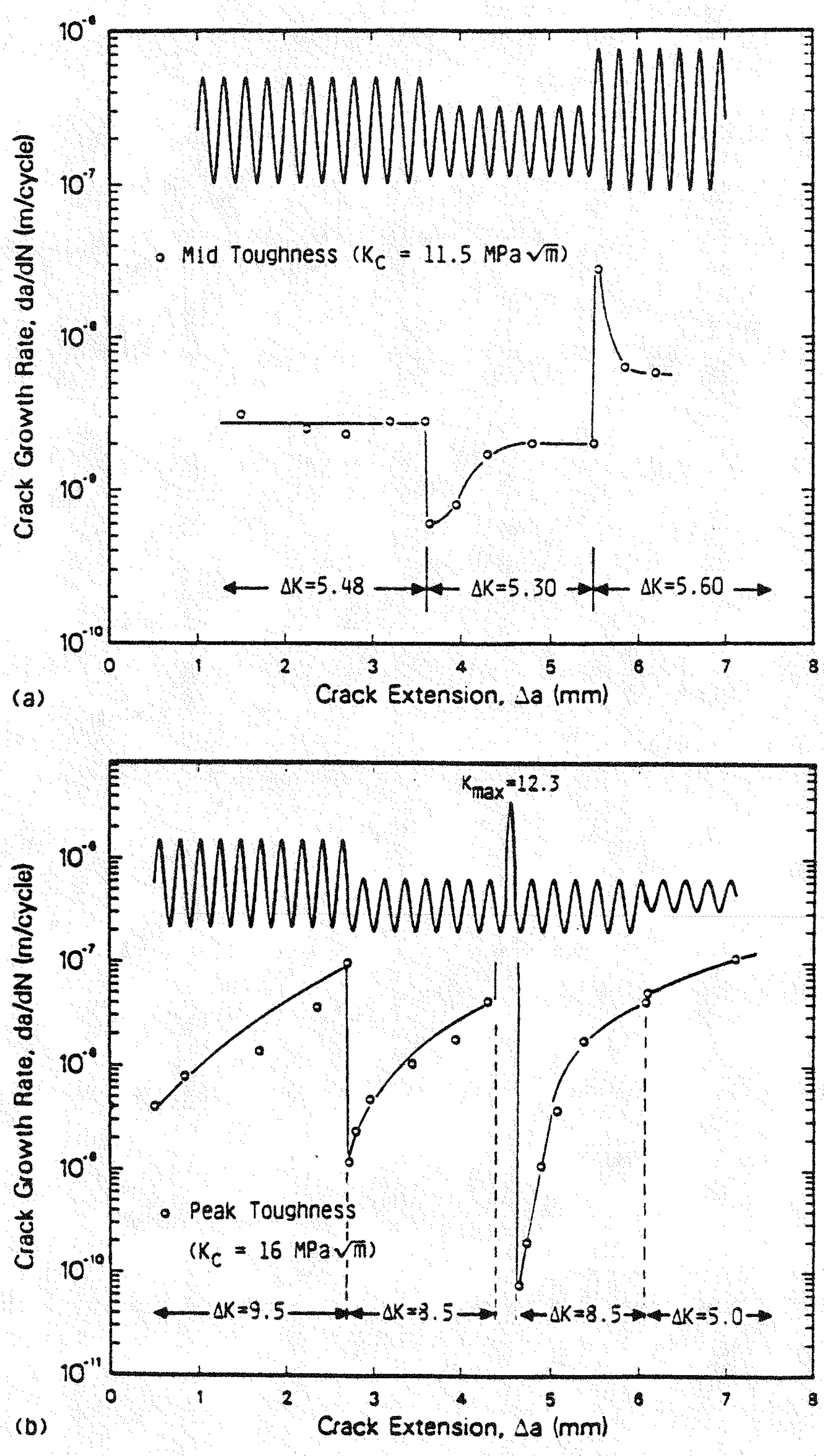

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Fig. 5. Transient fatigue-crack growth behavior in a) mid-toughness and b) peak-toughness MgO-PSZ due to variable-amplitude cyclic loads, showing immediate crack-growth retardations following high-low block overloads, immediate accelerations following low-high block overloads, and delayed retardation following a single tensile overload (room air environment).

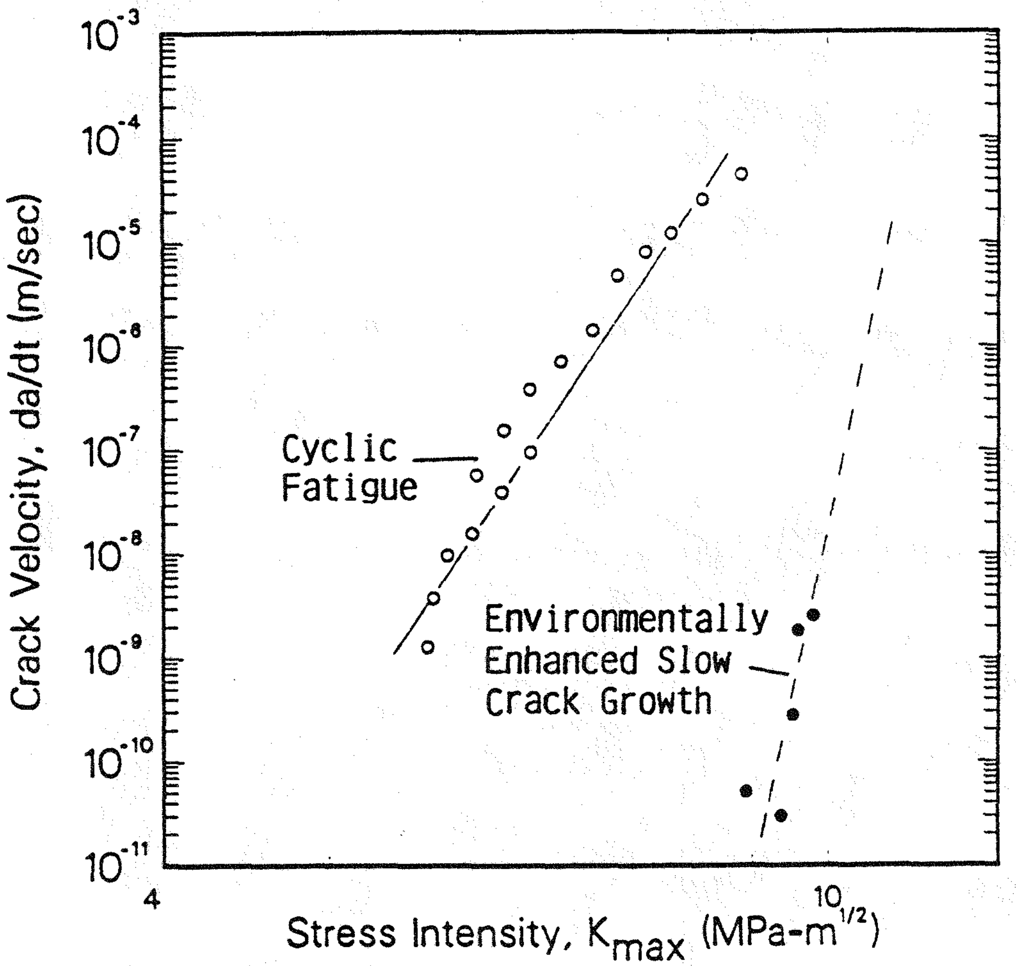

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Fig. 6. Subcritical crack-growth behavior in mid-toughness MgO-PSZ, showing a comparison of crack velocities da/dt, as a function of K_{max}, measured under monotonic and cyclic loading conditions in a moist air environment. Note how the cyclic crack velocities are up to 7 orders of magnitude faster at equivalent stress-intensity levels.