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Subcritical Crack Growth and Thresholds in a 3Y–TZP Ceramic Under Static and Cyclic Loading Conditions

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Abstract: Crack propagation behaviour of a 3Y–TZP ceramic is investigated under static and cyclic fatigue loading by means of the Double Torsion method, with a particular interest on the presence of thresholds under which no propagation occurs. A crack accelerating effect is shown under cyclic loading, correlated with a decrease in the threshold value.

1 INTRODUCTION

Subcritical Crack Growth (SCG), which consists of a slow propagation of flaws, is one of the major causes of damage to ceramics with time. SCG under constant loading is due to stress corrosion at the crack tip. It is well established that three distinct propagation stages, designated I, II and III, are observed in a crack velocity versus applied stress intensity factor (V–K_I) curve. Region I, corresponding to low velocities, has attracted the most attention, because it controls ceramic component lifetime. In this region, V is often related to K_I by an expression of the form:[1]

$$V = A \cdot K_I^n \tag{1}$$

An aspect of the SCG behaviour in ceramics of fundamental and practical interest is the tendency of V–K_I curves to show a threshold: it defines a stress intensity factor value K_{IO} under which no propagation occurs, thus leads to unlimited lifetime. Although the presence of thresholds in ceramics is thermodynamically well described,[2] and has been demonstrated in some glasses,[3] investigations in polycrystalline ceramics are still lacking because of the experimental difficulties involved in its determination.

Cyclic fatigue of ceramics recently became an attractive research field. The fact that reduced lifetimes are observed under cyclic loading, compared with static loading, is now accepted. Lifetime degradation occurs in different types of ceramics, either showing phase transformation or not.[4,5] In the former, cyclic fatigue effects are linked to a degradation of transformation induced crack-tip shielding.[6] For lifetime predictions of real components subjected to cyclic loading conditions, the integration of SCG measured under quasi-static conditions over the cycles is therefore inaccurate, and cyclic fatigue experiments must be conducted. The presence of a cyclic fatigue threshold is also of practical importance and can have a lower value than the one measured in static conditions, because of cyclic degradation.

SCG of 3Y–TZP has been investigated by several authors.[7–10] Under static conditions, crack propagation is caused by stress corrosion at the crack tip by the dissociated H_2O ions, and the V–K_I graph exhibits a standard three stages form.[7,8] Knechtel *et al.*[7] have suggested the presence of a threshold K_{IO}, but the few results presented are to be confirmed. Cyclic fatigue has been studied in detail by Liu and Chen[9] and Grathwohl and Liu.[10] Cyclic fatigue effects were clearly visible from the much shorter lifetimes under cyclic, as compared to static, conditions. Liu and Chen[9] have proposed a growth law in cyclic fatigue for short indentation cracks:

$$da/dt = A' \cdot (K_{max})^m \cdot (\Delta K)^2 \quad (2)$$

where K_{max} and ΔK are, respectively, the maximum value and the amplitude of the stress intensity factor over a cycle. Expression (2) can been written:

$$\frac{(da/dt)}{(1-R^2)} = A' \cdot K_{max}^{m+2} \quad (2b)$$

with $R = K_{min}/K_{max}$.

A threshold intensity factor in cyclic fatigue, lower than the threshold in static fatigue, has been presented by Grathwohl and Liu[10] from survivors to cyclic fatigue in bending during 200 h, with $R = \sigma_{min}/\sigma_{max} = 0.2$.

The purpose of the present investigation was to study and compare SCG under static and cyclic loading with a direct method, with a particular interest on the presence of thresholds under different loading conditions.

2 EXPERIMENTAL

A direct method, namely the Double Torsion, was chosen because measurements could be made on the plateau of the R-curve (steady-state). Indeed, 3Y–TZP exhibits a steeply rising R-curve over few microns (~10 μm), and a subsequent plateau toughness.[9] Therefore, studies using specimens containing large cracks are not perturbed by a rising toughness. The experiments were conducted on TZP, containing 3% mol of yttria (Ceramiques Techniques Desmarquest, France) with a grain size of 0.59 μm (additional information can be found in Ref. 11). The DT specimens consisted of plates of dimensions 20×40×2 mm^3. Testing and specimen geometry are detailed in a previous article.[11] A notch of dimensions: length $a_0 = 10$ mm and root $\varphi = 0.1$ mm was machined with a diamond saw. Subsequent pre-cracking at very low speed induced a sharp natural crack of length ~13 mm in each tested specimen. For such a crack, transformation zone was fully developed, and investigations were made on the plateau of the R-curve. Crack velocities were calculated directly from the crack advance measured optically on the surface of specimens. The surface of the specimens was therefore polished down to 1 μm to observe the crack length with a precision of ±2 μm with an optical microscope. Thus, with holding times of 7 days, crack velocities on the order of 10^{-11} m/s could be measured. Compliance analyses of the DT method[12] show that K_I is theoretically independent of the crack length. However, it has often been demonstrated that, in practice, stress intensity factor is slightly variant over the specimen length. Therefore, an empirical K_I expression proposed by Chevalier *et al.*[11] was used in the present study:

$$K_I = H \cdot P \cdot \left(\frac{a}{a_o}\right)^{6/32} \quad (3)$$

where a is the crack length measured on the tensile side of the DT specimen.

With expression (3), K_I could be calculated by means of the applied load and the crack length. Static and cyclic tests were conducted on separate specimens. Cyclic fatigue frequency was fixed to 1 Hz, and three R ratios (namely 0.1, 0.3, 0.5) were chosen for SCG laws determination. Measurements were carried out under laboratory conditions, in air.

V–K_I laws (or V–K_{Imax}) were practically determined by the following procedure: a specimen with a known crack size, a, was submitted to a constant (or cyclic) load for a given period of time Δt, chosen to induce a short propagation Δa of the crack (~5–50 μm). Crack velocity, given by $\Delta a/\Delta t$, was related to K_I (or K_{Imax}) calculated with expression (3) for $a + \Delta a/2$. Therefore, V–K_I (or V–K_{Imax}) laws could be determined by conducting several crack advance experiments. The presence of a threshold was investigated at low stress levels from long duration tests (several days). In the case where no crack advance was observed during the period, an upper value of 4 μm/Δt was calculated (4 μm corresponding to the precision of the measurement with the optical microscope).

3 RESULTS AND DISCUSSION

Figure 1 shows the V–K_I curve of the material in a log–log scale. The arrows indicate limit values for which no propagation was observed within the loading period. The crack velocity curve exhibits a three regimes shape, as already mentioned by Knechtel *et al.*[7] and Chevalier *et al.*[8] The tendency to a threshold K_{I0stat} at 3.5 MPa√m is clear from the curvature of the V–K_I graph at crack rates lower than 10^{-9} m/s and from the long time tests where no propagation takes place. In region I, data were correctly fitted by the power law (1) with $A = 1.8 \times 10^{-26}$ and $n = 32$.

Cyclic fatigue results are plotted in Fig. 2 in a log ($V/(1-R^2)$) versus log (K_{max}) graph in order to be consistent with expression (2) of Liu and Chen.[9] A single master curve is obtained for the three R ratios for crack velocities higher than 10^{-8} m/s, suggesting that expression (2) is still valid for macroscopic flaws with $m=18$ (results of Liu and Chen.[9] for microscopic flaws in a 3Y–TZP are presented in comparison). This result is of importance since it means that small and long cracks exhibit the same behaviour, at least for a crack speed higher than 10^{-8} m/s. This was already mentioned by Chevalier *et al.*[8] in the case of static fatigue.

Interesting is the presence of threshold values K_{IOmax}, dependent on the R ratio:

$$K_{IOmax} = 3.2\text{MPa}\sqrt{m} \text{ for } R = 0.5 - K_{IOmax} = 3\text{MPa}\sqrt{m} \text{ for } R = 0.3 - K_{IOmax} = 2.8\text{MPa}\sqrt{m} \text{ for } R = 0.1.$$

In any case, values are smaller than K_{IOstat}. Figure 3 shows a comparison of the V–K_I law obtained in static loading and the V–K_{max} law obtained under cyclic loading for $R=0.1$. Crack growth velocities are much higher under cyclic conditions, with a crack growth exponent m lower than n (respectively, 18 and 32). This definitely demonstrates a higher susceptibility to cyclic loading conditions, in agreement with previous works on zirconia materials.

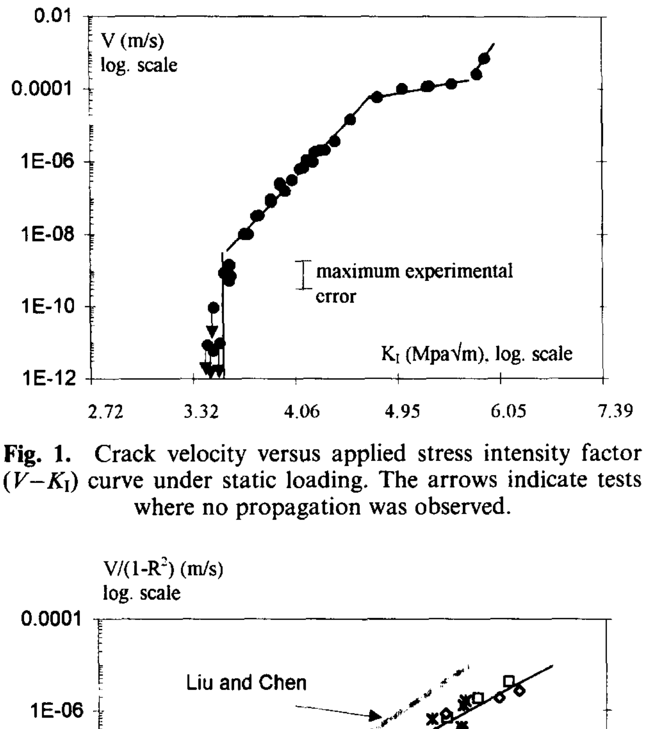


Fig. 1. Crack velocity versus applied stress intensity factor (V–K_I) curve under static loading. The arrows indicate tests where no propagation was observed.

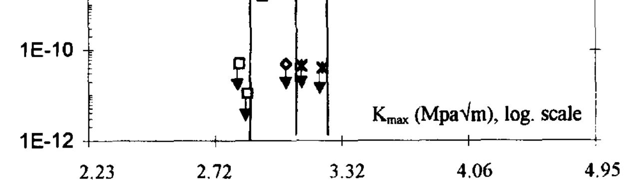


Fig. 2. ($V/(1-R^2)$) versus (K_{Imax}) diagram under cyclic loading for $R=0.1$ (□), $R=0.3$ (◇) and $R=0.5$ (*). Results of Liu and Chen[9] are shown in comparison.

The origin of cyclic fatigue crack accelerating effect in transforming ceramics has been studied in a CeTZP/Al_2O_3 composite by Tsai and Shetty.[6] The authors have clearly demonstrated that cyclic fatigue resulted in a degradation of transformation zone shielding. Transformation zones around the cracks consistently showed lower compressive stresses and higher stress intensity factors for the same loads during cyclic fatigue. Therefore, one can conclude that the plateau of the R-curve of zirconia materials is lower under cyclic compared to static conditions (cf. Fig. 4). The more realistic hypothesis is an environmentally driven crack growth, assisted by a shielding degradation under cyclic loading. Therefore, results presented above can be discussed in these terms:

Crack growth velocity is governed by the stress intensity at the crack tip K_{tip}, which is given by:

$$K_{tip} = K_I - K_{sh}$$

where K_{sh} is the shielding stress intensity factor, noted $K_{sh,stat}$ and $K_{sh,cycl}$ under static and cyclic loading, respectively.

Under cyclic solicitations shielding stress intensity factor $K_{sh,cycl}$ is lower, because of degradation of the transformed zone. If we call ΔK_{sh} the difference between $K_{sh,stat}$ and $K_{sh,cycl}$, ΔK_{sh} can be evaluated at low stresses by the difference between K_{IOstat} measured in static conditions and K_{IOmax} in cyclic fatigue. Indeed, the measured threshold in a given condition corresponds to the stress intensity factor K_{IO} at the crack tip necessary for the crack growth. The "real" threshold K_{IO} is in fact lower than the apparent thresholds K_{IOstat} and K_{IOmax}

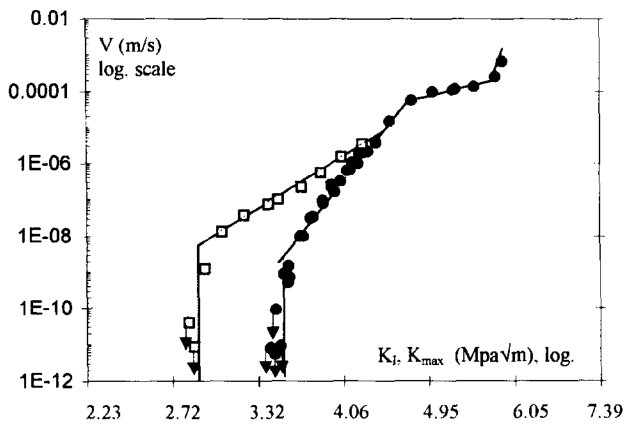
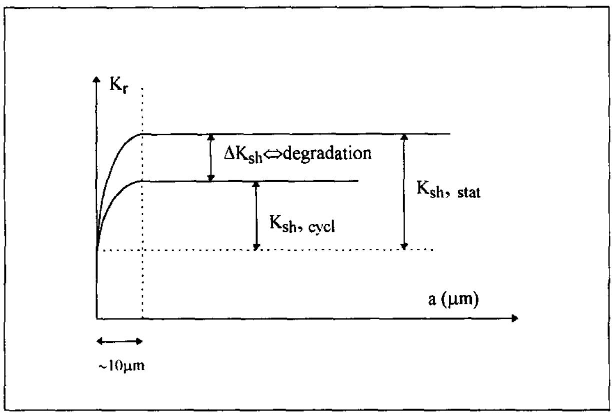


Fig. 3. Comparison of crack velocities under static (●) and cyclic (□) loading for $R=0.1$.



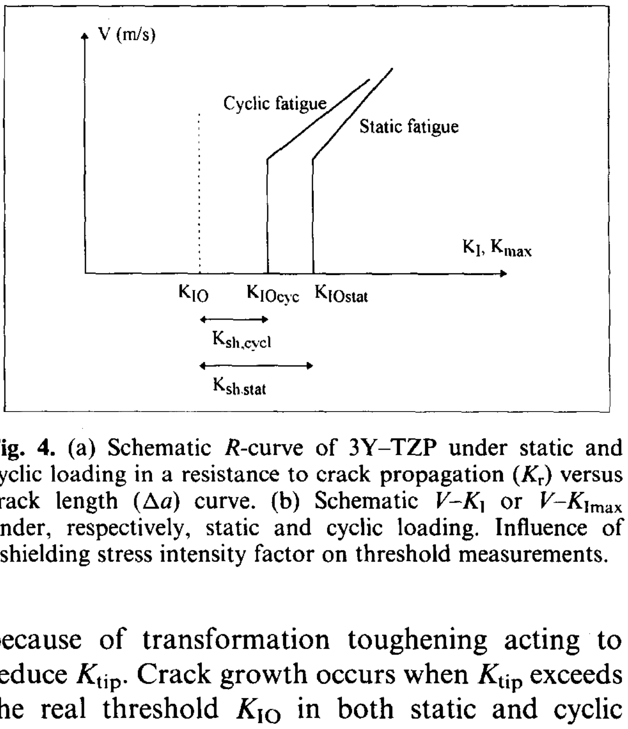


Fig. 4. (a) Schematic *R*-curve of 3Y–TZP under static and cyclic loading in a resistance to crack propagation (K_r) versus crack length (Δa) curve. (b) Schematic V–K_I or V–K_{Imax} under, respectively, static and cyclic loading. Influence of shielding stress intensity factor on threshold measurements.

because of transformation toughening acting to reduce K_{tip}. Crack growth occurs when K_{tip} exceeds the real threshold K_{IO} in both static and cyclic fatigue, but this corresponds to different applied stress intensity factors in the two cases, because of the difference of transformation toughening. From the difference in the measured thresholds in static and cyclic fatigue, ΔK_{sh} has been evaluated for the three cycling loading conditions: respectively, 0.7 MPa$\sqrt{m}$, 0.5 MPa$\sqrt{m}$ and 0.3 MPa$\sqrt{m}$ for R equal to 0.1, 0.3 and 0.5. Shielding degradation increases with decreasing R. It is to note that only tension–tension tests were presented in the article, and one can imagine an even stronger degradation in the case of negative R ratios. Note also that this discussion starts with the hypothesis that mechanical degradation and stress corrosion act in series. In fact, it is possible that such mechanisms acted in parallel and that a propagation took place under cyclic conditions even without stress corrosion at the crack tip, as recently demonstrated in non-transforming ceramics. Experiments in vacuum to avoid any corrosion effect would be interesting to separate mechanical fatigue from stress corrosion effects. Experiments at high temperatures, for which no transformation takes place, are also of great interest to investigate the role of transformation toughening on SCG behaviour.

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