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Static and cyclic crack propagation in Ce-TZP ceramics with different amounts of transformation toughening

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Abstract

Ceria partially stabilized zirconia ceramics (Ce-TZP) with identical grain size and different amounts of transformation toughening were processed to investigate the influence of phase transformation on static and cyclic fatigue crack growth.

Static crack growth is governed by environmentally stress induced corrosion at the crack tip and it is highly influenced by the crack shielding due to the phase transformation. Three fatigue mechanisms are expected to be operative at different proportions depending on the amount of transformation: wedge effect due to debris, degradation of bridging and modification of the shielding effect of the transformation zone. However, it is difficult to separate the contribution of the different mechanisms as grain bridging is induced by crack arrest due to phase transformation.
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Keywords: Ce-TZP; Fatigue; Transformation toughening

1. Introduction

It is now well recognized that monolithic engineering ceramics are prone to cyclic fatigue, particularly those undergoing crack growth resistance (R-curve behaviour), as alumina, silicon nitride and zirconia ceramics.[1–6] Cyclic loading enhances fatigue crack propagation compared to static loading at equivalent stress level, with a marked sensitivity to the maximum applied stress intensity factor, K_{max}. Efforts have been made to understand the mechanisms of cyclic fatigue and the toughening mechanisms are a major contributor to the material's vulnerability to cyclic degradation.

In this work, fatigue crack growth under static and cyclic loading conditions is investigated in Ce-TZP ceramics with identical grain size and different amount of phase transformation. The main motivation was to separate grain bridging due to the microstructure to phase transformation toughening.

2. Materials and procedure

Three Ce-TZP ceramics referred as 10Ce-TZP, 12Ce-TZP and 16Ce-TZP were processed from commercial stabilized zirconia powders (Zirconia Sales UK) with respectively 10, 12 and 16 mol% of ceria content. The as received powders were first compacted by uniaxial pressing at 20 MPa followed by an isostatic pressing at 350 MPa. The green compacts were sintered in air under different conditions (Table 1) so as to obtain an identical microstructure, i.e., an average grain size of 1.6 μm. The samples required for mechanical testing were cut from the sintered plates then ground and annealed at 1300 °C for 2 h to annihilate the t–m transformation, which had taken place during machining.

The materials are fully tetragonal after sintering; their properties are summarized in Table 1. From monoclinic content determined by X-ray diffraction (XRD) on surface fracture of bend bars, it can be concluded that the tetragonal phase is stable in 16Ce-TZP, whereas the amount of t–m is very high in the 10Ce-TZP material where a large number of autocatalytic transformation bands were observed on the tensile surface of bending specimens.

R-curve measurements were conducted at room temperature on SENB samples (4 mm × 6 mm × 40 mm) using 3-point flexural device with a span of 35 mm and a constant load point displacement rate of 5 μm/mn. Double torsion (DT) samples with dimensions 40 mm × 20 mm × 2 mm were used to obtain subcritical crack propagation curves, V–K_I (crack growth rate versus stress intensity factor), under static and cyclic loading conditions. Details of specimens geometry and loading con-

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Table 1
Material properties

Material	Sintering conditions	Relative density (%)	Young's modulous (GPa)[a]	Flexural strength (MPa)[b]	Monoclinic content (fracture surface) (%)[c]
10Ce-TZP	1450 °C–2 h	99	190	504	80
12Ce-TZP	1430 °C–2 h	99	192	540	60
16Ce-TZP	1450 °C–2 h	98	194	648	0

[a] Measured by resonance method.
[b] Measured in 4-point bending test.
[c] Measured by XRD.

figuration can be found in previous works.[7,8] Relaxation and constant loading tests were complementary performed to obtain static V–K_I curves. Cyclic fatigue experiments were performed under load control using a sine wave form with a ratio of minimum to maximum loads $R = P_{min}/P_{max} = 0.1$, at a frequency of 10 Hz.

3. Results and discussion

3.1. Crack growth resistance

The measured R-curves are shown in Fig. 1. The dashed line represents the toughness of the 16Ce-TZP material for which the fracture was unstable. The R-curve effect is limited for 12Ce-TZP with an initial and a plateau K_R values of 5.8 and 6.7 MPa m$^{1/2}$, respectively. The 10Ce-TZP shows a strong R-curve behaviour attributed to the autocatalytic phase transformation: starting from an initial value of 8.5 MPa m$^{1/2}$, K_R reaches 13.4 MPa m$^{1/2}$ after 700 μm of crack extension, and increases less steeply up to 15 MPa m$^{1/2}$, without evidence of a plateau value.

3.2. Static fatigue crack growth

The static V–K_I curves are shown in Fig. 2. The curves of 12Ce-TZP and 16Ce-TZP are roughly parallel with threshold values of 5.1 and 2.3 MPa m$^{1/2}$, respectively. The presence of three stages is consistent with the environmentally stress induced corrosion mechanism as it was observed for other zirconia ceramics.[7,9] The shift of the V–K_I curve of 12Ce-TZP to high K_I values is attributed to the increase of the transformation toughening capacity in this material. This shift is accentuated with 10Ce-TZP for which subcritical crack growth starts at stress intensity factor as high as 20 MPa m$^{1/2}$. This material shows atypical behaviour with serrated V–K_I curve correlated to discontinuous crack growth due to the autocatalytic transformation (Fig. 3). Crack arrests resulting from the high shielding effect of transformation zone occurred, followed by instabilities traduced by sudden load drops during the relaxation tests. The instabilities were always connected to a crack branching (Fig. 4). Similar behaviour, locally analogous to the "V" shaped V–K_I curve corresponding to small crack growth effect, was previously reported by Liu et al.[10] for Ce-TZP materials with different grain sizes.

3.3. Cyclic fatigue crack growth

Fig. 5 compare cyclic and static crack propagation results of 12Ce-TZP and 16Ce-TZP. Evidence of cyclic fatigue effects can be seen, even for the non transforming material, with significantly higher crack growth rates. No evidence of phase transformation or grain bridging was observed in 16Ce-TZP. Thus, the cyclic fatigue in this material can be attributed to frictional degradation of fractured surfaces that may lead to a wedging

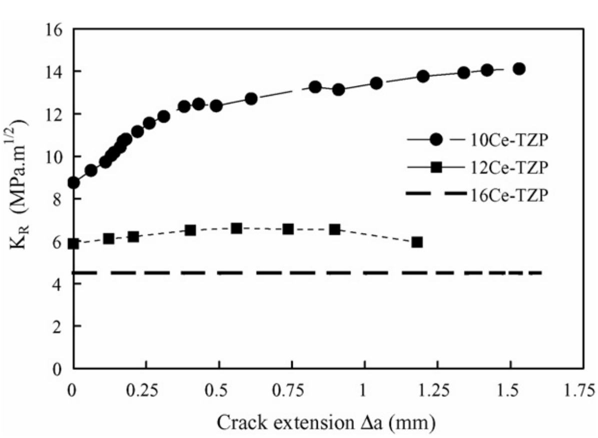

Fig. 1. R-curves measured on SENB samples, the dashed line represents the toughness of the 16Ce-TZP material.

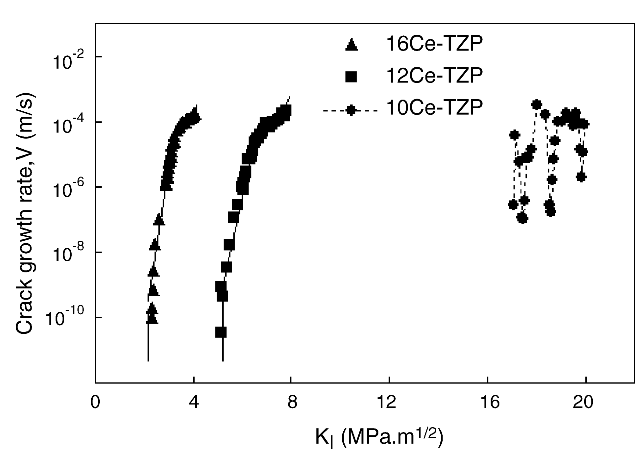

Fig. 2. Static crack propagation behaviour of 10Ce-TZP, 12Ce-TZP and 16Ce-TZP.

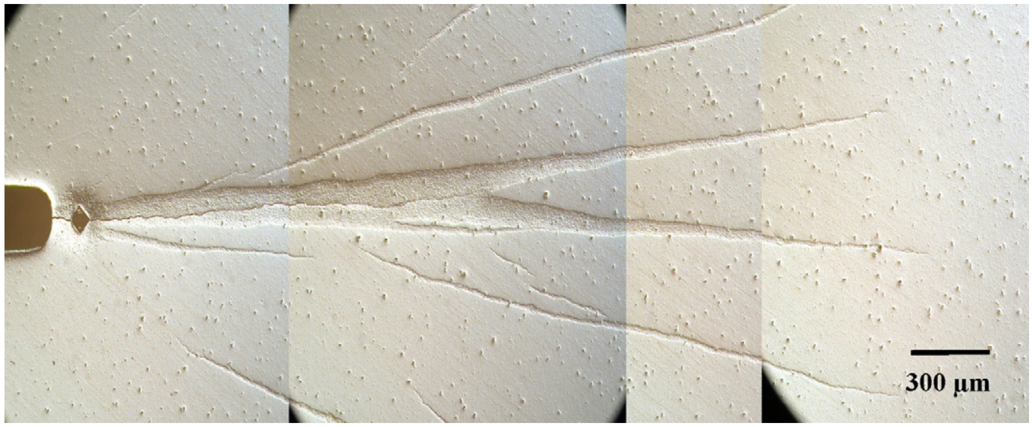

Fig. 3. Transformation zone of the 10Ce-TZP material during relaxation test.

action of fracture surface asperities by debris as it was involved for Ce-TZP–alumina composites.[11] AFM observations of static crack path in 12Ce-TZP (Fig. 6) show that the crack growth is mainly intergranular in this material with substantial crack bridging by monoclinic transformed grains. No change of the transformed zones was observed under cyclic loading of 12Ce-TZP. Rather than reduced crack shielding from transformation zone, the cyclic fatigue effect in this material can be mainly attributed to cyclic degradation of the observed crack bridging by the transformed grains as it occurs in non-transforming ceramics like alumina.[12]

For the autocatalytic transformation material 10Ce-TZP, unstable crack growth behaviour leading to the failure of the tested samples was observed at the early stage of cyclic loading, even at very low stress intensity factor (less than 50% of the value reached before cyclic loading). Although no cyclic crack growth measurements could be performed for this material, it can be concluded that it is more sensitive to cyclic crack growth than the two others. As the cyclic tests were conducted on pre-cracked specimens with established transformation zone, fast accumulation of cyclic fatigue damage may occur also by coalescence of secondary cracks due to substantial branching observed during static crack growth in this material (Fig. 4). This accentuates the anti-shielding effect at the crack tip, i.e., it increases drastically the crack tip stress intensity factor K_{tip}. As a consequence, the unstable crack extension observed during static loading is enhanced, resulting in complete failure of the sample.

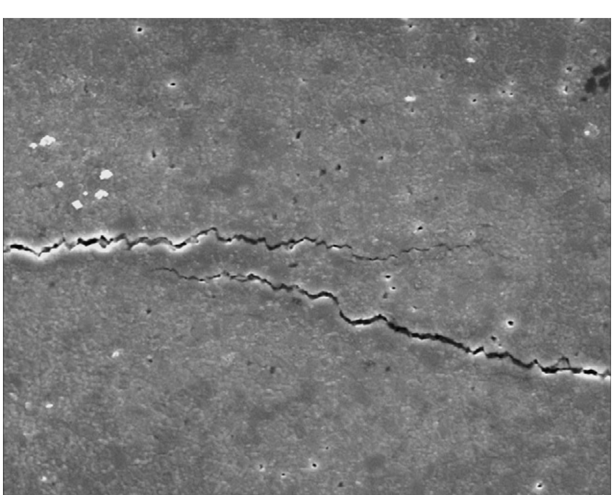

Fig. 4. SEM micrograph of crack branching associated with acceleration of crack rate in 10Ce-TZP (relaxation test).

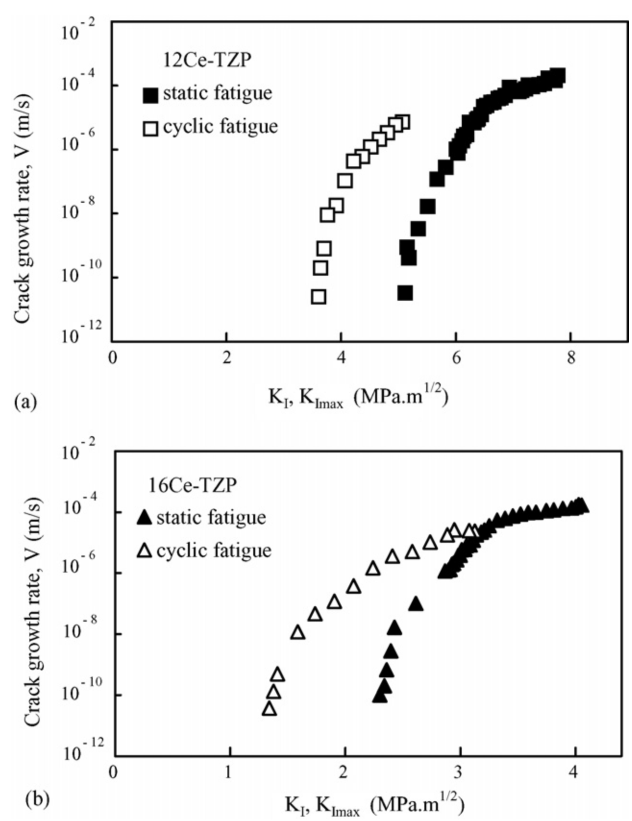

Fig. 5. Comparison of static and cyclic crack propagation behaviour of 12Ce-TZP (a) and 16Ce-TZP (b).

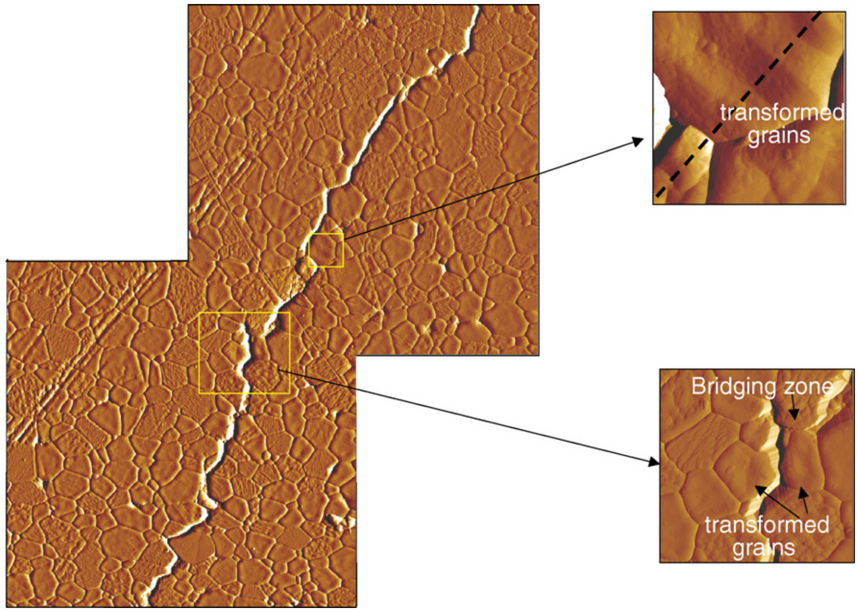

Fig. 6. AFM micrograph of static crack path in 12Ce-TZP showing crack bridging by monoclinic transformed grains.

4. Conclusions

From AFM and MEB observations of crack path, it can be concluded that phase transformation induces two other toughening mechanisms:

(i) crack bridging by monoclinic transformed grains resulting from crack arrest due to the shielding effect of the transformation zone.
(ii) crack branching operating within a large zone in the presence of autocatalytic transformation.

It is thus difficult to separate the contribution of each mechanism, particularly, the influence of grain bridging and transformation toughening, the initial objective of this work. The first mechanism is different from the simple grain bridging due to microstructure, generally observed in non transforming ceramics. Indeed, it cannot be dissociated from the phase transformation and its importance seams to increase with the amount of transformation.

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