Full Length Article

Fracture behavior of Ce-TZP/alumina/aluminate composites with different amounts of transformation toughening. Influence of the testing methods

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\begin{abstract}

The fracture behavior of four Ce-TZP zirconia composites containing 8 vol\% alumina and 8 vol\% strontium hexaaluminate was investigated. The composites exhibited different degrees of transformation toughening obtained by varying the amount of the CeO\textsubscript{2} stabilizer and the sintering temperature. The strength was measured by 4-point bending (4PB) and piston-on-three balls (POB) methods. Toughness and crack growth resistance (R-curve) were determined from Single Edge V-Notched Beam (SEVNB) and double torsion (DT) samples, and slow crack growth (SCG) curves were determined by DT method. Increasing the transformability of the composites enhanced their crack growth resistance and consequently, increased their resistance to SCG, which was completely inhibited for the most transformable composites. Simultaneously, flaw tolerance was also improved although a decrease in strength was observed. Under all configurations, the composites exhibited a plastic behavior and it was shown that their properties are correlated to the crack shielding due to autocatalytic phase transformation that not only depend on the material transformability, but is also strongly influenced by the testing method.

\end{abstract}

1. Introduction

Zirconia based ceramics are successfully used for a wide range wide range of technical and biomedical applications, due to their bio-compatibility and excellent mechanical properties, resulting from the stress-activated tetragonal to monoclinic (t-m) phase transformation \cite{1,2}. Yttria-stabilized zirconia polycristals (Y-TZP) are the mostly used, in particular for biomedical applications, due to their improved strength (\textasciitilde 1 G Pa), despite their moderate toughness and susceptibility to aging degradation \cite{3}. Besides, ceria-stabilized zirconia (Ce-TZP) received considerable attention due to their complete resistance to aging \cite{4} and high toughness \cite{5,6}. However, they have a modest formability, but is also strongly influenced by the testing method.

Another approach consists of developing “in situ” platelets reinforced composites with Ce-TZP zirconia reinforced with alumina and strontium or lanthanum aluminates \cite{8,14,15} that showed attractive combination of strength and fracture toughness \cite{16}. In the framework of the European “LongLife” project, a fully dense tri-phasic zirconia based composite composed of 84 vol\% Ce-TZP, 8 vol\% \textit{Al\textsubscript{2}}O\textsubscript{3} and 8 vol\% \textit{SrAl\textsubscript{12}}O\textsubscript{19} aluminate platelets was developed, with ceria contents ranging between 10 and 11.5 mol\% \cite{17}. By refinement of the microstructure and adjustment of the stabilizer content, excellent properties could be obtained for these materials \cite{18}. In the present study, the fracture behavior of similar composites with 11 and 11.5 mol\% CeO\textsubscript{2} contents obtained by industrial processing (i.e. isostatic pressing of a spray dried composite powder instead of post-doping of zirconia powder with alumina and aluminate precursors and slip casting in \cite{17,18}), is more deeply investigated by determining the strength, crack growth resistance and slow crack growth curves. A focus is made on the influence of the amount of transformability and the specimen geometry, with comparison to conventional 3Y-TZP.
2. Materials and methods

2.1. Materials

Four grades of Ce-TZP/8 vol% Al2O3/8 vol% SrAl12O19 composites, provided by DOCERAM GmbH (Germany), were used. The starting composite powders were synthesized and provided by DAIICHI KIGENSO LTD (Japan), on demand, in the form of spray-dried granules with organic binders to facilitate pressing. The spray dried powders were compacted under isostatic pressing at 300 MPa and sintered at different temperatures. The composites, hereafter named xCe-8A-8AS, differ by their CeO2 stabilizer content (x = 11 or 11.5 mol%) or sintering conditions (1 h at 1450 or 1500 °C), as detailed in Table 1. 3Y-TZP zirconia, sintered at 1480 °C for 3 h, was also delivered by DOCERAM and used as a benchmark for comparison. Its mean grain size initially of 0.6 μm was increased up to 1.9 μm by further annealing 2 h at different temperatures between 1520 °C and 1670 °C.

All the composites were fully dense (> 99.9%) and characterized by identical values of Young’s modulus and Poisson’s ratio respectively of 220 G Pa and 0.3 (determined by the resonance vibration method). The Vickers hardness was slightly lower for the composites sintered at 1500 °C (Table 1).

The transformability of the composites was characterized considering the size of the transformed zone around a Vickers indentation induced by an indentation load of 300 N as in [18]. It was also characterized by means of the measurement of the t-m transformation temperature. For this purpose, samples were cooled down progressively at low temperatures (below the ambient) above a liquid nitrogen bath and the temperature recorded by a thermocouple. The transformation was observed when spontaneous cracking/failure of the sample occurred, due to the bulk transformation of the specimens.

2.2. Strength measurements

Strength measurements were conducted in four-point bending (4PB) using rectangular bars (4 mm × 3 mm × 40 mm) with outer and inner spans of 35 and 10 mm respectively. They were also conducted by biaxial bending with disks (diameter of 15 mm and thickness of 1.2 mm) loaded in piston-on-three balls (POB) configuration as described in [19]. For both tests, the experiments were performed on polished samples (down to 1 μm) using a universal testing machine (Instron 8500). The cross-head speed was set at 5 mm/min until failure, to limit effects of slow crack growth.

2.3. Fracture toughness and R-curve measurements

The fracture toughness, KIC, and the crack growth resistance curves (R-curves) of the composites were determined by two different testing methods: single edge V notch beam (SEVNB) [20] and double torsion (DT) tests [21]. For the SEVNB tests, rectangular bars (4 mm × 3 mm × 40 mm) with one side polished to 1 μm finish for crack growth observations, using a diamond blade with a thickness of 0.2 mm. The notches were further sharpened with a fine razor blade and diamond paste of 1 μm and the samples were annealed at 1200 °C for 20 min to eliminate the machining residual stresses. For KIC measurements, the samples were notched to a relative depth of 0.4 and loaded in a 4-point-bending device (10–35 mm) at a cross-head speed of 5 mm/min. For R-curve determination, SEVNB samples with a relative depth of 0.5 were loaded in three-point bending with a span of 35 mm and a cross-head speed of 0.005 mm/min. The R-curves were determined from the recorded load-displacement curves, in terms of the stress intensity factor, K, and plotted versus the crack extension, Δa. K was calculated from the maximum recorded load and the corresponding crack length determined by a compliance formula [22] corrected by an empirical factor, adjusted so that the final calculated crack corresponded to the measured value at the end of the test.

DT torsion specimens consisted of plates measuring 40 mm × 20 mm × 2 mm, polished on the tensile surface to 1 μm finish to enable crack growth measurements and observation of the transformation behavior. The samples were notched to 10 mm with a diamond saw, then annealed at 1200 °C for 20 min. Subsequent pre-cracking was performed by loading the specimens at a low rate in order to induce a sharp crack with an initial length of 13 mm. To determine the fracture toughness, a set of DT samples were loaded to fracture at a high displacement rate of 5 mm/min and KIC was determined at the maximum load, using the stress intensity factor expression given in [21]. For R-curve measurements, DT samples were subjected to a series of loading-unloading sequences to follow crack extension and the evolution of transformation zone. K was determined from the stress intensity factor at each point of the load-deflection curve and plotted versus the crack extension Δa, optically measured.

Concerning the 3Y-TZP materials, the fracture toughness was also evaluated using two methods: SEVNB and stable indentation crack growth in bending (SIGB), described in [11,23]. It is to note that this latter method is not suitable to determine the fracture toughness of the Ce-TZP based composites as no cracks were formed from the indentation corners even when an indentation load as high as 300 N was applied.

2.4. V-KI curves

To investigate the slow crack growth behavior of the composites, V-KI curves (crack growth rate versus stress intensity factor) were determined in air at room temperature using the load-relaxation method during double torsion tests [24]. Pre-cracked DT specimens were rapidly loaded (at a displacement rate of 1 mm/min) to a certain load value from which the displacement was kept constant. The relaxation curve (load versus time) and the crack length deduced from compliance calibration curve were used to determine the V-KI curves as detailed in [21].

3. Results and discussion

3.1. Microstructure and transformability

The composites presented similar kinds of microstructures, an example of which is shown in Fig. 1, where three phases can be distinguished: a matrix of ceria stabilized zirconia, a secondary phase of alumina particles located both at the grain boundaries and inside zirconia grains, and strontium hexa-aluminate platelets at the zirconia grain boundaries. For all the composites, the mean grain size of alumina was of 0.3 ± 0.1 μm, the length of the aluminate platelets was of

<table>
<thead>
<tr>
<th>Nomenclature and characteristics of the studied composites.</th>
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<tbody>
<tr>
<td>Composite</td>
</tr>
<tr>
<td>-----------------</td>
</tr>
<tr>
<td>11Ce-8A-8AS</td>
</tr>
<tr>
<td>1450</td>
</tr>
<tr>
<td>11.5Ce-8A-8AS</td>
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<td>1450</td>
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1.7 ± 0.5 μm and their aspect ratio (length/width) of 5 ± 2. The mean grain size of the Ce-TZP phase was of 1 ± 0.3 μm for the composites sintered at 1450 °C and increased by a factor two when the sintering temperature was increased to 1500 °C, independently on the CeO₂ content (Table 1). It is to note that the industrial processing of the studied composites led to higher size of the zirconia grains and aluminate platelets, in comparison to those developed by a post-doping strategy and slip casting in [17,18].

In the following, the composites will be classified and denoted according to their transformability as shown in Table 1 and Fig. 2. The most transformable composite characterized by the largest transformation zone and the highest transformation temperature corresponds to the lower ceria content and the highest sintering temperature (11Ce-8A-8AS/1500 °C); it will be denoted by HT (for higher transformability). The less transformable one corresponds to the highest ceria content and the lowest sintering temperature (11.5Ce-8A-8AS/1450 °C) and it will be denoted by LT (for lower transformability). The other two composites exhibited similar, intermediate level of transformability (close transformation temperatures) and will be denoted by MTᵢ (ᵢ = 1 or 2) (for medium transformability). It was found that the MTᵢ composites present similar mechanical behavior; in the following, the results corresponding to one or the other of them will be presented and they will be designed by MT without distinction in the discussion.

3.2. Fracture toughness and R-curve behavior

Table 2 shows the fracture toughness of the studied composites measured by SEVNB and DT methods. For the SEVNB samples, no
significant dependence on the amount of transformability was observed for the fracture toughness (mean value of 10.4 M Pa m$^{1/2}$), which is comparable to reported values for 10Ce-TZP/Alumina composites [25]. DT toughness values are significantly higher, particularly for the two most transformable composites MT and HT, for which an extraordinary value of 25 M Pa m$^{1/2}$ was obtained. The lower fracture toughness value obtained by the SEVNB tests is however certainly more realistic as in this configuration, the fracture corresponds to small cracks. In comparison, for the 3-YTP materials results of which are shown in Fig. 3 as a function of the grain size, the fracture toughness values obtained by SEVNB and SIGB methods are close to each other and comparable to those reported in a previous work for DT samples [26]. This can be attributed to the low transformability of these materials (no transformed zones were observed around the indentation and crack path), which leads to a similar toughness values independently on the testing method.

More specifically, the influence of the testing method on the fracture toughness of the studied composites can be explained considering their R-curve behavior. For SEVNB samples, the $K_R$-curves (Fig. 4) start from an initial value of 6.6 M Pa m$^{1/2}$ and increased over 700 μm of crack extension to a steady state value (plateau value) of 10.5 M Pa m$^{1/2}$ and 12 M Pa m$^{1/2}$ respectively for the LT and the MT composites. For the DT samples, the $K_R$-curves (Fig. 5) start at a significantly higher level than for SEVNB. A limited increase of $K_R$ was observed for the composite with the lowest transformability, LT, for which a plateau value of 14 M Pa m$^{1/2}$ was reached. The MT and HT composites showed similar R-curve behavior with a steady increase up to 20 M Pa m$^{1/2}$ after 5 mm of crack extension, without reaching a plateau value. The increase of the crack growth resistance can be attributed to the transformation induced toughening that shields the crack tip from the applied stress [2,27,28]. Due to the volume expansion accompanying the transformation (about 4.5 vol%), compressive stresses are induced in the crack wake, thereby reducing the crack tip stress intensity factor, $K_{tip}$, corresponding to the driving force for crack growth, which may be expressed as:

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

(1)

where $K_I$ (=$K_R$) represent the applied stress intensity factor and $K_{sh}$ the shielding contribution of the phase transformation, proportional to the applied one [29]:

$$K_{sh} = C_{sh} K_I$$

(2)

where $C_{sh}$ is a parameter which increases with the material transformability. Due to this crack shielding, higher applied stress intensity

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**Table 2**

Fracture toughness values for SEVNB and DT tests.

<table>
<thead>
<tr>
<th>Composite</th>
<th>SEVNB</th>
<th>DT</th>
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<tbody>
<tr>
<td>HT</td>
<td>n.m.</td>
<td>25</td>
</tr>
<tr>
<td>MT₁</td>
<td>10.2 ± 0.2</td>
<td>n.m.</td>
</tr>
<tr>
<td>MT₂</td>
<td>10.3 ± 0.2</td>
<td>25</td>
</tr>
<tr>
<td>LT</td>
<td>10.6 ± 0.2</td>
<td>15</td>
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n.m. = not measured.
factor is required to induce crack growth, leading to crack growth resistance increase. Increasing the transformability of the material increases the shielding effect and thus the level of the crack resistance, which becomes particularly high for the composite with the highest transformability, HT. Crack bridging and branching were also observed in the studied composites (Fig. 6) and could contribute to the crack growth resistance. However, their contribution can only be minor compared to transformation toughening, as the crack bridging is limited to a very localized area and crack branching seems to be a consequence of phase transformation as in Ce-TZP ceramics with autocatalytic transformation [6], due to the high shielding effect of transformation zone, the main crack is arrested and a secondary crack is initiated from tensile region.

When the R-curve effect is important, which is the case for the studied composites, the fracture behavior is governed by the R-curve rather by a unique toughness $K_{IC}$, which correspond only to the particular point of fracture where the tangency conditions ($K_I = K_R$ and $dK_I/d\alpha = dK_R/d\alpha$) are satisfied [30]. Thus, depending on crack size and geometry, $K_{IC}$ measurements could fluctuate between the starting value (for short cracks) and the plateau value relative to long cracks. This explains the difference between SEVNB and DT fracture toughness measurement for the composites. The high DT toughness values (Table 2) correspond to the long crack stage, as they were obtained from samples pre-cracked over a relatively long length (2–3 mm from the initial notch), and thus correspond to pre-existing transformation zone and shielding effect. Furthermore, it has been shown that the R-curve of ceramic materials is influenced not only by the magnitude of the toughening but also by the sample geometry and crack length as well as by the test conditions [30–32]. The present results confirm this aspect as the difference between the R-curves obtained in SENVB and DT tests is clearly reflected in differences of the shape and extension of transformation zone around the crack path (Figs. 7 and 8). For SENVB samples, the transformation zone shown at the plateau value in Fig. 7 is limited to a narrow zone along the crack path, the width of which increases with the material transformability (50 $\mu$m for LT and 100 $\mu$m for MT). For DT samples, similar limited transformation behavior was observed in the less transformable composite, LT (Fig. 8), which is in accordance with the relatively low crack growth resistance of this material. In contrast, large transformed zones were observed for the MT and HT composites, with a marked autocatalytic transformation, characterized by an irregular dendritic shape and large number of transformation zone branches. Such transformation behavior was already observed for Ce-TZP [6,32] and Ce-TZP/Al$_2$O$_3$ composites [8,9,33]. This large ‘process zone’ is, in some aspects, analogous to the ductile behavior of metals. This analogy is also confirmed by the load-displacement curve during loading/unloading procedure of a notched DT sample (i.e. before pre-cracking), shown for the most transformable composite (HT) in Fig. 9. Nomarski optical micrographs of the transformation zone at different loading steps are also reported in this figure. Significant “plastic” deformation with no variation of the compliance was first observed before any crack propagation, due to a substantial extension of a transformation zone from the notch. The plasticity increased substantially with crack propagation (accompanied by a compliance increase) due to further expansion of the transformation zone at the crack path. By analogy with crack-tip plastic deformation, the difference of the transformation zones and related R-curves between SENVB and DT samples can be ascribed to differences in the thickness constraint between the two specimen types: the relatively lower thickness constraint in the DT geometry produces a larger transformation zone size as in [34] where short-double-cantilever-beam (s-DCB) specimens of Ce-TZP ceramics exhibited higher R-curve effects than single-edge-notched-beam (SENB) ones. This thickness effect is expected to be more pronounced in DT specimen as the crack propagates nearly at the tensile surface instead of through thickness, due to curved crack front [21]. To summarize, the DT results allowed providing evidence of the large-scale plastic deformation of the composites. However, in practice, small cracks are generally involved and the SENVB method provides an assessment of the mechanical properties at a more representative scale.

3.3. Strength and reliability

Fig. 10 shows representative load-displacement curves registered for the composites in POB or 4PB tests. In both configurations, the curves exhibited deviation from linearity, the onset of which decreased
with increasing transformability. This can be correlated to the appearance of transformation zones well before failure in these composites (Figs. 11–13). The most transformable composite, HT (tested only in 4PB), exhibited a particular behavior with a drastic drop of the load followed by a further increase until failure (Fig. 10a). For this composite, typical wedge-shaped transformation zones as reported by Sergo et al. [35], appeared at a very low stress level (< 250 MPa) at the side

Fig. 8. Optical micrographs of the transformation zone for DT samples of LT, MT₁ and HT composites.

Fig. 9. Load-displacement curve for the HT composite under DT test and Nomarski optical micrographs of the transformation zone at different steps of the loading/unloading procedure.

Fig. 10. Representative load displacement curves: a) 4PB test for LT, MT₁ and HT composites b) POB test for LT and MT₁ composites.
surface of the sample then expanded for further loading with a sudden formation of a large macroscopic transformed zone at the load drop (Fig. 11).

The influence of the testing method on the strength characteristics was investigated for the composites with the lower and medium transformation potentials, i.e. LT and MT. The results of the flexural tests are compared to those of Y-TZP in Fig. 14, where the mean strength values and the Weibull modulus, $m$, are reported respectively in bold and italic letters. It can be seen that the strength decreased when the material transformability is increased, with very high values...

Fig. 11. Evolution of the autocatalytic transformation on the side surface of the HT composite under 4PB test (the lower face correspond to tensile stress). Prismatic transformed bands at different loading stages (b, c) and generalization of the transformation before fracture (d).

Fig. 12. Transformation bands observed for LT and MT$_1$ composites under 4PB tests. (a) and (b) correspond to tensile surfaces respectively before fracture and at the fracture stress; (c) correspond to the side surface were the transformation was observed only for the MT composite.
for the biaxial POB test (> 1200 M Pa) compared to 4PB for LT and MT composites. Moreover, the gap between the two testing methods increased with increasing transformability, the POB strength being more than twice than that of 4PB for the MT composite. In comparison, for 3Y-TZP, the stress-strain curve of which was linear up to failure, the difference between the two measured strength values is only about 20%.

For the composite with the highest transformation potential, HT, a strength value of 290 ± 8 M Pa was obtained from 4PB measurements, confirming the tendency for the strength to decrease when the transformability is increased. This is a direct consequence of the strength limitation by the phase transformation, as discussed by Swain [7]. As shown above, the transformation in these composites occurs before crack propagation and as for metals, the transformation stress acts as a yield stress. The critical stress to trigger the t-m transformation is generally estimated from toughness characteristics and the transformation zone width, for a well-defined shape of the transformed zone [7,29,36], which is difficult to define in the case of the studied composites due to extended and non-homogeneous autocatalytic transformation. In this work, the evolution of the transformed zones was investigated by analogy with the plastic behavior of metals, considering two critical stresses corresponding to different levels of the phase transformation: (i) \( \sigma_t \), corresponding the onset of the autocatalytic phase transformation (analogous to the microscopic plastic deformation); and (ii) a macroscopic yield stress, \( \sigma_y \), corresponding to the deviation of the loading curve from linearity as the transformation becomes important (equivalent to the macroscopic yield point for metals). For POB disks, \( \sigma_t \) was calculated at the first apparition of the transformed bands (to this purpose, some samples were unloaded during the flexural tests for optical examinations with Normaski interference contrast). For 4PB bars, it was deduced from the linear stress...
distribution on the tensile surface, outside the supporting points. \( \sigma_t \) was taken as the stress value corresponding to the position of the outer transformed bands. The values of \( \sigma_T \) and \( \sigma_Y \) determined for representative samples are given in Table 3, where the mean strengths are also reported for comparison. It can be seen that in all configurations, the strength decrease is correlated to the decrease of both critical stresses when the material transformability is increased.

Another feature of the flexural results is the high Weibull modulus of the composites (in the range of 21–34 for LT and MT) that reached the value of 40 for the most transformable composite, HT. This reflects a significant increase of their reliability, traduced by a reduction of the strength scatter in comparison to Y-TZP (Fig. 14). Increasing the transformability increased the Weibull modulus in agreement with the observed increase in the R-curve effect. As discussed in [37–39], the induced stable crack propagation before failure tends to homogenize the size of the existing flaws, leading to an enhancement in flaw tolerance. For ceramic materials, the effect of the loading configuration on the fracture strength is generally well described by the Weibull analysis, considering the effective volume or the effective surface, for volume or surface defects respectively [40–42]. This analysis was successfully applied to the benchmark 3Y-TZP material for which an identical value of the Weibull modulus was found for 4PB and POB samples. Taking into account the effective surface subjected to tensile stress, the biaxial strength predicted from the mean value of 4PB measurement was of 1400 M Pa, which is within the range of the measured biaxial values for this material (1447 ± 127 M Pa). For the less transformable composite LT, an identical value of \( m \) was found for both tests. However, the predicted (effective surface corrected) POB strength was of 900 MPa, which is significantly less than the measured value (1306 M Pa). This finding, together with the variation of \( m \) with the testing method observed for the MT composite (Fig. 14), indicates a clear deviation from Weibull statistics for these materials, which is obviously related to their plastic behavior due to phase transformation.

The high difference between the strengths of 4PB and POB tests is in line with the results of our previous work [19] on another highly transformable 10Ce-TZP/Al\(_2\)O\(_3\)/La\(_2\)O\(_3\) composite. Rather than simple scaling effects, the influence of the sample geometry and the testing configuration on the flexural results can be directly correlated with the difference of transformation zones. For 4PB samples, irregularly spaced transformed bands with about 25 \( \mu \)m and 55 \( \mu \)m width were observed respectively on tensile surfaces of LT and MT composites (Fig. 12). Prismatic transformation zones reflecting the stress distribution were also observed on the sides of the MT samples (Fig. 12c). In the biaxial POB configuration, transformed bands appeared at the center of the sample then multiplied and propagated radially. For the less transformable composite LT, the transformation zone tended towards a netlike structure with randomly oriented bands (Fig. 13) whereas in the MT composite, it progressed towards a continuous circular zone with large number of individual branches. In both configurations, the fracture was initiated along transformation bands normal to the maximum principal stress direction. To characterize the transformation rate, the relative transformed area within the effective surface of the samples was estimated at the failure stress. The transformation ratio was about 50 and 80% respectively for LT and MT composites in the POB configuration whereas it didn’t exceed 1% in 4PB configuration for both composites. On the other hand, it can be seen from Table 3 that the critical stresses \( \sigma_T \) and \( \sigma_Y \) defined above are very close to each other for 4PB samples, whereas \( \sigma_Y \) is significantly high for POB tests. This can be understood considering the difference between the stress distributions in these configurations. A Finite element analysis has shown [19] that in POB configuration, the principal stress is maximal at the center of the sample and decreases rapidly toward its periphery. As a consequence, phase transformation starts at the center where the critical stress to trigger the transformation, \( \sigma_T \), is first reached. Due to the associated compressive stresses, the applied stress needs to be progressively increased to continue the transformation towards the periphery as shown schematically in Fig. 15. The non-uniform stress field in POB is therefore responsible of a circular zone under high compression from which cracks cannot escape. Only when the stress distribution becomes uneven (for very high applied loads), forming three lobes, cracks can propagate following one of the star shaped transformation band, as it can be observed in Fig. 13. In contrast, for the 4PB test where the stress is uniform between the two loading points, the transformation stress threshold is simultaneously reached within a large zone. The appearance of narrow transformation bands accommodate the stress only over very short distances, which explains the slight difference between the critical stresses \( \sigma_T \) and \( \sigma_Y \) and the fracture strength \( \sigma_f \) (Table 5).

The high variation of the strength with the loading configuration may thus be ascribed to the compressive residual stresses resulting from the autocatalytic phase transformation, which become particularly high under POB tests. Increasing their influence with the transformability is in accordance with the higher gap between the strength values for the MT composite. These results are of importance as they show that a direct comparison between reported strength values is not always valid for zirconia based ceramics and the commonly used Weibull statistics cannot be systematically applied to these materials. On the other hand, the results of the POB test used for example to qualify bioceramics for dental applications should be cautiously interpreted as overestimated strengths could be obtained for highly transformable materials. Strength characterization of highly transformable ceramics, must take into account representative sample size and shape as well as the real applied stress field.

Table 3

<table>
<thead>
<tr>
<th>Composite</th>
<th>( \sigma_T ) (M Pa)</th>
<th>( \sigma_Y ) (M Pa)</th>
<th>( \sigma_f ) (M Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HT</td>
<td>250</td>
<td>270</td>
<td>391</td>
</tr>
<tr>
<td>MT</td>
<td>385</td>
<td>391</td>
<td>700</td>
</tr>
<tr>
<td>LT</td>
<td>485</td>
<td>494</td>
<td>878</td>
</tr>
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Fig. 15. Schematic illustration of the stress distribution on the tensile plane of the POB disk at different loading levels (along the direction of maximal stress). The arrows show the evolution of the transformed zone as the applied stress reaches the critical stress to trigger the transformation.

...corrosion mechanism generally occurring in oxide ceramics as this composite is consistent with the environmentally stress induced crack path. The presence of two stages of crack propagation for relatively the same shape and size of the transformed zone around the threshold stress intensity factor, KI0, necessary to initiate slow crack growth. The threshold extrapolated from low crack velocity data, reached the exceptionally high values of 9, 16.2 and 21.5 MPa m1/2 for the LT, MT and HT composites, respectively for LT, MT and HT composites. It is to note that for the most transformable composites MT and HT, the shift of the V-KI curves as no single value of KIC could be defined (as explained above, the level of the V-KI curve varies from one test to another). It is commonly admitted that the higher the slope in the normalized diagram (i.e. the higher the KIC/KIC ratio), the lower the sensitivity to SCG and that this sensitivity decreases with the ionic to covalent bonding ratio. The MT and HT composites exhibit unusual behavior, with a normalized ratio KIC/KIC close to 1 and a very high slope, even higher than for covalent ceramics. The effect of phase transformation, extended to a large zone on either side of a growing crack as well as to its frontal area, is so high that it almost completely inhibited the crack extension due to SCG.

This suggests that no delayed failure would be expected in these highly transformable composites even at high level of stress intensity factor. However, the results must be considered only for qualitative comparison, due to the difficulty to determine a unique V-KI curve for...
MT and HT composites as the observed shift and steepness are artifacts of crack arrest and do not represent the real SCG behavior. This is comparable to toughness determination from direct length measurements of indentation cracks that could be trapped in the compressive transformed zones as discussed in [49] and observed for the studied composites (see 2.3, and Fig. 2), making this method unsuitable in this case. Moreover, the plastic behavior of these composites was not taken into account in the DT analysis, based on the stress intensity factor and suitable for linear elastic perfectly brittle materials. It is evident that V-K_{I} curves obtained on long crack with a wide transformation zone cannot be extrapolated to short cracks. Anyway, they show that shielding of the applied stresses by the transformation acts to limit slow crack growth in such highly transformable composites.

4. Conclusions

The fracture behavior of Ce-TZP/8 vol% Al_{2}O_{3}/8 vol% SrAl_{12}O_{19} composites with different amounts of phase transformation was investigated using different testing methods and the following conclusions may be drawn:

- In all configurations, the composites showed plastic like behavior with significant influence of the testing method on the size and extent of the transformation zones.

- With respect to resistance to crack propagation, the composites exhibited a rising R-curve behavior through extrinsic crack shielding that increased with the composite transformability. This effect was more pronounced in the DT samples due to larger transformation zones compared to SEVNb ones.

- As a consequence of the R-curve effects, the DT samples provided high toughness values (15–25 MPa m^{1/2}) corresponding to long crack regime. A more realistic toughness of 10.4 MPa m^{1/2} was obtained for all the composites with SEVNb samples, corresponding to short cracks.

- The strength is transformation limited and highly impacted by the testing method. In particular, the piston on tree balls test overestimates the strength and thus is not suitable for evaluating materials with high transformability, while it is often used in standards (e.g. ISO 6872 for dental ceramics).

- Improvement in flaw tolerance was evidenced, reflected by relatively high values of the Weibull modulus, m. On the other hand, it was shown that m depends strongly on the testing method and associated transformation zones, showing the unsuitability of the Weibull analysis for these highly transformable ceramics.

- Slow crack growth is highly influenced by the crack shielding due to phase transformation. The latter increases the crack propagation threshold and completely inhibits the SCG in the most transformable composites.

These findings outline the need to reassess the fracture testing methodology in the presence of transformation plasticity. Further work is required to better understand and describe the criteria applicable for the autocatalytic transformation, in order to apply correct mechanical behavior laws to highly transformable materials. In particular, investigation of through thickness transformation zones and integration of the plastic behavior could be helpful to define the most relevant methods for fracture characterization of these materials.

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- In all configurations, the composites showed plastic like behavior with significant influence of the testing method on the size and extent of the transformation zones.

- With respect to resistance to crack propagation, the composites exhibited a rising R-curve behavior through extrinsic crack shielding that increased with the composite transformability. This effect was more pronounced in the DT samples due to larger transformation zones compared to SEVNb ones.

- As a consequence of the R-curve effects, the DT samples provided high toughness values (15–25 MPa m^{1/2}) corresponding to long crack regime. A more realistic toughness of 10.4 MPa m^{1/2} was obtained for all the composites with SEVNb samples, corresponding to short cracks.

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