# FRACTURE RESISTANCE OF Y-TZP MATERIALS: INFLUENCE OF MICROSTRUCTURE

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Fracture properties of two microstructurally different Y-TZP (fine- and coarse-grained) have been studied. The coarse-grained zirconia exhibited a clear increase in fracture toughness, due to the larger effect of stress induced phase transformation. Strength results were fitted to a Weibull distribution function. The results of such a fitting indicated that the coarse-grained material presented a higher degree of flaw tolerance, and followed a three-parameter Weibull function, while the fine-grained one was rather described through a two-parameter function.

## INTRODUCTION

Zirconia-based ceramics show attractive mechanical properties which make them strong candidates for structural applications. From these properties, the main one is their large transformation toughening capability as related to the crack shielding effect promoted by the stress-induced phase transformation of tetragonal zirconia particles into the more stable monoclinic symmetry (1). Total or partial retention of metastable tetragonal zirconia at room temperature may be obtained through small additions of particular stabilising oxides (e.g., $Y_2O_3$, MgO, etc.). In this work it is investigated the effect of microstructure on the fracture behaviour of a zirconia-based ceramic with 2.8% molar of $Y_2O_3$.

From a microstructural viewpoint, zirconia materials containing yttria may be found as tetragonal zirconia polycrystals (Y-TZP) or partially stabilised zirconia (Y-PSZ). The former corresponds to stabiliser content between 2-3 mol% and is characterised by a very fine grain (0.2-2 μm) microstructure, high fracture strength (aprox. 1000 MPa), and rather moderate fracture toughness (4-6 MPa $m^{1/2}$) (2). On the

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other hand, the latter is obtained through additions of yttria up to 3-6 mol%, and may be described as consisting of tetragonal precipitates embedded within large cubic grains. PSZ generally possesses lower fracture strength than TZP, although associated with higher fracture toughness values (more than 10 MPa $m^{1/2}$) (2).

From a fracture strength viewpoint, it is well-known that failure of ceramic components is controlled by propagation of pre-existent flaws, originated in manufacture and processing steps. Thus, a relatively wide scatter is often found when testing ceramics. A statistical function that is commonly used for describing fracture strength results of brittle materials is the Weibull distribution function (3):

$$P_f = 1 - \exp\left[ - \int_V \left( \frac{\sigma - \sigma_{f0}}{\sigma_0} \right)^m dV \right] \quad (1)$$

where $P_f$ is the fracture probability, $\sigma$ the applied stress, $\sigma_{f0}$ the strength below which $P_f$ is zero, $\sigma_0$ is referred to as characteristic strength and it is defined as the strength which gives $P_f$ equal to 63.2%, and m is the Weibull modulus. Such m value is related to the scatter of results. The larger the m values are, the narrower the strength distribution is; i.e. the material is more flaw tolerant and more reliable.

As said above, the aim of this study is to determine microstructural effects on the fracture behaviour of a Y-TZP stabilised with 2.8% molar of $Y_2O_3$. In doing so, different microstructural features are attained through thermal treatment of the as-received material. Fracture characteristics are evaluated using statistical parameters. The obtained results are finally discussed with respect to the induced microstructural changes.

## EXPERIMENTAL PROCEDURE

### Microstructure characterisation

The starting material was in the shape of polished round bars of 8 mm diameter. Microstructural changes were promoted in as-received material (referred to as AR) via heat treatment at 1650 °C for two hours (material referred to as HT).

Grain size was characterised through the equivalent circular diameter. Measurements were taken from the polished and thermally etched sample surface (1400 °C, 1 hour) using scanning electron microscopy (SEM). Further, some samples were prepared for transmission electron microscopy (TEM) to obtain the crystallographic symmetry of particular grains.

### Mechanical tests

Bending strength was determined in cylindrical specimens of 8 mm diameter in three point bending with a span of 40 mm, using a total number of 30 specimens for each material condition. The obtained results were fitted to a Weibull distribution. Fracture

toughness was evaluated with different techniques: (a) measuring indentation crack length (IM), (b) using the fracture strength of indented specimens (ISB) and (c) from indented specimens without residual stresses (SCF) (4). Fractographic observations were performed by SEM.

## RESULTS

### Microstructure

The heat treatments produced a clear increase in grain size. In AR material microstructure was homogeneous with very fine grains ($0.30 \pm 0.01$ μm). Meanwhile, in HT material the microstructure was more heterogeneous. It was formed by quite large grains ($2.71 \pm 0.18$ μm), surrounded by smaller ones ($0.91 \pm 0.01$ μm). TEM studies, particularly in terms of electron diffraction, showed that grain symmetry was mainly tetragonal for AR. In HT material, small grains were either tetragonal and monoclinic depending on its size, whereas the large ones presented a tweed contrast. The latter observation is due to the presence of very small tetragonal precipitates within the large grains, formed by diffusional decomposition of the cubic matrix [5,6]. Moreover, preliminary studies with X-ray diffraction pointed out that only tetragonal transformable phase is found, with no presence of non-transformable t' phase. Hence, the microstructure of HT material may be considered as a mixture of Y-TZP and Y-PSZ.

### Fracture toughness and fracture strength

The obtained values of fracture toughness ($K_{IC}$) are shown in table 1, where it may be appreciated a significant increase in this parameter for HT material

TABLE 1- $K_{IC}$ values for each material obtained with different techniques.

| Material | IM | ISB | SCF |
|---|---|---|---|
| **AR** | $3.3 \pm 0.2$ | $5.5 \pm 0.1$ | $5.0 \pm 0.1$ |
| **HT** | $5.6 \pm 0.5$ | $7.4 \pm 0.4$ | $8.3 \pm 0.2$ |

The fracture strength values are shown in table 2. The experimentally obtained values hardly change, despite of the different grain and flaw sizes. Results were fitted to Weibull functions of two and three parameters. The obtained m and $\sigma_0$ values for the conducted fittings are shown in table 2. For AR material, two-parameter Weibull function produced a good fitting, whereas in HT a three-parameter function was the best choice. The corresponding fitting parameters for the latter are also included in table 2.

TABLE 2- Fracture strength and Weibull parameters of the studied materials.

| | | σ (MPa) | m | $\sigma_0$ (MPa) | $\sigma_{f0}$ (MPa) |
|---|---|---|---|---|---|
| **Two-parameter** | **AR** | $1068 \pm 93$ | 13 | 1107 | - |
| | **HT** | $1034 \pm 66$ | 20 | 1060 | - |
| **Three-parameter** | **HT** | $1034 \pm 66$ | 1.6 | 124 | 920 |

## DISCUSSION

### Microstructural effects on fracture behaviour

Heat treatment increased zirconia grain size, which is a well known controlling parameter of the stress induced phase transformation. Tetragonal grain size has a large influence on the stress required to nucleate transformation (7); thus, a critical grain size which offers the highest degree of toughening transformation may be defined. For the results here presented, for fine-grained microstructures, the critical stress for phase transformation may be described as large. This gives small transformation zones, which yields moderate fracture toughness values. On the contrary, in coarser microstructures transformation seems to be activated at lower stresses, which offers larger transformation zones and higher fracture toughness. This may explain the increase in fracture toughness values in HT material. Moreover, in Y-PSZ tetragonal precipitates within the cubic matrix effectively enhance mechanical properties (6). Thus, the observed fracture toughness increase in HT material may be rationalised in terms of both the larger mean grain size and the presence of fine tetragonal precipitates in large cubic grains.

Fracture strength did not change in HT material because the higher fracture toughness compensated the larger flaw sizes formed during microstructure coarsening. In order to support this statement, the fracture controlling defects were measured in SEM for AR and HT materials, and its ratio was compared to that of grain sizes (table 3). It can be seen that the size of fracture controlling defects are increased by a factor of 5 in the HT material which reasonably accounts for the close fracture strength of both materials. In addition, this increase in flaw size is similar to the increase in grain size.

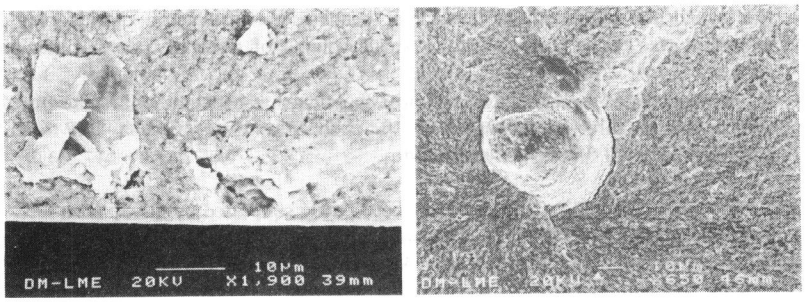
TABLE 3- Ratios between critical defects size and grain size for each material.

| $a_c$ (μm)AR | $a_c$ (μm) HT | $\frac{a_c\ (AR)}{a_c\ (HT)}$ | $\frac{d\ (AR)}{d\ (HT)}$ |
|---|---|---|---|
| $8 \pm 3$ | $40 \pm 8$ | $0.20 \pm 0.07$ | $0.30 \pm 0.15$ |

### Statistical functions

Experimental results were plotted following Weibull distribution functions and showed a clear downward curvature. This is specially true for the HT material. This behaviour could be explained by the presence of two defect populations, the specimen geometry, or by the presence of an R-curve. The former is rejected because the porous distribution was determined, and showed a single defect distribution. On the other hand, in cylindrical specimens the volume of material at a given distance from specimen surface is lower than in prismatic ones. So that, the probability of finding a large defect, that could lead to fracture, near the surface is considerably smaller in cylindrical bars than in prismatic bars. Hence, three point bending in cylindrical bars results in a relatively large number of specimens that failed due to bulk defects. To point out this aspect, fractographic observations were taken from the fracture surfaces. The results showed that samples with lower fracture strength values generally presented superficial controlling defects, whereas volumetric defects were found to control fracture in higher strength

specimens. In figure 1 some fracture micrographs corresponding to volumetric and superficial flaws in AR, are shown. Once the critical defect is located, the real fracture strength of the sample may then be estimated. Using these corrected values, scatter clearly diminished in both materials with respect to experimental strength (figure 2). In HT material these corrected results still presented a markedly curvature. This may be explained by the presence of a more pronounced R-curve behaviour (8,9). Although R-curve measurements were not conducted in this investigations, results from the literature suggest than the heterogeneous microstructure of HT material should induce a more pronounced R-curve behaviour.




(a) (b)

FIGURE 1.- Fractographies of AR material showing: (a) superficial defect, and (b) volumetric defect.

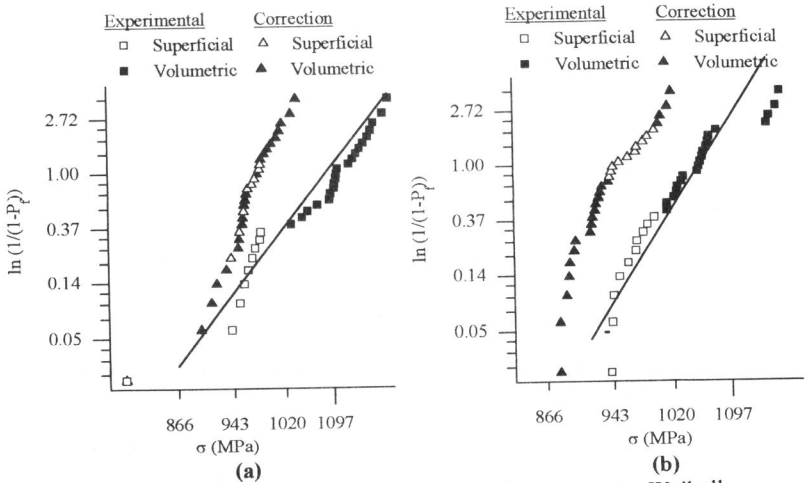



FIGURE 2.- Fitting fracture strength values to a two-parameter Weibull distribution function for: (a) AR and (b) HT. For each data set the location of the defect, i.e. surface or volume, it is indicated.

## CONCLUSIONS

On the basis of microstructural characterisation, fracture toughness and fracture strength distributions for a zirconia ceramic containing 2.8% mol $Y_2O_3$, the following conclusions can be drawn:

- Heat treatments at 1650 °C for two hours produced a significant increase in fracture toughness, with respect to fine-grained Y-TZP, without fracture strength degradation.
- Weibull modulus, as a consequence of the coarser microstructure of heat-treated material, clearly increases in the heat-treated material.
- For cylindrical specimens in three-point bending, the volumetric defects play an important role, since there is only a small volume of material near the specimen surface.
- In the heat-treated material, the statistical function that better describes the fracture behaviour is the three-parameter Weibull function. Meanwhile, in fine Y-TZP the two-parameter Weibull function may be used.

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