Cyclic fatigue of zirconia bioceramics

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Introduction

When ceramic is used for implant material such as artificial joints or dental abutment, it undergo loading for fairly long period [1].
There is a strong demand for generation of design-relevant fatigue data, which are required for many of the projected applications of structural ceramics. On the other hand, knowledge of fatigue in ceramics is insufficient so far [2-3].
In this paper, the fatigue behavior of tetragonal zirconia polycrystals (TZP) with 3mol.% of Y_2O_3 stabilized (3Y-TZP) under four-point bending in load cyclic conditions was investigated. Mechanical properties of hardness, fracture toughness and modulus of rupture were also evaluated.

Experimental procedure

High-purity Y-TZP (ZrO_2-3mol.%Y_2O_3 –Tosoh 3YSB-Japan) was used as starting-powder. The ZrO_2-powder was uniaxial cold-pressed into plates, under 80MPa. The compacts were sintered in air, at 1600^0C-2h (10^0C/min). Bulk density was measured by the Archimedes' method. The crystalline phase content was determined X-ray diffractometry (XRD). Monoclinic phase fraction was calculated using the *Garvie* [4] *and* Toraya [5] method. Polished-thermal etched surface and fractured surface of the mechanically tested specimens, were examined by scanning electron microscopy (SEM).
Microhardness and Fracture toughness, K_{IC}, were determined using Vickers Indentation method, using the relation proposed by Evans and Charles [6].
The Modulus of Rupture (MOR) and Fatigue were determined by 4 point bending testing, with outer and inner spans of 40 and 20mm, respectively, and speed of 0.5 mm/s at room temperature, using a servo-hydraulic testing machine. Samples with size of 3 x 4 x 45mm were machined from the as-received plate and polished to 1μm diamond surface finish.
The cyclic fatigue was studied under a sinusoidal stress wave within a frequency of 25Hz and stress ratio ($R=\sigma_{min}/\sigma_{max}$) of 0.1. The number of specimens used in fatigue tests was of 13 and 23 samples, under high stresses, and in the low stress levels 3 specimens tested. The tests were interrupted when the survival samples reached a number of cycles between 2 and 5 x 10^6 cycles.

Results and discussion

In XRD patterns of ZrO_2 was observed the starting powder, as received, presents 15% of residual monoclinic ZrO_2 phase. After sintering, the processing conditions allowed a total stabilizing of tetragonal ZrO_2 phase by incorporation of Y_2O_3 in the structure. The XRD patterns of the sample fracture surface undergo at bending strength test shows that there was a considerable monoclinic level, around 10 %, due at tensile stress which the grains of this surface were submitted. It is known that the application of stresses under tetragonal ZrO_2 grains can be to start the martensitic transformation [7].

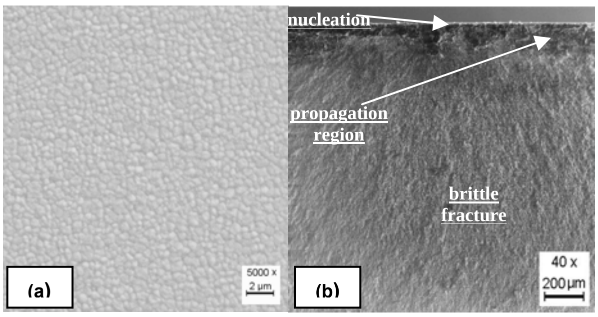

Figure 1 – Micrograph of the ZrO_2 samples: a) polished, b) fracture surface.

Figure 1 presents micrograph of the polished/etched ZrO_2 surface and fracture surface of the mechanically tested sample.
In the Fig.1(a) ,it can be observed the presence of refined microstructure with

equiaxial grains of mean size around 0.5 µm. In the Figure 1(b) is seen a typical fracture surface which show clearly that the initial crack nucleation and propagation region is located in the micrograph superior area, corresponding at tension surface submitted to bending test. Table 1 presents the relative density and mechanical properties.

Table 1 – Results of the sintered samples.

R.D. (%)	HV (GPa)	K_{IC} ($MPam^{1/2}$)	MOR (MPa)
99.7±0.2	13.5±0.2	8.15± 0.25	880±35

High densification associated with refined equiaxial grains morphology, Fig.1(a) were obtained. This typical microstructure indicated predominance total of tetragonal phase, justifying the high values of fracture toughness and bending strength presented in Tab. 1. Cracks deflections and toughening by phase transformation mechanisms are the main mechanisms actuating to improvement of mechanical properties of the material. The T-M transformation, allowed that the fracture strength reached the values elevated around 900MPa. The aspect of Fig. 2(b) indicate the event of brittle fracture where the crack start the propagation on the superior region of micrograph, which correspond to polished surface and subject to tension during the test. Furthermore, the hardness values were about 13,5 GPa consistent with literature data [8].

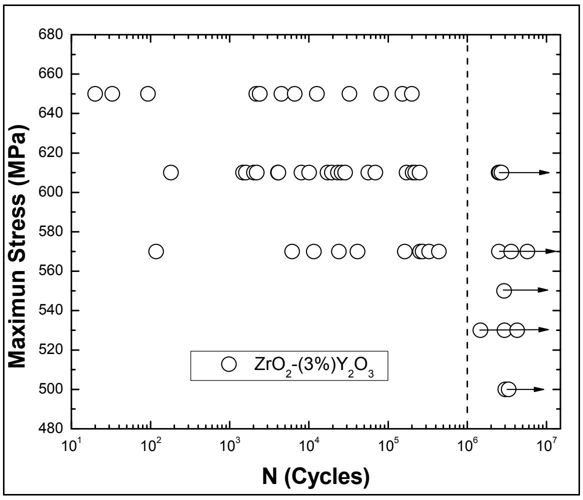

Figure 2 – Cyclic fatigue tests results of the ZrO_2 samples: σ_{max} x N curves.

The results of the cyclic fatigue are shown in Fig. 2. The specimens which did not fracture are marked by an arrow symbol. The five stress levels were selected in relation to the initial strength. It is found that fatigue strength limit over $5x10^6$ cycles is around of 550 MPa.

The Fig. 2 revealed that in stress above of 550 MPa, the most of specimens tested fail in the range of $10^3<N_f<2x10^6$ cycles. Samples that failed with low cycle ($N_f<10^3$ cycles), have a trend to be more representative while as bigger stresses are applied. Against, the stress reduction leads to an increasing significant of samples without failure.

The samples rupture in fatigue begin in the polished surface of tensile side tested, occurs in brittle mode and is a function of flaw size initiating fracture critical in the tension surface. This flaw, in turn, to initiate the propagation it suffers compression due to t-m transformation with associated volumetric expansion increasing the crack propagation strength until reach a critical value, above which, propagates the crack in catastrophic mode up to material fracture. The mechanism of the stress induced t-m transformation also was showed by Grathwohl [15].

Cyclic testing of Y-TZP provides interesting results concerning fatigue behavior, threshold phenomena, and the strengthening effect of this transformation-toughened ceramic. The range of fatigue is not very limited and it is clear that this fine-grained ceramic is particularly prone to cyclic fatigue.

Conclusions

Dense 3Y-TZP were obtained after sintering, and present HV, K_{IC} and MOR of 13.5 GPa, 8.15 $MPa.m^{1/2}$ and 880 MPa, respectively. In this ceramic submitted to the 4 point bending cyclic load, the propensity for cyclic fatigue is very strong and presents a considerable range of loading conditions, where cyclic fatigue can be detected. The results obtained indicates that the fatigue strength limit over $5x10^6$ cycles stress is about 550 MPa or around 63% static bending strengthl.

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