

**Performance of ZrO<sub>2</sub>-Bioglass Dental Ceramics Under Cyclic Fatigue Loading**

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**Abstract**

The cyclic fatigue life of 3mol.%Y<sub>2</sub>O<sub>3</sub>-stabilized ZrO<sub>2</sub> (3Y-TZP) ceramics doped with bioactive glass has been investigated. Pre-sintered 3Y-TZP-bioglass samples were sintered at 1300°C for 120 minutes. Sintered samples were characterized by relative density, XRD and SEM analysis. Hardness and fracture toughness were determined using Vicker's indentation method, and Modulus of Rupture was determined by four-point bending testing. Furthermore, the cyclic fatigue tests were realized under frequency of 25 Hz and stress ratio, R, of 0.1. Highly dense tetragonal ZrO<sub>2</sub> samples were obtained and presented hardness, fracture toughness and bending strength of 11.3GPa, 6.1MPa.m<sup>1/2</sup> and 320MPa, respectively. The increasing of stress level leads to decreasing of the number of cycles and the number of run-out specimens. The stress induced tetragonal-monoclinic (t-m)-ZrO<sub>2</sub> transformation, observed by X-Ray diffraction, contributes to the increasing of the fatigue life. Samples 3Y-TZP presents clearly a range of loading conditions where cyclic fatigue can be detected.

**Key words:**

Ceramics  
 Sintering  
 ZrO<sub>2</sub>  
 Mechanical  
 Properties  
 Fatigue  
 Characterization

**Palavras-chaves:**

Cerâmica  
 Sinterização  
 ZrO<sub>2</sub>  
 Propriedades  
 Mecânicas  
 Fadiga  
 Caracterizações

**Resumo**

A vida em fadiga cíclica de ZrO<sub>2</sub> estabilizada com 3%mol.Y<sub>2</sub>O<sub>3</sub> (3Y-TZP) dopada com vidro bioativo foi investigada. Amostras pré-sinterizadas 3Y-TZP-biovidro foram sinterizadas a 1300°C por 120 minutos. As amostras sinterizadas foram caracterizadas por densidade relativa, difração de raios X e MEV. Os valores de dureza e tenacidade à fratura foram determinados usando método de indentação Vickers e o módulo de ruptura por flexão foi realizado em ensaio de flexão em 4 pontos. Além disso, testes de fadiga cíclica foram realizados sob frequência de 25Hz e razão de tensão de 0,1. Amostras altamente densas de ZrO<sub>2</sub> tetragonal foram obtidas e apresentaram dureza, tenacidade à fratura de 11.3GPa, 6.1MPa.m<sup>1/2</sup> e 320MPa, respectivamente. O aumento do nível de tensão leva a diminuição do número de ciclos e o número de amostras com vida infinita. A tenacificação induzida por transformação de fase tetragonal-monoclinica (t-m), observada por difração de raios X, contribui para o aumento da vida em fadiga. Amostras de 3Y-TZP apresentaram claramente uma faixa de condições de carregamento onde fadiga cíclica pode ser detectada.

**1. Introduction**

Metal alloys have been currently used as crown or abutment materials in osseointegrated implant fixtures like dental prostheses. The material ought to be

compatible and must not promote plaque adherence, besides providing enough strength to endure and transmit occlusal forces to the implant and supporting the bone. Another

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significant factor is aesthetics, in order to replicate the visible aspect of the natural tooth. All these properties are necessary to fulfill intra-oral applications (ARDLIN *et al* -2002, GUAZATTO *et al*- 2004, KIM *et al*-2000). An important improvement has been possible by the use of all-ceramic systems.

All-ceramic dental restorations are attractive to the dental community because they provide higher strength and abrasion resistance, better biocompatibility and aesthetics, when compared with metal and resin restorations (GUAZATTO *et al*- 2004, SHENG *et al.*, 2004). On the other hand, the applications of all-ceramic crowns and bridges have been limited by their brittle behavior, long processing time and machinability performance (TANG *et al*-1999, STEVANS-1986). The most widely used ceramic materials are alumina ( $\text{Al}_2\text{O}_3$ ) and zirconia ( $\text{ZrO}_2$ ), due to their excellent biocompatibility. The main advantage of  $\text{Al}_2\text{O}_3$  is its high hardness and wears resistance, while  $\text{ZrO}_2$  exhibits higher mechanical strength and fracture toughness (BASU *et al*-1996, JANNEY *et al*-1998).

Cyclic fatigue of ceramics recently became a highly attractive research field for material scientists. There is a strong demand to generate design-relevant fatigue data which are required for many of the projected applications of structural ceramics as dental materials. On the other hand, knowledge of fatigue in ceramics is insufficient so far and information about the correlation between microstructural parameters and fatigue properties is still missing for most ceramic systems. Besides this lack of understanding a number of fundamental questions still have not been answered unambiguously for many of the most important ceramics (GRATHWOHL *et al.*, 1991).

The purpose of this study is to evaluate the mechanical properties and cyclic fatigue life of the  $\text{ZrO}_2$ -bioglass ceramics sintered at low temperatures, correlating these properties with densification and fraction of monoclinic- $\text{ZrO}_2$  phase.

## 2. Experimental Procedure

Pre-sintered  $\text{ZrO}$ -bioglass dental ceramic (ProtMat Materiais Avançados®) blocks presenting 12% of monoclinic phase

content, were cutted and sintered at  $1300^\circ\text{C}$ , in a  $\text{MoSi}_2$  furnace for 2h, with heating and cooling rate of  $10^\circ\text{C}/\text{min}$ .

Density of the sintered samples was obtained by Archimedes' method. The crystalline phases were determined by X-ray diffraction (XRD) using  $\text{Cu-K}\alpha$  radiation in the  $2\theta$  range of  $20^\circ$  to  $80^\circ$ , with a step width of  $0.05^\circ$  and 3s of exposure time per step. The monoclinic- $\text{ZrO}_2$  phase fraction was calculated using the Garvie and Nicholson method (1972).

Microstructural aspects of the sintered samples were examined by scanning electron microscopy (SEM), using LEO-1450VP microscope.

Microhardness and Fracture toughness, KIC, were determined using a Vickers Indentation method under 2000gf for 30s (EVANS & CHARLES-1976). Polished rectangular bars of  $4\times 3\times 45\text{mm}^3$  were obtained according ASTM C-1116-94. The tests were conducted using a four-point bending device with outer and inner spans of 40 and 20mm, respectively, by 0.5mm/s at room temperature using an MTS 310 Universal Test Machine.

Cyclic fatigue tests were carried out by four-point bending loading in air at room temperature, with a relative humidity near 60%. The specimen dimensions and the testing machine were the same as employed in the bending strength tests. The cyclic fatigue was studied under a sinusoidal stress wave form with a frequency of 25Hz and a constant stress ratio (R) between the minimum stress and maximum stress of 0.1. The number of specimens used in fatigue tests varied between 12 and 23 samples by stress level. The tests were interrupted when the surviving samples reached a number of stress cycles between 2 and  $5\times 10^6$  cycles.

## 3. Results and Discussion

Figure 1 shows the representative X-ray diffraction patterns and SEM micrograph of the sintered samples.

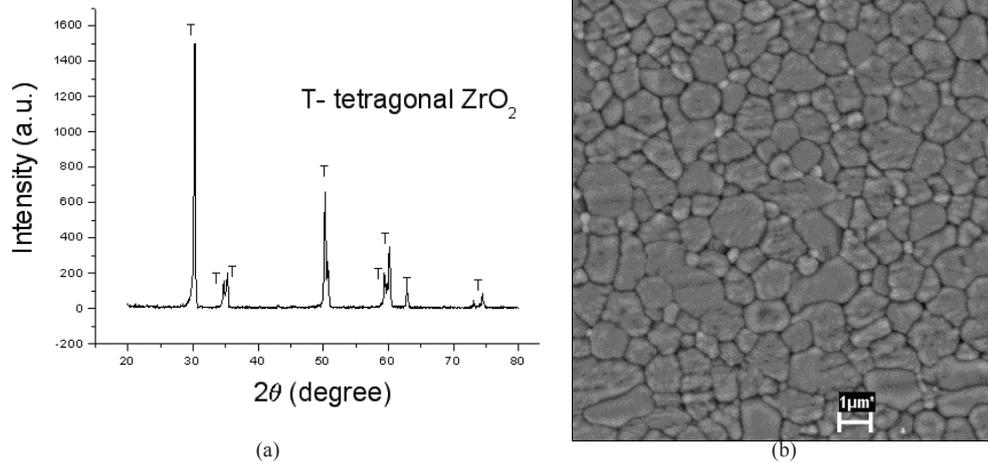


Figure 1 – XRD patterns (a) and SEM micrograph (b) of the samples sintered at 1300°C.

In Fig 1-a, only tetragonal  $ZrO_2$  phase was detected, indicating that all residual monoclinic phase was converted to tetragonal phase during sintering. Crystalline intergranular phase, starting of the glass were not detected. These ceramics presented high relative density, near to 98.5% of theoretical

density, indicating a low porosity content after sintering. The SEM results presented in Fig. 1-β show typical  $ZrO_2$  microstructures with grain size compatible with the starting particle size and sintering temperature used. Table 1 presents results of mechanical properties of sintered ceramics.

Table 1. Mechanical properties of the sintered samples.

Composition	Hardness, $HV_{(2000gf)}$ [GPa]	$K_{IC}$ [ $MPa \cdot m^{1/2}$ ]	Bending Strength, $\sigma_{F-(4p)}$ [MPa]
$ZrO_2$ -Bioglass (1300°C)	11.3±0.7	6.1±0.4	320±55

The results indicate that a hard, strength and toughened ceramic was obtained with hardness and fracture toughness of 1150HV and  $6.1 MPa \cdot m^{1/2}$ , respectively. These results are correlated with physical characteristics of the materials, with the best relative density and low martensitic transformation content previously showed.

Cyclic fatigue life. The results of cyclic fatigue of the  $ZrO_2$ -glass dental ceramics are shown in the Fig. 2. The specimens which did not fracture are marked by an arrow symbol. The five stress levels were selected in relation to the static bending strength (320MPa).

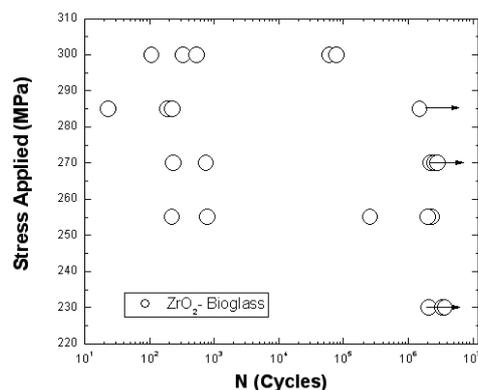


Figure 2 – Cyclic fatigue results of the  $ZrO_2$ -Bioglass samples:  $\sigma_{max} \times N$  curves

It is found that fatigue strength limit over  $2 \times 10^6$  cycles is around of 240MPa. These tests revealed that in stress higher than 240MPa, the most of specimens tested fail in the range of  $10^3 < N_f < 2 \times 10^6$  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. On the other side, the stress reduction leads to an increasing of samples without failure.

#### 4. Conclusions

The Y-TZP-Bioglass ceramics sintered at 1300°C showed good mechanical properties, presenting strength of 320MPa, hardness of 1150HV and fracture toughness of  $6.1 MPa \cdot m^{1/2}$ . These results are well established with the high relative density, low monoclinic- $ZrO_2$  phase percentage, presented in samples after sintering. Fatigue tests by four-point bending were conducted in order to obtain the  $\sigma \times N$  curve for the material. The experimental results clearly indicate that 3Y-TZP-Bioglass ceramic material suffered cyclic fatigue fracture. It was found that the fatigue strength limit is around

240MPa, which representing values around of 75% of static bending strength and is similar to the strength-mastigatory loads. The relatively-low sintering temperature used in this work, 1300°C, associated with good mechanical properties, can be attractive for development of ZrO<sub>2</sub>-Bioglass components to be used in dental implant parts.

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