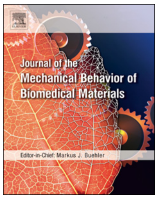
Journal of the Mechanical Behavior of Biomedical Materials 88 (2018) 305–312

Contents lists available at ScienceDirect

Journal of the Mechanical Behavior of Biomedical Materials

journal homepage: www.elsevier.com/locate/jmbbm



# Fatigue behavior and surface characterization of a Y-TZP after laboratory grinding and regeneration firing

Gabriela Scatimburgo Polli[a], Gabriel Rodrigues Hatanaka[a], Filipe de Oliveira Abi-Rached[a], Márcio de Souza Góes[b], José Mauricio dos Santos Nunes Reis[a,*]

[a] Department of Dental Materials and Prosthodontics, São Paulo State University (UNESP), School of Dentistry, Araraquara, Sao Paulo, Brazil
[b] Interdisciplinary Centre for Natural Sciences, UNILA - Federal University of Latin American Integration, Foz do Iguaçu, PR, Brazil

## ARTICLE INFO

*Keywords:*
Zirconia
Fatigue limit
Staircase method
Grinding
Regeneration firing

## ABSTRACT

This study evaluated the effect of grinding and regeneration firing on the flexural fatigue limit and surface characterization of Lava™ Y-TZP ceramic. Forty bar-shaped specimens with 20 × 4.0 × 1.2 mm constituted the as-sintered group (*AS* = control group), and 80 specimens with 20 × 4.0 × 1.5 mm were ground with cylindrical laboratory stone under water-cooling (*WG*) or in a dry condition (*G*) to reach 1.2 mm in thickness. Half of specimens were submitted to regeneration firing (1000 °C, 30 min), forming the groups *AS/R*, *WG/R* and *G/R*. Fatigue limit (500,000 cycles, 10 Hz) was determined by staircase method in a 4-point flexural fixture. Data were analyzed by 2-way ANOVA and Tukey HSD tests ($\alpha = 0.05$). The surface topography ($n = 3$) and fracture area ($n = 3$) were evaluated by SEM. Samples were also analyzed by Rietveld refinement from X-ray diffraction data. ANOVA revealed significant differences ($P < .001$) for grinding protocol, regeneration firing and their interaction. In the groups not submitted to regeneration firing, the mean flexural fatigue limit of *WG* was higher ($P < .05$) than that of *G* and *AS*, with no statistical difference between each other ($P > .05$). After regeneration firing the inequality *WG* > *AS* > *G* ($P < .05$) was observed. The regeneration firing increased the fatigue limit of *AS* group and decreased those of *G* and *WG* groups ($P < .05$). Grinding protocols created evident grooves on zirconia surface. Failures initiated on tensile side of all specimens. The percentages (wt%) of monoclinic phase before cyclic loading were: *AS* (7.4), *AS/R* (6.5), *G* (2.8), *G/R* (0.0), *WG* (4.4), *WG/R* (0.0); and after cyclic loading: *AS* (8.6), *AS/R* (1.2), *G* (2.4), *G/R* (5.7), *WG* (6.3), *WG/R* (0.0). Wet grinding did not compromise the fatigue limit of zirconia, increasing its mechanical strength. Regeneration firing reduced the fatigue limit of ground samples, despite reducing the amount of monoclinic phase in all experimental conditions.

## 1. Introduction

Due to the mechanical (Lee et al., 2016), optical (Magne et al., 2010) and biocompatibility properties (Lee et al., 2016; Josset et al., 1999) of yttria-partially-stabilized tetragonal zirconia (Y-TZP), it is widely used in Dentistry for the fabrication of dental fixed or implant supported prostheses (Kohal et al., 2011, 2008; Molin and Karlsson, 2008; Nakamura et al., 2010; Pilathadka et al., 2007; Sailer et al., 2007; Vagkopoulou et al., 2009), as these properties make it suitable for resisting the high stresses produced on single- or multi-unit prostheses (Studart et al., 2007b; Ryan et al., 2016). Fatigue studies (Studart et al., 2007a; Teixeira et al., 2007; Belli et al., 2014) have verified that Y-TZP was able to withstanding critical mechanical loading conditions. Nakamura et al. (2010) observed that zirconia is a suitable material for using as implant abutments; however, the authors affirmed that after mechanical and thermal aging, its mechanical properties might be compromised by progressive $t \rightarrow m$ phase transformation.

A concern is the influence of grinding with mounted stones or diamond burs on the mechanical properties of sintered Y-TZP (Lee et al., 2016; Ryan et al., 2016; Pereira et al., 2016), in order to obtain adequate interocclusal space for the veneering ceramic, framework marginal fit, suitable emergence profile and satisfactory axial contour of dental implant abutments. Although the CAD/CAM technology allows precise ceramic dental prostheses to be obtained, adjustments on frameworks and zirconia dental implant abutments are routine procedures in clinical and laboratory practice (Pereira et al., 2016; Adatia et al., 2009; Kim et al., 2010; Lopes et al., 2007; Sato et al., 2008; Wang et al., 2008; Zucuni et al., 2017; Candido et al., 2017).

According to Swain (1985), the flexural strength can be increased or diminished as a result of grinding/finishing and is related to the volume

---

* Corresponding author.
*E-mail address:* reis@foar.unesp.br (J.M.d.S.N. Reis).

https://doi.org/10.1016/j.jmbbm.2018.08.019
Received 28 June 2018; Received in revised form 7 August 2018; Accepted 19 August 2018

percentage of the zirconia phase transformation. It is known that procedures of finishing, polishing, airborne-particle abrasion (Aboushelib and Wang, 2010; Kosmac et al., 2000) and/or heat treatment (Denry and Holloway, 2006; Guazzato et al., 2005) may change its mechanical properties. The *t→m* phase transformation and the formation of superficial compressive stresses on zirconia structure may enhance the toughness of the material, improving its mechanical properties (Lee et al., 2016; Pereira et al., 2016; Zucuni et al., 2017; Garvie et al., 1975). In the study of Adatia et al. (2009) the preparation of zirconia abutments under copious water-cooling did not compromise the fracture strength of implant-abutment sets. Under cyclic loading, ceramic abutments present lower fracture resistance values (Nakamura et al., 2010; Gehrke et al., 2006; Yildirim et al., 2003) in comparison with monotonic tests (Yildirim et al., 2003); however, these resistance values are higher than the masticatory load (90–370 N) developed in the anterior region of the oral cavity. In spite of this, studies (Lee et al., 2016; Kim et al., 2010; Aboushelib and Wang, 2010; Scherrer et al., 2006) have verified deleterious action on the mechanical properties of zirconia after different surface treatments.

Grinding and finishing procedures may result in surface flaws, whose origin can be identified by means of fractographic analysis and is the possible reason for triggering the crack propagation of the ceramic material (Scherrer et al., 2006). Despite this, the effects of finishing/polishing treatments on the mechanical properties of zirconia have been approached in a conflicting manner in the literature (Wang et al., 2008; Kosmac et al., 2000, 1999; Curtis et al., 2006; Qeblawi et al., 2010). On the one hand, the t→m phase transformation causes volume increase within transformed grains creating compressive stresses (Denry and Kelly, 2008), which enhances the material toughness, leading to an increased mechanical strength (Kosmac et al., 1999; Karakoca and Yilmaz, 2009). Conversely, there may be harm to the mechanical properties when the length of microcracks exceeds the compressive layer, allowing the propagation of microcrack within the bulk of the material (Lee et al., 2016; Kim et al., 2010; Aboushelib and Wang, 2010; Guess et al., 2010b). According to Kosmac et al. (1999), an excessive increase in temperature from a more aggressive grinding protocol may induce *m→t* reverse phase transformation, which could also be responsible for the reduction in strength of the material due to the relief of compressive stresses. In the same way, Denry and Holloway (2006), by means of a microstructural and crystallographic study, affirmed that mechanical grinding might have a negative impact on the reliability of Y-TZP. These authors speculated about the possibility of using post-wear heat treatment as a beneficial means of avoiding damage on the zirconia after small adjustments. Sato et al. (2008) verified that the monoclinic phase concentration in partially stabilized zirconia after superficial grinding was reduced with heat-treatments, which is helpful in understanding the maintenance of the material mechanical properties.

Although there are many studies (Wang et al., 2008; Denry and Holloway, 2006; Qeblawi et al., 2010; Işerı et al., 2010; Luthardt et al., 2002; Scherrer et al., 2011) evaluating the mechanical properties of the zirconia, few of them have taken into account the influence of grinding with diamond stone followed by heat-treatments on the zirconia properties (Lee et al., 2016; Candido et al., 2017). To the best of the authors' knowledge, only two studies (Lee et al., 2016; Candido et al., 2017) were found in the literature regarding grinding with diamond stones and its influence on some of the characterization properties of Y-TZP.

The staircase method has been proved to be adequate for measuring the mechanical cyclic resistance of a material (Belli et al., 2014; Yamamoto and Takahashi, 1995; Amaral et al., 2016), and it is, therefore, widely used for defining the fatigue limit of several dental materials (Lopes et al., 2007; Frankenberger et al., 2003; Guess et al., 2010a; Mirmohammadi et al., 2010; Vergani et al., 2010). The study of the fatigue limit of a material allows knowing how resistant a material is under infinite cyclic loading without being fractured. Among fatigue tests, staircase method uses a few number of specimens and statistical inferences to calculate the fatigue limit within reasonable time constraints.

The aim of this study was to evaluate the effect of diamond stone grinding and regeneration firing on the flexural fatigue limit and surface characterization (surface topography and phase transformation) of a Y-TZP. The null hypothesis was that those procedures would not influence the fatigue behavior, surface topography and phase transformation of the Y-TZP.

## 2. Materials and methods

### *2.1. Preparation of Y-TZP specimens*

Zirconia bars of 25 × 5.0 × 1.5 mm (n = 40) and 25 × 5.0 × 1.9 mm (n = 80) were obtained by cutting pre-sintered milling blocks (Lava™; 3 M ESPE AG, LOT 1433000610, Seefeld, Baviera, Germany) using a saw (Isomet 1000; Buehler Ltd, Lake Bluff, IL, USA) with a water-cooled diamond-wafering blade (No. 11–4276; Buehler Ltd, Lake Bluff, IL, USA). The green state bar edges were finished using an abrasive rubber point (Exa Cerapol 0361HP; Edenta AG, Au, SG, Switzerland) with a low speed handpiece. After sintering (Lava Furnace 200; Dekema Dental-Keramiköfen GmbH, Freilassing, Baviera, Germany) in accordance with the manufacturer's instructions, the final dimensions of the bars were 20 × 4.0 × 1.2 mm (n = 40) and 20 × 4.0 × 1.5 mm (n = 80).

### *2.2. Grinding procedures*

The as-sintered group (*AS*) was composed of twenty thinner (1.2 mm) specimens, while the thicker ones (1.5 mm) were ground, either under water-cooling (*WG*) or not (*G*). The water-cooling was manually done by hypodermic syringe with 20 mL of distilled water per specimen. These specimens were placed in a custom-built apparatus with an opening measuring 20.05 × 4.05 × 1.20 mm to guide the ground of excess zirconia (0.3 mm) using an electric micromotor (NSK Ultimate XL; NSK Nakanishi Inc., Kanuma, Tochigi, Japan) at 10,000 rpm, with a cylindrical diamond stone (MCE 133 104, Master Ceram, Eurodental Comercial Importadora Ltda, São Paulo, SP, Brazil). A holding arm moved the specimen horizontally by the intimate contact between stone and zirconia surface, perpendicular to each other (Fig. 1). A digital caliper was used to verify the final thickness of 1.2 mm in four points of all zirconia bars (20 ×4.0 ×1.2 mm - ISO 6872, 1997).

### *2.3. Regeneration firing*

Half of as-sintered, *WG* and *G* specimens were submitted to regeneration firing (*AS/R*, *WG/R* and *G/R* groups; n = 20) at 1000 °C for 30 min (Fonseca et al., 2014) in a conventional porcelain oven (Aluminipress; EDG Equipamentos e Controles Ltda, São Carlos, SP, Brazil), following Lava™ manufacturer's recommendation. Specimens were

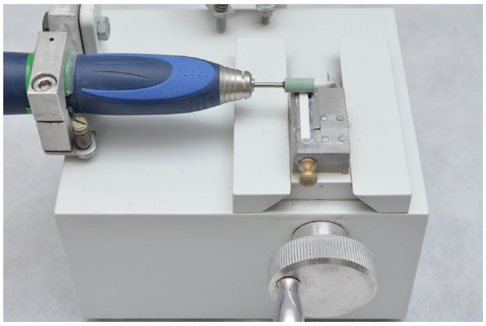


**Fig. 1.** Device for standardizing grinding.