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

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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

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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.

cooled to room temperature by opening the oven door.

2.4. Mechanical cycling test (Staircase fatigue limit test)

For determining the entry fatigue stress level, a monotonic test was performed before the cyclic fatigue experiment. For the monotonic test, 12 additional specimens (20 × 4.0 × 1.2 mm) from each group were tested in a servo-hydraulic mechanical testing machine (810 Material Test System; MTS Systems Corp, Eden Prairie, MN, USA) with a 10 kN load cell, at a crosshead speed of 0.5 mm/min (ISO 6872) (1997) to measure the four-point ultimate flexural strength. Using a four-point flexural fixture (16-mm span) immersed in a solution (Potassium chloride 0.096%, Sodium chloride 0.0674%, Magnesium chloride 0.0041%, Potassium phosphate 0.0274%, Calcium chloride 0.117%, Carboxymethylcellulose 0.375%, Sorbitol 2.4%, Methylparaben 0.1%, Distilled water; formula developed by School of Pharmaceutical Sciences, São Paulo State University (UNESP), Araraquara, São Paulo, Brazil) to simulate the salivary moisture at 37 ± 1 ºC, close to the humidity and temperature of the oral cavity, the specimens were placed over two 0.8 mm-radius rounded bearers and the as-sintered surfaces received a uniaxial compressive force applied by two rounded loading pistons (0.8 mm-radius, distance of 8.0 mm). The ultimate flexural strength was recorded in Newton (N) and calculated in MPa using the equation described in ISO 6872 (1997).

After being positioned in the MTS 810 testing machine, as previously described for the monotonic test, twenty specimens of each group received a cyclic load, by the staircase method (John Wiley and Sons, 1993), at a frequency of 10 Hz (Scherrer et al., 2011), for 500,000 cycles. A minimum of 15 specimens was needed for accurate data analysis by the staircase test method (John Wiley and Sons, 1993). The entry fatigue stress level was 60% of the monotonic stress at failure, to which a fixed increment (4.0% of mean flexural strength values) was added or subtracted (Vergani et al., 2010; Dixon and Mood, 1948). If a specimen survived after 500,000 cycles, the next specimen received a stress level one increment higher. If a specimen fractured in fewer than 500,000 cycles, the next one was submitted to a load one increment lower. Data analysis was based on the least frequent event (failure vs. survival).

The mean fatigue limit values (XL) and standard deviations (SD) were calculated by the equations below (Dixon and Mood, 1948):

$$XL = X_0 + d\,(A/N) \pm 0.5 \quad (1)$$

$$SD = 1.62\,d\,[NB - (A2/N2) + 0.029], \quad (2)$$

where X_0 was the lowest stress level recorded (MPa); d was the fixed stress increment applied (MPa); N was the sum of failures (or survivals) occurring at the different stress levels; A was the total sum of failures (or survivals) multiplied by the stress levels; and B was the total sum of failures (or survivals) multiplied by the square of the stress levels. In Eq. {1}, a positive or a negative sign was applied when the analysis was based on survivals or on failures, respectively.

2.5. Surface characterization (topography and phase transformation)

Surface topography and phase transformation analyses were made after ultrasonically cleaning all specimens in 99% isopropanol for 10 min and left them drying in silica gel desiccator for 24 h.

2.5.1. Surface topography analysis

For each experimental group, three non-fractured cycled specimens were randomly selected and their tensile sides were examined (×500; 12 kV) by scanning electron microscopy (SEM - JSM-6510LV; JEOL Ltd., Peabody, MA, USA). Three additional non-cycled specimens of groups *AS* and *AS/R* were also analyzed to determine the effect of the cyclic loading on zirconia topography.

SEM (×30 and ×100 magnification) was used for determining the origin of failures on the fracture surface of three cycled specimens of each experimental condition.

2.5.2. Phase transformation (XRD analysis)

X-ray diffraction (XRD) analysis was used to evaluate the effect of grinding protocols and regeneration firing on Y-TZP phase transformation. Data (n = 2) were collected from the treated surface of the specimens by a rotating anode diffractometer (RIGAKU® RINT2000, Akishima, Tokyo, Japan; 40 kV, 70 mA) with Cu kα radiation ($\lambda k\alpha 1 = 1.5405$ Å, $\lambda k\alpha 2 = 1.5443$ Å, $Ik\alpha 1/Ik\alpha 2 = 0.5$) monochromatized by a curved graphite crystal, the interval being from 20º to 80° (2θ), 0.02° (2θ) time per point of ~4.0 s, divergence 0.5 and open receiving slits. For the Rietveld refinements (Rietveld, 1969), the General Structure Analysis System (GSAS) (Larson and Von Dreele, 1994) program suite with EXPGUI interface (Toby, 2001) was used. The crystal structure parameter used as basis of the ICSD (Inorganic Crystal Structure Database) code was: 66781 (ZrO_2, tetragonal), 18190 (ZrO_2, monoclinic), and 53998 (ZrO_2, cubic). Some samples have unidentified Bragg`s peaks and, for this reason, were excluded during Rietveld refinements.

2.6. Statistical analysis

The results were submitted to the Levene and Shapiro-Wilk tests (IBM SPSS Statistics version 20; Statistical Package for Statistical Science Inc, Chicago, IL, USA) for analysis of the homogeneity of variance and normality, respectively. Two-way ANOVA was applied for the independent variables *grinding protocol* and *regeneration firing* using the software Microsoft® Excel for Mac 2011 (version 14.4.7, Microsoft Corporation, Redmond, WA, USA). When a significant effect was observed, the Tukey HSD post hoc test was applied ($\alpha = 0.05$).

3. Results

The two-way analysis of variance showed that there were significant differences ($P < .001$) in the flexural fatigue limit for the independent variables *grinding protocol*, regeneration firing and their interaction. The mean values, standard deviations and coefficients of variation for the four-point flexural fatigue limit and Tukey HSD ($\alpha = 0.05$) post-test results are shown in Table 1.

In the groups not submitted to regeneration firing, the mean flexural fatigue limit of *WG* was significantly higher ($P < .05$) than that of *AS* and *G* groups, which presented no significant difference between them ($P > .05$). In the samples submitted to regeneration firing, the inequality *WG* > *AS* > *G* ($P < .05$) was observed. Regeneration firing increased the fatigue limit of the *AS* group and decreased those of the ground groups (*G* and *WG*) ($P < .05$).

Grinding produced significant changes in the surface characteristics

Table 1
Mean values (MPa), standard deviations (in parentheses; ±) and coefficients of variation (%) of the flexural fatigue limit.

	Before regeneration firing	After regeneration firing (*R*)
As-sintered (*AS*)	452.46 (± 65.20) / 14.41% **B**^{b}	560.93 (± 32.46) / 5.79% **B**^{a}
Grinding (*G*)	488.90 (± 0.58) / 0.12% **B**^{a}	427.06 (± 18.44) / 4.32% **C**^{b}
Water-cooled grinding (*WG*)	758.86 (± 73.02) / 9.62% **A**^{a}	662.32(± 86.78) / 13.1% **A**^{b}

Equal capital letters, in the columns, and superscripted letters, in the lines, indicated statistical equality ($P \geq .05$).

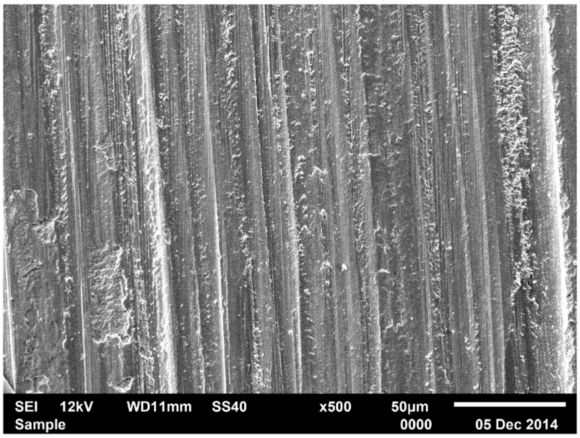

Fig. 2. Photomicroscopy (500×) of a specimen subjected to dry grinding (*G* group).

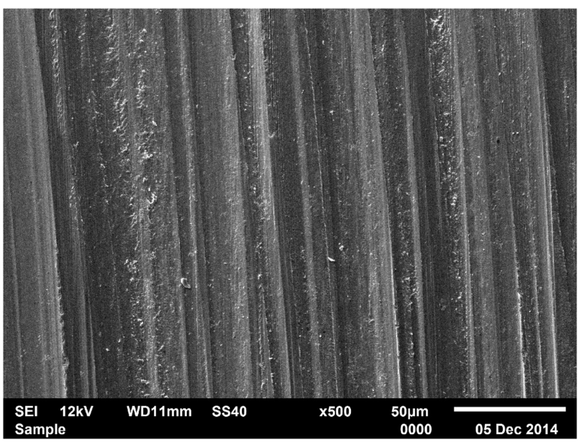

Fig. 3. Photomicroscopy (500×) of a specimen submitted to wet grinding (*WG* group).

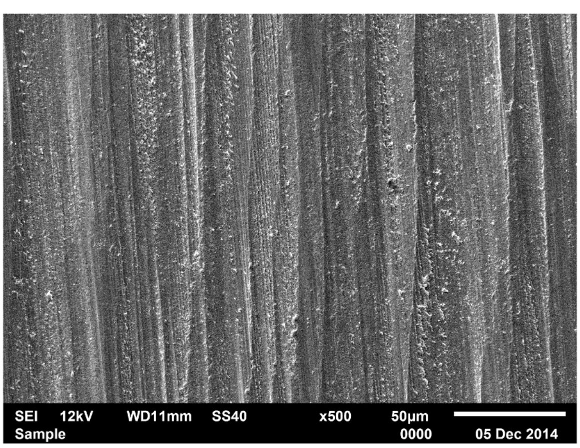

Fig. 4. Photomicroscopy (500×) of a specimen submitted to dry grinding after regeneration firing (*G/R* group).

of the samples, compatible with the grooves created by the diamond stone. Dry grinding produced more evident changes on the Lava™ zirconia surface (Figs. 2 and 3, 4 and 5).

Figs. 6 and 7 show that regeneration firing did not produce changes in the surface topography of zirconia. Similarly, by comparing the specimens submitted to the staircase method with those not cycled, it could be observed that the cyclic loading did not alter its surface topography (Figs. 6 and 8). Minimal defects with the absence of

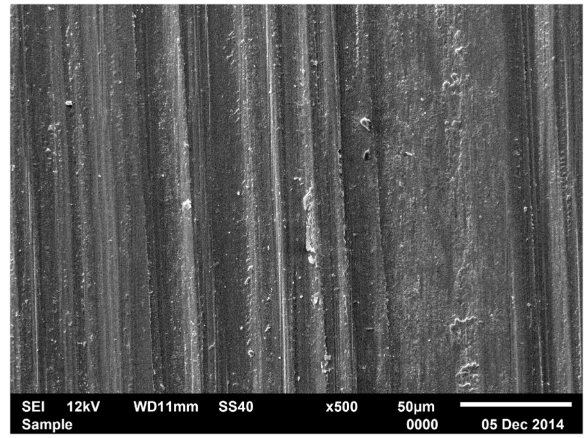

Fig. 5. Photomicroscopy (500×) of a specimen subjected to wet grinding after regeneration firing (*WG/R* group).

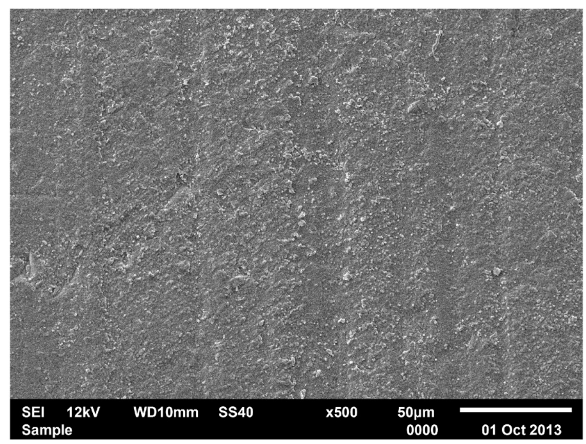

Fig. 6. Photomicroscopy (500×) of a specimen of the control group (*AS*).

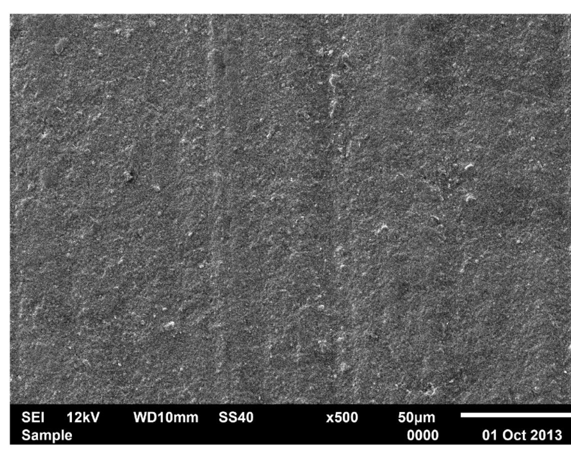

Fig. 7. Photomicroscopy (500×) of a specimen of the control group after regeneration firing (*AS/R*).

microcracks can be noted. Irrespective of the experimental groups, examination of the fracture sites revealed that all fractures started on the tensile side of the specimens (Fig. 9). Moreover, in a high magnification (Fig. 10A and B) it was possible to identify the fracture origin, mirror area and hackles, indicated by the semicircle and arrows, respectively. Crack propagation occurred from the tensile side towards the opposite side of the stress concentration (compression side).

Regeneration firing decreased or zeroed the monoclinic phase content (initial range before the thermal cycle = 2.4–8.6%; final range

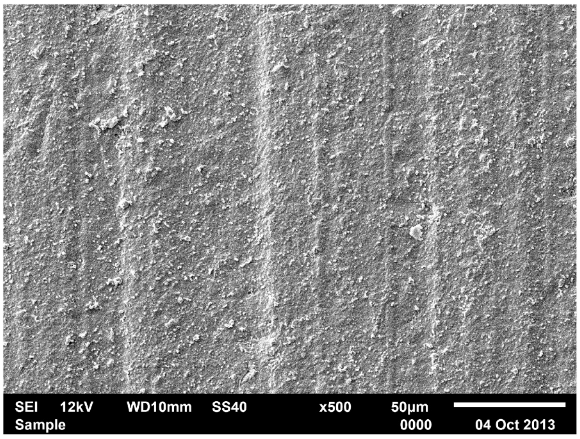

Fig. 8. Photomicroscopy (500×) of a specimen of the *AS* group previously to cyclic loading.

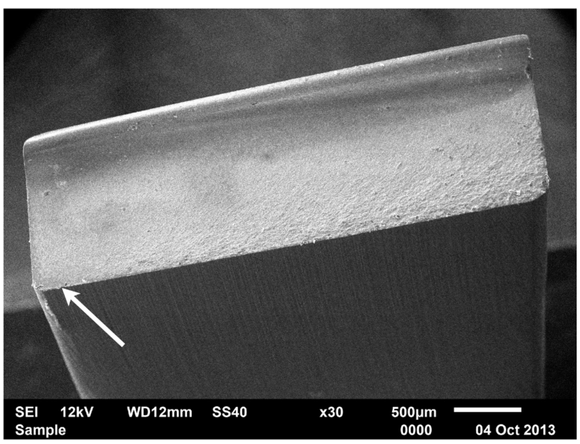

Fig. 9. Photomicroscopy (30×) of a control group specimen after regeneration firing, showing the beginning of the fracture on the tensile side (see arrow), opposite the compression surface where the chipping occurred.

after thermal cycle = 0.0–6.5%) and increased the tetragonal phase percentage of Y-TZP (initial range = 48.7–68.0%; final range = 54.6–100%). The grinding procedure, with or without cooling, before or after cycling, decreased the tetragonal phase content of Y-TZP (Table 2 and Fig. 11).

Table 2
Phase content (wt%) after Rietvelt refinement.

		Phases (wt%)		
		t-ZrO_2	m-ZrO_2	c-ZrO_2
non-cycled	*AS*	66.0(2)	8.6(1)	25.3(5)
	AS/R	74.7(8)	1.2(9)	24.1(2)
	G	52.8(7)	2.4(3)	44.8(9)
	G/R	54.6(5)	5.7(8)	39.6(8)
	WG	48.7(4)	6.3(4)	44.9(8)
	WG/R	100	–	–
Cycled	*AS*	68.0(2)	7.4(2)	24.4(3)
	AS/R	93.5(13)	6.5(13)	–
	G	51.5(5)	2.8(3)	45.5(7)
	G/R	89.8(1)	–	10.2(6)
	WG	55.2(5)	4.4(8)	40.4(8)
	WG/R	73.4(4)	–	26.0(1)

Codes: t - tetragonal; m - monoclinic; c - cubic.

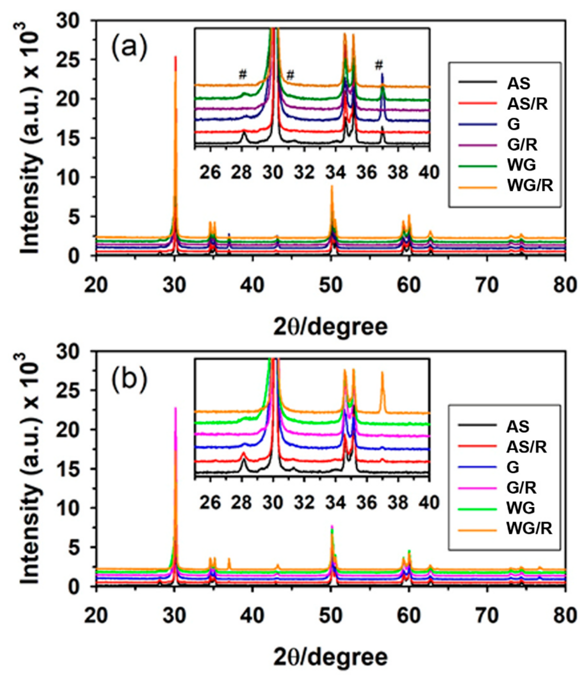

Fig. 11. X-ray diffraction patterns. (a) non-cycled specimens. (b) cycled specimens.

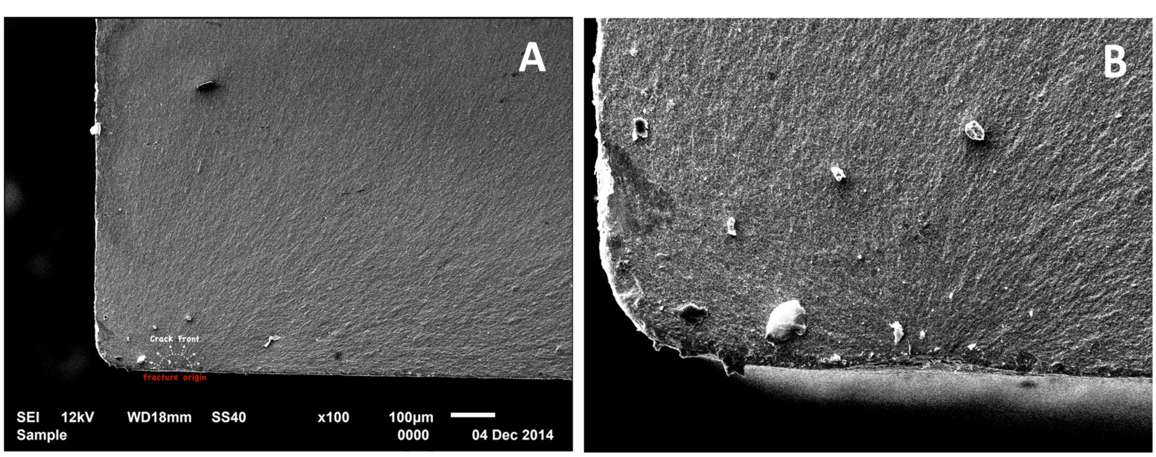

Fig. 10. A: Photomicroscopy (100×) of a specimen not submitted to regeneration firing, showing the origin of the fracture on the tensile side (half-circle). Arrows indicate the direction of crack propagation into opposite side (compression side – curl region). All specimens produced the same fractographic pattern, irrespective of the experimental condition. B: Closer view highlighting the initial crack propagation area.

4. Discussion

The mechanical strength of Y-TZP may be influenced by surface treatments that may cause different degrees of damage (Pilathadka et al., 2007; Wang et al., 2008; Qeblawi et al., 2010; Guess et al., 2010a; Garcia Fonseca et al., 2013; Ohkuma et al., 2011; Rungruanganunt and Kelly, 2012). Among the possible changes to which the zirconia surface may be subjected are grinding with stones or burs performed to obtain lab and clinical adjustments for adequate marginal fit, emergence profile and interocclusal space (Candido et al., 2017). In a recent study Candido et al. (2017) detected that grinding with diamond stone (MasterCeram) changed the crystallographic phases, surface roughness and wettability of a Y-TZP. However, there was no information about the effect of this procedure on fatigue behavior of the material. Fatigue limit of Y-TZP was determined under four-point flexural mechanical cycling test by staircase method, under conditions closer to those induced during masticatory function.

The null hypothesis was rejected, once water-cooling grinding increased the flexural fatigue limit of the Lava™ ceramic. Previous studies evaluating the monotonic flexural strength (Hatanaka et al., 2017) and flexural fatigue limit (Zucuni et al., 2017; Polli et al., 2016), after grinding with diamond burs specific for zirconia, also verified changes in the static and dynamic mechanical properties of Lava™ Y-TZP. These results were probably related to the compression stresses induced by *t*→*m* phase transformation (Zucuni et al., 2017; Curtis et al., 2006; Karakoca and Yilmaz, 2009; Amaral et al., 2016; Pittayachawan et al., 2009) accompanied by volumetric expansion of approximately 5.0% (Kim et al., 2010; Denry and Holloway, 2006) that resulted in compressive stresses in the micrometric layers of the surface, which not only contained the surface failures (Khayat et al., 2018), but at first increased the fracture strength of the material (Pereira et al., 2016; Khayat et al., 2018; Hjerppe et al., 2016). According to Teixeira et al. (2007) due to this process, there is a reduction in energy at the crack tips, limiting their propagation. Probably, the mechanical changes introduced during the grinding process produce more pronounced effect than the heat generated by friction with the diamond stones. Thus, the effect of *t*→*m* phase transformation induced by wet grinding (*WG* group) may have superimposed the effect of *m*→*t* phase transformation induced by the heating produced during that procedure (Guazzato et al., 2005). In spite of the positive results observed for the mechanical property evaluated, the increase in monoclinic phase may make the zirconia more susceptible to degradation in an aqueous or acid environment (Kosmac et al., 2000; Scherrer et al., 2011).

The mean flexural fatigue limit of the samples ground with stone under cooling was significantly higher in comparison with that of the control (*AS* specimens), differently from that which occurred with the specimens submitted to the dry grinding procedure. It is possible that the mechanical changes introduced during the grinding process produce less effect than the heat generated by not using cooling. Thus, the *m*→*t* reverse phase transformation induced by the heating produced during the grinding processes may have superimposed itself on the effect of the *t*→*m* phase transformation induced by the same procedures (Lee et al., 2016; Guazzato et al., 2005; Hjerppe et al., 2016). According to Hjerppe et al. (2016) this hypothesis is based on the low heat conductivity of zirconia when compared with other ceramics, which, in situations of grinding, have less potential for dissipating the heat produced. Thus, the surface and heat treatments may present opposite effects on the mechanical strength of Y-TZP (Wang et al., 2008; Guazzato et al., 2005; Işerı et al., 2010; Guess et al., 2010a). Further relevant information, when comparing the samples ground with or without cooling, is that significant changes were observed in the surface topography of the samples submitted to dry grinding: they presented less homogeneous surfaces with greater irregularity; something that must have influenced the flexural fatigue limit of the samples. Candido et al. (2017) observed higher roughness mean values on the surface of Y-TZP after wet grinding, and more homogeneous grinding in the absence of cooling; differently from conditions observed in the present study. According to the authors (Candido et al., 2017), the action of water during cooling contributed to clean the stone tip, favoring more effective cutting, while in the dry process, ceramic powder was impregnated onto the stone surface, diminishing its cutting power and contributing to polishing of the zirconia bar. Indeed, we clearly observed slurrying on the surface of the stones during grinding without irrigation. However, this slurrying was not homogeneous on the surface of the diamond stones, and contrary to that observed by Candido et al. (2017) this non-homogeneous slurrying certainly contributed to a more irregular grinding on the Y-TZP surface with larger flaws/grooves; which most probably were related to the worst fatigue limit results after dry grinding. Other authors (Vagkopoulou et al., 2009; Kosmac et al., 2000) have also affirmed that wet grinding are less deleterious, producing fewer surface defects, so that they do not damage the mechanical properties of zirconia.

Samples submitted to regeneration firing usually present reverse phase transformation, from monoclinic to tetragonal (*m*→*t*) (Zucuni et al., 2017; Guazzato et al., 2005), with release of compressive stresses at surface of the material (Zucuni et al., 2017). Considering that the regeneration firing of zirconia after sintering may revert the phase transformation induced by grinding, the damage created on the surface may culminate in fracture of the bulk material, due to crack propagation or larger persistent defects, because flaws/microcracks/ cracks may not be eliminated after heat-treatment (Zucuni et al., 2017; Denry and Holloway, 2006). These inferences help to explain the behavior observed for the ground samples submitted to regeneration firing, which presented a lower fatigue limit in comparison with the ground samples not submitted to heat-treatment.

Several authors (Sato et al., 2008; Guazzato et al., 2005; Thompson et al., 2011) have verified that heat-treatment may reduce the mechanical strength of the material, corroborating the findings of the present study. However, an increase in the fatigue limit of the as-sintered samples was verified. Based on the literature (Amaral et al., 2013), a possible explanation for this result may be based on the small quantity of residual monoclinic phase in *AS* group, transformed into tetragonal phase after regeneration firing, and thus contributing to increase the mechanical property. In a recent study (Polli et al., 2016), the Y-TZP Lava™ presented approximately 8.0 by wt% of monoclinic phase in the as-sintered (control) group, which corroborates this hypothesis. Ryan et al. (2016) also observed monoclinic phase content (7.3 wt%) in samples of as-sintered Y-TZP Lava™, which reinforces the inferred explanation. Another reasonable supposition may be related to a molecular rearrangement induced by heating, which could have altered the microstructure of the material. This possible change could have contributed to sealing the defects and microcracks initially present on the material surface, inherent to the sample fabrication process (e.g., cutting in a metallographic cutter), by means of a process cited in the literature as healing (Ho et al., 2009). Taking into consideration that this was an isolated effect, and based on the absence of surface changes detectable by SEM in the samples either submitted to the regeneration firing or not, future studies are necessary to confirm this finding. In other words, whether this effect is related only to *m*→*t* transformation, or whether it is associated with the molecular rearrangement of the tetragonal phase, with possible sealing of small cracks/flaws, additional investigations are necessary for this type of proof.

A limitation of this study is the use of specimens with simple geometry, instead of the use of anatomic specimens, with a complex design and/ or the presence of veneering ceramic. Additional studies evaluating the Weibull reliability analysis, as well as the low temperature degradation (LTD) of the zirconia are necessary to determine the long-term behavior of the material, especially because these analyses concern the post-grinding conditions simulating the oral environment, factors that possibly increase the monoclinic phase content of the material. In the maximum mechanical aging conditions applied (500,000 cycles), there was an increase on the flexural fatigue limit of Y-TZP after grinding. However, in the long term the material is expected to be more susceptible to degradation and failures, something of fundamental importance to additional studies.

5. Conclusions

Based on the results and on the limitations of the present study, the authors concluded that:

1. Grinding had distinct effect on the fatigue limit according to the use of water-cooling;
2. The fatigue limit was increased after water-cooled grinding, irrespective of regeneration firing;
3. Regeneration firing increased the fatigue limit of the as-sintered Y-ZTP, while reduced its mechanical strength after grinding;
4. Grinding modified the Y-TZP topography, incorporating evident grooves at surface, whereas regeneration firing did not change its topography;
5. In general, regeneration firing decreased or zeroed the monoclinic phase.

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Declaration of interest

None.

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