

Effect of Grinding and Resintering on the Fatigue Limit and Surface Characterization of a Y-TZP Ceramic

Gabriela Scatimburgo Polli¹, Gabriel Rodrigues Hatanaka¹, Filipe de Oliveira Abi-Rached¹, Lígia Antunes Pereira Pinelli¹, Márcio de Sousa Góes², Paulo Francisco Cesar³, José Maurício dos Santos Nunes Reis¹

¹Department of Dental Materials and Prosthodontics, Araraquara Dental School, UNESP - Univ Estadual Paulista, Araraquara, SP, Brazil
²UNILA - Universidade Federal da Integração Latino-Americana, Foz do Iguaçu, PR, Brazil
³Department of Biomaterials and Oral Biology, Dental School, USP - Universidade de São Paulo, São Paulo, SP, Brazil

Correspondence: José Maurício dos Santos Nunes Reis, Rua Humaitá, 1680, Centro, 14801-903 Araraquara, SP, Brasil. Tel: +55-16-3301-6503. e-mail: reis@foar.unesp.br

This study evaluated the effect of grinding protocols and resintering on flexural fatigue limit and surface characterization of Lava™ Y-TZP. Bar-shaped specimens (20×4.0×1.2 mm, n=40; 20×4.0×1.5 mm, n=80) were obtained. Half of the thinner specimens (1.2 mm) constituted the as-sintered group (AS), while the thicker ones (1.5 mm) were ground with diamond burs under irrigation (WG) or not (G). The other half of thinner and half of ground specimens were resintered (1000 °C, 30 min), forming the groups ASR, WGR and GR. Fatigue limit (500,000 cycles, 10 Hz) was evaluated by staircase method in a 4-point flexural fixture. Data were analyzed by 2-way ANOVA and Tukey's test ($\alpha=0.05$). Surface topography (n=3) and fracture area (n=3) were evaluated by SEM. X-ray diffraction data (n=1) was analyzed by Rietveld refinement. ANOVA revealed significant differences ($p<0.001$) for the grinding protocol, resintering and their interaction. Grinding increased the fatigue limit of non-resintered groups. There was no significant difference among the resintered groups. Resintering significantly increased the fatigue limit of the AS group only. Both protocols created evident grooves on zirconia surface. The failures initiated at the tensile side of all specimens. The percentages (wt%) of monoclinic phase were AS (8.6), ASR (1.2), G (1.8), GR (0.0), WG (8.2), WGR (0.0) before, and AS (7.4), ASR (6.5), G (3.2), GR (0.2), WG (4.6), WGR (1.1) after cyclic loading. Grinding increased the fatigue limit of non-resintered Y-TZP and formed evident grooves on its surface. Resintering provided significant increase in the fatigue limit of as-sintered specimens. In general, grinding and resintering decreased or zeroed the monoclinic phase.

Key Words: zirconia, fatigue limit, staircase method, grinding, resintering.

Introduction

The yttria partially stabilized tetragonal zirconia polycrystalline (Y-TZP) is widely used in dental practice, mainly for manufacturing metal-free frameworks for single units and long span fixed dental and implant-supported prostheses. However, mechanical and thermal aging may induce tetragonal (t) to monoclinic (m) phase transformation (toughening mechanism) that may modify the mechanical behavior of the zirconia (1,2).

Zirconia frameworks/implant abutments are usually designed and milled in computer-aided design/computer-aided manufacturing (CAD/CAM) systems with minimal excess in the finishing line of tooth/abutment preparation. After sintering, this excess is finished in the lab by grinding on individual dies. Clinical grinding may also be required to obtain enough interocclusal space for the veneering porcelain, accurate marginal fit, as well as to correct the emergence profile, and the design of frameworks and implant abutments. A minimal or an extensive redesign is defined by several factors, like virtual 3D project of the framework, precision milling of CAD/CAM systems and the shape of tooth preparation (3). According to Swain (4), the effect of grinding on the flexural strength of zirconia is

contradictory and influenced by its severity and the attained local temperature, as well as by the amount, size and depth of the flaws/grooves, which are strongly related to the volume of t→m transformation.

Despite the possibility of toughening mechanism, Iseri et al. (5) did not find significant differences on the flexural strength of a Y-TZP submitted to different grinding methods. In turn, Luthardt et al. (6) recommend that grinding should be carefully performed ensuring higher zirconia reliability. Similarly, Denry and Holloway (7) suggested that grinding could damage the longitudinal mechanical strength of Y-TZP. Nevertheless, heat treatment ("resintering") after grinding may be beneficial by the reverse phase transformation (m→t) and elimination of the compressive stress at the zirconia surface (7). The consequence of this phenomenon occurs according to the severity of condition to which the material is subjected and related to the amount of flaws/cracks. The m→t transformation may improve the aging resistance (1) and, consequently, the reliability of the zirconia (8). On the other hand, its influence on the mechanical behavior of Y-TZP has been scarcely investigated (9).

Although several studies (3,5-9) evaluated the

consequences of grinding, finishing, polishing and sandblasting on the mechanical properties of Y-TZP, very little is known about the influence of grinding and resintering treatments on its fatigue limit. A widely used method that has proven to be suitable for evaluating the zirconia fatigue limit is the staircase approach (10-15). Mechanical fatigue tests are usually performed to analyze the effect of aging on several restorative materials and often only analyze the ability of materials to resist a given number of cycles. The staircase approach is an outstanding method for estimating the fatigue behavior, investigating the survival rates and for calculating the maximum stress supported by a material in cyclic conditions (16). The threshold level is calculated from an up-and-down test condition, in which the specimens are repeatedly cycled under different stresses until a given number of runouts and failures have been reached. In addition, the staircase method requires fewer tests to calculate reliable threshold values than any other cycling method, because it concentrates testing near the mean (11).

Zirconia under mechanical stress may undergo changes in its toughening mechanism, favoring a subcritical propagation of microcracks and the low-temperature degradation phenomenon (17). Thus, its long-term stability and fatigue properties should be studied warranting safe therapeutic procedures. Therefore, it is important to evaluate the effect of resintering (regeneration firing) and different grinding protocols on the fatigue limit of Y-TZP, as well as on its surface characteristics (topography and phase transformation). The null hypothesis was that grinding and resintering would not influence the Y-TZP fatigue limit.

Material and Methods

Preparation of Zirconia Specimens

Pre-sintered CAD/CAM zirconia milling blocks (Lava Frame; 3M ESPE AG, Seefeld, Germany) were cut into bars of 25×5.0×1.5 mm (n=40) and 25×5.0×1.9 mm (n=80) using a water-cooled diamond disc (Diamond Wafering Blade, Series 15LC Diamond #11-4276; Buehler Ltd, Lake Bluff, IL, USA) by a high-precision saw (Isomet 1000; Buehler Ltd). The zirconia bars were washed in tap water and their edges were carefully finished using a ceramic polisher (Exa Cerapol 0361HP; Edenta AG, Au, Switzerland) in a low-speed hand piece.

The sintering process was performed in Lava Furnace 200 (Dekema Dental-Keramiköfen GmbH, Freilassing, Germany), according to the manufacturer's recommendations (heating rate = 20 °C/min: 0-1000 °C; 10 °C/min: 1000-1500 °C; holding time = 2.0 h and cooling rate = 15 °C/min: 1500-800 °C; 20 °C/min: 800-250 °C, when oven was opened). The dimensions of the zirconia bars after sintering shrinkage were 20×4.0×1.2 mm (n=40) and 20×4.0×1.5 mm (n=80).

Grinding Procedures

Half of the thinner specimens constituted the as-sintered group (AS - control). The thicker ones were ground under constant irrigation in distilled water (WG) or not (G). For this procedure, the specimens were positioned into a custom-made stainless steel apparatus with a rectangular central opening (20.05×4.05×1.20 mm) and the 0.3 mm excess of zirconia was ground with 151 µm-grit cylindrical diamond burs (4ZR; Komet-Brasseler, Lemgo, Germany) in a high-speed hand piece (Kavo Extra Torque 605, Kavo do Brasil Ind. e Com. Ltda, Joinville, SC, Brazil) at 350,000 rpm, until the final thickness of 1.2 mm, measured by a digital caliper (500-144B; Mitutoyo Sul Americana, Suzano, SP, Brazil) in four different points. The high-speed handpiece was clamped to a holding arm, which moved horizontally, assuring the application of a constant load of the diamond bur in contact with the specimen surface and perpendicular to its length. After grinding, all zirconia bars (20×4.0×1.2 mm - ISO 6872 standard) (18) were washed in tap water and left to dry at room temperature for 24 h.

Resintering (Regeneration Firing)

The other half of the thinner specimens and half of the WG and G groups were submitted to a resintering treatment (1000 °C for 30 min) in a conventional porcelain oven (Aluminipress; EDG Equipamentos e Controles Ltda, São Carlos, SP, Brazil), according to the Lava™ manufacturer's recommendation, resulting in ASR, WGR and GR groups (n=20). After this procedure, the specimens were cooled to room temperature by opening the oven door.

Cyclic Fatigue Testing (Staircase Method)

For determining the entry fatigue stress level, it was necessary to perform monotonic tests before the cyclic fatigue testing. For each designed experimental condition (AS, WG, G, ASR, WGR and GR), 12 additional specimens (20×4.0×1.2 mm) were used to measure the monotonic four-point ultimate flexural strength in a servo-hydraulic mechanical testing machine (810 Material Test System; MTS Systems Corp, Eden Prairie, MN, USA) with a 10 kN load cell and a constant crosshead speed of 0.5 mm/min (ISO 6872) (18). The specimens were positioned in a four-point flexural fixture (16-mm span) over two 0.8-mm radius rounded bearers in artificial saliva, thermostatically maintained at 37±1 °C. A uniaxial compressive force was applied over the non-ground surface of each specimen by two-round loading pistons (0.8 mm-radius, 8.0 mm distance), the ground surfaces being loaded in tension until failure. The ultimate flexural strength was recorded in Newton (N) and calculated in MPa using the equation listed in ISO 6872 (18).

Twenty specimens of each group were positioned in the MTS 810 testing machine, under the same conditions as for the monotonic flexural strength test, and subjected to a cyclic load in a four-point bending mode at 10 Hz frequency (19), for 500,000 cycles in artificial saliva at 37 ± 1.0 °C. Cyclic fatigue test was determined by the staircase method (20). According to Collins (20), a minimum of 15 specimens is required for accurate data analysis by staircase. The entry fatigue stress level was set at 60% of the monotonic stress at failure, which was increased or decreased by a fixed increment (4.0 % of mean flexural strength values) (16,21), depending on the occurred event (failure or survival) during the application of 500,000 cycles. If the specimen survived to 500,000 cycles of loading, a stress level one increment higher was applied to the next specimen. If the specimen fractured in fewer than 500,000 cycles of loading, a load one increment lower was applied to the next one. This procedure was performed until all specimens of each group were expended. The data analysis was based on the least frequent event (failure vs. survival).

The mean fatigue limit values (XL) and standard deviations (SD) were calculated using the following equations (16):

$$XL = X_0 + d (A/N) \pm 0.5 \{1\}$$

$$SD = 1.62 d [NB - (A^2/N^2) + 0.029] \{2\},$$

where X_0 is the lowest recorded failure stress (MPa), d is the fixed increment in MPa, N is the sum of failures or survivals occurring at the different stress levels, independent of the load applied, A is the total sum of failures or survivals, multiplied by the stress levels and B is the total sum of failures or survivals, multiplied by the square of the stress levels. In the first equation, a negative sign was used when the analysis was based on failures, in other words, when they occurred in lower number within the 20 specimens evaluated for each experimental group.

Statistical Analysis

Data from cyclic fatigue tests were statistically analyzed by 2-way analysis of variance, considering the grinding protocol and resintering as independent variables (Microsoft® Excel for Mac 2011, version 14.4.7, Microsoft Corporation, Redmond, WA, USA). If there was a significant effect, the Tukey's post-hoc Honestly Significant Difference (HSD) test ($\alpha=0.05$) was applied.

Surface Characterization (Topography and Phase Transformation)

For the analyses of surface topography and phase transformation, all specimens were ultrasonically cleaned in 99 % isopropanol for 10 min and dried in silica gel desiccator for 24 h.

Surface Topography Analysis

The non-fractured cycled specimens ($n=3$) of each experimental group were randomly selected, gold-sputtered, mounted on metallic stubs and their tensile sides were examined by scanning electron microscope (SEM - JSM-6510LV; JEOL Ltd., Peabody, MA, USA) at $\times 500$ magnification with a 12 kV accelerating voltage. Non-cycled additional specimens of the groups AS and ASR were also characterized ($n=3$) to evaluate the effect of cyclic loading on the zirconia topography without grinding influence.

In order to determine the origin of the failures, the fractured surfaces of three cycled specimens from each group were examined by SEM at $\times 30$ magnification.

Surface Phase Transformation Analysis

The X-ray diffraction (XRD) analysis assessed the effect of the grinding with or without cooling and the resintering on the phase transformations of Y-TZP. The XRD data ($n=1$) were collected using a RIGAKU® RINT2000 rotating anode diffractometer (40 kV, 70 mA) with Cu $k\alpha$ radiation ($\lambda_{k\alpha 1}=1.5405$ Å, $\lambda_{k\alpha 2}=1.5443$ Å, $I_{k\alpha 1}/I_{k\alpha 2}=0.5$) monochromatized by a curved graphite crystal. An interval from 20° to 120° (2θ), with a step size of 0.02° (2θ), 4 s per step and divergence 0.5, and open receiving slits were the selected conditions for Rietveld refinement (22). The Rietveld refinements were performed using the General Structure Analysis System (GSAS) program suite with EXPGUI interface. The following parameters were refined: atomic coordinates, occupancies, unit cell, scale factor, sample displacement, atomic displacement and full width at half maximum (FWHM). The crystal structure parameter used as basis of the ICSD (Inorganic Crystal Structure Database) code was: 66781 (ZrO₂, tetragonal), 18190 (ZrO₂, monoclinic) and 53998 (ZrO₂, cubic).

Results

Flexural Strength Fatigue Limit

The 2-way analysis of variance revealed significant effect ($p<0.001$) on the flexural strength fatigue limit for the variables grinding protocol, resintering and their interaction. The mean flexural strength fatigue limit values, standard deviations and the results of Tukey's HSD post-hoc test are presented in Table 1. The AS group presented the lowest mean flexural strength fatigue limit ($p<0.05$), which was increased by the resintering procedure, showing no significant difference among the ground groups.

Surface Topography

Representative SEM micrographs of cycled and non-cycled groups are presented in Figure 1. The ground specimens (WG and G) produced evident changes on the

surface feature, compatible with hazards created by the diamond bur (Figs. 1C and 1E). Resintering did not produce changes in the zirconia surface topography (Figs. 1A vs. 1B; 1C vs. 1D and 1E vs. 1F). Similarly, no apparent surface changes were observed comparing the cycled with the non-cycled specimens, with minimal flaws noted in both (Figs. 1A and 1G). Regardless of the experimental groups, examination of the fracture sites revealed that all failures initiated at the tensile side of the specimens (Fig. 2).

Surface Phase Transformation

Table 2 shows the results of quantitative phase analysis and Figure 3 presents the representative diffraction patterns of the experimental groups. In general, before and after cyclic loading, the grinding with or without cooling provided a decrease in the monoclinic phase, and a "decomposition" of t-ZrO₂, m-ZrO₂ and c-ZrO₂ phases in other (t-ZrO₂*). For the G and WG non-cycled groups, the resintering promoted total incorporation of monoclinic phase into tetragonal phase. For the other experimental conditions, this procedure decreased the monoclinic phase content.

Discussion

Although the CAD/CAM technology allows obtaining accurate metal-free prostheses, adjustments in zirconia frameworks/implant abutments are routinely performed in laboratory and clinical practice (8,9,23). Several authors (9,19,24) have claimed that these adjustments and surface treatments may modify significantly the physical, chemical and mechanical properties, influencing the strength of the Y-TZP with different degrees of damage (3,10).

This study revealed that grinding increased the flexural fatigue limit of the Lava™ zirconia, regardless of water-cooling. Pereira et al. (15) also observed an increase in the fatigue limit of two Y-TZP ceramics after grinding with coarse diamond bur (181 μm). Probably, this finding is related to a "decomposition" of t-ZrO₂, m-ZrO₂ and c-ZrO₂ phases in another tetragonal (t-ZrO₂*; see Table 2) phase. Therefore, in the ground groups it can be observed

the prevalence of the tetragonal phase in relation to the monoclinic one, as observed by Denry and Holloway (7). According to Mochales et al. (25), the higher monoclinic phase content impairs the mechanical behavior of zirconia along time, making it critically brittle. Considering that in the present study was simulated a long-term clinical condition, it may be inferred that the higher percentage of tetragonal phase was probably responsible for the best mechanical performance of the ground specimens. Other hypothesis could be related to a molecular rearrangement induced by grinding, which may have altered the physicochemical structure of the material, increasing its mechanical strength. Zirconia grains are not rigid and their three-dimensional movement makes deformation easier after a switching event (26). Maybe the m→t transformation induced by heat during the grinding (27) was masked by the effect of "decomposition" of phases and/or molecular rearrangement induced by grinding. Water-cooling has been used as an aid to the grinding process of Y-TZP surface, cleaning off debris and avoiding overheating with possible phase transformation consequences (3). In this study, before and after cycling, uncooled or cooled grinding provided a decrease in the monoclinic phase and enhanced the mechanical strength of Y-TZP. Further studies are required for a better understanding of how grinding can change the atomic/molecular structure, influencing the zirconia strength. Despite these positive results observed for the evaluated mechanical property, creation of microcracks by grinding can make the zirconia more

Table 1. Mean flexural strength fatigue limit values (MPa) and standard deviations (±) of the experimental groups

Group	Non-resintered	Resintered (R)
As-sintered (AS)	448.55 (±56.22) B ^b	550.49 (±42.33) A ^a
Grinding (G)	520.92 (±34.95) A ^a	557.65 (±59.78) A ^a
Water-cooled grinding (WG)	510.44 (±27.00) A ^a	525.79 (±21.81) A ^a

Vertically, entries with the same capital letter, and horizontally, entries with the same superscript letter, were not statistically different (p≥0.05).

Table 2. Phase content (wt%) of the experimental groups

		Phases (wt%)			
		t-ZrO ₂	m-ZrO ₂	c-ZrO ₂	t-ZrO ₂ *
Non-cycled	AS	66.0(2)	8.6(1)	25.3(5)	-
	ASR	74.7(8)	1.2(9)	24.1(2)	-
	G	60.7(2)	1.8(2)	-	37.4(4)
	GR	61.1(1)	-	-	38.8(7)
	WG	47.3(1)	8.2(1)	-	44.4(1)
	WGR	100.0	-	-	-
Cycled	AS	68.0(2)	7.4(2)	24.4(3)	-
	ASR	93.5(13)	6.5(13)	-	-
	G	51.2(7)	3.2(2)	-	45.6(9)
	GR	70.8(4)	0.2(6)	-	28.9(1)
	WG	56.7(6)	4.6(4)	-	38.6(9)
	WGR	72.4(4)	1.1(2)	-	26.4(1)

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

susceptible to degradation in aqueous or acid environment (2,8,19). Moisture can strongly exacerbate fatigue crack propagation, with slow growth from its nucleation points (2,10,23,24).

Cyclic loads lead to stresses that initiate the propagation of subcritical cracks along the surface of material, which may contribute to its failure (10,13). The nucleation sites and propagation of cracks usually appear near the site

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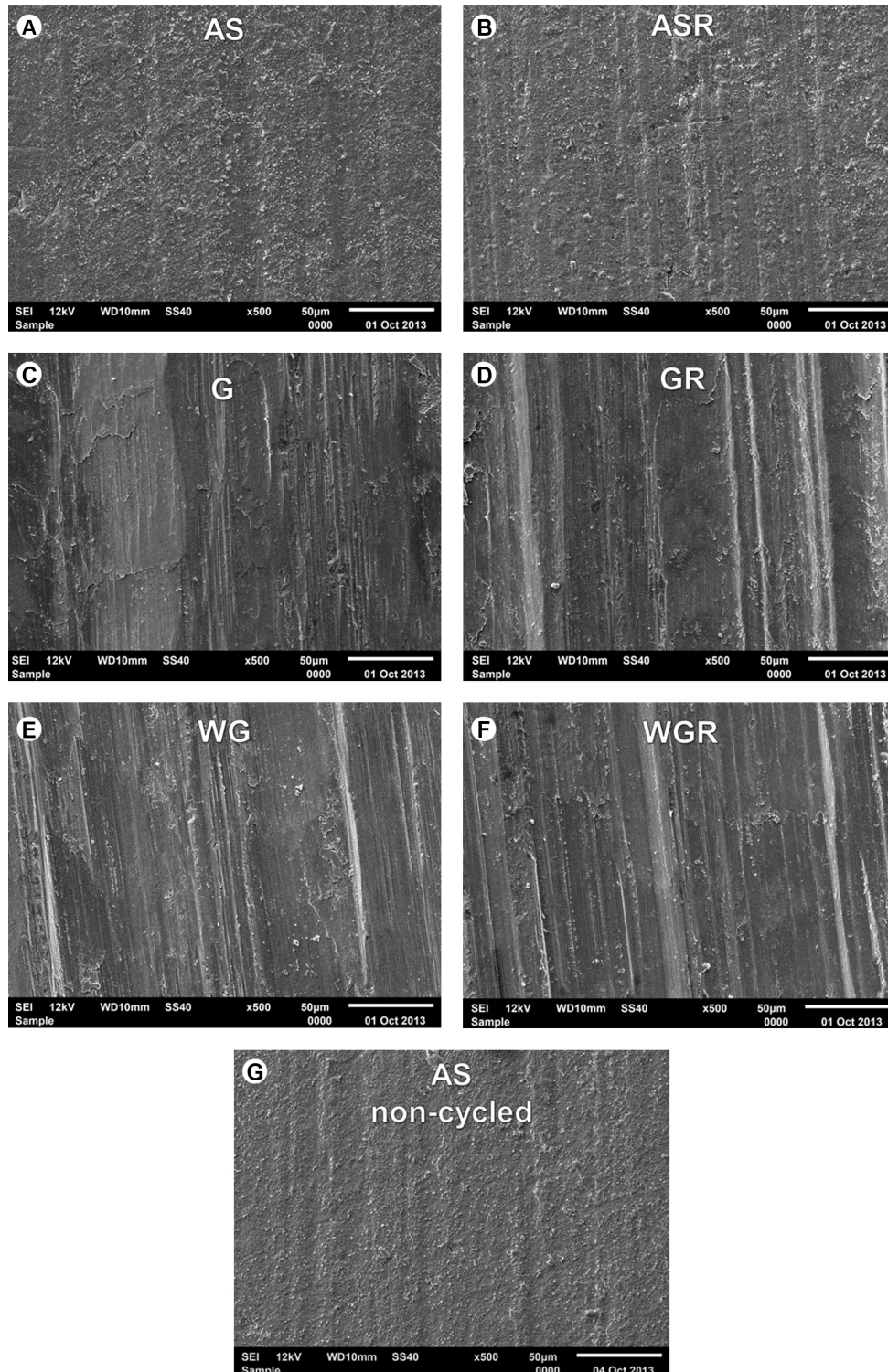


Figure 1. Representative SEM micrographs of cycliced (A-F) and non-cycled (G) groups. A: as-sintered specimen; B: resintered as-sintered specimen; C: ground specimen without cooling; D: resintered ground specimen without cooling; E: ground specimen with cooling; F: resintered ground specimen with cooling; G: as-sintered specimen.

of load application, in the tensile region (10,13,23). Due to repetitive loads during chewing, small defects tend to increase until a critical size, in which catastrophic failures frequently occur (8). Deep surface defects may act as stress concentration areas, being a strength-limiting factor if the length of the flaw extends beyond the superficial compression layer (15-20 μm) on the material's surface (5-7,10). In this study, cyclic loading appears not to have changed the surface topography of the samples. In Figures 1A and 1G, non-detectable differences can be observed

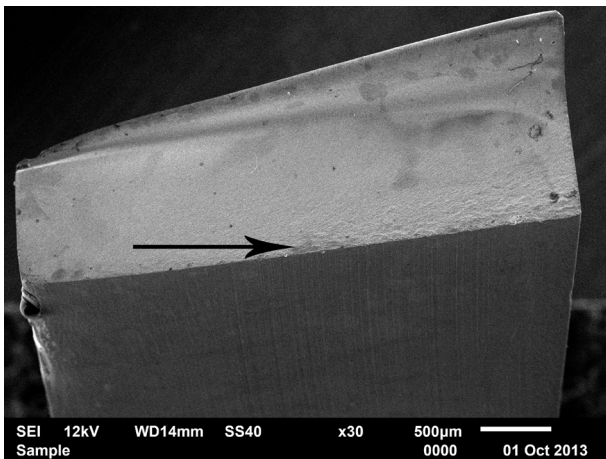


Figure 2. SEM micrograph illustrating the fractured surface of a non-resintered control group specimen. The arrow indicates the beginning of fracture in tensile side. Delamination failure may be seen at the opposite side (compressive area).

between cycled and non-cycled surfaces. This observation may be possibly attributed to the high elasticity modulus of zirconia and its low deflection under flexural cyclic loading. Thus, the mechanical fatigue appears to be not enough to change or damage the topography of the zirconia bars. Similar findings were reported by Amaral et al. (13), in which no signs of crack growth were observed on the surface of a Y-TZP ceramic after a high number of cycles. Further analyzes other than SEM evaluation must be performed to confirm these observations. As expected, the grinding produced apparent changes in the characteristics of the surface. Grinding procedures usually produce grooves, microcracks or even cracks, depending on the used grain of the bur, load and speed (5,10,23,27). The mechanism by which surface damages occur after phase transformation may be explained by the grain loss associated with the increase in the volume of the crystalline material (7). Guazzato et al. (27) showed that coarse grinding (125-150 μm) leads to an increased surface roughness, resulting in reduced bending resistance of Y-TZP. Conversely, the coarse-grit (151 μm) burs used in the present study increased the fatigue limit of Y-TZP. Based on these results, it may be inferred that the cracks did not present a significant critical size to detrimentally affect the fatigue strength of Lava™ Y-TZP. On the other hand, these microcracks may promote degradation of the material in the oral environment and may cause deterioration of its long-term strength (23).

In this study, a reverse phase transformation ($m \rightarrow t$) occurred after resintering. This phenomenon is accompanied

Zirconia fatigue after grinding/resintering

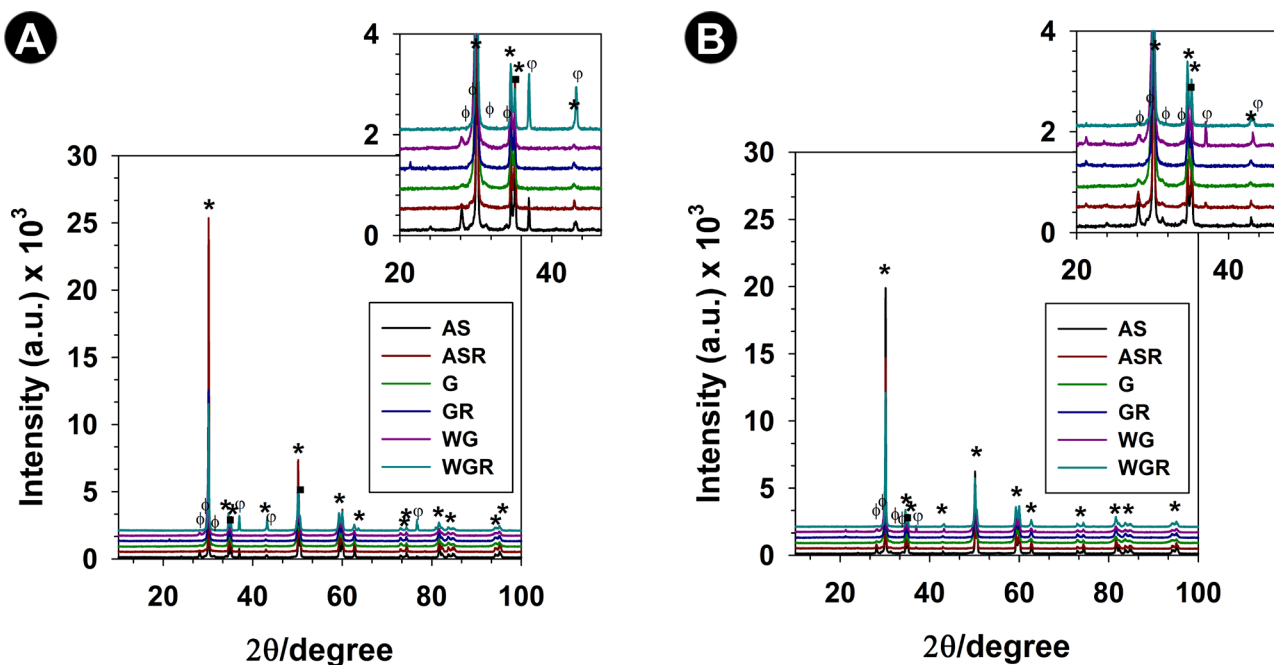


Figure 3. X-ray diffraction patterns in all groups.

by the relief of compressive stresses on the material surface, reducing its mechanical strength (27). Although the heat treatment of the ground zirconia may reverse the phase from $m \rightarrow t$, the damage created in the surface could lead to failure by crack propagation or persistent major defects, since the flaws are not eliminated (7). However, this phase reversibility should not be confused as a mechanism for healing of the previously introduced flaws (28). Resintering only provides mechanical strength improvement to as-sintered specimens, but it may improve the aging resistance of the material, favoring its longevity, due to the decrease of the monoclinic phase content (2,23).

Surface and thermal treatments may have opposite effects on the flexural strength of Y-TZP (5,8,10,27). In the present study no significant differences were observed in the flexural fatigue limit among the resintered groups, although studies (9,27) have found that heat treatment lowers the mechanical strength of zirconia. This may be attributed to the increase in the fatigue limit of the ASR specimens, considering the absence of effects on the strength of the ground samples (WGR and GR). A possible hypothesis for these findings is that the AS group presented an initial amount of monoclinic phase, as observed by the authors (7.4 wt%) and Amaral et al. (1.4 wt%) (29). Thus, when Y-TZP was resintered, part of this monoclinic phase was transformed to tetragonal, favoring its mechanical properties, since in the AS group there was no stress-induced toughening mechanism by grinding. This result may be also explained by a molecular rearrangement and change on the chemical structure of the material induced by resintering. Since this was an isolated effect and considering the absence of detectable surface changes on the samples resintered or not, future investigations should clarify if this behavior is related to $m \rightarrow t$ transformation and/or associated to the molecular rearrangements.

From the results of this study, it was observed that resintering had no effect on the fatigue limit of the ground zirconia, regardless of water-cooling. Possibly, in these samples, the thermal effect of the resintering was superposed by the significant effect of "decomposition" of phases and/or molecular rearrangement produced by grinding. In turn, Wang et al. (8) observed changes in the properties of zirconia after heat treatment. According to those authors (8), thermal firing increased the Weibull modulus of zirconia. Based on this observation and on the results of the current study, it seems that resintering can influence the long-term behavior of mechanically treated zirconia. Furthermore, resintering seems to have a beneficial effect on the ground zirconia, since this step tended to increase the absolute values of flexural fatigue limit.

Among the limitations of this study are the use of geometrical specimens instead of anatomical ones, and

no application of veneering porcelain over the zirconia. Additional studies including Weibull reliability and low temperature degradation analyses are required to evaluate the long-term behavior of Y-TZP under the tested conditions.

Based on the experimental design of this study, the following conclusions can be drawn: grinding with and without cooling increased the flexural fatigue limit of the non-resintered Y-TZP; resintering increased the fatigue limit of the as-sintered zirconia, while it did not change the fatigue strength of the ground groups; grinding changed the surface topography, forming evident grooves on the zirconia surface, while resintering and cyclic loading seemed not to be able to modify its surface feature; in general, grinding and resintering decreased or zeroed the monoclinic phase content.

Resumo

Este estudo avaliou o efeito de protocolos de desgaste e resinterização no limite de fadiga flexural e na caracterização da superfície da zircônia Lava™. Corpos-de-prova em forma de barra (20x4,0x1,2 mm, n=40; 20x4,0x1,5 mm, n=80) foram obtidos. Metade das barras com menor altura (1,2 mm) constituiu o grupo controle (AS), enquanto as de maior altura (1,5 mm) foram desgastadas com fresas diamantadas com (WG) ou sem (G) irrigação. A outra metade dos corpos-de-prova de menor altura e metade dos desgastados foram resinterizadas (1000 °C, 30 min), obtendo-se os grupos ASR, WGR e GR. O limite de fadiga (500.000 ciclos, 10 Hz) foi avaliado pelo método de escada em dispositivo de flexão em 4 pontos. Os dados foram analisados pelos testes ANOVA a dois fatores e Tukey ($\alpha=0,05$). A topografia de superfície (n=3) e área de fratura (n=3) foram avaliadas por MEV. Os dados de difração de raios-X (n=1) foram analisados pelo refinamento de Rietveld. ANOVA revelou diferenças significantes ($p<0,001$) para desgaste, resinterização e interação entre os fatores. O desgaste aumentou o limite de fadiga para os grupos não resinterizados. Não houve diferença significante entre os grupos resinterizados. A resinterização aumentou o limite de fadiga somente para o grupo AS. Ambos os protocolos criaram riscos evidentes na superfície da zircônia. As falhas iniciaram sempre no lado de tração. A porcentagem de fase monoclinica foi: AS (8.6), ASR (1.2), G (1.8), GR (0.0), WG (8.2), WGR (0.0) antes e AS (7.4), ASR (6.5), G (3.2), GR (0.2), WG (4.6), WGR (1.1) após a ciclagem. O desgaste aumentou o limite de fadiga da zircônia não resinterizada e formou ranhuras evidentes na sua superfície. A resinterização promoveu aumento significativo no limite de fadiga somente para o grupo controle. Em geral, o desgaste e a resinterização diminuíram ou anularam o conteúdo de fase monoclinica.

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