Effect of Grinding and Multi-Stimuli Aging on the Fatigue Strength of a Y-TZP Ceramic

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This study aimed to investigate the effect of grinding and multi-stimuli aging on the fatigue strength, surface topography and the phase transformation of Y-TZP ceramic. Discs were manufactured according to ISO-6872:2008 for biaxial flexure testing (diameter: 15 mm; thickness: 1.2 mm) and randomly assigned considering two factors "grinding" and "aging": C- control (as-sintered); CA- control + aging; G- ground; GA- ground + aging. Grinding was carried out with coarse diamond burs under water-cooling. Aging protocols consisted of: autoclave (134°C, 2 bars pressure, 20 hours), followed by storage for 365 days (samples were kept untouched at room temperature), and by mechanical cycling (106 cycles by 20 Hz under a load of 50% from the biaxial flexure monotonic tests). Flexural fatigue strengths (20,000 cycles; 6 Hz) were determined under sinusoidal cyclic loading using staircase approach. Additionally, surface topography analysis by FE-SEM and phase transformation analysis by X-ray Diffractometry were performed. Dixon and Mood methodology was used to analyze the fatigue strength data. Grinding promotes alterations of topographical pattern, while aging apparently did not alter it. Grinding triggered t-m phase transformation without impacting the fatigue strength of the Y-TZP ceramic; and aging promoted an intense t-m transformation that resulted in a toughening mechanism leading to higher fatigue strength for as-sintered condition, and a tendency of increase for ground condition (C < CA; G = GA). It concludes that grinding and aging procedures did not affect deleteriously the fatigue strength of the evaluated Y-TZP ceramic, although, it promotes surface topography alterations, except to aging, and t-m phase transformation.

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Introduction

In Prosthetic Dentistry, yttrium-stabilized tetragonal zirconia polycrystal ceramics (Y-TZP) has been used for frameworks of single-/multi-unit fixed dental prosthesis, which are veneered with feldspathic ceramic, and for monolithic full-contour restorations (1). The major advantages of monolithic restorations are that this assembly allows a substantial ceramic thickness reduction, by eliminating the veneer layer, without compromising the final strength of the system. Thus, this assembly results in a more conservative tooth preparation and by that it decreases tooth removal (1). Additionally, it eliminates the fracture/chipping of the veneer porcelain, which it has been noticing by clinical studies as the main failure of veneered zirconia restorations (2).

Zirconia is a polymorphic ceramic that may be considered a bio-smart material, owing to phase transformation mechanism from tetragonal (t) to monoclinic (m) phase (t \rightarrow m) when the material is stimulated (e.g., humid environment, chemical events, cyclic loading, temperature changes, etc.), resulting in superficial compressive stress concentration around any existing superficial defects/ cracks and preventing (making it difficult) further crack

propagation, increasing its toughness (3).

However, the development of the transformation mechanism (i.e. spreading of m-phase on surface and subsurface) may result in grain detachment (pull-out), increase in surface roughness, decrease in density; consequently it could impair the material's mechanical properties (4,5). Kobayashi and collaborators (6) described this mechanism as low-temperature degradation (LTD) and showed that it is accelerated under presence of water and temperature changes.

Clinically, Y-TZP ceramics are subjected to an environment with plenty moisture (saliva), variation of temperatures, intermittent loading, and oral microorganisms for a long time. All of those conditions might allow LTD to take place and until now few studies evaluated the fatigue strength of Y-TZP ceramic after stimuli that simulate some oral environment aging conditions (especially considering the combination of stimuli) (7-9).

Additionally the Y-TZP restorations (produced by CAD/CAM system, computer-aided design / computer-aided machining) might require laboratorial and/or in-office adjustments to enhance their adaptation, emergency profile and occlusion relations (10). Those adjustments will lead to

 $t \rightarrow m$ phase transformation and also to the introduction of defects (11–13). On this sense, literature shows that the outcome (toughness mechanism or LTD) will depend on the balance between these two factors: phase transformation and introduction of defects (14).

Clinical failure of restorations occurs by fatigue effects. The failure under fatigue is defined as the progressive fracture in response to cyclic forces below the monotonic strength of the material (15). Basically, cyclic forces work on existing defects resulting in the progressive growth of these defects until it achieves a critical size where the fracture occurs; this mechanism is known as slow crack growth (16). In vitro studies seek to simulate those conditions under a standardized environment simulating as close as possible the oral environment. Thus, some parameters have to be defined, such as: frequency, number of cycles, applied load. From this standpoint, Wiskott and collaborators (16) stated that the minimum number of cycles to promote clinical relevant data is 106; and Fraga and collaborators (17) state that a frequency of up to 20 Hz can be used for fatigue testing of Y-TZP material, without biasing the fatique strength findings.

Hence, this current study aimed to evaluate the effect of grinding and multi-stimuli aging on the fatigue strength, surface topography and the phase transformation of a Y-TZP ceramic.

Material and Methods

Specimen Preparation

Pre-sintered zirconia blocks (Lava Frame, 3M ESPE) were shaped into discs for biaxial flexure strength testing following the guidelines of ISO-6872:2008 (18), using the methodology previously described by Pereira and collaborators (19).

Basically, as the blocks presents an rectangular shape (40 mm in length), and ISO-6872:2008 (18) requires disc-shaped specimens for biaxial flexural strength testing, two metal cylinder guides with 18 mm diameter were glued parallel in both sides of the block and then, a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, Illinois, USA) was used with 600 grit silicon carbide (SiC) papers shaping the blocks into cylinders. Afterward, the cylinders were cut under water irrigation with a diamond saw (ISOMET 1000, Buehler) in slices of 1.65 mm

saw (ISOMET 1000, Buehler) in slices of 1.65 mm thickness. Then the specimens were polished with a 1200-grit Sic paper (on both sides) and sintered according to manufacturer's recommendation, resulting in specimens with 15 mm in diameter and 1.2 mm in thickness.

Immediately after sintering, all specimens had their dimensions inspected with a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper, Takatsu-ku, Kawasaki, Kanagawa, Japan) to confirm that they presented values inside the range recommended by ISO-6872:2008 (18), and then, they were randomly allocated (n= 25) into four groups considering two factors, 'grinding' and 'aging', as described in Table 1.

Specimens from the control group remained untouched (without grinding and aging) after the sintering process – "as-sintered" specimens (group C).

Grinding (G)

Grinding was performed by a single trained operator using diamond burs (#3101G – grit size 181 µm; KG Sorensen) in a slow-speed motor (Kavo Dental, Biberach, Baden-Württenberg, Germany) coupled to a contra-angle handpiece (T2 REVO R170 contra-angle handpiece up to 170,000 rpm, Sirona, Long Island, New York, USA) under constant water-cooling (≈30 mL/min). The diamond bur was replaced after each specimen (14).

In order to standardize the wear thickness and to ensure a complete and standardized surface wear, the specimens were marked with a permanent marking pen (Pilot, Sao Paulo, Brazil) previously to the wearing. Then, the specimens were attached to a device specially designed for this aim, keeping the diamond bur and specimen surface parallel to each other. After that, the grinding procedure was performed with horizontal movements, up to the point that the marking was completely eliminated (protocol previously described by Pereira and collaborators (19)).

Aging (A)

As literature states that the LTD mechanism is a time dependent spontaneous process (6), accelerated on the presence of water, and mechanical cycling, and as the aim of this study was to combine the stimuli for LTD effects, an aging protocol by 3 consecutive steps was chosen:

1. Autoclave (more common and effective protocol to stimulate LTD as it considers moisture and temperature) (4,5); 2. Storage (aging step that would lead to progression of the LTD mechanism); and 3. Mechanical cycling (commonly used to simulate clinical loading by intermittent load application, leading to crack propagation).

Table 1. Experimental design

Material brand	Groups	Surface Treatment	Aging
Lava Frame (3M ESPE, Seefeld, Germany; lot 70201131797)	С	Control, as-sintered (without	Without
	CA	any additional treatment)	With
	G	Grinding with coarse diamond	Without
	GA	bur (3101G – grit size 181 µm, KG Sorensen, Cotia, Brazil)	With

Aging in Autoclave

The specimens were aged in an autoclave (Sercon HS1-0300 n11560389/1, Sercon & Steris Corporation, Mogi das Cruzes, Brazil) at 134°C, under 2 bar pressure, over a period of 20 h (4,5).

Storage at Room Temperature

Specimens were kept into a sealed plastic vessel, where each specimen was sided with each other without direct contact. After that, these vessels were stored at room temperature, without direct exposure to day light, for 365 days.

Mechanical Cycling

For this procedure it was used a biaxial flexural assembly (piston-on-three-balls according to ISO-6872:2008) in an electro-dynamic fatigue simulator (Instron Electro Puls E3000, Instron Corporation, Norwood, Massachusetts, USA; maximum estimated error 0.5% from the maximum load cell capacity, as we used a 5 KN load cell, it would be expected a maximum error of 25 N) under 10⁶ cycles with 20 Hz frequency and a ranging load of 20 N (minimum applied load during cycle) to the maximum of 50% of the mean (C= 432.95; CA= 490.05; G= 538.45; GA= 415) biaxial flexural strength observed in a previous study that considered each evaluated condition on same material under a biaxial flexural static test assembly (12).

For that, the specimens were positioned with the treated surface facing down (tensile stress) on three support balls (\emptyset = 3.2 mm), which were positioned 10 mm apart from each other in a triangular position. The assembly was immersed into water and a flat circular tungsten piston (\emptyset = 1.6 mm) was used to apply the load at the center of the disc. Before mechanical cycling, a film of a non-rigid material (cellophane, 2.50 µm) was placed between the supporting balls and the specimen (tensile surface) and an adhesive tape (3.50 µm) was fixed on the compression side of the discs in order to avoid the fragments to be spread (20) and to provide better contact between the piston and the specimens (18).

Surface Topography Analysis

Specimens (n= 3) from each condition received sputter coating with a gold-palladium alloy and surface topography images were obtained with 1000× magnification in a Field Emission - Scanning Electron Microscope (FE-SEM Inspect F50, FEI; Hillsboro, Oregon, USA).

Phase Analysis by X-Ray Diffraction (XRD Analysis)

Quantitative analysis of phase transformation (n= 2) was conducted, for each condition evaluated, using a x-ray diffractometer (D8 Advanced XRD, Bruker AXS

GmbH, Karlsruhe, Baden-Württenberg, Germany) with CuK_{α} radiation. Spectra were collected using the Bragg-Brentano geometry in the 2θ range from 25 to 35 degrees, at a step interval of 1 s, and step size of 0.03 degrees/step. The amount of m-phase (Xm) and the volumetric fraction (Fm) was calculated using the method developed by Garvie & Nicholson modified by Toraya and collaborators (21), extensively used and described on previous literature (4,5,13).

Flexural Fatigue Strength Testing

For this test, the same assembly described previously for mechanical cycling (ISO-6872:2008 (18)) was utilized, where specimens (n= 20) from each evaluated condition were subjected to a fatigue strength test with a lifetime of 20,000 cycles using the staircase method described by Collins (22).

Sinusoidal loading was applied, with amplitude ranging from a minimum of 20 MPa, just to avoid the movement of the specimen, to the maximum tensile applied with a frequency of 6 Hz (6 cycles per second). The initial stress level (70% of the monotonic biaxial flexural strength mean) and the step size (5% of initial strength) were determined based on the results of the monotonic biaxial tests of three specimens of each condition (n= 3) using the same testing assembly described previously for mechanical cycling (ISO-6872:2008 (18)). Then the first specimen of each group was tested and depending on the survival or failure of this specimen, the next disc was tested with a tensile increment higher or lower than the initial tensile, respectively. Thus, stress controlled all fatigue tests, and the load (N) required to achieve the desired stress (MPa) was calculated according to ISO-6872:2008 (18), for each tested specimen.

After testing, the biaxial flexure fatigue strength mean (σ f), standard deviation (SD) and 95% confidence intervals (CI α = 0.05) was calculated, according to Collins (22), based on the data of the less frequent event (survival or failure), as described on Villefort et al. (23).

Fractography Analysis

A fractography examination was performed first on all fractured specimens using a light microscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany), from which representative specimens were selected (from each group evaluated), and after, they were analyzed at the Field Emission - Scanning Electron Microscope (n= 1, FE-SEM Inspect F50, FEI; Hillsboro, Oregon, USA) to determine fracture origin.

Data analysis

Data were subjected to the Dixon and Mood method,

which involves maximum-likelihood estimation techniques for analytical solutions to the problem of determining the mean and standard deviation (22,23). This method assumes that the fatigue strength follows a normal distribution. The two statistical properties were determined using only the failures or only the runouts (survivals), depending on the least frequent event, which is determined by the lower total numbers of events that occurred for each group.

Results

The surface topography analyses (FE-SEM) shows that grinding promotes an alteration of topographical pattern, deforming the surface and introducing scratches parallel to

the movement of the grinding tool; while, aging apparently did not lead to any alteration (Fig. 1).

Data from the fatigue tests (staircase approach) in addition to X-ray Diffractometry (Fig. 2, Table 2) show that grinding triggered t-m phase transformation without impacting the fatigue strength of the Y-TZP ceramic (C = G). Regarding aging, an intense t-m phase transformation was observed, triggering the toughening mechanism and leading to higher fatigue strength for as-sintered condition, and depicting a tendency of increase on ground condition, although without reaching a statistical difference level (C < CA; C = GA). Although, it may be highlighted that different susceptibilities to t-m phase transformation were

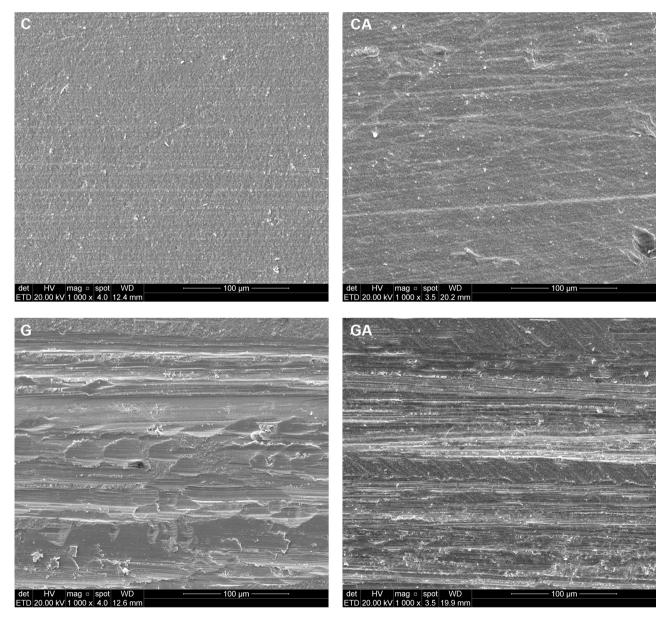


Figure 1. SEM micrographics (C = control (as-sintered); CA = control subjected to aging; G = ground; GA = ground and aged) of the surface topography (1000x magnification) of the different tested conditions, elucidating the surface alterations generated by grinding and no notable modification by aging.

observed (Table 2).

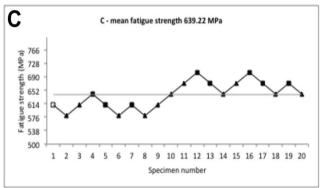
Fractography analysis (Fig. 3) shows that all fractures originated from the surface defects at the center of the side under tensile stress, which is the region with highest stress, as stated by ISO-6872:2008 (18), and that the fracture propagated into the opposite side where compression stress is concentrated (compression curl region).

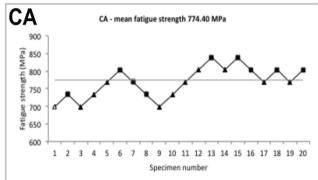
Discussion

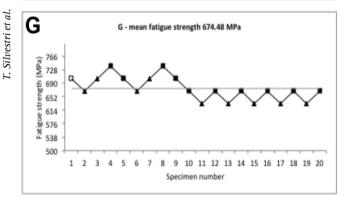
Our data support that grinding led to increase in

m-phase content, surface topography alterations, although it did not affect deleteriously the fatigue strength of the Y-TZP ceramic. Regarding aging, it observes an even higher m-phase content increase that resulted in an increase in fatigue strength for as-sintered condition, and a tendency of increase on ground condition, without promoting any topography alterations.

A recent systematic review (14) evaluated the effect of grinding on Y-TZP ceramic and demonstrated that the methodology (protocol) for grinding plays a main role in







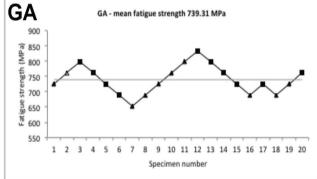


Figure 2. Pattern of runouts (survival, highlighted as \blacksquare) and failures (highlighted as \blacksquare) for each group (C = control (as-sintered); CA = control subjected to aging; G = ground; GA = ground and aged) observed during fatigue testing, where the unfilled marker represent the start of the fatigue test according to Collins, 1993.

Table 2. Monotonic biaxial strength mean, initial strength (70% of monotonic biaxial strength mean) and the step size (5% of the initial strength) in MPa for fatigue testing (staircase); fatigue strength mean (of), standard deviation (SD) and 95% confidence interval (CI) in MPa obtained from staircase tests, in addition to percentage (%) of m-phase content.

Groups	Monotonic	Initial strength		Fatigue strength in MPa		XRD Analysis
	strength mean	for fatigue test	Step size	of ± SD*	95% CI	Mean m-phase content ± SD (%)
С	872.00	610.40	30.52	639.22 ± 68.58 ^a	597.56 - 680.89	0.00
CA	998.14	698.70	34.94	774.40 ± 64.53°	733.93 - 814.88	60.47 ± 2.53
G	1003.69	702.58	35.13	674.48 ± 36.37 ^{ab}	652.16 - 696.79	13.95 ± 1.59
GA	1036.02	725.21	36.26	739.31 ± 85.83 ^{bc}	686.60 - 792.02	30.75 ± 0.55

C: control (as-sintered); CA: control subjected to aging; G: ground; GA: ground and aged. *Different letters indicate statistically significant differences, while same letters indicate no statistical difference.

defining the final outcome: if the balance tends to the introduction of defects it may be observed a negative impact (8,9,24), while if the balance favors phase transformation it could be observed the toughening mechanism (12,13,19). Our study corroborates that assumption, since we used the methodology preconized by this systematic review (14) to avoid defect introduction (handpieces coupled to slow speed motors and the use of plenty coolant) and we did not notice a deleterious effect, even using coarse diamond burs during grinding.

As stated previously, restorations clinically fail by fatigue effects (15). Previous studies about fatigue behavior of ground Y-TZP ceramics present high heterogeneity and by

that gave inconclusive results. Kosmac & Dakskobler (8) and Kosmac and collaborators (9) used a high-speed hand-piece during grinding and it leads to deleterious impact on mechanical properties. While, Pereira and collaborators (12) applied a gentle protocol of grinding, and observed no damage effect on fatigue strength even after aging in autoclave, even though a small number of cycles (20,000) had been applied for fatigue strength assessment.

On this sense, to the authors knowledge, there are only two studies on literature that considered the fatigue behavior of aged Y-TZP ceramics: Cotes and collaborators (7) did not notice any damaging effect on mechanical properties even after 15x10⁶ cycles at 200N; and Pereira

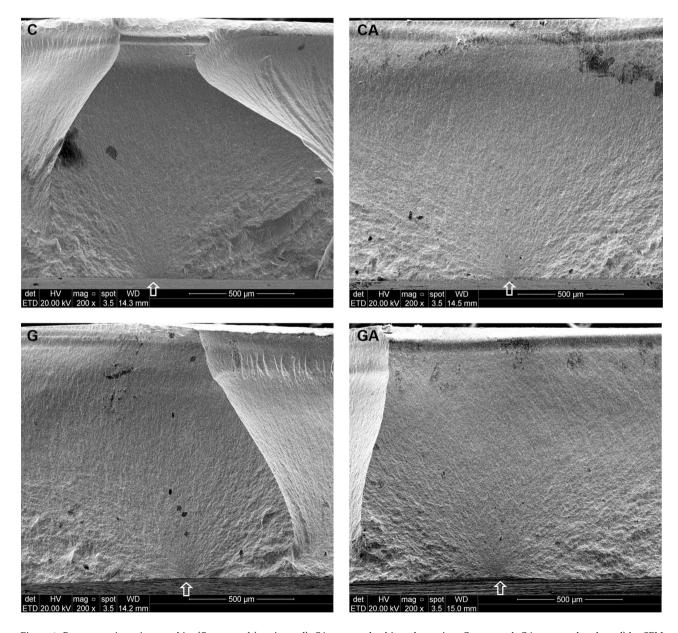


Figure 3. Representative micrographics (C = control (as-sintered); CA = control subjected to aging; G = ground; GA = ground and aged) by SEM of fractured surfaces (fractography examination). The arrow indicates crack origin at a superficial defect where concentrated tensile stress, from which the fracture propagated toward the opposite side (compression curl region).

and collaborators (12) found congruent finding, i.e. no deleterious effect even when applying an accelerated fatigue strength test. Thus, our findings are in agreement with the results depicted by those investigations.

Among the many available protocols used for inducing aging effects (LTD) on Y-TZP ceramics, Pereira and collaborators (5) showed that autoclave aging, under at least 134°C, 2 bar pressure for 20 h, is an effective way to produce and to evaluate the effects of LTD, since this aging method leads to increase of 60% in m-phase content approximately. Additionally, Chevalier and collaborators (4) stated that an autoclave cycle at 134°C, 2 bar pressure for 5h would simulate 15-20 years of exposure to environment at 37°C temperature. Besides, ISO-13356:2008 states that the monoclinic phase content should not exceed the maximum of 25%, for an Y-TZP to be considered suitable for biomedical applications, after aging at 134°C, 2 bar pressure for 5h. However, we may emphasize that a direct correlation between LTD and clinical exposure (allowing prediction of the behavior under clinical environment) is still absent on literature, so all the extrapolations should be considered with extreme caution.

Flury and collaborators (25) stated that surface roughness might play a crucial role in the resistance of ceramics, usually showing a significant negative correlation with flexural strength (the higher roughness the lower flexural strength). It is already known that grinding leads to an increase in roughness (12,13), however an extensive number of studies have been showing that the phase transformation toughening mechanism may counterbalance to some extent this effect closing this defects and even resulting in increased flexural strength (3, 12,13,19). Our data support the aforementioned statement, since no negative influence of increased roughness (grinding) on fatigue strength. In fact, it seems that for Y-TZP the phase transformation toughening mechanism triggered by grinding and aging counterbalanced the defects introduced.

In our micrographics (Fig. 1), it is clear that grinding deforms the superficial grains and change the microstructure of the surface. The current XRD analysis also shows that grinding increase m-phase content. However, during aging, the ground conditions exhibited a decreased susceptibility to new phase transformations (30.75% of m-phase), when compared with the aged condition as-sintered, which it had higher m-phase content (60.47%).

On this sense, Muñoz-Tabares and collaborators (11) stated that microstructural changes induced by grinding of Y-TZP consist of three well defined layers which are described as follows, from the surface to the interior: (1) a superficial crystallized zone, where the grains diameter range from 10 to 20 nm approximately; (2) a plastically

deformed zone; (3) a zone in which tetragonal to monoclinic phase transformation has taken place, which is mainly responsible for the formation of compressive residual stresses that usually increases the flexure strength and apparent fracture toughness of ground specimens.

Some studies (11,14) explains that the higher resistance to hydrothermal degradation (increase in m-phase content – low-temperature degradation) of ground Y-TZP would be possibly related to the existence of this very thin layer of tetragonal re-crystallized nano-grains (10-20 nm), whose size is smaller than the critical size for transformation in humid environment, in addition to the presence of residual stress.

One issue that remains unclear on literature and needs more attention on future researches is about the best alternative to proceed after an adjustment of Y-TZP surface (post-processing treatments: polishing and/or glazing and/or heat treatment). Those treatments will influence the surface microstructure and roughness acting on the defects introduced by grinding and also on the m-phase content generated by t-m phase transformation and by that they may impact the materials aging susceptibility and fatigue behavior. A recent investigation by Zucuni and collaborators (26) shows that the polishing after grinding appears to play a relevant role for fatigue behavior improvements of polycrystalline zirconia material.

It highlights that more studies should be performed to correlate the effect of in vitro simulated LTD and its respective correspondence to the clinical environment scenario. Another important question is if a higher number of cycles would lead to a deleterious effect. Thus, those issues (extrapolation to clinical environment; number of cycles) may be considered as the main limitations of our study.

As conclusion, our study shows that even after grinding under a multi-stimuli aging protocol no deleterious effect on fatique strength of Y-TZP material is noticed.

Resumo

Este estudo objetivou investigar o efeito do desgaste e envelhecimento (estímulos combinados) na resistência à fadiga, topografia superficial e transformação de fase de uma cerâmica Y-TZP. Discos para teste de flexão biaxial foram confeccionados segundo as normas da ISO-6872:2008 (15 mm Ø; 1,2 mm espessura) e randomicamente distribuídos de acordo com os fatores "desgaste" e "envelhecimento": C- controle (sinterizado); CA- controle envelhecido; G- desgaste; GA- desgaste envelhecido. O desgaste foi feito com pontas diamantadas de granulação grossa sob irrigação com água. Os protocolos de envelhecimento consistiram em: autoclave (134°C, 2 bar de pressão, 20 horas), 365 dias armazenados em temperatura ambiente, e ciclagem mecânica (106 ciclos a 20 Hz em um intervalo de carga de até 50% da carga monotônica observada em testes estáticos). A resistência à fadiga foi determinada para 20.000 ciclos à 6 Hz sob carregamento sinusoidal através do método de escada. Adicionalmente, análises de topografia superficial em microscopia eletrônica de varredura e de transformação de fase por difração de raios-X foram executadas. A metodologia de Dixon e Mood foi usada para

analisar os dados de resistência à fadiga. Foi observado que o desgaste promove uma alteração de padrão topográfico superficial; enquanto o envelhecimento aparentemente não demonstra influência. Em relação à transformação de fase e resistência à fadiga, o desgaste desencadeou um aumento de fase m sem impactar na resistência à fadiga da cerâmica Y-TZP; já o envelhecimento promoveu uma intensa transformação de fase, resultando no mecanismo de tenacificação, que gerou um aumento na resistência à fadiga para a condição sinterizada e uma tendência a aumento na condição desgaste (C < CA; G = GA). Os dados elucidam que o desgaste e o envelhecimento não impactaram negativamente na resistência à fadiga da cerâmica Y-TZP, apesar de promoverem alterações de topografia superficial e intensa transformação de fase de t-m respectivamente.

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