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Camila Pauleski Zucuni

COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP: EFEITO DE TRATAMENTOS DE SUPERFÍCIE E TÉCNICAS DE PROCESSAMENTO

Santa Maria, RS
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Dissertação apresentada ao Programa de Pós-Graduação em Ciências Odontológicas da Universidade Federal de Santa Maria (UFSM, RS). Área de Concentração em Odontologia, ênfase em Prótese Dentária, como requisito parcial para a obtenção do título de **Mestre em Ciências Odontológicas**.

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(Martin Luther King Jr.)

RESUMO

COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP: EFEITO DE TRATAMENTOS DE SUPERFÍCIE E TÉCNICAS DE PROCESSAMENTO

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A presente dissertação está composta por três estudos com respectivos objetivos: estudo 1- avaliar o efeito de tratamentos de superfície (polimento, tratamento térmico e glaze), associados ou não ao envelhecimento em autoclave (LTD), na micromorfologia de superfície, na transformação de fase (*t-m*), na resistência à flexão biaxial e na confiabilidade estrutural (Análise de Weibull) de uma cerâmica Y-TZP desgastada por ponta diamantada; estudo 2- avaliar o efeito de diferentes tratamentos de superfície e de suas associações (polimento, tratamento térmico, glaze, polimento + tratamento térmico, polimento + glaze) na micromorfologia de superfície, na transformação de fase (*t-m*) e no comportamento em fadiga de uma cerâmica Y-TZP desgastada por ponta diamantada; estudo 3- avaliar o efeito de diferentes métodos de confecção de amostras pré-sinterizadas (usinagem em CAD/CAM x técnicas de confecção em laboratório) nos desfechos acima mencionados. Para os três estudos, discos de cerâmica Y-TZP foram confeccionados conforme ISO 6872/2008. No estudo 1, os espécimes foram alocados em 10 grupos de acordo com o fator 'tratamento de superfície'(*Ctrl*: sem tratamento; *Ctrl + Aut*: sem tratamento e envelhecido; *Gr*: desgastado; *Gr + Aut*: desgastado e envelhecido; *Gr + HT*: desgastado e tratado termicamente; *Gr + HT + Aut*: desgastado, tratado termicamente e envelhecido; *Gr + Pol*: desgastado e polido; *Gr + Pol + Aut*: desgastado, polido e envelhecido; *Gr + Gl*: desgastado e glazeado; *Gr + Gl + Aut*: desgastado, glazeado e envelhecido e o fator'envelhecimento' (com ou sem). No estudo 2, os espécimes foram alocados em 7 grupos de acordo com o tratamento de superfície: *Ctrl*: sem tratamento; *Gr*: desgastado; *Gr + HT*: desgastado e tratado termicamente; *Gr + Pol*: desgastado e polido; *Gr + Pol + HT*: desgastado, polido e tratado termicamente; *Gr + Gl*: desgastado e glazeado; *Gr + Pol + Gl*: desgastado, polido e glazeado. Para o estudo 3, os espécimes cerâmicos foram divididos em 5 grupos: *Polished*: grupo polido (confecção convencional); *Machined*: usinados pelo sistema CAD/CAM; grupos *Fine*, *XFine* e *SiC*: discos obtidos em máquina de corte e desgastados com ponta diamantada fine, com ponta diamantada xfine, e com lixa de carbeto de silício, respectivamente. Todos os desgastes do estudo 3 foram realizados antes da sinterização. Em relação ao estudo 1, observou-se que o polimento gerou maior resistência característica quando comparado aos demais tratamentos de superfície, além de ter reduzido a rugosidade. Esses dados corroboram com os resultados do estudo 2: polimento gerou maior resistência a fadiga. De acordo com o estudo 3, o desgaste com ponta diamantada de granulação fina (46µm) previamente a sinterização, apresentou o maior potencial em mimetizar tanto as características superficiais, quanto o comportamento mecânico gerado pela usinagem CAD/CAM.

Palavras-chave: CAD/CAM. Degradação hidrotérmica. Envelhecimento em baixa temperatura. Fadiga. Resistência à flexão. Tratamento de superfície. Zircônia policristalina parcialmente estabilizada por óxido de ítrio.

ABSTRACT

MECHANICAL BEHAVIOR OF A Y-TZP CERAMIC: EFFECT OF SURFACE TREATMENTS AND PROCESSING TECHNICS

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ADVISOR: LUIZ FELIPE VALANDRO

This study is composed by three studies with respective goals: 1st study- to evaluate the effect of post-processing surface treatments (polishing, heat treatment/annealing and glazing), associated or not with autoclave aging (LTD– Low Temperature Degradation) on the surface micro-morphology, phase transformation (*t-m*) and mechanical behavior (flexural strength and structural reliability) of a Y-TZP ceramic; 2nd study- to evaluate the effect of post-processing surface treatments and associations (polishing, heat treatment, glazing, polishing + heat treatment and polishing + glazing) on the surface micro-morphology, phase transformation (*t-m*) and fatigue behavior of Y-TZP ceramic; 3rd study- to evaluate the effect of different pre-sintering fabrication processing techniques (CAD/CAM milling x in-lab fabrication processing techniques) on the outcomes aforementioned. For three studies, discs of Y-TZP ceramic were manufactured according to ISO: 6872-2008. To the first study, the specimens were divided into 10 groups according to the aging (with or without) and surface treatment (*Ctrl*; "as-sintered"; *Ctrl + Aut*: 'as-sintered" + aging; *Gr*: ground with diamond bur; *Gr + Aut*: ground with diamond bur + aging; *Gr + HT*: ground with diamond bur + heat treatment; *Gr + HT + Aut*: ground with diamond bur + heat treatment+ aging; *Gr + Pol*: ground with diamond bur + polishing; *Gr + Pol + Aut*: ground with diamond bur + polishing + aging; *Gr + Gl*: ground with diamond bur + glazing; *Gr + Gl + Aut*: ground with diamond bur + glazing + aging. To the second study, the specimens were divided into 7 groups: *Ctrl*: "as-sintered"; *Gr*: ground; *Gr + HT*: ground and heat treatment; *Gr + Pol*: ground and polishing; *Gr + Pol + HT*: ground, polishing and heat treatment; *Gr + Gl*: ground and glazing; Gr + Pol + Gl: ground, polishing and glazing. To the third study, the specimens were divided into 5 groups: *Polished*: polishing group (conventional process); *Machined*: CAD/CAM milling; *Fine*, *Xfine* and *SiC*: fabrication using a cutting machine followed by grinding with diamond bur fine, xfine and 220-grit silicon carbide, respectively. All of the grinding procedures of the third study were performed before sintering. Regarding the first study, it observed that polishing generated higher characteristic strength than the other surface treatments and reduced the surface roughness. These findings corroborate to ones from the second study: the polishing promoted higher fatigue strength. According to the third study, the grinding with fine diamond burs (46µm) before sintering showed to be the best protocol to mimic the surface and the mechanical behavior of Y-TZP ceramic milled by CAD/CAM.

Key words: CAD/CAM. Fatigue. Flexural strength. Hydrothermal degradation. Low temperature aging. Surface treatments. Zirconium oxide partially stabilized by yttrium.

SUMÁRIO

1. INTRODUÇÃO GERAL

As cerâmicas à base de zircônia tetragonal policristalina parcialmente estabilizada com 3mol% de ítrio (Y-TZP) têm sido indicadas como infraestrutura de próteses dentais fixas, assim como restaurações monolíticas (KIM et al., 2010), devido a sua biocompatibilidade (XIE et al., 2016), maior resistência à flexão e tenacidade à fratura quando comparada a todas as outras cerâmicas odontológicas disponíveis atualmente (DENRY & KELLY, 2014).

A zircônia é um polimorfo que ocorre na natureza sob três formas cristalinas, cada qual relacionada primariamente à temperatura em que se encontram. São elas (com os valores mencionados de temperatura referentes à zircônia pura): monoclínica (m), desde a temperatura ambiente até 1170°C; tetragonal (t), para temperaturas acima de 1170°C; e cúbica, quando a temperatura se eleva acima de 2370°C (PICONI & MACCAURO, 1999). Para que a zircônia se mantenha na fase tetragonal à temperatura ambiente, alguns óxidos estabilizadores são adicionados, tais como: CaO, MgO, CeO_2, Y_2O_3 (ALGHAZZAWI et al., 2012).

Porém, mesmo com a adição de óxidos que estabilizem a fase tetragonal em temperatura ambiente, a zircônia pode sofrer transformação de fase (*t-m*) quando submetida a estímulos físico-químicos, como procedimentos de desgaste e jateamento, e pela degradação em baixas temperaturas (*Low Temperature Degradation* - LTD) (DENRY & KELLY, 2008; CHEVALIER & GREMILLARD, 2009). A transformação de fase (*t-m*) também está associada a outros fatores, tais como: tipo e concentração do óxido estabilizador, tamanho do grão da cerâmica (CHEVALIER & GREMILLARD, 2009) e condições de sinterização (DENRY & KELLY, 2008).

A transformação de fase (*t-m*) é acompanhada por um aumento restrito de volume de aproximadamente 3-5% nas regiões de trincas (KOSMAC, 1999), pois os grãos monoclínicos são maiores em volume que os grãos tetragonais (HJERPPE et al., 2016). Este aumento de volume ao redor da trinca está associado ao desenvolvimento de uma camada de tensão compressiva, que dificulta a propagação da trinca para o interior do material, melhorando as propriedades mecânicas da zircônia (DENRY & HOLLOWAY, 2006; GARVIE, HANNINK e PASCOE, 1975).

Como mencionado anteriormente, a zircônia é susceptível à LTD quando exposta a um ambiente úmido (CHEVALIER, CALES e DROUIN, 1999). A LTD é dependente do tempo, sendo que ocorre mais rapidamente em temperaturas entre 200-300°C (CHEVALIER, CALES e DROUIN 1999). O processo de envelhecimento é considerado indesejável, pois o excesso de tensões residuais geradas pela transformação de fase também pode levar a introdução de micro-trincas, que podem comprometer as propriedades mecânicas da cerâmica ao longo do tempo (ALGHAZZAWI et al., 2012; PAPANAGIOTOU et al., 2006).

Dentre os métodos utilizados para a fabricação de restaurações cerâmicas, os sistemas CAD/CAM (*Computer Aided Design-Computer Aided Machining*) vêm sendo utilizados cada vez mais, pois apresentam como vantagens: maior precisão, eficácia e acurácia, redução no tempo de trabalho e menor possibilidade de erro para a confecção destas restaurações quando comparadas às restaurações confeccionadas de forma convencional (manual) (MIYAZAKI et al., 2009).

Porém, apesar da alta precisão deste sistema de processamento, geralmente ajustes da restauração se fazem necessários, os quais usualmente são executados com instrumentos diamantados acoplados a motores de baixa/alta rotação, visando o aprimoramento da anatomia final, adaptação marginal, perfil de emergência e relações oclusais (PREIS et al., 2015[a]). A literatura mostra que este procedimento pode desencadear dois principais eventos: 1- um aumento dos valores de resistência à flexão através do desenvolvimento de uma camada de tensão residual superficial (LUTHARD et al., 2004); 2- a introdução de diferentes tipos de danos, como trincas, que variam em profundidade e que podem penetrar em direção ao interior do material, o que comprometeria as propriedades mecânicas da cerâmica (PEREIRA et al., 2014; PREIS et al., 2015[a]; KOSMAC et al., 1999; GUAZZATO et al., 2005); além de gerar a perda do glaze e da lisura superficial (PREIS et al., 2015[a]).

De acordo com as recomendações dos fabricantes, após a execução de ajustes que visam restabelecer a integridade (remoção dos danos introduzidos), o brilho e a lisura superficial, torna-se necessário a realização de um protocolo de polimento e/ou a aplicação de uma fina camada de porcelana vítrea de baixa fusão (*glaze*) e/ou a realização de um tratamento térmico específico. Ainda não está estabelecido na literatura qual o tratamento de superfície mais adequado para ser realizado após a execução do desgaste, visto que cada tipo de tratamento interage através de diferentes tipos de mecanismos com a superfície cerâmica, e dessa forma, podem influenciar positiva ou negativamente as propriedades mecânicas do material.

Outro importante aspecto que dificilmente é levado em consideração por trabalhos *in vitro* que estudam materiais à base de Y-TZP, é a simulação da superfície usinada pelo CAD-CAM. A porção interna de restaurações obtidas por usinagem CAD/CAM apresenta uma rugosidade e topografia superficial decorrente do desgaste pela brocas usadas durante a usinagem, sendo que, ao contrário da superfície externa, estas não podem passar por etapas de acabamento e polimento, pois isto desencadearia uma desadaptação da peça protética, além de ser operacionalmente difícil. Nesse ínterim, sabe-se que diferentes rugosidades e parâmetros de topografia superficial podem afetar o comportamento mecânico do material, principalmente quando estas estão localizadas em regiões de concentração de tensão.

Levando em consideração que a confecção de espécimes para estudos *in vitro* pela usinagem com sistemas CAD/CAM torna-se muito onerosa, pois em geral, com um bloco de zircônia é possível produzir um ou dois espécimes, dependendo das dimensões do bloco pré-sinterizado (ADDISON et al., 2012; FRAGA et al., 2015), alguns estudos têm utilizado técnicas simplificadas de confecção, para produzir maior número de amostras cerâmicas, mas que não simulam a superfície gerada pela usinagem (AMARAL et al., 2013; SOUZA et al., 2013; OZCAN et al., 2013; PEREIRA et al., 2016; HJERPPE et al., 2016; GUILARDI et al., 2017). Segundo Wang et al., (2008), a usinagem de blocos pré-sinterizados gera danos na superfície da cerâmica, que podem reduzir sua resistência a fratura.

Tendo em vista que a forma de obtenção de espécimes irá gerar danos no material, e que estes poderão interferir no desempenho mecânico do mesmo, torna-se imprescindível o uso de um método de confecção de amostras em laboratório que possa simular com mais acuidade a superfície gerada pela usinagem em sistema CAD/CAM, para que a avaliação das propriedades mecânicas de cerâmicas Y-TZP seja realizada de forma mais consistente.

Com base nos conhecimentos acima expostos, o presente trabalho apresenta como objetivos:

- **Estudo 1 (*Polishing of ground zirconia is mandatory for improving the mechanical behavior of polycrystalline zirconium oxide ceramics*):** avaliar os efeitos do polimento, do tratamento térmico (*annealing*) e da aplicação de *glaze* na micromorfologia de superfície, na transformação de fase (*t-m*), na resistência à flexão biaxial e na confiabilidade estrutural (Análise de *Weibull*) de uma cerâmica Y-TZP desgastada por ponta diamantada de granulação grossa;

-**Estudo 2 (*Fatigue strength of yttria-stabilized zirconia polycrystals: Effects of grinding and post-processing treatments*):** avaliar os efeitos do polimento, tratamento térmico (*annealing*), aplicação de *glaze* e da associação destes tratamentos (polimento + glaze e polimento + tratamento térmico) na micromorfologia de superfície, na transformação de fase (*t-m*) e na resistência a fadiga de uma cerâmica Y-TZP desgastada por ponta diamantada de granulação grossa;

- **Estudo 3 (*CAD-CAM Machining Vs. pre-sintering in-lab fabrication techniques of Y-TZP ceramic specimens: effects on their mechanical fatigue behavior*):** avaliar e comparar o efeito de diferentes métodos de confecção de amostras pré-sinterizadas (usinagem em CAD/CAM x técnicas de confecção em laboratório), na micromorfologia de superfície, na transformação de fase (*t-m*), na resistência à flexão biaxial, no comportamento em fadiga e na confiabilidade estrutural (análise de *Weibull*).

Para efeitos de apresentação, esta Dissertação está apresentada sob a forma de três artigos:

ARTIGO 1 - Polishing of ground zirconia is mandatory for improving the mechanical behavior of polycrystalline zirconium oxide ceramics

Submetido para "*Applied Surface Science*" – Fator de Impacto: 3.150

ARTIGO 2 - Fatigue strength of yttria-stabilized zirconia polycrystals: Effects of grinding and post-processing treatments

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ARTIGO 3 - CAD-CAM Machining Vs. pre-sintering in-lab fabrication techniques of Y-TZP ceramic specimens: effects on their mechanical fatigue behavior

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2. ARTIGO 1 - Polishing of ground zirconia is mandatory for improving the mechanical behavior of polycrystalline zirconium oxide ceramics

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Running title: Post-processing treatments and aging of ground Y-TZP.

ABSTRACT

This study evaluated and compared the effect of post-processing surface treatments (polishing, heat treatment/annealing and glazing) on the superficial characteristics (micromorphology and roughness), structural stability (phase transformation and susceptibility to aging - low-temperature degradation) and mechanical behavior (flexural strength and structural reliability) of a ground Y-TZP ceramic. Discs of Y-TZP ceramic (VITA In-Ceram YZ) were manufactured (ISO: 6872-2008; final dimensions of 15 mm diameter and 1.2±0.2 mm thickness) and randomly assigned to 10 groups based on the factors "aging" (with and without) and "surface treatment" (Ctrl – as-sintered; Gr – ground with coarse diamond bur; Gr + HT – ground and heat treatment; Gr + Pol – ground and after polished; Gr + Gl – ground and glazing). Roughness (n=30), biaxial flexure (n=30), phase transformation (n=2), and surface topography (n=2) evaluations were performed. The roughness analysis showed that the treatments increased the roughness values (Ra and Rz parameter) when compared to the as-sintered samples, even though a smoothening effect was noticed after polishing and glazing. Aging led to an intense increase in m-phase content for all tested conditions, even though the highest susceptibility to t-m phase transformation was observed for the as-sintered samples. Aging did not deleteriously change the major evaluated outcomes (roughness, structural reliability and characteristic strength). No treatment reduced the Weibull modulus for all conditions evaluated. Additionally, heat treatment and glazing after grinding led to a decrease in characteristic strength, while polishing presented the highest characteristic strength values. Thus, polishing is mandatory after grinding the Y-TZP ceramic, while performing glazing or heat-treatment alone after grinding lead to the worst mechanical performance.

Key words: Dental Ceramics. Zirconium oxide partially stabilized by yttrium. Glazing. Annealing. Grinding. Weibull Analysis.

1. Introduction

All-ceramic restorations have been widely used due to their superior aesthetics and biocompatibility (Matsuzaki et al., 2015). When considering the existing ceramics, yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) has been noted for its superior fracture toughness and has been recommended for manufacturing prosthetic frameworks of fixed dental prosthesis (FDPs), which are then covered by feldspathic porcelain (Denry & Kelly, 2014), as well as for *full-contour* monolithic restorations, where coverage by feldspathic porcelain is not necessary (Denry & Kelly, 2014).

Zirconia is a polymorphic metastable material (Piconi & Maccauro, 1999) that triggers a phase transformation mechanism (tetragonal – *t* to monoclinic – *m*) in response to different stimuli (e.g. stress concentration or exposure to environment with humidity). This transformation results in a restricted volumetric expansion (3%–5%) around the defect, which creates a compressive stress layer at the crack origin and actively restricts crack propagation (Hannink et al., 2000).

However, surface roughness increases as that phase transformation mechanism progresses, primarily on superficial grains with water incorporation into the open spaces and spreading to the surface. This increased surface roughness leads to a decrease in hardness, fracture toughness and density of zirconia, a process known as low-temperature degradation - LTD (Kobayashi et al., 1981; Chevalier et al., 2007; Pereira et al., 2015[b]).

It is important to consider that clinical adjustments (with diamond grinding instruments) are usually needed to achieve a better fit, create an adequate emergence profile, or to improve the occlusal relations for both recommended restorative applications of Y-TZP ceramic (infrastructure of FDPs or *full-contour* monolithic restorations) after the restorations have been manufactured by CAD/CAM systems (Computer-Aided Design / Computer-Aided Machining) (Aboushelib et al., 2009; Preis et al., 2015[b]; Jing et al., 2014; Pereira et al., 2016[c]).

Thus, when the Y-TZP surface is ground, clinicians need to be aware that superficial defects are introduced and *m-phase* content will appear (Kosmac et al., 2007; Pereira et al., 2014;Pereira et al., 2016[b]). The relation between the size of the resulting scratches/cracks and the depth of the transformed layer determines the crack growth and whether the material will fail at loads below the critical load value (Zhang et al., 2006; Kosmac et al., 2008; Pereira et al., 2016[c]).

After grinding, manufacturers usually recommend a polishing protocol, which should be executed to achieve a smooth and shining surface (Goo et al., 2016; Preis et al., 2015[a]); and/or a glazing procedure, i.e., application of a thin layer of glaze (silica-based amorphous material) into the surface to promote a smooth and shining surface (Manjuran et al., 2014); and/or a heat treatment/annealing that consists generally in subjecting the zirconia to a specific heat treatment, with temperatures around 1000 ºC for 15 min, with an aim for complete m-t phase reversion (Guazzato et al., 2005).

The literature has shown some advantages for each aforementioned post-processing treatment related to polishing (surface smoothening effect and decreased wear) (Preis et al, 2015[b]; Schmitter et al., 2015; Manjuram et al., 2014), glazing (decrease in roughness and in biofilm formation) (Haralur, 2012), and heat treatment (it leads to m-t reverse transformation) (Guazzato et al., 2005), even though data regarding how those post-processing treatments affect the mechanical performance of Y-TZP surface are scarce. From these points of view, it is clinically relevant to investigate the procedures to be performed after grinding the zirconia surface, since as those procedures might affect the mechanical properties, fatigue behavior and bacterial adhesion.

Thus, based on the absence of studies that may help clinicians to define a gold-standard finishing protocol for maintaining the surface quality, smoothness, and structural stability, this study evaluated and compared the effects of polishing, heat treatment/annealing and glazing on the superficial characteristics (micromorphology and roughness), structural stability (phase transformation and susceptibility to aging – low-temperature degradation) and mechanical behavior (flexural strength and structural reliability – Weibull analysis) of a ground Y-TZP ceramic.

2. Materials and Methods

2.1. Specimen Preparation

Disc shaped specimens (final dimensions of 15 mm in diameter and 1.2 ± 0.2 mm in thickness) were manufactured from Y-TZP ceramic blocks (VITA In-Ceram YZ for inLab YZ-40/19, 40x19x15 mm^3, VITA Zahnfabrik, Bad Sackingen, Germany), according to ISO 6872-2008 guidelines for ceramic testing.

The ceramic blocks were detached from the metal holder and 18mm cylindrical metal rings were carefully fixed to both extremities of the block, caution was taken to keep both rings parallel. The block was then ground using 600 grit silicon carbide paper attached to a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA) until a cylindrical form was achieved. The cylinders were then sectioned under water irrigation using a diamond saw (ISOMET 1000, Buehler), polished with 1200 grit silicon carbide paper and sintered according to the manufacturer's recommendations (Zyrcomat T, Vita Zahnfabrik).

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 guarantee that they presented values inside the range recommended by ISO 6872-2008 for ceramic biaxial flexure strength testing (diameter of 12-15 mm; thickness of 1.2 ± 0.2 mm). The samples were cleaned in an ultrasonic bath (1440 D – Odontobras, Ind. E Com. Equip. Méd. Odonto. LTDA, Ribeirao Preto, Brazil) using 78% isopropyl alcohol for 5 min, and randomly allocated to ten groups according to the factors under study (Table 1).

2.2. Surface Treatments

Samples from the control group (Ctrl) remained untouched after the sintering process – "as-sintered" samples.

2.2.1. Grinding

Grinding was performed by a single trained operator using diamond burs (#3101G – grit size 181 µm; KG Sorensen, Cotia, Brazil) in a slow-speed motor (Kavo Dental, Biberach, Germany) associated with a contra-angle handpiece (T2 REVO R170 contra-angle handpiece up to 170,000 rpm, Sirona, Bensheim, Germany) under constant water-cooling (≈30mL/min). Samples were attached to a device that guaranteed parallelism between the surface to be ground and the diamond bur. The diamond bur was replaced after each specimen (Guilardi et al., 2017).

For standardization of the wear thickness and to guarantee that the entire surface was ground, the specimens were marked with a permanent marking pen (Pilot, São Paulo, Brazil). Then, the grinding procedure was performed manually, using oscillatory movements, up to the point that the marking was completely eliminated (Guilardi et al., 2017).

2.2.2. Polishing

Polishing was executed using the Eve Diapol ceramic polishing kit (Eve Ernst Vetter, Pforzheim, Germany), which uses rubber tips in three granulations (blue – coarse grit size; pink – medium grit size; grey – fine grit-size), coupled to an electric motor (W&H Perfecta 300, W&H Dentalwerk, Bürmoos, Austria) that allows control of speed (12,000 rpm). Samples were attached to a device that guaranteed parallelism between the surface to be polished and the polishing tips.

For standardization, each specimen was divided into 7 regions (considering the size of the active point of the rubber tip) and then polishing was executed moving the tip forward and backwards (whole specimen extension) for approximately 7 seconds in each region of the specimen (total of approximately 49 seconds for each tip per specimen).

2.2.3. Heat treatment/Annealing

The heat treatment/annealing was executed as recommended by the manufacturers using a Vacumat 600MP furnace (Vita Zahnfabrik): initial temperature of 500 °C, heating time of 5 min, heating rate of 100 per minute, and maintenance of the final temperature of 1000 °C for 15 min.

2.2.4. Glazing

A thin layer of glaze material (Vita Akzent, Vita Zahnfabrik) was applied to the ground surface, following the manufacturer's instructions. The powder was mixed with distilled water until a homogeneous paste was achieved. The paste was then applied to the surface using a specific brush. After drying, the specimens were submitted to glaze firing in the Vacumat 600MP furnace (Vita Zahnfabrik): maintaining the initial temperature of 500 °C for 4 min, heating time of 5 min, heating rate of 80°C/min, and maintaining the final temperature of 900 °C for 1 min.

2.3. Low-temperature aging

Low-Temperature Degradation (LTD) was simulated using an autoclave (Sercon HS1-0300 n11560389/1, Mogi das Cruzes, Brazil) at 134°C, under a 2 bar pressure, over a period of 20 h (Chevalier et al., 2007; Pereira et al., 2015[a]).

2.4. Surface topography and roughness analysis

For the qualitative and quantitative determination of the surface topography pattern for each evaluated condition, specimens were analyzed in a surface roughness tester (n=30, Mitutoyo SJ-410, Mitutoyo), scanning electron microscope (SEM) (n=2, FE-SEM Inspect F50, FEI, Hillsboro, Oregon, USA), and atomic force microscope (AFM) (n=2, Agilent Technologies 5500 equipment, Chandler, Arizona, USA).

For surface roughness analysis, six measurements (measured range up to 80 μm with an expected accuracy of 0.001μm) were conducted for each specimen (3 along the grinding direction, 3 in the perpendicular direction), according to the ISO:1997 parameters (Ra – arithmetical mean of the absolute values of peaks and valleys measured from a medium plane (μm) and Rz – average distance between the five highest peaks and five major valleys found in the standard (μm)) with a cut-off (n=5), λC 0.8 mm and λS 2.5 μm. Arithmetic mean values of all measurements from each specimen were obtained.

For scanning electron microscopy, two specimens from each group were submitted to sputter-coating with a gold-palladium alloy and images were obtained at a 5000x magnification.

For atomic force microscopy, two specimens from each group were submitted to the analysis and images were obtained using a non-contact methodology and specific probes of a 10 x 10µm area (PPP-NCL probes, Nanosensors, Force constant = 48 N/m) and manipulation with a specific computer software (Gwyddion™ version 2.33, GNU, Free Software Foundation, Boston, MA, USA).

Prior to the surface topography analysis, all specimens were cleaned in an ultrasonic bath as described previously.

2.5. Phase analysis (X-Ray Diffractometry - XRD)

Quantitative analysis of phase transformation was conducted (n=2) to determine the relative amount of *m*-phase and depth of the transformed layer for each condition. The analysis was performed using an x-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany). Spectra were collected in the 2θ range of 25–35° at a step interval of 1 s and step size of 0.03°. The amount of m-phase (X_M) was calculated using the method developed by Garvie & Nicholson (1972):

$$Xm = \frac{(\bar{1}11)M + (111)M}{(\bar{1}11)M + (111)M + (111)T} \qquad \text{Eq. (1)}$$

where: $(\bar{1}11)_M$and $(111)_M$ represent the monoclinic peaks (2θ=28° and 2θ=31.2°, respectively) and $(111)_T$ indicates the intensity of the respective tetragonal peak (2θ=30°). The volumetric fraction (F_m) of the m-phase was calculated according to Toraya and collaborators (1984):

$$Fm = \frac{1.311 \cdot Xm}{1 + 0.311 \cdot Xm} \qquad \text{Eq. (2)}$$

The depth of the transformed layer (TZD) was calculated on the basis of the amount of the *m*-phase, considering that a constant fraction of grains had symmetrically transformed to m-phase along the surface, as described by Kosmac and collaborators (1981):

$$TZD = \left(\frac{sen\theta}{2\mu}\right)\left[\ln\left(\frac{1}{1-Fm}\right)\right] \qquad \text{Eq. (3)}$$

where θ=15° (the angle of reflection), µ=0.0642 is the absorption coefficient, and F_M is the amount of m-phase obtained using Eqs. (1) and (2).

2.6. Biaxial flexure test

The biaxial flexure test was performed by a single trained operator. Samples (n=30) were subjected to a biaxial flexure strength test according to ISO:6872-2008. Disc-shaped specimens were positioned with the treated surface facing down (tensile stress) on three support balls (Ø=3.2 mm), which were placed 10 mm apart from each other in a triangular position. The assembly was immersed in water and a flat circular tungsten piston (Ø=1.6 mm) was used to apply an increasing load (1 mm/min) until catastrophic failure using a universal testing machine (EMIC DL 2000, São José dos Pinhais, Brazil). Before testing, adhesive tape was fixed on the compression side of the discs to avoid spreading the fragments (Quinn, 2007) and also to provide better contact between the piston and the sample (Wachtman et al., 1972). Flexural strength was calculated according to ISO:6872-2008:

$$\sigma = -0.2387 \cdot \frac{p(X-Y)}{b^2} \qquad \text{Eq. (4)}$$

where σ is the maximum tensile stress (MPa), P is the total load to fracture (N), b is the thickness at fracture origin (mm), and X and Y are calculated according to:

$$X = (1+v)\ln\left(\frac{r_2}{r_3}\right)^2 + \left[\frac{(1-v)}{2}\right]\left(\frac{r_2}{r_3}\right)^2 \qquad \text{Eq. (5)}$$

$$Y = (1+v)\left[1+\ln\left(\frac{r_1}{r_3}\right)^2\right] + (1-v)\left(\frac{r_1}{r_3}\right)^2 \qquad \text{Eq. (6)}$$

where ν is Poisson's ratio (ν = 0.25), $r_1$is the radius of the support circle (5 mm), r_2 is the radius of the loaded area (0.8 mm), and r_3 is the radius of the specimen (7.5 mm).

2.7. Data Analysis

As roughness data (Ra and Rz) assumed a nonparametric distribution (tested by Shapiro-Wilk normality test), Kruskal-Wallis and the post-hoc Dunn's tests were performed, in addition to the Spearman Correlation test between the Ra and Rz roughness data and biaxial flexural data.

The statistic used to describe the reliability of the ceramic material was based on the Weibull statistical analysis (Weibull, 1951), which is a way to describe the variation of resistance obtaining the Weibull modulus (m) and the characteristic strength (σ_C) with a confidence interval of 95%, as determined in a diagram according to DIN ENV 843-5(2007):

$$\ln\ln\left(\frac{1}{1-F}\right) = m\ln\sigma_c - m\ln\sigma_0 \qquad \text{Eq. (7)}$$

where F is the failure probability, σ_0 is the initial strength, σ_c is the characteristic strength, and m is the Weibull modulus. The characteristic strength is considered to be the strength at a failure probability of approximately 63%, with the Weibull modulus used as a measure of the distribution of strengths, expressing the reliability of the material.

2.8. Fractography analysis

A fractography examination was performed by a single trained operator using a light microscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany), where representative specimens for each evaluated condition were chosen and submitted to further evaluation in a Scanning Electron Microscope to determine the origin of the fracture.

Additional SEM analysis (n=5) was made on fractured specimens to check the mean average thickness of the glaze layer.

3. Results

SEM and AFM analysis showed that grinding produces a more irregular surface, introducing scratches following the direction of the grinding movement. Heat treatment did not promote any noticeable topographical alteration, resulting in a surface that was similar to the ground samples. Polishing and glazing result in a more regular surface, demonstrating a smoothening potential. Aging did not promote any relevant effect on topography (Figure 1). Additionally, glazing presented an average thickness ranging from 30-55μm (Fig. 2).

Roughness analysis (Ra and Rz parameters) showed that there was a statistically significant increase in roughness after grinding; heat treatment/annealing did not affect surface roughness, while polishing and glazing lead to a decrease in roughness when compared to the ground condition. However, polishing and glazing did not re-establish the roughness observed in the as-sintered condition (Ctrl). Generally, aging did not affect the surface roughness, although ground aged specimens demonstrated smaller roughness values (Table 2).

The Spearman correlation tests between roughness (Ra and Rz parameters) and biaxial flexure strength parameters (Table 2) demonstrated coefficient values smaller than 0.4 ($0.0 < (r) < 0.4$). According to Crespo (1997), this range of coefficients indicates very weak/weak correlations (very weak $0.0 < (r) < 0.3$; weak $0.3 < (r) < 0.6$).

XRD analysis showed that the m-phase was present in all evaluated conditions, except for the as-sintered (very small quantity, 1.4%) and heat treatment/annealing after grinding (Gr + HT) groups. Thus, grinding led to an increase in m-phase content, which was reverted in a small scale under polishing and glazing, and was completely reverted under heat treatment/annealing. Aging led to an intense increase in m-phase content under all evaluated conditions, although different susceptibilities to t-m phase transformation were observed (Table 2).

Weibull analysis demonstrated higher characteristic strength values for ground and ground + polished conditions and decreased characteristic strength for heat treatment and glazing. Aging in an autoclave differently influenced each evaluated condition, although no deleterious effect was observed for structural reliability (Weibull moduli) (Table 2).

Fractography analysis showed two patterns of fracture. For the glazed groups, the fractures originated at the interface between the glaze and Y-TZP or in defects (bubbles) presented in the glaze layer at the side of the specimen subjected to tensile stress (treated surface) at the center region. For the other groups, the fracture started at superficial defects on the side subjected to tensile stress (treated surface) at the center region (Fig. 3).

4. Discussion

Based on our data, polishing appears to be mandatory after grinding Y-TZP ceramic, as it promotes a decrease in roughness (smoother topography), provides the highest characteristic strength, and demonstrates the lowest susceptibility to t-m phase transformation during aging in an autoclave (less prone to LTD).

SEM micrographs demonstrated that, since Y-TZP ceramic exists as a polycrystalline material without any glass content (Denry & Kelly, 2014), there was no observed healing effect (decrease in defect size) due to heat treatment; in fact, the defects introduced by grinding and the resulting topography were maintained after heat treatment, which is in accordance with previous studies (Çaglar & Yanikoglu, 2016; Fonseca et al., 2014). Alternatively, the heat treatment affected the m-phase content, promoting a complete reversal of m-phase back to t-phase configuration (Guazzato et al., 2005; Çaglar & Yanikoglu, 2016). As a result, the defects introduced by grinding act as stress concentration factors and lead to a higher risk of failure under smaller loads (Kosmac et al., 1999; Kosmac et al., 2007; Ramos et al., 2016; Çaglar & Yanikoglu, 2016). Thus, as expected, a decrease in characteristic strength after heat treatment was found in the current study.

Polishing and glazing demonstrated a smoothening effect (to some extent), although they did not lead to similar roughness values when compared to the as-sintered condition. This may indicate that the defects introduced by grinding were partially removed by those treatments. Additionally, a slight decrease in m-phase content by polishing and glazing was noted when compared to the ground condition. Almost certainly, this m-phase reduction may have been caused by slight heating when performing the polishing and by the high temperature required to sinter the glaze. During polishing, the superficial temperature may rise to values above the critical point, from which the m-t reverse transformation may occur (Kosmac et al., 1999, Kosmac et al., 2007; Íseri et al., 2012), which might have led to reduced m-phase content. Alternatively for glazing, the specimens are subjected to firing; although the cycle is very short (only 1 min at 900°C, while heat treatment is 15 min at 1000°C), it was enough to promote some reversion (m to t phase).

It is important to note that the worst mechanical performance (characteristic strength) was observed for the glazed groups. When the stress applied on a material becomes higher than the strength, mechanical failure may occur (Della Bona et al., 2003). The stress concentration is very dependent to the size of preexisting cracks (Mecholsky, 1995). Thus, in these bilayer assemblies (e.g. Y-TZP + glaze), the strength and fracture mode are influenced by the properties of the material under tension (Borba et al., 2011) (glaze material in this case). Our micrographics show the presence of many bubbles (defects) inside the glaze layer (Fig. 2), additionally the fractography analysis (Fig. 3) showed that the fractures originated at the glaze/Y-TZP interface in these group. These facts could be the reason why the characteristic strength decreased in our glaze group.

Anusavice & Philips (2003) states that the application of a thin layer of glaze results in a fill-up mechanism, from which the glaze filled any existing microcrack and porosity on the ceramic surface, leading to a smoother surface, with less defects and an increased fracture strength. These were also observed by Aurelio and collaborators (2015), in which an alternative glaze protocol enhanced the mechanical performance of a hard-machined leucite glass ceramic. The current study did not demonstrate this positive effect; on the contrary, the glaze group had lower strength when compared to the other groups, since the fracture started at the glaze layer, which is a weaker material. Additionally, there is a weak adhesion between the zirconia and veneering ceramic, which might have been the cause for failure initiation at the glaze layer (Aboushelib et al., 2008).

Our findings show that an increase in m-phase content led to higher characteristic strength (transformation toughening mechanism), which corroborated with the literature (Hannink et al., 2000; Pereira et al., 2016[a]). Even when 60% of m-phase content is observed (Ctrl + Aut), an increased characteristic strength is noticed. However, this outcome may change when the Y-TZP surface is ground: since the grinding + aging condition demonstrated a decrease in strength, even though it presented a smaller m-phase content when compared to the Ctrl + Aut group (approximately 40%). This fact points out that the defects introduced by grinding may become deleterious when associated with aging (even with an increase in m-phase content), which confirms the findings observed by Pereira and collaborators (2016[b]).

At the same time, our study shows that grinding before aging decreases the susceptibility to t-m phase transformation – this fact is more evident for post-processing treatments (polishing, glazing and heat treatment/annealing) subjected to aging. Some studies report that grinding introduces superficial compressive residual stresses (Chevalier et al., 2007; Chevalier et al., 2009) and Whalen and collaborators (1989) demonstrated that compressive residual stress decreased the susceptibility to t-m phase transformation. Additionally, Muñoz-Tabares & Anglada (2012) stated that the superficial microstructure change induced by grinding could play an important role on the susceptibility of m-phase transformation.

The microstructural changes induced by grinding 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 grain diameter ranges 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 (Muñoz-Tabares et al., 2011).

We observed that heat treatment completely reverted the m-phase content back to the t-phase; however, a higher resistance to transformation was observed for that scenario, even though there is no expected residual stress on that surface. Thus, the increased resistance to aging (transformation) could be possibly related to the existence of this very thin layer of tetragonal recrystallised nano-grains (10-20 nm), which were generated by grinding, whose size are smaller than the critical size for transformation in a humid environment (Evans et al., 1981; Lange, 1982; Muñoz-Tabares et al., 2011).

Even with the limitations of this *in vitro* study, our data clearly supports the benefit of polishing after grinding Y-TZP ceramic, instead of glazing and heat treatment. However, we did not consider an association of factors, such as grinding followed by polishing and heat treatment, thus more studies are recommended. Additionally, no intermittent mechanical loading or fatigue were conducted – since dental restorations are known to fail due to fatigue effects, where cyclic forces below the characteristic strength of the material act on existing defects, resulting in the progressive growth of these defects until they achieve a critical size where a fracture occurs (slow crack growth) (Wiskott et al., 1995; Gonzaga et al., 2011). Therefore, more studies are needed to determine the effects of loading on surface treatments and crack formation.

5. Conclusions

When taking into account the mechanical outcomes, it can be concluded that:

- Polishing of ground zirconia surfaces improves the mechanical behavior, thus polishing is the most appropriate surface treatment to be performed after grinding Y-TZP ceramics.
- The strength reduces when only heat treatment is performed after grinding.

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TABLES

Table 1 – Experimental design

Surface treatment	LTD	Groups' Codes
As-sintered (without treatment, control)	Without	**Ctrl**
	With	**Ctrl + Aut**
Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm)	Without	**Gr**
	With	**Gr + Aut**
Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm) + Heat treatment	Without	**Gr + HT**
	With	**Gr + HT + Aut**
Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm) + Polishing	Without	**Gr + Pol**
	With	**Gr + Pol + Aut**
Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm) + Glazing	Without	**Gr + Gl**
	With	**Gr + Gl + Aut**

Table 2 – Roughness analysis (parameter Ra and Rz), Spearman Correlation, X-ray Difractometry analysis (% of monoclinic phase and depth of transformed layer), Weibull modulus (*m*), Characteristic strength (σ_c) and respective confidence intervals (CI – 95%).

Groups	Roughness Analysis		Spearman Correlation		XRD Analysis		Weibull Analysis	
	Ra (µm)	Rz (µm)	(σ x Ra)	(σ x Rz)	m-phase content (%)	TZD (µm)	*m* (CI 95%)	σ_c (CI 95%)
Ctrl	0.24 ± 0.83^{A}	1.93 ± 0.50^{A}	-0.239 (p=0.203)	-0.349 (p=0.059)	1.4	0.1	9.7 (6.8-12.5)^{AB}	964.7 (922.5-1007.5)^{D}
Ctrl + Aut	0.21 ± 0.34^{A}	1.77 ± 0.36^{A}	0.018 (p=0.926)	-0.193 (p=0.306)	65.6	5.4	26.0 (18.2-33.4)^{C}	1030.3 (1013.2-1047.3)^{E}
Gr	1.55 ± 0.65^{D}	8.26 ± 1.26^{D}	0.029 (p=0.880)	-0.166 (p=0.381)	13.9	0.8	10.7 (7.5-13.7)^{AB}	1183.3 (1136.0-1231.3)^{G}
Gr + Aut	1.33 ± 0.12^{C}	8.09 ± 0.89^{D}	0.086 (p=0.653)	0.037 (p=0.845)	43.2	2.9	14.0 (9.8-18.0)^{B}	1085.7 (1052.5-1118.9)^{F}
Gr + HT	1.35 ± 0.15^{C}	8.33 ± 0.98^{D}	-0.254 (p=0.175)	-0.399 (p=0.029)	0	0.0	15.3(10.7-19.6)^{BC}	818.5 (795.4-841.3)^{B}
Gr + HT + Aut	1.35 ± 0.19^{C}	8.27 ± 1.15^{D}	-0.081 (p=0.671)	-0.162 (p=0.392)	26.6	1.6	29.2 (20.5-37.6)^{C}	882.0 (869.0-894.8)^{C}
Gr + Pol	0.60 ± 0.14^{B}	3.76 ± 0.79^{C}	-0.069 (p=0.716)	-0.089 (p=0.639)	9.6	0.5	10.0 (7.0-12.8)^{AB}	1286.2 (1231.2-1342.1)^{G}
Gr + Pol + Aut	0.53 ± 0.12^{B}	3.16 ± 0.60^{B}	0.185 (p=0.327)	0.233 (p=0.215)	20.3	1.2	7.0 (4.9-9.1)^{A}	1309.3 (1230.9-1390.4)^{G}
Gr + Gl	0.62 ± 0.26^{B}	3.54 ± 1.91^{C}	0.133 (p=0.482)	0.122 (p=0.519)	7.3	0.4	13.8 (9.6-17.7)^{B}	745.4 (722.2-768.6)^{A}
Gr + Gl + Aut	0.64 ± 0.21^{B}	3.54 ± 1.43^{C}	0.321 (p=0.083)	0.259 (p=0.166)	17.2	1.0	14.6 (10.2-18.7)^{BC}	732.1 (710.6-753.7)^{A}

* Same letters mean no statistical difference. Different letters correspond to statistical difference.

FIGURES

Figure 1 –Representative SEM images (5000x magnification) and AFM images (20 µm X 20 µm) of Y-TZP surface for different conditions, demonstrating that grinding with diamond burs (Coarse) modified the topographic pattern of "as sintered" samples creating parallel scratches following the movement of the grinding tool and deforming the surface. Heat treatment did not promote any change on those aspects, while polishing and glazing successfully demonstrate a smoothening potential. Aging cause none relevant alteration for any evaluated condition.

SEM images	AFM images	SEM images	AFM images
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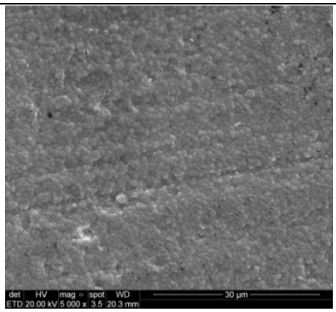	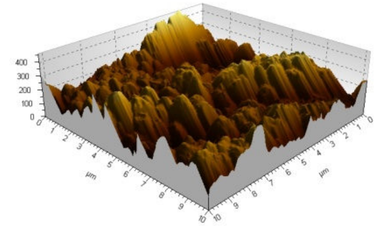	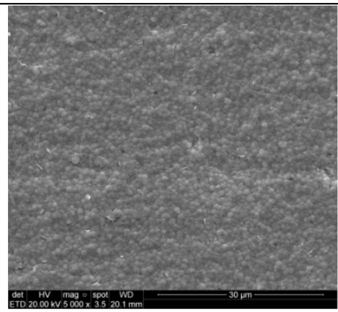	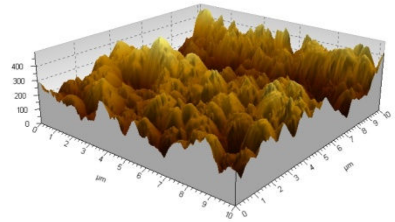
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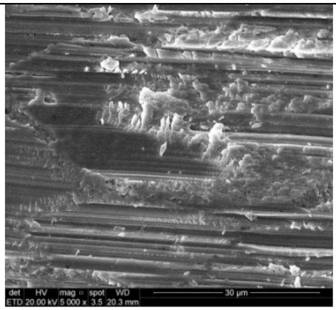	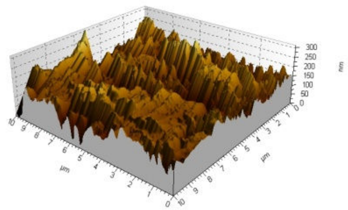	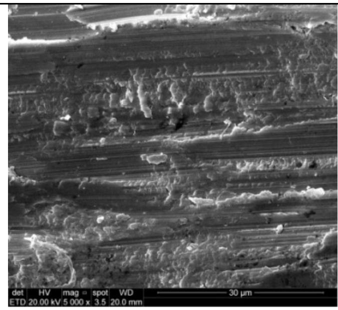	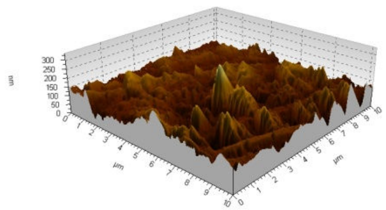
Gr + Ht		Gr + Ht + Aut	

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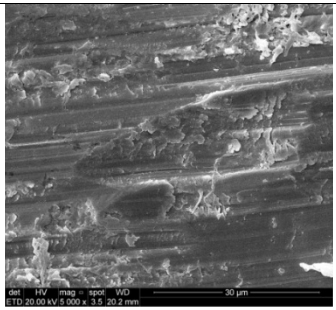
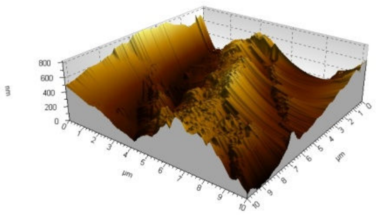
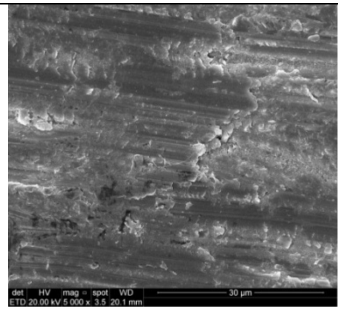
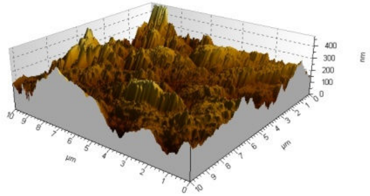
<tr><td></td><td></td><td></td><td></td></tr>
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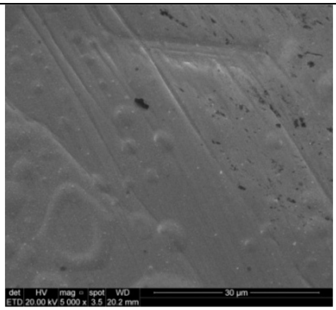
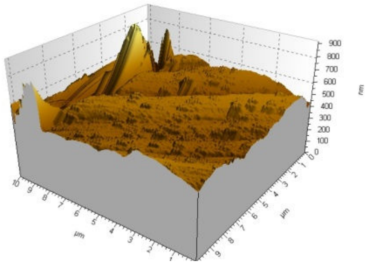
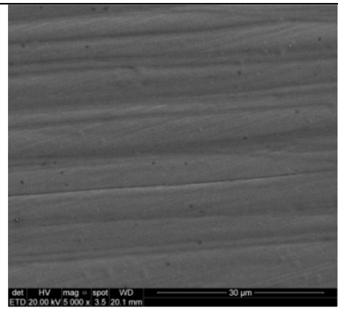
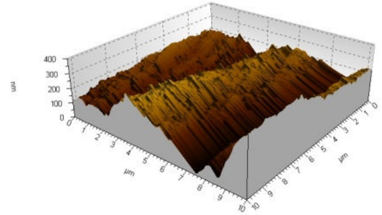
<tr><td></td><td></td><td></td><td></td></tr>
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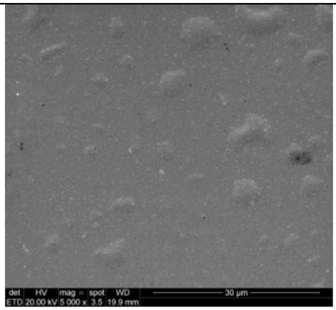
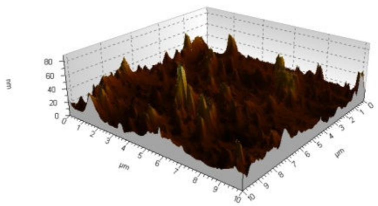
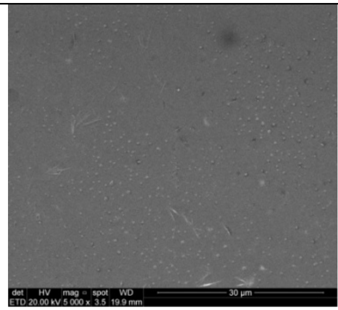
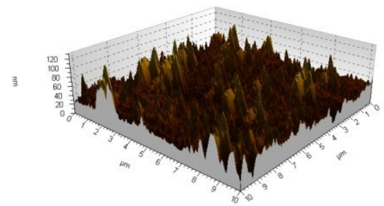
<tr><td></td><td></td><td></td><td></td></tr>
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Figure2–Representative micrographs (1000x magnification) demonstrating to some extent an apparent potential of glaze application to fill the defects introduced by grinding and form an external smooth surface. Additionally it notices that a thin thickness of glaze (approximately 32 – 55 μm) was achieved. Further, it highlights that the glaze layer presents of many air bubbles and defects in the bulk.

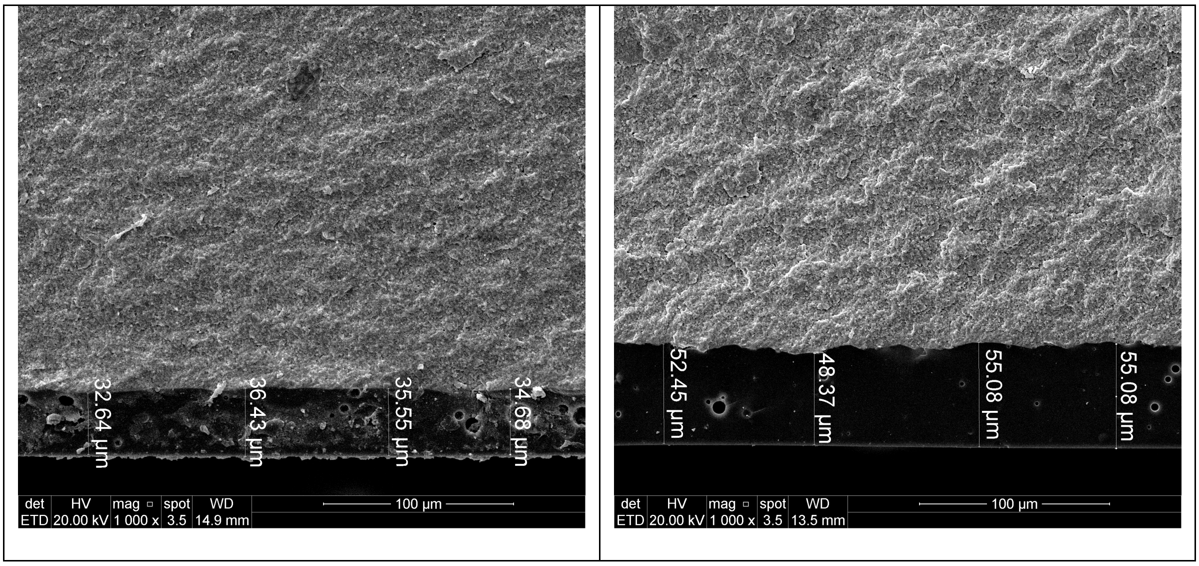
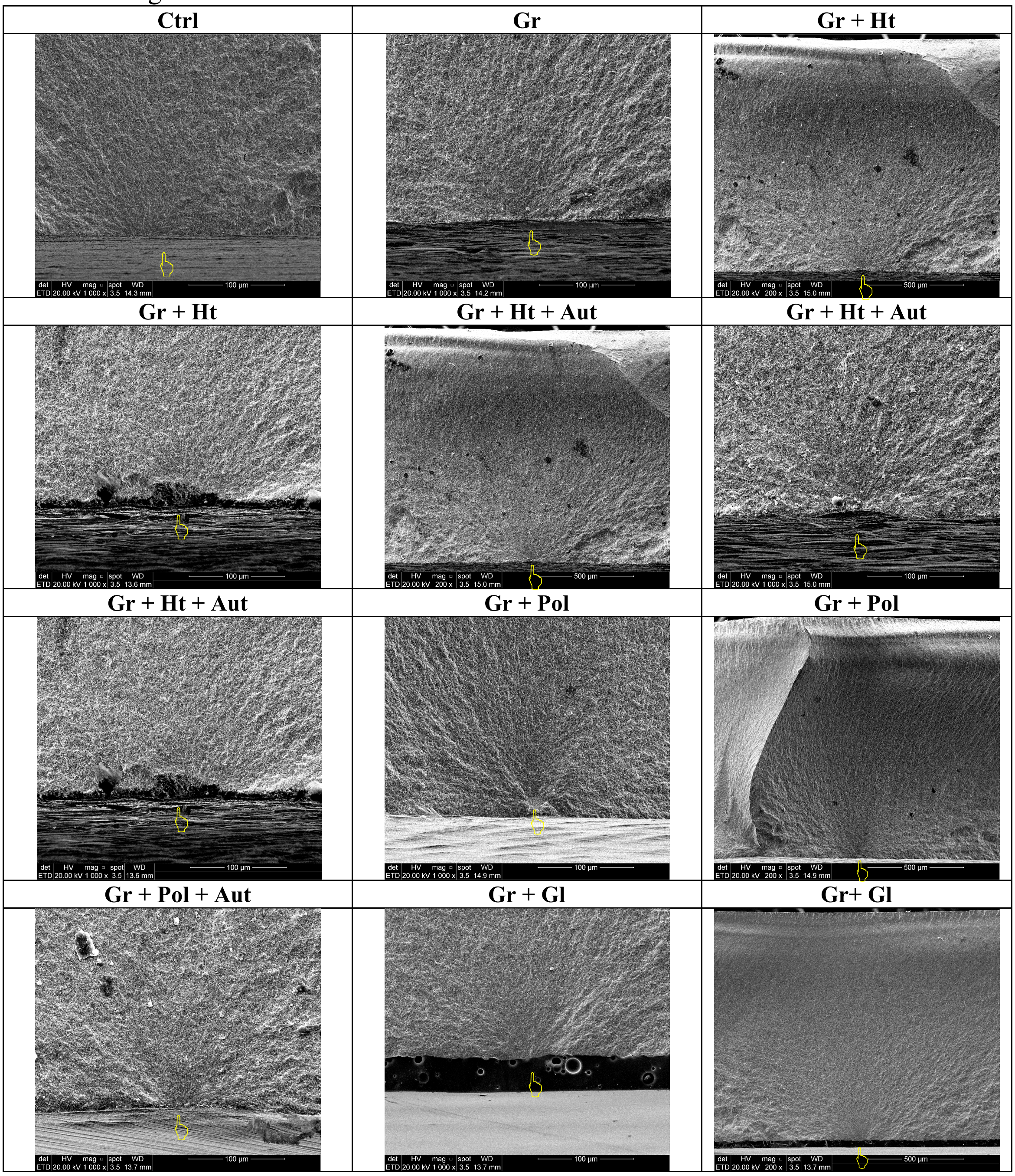


Figure 3 –Representative images of the fractography analysis under SEM (200x and 1000x magnification): Typical brittle fracture surfaces, which clearly shows that the initial crack nucleation and propagation region are located in the lower side of the micrographs, corresponding to the region of maximum tensile stress in the biaxial test. For Ctrl, Gr, Gr + Ht (and respective aged conditions), it observes that the fracture originates on superficial defects; for Gr + Pol (with and without aging), it starts on subsurface defects, while Gr + Gl (with and without aging) the fracture originates on the glaze/Y-TZP interface. The 👆 pointer indicates the fracture origin.



3. ARTIGO 2 - Fatigue strength of yttria-stabilized zirconia polycrystals: Effects of grinding and post-processing treatments.

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Running title: Post-processing treatments and fatigue of Y-TZP.

ABSTRACT

This study aimed to evaluate and compare the effect of different surface post-processing treatments (polishing, heat treatment, glazing, polishing + heat treatment and polishing + glazing) on the superficial characteristics (micromorphology and roughness), phase transformation and fatigue strength of a Y-TZP ceramic ground with diamond bur. Discs of Y-TZP ceramic were manufactured (ISO:6872-2015; final dimensions of 15mm in diameter and 1.2 ± 0.2mm in thickness) and randomly allocated according to the surface condition: Ctrl- as-sintered; Gr- ground with coarse diamond bur; Gr+HT- ground and subjected to the heat treatment; Gr+Pol- ground and polished; Gr+Pol+HT- ground, polished and heat treated; Gr+Gl- ground and glazed; Gr+Pol+Gl- ground, polished and glazed. The following analyses were performed: roughness (n=25), surface topography (n=2), phase transformation (n=2) and fatigue strength by staircase method (n=20). All treatments influenced to some extent the surface characteristics of Y-TZP, being that polishing reduced the surface roughness, the m-phase content and improved the fatigue strength; glazing led to the lowest roughness values (Ra and Rz), although it showed the worst fatigue strength; heat treatment showed limited effect on surface roughness, led to complete reversion of the existing m-phase content to t-phase, without enhancing fatigue performance. Thus, a polishing protocol after clinic adjustment (grinding) of monolithic restorations based on polycrystalline zirconia material is mandatory for surface characteristics and fatigue performance improvements.

Key words: Dental Ceramics. Zirconium oxide partially stabilized by yttrium. Glazing. Annealing.

1. INTRODUCTION

Monolithic restorations made of polycrystalline zirconia material have been proposed for manufacturing high strength and aesthetic (to some extent) rehabilitation on Prosthetic Dentistry (Marchack et al., 2011). One of the main advantages of this restorative strategy is the reduced tooth preparation (0.5 mm thickness), which allows a minimally invasive dental approach (Nakamura et al., 2015). Besides, it avoids the main reason to the reported failure of conventional zirconia restorations (framework of zirconia + veneering porcelain) namely chipping of the veneer material (Shi et al., 2016).

The use of zirconia restorations in Dentistry was only possible with the advances/enhancement on CAD/CAM manufacturing systems (Computer-Aided Design/ComputerAided Machining). Miyazaki and collaborators (2009) states that

CAD/CAM milling permit a substantial reduction on working time and higher precision of the final restoration. However it is still commonly necessary adjustments (grinding, finishing and polishing procedures) to enhance emergence profile and occlusal/proximal relations (İşeri et al., 2012; Pereira et al., 2016[a]). From this point of view, literature has been showing that those adjustments may introduce different types of damage (defects) into the materials' surface, and by that they may impair the mechanical performance of zirconia materials and the longevity of such restorations (Green,1986; Kosmac et al., 1999; Pereira et al., 2016[a]).

Zirconia ceramics is a metastable polymorphic material that in response to stimuli (such as adjustments or aging) triggers a phase transformation mechanism (tetragonal – t to monoclinic – m)(Piconi, 1999; Lazar et al. 2008). The t-m phase transformation mechanism results in a local volumetric expansion of 3-4% and by that it creates a layer of residual compressive stress concentration (Hannink et al., 2000). This residual stress layer will act preventing crack propagation and enhancing the material's toughness (Chevalier & Gremillard, 2009).

Though, this protective effect (increase in toughness, i.e, making crack propagation more difficult) is not constant and predictable; if the defect introduced by the stimuli (like restoration adjustments) is bigger than the layer of residual stress (Green, 1983); or with the progression of a slow-crack growth (in response to fatigue stimuli) (Papanagiotou et al., 2006) a catastrophic failure may still happen.

Consequently, post-processing treatments after adjustments/grinding, such as polishing, heat treatment or glazing, appear be required, to restore the surface smoothness, to prevent damaging to the mechanical performance and antagonist tooth wear (Guazzato et al., 2005; Preis et al., 2012; Preis et al., 2013).

Literature shows that polishing may decrease the surface roughness (Preis et al., 2015), which is a positive outcome. Although it also may trigger surface temperature increase above the limit of m-t phase transformation (Guazzato et al., 2005; Juy & Anglada, 2007), so if the polishing procedure does not remove all the surface defects and at the same time promotes a reversion of m-t phase eliminating any potential residual stress concentration, it could be observed a negative result on mechanical performance.

Regarding heat treatment, literature shows that this procedure successfully reverts back m phase to t-phase and eliminates existing residual stress (Ramos et al., 2016), although it does not present any potential for defect reduction (healing of defects) on Y-TZP ceramics, as could be observed on glass ceramics (Aurélio et al., 2017).

Another post-processing alternative is glazing, which consists on applying a thin layer of glassy material on zirconia surface aiming to reduce the surface roughness, to increase the light reflection (shining surface), to enhance the aesthetic and to decrease the biofilm formation (Haralur, 2012). Although limited information exists on literature on the effects of glazing on the mechanical performance of zirconia restorations.

Thus, as each existing post-processing option after adjustments (grinding) of zirconia restorations may interact by different mechanisms with the zirconia surface (which could result in different mechanical performance), as well as, once scarce information exists to elucidate and help the clinicians to define a gold-standard protocol to this aim, this study evaluated and compared the effect of different post-processing treatments (heat treatment, polishing, glaze, polishing + heat treatment and polishing + glaze) on the fatigue strength, surface characteristics (topography and roughness), and phase transformation of a translucent zirconia ceramic for monolithic restorations. The assumed hypotheses were: 1- grinding will change the material's surface characteristics (introduce surface flaws and increase roughness), promote t-m phase transformation and influence the mechanical properties; 2- post-processing treatments would also influence the surface characteristics and the mechanical properties in distinct intensities (based on the differences among each treatment) as an attempt to revert any deleterious effect exhibited by grinding.

2. MATERIALS ANDMETHODS

The materials utilized in this study are described in table 1.

Disc-shaped specimens of Y-TZP (yttrium-stabilized tetragonal polycrystalline zirconia – Zenostar T) were produced following the guidelines of ISO 6872-2015 for biaxial flexural strength testing of ceramic materials (final dimensions: 15 mm in diameter and 1.2 ± 0.2 mm in thickness).

For production of those ceramic discs, a Zenostar blank (98 mm diameter – as available by manufacturer) was shaped manually into smaller blocks of 20 mm x 20 mm with diamond discs and after metallic rings of 18 mm diameter were attached to parallel surfaces, serving as a guide for obtaining a cylinder of zirconia by grinding in polishing machines (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA) with silica carbide (SiC) papers (600-1200 grit). After that, slices (discs) of 1.65 mm thick were obtained by cutting under water-cooling with diamond blades (ISOMET 1000, Buehler, Lake Bluff, USA).

Afterward, the specimens were polished on both sides with SiC paper (1200 grit) to remove any irregularity introduced by processing (until 1.5 mm thick), sintered according to manufacturers instructions (1450°C for 2 h) in a specific special furnace (Zyrcomat T, Vita Zahnfabrik, Bad Sackingen, Germany), inspected with a digital caliper (Mitutoyo ABSOLUTE 500-196-20 Digital Caliper, Takatsu-ku, Kawasaki, Kanagawa, Japan) for guarantee that the dimensions were in accordance to ISO 6872-2015, and then they were randomly allocated considering the surface post-processing treatments to be evaluated (table 2).

2.1 Surface Post-Processing Treatments

A single trained (specialist in Prosthetic Dentistry – CPZ) operator executed all post-processing treatments. All groups are summarized in table 2.

2.1.1 As-sintered – CTRL

Specimens were kept untouched after sintering.

2.1.2 Grinding – Gr

Grinding was executed with coarse diamond bur (#3101G – grit-size 181 μm; KG Sorensen) coupled to a speed multiplier handpiece (T2 REVO R170 contra-angle handpiece up to 170,000 rpm, Sirona, Bensheim, Germany) attached to a low-speed motor (Kavo Dental, Biberach, Germany) under constant water-cooling (≈30 mL/min).

Specimens were attached to a metal base that served as reference for maintaining the diamond bur tip parallel to the specimen surface, and then grinding was executed manually with oscillatory motion. Cautious were taken for this procedure to be standardized, increasing its reproducibility: for that the entire zirconia surface to be ground was marked with a permanent marking pen (Pilot, São Paulo, Brazil); grinding was executed until the complete elimination of this marking; besides the diamond bur was replaced after each specimen (1 bur per specimen). Methodology previously employed by Guilardi and collaborators (2017).

2.1.3 Polishing – Pol

Polishing was executed in two consecutive steps aiming to achieve a smoothened surface following the guideline of the Y-TZP manufacturer's (Zenostar T, Ivoclar Vivadent).

Firstly, finishing procedure was executed using fine (#3101F – grit-size 46 μm; KG Sorensen) and extra-fine (#3101FF – grit-size 30 μm; KG Sorensen) diamond burs under the same execution protocol described on grinding procedure.

After that, the Optrafine System (Ivoclar Vivadent) was used. This polishing kit consists in 2 tips (Light-blue and Dark-blue) and a nylon brush that is used combined with a diamond paste (OptraFine HP Polishing Paste – diamond particle size of 2 – 4 μm) to generate a shining smooth surface. Also, procedures were cautiously performed for standardization and reproducibility: the specimen was affixed in a similar metal base to the one used on grinding procedure, and the area to be polished was divided into two regions considering the size of the polishing tips; this procedure was executed for 25 seconds in each region for each specific tip.

2.1.4 Glazing – Gl

A thin glaze layer was applied into the ceramic surface (IPS Ivocolor Glaze Paste, Ivoclar-Vivadent) following the manufacturer's guidelines. For that, the paste was mixed with distilled water until obtaining an adequate consistency, and then applied into the whole ceramic surface with a specific brush, and sintered on the Vacumat 6000MP furnace (Vita Zahnfabrik; drying temperature 403°C, furnace closing time 6 min, heating rate 45°C/min, final temperature 710°C, maintenance of 1 min, with vacuum at 450°C and at 709°C).

Prior to the final execution of this step, the researcher (C.P.Z.) developed pilot studies to standardize and improve reproducibility of this protocol until it become noticeable a homogenous glaze layer under Scanning Electron Microscopy (SEM - Vega3, Tescan, Czech Republic; methodology described below on 2.2 section) with a final thickness ranging from 24 to 28 μm as depicted on Figure 1.

2.1.5 Heat Treatment – HT

Heat treatment is proposed by manufacturers to remove any potential residual stress introduced by processing. This procedure was executed on the Vacumat 600MP furnace (Vita Zahnfabrik) following the manufacturer's guidelines (closing time 18s, heating rate 65°C/min, final temperature 1050°C, maintenance of 15 min, followed by slow cooling 25°C/min).

2.1.6 Pol + HT:

After grinding the specimens were subjected to the polishing and heat treatment procedures as aforementioned.

2.1.7 Pol + Gl:

After grinding, the specimens were subjected to the polishing and glazing procedures as aforementioned.

2.2 Surface topography

Firstly, two specimens per group were cleaned in ultrasonic bath (1440 D – Odontobras, Ribeirao Preto, Brazil) with 78% isopropyl alcohol for 5 min, and then they were gold sputtered and analyzed on Scanning Electron Microscopy (SEM - Vega3, Tescan, Czech Republic) under 5000x magnification to observe the superficial topographical pattern and under 2000x magnification to observe cross-sectional topography.

2.3 Roughness analyses

All specimens from each evaluated condition were submitted to a roughness analysis on a specific profilometer (Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan) considering the parameters recommended by ISO:4287-1997 (Ra and Rz). Three readings were executed per specimen on the opposite direction of the one used during polishing with a cut-off (n=5), λC 0.8 mm e λS 2.5 µm, after mean average values were obtained for each specimen.

2.4 X-ray Diffraction (XRD) for phase transformation analysis

Two specimens from each condition were analyzed by X-ray Diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) to quantify of monoclinic phase content. Data was collected in a Bragg-Brentano assembly on 2θ, considering the angle interval of 24-35° with a step size of 0.03° for each second, and the m-phase content was obtained using the Garvie and Nicholson methodology modified by Toraya and collaborators (Garvie & Nicholson, 1972; Toraya et al., 1984). After that, means and standard deviations were calculated.

2.5 Biaxial flexural fatigue test

Initially, three specimens from each condition were subjected to a monotonic static biaxial flexural test on a universal testing machine (EMIC DL 2000, São José dos Pinhais, Brazil), to obtain a mean strength value for defining the fatigue protocol. The test setup (piston-on-three balls) followed the ISO:6872-2015 guidelines for biaxial flexural strength testing of ceramic materials extensively described on previous literature (Amaral et al., 2013; Guilardi et al., 2017; Pereira et al., 2014; Pereira et al., 2016[b]).

Afterward, a fatigue test (n=20) was executed under the same biaxial test setup in an electric machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, USA) using the staircase approach, described by Collins (1992). The reasons that guided the choice of this testing assembly (staircase method) was based on the fact that this method is widely used, and is known to be a fast and precise manner for inducing fatigue of dental ceramics with low variability (Collins, 1992; Villefort et al., 2017). The parameters for fatigue tests were defined based on the relation between a monotonic and fatigue test elucidated on previous literature (Pereira et al., 2016[c]; Villefort et al., 2017): the initial load consisted in 60% of the static flexural strength mean, and the step size was considered as 5% of the calculated initial strength mean for fatigue test. The fatigue tests were executed under a lifetime of 20,000 cycles and a frequency of 6 Hz. If the tested sample endured the test, one step-size was incremented and a new sample was tested. If the observed outcome was fracture, the step was reduced. This procedure was followed until all specimens from each group were tested.

2.6 Fractography analysis

All fractography examinations were executed by a single trained researcher (LFG). Firstly, all specimens were examined in an optical stereomicroscopy (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany), from which representative fracture patterns were recognized for each group and then subjected to SEM analysis (Vega3, Tescan, Czech Republic) to determine the crack origin and fractography characteristics. Additionally, specimens that received glaze procedure were examined in SEM for measurement of glaze layer thickness (transversal/cross sections were examined).

1.7 Data analysis

A descriptive analysis of the roughness data was made (Ra and Rz parameters) to obtain mean and standard deviation values, and as the data assumed a non-parametric distribution (Shapiro Wilk test), the Kruskal-Wallis and post hoc LSD was used ($\alpha = 0.05$). Regarding the fatigue strength (σ_f), means, standard deviations and 95% confidence intervals of both parameters were obtained in accordance to the methodology of Collins, 1992 (previously described on Fraga et al., 2016 and Villefort et al., 2017), and then, the statistical comparisons were made by one-way ANOVA (analysis of variance) and post-hoc Bonferroni tests.

2. RESULTS

SEM images (Figure 2 and 3) shows that grinding creates an irregular topography surface and that heat treatment did not promote any relevant changes on this pattern. Polishing and glazing demonstrate a potential to decrease the irregularities achieving an apparent smoother surface.

Roughness analysis (Ra and Rz parameters) corroborates SEM findings: heat treatment did not change roughness; polishing and/or glazing successfully re-established the smoothness observed on as-sintered samples (Gr+Pol = Ctrl = Gr+Gl). When polishing was combined with glazing, there was a tendency of decrease on roughness values, which in fact resulted on the smoothest condition (Table 3).

XRD data shows that grinding leads to higher superficial m-phase content. Polishing and/or glazing triggered some reverse phase transformation (m-phase back to t-phase), decreasing m-phase content. While heat treatments promoted a complete phase reversion (0% of m phase) (Table 3).

Regarding mechanical fatigue behavior, it could be withdrawn that: the group polished after grinding (Gr+Pol group) demonstrates the best performance (highest values of fatigue strength); heat treatment protocols (Gr+HT and Gr+Pol+HT groups) led to similar fatigue strength to as-sintered group; glazed group (Gr+Gl group) showed the worst results (lowest values of fatigue strength); and that polishing previous to glazing (Gr+Pol+Gl group) significantly improved the fatigue strength compared to glazing alone (Gr+Gl group) (Table 3 and Figure 4).

Fractography (Figure 5) showed that all fractures started on the surface subjected to tensile stress concentration (side opposite to load application), although the pattern of fracture on glazed groups was different from the other groups. On glaze groups, the origin of fracture is located on the interface ceramic/glaze and on all other conditions the fracture origins on superficial defects (Figure 5).

3. DISCUSSION

Based on the current findings, it may be noticed that the first hypothesis was accepted, since grinding generated the most irregular surface, with the highest values of roughness, promoted t-m phase transformation, although it not deleteriously impact fatigue strength in comparison to as sintered condition (Gr > Ctrl). As for the second hypothesis it was also accepted, because different post-processing treatments affected differently the Y-TZP surface characteristics and mechanical properties, where an adequate polishing protocol prove to be mandatory after adjustment (grinding) of Y-TZP ceramics, promoting a decrease on surface roughness, improving fatigue strength and also decreasing m-phase content (reversion of m-phase back to t-phase). Still on this sense, despite glazing promoted surface smoothing (decrease in roughness), it impacted deleteriously on the material's fatigue strength, as also did the heat treatment protocol.

SEM images (Figures 2 and 3) show that heat treatment has no potential to eliminate surface defects (healing) introduced by grinding; in other words, after heat treatment, the topography pattern was kept very similar to that one observed after grinding. This finding is in accordance with previous literature that considered Y-TZP ceramic (Çaglar & Yanikoglu, 2016; Deville et al., 2006; Ramos et al., 2016). However, it has to be emphasized that regardless of the association with polishing or executed solely, heat treatment eliminated any existing m-phase content (complete reversion from m to t phase) (table 3), which could be an indicative of residual stress removal as it also led to a decrease on fatigue strength in response to the elimination of any toughening mechanism generated (Guazzato et al., 2005; Ramos et al., 2016).

Different stimuli (grinding, sandblasting, exposure to water and temperatures) may trigger t-m phase transformation (Denry & Kelly 2014; Kim, 2010). Kobayashi et al., 1981 described this mechanism, as a time dependent material response mechanism that, as it develops, it spreads into the ceramics surface and core leading to grain detachment, increased porosity and roughness, that ultimately would lead to decrease on density and mechanical performance. This mechanism was named low-temperature degradation (LTD) and is categorized as the degradation of the material in response to m-phase content increase above a critical point (according to ISO 13356-2008 the monoclinic phase content must not be greater than 25%) were mechanical stability is lost (Kim et al., 2010; Pereira et al., 2016[d]).

Some factors are known to predispose Y-TZP ceramic to LTD effects, e.g. grain size and processing protocols (Chevalier & Gremillard, 2009). Recently introduced translucent zirconias present smaller grain sizes, which enhances aesthetic characteristics (e.g.translucency) (Zhang, 2014). Lucas t al., 2015 showed that a reduced grain size also decreases LTD susceptibility. On this sense, the amount of m-phase content observed on our study, in addition to the observed performance among all other evaluated outcomes, does not represent a worst performance and predictability of an inadequate performance clinically. On contrary, a higher level of tension is required for a fracture to occur (Kim et al., 2009).

Besides, literature shows that an increase in m-phase content does not necessarily damage the mechanical performance, on contrary, it is already well established that to some extent Y-TZP may respond with a toughening effect related to residual stress concentration by this phase transformation mechanism arresting crack propagation (Guilardi et al., 2017; Gupta, 1980). Those statements are in accordance with the behavior observed on the group subjected to grinding, in which an enhanced fatigue performance was observed (increase on fatigue strength in comparison to as-sintered samples - Ctrl group) (table 3).

Hence, a toughened surface generated by residual stress concentration in response to a layer of transformed grains (m-phase content increase) (Pereira et al., 2014) may be desirable when adjustments are executed and defects are introduced and need to be restrained by a protective mechanism preventing premature failure (Guilardi et al., 2017). However, if the defects are bigger than this transformed layer zone or if a slow-crack growth mechanism triggered by fatigue, stimulate this defects it may be expected a deleterious impact (Guazzato et al., 2005; Green 1983; Kosmac et al., 1999).

Thus it becomes essential (as demonstrated on our experiments) that an adequate polishing be executed to remove any defect introduced (decrease roughness) that could be harmful to the mechanical performance of zirconia-based restorations. The tested polishing protocol was able to re-establish the smoothness observed on as-sintered samples (Gr+Pol = Ctrl). Gönülol et al., (2012) stated that a high quality polishing enhances aesthetic characteristics and restorations longevity; on the other hand, a roughened surface would enhance biofilm accumulation, gingival inflammation and increase the risk for secondary caries. Still on this sense, Preis et al., 2012 showed that a roughened surface would lead also to higher antagonist tooth wear.

Among all evaluated protocols, the polished group demonstrated the highest fatigue strength, one of the smoother surfaces and led to a reduction of m-phase content compared to the group just ground (Table 3). The potential for m-phase reversion (m-t transformation) was already elucidated by previous literature (Guazzato et al., 2005; Kosmac et al., 1999) and may be explained by temperature rising locally above the critical point were m-phase could be sustained.

Another important observation to be highlighted is the worst mechanical performance for the glazed groups (alone or in association with other post-processing treatments). According to Anusavice (2003), a thin glaze layer would fill any existing crack and porosity and enhance mechanical performance on glass-based ceramics. However, owing to the fact that Y-TZP ceramics is polycrystalline (without any glassy content), an adequate bonding among these substrates (Y-TZP ceramics and glaze) is difficult (Denry & Kelly 2014). Besides, in such scenario where a bi-layer interface is created; Borba et al., 2011 showed that the material under tension would dictate the mechanical performance of the system (i.e., the glaze layer, in our scenario), especially when an incompatibility between the substrates (poor adhesion between layers) is presented (Wuttiphan et al., 1996).

Still on this sense, as shown by Pozzobon et al., 2017, there may be an incorporation of bubbles inside the glaze layer, which are introduced as inherent consequence to the application procedures (air during mixing and sintering of the glaze material), this factor may increase the risk of failure originating on this interface. On this sense, the thickness of glaze layer also may affect this process, factor that could also help to explain the reduced strength of this group. However, as we took precautions for standardizing these factors on our testing set-up, we did not observed a larger variability on this group and by that we did not expect bias on our obtained data, which otherwise could lead to a misinterpretation.

Interestingly, our data support that polishing rather than glazing (Gr+Pol+Gl group) showed a potential to enhance the fatigue behavior – higher fatigue strength than Gr+Gl group (i.e. no polishing). Hence, Evans & Hutchinson, 1989 showed that the roughness on the interface influences the strength of the system, as roughened interfaces (Figure 5) may predispose cracks to propagate inside the glass material (glaze) and compromise mechanical performance. As some clinical scenarios request glaze application to enhance the final aesthetic characteristics of restorations, especially when using Y-TZP ceramics in monolithic application, it seems mandatory to execute an adequate polishing to remove surface defects and generate a smooth surface previously to glaze application for fatigue improvements.

From the mechanical point of view, our data support that polishing after grinding to reestablish surface roughness and remove defects introduced by grinding is mandatory. Even though, it is important to highlight that this in vitro study presents inherent limitations such as: no optical properties was evaluated; only 1 polishing kit and protocol was considered; important clinical conditions could not be simulated, such as long-term aging, temperature changes, sliding which occurs during chewing. Thus, more studies are necessary to better fulfill the demand of establishing a gold-standard post-processing protocol of Y-TZP monolithic restorations.

CONCLUSIONS

- An adequate polishing after clinical adjustment on Y-TZP ceramics seems to be mandatory for fatigue enhancement and predictability of preventing crack propagation under smaller stress.

- When glazing is indispensable in a clinical scenario, a previous polishing protocol is essential for fatigue behavior improvements.

- Heat treatment alone of ground Y-TZP ceramics seems not to be a good alternative, as it does not restore surface smoothness and it led to a decrease in mechanical performance.

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TABLES

Table 1 – Materials' main compositions.

Materials	Composition	Manufacturers
Y-TZP ceramic	Zirconium oxide ($ZrO_2 + HfO_2 + Y_2O_3$) > 99.0%; Yttrium oxide (Y_2O_3) > 4.5 – ≤ 6.0%, Hafnium oxide (HfO_2) ≤ 5.0 %; other oxides ≤ 1.0%	Ivovlar-Vivadent
Diamond bur #3101G – gritsize 181µm	Diamond and stainless steel	KG Sorensen
Diamond bur #3101F – gritsize46 µm	Diamond and stainless steel	KG Sorensen
Diamond bur #3101FF – gritsize30µm	Diamond and stainless steel	KG Sorensen
Optrafine System	***Tip Light-blue and Dark-blue*:** synthetic rubber, diamond granulate, titanium dioxide and stainless made of steel; ***Nylon brush*:** nylon fibres and stainless made of steel; ***Past diamond*:** diamond dust (2-4µm), glycerine, sodium lauryl, sulphate and propylene glycol.	Ivovlar-Vivadent
IPS Ivocolor Glaze Paste	Alkali Alumino-silicate glass	Ivovlar-Vivadent

Table 2 –Experimental design

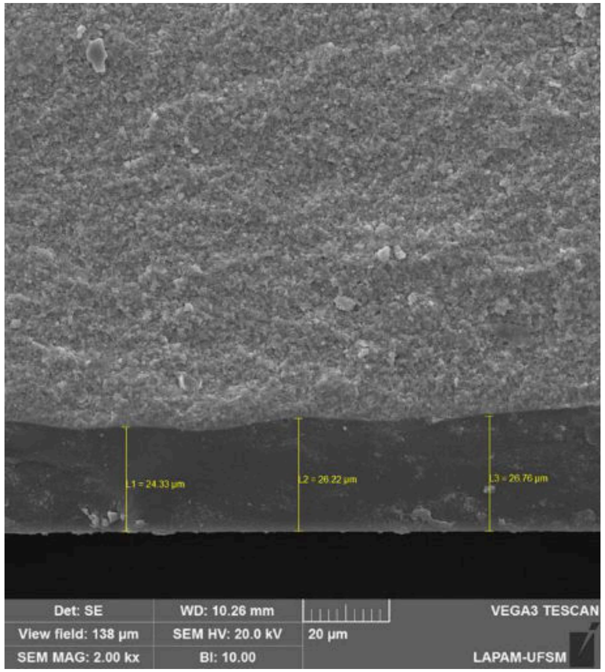
Groups' codes	Surface Treatment
Ctrl	As-sintered (without treatment, control)
Gr	Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm)
Gr+HT	Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm) + Heat treatment
Gr+Pol	Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm) + Polishing
Gr+Pol+HT	Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm) + Polishing + Heat treatment
Gr+Gl	Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm) + Glazing
Gr+Pol+Gl	Grinding with Coarse Diamond Bur # 3101G (average grit size 181µm) + Polishing + Glazing

Table 3 – Data from monotonic biaxial strength mean, initial fatigue strength (60% of monotonic biaxial mean strength) and step size (5% of initial strength) for fatigue testing (staircase); Fatigue strength (σ_f), standard deviation (SD) and 95% confidence interval (CI) obtained on the staircase tests. Besides, percentage (%) of m-phase content and Roughness (Ra and Rz parameter) data are showed.

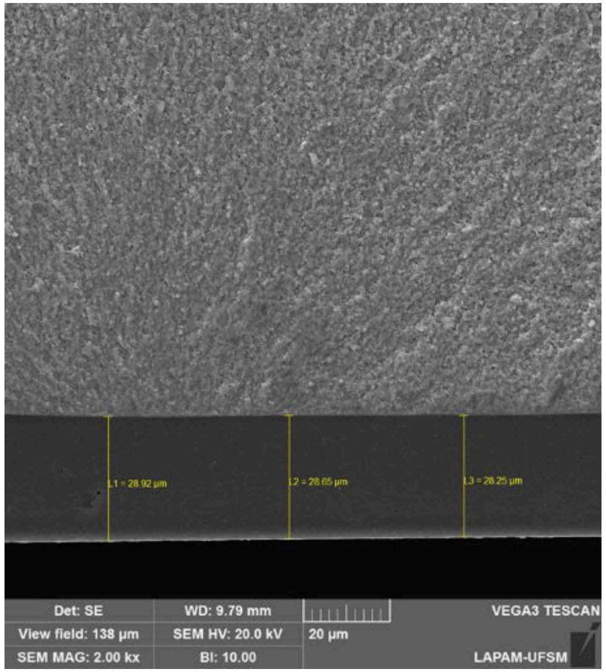
Groups	Monotonic strength means (MPa)	Initial fatigue test strength (MPa)	Step (MPa)	Fatigue strength inMPa		XRD Analysis	Roughness	
				σ_f ± SD*	95% CI	m-phase%	Ra	Rz
Ctrl	765.20	459.12	22.95	525.1 ±14.4 ^{b}	514.29 – 535.91	0.00	0.22 ± 0.10^{a}	1.55 ± 0.59^{bc}
Gr	1120.88	672.53	33.62	719.2 ± 140.1 ^{c}	635.02 – 803.43	14.04 ±0.41	1.10 ± 0.16^{d}	4.97 ± 0.86^{d}
Gr+HT	783.0	469.80	23.49	522.6 ± 17.7 ^{b}	505.43 – 539.87	0.00	0.96 ± 0.13^{c}	4.90 ± 0.48^{d}
Gr+Pol	1003.55	602.13	30.10	651 ± 67.7 ^{c}	607.41 – 694.67	9.78 ± 1.39	0.29 ± 0.05^{b}	1.80 ± 0.32^{c}
Gr+Pol+HT	873.85	575.37	28.76	529 ± 26.7 ^{b}	511.62 – 546.45	0.00	0.28 ± 0.06^{ab}	1.63 ± 0.43^{c}
Gr+Gl	715.44	429.27	21.46	464.5 ± 54.9 ^{a}	426.68 – 502.37	12.64 ± 0.38	0.24 ± 0.11^{ab}	1.24 ± 0.60^{ab}
Gr+Pol+Gl	891.35	534.81	26.74	534.8 ± 64.1 ^{b}	497.48 – 572.14	8.98 ± 2.50	0.17 ± 0.05^{a}	0.93 ± 0.37^{a}

FIGURES

Figure 1 – Representative micrographs (2000x magnification) demonstrating a thin and apparent homogeneous surface of glaze layer (ranging between 24 – 28 µm).



A (Gr+Gl)



B (Gr+Pol+Gl)

Figure 2 – Representative SEM images (5000x magnification) of Y-TZP surface, where it notices that grinding (B) introduced scratches with varying depths (surface topographical alterations), whose alterations were kept after heat treatment (C). Also it notes that the polishing and glazing promoted smoother surfaces (D-G).

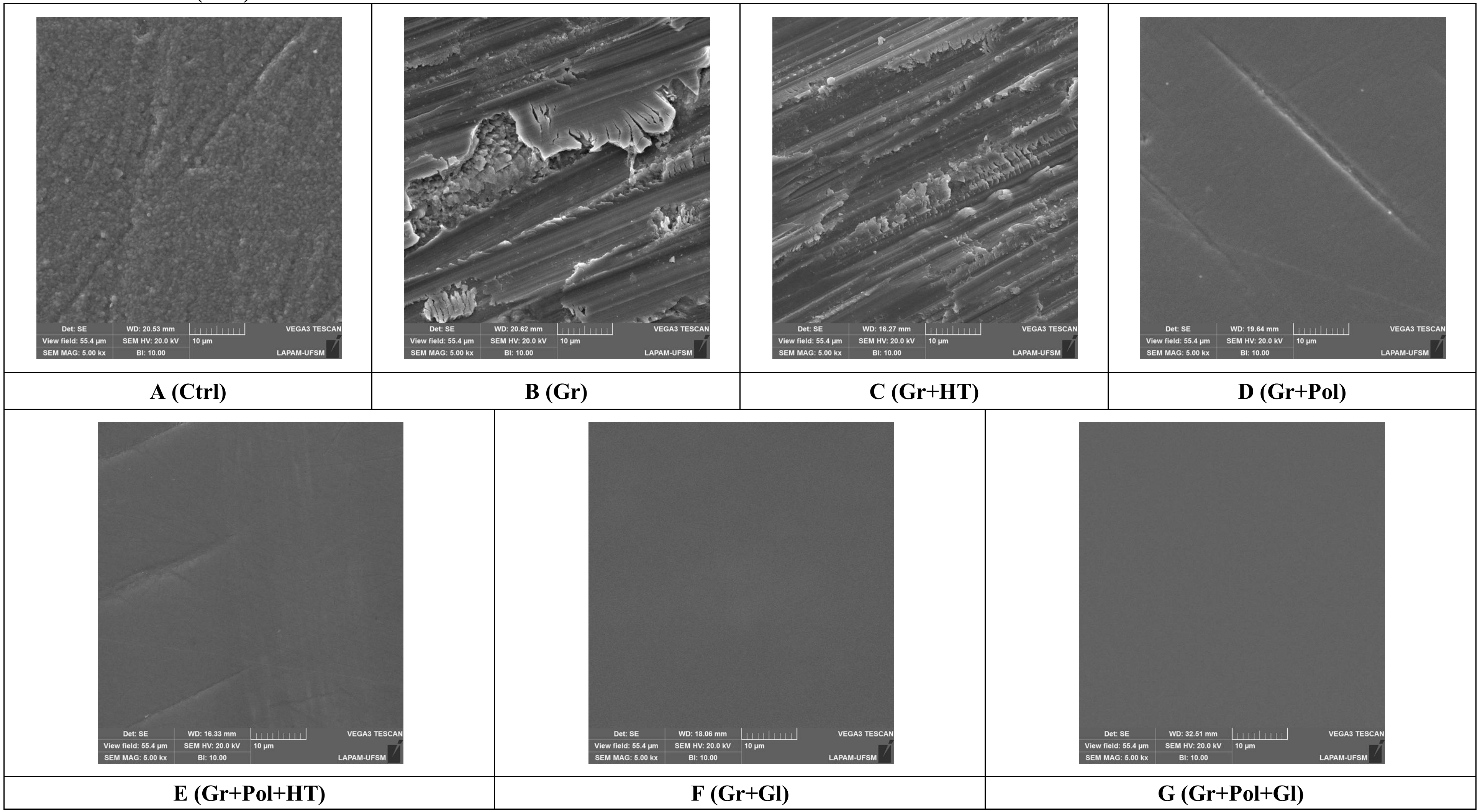
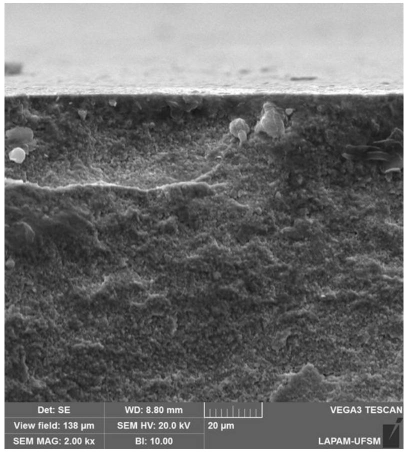
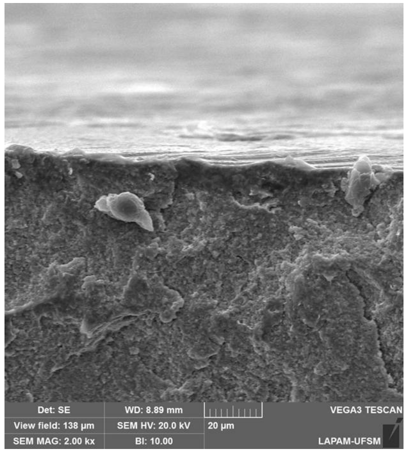
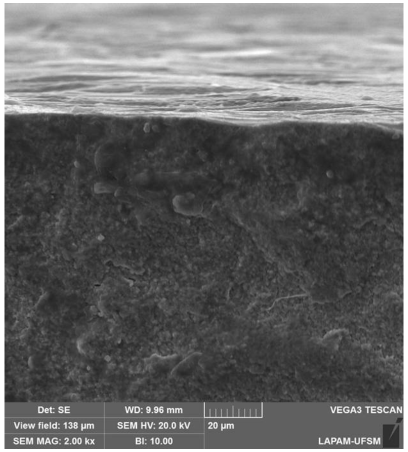
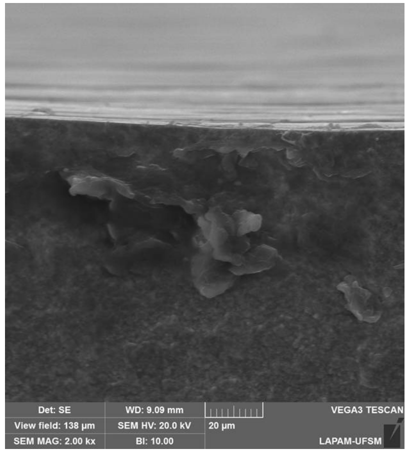


Figure 3 – Representative images of the cross-section regions for the different conditions under SEM (2000x magnification), where it elucidates the presence of surface defects and the surfaces alterations produced by each condition – these results corroborate the findings described in the Figure 2.

			
A (Ctrl)	**B (Gr)**	**C (Gr+HT)**	**D (Gr+Pol)**

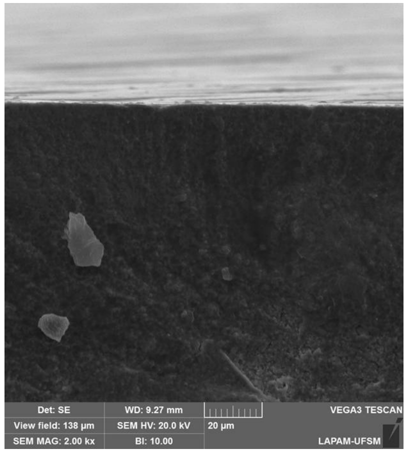
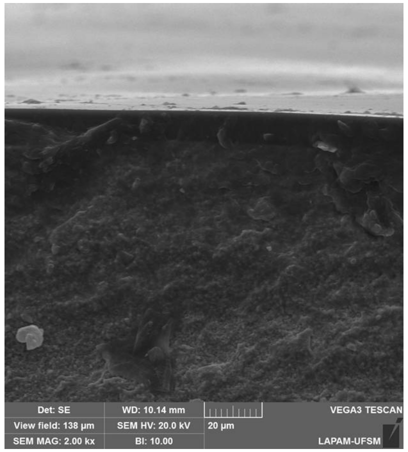
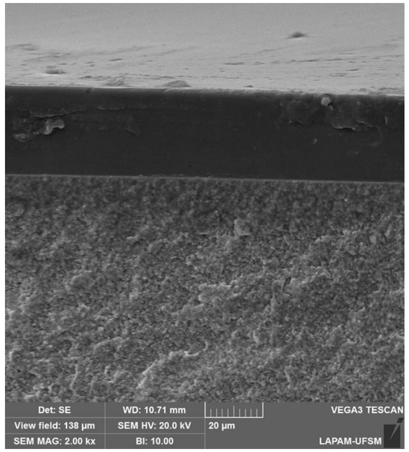
		
E (Gr+Pol+HT)	**F (Gr+Gl)**	**G (Gr+Pol+Gl)**

Figure 4 – Staircase plots obtained through the fatigue testing of all evaluated conditions.

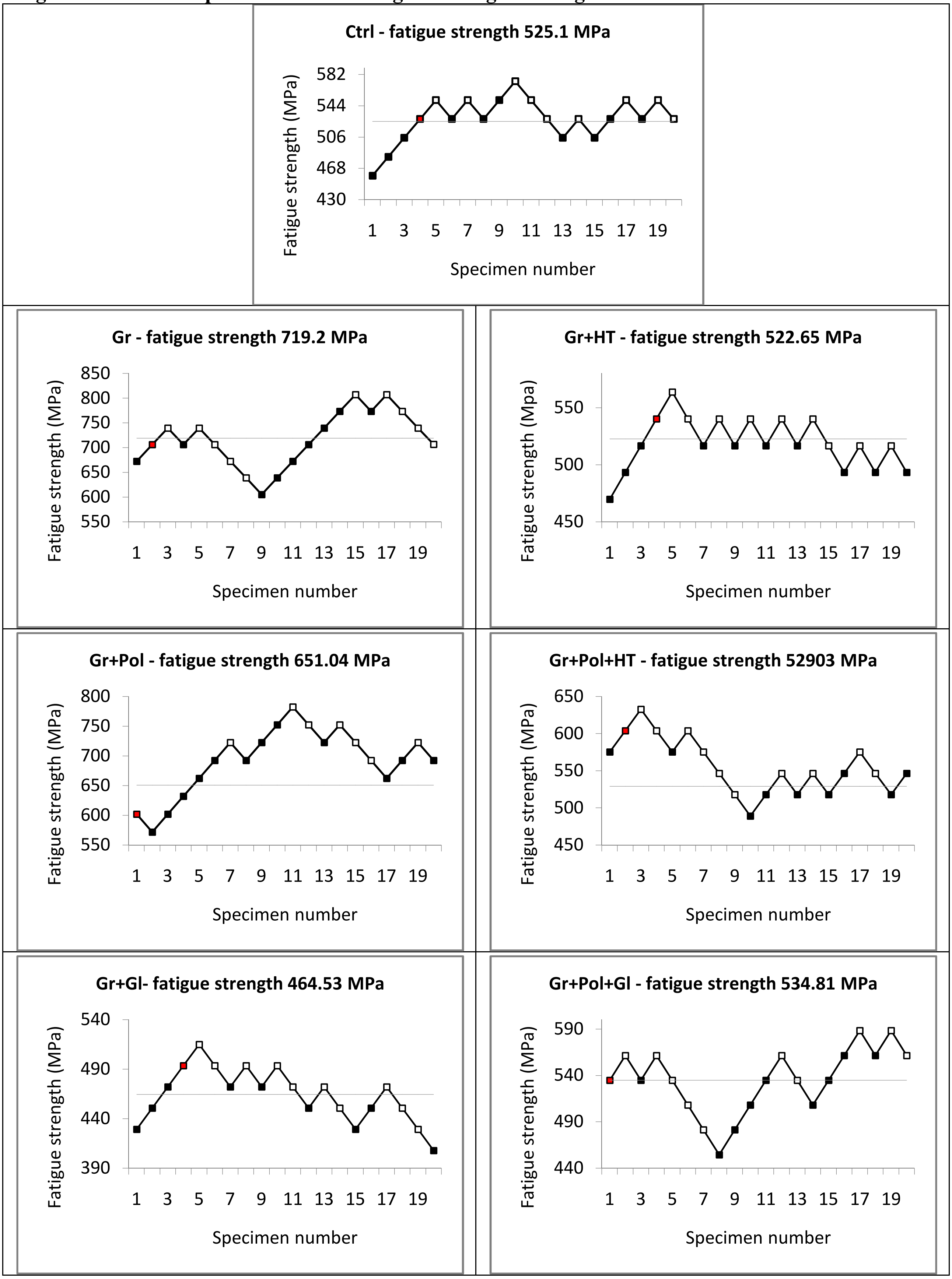
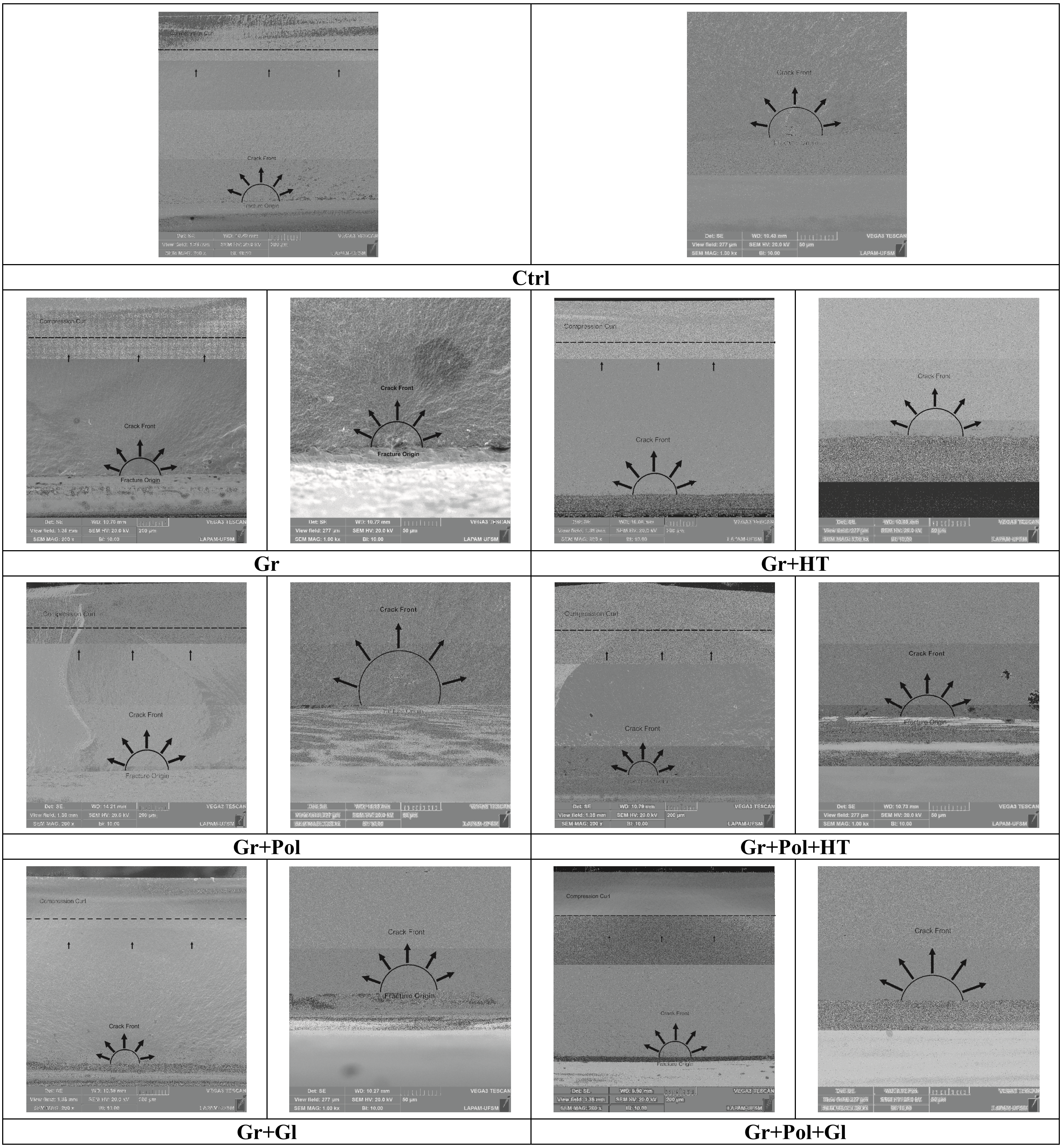


Figure 5 – Representative images of the fractography analysis from all evaluated conditions under SEM (magnification: 200x - left and 1000x - right). It notes that all fractures started (region under the half-circle) on a superficial or sub-superficial (in case of glazed groups) defects on the side where the tensile stresses concentrated during fatigue testing (opposite to the load application). Also depicts that the cracks propagated (direction pointed by arrows) to the opposite site where compression stresses concentrated during testing (compression curl region).



4. ARTIGO 3 - CAD/CAM machining Vs pre-sintering in-lab fabrication techniques of Y-TZP ceramic specimens: effects on their mechanical fatigue behavior

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Running title: Effect of processing on mechanical properties of Y-TZP.

ABSTRACT

This study evaluated the effects of different pre-sintering fabrication processing techniques of Y-TZP ceramic (CAD/CAM Vs. in-lab), considering surface characteristics and mechanical performance outcomes. Pre-sintered discs of Y-TZP ceramic (IPS e.max ZirCAD, Ivoclar Vivadent) were produced using different pre-sintering fabrication processing techniques: Machined- milling with a CAD/CAM system; Polished- fabrication using a cutting device followed by polishing (600 and 1200 SiC papers); Xfine- fabrication using a cutting machine followed by grinding with extra-fine diamond bur (grit size 30 μm); Fine- fabrication using a cutting machine followed by grinding with fine diamond bur (grit size 46 μm); SiC- fabrication using a cutting machine followed by grinding with 220 SiC paper. Afterwards, the discs were sintered and submitted to roughness (n=35), surface topography (n=2), phase transformation (n=2), biaxial flexural strength (n=20), and biaxial flexural fatigue strength (fatigue limit) (n=15) analyses. No monoclinic-phase content was observed in all processing techniques. It can be observed that obtaining a surface with similar characteristics to CAD/CAM milling is essential for the observation of similar mechanical performance. On this sense, grinding with fine diamond bur before sintering (Fine group) was the best mimic protocol in comparison to the CAD/CAM milling.

Key words: Dental Ceramics. Yttrium-stabilized tetragonal zirconia polycrystal. Machining. CAD/CAM fabrication. Fabrication Processing. Mechanical Properties.

1. Introduction

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic has been highlighted due its superior fracture toughness. Thus, it has been recommended for manufacturing frameworks of fixed dental prosthesis (FDPs), which are covered by feldspathic porcelain, and for full-contour monolithic restorations, where coverage by feldspathic porcelain is not necessary (Denry & Kelly, 2014).

The use of Y-TZP ceramics is strictly related to the development/enhancement of Computer-Aided Design/Computer-Aided Machining (CAD/CAM) systems. The advances in CAD/CAM milling have resulted in a high precision, efficient, and accurate system that has reduced the processing timework (Miyazaki et al., 2009). There are two main options for CAD/CAM milling of Y-TZP ceramics: the use of pre-sintered blocks (soft milling) or completely sintered blocks (hard milling) (Zarone et al., 2011).

The main advantage of soft milling in comparison to hard milling of Y-TZP ceramics is the fact that, after manufacturing, the restoration is submitted to a final sintering (Miyazaki & Hotta, 2011) that removes any potential residual stress introduced by milling (e.g. any potential existing monoclinic phase (m-phase) content is removed during final sintering) (Corazza et al., 2015). Besides that, fully sintered Y-TZP blocks are harder to mill, introducing more superficial defects, decreasing the diamond burs longevity, and increasing the time and costs involved in the manufacturing of an all-ceramic restoration (Zarone et al., 2011; Miyazaki et al., 2009; Miyazaki & Hotta, 2011).

In vitro studies that investigate the mechanical behavior of Y-TZP ceramics commonly disregard the damage introduced by CAD/CAM milling. These studies use only simplified fabrication processing techniques, including the use of polished samples (prior to sintering), which do not simulate milling (Flinn et al., 2012; Amaral et al., 2013; Ozcan et al., 2013; Hjerppe et al., 2016; Guilardi et al., 2017).

The main reason in disregarding clinical parameters is that a higher number of ceramic blocks is necessary to prepare simplified specimens (discs and bars) by CAD/CAM milling, when compared with pre-sintering simplified in-lab techniques. In the studies of Fraga and collaborators (2015) and Addison and collaborators (2012), one glass-ceramic block resulted in just one machined disc. If a conventional technique, simulating the damage introduced by milling, had been used to prepare the samples, at least six discs could have been prepared by using the same ceramic block, reducing the costs of the research.

However, the question is: do the pre-sintering simplified in-lab techniques for producing Y-TZP samples mimic the surface aspects and mechanical performance comparable with Y-TZP samples produced by CAD/CAM milling?

Therefore, this study aimed to evaluate the effects of pre-sintering fabrication processing of Y-TZP samples (CAD/CAM milling Vs different in-lab techniques to simulate milling) on the surface characteristics (micrometric and nanometric roughness; topography; phase transformation) and mechanical performance (flexural biaxial strength, fatigue limit, and structural reliability).

2. Materials and Methods

2.1. Specimens manufacturing

Y-TZP (IPS e.max ZirCAD for inLab MO 0 / B-40L, 15.4x19x39 mm^3, Ivoclar Vivadent, Schaan, Liechtenstein) disc shaped specimens were manufactured using 5 different pre-sintering fabrication processes, with final dimensions (post-sintering) of 15 mm in diameter and 1.2 mm (± 0.2 mm) of thickness, according to ISO: 6872-2008 for biaxial flexure strength testing of ceramics.

2.1.1 CAD/CAM Milling (control group)

Y-TZP specimens were milled, as described in a prior study (Fraga et al., 2015), from pre-sintered Y-TZP blocks using a CEREC CAD/CAM system (CEREC inLAB MC XL, Sirona Dental Systems Gmbh, Bensheim, Germany). Each block generated 2 specimens that had been cautiously identified to assure the block origin and sequence of milling. Sintering was performed in a high temperature furnace (VITA Zyrcomat 6000 MS, Vita, Germany) according to manufacturer guidelines.

2.1.2 In-lab fabrication processes

Pre-sintered Y-TZP blocks were detached from the metal holder and 18mm cylindrical metal rings were glued at both ends of the block. The block was ground using 600SiC paper attached to a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA), under water-cooling, until a cylindrical form was achieved. Then, the cylinders were sectioned, under water irrigation, in a cutting machine (ISOMET 1000, Buehler, Lake Bluff, USA) to produce disc samples, which were polished using a 1200 SiC paper (methodology previously elucidated on Pereira et al., 2014). Afterwards, the specimens were subjected to 4 different fabrication processes before sintering:

- *Polished surface:* Samples remained untouched – "polished as-sintered" samples.

- *Grinding with Xfine and Fine Diamond burs:* Two alternative protocols based on grinding with diamond burs were evaluated, one using a fine diamond bur (#3101F – grit size 46µm) and the other using an extra-fine diamond bur (#3101FF – grit size 30µm; KG Sorensen, Cotia, Brazil). Grinding was performed by a single trained operator with a slow-speed motor (Kavo Dental, Biberach, Germany) associated with a contra-angle handpiece (T2 REVO R170 contra-angle handpiece up to 170,000 rpm, Sirona, Bensheim, Germany) under constant water-cooling (≈30mL/min), and the diamond bur was replaced after each specimen. To improve the reproducibility, to standardize the wear thickness, and to guarantee that the entire surface was ground, the specimens were marked with a red pencil (Pentel, Tokyo, Japan). Grinding was performed manually with horizontal movements until the complete removal of the marking pencil.

- *Grinding with SiC papers:* Grinding was performed manually using a 220 SiC paper under water-cooling. In order to provide better reproducibility, standardize the wear thickness, and guarantee that the entire surface was ground, the specimens were marked with a red pencil

(Pentel, Tokyo, Japan). Then, the grinding procedure was performed manually with horizontal movements for 25 cycles, which completely eliminated the marking.

After performing the pre-sintering treatments the specimens were sintered according to manufacturer guidelines.

2.2. Surface topography and roughness analysis

The surface topography presented on each evaluated condition were analyzed using a surface roughness tester (n= 35, MitutoyoSJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan), a field emission scanning electron microscope (FE-SEM) (n = 2, FE-SEM Inspect F50, FEI, Hillsboro, Oregon, USA), and an atomic force microscope (AFM) (n = 5, Agilent Technologies 5500 equipment, Chandler, Arizona, USA).

For the analysis with the surface roughness tester (micrometric analysis) six measurements (measured range up to 80µm, with an accuracy of 0.001µm) were conducted for each specimen (3 along the grinding direction, 3 in a perpendicular direction), considering the ISO: 4287-1997 parameters (Ra and Rz) with a cut-off (n=5), λC 0.8 mm and λS 2.5 µm. Arithmetic mean values of all measurements from each specimen were obtained.

For field emission scanning electron microscopy, two specimens from each group were subjected to sputter-coating with a gold-palladium alloy and images were obtained at 5000x magnification.

For atomic force microscopy (nanometric analysis), five specimens from each group were subjected to the analysis. The images were obtained using a non-contact methodology and specific probes from an area of 10 x 10µm (PPP-NCL probes, Nanosensors, Force constant = 48 N/m) and a specific software (Gwyddion™ version 2.33, GNU, Free Software Foundation, Boston, MA, USA) was used to obtain the Sq roughness parameter (nanometric root mean square roughness of the evaluated area).

2.3. Phase analysis (X-Ray Diffractometry - XRD)

In order to evaluate the content of the monoclinic phase, samples (n = 2) were submitted to XRD analyses (Bruker AXS, D8 Advance, Karlsruhe, Germany; Cu Kα radiation with 0.15406 nm, spectra collected in the 2θ range of 25–35°, step of 1 s, and step size of 0.03°) through the Garvie & Nicholson method (1972) modified by Toraya and collaborators (1984). This method has being extensively used (Pereira et al., 2015 [b]) and is described at previous literature (Amaral et al., 2013; Guilardi et al., 2017; Flinn et al., 2012; Pereira et al., 2016[a]; Pereira et al., 2016[b]; Fonseca et al., 2014).

2.4. Biaxial flexure test

Samples (n = 20) were subjected to biaxial flexure strength test according to ISO: 6872-2008.

Disc-shaped specimens were positioned with the treated surface facing down (tensile stress) on three support balls (Ø=3.2 mm), which were placed 10 mm apart from each other in a triangular position. The assembly was immersed in water and a flat circular tungsten piston (Ø=1.6 mm) was used to apply an increasing load (1 mm/min) until catastrophic failure using an universal testing machine (EMIC DL 2000, São José dos Pinhais, Brazil). Before testing, an adhesive tape was fixed on the compression side of the discs to avoid dispersal of fragments (Quinn, 2007), and also to provide better contact between the piston and the sample (Wachtman et al., 1972). Flexural strength was calculated according to ISO:6872-2008, as described on previous literature (Amaral et al., 2013; Pereira et al., 2014; Guilardi et al., 2017; Fonseca et al., 2014).

2.5. Flexural fatigue strength test

Samples (n=15) were subjected to a biaxial flexural fatigue test in an electrical machine (InstronElectroPuls E3000, Instron Corporation, Norwood, MA, USA) according to ISO:6872-2008. The test assembly was executed as described previously for biaxial flexure test.

The biaxial flexure fatigue limit was determined for each group with a lifetime of 20,000 cycles using the staircase approach method described by Collins (1993). Sinusoidal loading was applied, with amplitude ranging from a minimum of 10 MPa (in order to avoid the movement of the specimen) to the maximum force, with a frequency of 6 Hz (Pereira et al., 2016[b]).

The initial stress and the step size were determined for each condition based on the results of the monotonic biaxial tests (Table 2). 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 stress, respectively. The fatigue testing was controlled by stress, and the load (N) required to achieve the desired stress (MPa) was calculated according to ISO: 6872-2008, for each tested sample.

After testing, the mean biaxial flexure fatigue limit (σ_f) and confidence interval (CI - 95%, α = 0.05) was calculated, according to Collins (1993), based on the data of the less frequent event (survival or failure), as described by Villefort and collaborators (2017) and by Pereira and collaborators (2016[b]).

2.6. Data Analysis

Micrometric roughness data (Ra, Rz) assumed a nonparametric distribution (Shapiro-Wilk test); therefore Kruskal-Wallis and Dunn's post-hoc tests were performed. The Pearson Correlation test was carried out between Ra and monotonic biaxial flexural data. Nanometric roughness data (Sq) assumed a parametric distribution (Shapiro-Wilk test); therefore, one-way ANOVA and Tukey's post-hoc tests were performed.

The statistic used to describe the reliability of the ceramic material was based on the Weibull statistical analysis (Weibull, 1951), which is a way to describe the variation of resistance obtaining the Weibull modulus (m) and the characteristic strength (σ_C) with a confidence interval of 95%, as determined in a diagram according to DIN ENV 843-5(2007).

The characteristic strength is considered the strength at a failure probability of approximately 63%, with the Weibull modulus used as a measure of the distribution of strengths, expressing the reliability of the material.

The biaxial flexural fatigue limit data assumed a parametric distribution (Shapiro-Wilk test); therefore, one-way ANOVA and Tukey's post-hoc tests were performed.

2.7. Fractographic analysis

A fractographic examination was performed by a single trained operator using a light microscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany), where representative specimens for each evaluated condition were chosen and submitted to further evaluation in a Field Emission Scanning Electron Microscope to determine the origin of the fracture.

3. Results

FE-SEM and AFM topography analyses (Fig. 1) indicated that the Polished group presented a smoothed surface, which was a completely different topographical pattern when compared to the other conditions. The alternative fabrication techniques (Xfine, Fine, SiC) for simulating CAD/CAM milling produced an increase in surface roughness, approximating to the surface pattern produced by CAD/CAM milling. Grinding with fine diamond burs (Fine group) produced the most similar topographical pattern to the Machined condition.

Roughness analysis (Table 2), considering both micrometric (Ra and Rz) and nanometric roughness (Sq) parameters, indicated that Machined and Fine groups had similar and higher Ra and Rz roughness values. The correlation test demonstrated that there was a statistically significant negative correlation between Ra roughness and monotonic flexural strength ($r = -0.682$; $P<0.001$).

XRD analysis (Table 2) shows a lack of m-phase content for all evaluated conditions, indicating that any possible residual stress or phase transformation triggered by the fabrication process was completely eliminated during sintering.

Weibull analysis of the monotonic biaxial flexural strength data shows that the Machined condition presented the lowest values, which was only comparable to the fine diamond bur condition. The pre-sintering treatments had no deleterious effect on the Weibull moduli indicating that the variability of strength data was similar between all evaluated conditions (Table 2).

The fatigue limits of the pre-sintering treatments (Xfine, Fine, SiC) were similar to the Machined group, while the Polished group had the highest fatigue limit value (Table 2; Fig. 3).

Fractographic analysis (Fig. 2) shows that the initial crack nucleation sites were located in superficial defects on the lower side of the specimen, corresponding to the region of maximum tensile stress during biaxial test. Additionally, the shape of the defects (Fig. 2 – 5000x magnification) for fracture initiation was directly related to the pre-sintering treatment: grinding with fine diamond burs (Fine group) more closely simulated the shape of the Machined condition.

4. Discussion

The current findings generally indicate that only fabrication processing with a fine diamond bur was able to successfully simulate the effects of the CAD/CAM milling technique on the Y-TZP surface, considering both surface characteristics and mechanical performance. These results indicate that the fine diamond bur technique (Fine group) was the most similar technique to CAD/CAM milling and that additional polishing before sintering might result in misinterpretation of mechanical performance (possible overestimation).

The knowledge and the characterization of the effects of milling on the mechanical performance and surface properties of ceramics are essential (Addison et al., 2012). Wang and collaborators (2008) stated that different fabrication processes might influence the strength of Y-TZP ceramics, as they introduce different sizes and shapes of defects, cracks and damage on the material's surface. With this in mind, Jing and collaborators (2014) and Addison and collaborators (2012) stated that micro-defects and residual stresses are introduced during zirconia restoration production from industrial production, lab preparations, and clinical adjustments. These micro-defects and residual stresses are cumulative and determine the microstructure resilience and the final mechanical properties of Y-TZP ceramics.

Flury and collaborators (2012) stated that surface roughness might play a crucial role in the resistance of ceramics, usually with a significant negative correlation with flexural strength (higher roughness produce slower flexural strength). Our findings corroborate that affirmation, since the roughness data and respective monotonic flexural data for all tested conditions indicated a significant negative correlation between those outcomes (Guess et al., 2010; Fraga et al., 2015; Pereira et al., 2014).

It is already well established that deep surface flaws can act as stress concentrators, reducing the strength values of ceramics (Green, 1983; de Jager et al., 2000). The current literature (Yin et al, 2006; Quinn et al., 2005; Pereira 2016[c]) shows that grinding could introduce damage that varies from deep scratches (in addition to chipping) associated with penetrating median cracks, to subsurface lateral cracks and shallow scratches, depending on the grit-size, applied load, and grinding speed.

Literature has shown that, when submitted to stress, Y-TZP ceramics trigger a defensive transformation toughening mechanism (t- to m-phase), leading to local volumetric expansion (approximately 3-5%) that can result in compressive stress around an existing crack tip (or existing defects), making it more difficult for a crack to initiate/propagate (Gupta et al., 1978; Hannink et al., 2000; Pereira et al., 2015[a]; Pereira et al., 2015[b]; Pereira et al., 2016[a]; Hjerppe et al., 2016). Our data show that the final mechanical performance was determined by the presence of defects introduced by the pre-sintering fabrication techniques. Since these procedures were executed before sintering, no m-phase content was observed after sintering (i.e. any existing m-phase content was reverted to t-phase configuration).

Polishing or glazing treatments can control the external surface roughness of a restoration; however, it is not possible to modify the internal surface of a restoration because this would affect the adaptation to the prepared tooth (de Kok et al., 2015). The literature has already developed a consensus that the clinical reason for a restoration to fracture is mainly caused by masticatory stresses concentrating on minor surface flaws at the cement interface, leading an initial crack to propagate radially towards the surface under tensile pressure (Kelly et al., 2010; de Jager et al., 2000; Aboushelib et al., 2007). Therefore, if we consider an *in vitro* scenario where we assume that a polished surface simulates an intaglio surface of a restoration, a biased outcome might be observed (e.g. fractures originating on the top of the surface – surface submitted to compression stress; or higher fracture strength – overestimation).

Finally, our findings corroborate that the introduction of approximately similar size and shape of defects into the surface of Y-TZP, as occurs by CAD/CAM milling, is crucial for more realistic *in vitro* evaluations. We observed that the best condition for mimicking the behavior of the machined scenario is grinding the pre-sintered zirconia with fine diamond burs (Table 2, Fig. 1 and Fig. 2). The main limitation of this study is: when cementing a milled zirconia restoration, the cement may interact with the existing defects on the intaglio surface of the restoration, which might modify the outcomes.

5. Conclusion

- It is necessary to simulate the surface of Y-TZP ceramic milled by CAD/CAM to evaluate and characterize the mechanical behavior of this material; otherwise, the mechanical properties might be misinterpreted.
- Grinding of a zirconia surface with fine diamond burs before sintering is the closest method of reproducing a surface milled by CAD/CAM.

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TABLES

Table 1– Micrometric (from roughness tester) and nanometric (from AFM analysis) roughness data (mean ± standard deviation- SD); Structural stability analysis (phase transformation analysis for m-phase content determination under XRD); Monotonic biaxial flexural strength data (mean strength -σ and standard deviation – SD; and Weibull analysis with Weibull moduli – m and characteristic strength σ_c and respective confidence intervals (CI 95%)); and Biaxial flexural fatigue strength data with initial load (correspondent to 60% of monotonic mean biaxial strength load), step size (correspondent to 5% of monotonic mean biaxial strength load) and fatigue limits (mean ± standard deviation).

Pre-sintering fabrication techniques	Roughness Analysis			XRD analysis (m-phase %)	Monotonic biaxial flexural strength test (MPa)			Biaxial flexural fatigue strength testing (MPa)		
	Ra (μ*m*)	Rz (μ*m*)	Sq (*nm*)		σ ± SD	Weibull Analysis: m (CI 95%)	Weibull Analysis: σ_c (CI 95%)	Initial load (0.6 σ)	Step size (0.05 σ)	Fatigue Limit
Machined	1.8 ± 0.2[b]	11.4 ± 0.9[b]	102.2 ± 16.5[b]	0	598.6 ± 39.9	17.9 (11.2 - 24.3)[ab]	620.4 (601.1 - 640)[c]	359.2	29.9	391.2 ± 27.1[a]
Polished	0.4 ± 0.7[a]	3.2 ± 0.4[a]	61.9 ± 10.3[a]	0	933.6 ± 102.5	12.5 (7.8 - 17)[a]	771.5 (737.4 - 806.6)[a]	560.1	46.7	556.8 ± 24.7[b]
Xfine	1.4 ± 0.3[a]	8.3 ± 1.4[a]	76.0 ± 13.4[ab]	0	627.0 ± 78.1	10.7 (6.7 - 14.4)[ab]	689.3 (653.6 - 726.3)[b]	376.2	31.4	418.0 ± 93.2[a]
Fine	1.8 ± 0.2[b]	10.7 ± 1.3[b]	100.6 ± 16.1[b]	0	746.1 ± 115.2	8.1 (5.0 - 10.9)[a]	638.8 (595.5 - 684.7)[bc]	447.7	37.3	397.0 ± 26.4[a]
SiC	1.2 ± 0.2[a]	7.1 ± 1.4[a]	105.3 ± 28.8[b]	0	552.6 ± 80.5	11.7 (7.3 -15.9)[ab]	658.6 (627.6 - 690.8)[b]	331.6	27.6	373 ± 52.5[a]

*Similar letters indicate no statistically significant difference among evaluated conditions at the same column.

FIGURES

Figure 1- SEM images (5000x magnification – left columns) and AFM images (10 µm X 10 µm – right column) of Y-TZP surface demonstrating the topographical patterns obtained on sintered samples after each surface treatment executed previous to sintering.

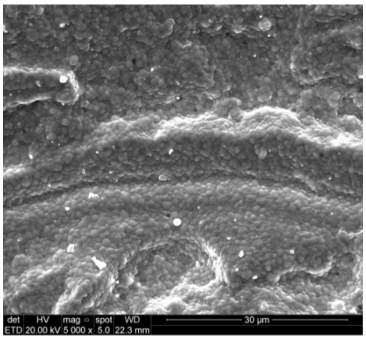
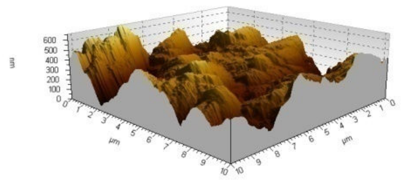
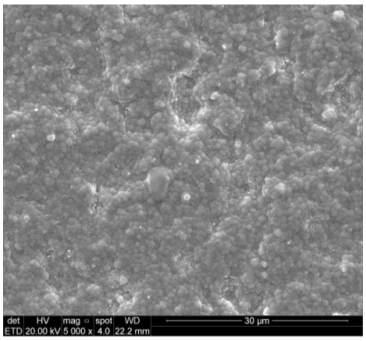
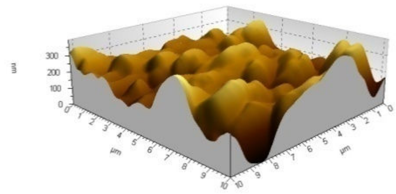
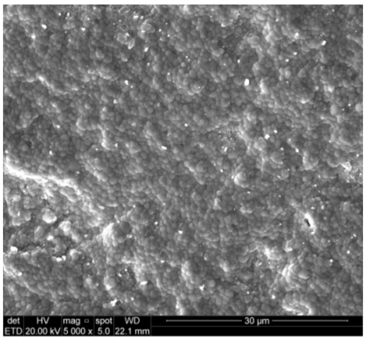
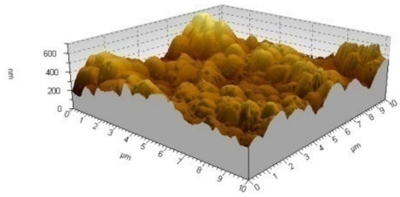
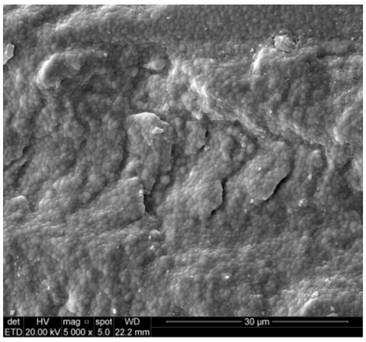
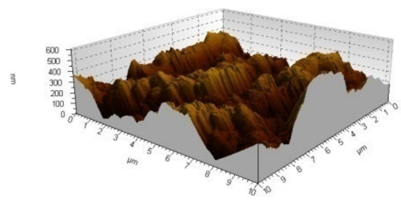
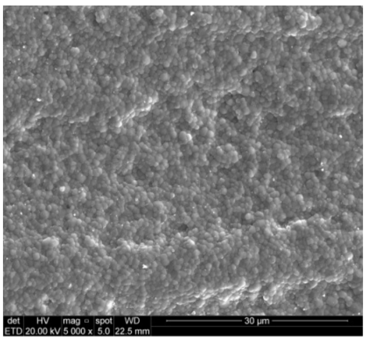
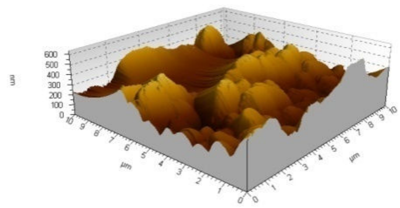
	SEM (5000x)	AFM (10 µm X 10 µm)
Machined		
Polished		
Xfine		
Fine		
SiC		

Figure 2 – Fractography analysis on SEM (200x and 5000x magnification), showing typical brittle fractured surfaces, with initial crack nucleation sites located on superficial defects at the region of maximum tensile stress (opposed side to compression tip) during the biaxial tests. The pointer indicates the fracture origin.

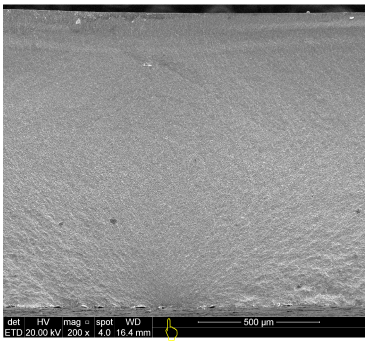
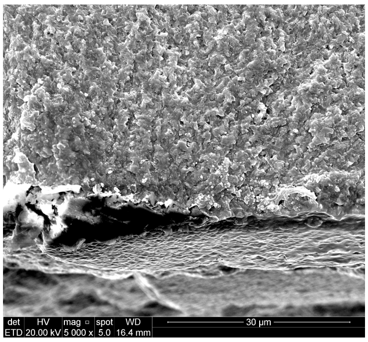
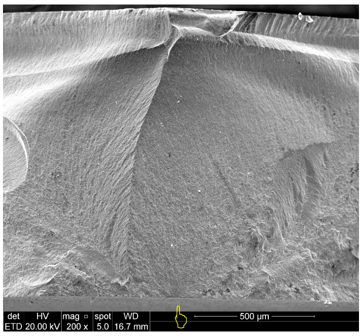
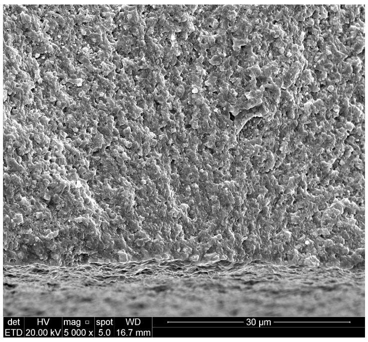
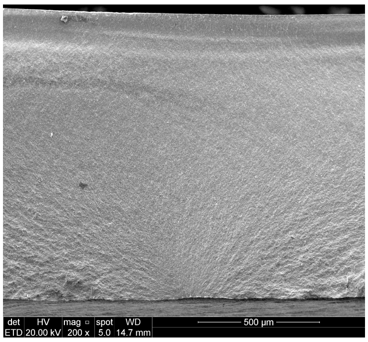
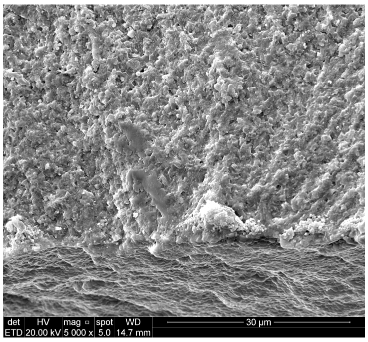
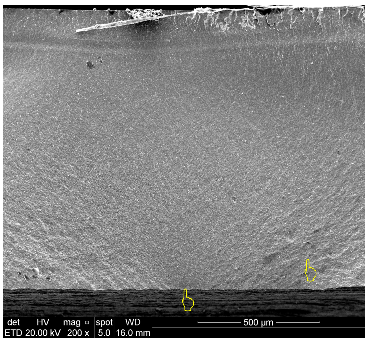
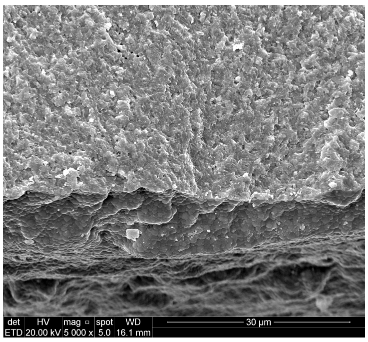
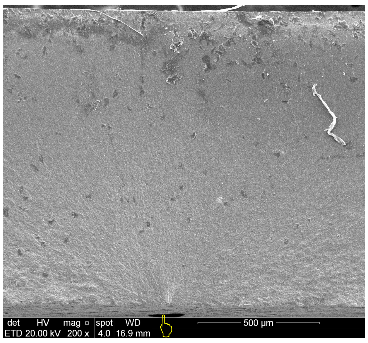
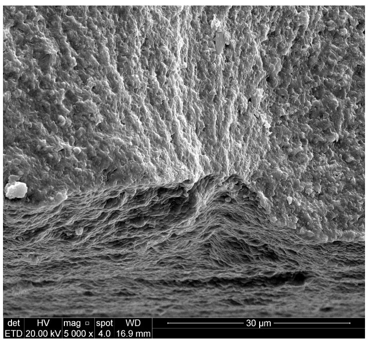
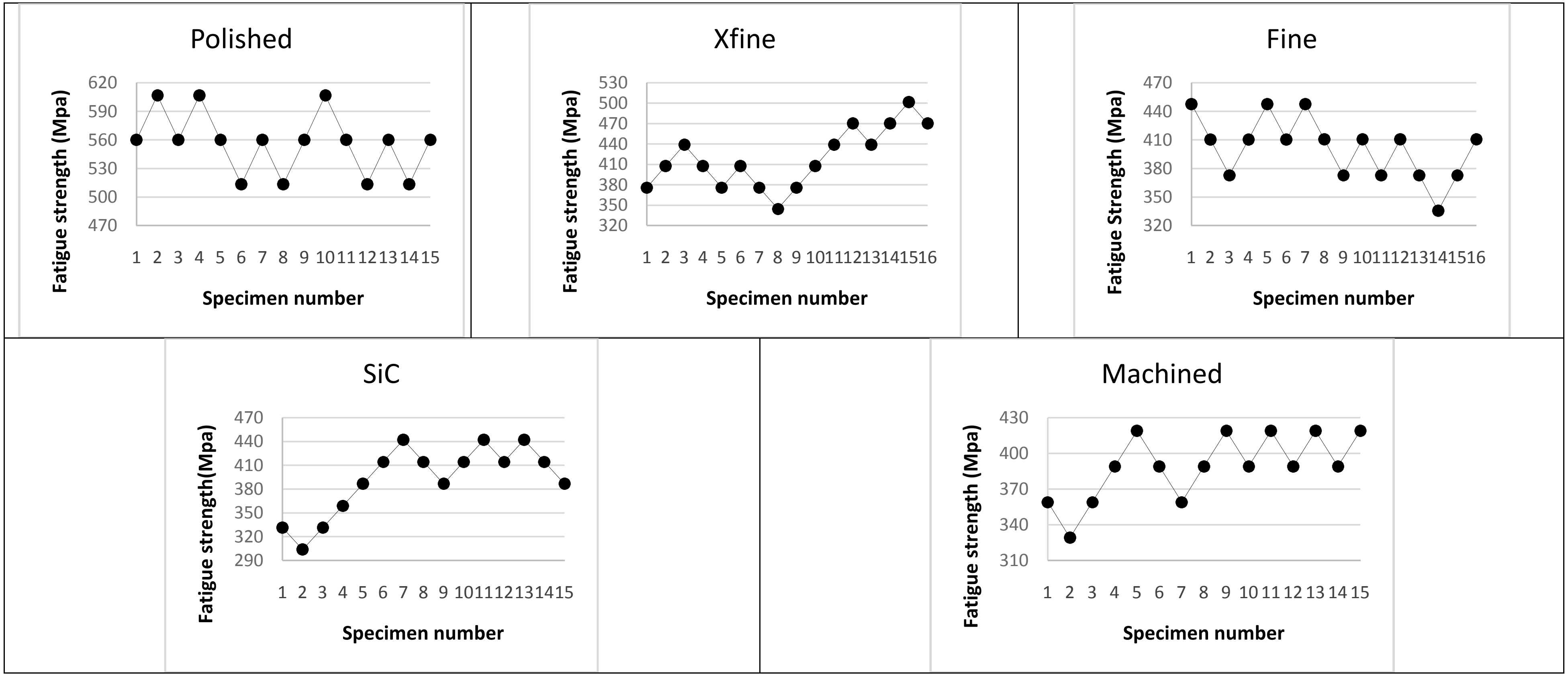
	SEM	
	200x	**5000X**
Machined		
Polished		
Xfine		
Fine		
Sic		

Figure 3– Pattern of runouts (survival) and failures for each group observed during fatigue testing.



5. DISCUSSÃO

Com base nos estudos 1 e 2 desta dissertação, verificamos que o desgaste com ponta diamantada introduziu defeitos superficiais no material. Porém, devido ao mecanismo de tenacificação desencadeado pela transformação de fase (*t-m*) (CHEVALIER, GREMILLARD e DEVILLE, 2007; DENRY & HOLLOWAY 2006; PEREIRA et al., 2014), a resistência a fratura e o comportamento a fadiga do material foi otimizado.

Pelo fato da zircônia ser uma cerâmica policristalina sem qualquer conteúdo vítreo (RAIGRODSKI et al., 2012), o tratamento térmico não promoveu qualquer alteração na rugosidade superficial após o desgaste. Entretanto, devido ao relaxamento das tensões ocasionado pela transformação reversa de fase (*m-t*) (GUAZZATO et al., 2005), o grupo que recebeu este tratamento apresentou uma deterioração no comportamento mecânico (resistência característica, fadiga) em relação ao grupo desgastado e polido.

Em ambos os estudos 1 e 2, a aplicação do glaze reduziu significativamente a rugosidade superficial gerada pelo desgaste. Porém, todos os grupos que receberam glaze apresentaram os piores desempenhos mecânicos. De acordo com BORBA et al., 2011, a interface de restaurações bicamada (neste caso glaze/zircônia), pode ter uma forte influência no desempenho mecânico da restauração, especialmente quando há uma grande incompatibilidade entre os sistemas (WUTTIPHAN et al., 1996). Além disso, a eventual presença de bolhas na camada do glaze (resultado do aprisionamento de ar entre a mistura), como observado na figura 2 do estudo 1, pode aumentar o risco de falha nesta interface (CAZZATO & FABER, 1997).

O glaze quando associado ao polimento apresentou uma melhora no comportamento mecânico quando comparado ao grupo que recebeu glaze somente. Assim, em regiões estéticas quando a aplicação de uma fina camada de glaze na restauração cerâmica é indispensável para se reproduzir as características ópticas dos dentes naturais, torna-se essencial a realização de um polimento prévio.

Como mencionado anteriormente, o polimento demonstrou ter um efeito benéfico na resistência mecânica da zirconia, tanto em ensaio monotônico (estudo 1) quanto no comportamento em fadiga (estudo2). Durante o polimento, pode ocorrer um aumento da temperatura local, e dessa forma desencadear a transformação reversa de fase (*m-t*) (KOSMAC et al., 2000), o que foi observado em nossos achados de maneira sutil (pequena redução de conteúdo de fase *m*). Além disso, o polimento causou uma significativa redução da rugosidade superficial, restabelecendo a lisura superficial inicial. Estes achados estão de acordo com a literatura (HUSIAN, CAMILLERI e ÖZCAN, 2016; PREIS et al., 2015[a]; PREIS et al., 2015[b]).

Ainda neste contexto, o estudo 1 também avaliou o efeito da LTD (Low Temperature Degradation) associada aos diferentes tratamentos posteriores ao desgaste. O envelhecimento em autoclave não promoveu efeitos na rugosidade superficial, porém produziu um aumento no conteúdo de fase monoclínica (tabela 2, estudo 1). A LTD é considerada indesejável, pois, o excesso de tensão residual introduzida pela transformação de fase gera microtrincas que podem se propagar para o interior do material, reduzindo assim as propriedades mecânicas deste (PAPANAGIOTOU et al., 2006). Entretanto, em nenhuma das condições avaliadas neste estudo, foi observada a degradação das propriedades mecânicas causada pelo envelhecimento, o que corrobora com outros achados em termos de comportamento mecânico das cerâmicas Y-TZP avaliadas frente à LTD (AMARAL et al., 2013; KIM et al., 2009; XIE et al., 2016).

No que se refere às técnicas de confecção de espécimes (estudo 3), observamos que a utilização de técnicas que mimetizem a superfície obtida pelo CAD/CAM é essencial para melhor relacionar as condições clínicas-laboratoriais, tendo em vista que a superfície usinada apresenta uma rugosidade aumentada, com defeitos superficiais, o que consequentemente afeta o desempenho mecânico da cerâmica. Estes achados estão de acordo com as observações de Wang et al., (2008) que declararam que diferentes processos de fabricação podem influenciar na resistência do material.

Neste sentido, nota-se que o método comumente empregado em vários estudos para a obtenção de espécimes (AMARAL et al., 2013; SOUZA et al., 2013; OZCAN et al., 2013; PEREIRA et al., 2016; HJERPPE et al., 2016; GUILARDI et al., 2017) não leva em consideração esses aspectos, o que pode conduzir a uma avaliação enviesada. Portanto, para que as características da cerâmica sejam avaliadas com maior precisão, torna-se necessária a confecção de espécimes que simulem os efeitos gerados pela usinagem. De acordo com os resultados do estudo 3, a técnica *in-lab* que mais se aproxima tanto do ponto de vista de características superficiais quanto do comportamento mecânico, é a confecção de espécimes em máquina de corte com disco diamantado, seguido pelo desgaste com ponta diamantada de granulação Fina (46µm) previamente a sinterização.

4. CONCLUSÃO

Estudos 1 e 2:

- Após a realização de desgaste com ponta diamantada de uma cerâmica Y-TZP, torna-se essencial a realização de um protocolo de polimento para redução da rugosidade e otimização do comportamento mecânico;
- Em situações estéticas em que a aplicação de glaze é indispensável, o polimento da superfície deve ser realizado previamente ao glaze;
- O tratamento térmico, deve ser utilizado com cautela, visto que seus efeitos causam degradação das propriedades mecânicas do material.

Estudo 3:

- A utilização de uma técnica de confecção de espécimes que reproduza os efeitos gerados pela usinagem em CAD/CAM é necessária para a correta avaliação das propriedades superficiais e mecânicas do material;
- O desgaste do espécime com ponta diamantada com granulação 'fina' (46µm) é o método que melhor reproduz os efeitos gerados pela usinagem em CAD/CAM.

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ANEXO A – AUTHOR GUIDELINES FOR APPLIED SURFACE SCIENCE

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ANEXO B - AUTHOR GUIDELINES FOR JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS.

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