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COMPORTAMENTO MECÂNICO EM FADIGA DE YSZ: EFEITOS DE DIFERENTES TRATAMENTOS DE SUPERFÍCIE E MATERIAIS A BASE DE ZIRCONIA

Santa Maria, RS
2019

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Tese apresentada ao Programa de Pós-graduação em Ciências Odontológicas, da Universidade Federal de Santa Maria (UFSM, RS). Área de Concentração em Odontologia, como requisito para a obtenção do título de **Doutora em Ciências Odontológicas**.

Orientador: Prof. Dr. Luiz Felipe Valandro
Co-orientador: Prof. Dr. Gabriel Kalil Rocha Pereira

Santa Maria, RS
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“Algum dia os seus olhos verão aquilo que você tanto pediu à Deus”.

Autor Desconhecido

RESUMO

COMPORTAMENTO MECÂNICO EM FADIGA DE YSZ: EFEITOS DE DIFERENTES TRATAMENTOS DE SUPERFÍCIE E MATERIAIS A BASE DE ZIRCÔNIA

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A presente tese está composta por quatro estudos com respectivos objetivos: estudo 1- avaliar a carga para falha em fadiga, número de ciclos para falha e taxa de sobrevivência em discos de zircônia de segunda e terceira geração cimentados adesivamente em substrato análogo à dentina; estudo 2- avaliar e comparar o efeito de três sistemas de polimento associados ou não ao acabamento prévio com pontas diamantadas de granulação fina e extra-fina, nas características superficiais (rugosidade e topografia), transformação de fase e comportamento à fadiga de uma cerâmica Y-TZP desgastada por ponta diamantada grossa; estudo 3- avaliar e comparar o efeito de duas formas de aplicação de glaze (pincel e spray) no comportamento à fadiga e características superficiais (topografia e rugosidade) de uma cerâmica Y-TZP antes e após o desgaste com ponta diamantada; estudo 4- avaliar o efeito de tratamentos de superfície (desgaste, polimento e glaze) realizados na superfície oclusal, nas características superficiais (topografia e rugosidade), carga para falha em fadiga, número de ciclos para falha e transformação de fase de uma zirconia FSZ cimentada adesivamente em substrato análogo de dentina. No estudo 1, foram utilizadas quatro cerâmicas de segunda geração (Lava Plus, 3M ESPE; Vita In-Ceram YZ-HT, VITA Zahnfabrik; Zirlux FC, IvoclarVivadent; Katana ML-HT, Kuraray) e duas de terceira geração (Katana UTML and Katana STML, Kuraray). Os discos cerâmicos foram cimentados adesivamente em discos de resina epóxi. Testes de fadiga, análise de fase e fractográfica foram realizadas. No estudo 2, discos de Y-TZP (Zenostar T) foram confeccionados com: Ø=15 mm, 1.2 ± 0.2 mm de espessura e divididos em 8 grupos: Ctrl– sem tratamento; Gr– desgastado; Gr+Eve– desgastado e polido com EveDiacera (sistema de dois passos); Gr+Fin+Eve– desgaste+ acabamento + polimento com EveDiacera; Gr+Kg– desgaste + polimento Kg Viking (sistema de dois passos); Gr+Fin+Kg– desgaste + acabamento + polimento com Kg Viking; Gr+Op– desgaste + polimento com Optrafine (sistema de três passos); Gr+Fin+Op– desgaste + acabamento + polimento com Optrafine. Após, análise de topografia, rugosidade, transformação de fase, resistência a fadiga e fractografia foram realizadas. Estudo 3, os espécimes cerâmicos foram divididos em 6 grupos: Ctrl sem tratamento; Gr – desgastado com ponta diamantada; Br – aplicação de glaze pó/líquido; Sp – aplicação de glaze spray; Gr+Br – desgaste +glaze pó/líquido; Gr+Sp – desgaste + glaze spray. Análises de rugosidade de superfície, resistência à fadiga e fractografia foram realizadas. Estudo 4 discos de IPS e.max Zircad MT Multi foram produzidos e divididos em 5 grupos: Ctrl– sem tratamento; Gr– desgastado; Gr+Pol– desgaste + polimento com sistema de dois passos; Gr+Gl– desgastado + aplicação de glaze; Gr+Pol+Gl– desgaste + polimento + aplicação de glaze. Após, os discos foram cimentados com cimento resino em discos de resina epóxi. Análises de rugosidade superficial, teste de fadiga, fractografia e drx foram realizados. Em relação ao estudo 1 observou-se que zircônias de segunda geração apresentam maior carga para falha em fadiga e número de ciclos até a falha do que zircônias de terceira geração. No estudo 2 verificou-se que o polimento após desgaste reduziu a rugosidade superficial e aumentou a resistência à fadiga quando comparado ao grupo controle. Porém, o acabamento previamente ao polimento não afeta a rugosidade superficial e resistência à fadiga. Em relação ao estudo 3, observou-se que a aplicação de glaze, independentemente do método de aplicação, não promove efeitos deletérios na resistência à fadiga da cerâmica. No estudo 4 verificou-se que nenhum dos tratamentos de superfície realizados influenciaram negativamente na carga para falha em fadiga da cerâmica; contudo, o polimento e o glaze devem ser realizados após desgaste da cerâmica, por reduzirem a rugosidade superficial.

Palavras-chave: Fadiga. Resistência à flexão. Desgaste. Polimento. Acabamento. Staircase.

ABSTRACT

FATIGUE BEHAVIOR OF YSZ: EFFECTS OH DIFERENTS SURFACE TEATMENTS AND MATERIAL BASED ZICONIA

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This thesis are composed of four studies: study 1 – evaluation of fatigue failure load, number of cycles for failure and survival rate in disc of second and third-generation zirconia bonded in the analogue to dentin; study 2 – evaluation and comparison of effect of three polishing systems associated or not to finishing with fine and extra-fine diamond bur, on the surfaces characteristics (roughness and topography), phase transformation and fatigue behavior of a ground Y-TZP ceramic; study 3 – evaluation and comparison of effect of two techniques of glaze application (spray and brush) on the fatigue behavior and surface characteristics (roughness and topography) of Y-TZP ceramic, before and after grinding with diamond bur; study 4 – evaluation of effects of surface treatments (grinding, polishing and glaze) executed on the occlusal surface on its characteristics (roughness and topography), fatigue failure load, number of cycles for failure and phase transformation of an FSZ zirconia cemented adhesively into a similar dentin substrate. In study 1, four second-generation zirconia (Lava Plus, 3M ESPE, Vita In-Ceram YZ-HT, VITA Zahnfabrik, Zirlux FC, Ivoclar Vivadent, Katana ML-HT, Kuraray) and two third-generation zirconia (Katana UTML and Katana STML, Kuraray) were used. Ceramic discs were adhesively cemented onto epoxy resin discs. Fatigue test, fractographic and phase analyzes were performed. In study 2, Y-TZP (Zenostar T) discs were made with: Ø = 15 mm, 1.2 ± 0.2 mm thickness and divided into 8 groups: Ctrl- without treatment; Gr=grinding; Gr + Eve- grinding + polishing with Eve Diacera (two-step system); Gr + Fin + Eve- grinding + finishing + polishing with Eve Diacera; Gr + Kg- grinding + polishing with Kg Viking (two step system); Gr + Fin + Kg- grinding + finishing + polishing with K Viking; Gr + Op- polishing with Optrafine (three-step system); Gr + Fin + Op- grinding + finishing + polishing with Optrafine. In the sequence, topography, roughness, phase transformation analysis, fatigue test and fractography were performed; in study 3, the ceramic specimens were divided into 6 groups: Ctrl- without treatment; Gr- grinding with diamond bur; Br - application of glaze powder / liquid; Sp - application of glaze spray; Gr + Br - grinding + glaze powder / liquid; Gr + Sp - grinding + glaze spray. Surface roughness, fatigue test and fracture analysis were performed. In study 4- discs of IPS e.max Zircad MT Multi were produced and divided into 5 groups: Ctrl- without treatment; Gr- grinding with diamond bur; Gr + Pol- grinding + polishing with two step system; Gr + Gl- grinding + application of glaze; Gr + Pol + Gl- grinding + polishing + glaze application. Afterwards, the discs were cemented with resin cement into epoxy resin discs. Surface roughness analysis, fatigue test, fractography and xrd were performed. Regarding the study 1, it was observed that second-generation zirconia presents higher load for fatigue failure and number of cycles until failure than third-generation zirconia. In study 2, it was verified that the polishing after grinding reduced the surface roughness and increased the fatigue strength when compared to the control group. However, finishing previously to polishing does not affect surface roughness and fatigue strength. Regarding the study 3, it was observed that the application of glaze, regardless of the application method (brush or spray), does not promote deleterious effects on the fatigue strength of ceramics. In study 4, it was verified that none of the surface treatments performed influenced negatively the fatigue failure load of the ceramic. However, polishing and glaze should be performed after ceramic grinding, as they reduce surface roughness.

Key words: Fatigue. Flexural strength. Grinding. Glazing. Polishing. Finishing. Staircase method.

SUMÁRIO

1. INTRODUÇÃO GERAL

As cerâmicas à base de zircônia parcialmente estabilizadas por ítrio (YSZ – “*yttrium-stabilized zirconia*”) têm sido amplamente usadas na confecção de infraestruturas de próteses dentais, bem como na fabricação de coroas monolíticas (SHI *et al*., 2016; AMARAL et al., 2014) pois, apresentam as melhores propriedades mecânicas quando comparadas às demais cerâmicas odontológicas (DENRY & KELLY, 2014). A zircônia é um material que possui três fases cristalinas principais de acordo com a temperatura em que se encontra, as quais podemos citar: monoclínica (m), que se apresenta desde a temperatura ambiente até 1170°C; tetragonal (t), para temperatura acima de 1170ºC; e cúbica, quando a temperatura está acima de 2370°C (PICONI & MACCAURO, 1999). Entretanto, alguns óxidos estabilizadores podem ser adicionados ao material, tais como: CaO, MgO, CeO_2, sendo que na odontologia, o mais comumente usado é o óxido de ítrio (Y_2O_3) (ALGHAZZAWI *et al*., 2012). Ao longo dos anos, a zircônia sofreu algumas mudanças em sua microestrutura, sendo atualmente, classificada em três gerações de acordo com fase predominante e quantidade de óxido estabilizador (STAWARCZYK *et al*., 2017; ZHANG & LAW, 2018).

A primeira geração de zircônia também chamada de zircônia convencional (STAWARCZYK *et al*., 2017), apresenta em torno de 0.25% de alumina (AL_2O_3) e exibe uma resistência flexural superior a 1 GPa (ZHANG & LAW, 2018). Está indicada para a confecção de infraestruturas de prótese dental fixa na região posterior (ZHANG & LAW, 2018). Contudo, à longo prazo, as taxas de insucesso estão intimamente associadas ao lascamento e à delaminação da cerâmica de cobertura (ZHANG & LAW, 2018). Em relação às propriedades ópticas, apresenta alta opacidade devido as fases não cúbicas que resultam em dispersão da luz (ZHANG & LAW, 2018). Neste material a estrutura do cristal é muito pequena, assim, há um alto índice de refração de luz contribuindo para que a zircônia se torne opaca (STAWARCZYK *et al*., 2017).

Na tentativa de tornar a zirconia mais estética, surgiram as zircônias de segunda geração (PSZ – “*partially stabilized zirconia*”) (ZHANG & LAW, 2018), as quais continuam sendo parcialmente estabilizadas com 3% mol de ítria, mas apresentam uma incorporação de grãos de alumina de menor tamanho o que possibilita melhor característica óptica e de estabilidade cristalográfica (STAWARCZYK *et al*., 2017). Entretanto, salienta-se que tanto a zircônias de primeira, quanto as de segunda geração, podem sofrer transformação de fase (t-m), a qual pode ocorrer quando o material é submetido à estímulos mecânicos e/ou térmicos (AMARAL *et al*., 2016; BARTOLO *et al*., 2016; PEREIRA *et al*., 2016). Esta transformação de fase promove o desenvolvimento de uma camada de tensão compressiva, a qual dificulta a propagação de trincas para o interior do material, melhorando as suas propriedades mecânicas (DENRY & HOLLOWAY, 2006; GARVIE, HANNINK, PASCOE, 1975). Em relação à microestrutura, zircônias de segunda geração necessitam de uma temperatura de sinterização maior para reduzir suas porosidades, além de possuírem uma redução da quantidade de alumina (ZHANG & LAW, 2018). Estas alterações geram uma melhora na translucidez do material (TONG *et al*. 2016). Zircônias de segunda geração são indicadas para a confecção de restaurações monolíticas posteriores, porém, para o uso na região anterior, as características estéticas ainda não são suficientes para reproduzir as características dos dentes naturais (ZHANG & LAW, 2018).

Assim, surgiram as zircônias de terceira geração, chamadas também de zirconia totalmente estabilizada (FSZ – "*fully-stabilized zirconia"*), por apresentarem em sua composição 5% mol de ítrio (STAWARCZYK *et al*., 2017). Este aumento na concentração de óxido de ítrio estabiliza o material na fase cúbica (STAWARCZYK *et al*., 2017). Os grãos cúbicos por sua vez, são maiores em volume que os grãos tetragonais, assim, a luz passa a ser melhor transmitida (STAWARCZYK *et al*., 2017). Contudo, no que se refere às propriedades mecânicas, zircônias de terceira geração possuem menor resistência à fratura em relação às zircônias de segunda geração (SULAIMAN *et al*., 2017; PEREIRA *et al*., 2018), por não apresentarem mecanismo de tenacificação por transformação de fase (PEREIRA *et al*., 2018). Assim, os defeitos introduzidos pela usinagem durante a fabricação da restauração e pelos tratamentos de superfície realizados previamente à sua cimentação ficam livres para se propagarem, pois nesse caso, não há o efeito protetor do mecanismo de tenacificação (SULAIMAN *et al*., 2017). Estes materiais estão indicados na confecção de coroas anteriores (ZHANG & LAW, 2018).

No que tange aos tratamentos de superfície, frequentemente na prática clínica, torna-se necessário a realização de ajustes com pontas diamantadas da restauração cerâmica para se obter uma melhora da anatomia, perfil de emergência, contatos proximais/oclusais (İŞERI *et al*., 2012). Entretanto, este procedimento introduz defeitos no material que poderão atuar da seguinte forma: no caso de zircônia parcialmente estabilizadas (no caso de zircônias de primeira e segunda geração), quando estes defeitos forem mais profundos que a camada de tensão compressiva, pode ocorrer uma redução das propriedades mecânicas, pois estes defeitos atuarão como fator de concentração de tensão levando a fratura catastrófica da restauração (GREEN, 1983); porém, se os defeitos forem menores que a camada de tensão compressiva, há um efeito protetor, pois o mecanismo de tenacificação dificulta a propagação da trinca para o interior do material (CHEVALIER & GREMILLARD, 2009). Entretanto em zircônias totalmente estabilizadas o mecanismo de tenacificação é inexistente, pois estas não sofrem transformação de fase devido à alta concentração de fase cúbicas (INOKOSHI *et al*., 2018). Assim, os defeitos introduzidos no material ficam livres para se propagarem quando submetidos à estímulos mecânicos (SULAIMAN *et al*., 2017, ZHANG & LAWN 2018).

Ainda nesse contexto, o desgaste promove um aumento da rugosidade de superfície (PEREIRA *et al*., 2014; GUILARDI *et al*., 2017), de tal forma que causam maior desgaste do dente/restauração antagonista, maior acúmulo de placa, contribuindo para irritação gengival e cáries recorrentes, além de interferir negativamente nas propriedades estéticas e mecânicas da restauração (GÖNÜLOL *et al*., 2012). A fim de se reduzir a rugosidade após o desgaste, a literatura tem demonstrado que um protocolo de polimento é indispensável para a melhora das características superficiais e do comportamento mecânico do material (PREIS *et al*., 2015; ZUCUNI *et al*., 2017). Contudo, ainda não há achados na literatura acerca do efeito do desgaste e protocolo de polimento da superfície oclusal de restaurações a base de zircônia no comportamento mecânico em fadiga destas restaurações.

Outro tratamento também indicado para promover maior lisura superficial é a aplicação de um material vítrocerâmico amorfo, conhecido como "*glaze*". Segundo Anusavice (2003), o glaze tem um impacto positivo no comportamento mecânico de cerâmicas vítreas, pois ele penetra nas irregularidades superficiais unindo os defeitos, aumentando assim, a resistência da cerâmica. Porém, os dados presentes na literatura, apresentam-se controversos em relação a este tratamento quando realizado em cerâmicas à base de zircônia, pois há estudos que demonstraram uma degradação das propriedades mecânicas do material quando uma fina camada de glaze é aplicada na superfície da cerâmica. (YENER *et al*., 2011; ZUCUNI *et al*., 2017). A zircônia por ser um material policristalino apresenta uma certa incompatibilidade com o glaze (material vítreo), tanto no que se refere ao coeficiente de expansão térmica quanto na adesão entre estes dois materiais (zircônia e glaze) (ABOUSHELIB *et al*., 2008; TAN *et al*., 2012; DENRY& KELLY 2014). Outro possível fator que pode interferir na divergência destes dados é a forma de aplicação do glaze, ou seja, durante aplicação do glaze na conformação pó/líquido pode ocorrer uma maior de formação de bolhas, as quais poderão atuar como fatores de concentração de tensão levando a fratura catastrófica do material.

No ambiente oral, as restaurações cerâmicas estão susceptíveis à falha em fadiga, a qual é definida como fratura do material devido ao crescimento progressivo de trincas quando submetidas à tensões cíclicas de baixa intensidade provenientes do ciclo mastigatório (WISKOTT *et al*., 1995; ZHANG *et al*., 2013). A falha por fadiga pode ocorrer de forma mais acelerada na presença de umidade, pois a água penetra nas fissuras/trincas do material quebrando as ligações coesivas, a qual resulta no crescimento “subcrítico” ou “lento” que progride ao longo do tempo, levando à falha da restauração (ZHANG *et al.*, 2013). Dessa forma, testes de fadiga reproduzem com maior precisão e previsibilidade o comportamento mecânico do material do que testes estáticos (como por exemplo, testes de resistência flexural).

Com base nos conhecimentos acima expostos, a presente tese está composta por quatro artigos, os quais apresentam como objetivos:

- Estudo 1: “*Load-bearing capacity under fatigue and survival rates of adhesively cemented yttrium-stabilized zirconia polycrystal monolithic simplified restorations”.* Onde buscou-se avaliar a carga para falha em fadiga, número de ciclos para falha e taxa de sobrevivência em discos de zircônia de segunda e terceira geração cimentados adesivamente em substrato análogo à dentina;

- Estudo 2: “*Influence of finishing/polishing on the fatigue strength, surface topography, and roughness of an yttrium-stabilized tetragonal zirconia polycrystals subjected to grinding”.* Cujo objetivo foi avaliar e comparar o efeito de três sistemas de polimento associados ou não ao acabamento prévio com pontas finas e extra-finas, nas características superficiais (rugosidade e topografia), transformação de fase e comportamento à fadiga de uma cerâmica Y-TZP desgastada por ponta diamantada grossa;

- Estudo 3: “*Low-fusing porcelain glaze application does not damage the fatigue strength of Y-TZP”*. Que objetivou avaliar e comparar o efeito de duas formas de aplicação de glaze (pincel e spray) no comportamento à fadiga e características superficiais (topografia e rugosidade) de uma cerâmica Y-TZP antes e após o desgaste com ponta diamantada;

- Estudo 4: “*Surface treatments executed on the occlusal surface and heat-treatment: effect on the fatigue performance of bonded 4Y-PSZ monolithic restorations”.* Onde avaliou-se o efeito de tratamentos de superfície (desgaste, polimento e glaze) nas características superficiais (topografia e rugosidade), carga para falha em fadiga, número de ciclos para falha e transformação de fase de uma zirconia FSZ cimentada adesivamente em substrato análogo de dentina.

Para efeitos de apresentação, esta Tese está apresentada sob a forma de quatro artigos:

ARTIGO 1
Publicado em "*Journal of the Mechanical Behavior of Biomedical Materials*".
Fator de Impacto: 3,239.
DOI: https://doi.org/10.1016/j.jmbbm.2018.11.009.

Zucuni CP, Venturini AB, Prochnow C, Pereira GKR, Valandro LF. Load-bearing capacity under fatigue and survival rates of adhesively cemented yttrium-stabilized zirconia polycrystal monolithic simplified restorations. J Mech Behav Biomed Mater. 2019 Feb; 90:673-680. Epub 2018 Nov 12.

ARTIGO 2
Publicado em "*Journal of the Mechanical Behavior of Biomedical Materials*".
Fator de Impacto: 3,239.
DOI: https://doi.org/10.1016/j.jmbbm.2019.02.013.

Zucuni CP, Dapieve KS, Rippe MP, Pereira GKR, Bottino MC, Valandro LF. Influence of finishing/polishing on the fatigue strength, surface topography, and roughness of an yttrium-stabilized tetragonal zirconia polycrystals subjected to grinding. J Mech Behav Biomed Mater. 2019 May; 93:222-229. Epub 2019 Feb 13.

ARTIGO 3
Publicado em "*Journal of the Mechanical Behavior of Biomedical Materials*".
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DOI: https://doi.org/10.1016/j.jmbbm.2019.07.022.

Zucuni CP, Pereira GKR, Dapieve KS, Rippe MP, Bottino MC, Valandro LF. Low-fusing porcelain glaze application does not damage the fatigue strength of Y-TZP. J Mech Behav Biomed Mater. 2019 Nov;99:198-205. Epub 2019 Jul 20.

ARTIGO 4
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DOI: https://doi.org/10.1016/j.jmbbm.2019.103528.

Zucuni CP, Pereira GKR, Valandro LF. Grinding, polishing and glazing of the occlusal surface do not affect the load-bearing capacity under fatigue and survival rates of bonded monolithic fully-stabilized zirconia simplified restorations. Available online 11 November 2019, 103528, In Press, Journal Pre-proof.

2. ARTIGO 1 - Load-bearing capacity under fatigue and survival rates of adhesively cemented yttrium-stabilized zirconia polycrystal monolithic simplified restorations

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Running Title: Fatigue behavior of monolithic zirconia restorations

Abstract

This study aims to evaluate the fatigue failure load, number of cycles for failure and survival probability of 2nd and 3rd generation yttrium-stabilized zirconia (YSZ) adhesively cemented to a dentin analogue substrate. Disc-shaped specimens (n = 10; Ø = 10 mm; thickness = 1.0 mm) were produced from four 2nd generation YSZs (Lava Plus, 3M ESPE; Vita In-Ceram YZ-HT, VITA Zahnfabrik; Zirlux FC, IvoclarVivadent; Katana ML-HT, Kuraray) and two 3rd generation YSZs (Katana UTML and Katana STML, Kuraray). Each YSZ disc was adhesively cemented (Multilink Automix System) onto its dentin analogue pair (epoxy resin, Ø = 10 mm; thickness = 2.5 mm). Fatigue tests were conducted through step-stress approach (load ranging from 400 to 2600 N; step-size of 200N; 20,000 cycles per step, 20 Hz) and the obtained data were analyzed using Kaplan Meier and Mantel-Cox tests. Surface topography and phase transformation (*m*-, *t*-, and *c*-phases) inspections after particle air-abrasion of the YSZs were performed, as well as fractographic analysis of the failed specimens. Second-generation zirconia materials presented higher fatigue failure load, number of cycles for failure, and survival probability than 3rd generation. Similar topographical characteristics of the YSZs could be noted. Phase transformation (t- to m-phase) after YSZ air-abrasion was only observed for 2nd generation materials. All failures started from the surface/sub-surface defects located at the cementation interface. 2nd generation zirconia presented higher load-bearing capacity in cyclic loading than 3rd generation materials.

Keywords: Survival analysis. Fatigue testing. Full-contour restorations. All-ceramic. Polycrystalline zirconia. Fractographic analysis.

1. Introduction

Zirconia ceramics have been widely used in restorative dentistry due to their great flexural strength and fracture toughness when compared to glass-ceramics (Denry et al., 2014). Zirconia is a polymorphic metastable material that exists under the form of three crystalline structures: monoclinic (room temperature up to 1170 °C), tetragonal (1170^o up to 2370 °C) and cubic (above to 2370 °C) (Piconi and Maccauro, 1999). The tetragonal and cubic phases may be stabilized in room temperature by the addition of oxides such as CaO, MgO, CeO2, and Y2O3 in specific concentrations (Harada et al., 2016; Chevalier et al., 2007).

Recently, the possibility of manufacturing monolithic restorations made from yttrium-stabilized zirconia polycrystal materials (YSZs) has been considered (Stawarczyk et al., 2017^a). Many YSZ ceramics were developed, and today three generations may be encountered:

1) 1st generation zirconia (conventional zirconia-3Y-TZPs) contains 0.25 wt% alumina (Al2O3) and presents flexural strength above to 1 GPa (Zhang and Lawn, 2018). The structure of this material presents mainly very small crystals of zirconium oxide with alumina dispersed into its structure with a coarse grain size, which produced a high light refraction index, exhibiting high opacity (Stawarczyk et al., 2017^a; Zhang and Lawn, 2018). 1st generation zirconia is indicated for framework in porcelain-veneered crowns. However, in long-term, these crowns have presented some clinical failure rates, as chipping and delamination (Zhang and Lawn, 2018);

2) the 2nd generation ones firstly appeared as an available option for monolithic restoration (Stawarczyk et al., 2017^a). Second generation YSZs presents a decreased number and grain size of aluminum oxide (Al_2O_3) particles that were positioned around the zirconium oxide crystals, which allowed an improvement of the refraction index, increasing the translucency in comparison to the 1st generation and maintaining high strength and long term stability (Stawarczyk et al., 2017^a). Both 1st and 2nd generation materials are known as partially stabilized zirconia (PSZ) (Stawarczyk et al., 2017^a). As their main feature, PSZs have a metastable mechanism, which enables responding to stimuli via phase transformation toughening, making crack propagation less probable (Chevalier et al., 2009). Even though PSZs present excellent mechanical behavior based on the toughening mechanism, the need to improve their optical properties was still present (Shenoy and Shenoy, 2010; Stawarczyk et al., 2017^b);

3) several efforts have been performed to enhance the optical properties of YSZ materials, especially for translucency improvements (Sulaiman et al., 2015). From this

standpoint, 3rd generation zirconia (fully-stabilized zirconia – FSZ, by using 5%mol yttrium) (Stawarczyk et al., 2017[a]) were developed for the similar clinical indications to the PSZ material. The increased percentage of stabilizing/dopant oxides into FSZ materials leads to the presence of cubic phase crystals on their microstructure (Stawarczyk et al., 2017[a]). The cubic crystals show larger volume than tetragonal grains and are also more isotropic, thus light is better transmitted throughout the restorations (Stawarczyk et al., 2017[a]).

Studies have shown that FSZs present lower physical and mechanical properties than those of PSZs (Sulaiman et al., 2017; Pereira et al., 2018). Sulaiman et al. (2017) showed a flexural strength reduction up to 40% of FSZ compared to the PSZ. Pereira et al. (2018) corroborated this assumption, finding a decrease in fatigue strength of up to 60% for 3rd generation zirconia. Both studies have examined simplified testing assemblies (bars or discs) which present the advantage of evaluating the isolated influence of the material's microstructure on its mechanical performance, but did not consider any factors involved in a clinical scenario, such as surface conditioning of the intaglio surface and zirconia cementation set-up.

As aforementioned, zirconia is a crystalline material (Denry et al., 2014), being classified as acid resistant (i.e. its surface is not topographically changed by hydrofluoric acid etching) (Valandro et al., 2005), therefore it demands surface treatments such as particle air-abrasion for bond improvements (Denry et al., 2014). Adhesively-cemented restorations usually show higher fatigue strength and survival rates (Anami et al., 2016; Campos et al., 2017; Monteiro et al., 2018), because the resin cement fills the irregularities and defects of the bonding surface, somewhat healing the cracks and improving the mechanical behavior (Addison et al., 2007).

The surface characteristics of intaglio of ceramic crowns play a key role in initiating failure in clinical scenarios (Pagniano et al., 2005; Prochnow et al., 2018; Venturini et al., 2018). Some studies show that fractures usually initiate from the interfacial zones between resin cement and the ceramic intaglio surface, where the highest tensile stresses are concentrated (Kelly, 1999; Kelly et al., 2010; Campos et al., 2017). Also, considering a cementation circumstance, particle air-abrasion as zirconia surface treatment is commonly applied for increasing the intaglio surface roughness and to enhance the adhesion (Sulaiman et al., 2017). However, as an inherent consequence, it means that the number of surface defects (flaw population) on the intaglio surface is also increased.

As already known, the air-abrasion of 2nd generation zirconia generates phase transformation (t-m), triggering the toughening mechanism, and increasing its flexural strength (Aurélio et al., 2016; Moon et al., 2016). On the other hand, the air-abrasion might impair the mechanical fatigue behavior of 3rd generation zirconia, since this kind of material is fully stabilized and unable to present phase transformation to trigger the toughening mechanisms. Regarding this situation, one question has yet not been answered in the scientific literature: Do fully and partially stabilized zirconia polycrystal materials subjected to air-abrasion and bonded on a dentin analogue substrate behave similarly when tested under intermittent cyclic loading?

From the aforementioned viewpoints, this study evaluated the fatigue failure load, number of cycles for failure and survival probability of 2nd and 3rd generation zirconia polycrystal restorations adhesively cemented on a dentin analogue substrate. The hypothesis assumed was that 2nd generation zirconia would have better fatigue behavior than 3rd generation.

2. Material and Methods

Table 1 lists the materials used in this study with their respective composition, sintering cycle, and clinical indication alleged by the manufacturers.

2.1 Specimen preparation

Zirconia discs (Ø= 98 mm) were manually sliced into square blocks of 16×16mm, where the thickness ranged from 14 up to 20 mm depending on the initial thickness of the zirconia disc. Metal rings (Ø= 12 mm) were glued at each side of the obtained blocks to serve as reference for grinding (with 600- to 1200-grit silica carbide papers) on a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA), obtaining cylinders measuring 12 mm in diameter as well-described by Pereira et al. (2014).

Next, slices (thickness of 1.4 mm) were obtained in a precision cutting machine (Isomet 1000, Buehler) under water-cooling. All ceramic discs were polished (up to 2000-grit SiC papers) until 1.2 mm thickness on both sides to remove any irregularity introduced by cutting, then sintered as recommended for each material (Zyrcomat T, Vita Zahnfabrik, Bad Säckingen, Germany; Table 1). After sintering, all zirconia discs (n= 10) presented 1.0 mm thickness and 10 mm diameter.

Epoxy resin plates (Epoxy Plate 150Plate 150 × 350 × 2.5 mm, Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany) were shaped into cylinders using a diamond drill (Diamant Boart, Brussels, Belgium) under refrigeration, resulting in epoxy discs with 10 mm diameter.

Zirconia pairs and epoxy discs were randomly allocated and a simplified tri-layer setup (Monteiro et al., 2018) was used, simulating a molar restoration with a final thickness of 3.5

mm after cementation, where the ceramic disc represented the occlusal part of the simplified restoration and the epoxy resin disc simulated dentin. Those dimensions (3.5 mm in thickness, 10 mm in diameter) were respectively chosen based on the average thickness from the pulp wall to the occlusal surface described by Sulieman et al. (2005), and the average surface area of a first posterior molar postulated by Ferrario et al. (1999).

2.2 Adhesive cementation

Prior to the cementation procedure, the zirconia and epoxy resin specimens were cleaned in an ultrasonic bath (1440 D – Odontobras, Ribeirao Preto, Brazil) with 78% isopropyl alcohol for 5 min.

The intaglio surface of zirconia discs was air-abraded with 45 µm aluminum oxide powder (Polidental, Cotia, Sao Paulo, Brazil) for 10 s at 15 mm of distance under 2 bar pressure (Aurélio et al., 2016), followed by a primer application containing multiple bond promoters (Monobond Plus, Ivoclar-Vivadent, Schaan, Liechtenstein), which was actively applied for 15s, kept to react for 45s, and gentle air-dried.

The bonding surface of the respective counterpart epoxy resin disc was etched by 10% hydrofluoric acid (CondacPorcelana, FGM, Joinville, Brazil) for 60 s, followed by washing for 30s, ultrasonic cleaning in distilled water for 5 min, and air-drying. Then, Multilink Primers A and B (Ivoclar-Vivadent) were mixed in a 1:1 ratio, scrubbed on the treated surfaces (30 s), and gentle air-dried to obtain an even thin layer.

Afterward, each zirconia disc was adhesively cemented (Multilink Automix, IvoclarVivadent) to its counterpart epoxy disc. When positioned to each other, the assembly was seated under a constant load of 2.5 N, followed by removal of the cement excesses and light-activation (Radii-cal, SDI, Bayswater, Australia) for five exposures of 20 s each (0°, 90°, 180°, 270°, and top from the occlusal part – i.e. zirconia). All the specimens were stored in distilled water (37 °C) for around 7 days before conducting the step-stress fatigue tests.

2.3 Step-stress fatigue tests

The cemented assemblies (n=10) were tested using the step-stress test method (Magne et al., 2010; Dapieve et al., 2018) in an electric machine (Instron Electro Puls E3000, Instron Corp, Norwood, United States). Incremental cyclic loads ranging from 10 N up to the maximum desired load in each step were applied with a 40-mm diameter stainless-steel hemispheric piston (Kelly et al., 2010) under distilled water at a frequency of 20 Hz. An adhesive tape was placed on the occlusal surface and a polyethylene sheet (0.1 mm thick) was placed between the piston and the ceramic surface to reduce contact stress concentration (ISO 6872:2015) (Figure 1).

An initial maximum load of 200 N was applied for 5,000 cycles to adjust piston/specimen relations; after that, a step-size of 200 N and a lifetime of 20,000 cycles at each load step were used. Therefore, the maximum load ranged from 400 N up to 2600 N until the run-out (survival) of the specimen or failure in a distinct step. The specimens were checked for cracks at the end of each step by light oblique transillumination (Dibner & Kelly, 2016).

2.4 Fractographic analysis

After the cyclic loading tests, all failed specimens were analyzed under a stereomicroscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany) for contact damage and by light oblique transillumination to identify the crack direction. Then, those specimens were longitudinally sectioned into two halves, perpendicularly to the radial crack direction with a diamond blade under water-cooling (Isomet 1000, Buehler). Representative specimens from each group were ultrasonically cleaned in distilled water (5 min), gold-sputtered, and analyzed under scanning electron microscopy (SE+BSE detector, 20Kv; SEM - Vega3, Tescan, Czech Republic).

2.5 X-ray Diffraction (XRD analysis)

To identify the superficial crystalline phase content (*m*-, *t*-, and *c*-phases) at the intaglio ceramic surface after air-abrasion, two additional specimens of each zirconia material were analyzed on an X-ray Diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) with CuKα radiation (40kV, 40mA) in a 2θ angular interval of 20 to 90°, with a step-size of 0.01° every 3 s following a previously-described methodology (Inokoshi et al., 2015; Pereira et al., 2018).

2.6 Surface topography

To depict surface morphology, topography, and the defects introduced by air-abrasion, two additional specimens of each zirconia material were cleaned in an ultrasonic bath (1440 D–Odontobras) with 78% isopropyl alcohol for 5 min, gold-sputtered and then analyzed by Scanning Electron Microscopy (SEM - Vega3, Tescan) under 1000× and 5000× magnification.

2.7 Statistical analysis

The obtained fatigue failure load (in N) and the number of cycles for failure were subjected to the Kaplan Meier and Mantel-Cox survival tests (α= 0.05) (SPSS 21.0, IBM, Chicago, USA).

3. Results

The 2nd generation zirconia ceramics presented statistically higher fatigue failure load, number of cycles for failure and survival probabilities than 3rd generation zirconia ceramics (Table 2; Fig. 2). The 2nd generation zirconia materials obtained high load-bearing capacities and presented slightly different fatigue performance among each other statistically (Table 2; Fig. 2). Table 3, Table 4 and Fig. 2 depict that 2nd generation materials obtained higher survival rates, not only for load parameters, but also for number of cycles.

SEM analysis showed similar topographical characteristics after air-abrasion for all tested YSZs, regardless of the zirconia generation (Fig. 3). Thus, it is possible to observe that air-abrasion generated rough surfaces with regular and homogeneous defects, completely different from a typical micrograph of a non-treated zirconia, where crystals aggregations are easily observed.

XRD data depicted that 2nd generation materials had phase transformation (t-m, monoclinic peaks -111 and 111) after air-abrasion, while no m-phase could be detected in 3rd generation zirconia, presenting cubic phase (peak 400), as well as the conventional tetragonal phase (Fig. 4).

Finally, fractographic micrographs (Fig. 5) depicted that failures always started from defects of the adhesive zone (cementation zone).

4. Discussion

Our findings support that the 2nd generation zirconia polycrystal ceramics present higher fatigue performance than 3rd generation ones, thus the assumed hypothesis was accepted.

PSZ (2ndgeneration zirconia) ceramics are metastable (Piconi & Maccauro 1999) due to the tetragonal-monoclinic phase transformation. This phase transformation takes place as response to stimuli: thermal (e.g. cooling after sintering, thermal variations, low-temperature degradation) (Shahmiri et al., 2018) or mechanical (e.g. grinding, air-abrasion, polishing) (Lucas et al., 2015). The phase transformation (t-m) triggers the toughening mechanism (Garvie and Nicholson et al., 1972), which acts in hindering defects and crack propagation through the material (Denry & Kelly, 2014).

The 3rd generation zirconia materials are fully stabilized and present high cubic phase content (up to 15% according to the manufacturers) (Inokoshi et al., 2018). Thus, phase transformation (t-m) does not take place for this ceramic (Inokoshi et al., 2018), and consequently no toughening mechanism occurs. For this reason, this kind of material is more likely to present cracking and its propagation when stimuli are applied (Sulaiman et al., 2017, Zhang & Lawn 2018). Recently, Inokoshi et al. (2018) observed a decrease in strength and fracture toughness for this class of material. Our data corroborate those findings, since the fatigue failure load, number of cycles for failure and survival probabilities were lower for 3rd generation zirconia than 2nd generation ones (Table 2, Figure 4).

Another explanation for distinct mechanical behaviors is based on the differences in the material's microstructure. The 3rd generation zirconia presents higher grain size and yttria content in its composition, and it shows more intergrain spaces when compared to 2nd generation as well (Sulaiman et al., 2017). In addition, cubic crystals have higher volume and are less strongly bonded, decreasing the threshold strength (Sulaiman et al., 2017). On the other hand, the 2nd generation zirconia presents small grains allied to a toughening mechanism triggered by phase transformation, despite the low esthetic properties (Stawarczyk et al., 2017[a]; Lucas et al., 2015). Hence, the literature supports that the yield stress and microhardness of nanocrystalline materials can be 2-10 times higher than the corresponding larger-grained materials with the same chemical composition (Palmero, 2015; Pande and Cooper, 2009). Therefore, higher mechanical performance would be expected for 2nd generation zirconia than 3rd generation.

Taking into consideration a clinical scenario, alumina particle air-abrasion as pre-treatment has been applied for topographical alterations (increase in roughness) of zirconia surface, enabling mechanical interlocking of the luting agent and bond improvement (Sulaiman et al., 2017). Figure 3 depicts topographic changes and defects generated by air abrasion in the different zirconia materials we tested. Meanwhile, these introduced flaws might also be potentially damaging to the mechanical behavior of these materials (Coti et al., 2017). The stress generated by air abrasion can modify the structural stability, increasing the susceptibility to degradation and promoting cracks and damage of zirconia materials (Kosmac et al., 1999; Aurelio et al., 2016).

A systematic review by Aurélio et al. (2016) stated that the airborne-particle abrasion using gentle protocols (2.8 bar for 15 s) applied onto a 2nd generation zirconia promotes phase (t-m) transformation, increasing the flexural strength. However, the application of air-abrasion particles on 3rd generation zirconia is somewhat debatable, since this kind of zirconia has no toughening mechanism by phase transformation. In fact, Sulaiman et al. (2017) presented a decrease of up to 60% in flexural strength after air-abrasion for 3rd generation zirconia (FSZ).

In terms of cyclic loading in clinical service, it should consider that the maximum chewing force can reach 800 N for 7 seconds during sleeping and bruxism (Nishigawa et al., 2001). From the chewing force standpoint, 3rd generation zirconia (Katana UTML and STML) would only have a 30% probability to exceed 800 N without occurring failure, while all the 2nd generation zirconia materials would present 100% probability to exceed this load (Table 3). Thus, it is logical to assume that the 3rd generation material is more likely to present cracks and fractures in such a scenario than a 2nd generation one. Corroborating the results of the present study, a recent study using a Hertzian indentation flexural radial fracture test showed that lithium disilicate exhibits similar load bearing properties to 4Y-PSZ (containing 4%mol yttrium) and better than 5Y-PSZ (containing 5%mol yttrium) when bonded to and supported by a dentin-like substrate, meanwhile 3Y-TZP (2nd generation) still holds the highest load-bearing capacity (Yan et al., 2018).

As a contradictory issue, it is important to emphasize that 2nd generation materials might be more prone to aging than 3rd generation ones (Pereira et al., 2018). Second generation zirconia suffers phase transformations during aging that may lead to mechanical impairment (Chevalier et al., 2007), which is known as low-temperature degradation. However, until now there is no data supporting that the clinical failures of zirconia crowns occur as a consequence of zirconia degradation.

Regarding limitations, we can point out the absence of long-term and thermal cycling aging for bond degradation (bond hydrolysis), the use of a simplified model to mimic posterior restorations (disc-disc set-up), and the absence of sliding movement during cyclic loading application.

5. Conclusion

Based on our findings, it is concluded that the tested 2nd generation zirconia materials present better fatigue behavior than the 3rd generation zirconia materials due to their inherent structural and phase composition differences.

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TABLES

Table 1. Description of materials, their composition, sintering cycle and clinical indications alleged by the manufacturers guidelines.

Yttrium-stabilized zirconia polycrystals	Commercial name (Manufacturer)	Composition	Sintering cycle	Alleged clinical indications
2nd generation zirconia (partially-stabilized zirconia)	Lava Plus (3M ESPE)	ZrO_2, Y_2O_3, Al_2O_3	2h at 1450°C	Full-contour crowns, full-contour bridges, inlays, onlays and layered ceramics
	In-Ceram YZ-HT (VITAZahnfabrik)	ZrO_2, Y_2O_3, HfO_2, Al_2O_3, Er_2O_3, Fe_2O_3	2h at 1450°C	Frameworks, bridges and fully-anatomical anterior and posterior restorations
	Zirlux FC (IvoclarVivadent)	ZrO_2, Y_2O_3, Al_2O_3, HfO_2	2h at 1500°C	Copings, multi-unit frameworks, multi-unit bridges, inlays/onlays, full-contour crowns, full-contour bridges, implant abutment, full arch/hybrid bridges
	Katana ML (Noritake/Kuraray)	ZrO_2, Y_2O_3	2h at 1550°C	Frameworks, FCZ crowns, FCZ bridges, inlays, onlays and veneers
3rd generation zirconia (fully-stabilized zirconia)	Katana UTML (Noritake/Kuraray)	ZrO_2, Y_2O_3	2h at 1550°C	Frameworks, FCZ crowns, FCZ bridges, inlays, onlays and veneers
	Katana STML (Noritake/Kuraray)	ZrO_2, Y_2O_3	2h at 1550°C	Frameworks, FCZ crowns, FCZ bridges, inlays, onlays and veneers

Table 2. Step-stress fatigue tests results: mean fatigue failure load and mean cycles until failure with respective 95% confidence interval for each tested material.

Yttrium-stabilized zirconia polycrystals		**Fatigue failure load (N)**		**Cycles until failure**	
		Mean	**CI (95%)**	**Mean**	**CI (95%)**
2ndgeneration zirconia	Lava Plus	2540^{ab}	2422.4−2657.6	239.000^{ab}	227.240−250.760
	In-Ceram YZ-HT	2200^{b}	1959.1−2440.9	205.000^{b}	180.906−229.093
	Zirlux FC	2580^{a}	2540.0−2619.2	243.000^{a}	239.080−246.920
	Katana ML	2380^{ab}	2200.4−2559.6	223.000^{ab}	205.036–240.964
3rdgeneration zirconia	Katana UTML	920^{c}	699.8−1140.2	77.000^{c}	54.979–99.020
	Katana STML	860^{c}	800.1−919.9	71.000^{c}	65.012−76.988

*Different letters indicate statistically significant difference among evaluated conditions at the same column.

Table 3. Survival rates (specimens' probability to exceed the respective fatigue failure load without fracture, and the respective standard error measurements) for the different zirconia polycrystal ceramics.

Yttrium-stabilized zirconia polycrystals		**Survival rates on the respective load steps**											
		400	**600**	**800**	**1000**	**1200**	**1400**	**1600**	**1800**	**2000**	**2200**	**2400**	**2600**
2nd generation zirconia	Lava Plus	1	1	1	1	1	1	1	1	0.9 (0.1)	0.9 (0.1)	0.9 (0.1)	0.9 (0.1)
	In-CeramYZ-HT	1	1	1	1	1	0.9 (0.1)	0.9 (0.1)	0.9 (0.1)	0.5 (0.2)	0.5 (0.2)	0.3 (0.2)	0.1 (0.1)
	Zirlux FC	1	1	1	1	1	1	1	1	1	1	0.9 (0.1)	0.6 (0.2)
	Katana ML	1	1	1	1	1	1	1	0.9 (0.1)	0.8 (0.1)	0.7 (0.2)	0.5 (0.2)	0.3 (0.2)
3rd generation zirconia	Katana UTML	1	0.8 (0.1)	0.3 (0.2)	0.2 (0.1)	0.1 (0.1)	0.1 (0.1)	0.1 (0.1)	0.0	-	-	-	-
	Katana STML	1	1	0.3 (0.2)	0.0	-	-	-	-	-	-	-	-

*the sign '-' indicates absence of specimen being tested on the respective load step.

Table 4. Survival rates (specimens' probability to exceed the respective number of cycles without failure, and the respective standard error measurements) for the different zirconia polycrystalline ceramics.

Yttrium-stabilized zirconia polycrystals		**Survival rates on the respective number of cycles steps**											
		20,000	**40,000**	**60,000**	**80,000**	**100,000**	**120,000**	**140,000**	**160,000**	**180,000**	**200,000**	**220,000**	**240,000**
2nd generation zirconia	Lava Plus	1	1	1	1	1	1	1	1	0.9 (0.1)	0.9 (0.1)	0.9 (0.1)	0.9 (0.1)
	In-Ceram YZ-HT	1	1	1	1	1	0.9 (0.1)	0.9 (0.1)	0.9 (0.1)	0.5 (0.2)	0.5 (0.2)	0.3 (0.2)	0.1 (0.1)
	Zirlux FC	1	1	1	1	1	1	1	1	1	1	0.9 (0.1)	0.6 (0.2)
	Katana ML	1	1	1	1	1	1	1	0.9 (0.1)	0.8 (0.1)	0.7 (0.2)	0.5 (0.2)	0.3 (0.2)
3rd generation zirconia	Katana UTML	1	0.8 (0.1)	0.3 (0.2)	0.2 (0.1)	0.1 (0.1)	0.1 (0.1)	0.1 (0.1)	0.0	-	-	-	-
	Katana STML	1	1	0.3 (0.2)	0.0	-	-	-	-	-	-	-	-

*the sign '-' indicates absence of specimen being tested on the respective step.

FIGURES

Figure 1. **S**chematic figure of the specimen set and the load application during fatigue testing.

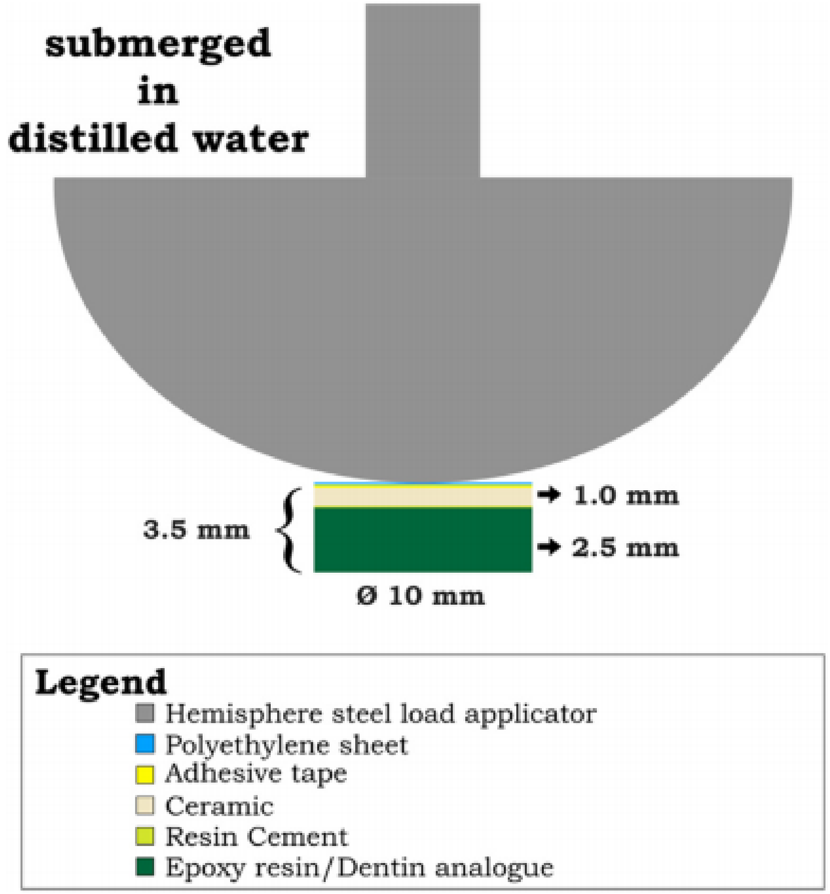


Figure 2. Illustrative graphs depicting the survival probability in function of the load steps (A) and number of cycles steps (B).

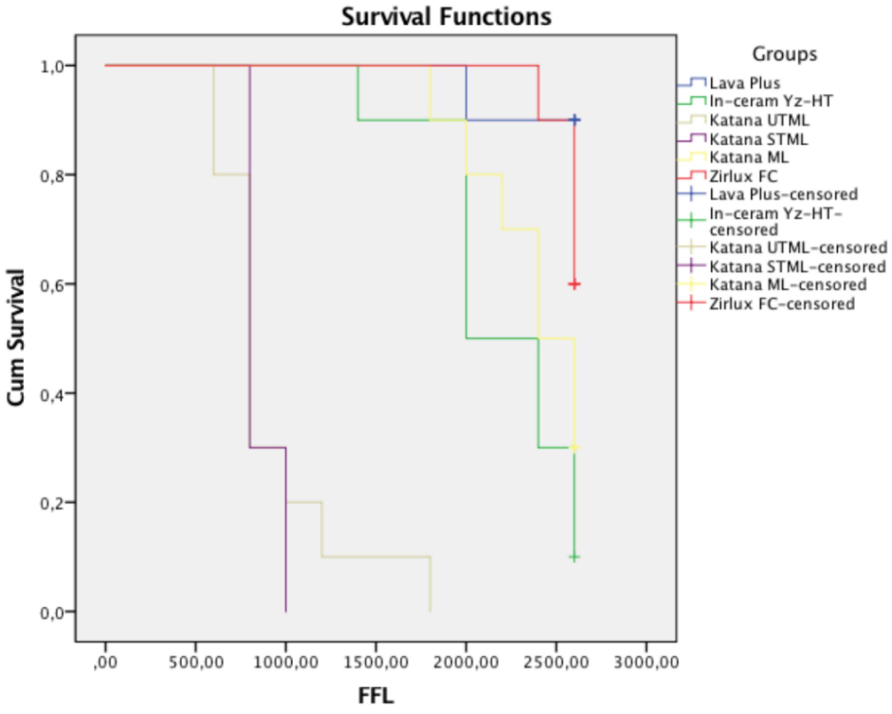
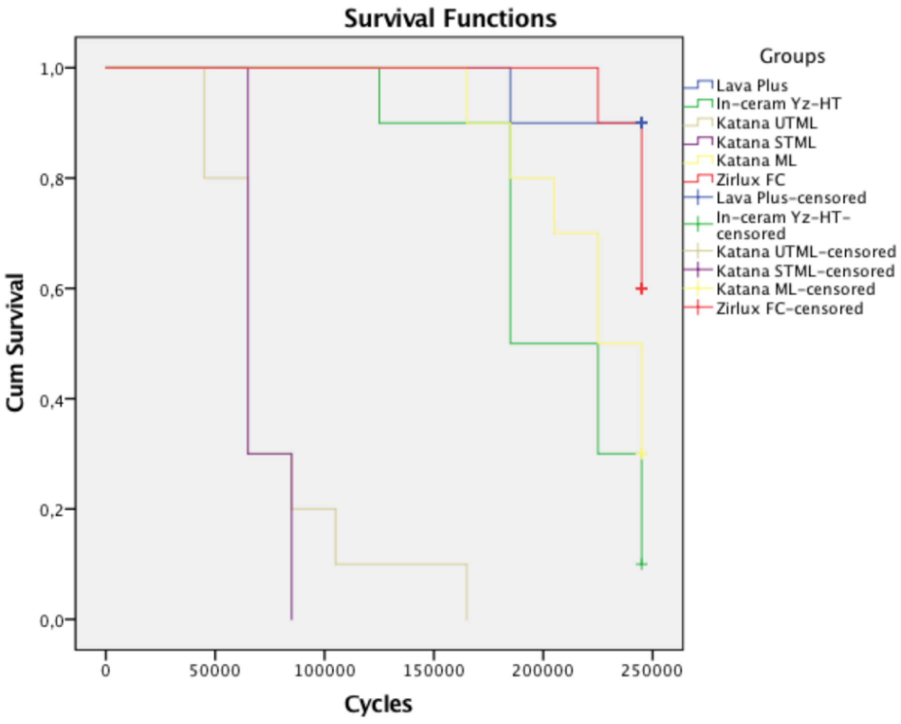
Survival curves considering the load parameters	**Survival curves considering number of cycles**
	

Figure 3. Representative SEM images at 5000× magnification: the bigger image represents the air abraded zirconia surface while the image located in the upper right corner shows the occlusal surface without treatment.

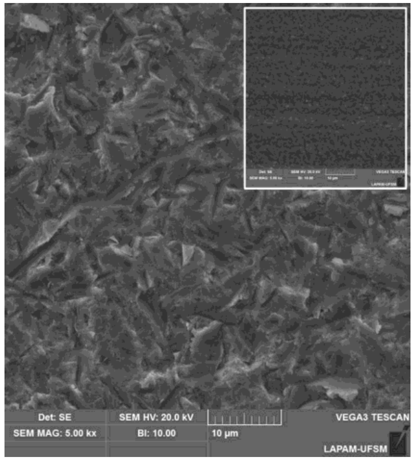
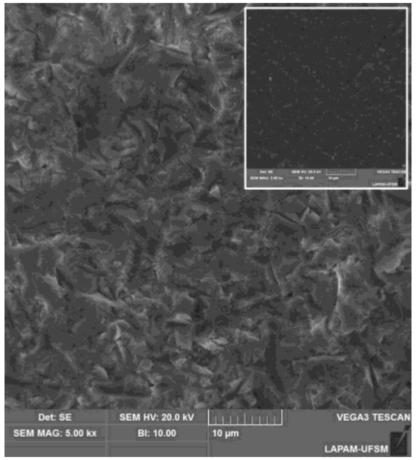
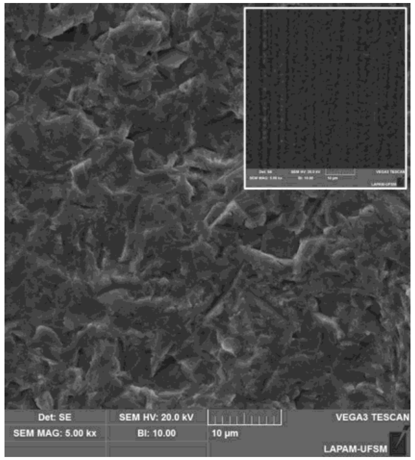
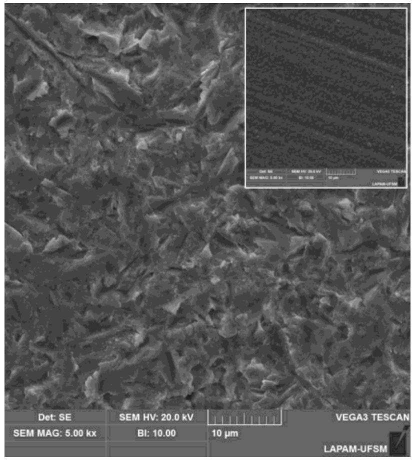
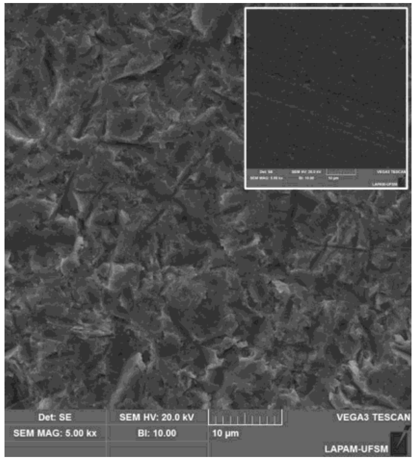
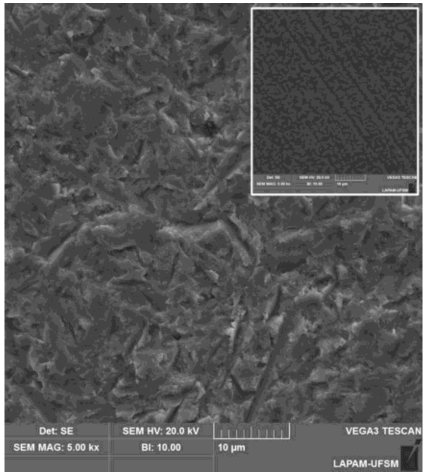
2nd generation zirconia		**3rd generation zirconia**
		
Lava Plus	**In-Ceram YZ-HT**	**Katana UTML**
		
Katana ML	**Zirlux FC**	**Katana STML**

Figure 4. XRD graphs of the materials tested showing the peaks of each crystallographic phase.

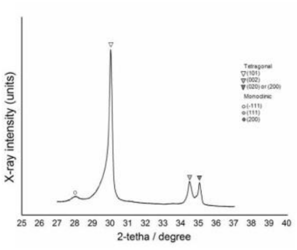
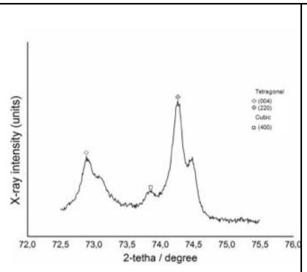
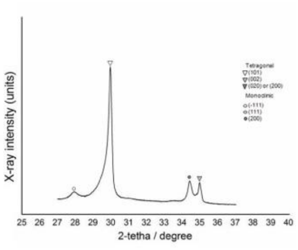
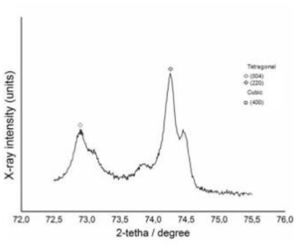
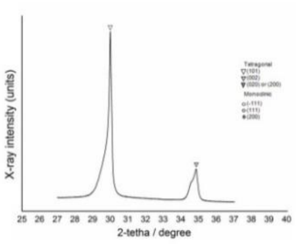
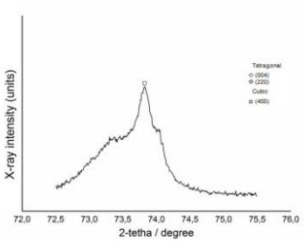
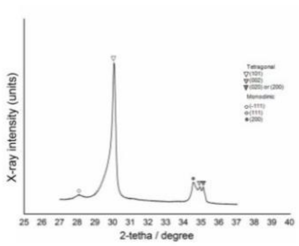
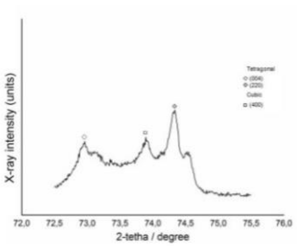
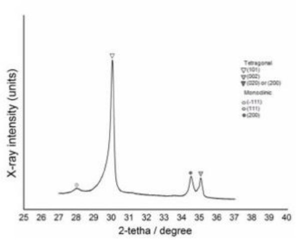
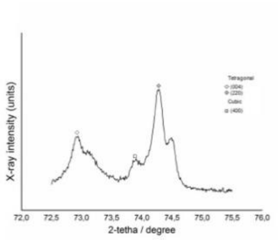
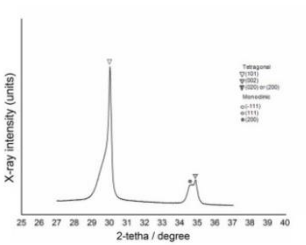
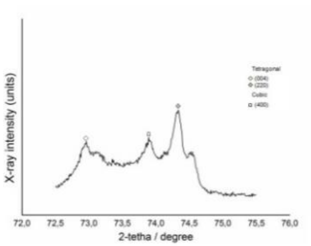
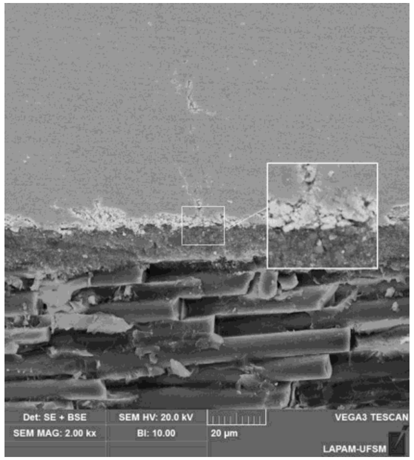
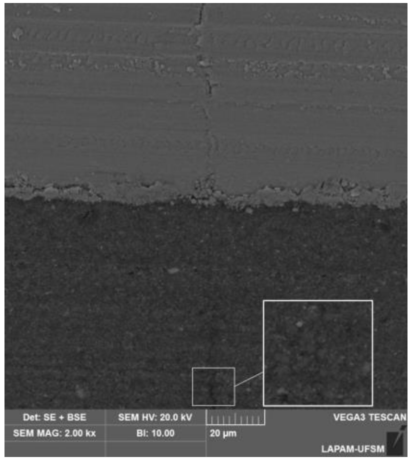
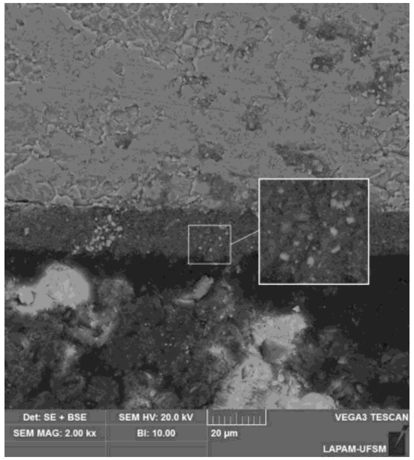
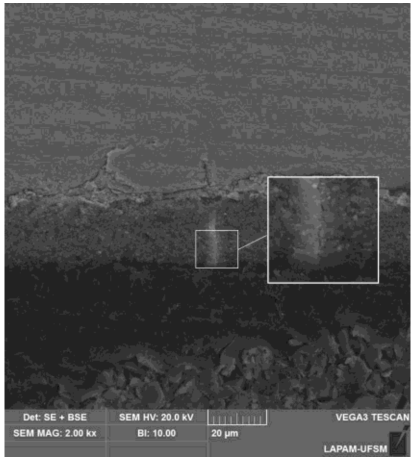
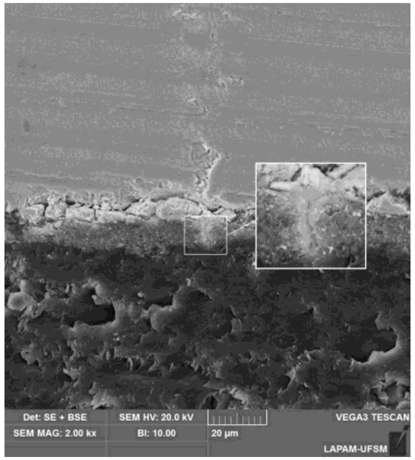
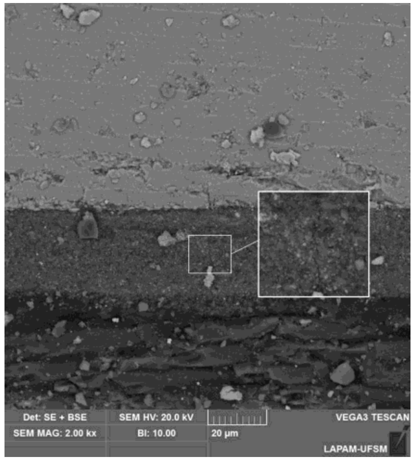
2nd genetaion zirconia				**3rd generation zirconia**	
					
Lava Plus		**In-Ceram YZ-HT**		**Katana UTML**	
					
Katana ML		**Zirlux FC**		**Katana STML**	

Figure 5. Fractographic analysis obtained by Scanning Electron Microscopy at 2000× and 5000× (expanded image) magnification, showing all the failures originating from the cementation surface.

2nd genetaion zirconia		**3rd generation zirconia**
		
Lava Plus	**In-Ceram YZ-HT**	**Katana UTML**
		
Katana ML	**Zirlux FC**	**Katana STML**

3. ARTIGO 2 - Influence of finishing/polishing on the fatigue strength, surface topography, and roughness of an yttrium-stabilized tetragonal zirconia polycrystals subjected to grinding

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Running Title: Finishing, polishing and fatigue of Y-TZP ceramic.

Abstract

This study aimed to evaluate the effects of various polishing systems associated or not to finishing with diamond burs of lower-grit size on the topography, roughness, and fatigue behavior of a ground yttrium-stabilized tetragonal polycrystalline zirconia (Y-TZP). Disc specimens of Y-TZP (Zenostar T, Ivoclar-Vivadent) were produced (diameter=15 mm, thickness= 1.2 ± 0.2 mm; ISO 6872-2015) and randomly allocated into 8 groups: [Ctrl] as-sintered; [Gr] ground with coarse diamond bur; [Gr+Eve] grinding + polishing with EveDiacera (2-step polishing system); [Gr+Fin+Eve] grinding + finishing + polishing with EveDiacera; [Gr+Kg] grinding + polishing with Kg Viking (2-step polishing system); [Gr+Fin+Kg] grinding + finishing + polishing with Kg Viking; [Gr+Op] grinding + polishing with Optrafine (3-step polishing system); and [Gr+Fin+Op] grinding + finishing + polishing with Optrafine. Next, surface topography, roughness, phase transformation, fatigue strength (staircase method), and fractography analyses were performed. Grinding changed the surface topography and generated higher roughness (Ra in μm) (1.214); the subsequent finishing/polishing procedures were able to reduce the roughness (0.326-0.839); however, it remained higher than the control [ctrl] group (0.221). All samples subjected to surface treatment presented an increase in m-phase content (8.04% – 17.46%). In terms of fatigue strength (in MPa), the grinding group (677.36) and polishing/finishing groups (641.66-707.20) presented higher fatigue strength than the control [ctrl] group (592.48). Finishing before polishing had no effect on fatigue strength (645.37-707.20). Grinding altered the Y-TZP surface features and increased their fatigue strength by phase transformation mechanism, while the finishing/polishing procedures promoted surface smoothening, while maintaining high fatigue strengths. Finishing as an additional step before polishing had no effect on roughness reduction and fatigue strength improvements; thus, the finishing procedure might be unnecessary.

Keywords: Polycrystalline zirconia. Fatigue behavior. Surface alterations. Staircase method.

HIGHLIGHTS

- As-sintered samples had the smoothest surfaces, but the lowest fatigue strength.
- Grinding altered the topography and increased roughness of the Y-TZP surface.
- Finishing/polishing promoted surface smoothening of the ground Y-TZP surface, thus maintaining high fatigue strengths.
- The additional finishing step does not promote relevant effects on fatigue performance.
- No deleterious impact on mechanical fatigue performance could be observed.

1. Introduction

Clinical adjustments of fixed dental prostheses (FDP) are frequently needed to improve proximal/occlusal contact, emergence profile, restoration marginal fit, contouring of the restoration or cementation areas (Hmaidouch et al., 2014; Dutra et al., 2017). These adjustment procedures may be performed through a grinding process using diamond burs of medium and/or coarse grit-size (Bollen et al., 1997; Pereira et al., 2016). However, it is known that grinding leads to an increase in surface roughness, alters topography (Guilardi et al., 2017), and introduces different surface damages such as cracks and scratches, thus increasing the defect population onto the surface. Indeed, the changes in surface topography allied to a higher defect population may cause the following: i) compromise the mechanical performance of restorative materials (Pereira et al., 2016), ceramics are brittle materials (without plastic deformation), thus defects introduced in the surface can generate stress concentration, exceeding the materials' fracture toughness which can lead to a catastrophic failure (Scherrer et al., 2017); ii) increase the susceptibility to staining, plaque accumulation, gingival irritation, recurrent caries, thus impacting restoration longevity (Gunolol et al., 2012); and iii) intensify the wear of the antagonistic restoration/tooth (Sabrah et al., 2013; Luangruagrong et al., 2014; Preis et al., 2015[a]), dentin hypersensitivity, esthetic impairment, and degrades masticatory function (Goo et al., 2016). Furthermore, Hmaidouch and Weigl (2013) strongly emphasized that the maintenance of a smooth ceramic surface during clinical use is key to avoiding initiation and progression of microcracks, and minimizing abrasion of the opposing teeth.

Consequently, in order to reduce all of these drawbacks, the literature has shown that polishing protocols are mandatory (Preis 2015[b]; Goo et al., 2016; Park et al., 2016; Zucuni et al., 2017; Zucuni et al., 2018). Polishing is the process of producing a smooth, shiny surface through the use of abrasives (Goo et al., 2016). For instance, it can be performed by means of rubber polishers coated with diamond abrasive particles, starting from coarser-to-finer ones (Al-Haj Husain et al., 2016). As demonstrated by Steiner (2015), Huh et al. (2016), and Preis (2015[b]), there are different ceramic polishing systems specially based on 2- or 3-step systems, and all of them attempt to produce acceptable roughness parameters. Worth mentioning, these authors also highlight different degrees of surface smoothness associated with the different polishing systems. Furthermore, another debatable point is the necessity of performing finishing procedures prior to polishing. Finishing steps consist of using fine-grained diamond burs after grinding (Dutra et al., 2017). Hypothetically, finishing would aid in reducing intense heterogeneous topographical and rougher characteristics, enabling enhanced performance of polishing systems subsequently employed and which present even lower grit-size. However, comparisons and evaluations of the effects of polishing systems combined or not with finishing on the fatigue performance of restorative materials are scarce. Thus, studies aimed at elucidating the mechanism and performance of finishing and polishing systems, depicting their interaction with the surface of restorative materials are encouraged.

In regard to the influence of restorative materials, the fabrication of monolithic restorations using yttrium-stabilized tetragonal polycrystalline zirconia (Y-TZP) ceramics has been promoted due to superior mechanical and biological properties (Stawarczyk et al., 2017). When considering Y-TZP ceramics of second-generation, its already-known potential for t→m phase transformation triggering due to grinding or any surface treatments (air-abrasion, finishing, polishing) is highlighted (Aurelio et al., 2016; Zucuni et al., 2017). From this standpoint, finishing/polishing of the ground surface should be made mandatory, not only to achieve a smooth surface and reduce the surface defect/flaw population, but also to avoid a high content of monoclinic phase to prevent its degradation, which might improve Y-TZP restorations' longevity. Thus, the question is: do these finishing/polishing procedures affect the fatigue strength of a ground Y-TZP ceramic, as well as promote potential surface smoothing?

Based on the aforementioned premises, the present study aimed to evaluate and compare the effects of three polishing systems associated or not to finishing with fine and extra-fine diamond burs, assessing their effects on the surface characteristics (roughness and topography), phase transformation, and fatigue strength of a ground Y-TZP ceramic. The first null hypothesis was that distinct polishing systems generate similar fatigue strength. The second null hypothesis was that finishing before polishing does not affect fatigue strength.

2. Materials and Methods

The materials used in this study are presented in Table 1. Disc-shaped specimens of 2nd generation yttrium-stabilized tetragonal polycrystalline zirconia (Y-TZP - Zenostar T, Ivoclar Vivadent) were produced to test the biaxial flexural strength of ceramic materials following the guidelines of ISO 6872-2015 (final dimensions: 15 mm in diameter and 1.2 ± 0.2 mm in thickness).

For this purpose, a Zenostar blank (98-mm diameter; 16-mm thick) was cut with a diamond disc, which was coupled to a handpiece (W&H, Burmoos, Austria) to produce 20 mm^3 blocks. Next, metallic rings 18 mm in diameter were glued at two flat opposite ends and then the lateral part of the blocks was ground in a polisher (EcoMet/AutoMet 250, Buehler, Lake Bluff, IL, USA) using silicon carbide papers (SiC 600-1200 grit; 3M, Sumare, Brazil) until reaching the rings to shape zirconia cylinders (18 mm in diameter and 18 mm in height). Then, discs ~ 1.6-mm thick were obtained by cutting under water-cooling, using diamond blades in the cut machine (ISOMET 1000, Buehler).

The specimens were then standardized and polished on both sides with SiC paper (1200-grit) until they reached a thickness of 1.5 mm. Next, the specimens were sintered at 1450°C for 2 h (Zyrcomat 6000MS, Vita Zahnfabrik, Bad Sackingen, Germany). All specimens were measured using a digital caliper (Mitutoyo Series 209 Caliper Gauge, Takatsu-ku, Kawasaki, Kanagawa, Japan) to confirm that the dimensions conformed to ISO 6872-2015 recommendations, then allocated into 8 groups (n=15) according to the surface treatment (Table 2).

2.1 Surface Treatments

2.1.1 CTRL - As-sintered

Samples remained untouched after sintering.

2.1.2 Gr - Grinding

The grinding process was manually performed using a coarse diamond bur (#3101G–grit-size 181 µm; KG Sorensen, Cotia, Brazil), 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).

The specimens were attached to a metal base to maintain the diamond bur parallel to the specimen surface, and the grinding process was executed with horizontal movements. The side to be ground was marked with a permanent marking pen (Pilot, São Paulo, Brazil) to ensure that the entire zirconia surface was ground, and the grinding process was performed until complete elimination of the marking, with each specimen being ground with a new diamond bur (1 bur per specimen). This methodology was previously described and employed by Pereira et al. (2014).

2.1.3 Finishing

The finishing process was sequentially performed with fine (#3101F–grit-size 46 µm; KG Sorensen) and extra-fine (#3101FF–grit-size 30 µm; KG Sorensen) diamond burs, following the same protocol described for surface grinding (2.1.2 section).

2.1.4 Polishing

Three polishing systems were selected for this study. The factors considered in selecting such systems were based on the manufacturer's indications (specific to zirconia or generic for restorative materials) and the treatment protocol used (simplified systems with a 2- step technique, or more complex ones that use a 3-step technique), as described in Table 2.

The polishing process was executed according to the manufacturer's recommendation for each polishing system:

- *Optrafine System (Ivoclar-Vivadent)*: consists of 2 tips (Light-blue and Dark-blue) and a nylon brush, which is used with a diamond paste (OptraFine HP Polishing Paste);
- *EveDiacera (Eve Ernst Vetter*): consists of 2 tips (salmon and green);
- *Kg Viking (Kg Sorensen)*: is composed of 2 tips (blue and gray).

All of the specimens were affixed onto a metal base to guarantee parallelism between the sample and polishing tip. The area to be polished was divided into two regions (considering the size of the polishing tip) and the polishing was executed for 25 s in each region for each specific tip.

2.2 Surface topography

Two specimens per group were cleaned in an ultrasonic bath (1440 D–Odontobras, Ribeirao Preto, Brazil) with 78% isopropyl alcohol for 5 min prior to analysis. Next, they were gold-sputtered and analyzed with Scanning Electron Microscopy (SEM - Vega3, Tescan, Czech Republic) under 2000× and 5000× magnifications to inspect the surface topographic pattern.

2.3 Roughness analyses

The roughness of all specimens from each condition (n=15) was measured by profilometry (Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan) for Ra and Rz parameters, as recommended by ISO 4287-1997. Three measurements were carried out per specimen with a cut-off (n= 5): λC 0.8 mm, λS 2.5 µm, being that the mean average value was calculated for each specimen to be used for statistical analysis.

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

Two specimens from each group were analyzed using an X-ray Diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) to quantify the monoclinic/tetragonal phase content. Data was collected in a Bragg-Brentano assembly on 2θ, considering an angle interval of 24-35° with a step size of 0.03° for each second, and the m-phase content was obtained through the Garvie and Nicholson methodology modified by Toraya and collaborators (Pereira et al., 2014; Pereira et al., 2015).

2.5. Biaxial flexural fatigue test (staircase method)

First, three specimens from each condition were subjected to a monotonic biaxial flexural test (piston-on-three balls; ISO: 6872-2015 guidelines) in a universal testing machine (EMIC DL 2000, São José dos Pinhais, Brazil) in order to define the fatigue protocol.

Next, a fatigue test (n = 15) was performed in an electric machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, USA) using the staircase approach described by Collins (1993). The parameters for the fatigue test were defined from the mean obtained in the monotonic tests: 60% from the static flexural strength mean as "*the initial strength for fatigue test*", and 5% from the aforementioned initial strength mean as "*step size for fatigue test*". The fatigue tests were executed under 20,000 cycles, with a frequency of 6 Hz. If the tested specimen survived (no fracture) until the end of the test, one step-size was increased for the subsequent sample to be tested. If fracture occurred, the step decreased. Thus, 15 specimens from each group were tested after the first outcome inversion (survival or fracture) (Collins, 1993).

2.6. Fractography analysis

All specimens were inspected by optical stereomicroscopy (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany) and the representative fracture patterns of each group were selected for SEM analysis (Vega3, Tescan, Czech Republic) to determine the fracture and crack origin characteristics.

2.7. Data analysis

SPSS statistical software was used to perform the descriptive analysis of the roughness (Ra and Rz parameters) and fatigue strength (σf) to obtain the mean and standard deviation values. As the data assumed a non-parametric distribution (Shapiro Wilk test), the Kruskal-Wallis and post hoc LSD were used (α= 0.05).

3. Results

3.1 Roughness measurements and topography

The grinding process with diamond bur generated the highest surface roughness (Table 3), introducing defects and scratches on the surface. All of the polishing procedures, either associated or not to the finishing, reduced surface roughness (Figure 1 and Table 3), thus lessening the number and scratch marks (Figure 1), even though it was not able to restore the initial smoothness (as-sintered samples). The Eve Diacera system showed the best potential to smoothen the surface (both to Ra and Rz parameter - Table 3), since the finishing procedure had no effect (no need to be performed). The finishing also presented a positive effect on reducing the roughness when associated with Kg Viking and Optrafine systems (Table 3).

3.2 Phase transformation

All treatment groups led to an increase in monolithic phase content compared to the as-sintered samples (Table 3). Grinding generated a higher m-phase percentage, with a slight reduction in the m-phase after finishing/polishing procedures were noted (Table 3).

3.3 Fatigue strength

The fatigue findings are shown in Table 3 and Figure 2. Grinding generated an increase in fatigue strength compared to the control group. Furthermore, the polishing/finishing groups had higher fatigue strength than the as-sintered group; i.e., no deleterious effects occurred in the fatigue behavior. Finishing before polishing had no effect on fatigue strength when comparing the groups with Vs without finishing for the same polishing system (Gr+Eve = Gr+Fin+Eve; Gr+Kg = Gr+Fin+Kg; Gr+OP = Gr+Fin+OP).

3.4 Fractography analysis

The fracture in all of the tested samples started from the surface/sub-surface defects at the opposite side to the load application; i.e., the side where there was a concentration of tensile stress (treated surface).

4. Discussion

Our data showed that distinct polishing systems produced different effects on surface fatigue strength, thus rejecting our first null hypothesis. However, finishing had no effect on the fatigue behavior; thus, the second hypothesis was accepted. In terms of the roughness effect, finishing with fine and extra-fine diamond burs had no effect when performing before the Eve System, while it contributed to smoothening when associated with the Kg Viking and Optrafine systems.

Zirconia is a very difficult material to polish (Kou et al., 2006), because it is composed of homogeneous and dense polycrystalline structures, which present high hardness (1140 Knoop value) (Apholt et al., 2001; Huh et al., 2016). Thus, diamonds are frequently used as the main abrasive in tools for grinding and polishing. In addition, the SiC can be used as supplementary abrasive (Huh et al., 2016). Our SEM images (Figure 1) and Ra/Rz data (Table 3) show that all polishing systems led to surface roughness reduction (i.e., smoothening effect). However, none of the polishing systems were able to re-establish the initial surface smoothness found in as-sintered samples (control group). Our results are in accordance with previous studies (Manawi et al., 2012; Al-Haj Husain et al., 2016).

The literature has been showing that diamond pastes are effective in reducing surface roughness generated by clinical adjustments when used after initial polishing (Sarac et al., 2006). However, in our study, the polishing with diamond paste associated with a nylon brush (Optrafine Polishing) had a somewhat restricted smoothening effect, since it presented intermediate roughness results (higher than the KG Sorensen system and lower than the EVE system) (Figure 1, Table 3). Polishing systems that are not specifically indicated for zirconia, such as those utilized to polish any ceramics (e.g., KG Viking), might be composed of abrasive particles with insufficient hardness. Thus, they might be ineffective in terms of promoting a smoothening effect when used to polish harder surfaces, such as Y-TZP ceramics (Park et al., 2017). Our results corroborate this assumption, since the Kg Viking polishing system, which is indicated for all types of ceramics and metal, reduced surface roughness compared to the grinding group, but it was less effective than EveDiacera and Optrafine Polishing (Figure 1, Table 3).

While conducting polishing, an increase in the local temperature (heating) might occur, which might trigger the m→t reversal phase transformation (Juy & Anglada 2007), whose reversion could be noted in our experiment (Table 3). In this context, other factors, such as grit-size, magnitude, speed, and load applied on the polishing and thermal conductivity of zirconia can influence the change of the crystal structure (Husian et al., 2016).

From a mechanical behavior standpoint, it is well known that the flexural strength of glass ceramics might be influenced by their surface roughness (Nakazato et al., 1999). Also, its fatigue behavior is significantly increased through surface smoothening via polishing (Nakazato et al., 1999; Isgro et al., 2003; Nakamura et al., 2010). However, the surface roughness does not seem to play a significant role in the mechanical behavior of Y-TZP-based ceramics (Zucuni et al., 2017), as these materials present t→m phase transformation when subjected to mechanical stimuli (i.e., grinding, polishing) (Preis et al., 2015^{a}). The t→m phase transformation creates a residual compressive stress layer that hinders crack propagation;

thereby improving the mechanical behavior of the ceramic (Chevalier et al., 2009). Our results corroborate these considerations for Y-TZP (Figure 2, Table 3).

In addition, a toughened surface generated by residual stress concentration in response to a layer of transformed grains (m-phase content increase) (Pereira et al., 2014) due to surface adjustments may be desirable, unless the flaws/defects are removed as much as possible (Guilardi et al., 2017). If the defects are deeper than the transformed layer zone or if a slow-crack growth mechanism is triggered by fatigue stimuli over time, a deleterious impact may be expected (Guazzato et al., 2005; Green, 1983; Kosmac et al., 1999). Damage caused by the occlusal adjustment of contact points (i.e., grinding grooves, defects, flaws) might further lead to the origin (starter) of cracking and catastrophic failure (Preis et al., 2012). Therefore, accurate polishing of ground Y-TZP surfaces with appropriate systems for zirconia seems to be important for clinical long-term success (Preis et al., 2015), not only for fatigue improvements, but also for the smoothening effect (Bollen et al., 1997; Preis et al., 2012; Dutra et al. 2017; Dutra et al. 2018).

Taken together, polishing systems specifically indicated for zirconia show better potential for surface smoothness, being that the finishing might be dispensable. On the other hand, when universal polishing systems are used, perhaps finishing with a fine and extra-fine diamond bur before polishing should be considered. In terms of fatigue outcome, the tested polishing/finishing procedures resulted in no damaging effects on the current Y-TZP material. However, it is important to emphasize the inherent limitations of this *in vitro* work, such as: no evaluation under sliding motion, no changes in temperature and in pH, no color change assessments, Y-TZP samples were not bonded to the substrates (monolithic samples were tested), and being subjected to intermittent loading to assess the effects on fatigue behavior of the restorations.

5. Conclusion

- As-sintered samples had the smoothest surfaces, lower m-phase content, and lowest fatigue strength.
- The finishing/polishing procedures smoothed the ground Y-TZP surface, keeping their fatigue strengths higher than as-sintered surfaces (no damaging effect).
- Finishing with fine and extra-fine diamond burs as an additional step before polishing had no effect on roughness reduction or fatigue strength improvements; thus, the finishing procedure might be dispensable, maintaining the polishing of ground Y-TZP surface as essential.

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TABLES

Table 1. Materials their main compositions and manufactures.

Materials	**Composition**	**Trade mark and Manufacturer**	**Batch number**
Yttrium-stabilized partially tetragonal zirconia polycrystals 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%	Zenostar T, Ivoclar Vivadent, Schaan, Liechtenstein.	U20833
Diamond bur #3101G – grit size 181 µm	Diamond and stainless steel	KG Sorensen, Cotia, Brazil.	14763
Diamond bur #3101F – grit size 46 µm	Diamond and stainless steel		14720
Diamond bur #3101FF – grit size 30 µm	Diamond and stainless steel		19624
3-step polishing system	**Light-blue and Dark-blue tips**: synthetic rubber, diamond granulate, titanium dioxide and stainless made of steel; **Nylon brush**: nylon fibres and stainless made of steel; **Diamond past**: diamond dust (2-4 µm), glycerine, sodium lauryl sulphate and propylene glycol.	Optrafine System, Ivoclar Vivadent, Schaan, Liechtenstein.	UL0793
2-step polishing system	**Tip green**: medium grit; **Tip Salmon**: fine	Eve Diacera, Eve Ernst Vetter, Keltern, Germany.	302410
2-step polishing system	Sulphonated chlorine polyethylene silicon carbide, stainless made of steel. **Blue tip**: fine; **Gray tip**: extra-fine	Kg Viking, Kg Sorensen	16219

Table 2. Study design.

Group's codes	Surface treatment			Manufacturer's recommendation for
	Grinding (Coarse)	**Finishing** (Fine and Extra-fine)	**Polishing**	
Ctrl	Without treatment (as-sintered)			-
Gr	with	-		-
Gr+Eve	with	without	Eve Diacera (two step system)	Zirconia restorations
Gr+Fin+Eve	with	with		
Gr+Kg	with	without	Kg Viking (two step system)	Ceramic and metal restorations
Gr+Fin+Kg	with	with		
Gr+OP	with	without	Optrafine (three step system)	Lithium disilicate, leucita and zirconia restorations
Gr+Fin+OP	with	with		

Table 3. Fatigue strength (σf) and 95% confidence interval (CI) via the staircase tests; roughness (Ra and Rz parameters in micrometers) and percentage (and SD) of m-phase content and (and SD).

Group	Fatigue strength (MPa)		Roughness Surface		Monoclinic phase (%)
	σf	**95% CI**	**Ra**	**Rz**	
Ctrl	592.48	579.15-605.82^{a}	0.221^{a}	1.865^{a}	3.84 (0.3)
Gr	677.36	662.12-692.60^{c}	1.214^{f}	7.416^{e}	17.46 (0.9)
Gr+Eve	648.98	620.70-677.25^{bc}	0.328^{b}	2.336^{a}	8.04 (1.4)
Gr+Fin+Eve	707.20	678.40-736.01^{c}	0.326^{b}	2.068^{a}	7.01 (1.2)
Gr+Kg	641.66	624.88-658.43^{b}	0.839^{e}	5.376^{d}	13.36 (1.2)
Gr+Fin+Kg	654.71	619.68-689.77^{bc}	0.572^{c}	3.845^{c}	14.21 (0.4)
Gr+OP	697.56	666.71-728.40^{c}	0.628^{c}	4.162^{c}	11.47 (0.6)
Gr+Fin+OP	645.37	628.51-662.26^{bc}	0.472^{d}	3.270^{b}	8.93 (2.1)

* Different letters indicate statistical differences depicted by Kruskal-Wallis and post hoc LSD tests.

FIGURES

Figure 1. Representative SEM images (2000× and 5000× magnification) of Y-TZP surface. It notices that grinding (Gr) introduces scratches and flaws in the ceramic surface; whereas all polishing systems depicted a potential to reduce such alterations. It highlights that finishing as additional step before polishing does not promote surface topographical improvements.

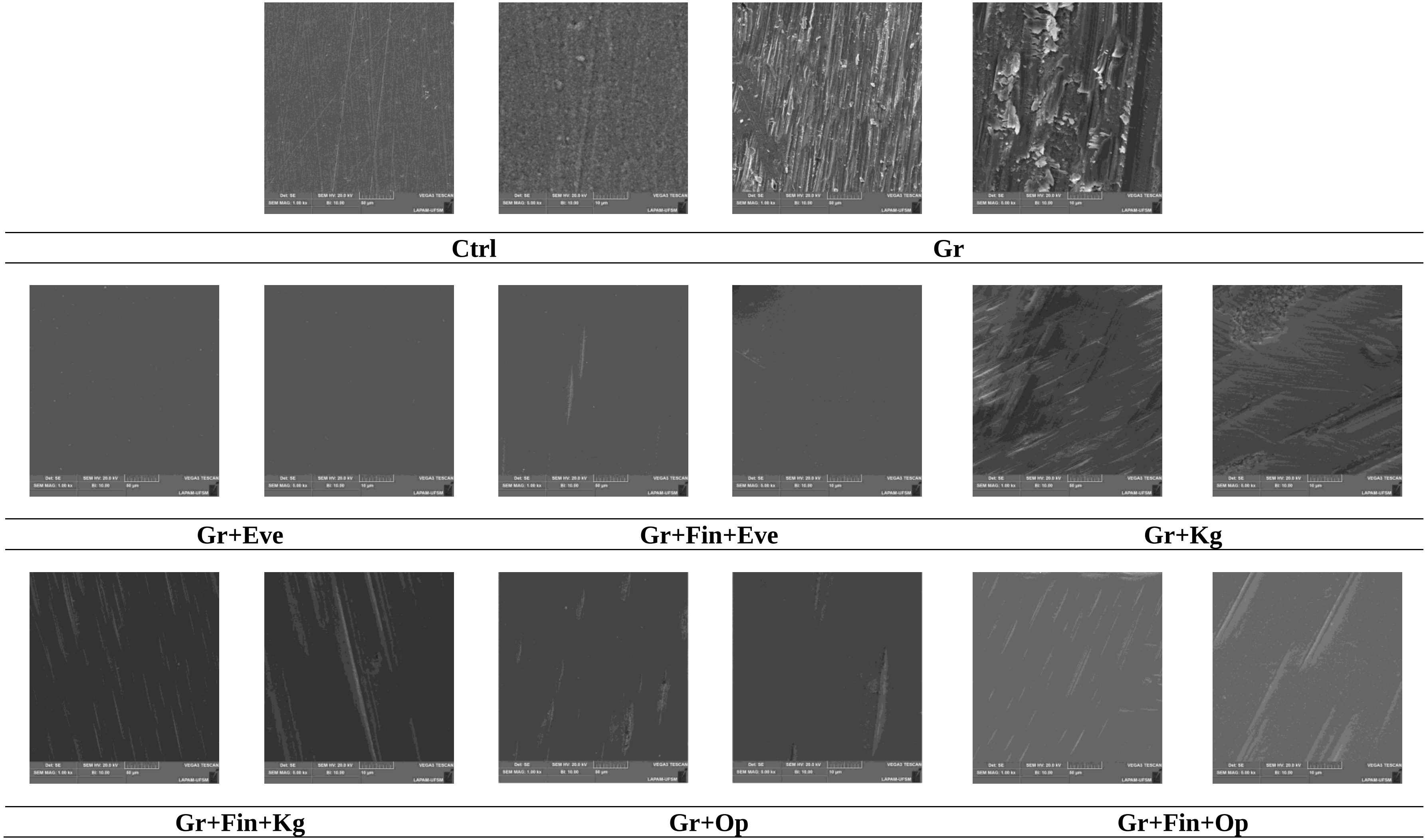


Figure 2. Staircase plots obtained through the fatigue test of all evaluated conditions. The red point indicates where test starts; the black points show the failed specimens and the white points indicate the survived specimens.

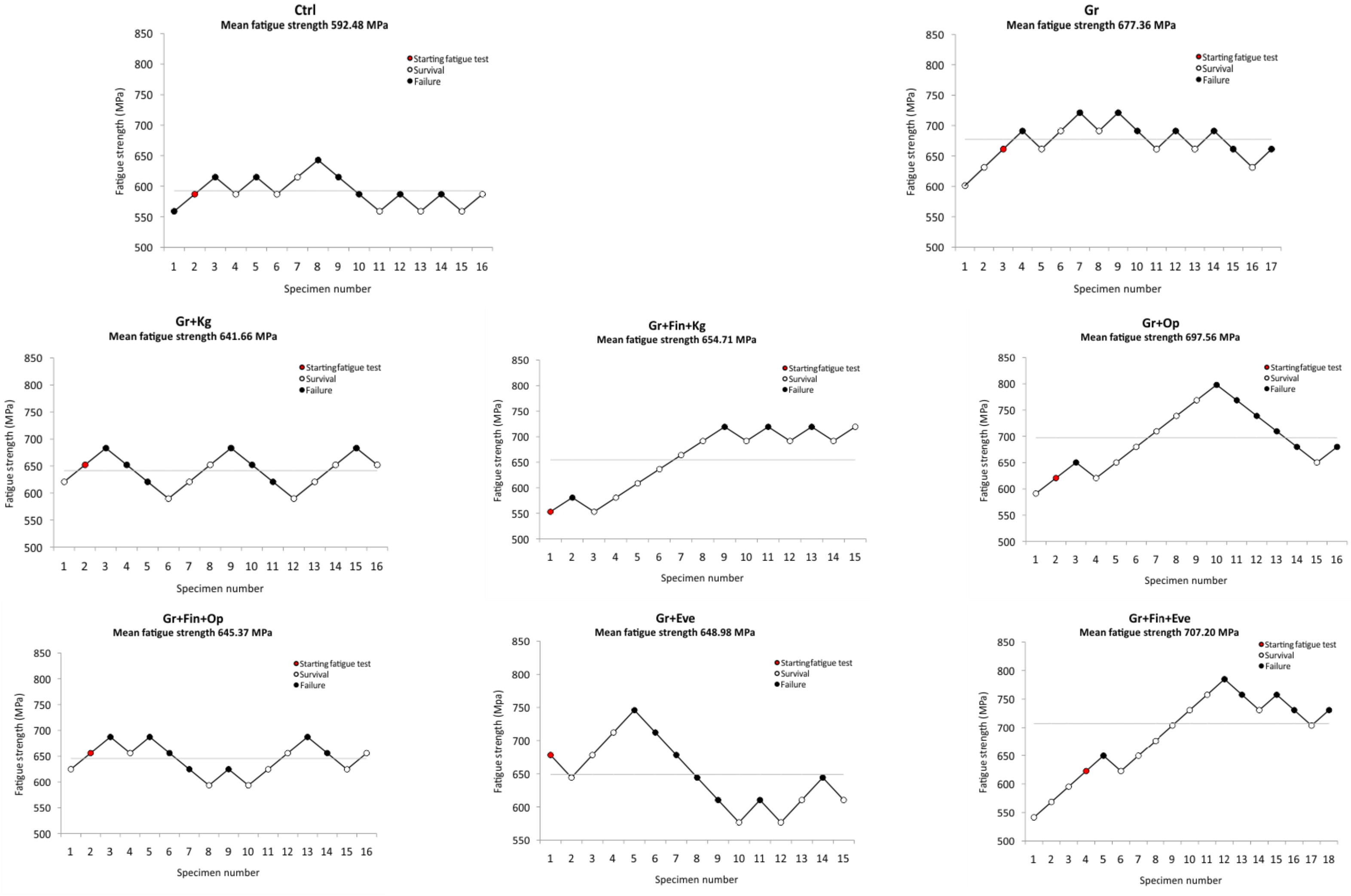
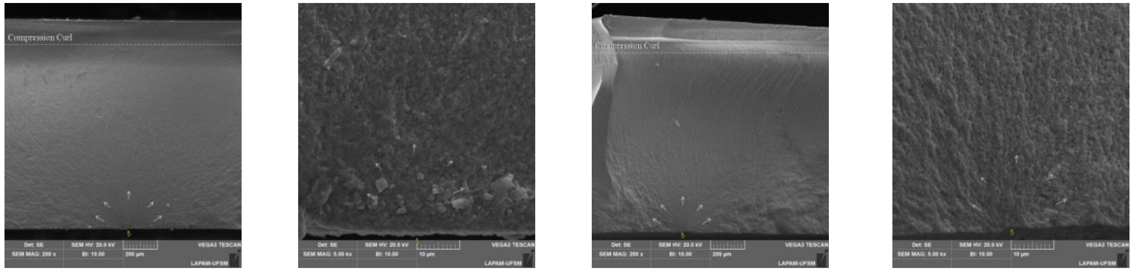
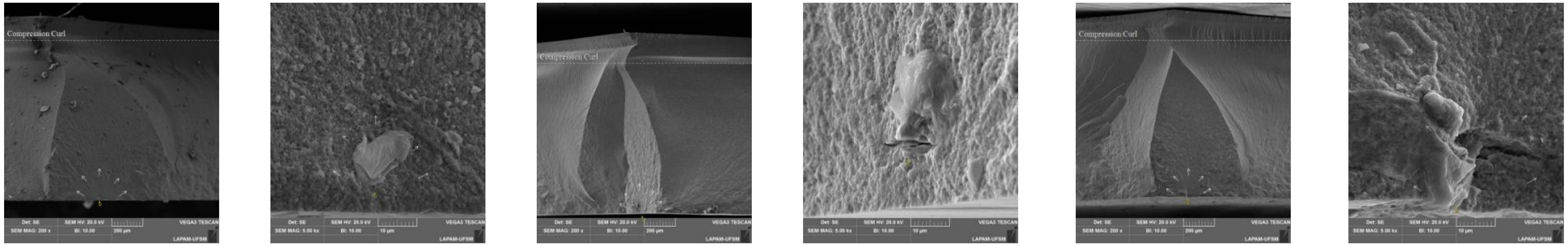


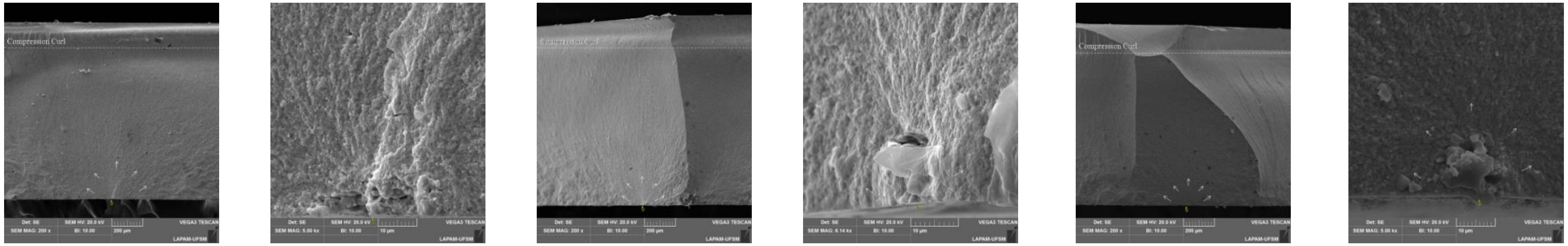
Figure 3. Representative SEM images of the fracture surfaces (magnification: 200× - left and 5000× - right), depicting that all fractures (region indicated by point 👆) started at the superficial/subsuperficial defects from the side where the tensile stresses are generated during the fatigue test. The white arrow indicates the cracks propagated (opposite to compression stresses concentrated during fatigue test - compression curl).



Ctrl **Gr**



Gr+Eve **Gr+Fin+Eve** **Gr+Kg**



Gr+Fin+Kg **Gr+Op** **Gr+Fin+Op**

4. ARTIGO 3 - Low-fusing porcelain glaze application does not damage the fatigue strength of Y-TZP

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Running Title: Glaze and fatigue of high translucent Y-TZP

Abstract

This study evaluated and compared the effects of two glaze application methods (brush and spray) on the fatigue strength and surface characteristics (topography and roughness) of a translucent yttrium stabilized partially tetragonal zirconia polycrystal ceramic (Y-TZP) prior to and after grinding. Disc-shaped specimens of translucent Y-TZP (Vita YZ-HT; Vita-Zahnfabrik) were processed (ISO 6872–2015) and randomly allocated into 6 groups, according to the surface treatments performed on the tensile surface: Ctrl–as-sintered (no treatment); Gr–grinding with a diamond bur (181μm-grit; #3101G); Br–glaze obtained from a powder–liquid mix and applied by brush (Vita Akzent; Vita Zahnfabrik); Sp–glaze application via spray (Vita Akzent Plus; Vita Zahnfabrik); Gr + Br and Gr + Sp–association of grinding + respective glaze method. Analyses of surface roughness (Ra and Rz), fatigue strength (staircase method), surface topography and fractography were carried out. The assintered condition had the smoothest surface, while grinding led to the rougher and more heterogeneous topography. Both glaze application methods showed a potential for topography evenness (smoothening effect), while the glaze spray method led to thinner layers of material, showing a limitation in reducing the roughness compared to the brush method. No deleterious effect on fatigue strength of the Y-TZP could be observed, as the glaze-spray application on the as-sintered surface showed the highest values. Fractography depicted two distinct fracture origin regions: from defects in the surface/sub-surface region for the Ctrl and Gr groups; and at the zirconia-glaze layer interface for Br, Sp, Gr + Br and Gr + Sp. The clinical relevance of this work is that the tested glaze application methods did not damage the fatigue strength of the tested Y-TZP.

Keywords: Yttrium stabilized tetragonal polycrystal zirconia. Post-processing treatments. Grinding. Glazing. Fatigue test.

Highlights:

- Glaze application had no deleterious effect on the fatigue strength of the Y-TZP ceramic.
- The brush technique for glazing promoted better results regarding surface roughness.
- The spray technique on the as-sintered surface was superior regarding fatigue behavior.

1. Introduction

Monolithic full-contour zirconia restorations have been indicated as an alternative to classic veneered bilayer fixed dental prostheses (FDPs) (Konstantinidis et al., 2018). Milling scratches are introduced during processing in CAD/CAM systems and inherently result in a roughened restoration surface (Addison et al., 2012; Fraga et al., 2015). Rough external surfaces may predispose to caries and the occurrence of periodontal disease (Gonulol and Yılmaz, 2012), as well as potentiate the wear of opposing teeth (Preis et al., 2016), while rough inner surfaces (cementation surface) may further reduce the mechanical competence of all-ceramic restorations, since the radial cracks starting from the defects at the inner surface induct consequential failures when under clinical service (i.e., cyclic intermittent loading) (Kelly et al., 1990; Bindl and Mormann, 2004; Prochnow et al., 2018;Zucuni et al., 2018). Moreover, both internal and external surfaces may interfere in the optical properties and impair aesthetics (Lee et al., 2016). Thus, all of these outcomes might influence the longevity of such restorations, and therefore finishing, polishing and/or glazing protocols should be considered as mandatory for smoothening effect (reduction in surface roughness and to even the topography), particularly on the external surfaces (Carrabba et al., 2017).

Glazing consists of the application of a thin layer of a low-fusing silica-based material (vitreous or glassy material) onto the restoration's outer surface (Manjuran and Sreelal, 2014). The glaze application results in a fill-up mechanism (healing effect), from which the glaze penetrates the existing scratches, microcracks, and porosity on the ceramic surface, and in doing so, leads to a smoother surface with less defects (Anusavice and Phillips, 2003). However, the effects on the mechanical behavior of glass ceramic (Anusavice and Phillips, 2003; Aurelio et al., 2017) and of yttrium-stabilized tetragonal zirconia polycrystal ceramic (Y-TZP) (Amaral et al., 2016; Chun et al., 2017; Zucuni et al., 2017) can still be considered controversial. In particular, Y-TZP is a ceramic mainly composed of a polycrystalline microstructure without any glass content (or tiny glass traces) (Denry et al., 2014), resulting in two problems: i) an inherent incompatibility between glaze and zirconia, which leads to a weak adhesion between these materials (Aboushelib et al., 2008; Yamamoto et al., 2016); and ii) a thermal coefficient mismatch between them, which may lead to residual stress introduction and increased probability of failure, thereby compromising the mechanical properties (Tan et al., 2012; DeHoffet al., 2008). Furthermore, as known in material science, highly glassy materials (such as glaze) present very low tensile strength, because they are extremely brittle. These conditions might damage the mechanical performance of Y-TZP based materials when glazed and undergoing intermittent loading (Della Bona et al., 2003; Borba et al., 2011; Pozzobon et al., 2017), being a relevant research question.

From both clinical and laboratory standpoint, glaze application is very common in Y-TZP restorations, particularly in monolithic full contour ones, which (if applicable) receive the shaded glaze application (material with pigments) for esthetic (external pigmentation) and optical improvements for matching the natural dentition (Kumchai et al., 2018). In terms of the glaze application technique, there are two approaches: classical, which requires the powder being mixed to a specific liquid, so that the mixture is applied on the ceramic surfaces with a brush; or the spray option, applied by spraying a similar glassy material (Antunes et al., 2018). The literature (Pozzobon et al., 2017; Zucuni et al., 2017) shows that the glaze application, which is applied by brush, generates a layer with many internal defects (i.e., bubbles), despite its homogeneous outer surface. These defects may act as stress concentration factors and, as a consequence, contribute to a higher risk of failure originating from the interface of the set (zirconia/glaze) (Zucuni et al., 2017), thus affecting the survival of Y-TZP restorations, as previously mentioned. On the other hand, the spray glaze application seems to have a beneficial effect on the mechanical behavior of Y-TZP (Chun et al., 2017), because it makes the glaze layer thinner and more homogeneous, thus reducing the defect population (Chun et al., 2017).

Nevertheless, until now, there is no study that compares the fatigue behavior of Y-TZP ceramics subjected to the two distinct glaze application methods (brush vs. spray).

Another clinical standpoint involves evaluating Y-TZP restorations before cementation and glazing; clinical adjustments are commonly executed to improve adaptation of the restoration (Hmaidouch et al.,2014). These adjustments are performed through grinding with a diamond bur and introducing defects (damage, scratches) on the ceramic surfaces, thus increasing surface roughness (Guilardi et al., 2017; Zucuni et al., 2017). As well-known, the strength of ceramic materials is directly dependent on its surface roughness (the rougher surface, the lower the strength) (Fischer et al., 2003; Schmitter et al., 2015), and it also has to be highlighted that the roughened interfaces might induce cracks propagation and reduce the material's strength (Evans and Hutchinson, 1989). Therefore, as already mentioned, a ground surface should be smoothened by finishing, polishing, and/or glazing.

Considering the aforementioned aspects, some relevant clinical issues should be answered: How is the fatigue behavior of ground Y-TZP subjected to glazing procedure? Would the glassy-based glaze material “heal” the surface defects (scratches) created by grinding, making the surface smoother and having a strengthening effect of the Y-TZP? Or would the glassy material weaken the Y-TZP, since this vitreous material has low tensile strength (is more brittle) and due to thermal incompatibility between the Y-TZP and the glaze material? Also, do glazing by brush and spray techniques present similar effects on the fatigue strength of Y-TZP? Therefore, this study aimed to evaluate and compare the effects of two glaze application methods (brush and spray) on fatigue strength and surface characteristics (topography and roughness) of a Y-TZP ceramic prior to and after grinding. The null hypothesis assumed that the glaze application by brush (powder/liquid mix) and the spray techniques would have no effect on the fatigue behavior of the Y-TZP ceramic.

2. Materials and Methods

Table 1 describes the materials utilized in this study. The methodology for shaping Y-TZP blocks into discs has previously been described by Guilardi et al. (2017). For this, a Vita YZ-HT disc (98 mm diameter) was cut with diamond discs into blocks of 20 mm × 20 mm. Next, metallic rings of 18 mm in diameter were attached at both ends of each block and a cylinder was obtained by grinding with silica carbide papers (600–1200 grit) in a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA). Then, 1.65 mm-thick discs were obtained by cutting the cylinders with a diamond blade (ISOMET 1000, Buehler) under water-cooling. The discs were subsequently regularized and polished on both sides with silicon carbide paper (1200 grit) until 1.5 mm thick, and then sintered at 1450 °C for 2 h (Zyrcomat 6000MS furnace, Vita Zahnfabrik). After sintering, all discs were inspected with a digital caliper (Mitutoyo Series 209 Caliper Gauge, Takatsu-ku, Kawasaki, Kanagawa, Japan) to ensure the dimensions were in accordance with ISO:6872-2015 (15 mm in diameter and 1.2 ± 0.2 mm in thickness) and then they were randomly allocated as described in Table 2.

2.1 Surface treatments

Importantly, all surface treatments were only performed on the Y-TZP discs' surface, which was subjected to tensile stress in the mechanical fatigue tests. Several training experiments were performed as pilot studies to enhance reproducibility and standardization of these critical processing steps, followed by topographical and surface characteristics analysis until a standardized protocol was finally achieved. Then, a single trained operator executed all the processing steps (CPZ).

2.1.1 Ctrl - As-sintered

Specimens that did not receive any treatment after sintering, were kept as-sintered.

2.1.2 Grinding -Gr

This methodology was previously employed by Pereira et al. (2014). Basically, specimens were attached to a metal base in order to maintain the specimen surface parallel to the diamond bur tip. The surface to be ground was then marked with a permanent marking pen (Pilot, São Paulo, Brazil), followed by grinding, which was manually performed until the total removal of such marking. Grinding was executed using a 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). The diamond bur was replaced after grinding each specimen (1 bur per specimen).

2.1.3 Glazing - Gl

Two glaze application techniques were used in this study, following strict guidelines of the manufacturer: Vita Akzent (powder/liquid) and Vita Akzent Plus (spray) (Table 1).

- Application of Vita Akzent powder/liquid by brush: The powder was mixed with distilled water at a ratio of 1 spoon of powder to 2 drops of liquid, following the manufacturer's instructions, until a homogeneous and creamy paste was achieved. The mixture was then applied onto the Y-TZP surface with a specific brush (# 0, Condor, São Bento do Sul, Brazil), followed by the appropriate glaze firing (Vacumat 6000MP furnace, Vita Zahnfabrik): initial temperature of 500 °C, maintained for 4 min for pre-dry at a heating rate of 80°C/min until a final temperature of 900°C and maintained for 1 min.

- Application of Vita Akzent Plus Spray: First, the recipient containing the glaze (bottle) was shaken for about 1 min to mix the material, as recommended by the manufacturer. Subsequently, the spray was applied at a distance of 15 mm between the applicator's tip and the ceramic surface. A slight oscillatory movement had to be performed until the ceramic surface was entirely covered prior to the glaze firing (Vacumat 6000MP furnace, Vita Zahnfabrik) as recommended: Initial temperature: 500°C, maintained for 4 min for pre-dry, at a heating rate of 80°C/min until the final temperature of 950°C, maintained for 1 min.

2.2 Surface topography

Two specimens per group were cleaned in an ultrasonic bath (1440 D–Odontobras, Ribeirao Preto, Brazil) with 78% isopropyl alcohol for 5 min, and then gold sputtered before being analyzed under Scanning Electron Microscopy (SEM - Vega3, Tescan, Czech Republic) to observe the surface topography on the top and cross-sectional regions. For cross-sectional imaging, specimens were transversally cut with a diamond blade (ISOMET 1000, Buehler). In addition, representative specimens (n=2) of each evaluated condition were selected for analyses of the glaze layer thickness, from which 2 measures per specimens were performed.

2.3 Roughness analyses

All specimens from each group were analyzed using a profilometer (Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan). Roughness measurements were determined by parameters recommended by ISO:4287-1997 (Ra and Rz). Three readings were performed per specimen in a perpendicular direction to the grinding with a cut-off (n=5), λC 0.8 mm, and λS 2.5 µm, and the mean average values were then obtained for each specimen.

2.4 X-ray Diffraction (XRD)

One additional specimen from each group was manufactured and analyzed by X-ray Diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) to verify the presence/absence of monoclinic phase. In the correspondent glaze groups application, only the firing cycle was performed (i.e without glaze layer). Data was collected in a Bragg-Brentano assembly on 2θ, with the angle interval of 27-37° 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).

2.5 Biaxial flexural fatigue test (by the staircase method)

Prior to the fatigue test, a monotonic static biaxial flexural test (n=3) was performed using a universal testing machine (EMIC DL 2000, São José dos Pinhais, Brazil) to determine the stress parameters for fatigue tests. This followed the ISO:6872-2015 guidelines for biaxial flexural strength testing of ceramic materials. The specimens were positioned with the treated surface down (tensile stress side) onto three support balls (Ø=3.2 mm), which were placed 10 mm apart from each other in a triangular disposition. The specimens were immersed in water and a flat circular tungsten piston (Ø=1.6 mm) applied an increasing load (1 mm/min) until sample failure. The fatigue test (n=15) was then performed (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, USA) using the staircase method described by Collins (1993).

The parameters for the fatigue tests were defined as follows: *initial stress* – consists of 60% of the monotonic flexural strength mean; *step-size (increment)* – 5% of the calculated initial strength mean for the fatigue test. The fatigue test was executed under 20,000 cycles for each step stress and a frequency of 6 Hz. The first specimen was tested applying the initial stress according to the parameters for each group. The next specimen was tested according to the outcome (survival or fracture) with a variation in the stress increment; i.e., if the first specimen survived, the stress applied to the next one was increased by one increment. On the other hand, if the specimen fractured, the stress was decreased in the next step. After curve inversion (survival or fracture), fifteen (15) specimens from each evaluated condition were tested following this methodology (Collins, 1993).

2.6 Fractography analysis

All specimens were examined by a single trained researcher (CPZ) in an optical stereomicroscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany). Representative fracture patterns for each group were selected and subjected to SEM analysis (Vega3, Tescan, Czech Republic) to determine the crack origin and fractography characteristics.

2.7 Data analysis

The flexural strength data assumed a parametric, homoscedastic distribution (Shapiro Wilk and Levene tests), thus, analysis of variance (two-way ANOVA) and post-hoc Tukey tests were used ($\alpha = 0.05$). The roughness (Ra and Rz parameters) data assumed a non-parametric distribution, thus Kruskal-Wallis and post hoc LSD were used ($\alpha = 0.05$).

3. Results

3.1 Roughness and topography analyses

The as-sintered condition presented an already-known characteristic pattern of topography of Y-TZP ceramics (dense polycrystalline structure) and showed lower roughness measurements (Figure 1; Table 3). Grinding with a diamond bur deformed the surface, introducing defects, scratches, and completely altering the topographic pattern (uneven surface), thus generating the highest surface roughness (only statistically similar to the Gr+Sp group). Both glazing applications led to a more homogeneous surface topography having a smoothening effect; however, it was rougher than the as-sintered surface. Comparing the two glazing techniques, the brush technique promoted smoother surfaces than the spray technique by either applying on the as-sintered surface or on the ground surface. The spray technique resulted in a thinner layer of material (Figures 2 and 3) compared to the brush technique.

3.2 X-ray Diffraction (XRD)

XRD data shows that grinding promotes the (t-m) phase transformation. Besides, the glaze firing cycle, when executed in the surface previously ground, it triggered some reversal phase transformation (m-t) (Table 3). When no grinding was executed, no m-phase could be detected.

3.3 Biaxial flexural fatigue test

No deleterious impact on fatigue strength was observed after any treatment in which the group only glazed with spray (Sp) showed the highest values (Table 3; Figure 4).

3.4 Fractography analysis

First, it was found that all the fracture originated from the tensile side (surface treatment side) and propagated to the compression side, where a compression curl feature can be seen (Figure 5). Two distinct fracture origin regions could be observed: for Ctrl and Gr, the fracture originated at the defects of the surface/sub-surface region; for Br, Sp, Gr+Br, and Gr+Sp, the fracture originated in the interface between the zirconia and glaze layers (Figure 5).

4. Discussion

Our data show that the application of a thin layer of porcelain glaze either by brush or spray method did not impair the fatigue strength of a highly translucent Y-TZP, highlighting that the group only glazed with the spray technique (Sp) showed the highest fatigue strength values. Therefore, the null hypothesis could be accepted. In terms of surface characteristics, our data support the glaze application by brush since it promoted lower roughness values (more homogeneous topography) than the spray technique.

When yttrium-stabilized tetragonal zirconia polycrystal (recently referred as “2nd generation zirconia” and used in this study) are subjected to thermal and mechanical stimuli, tetragonal to monoclinic phase (t-m) transformations occur (Pereira et al., 2016[b]; Stawarczyk et al., 2017). Thus, grinding with a diamond bur can increase the monoclinic phase content (Guilardi et al., 2017), triggering the toughening mechanism (Chevalier & Gremillard, 2009), that leads to an increase in fatigue strength of Y-TZP (Guilardi et al., 2017). This might act against the negative effects of introducing surface defects by grinding and low strength of the glaze material under stress, thus maintaining a high fatigue strength of the Y-TZP. Our findings appear to corroborate with these explanations, since the fatigue strength was not damaged by surface treatments (Table 3).

When comparing the two methods for glaze application solely on the as-sintered surface, the glaze applied by brush (633.39 MPa) showed lower fatigue strength than the glaze spray (673.40 MPa) (Table 3). The lower results for the glaze brush were probably caused by bubbles (flaws) in the interior (bulk) of the glaze layer (Figure 3), whose inclusion was caused by the inherent imprisonment of air during mixing of the liquid and/or sintering process. According to Cazzato & Faber (1997), this factor may increase the risk of failure originated on this interface (glaze-zirconia), because the stress concentration is very dependent on the size of preexisting cracks/defects (Mecholsky, 1995). Hence, as demonstrated by Borba et al. (2011) in bilayer assemblies (i.e., Y-TZP + glaze), the strength and fracture mode are influenced by the properties of the material under tensile stress (as observed by us, i.e., the fracture starting at the interface between the glaze layer and zirconia in Br, Gr+Sp, and Gr+Br) (Figure 5). Another possible explanation is that the glaze spray promoted a thinner layer than the glaze layer obtained from the brush application; thus, the negative effects of the lower glaze material strength might have been decreased for the glaze-spray group compared to the glaze-brush group.

In contrast to the aforementioned statistical difference, the two glaze techniques had similar fatigue strengths (Brush: 612.49 MPa, Spray: 623.57 MPa) when considering the application on the ground surface. One possible explanation is that the grinding introduces surface defects, as shown in Fig. 1, as well as induces inherent increase in m phase; but the firing cycle of glaze induces phase transformation reversion (as consequence, it removes the compression layer in part), keeping the surface defects, thus damaging its fatigue strength. To corroborate this assumption, it is noted that the glaze spray applied on the “as-sintered” surface (673.40 MPa) had a higher fatigue strength than the glaze spray applied on the “ground” surface (623.57 MPa), statistically demonstrating a strong influence of grinding when the thin glaze layer is applied. This negative effect of grinding might be somewhat lower for the glaze applied by brush, probably because this group had a strong influence on the thicker layer and its effects (internal bubbles and flaws) on strength, as previously assigned.

In terms of surface characteristics, the brush glaze application (Br group) showed lower roughness (Ra and Rz parameter) compared to the glaze spray application. Chun et al. (2017) showed that glaze spray accumulates on certain regions of the material’s surface. This fact can explain the higher surface roughness for this group (Figure 3; Table 3). Therefore, the glaze layer in the spray technique is thinner than the brush method (Figure 2), and so the glaze follows the irregularities generated by grinding. Surface roughness was consequently higher in the Sp group than in the Br group.

X-ray diffraction (XRD) of the Y-TZP surface with glaze was not performed, because the glaze presence makes this analysis difficult, since the glaze layer (83.68 - 355.51 µm) on the Y-TZP surface is thicker than the depth that the X-ray can reach, thus biasing the phase transformation analysis of only Y-TZP (Pozzobon et al., 2017; Feitosa et al., 2018). However, in this study, additional specimens were manufactured simulating only the glaze firing cycle, i.e., without glaze application. X-ray diffraction analyses (Table 3) showed that the glaze firing cycle led to (m-t) reversal phase transformation regardless the cycle (for brush or spray) used. Even so, we emphasize that the absence of application of the glaze material can be considered as a limitation of such analysis, as the glaze's presence may change this phase analysis.

We can also include the following as further limitations: the fatigue test was not performed under conditions that reproduce the oral environment (surfaces with no milling, temperature and pH changes, sliding motion, and cementation set-up), and optical properties that might be influenced by glaze were not accessed.

5. Conclusions

- The application of a thin layer of porcelain glaze, either by brush or the spray technique, did not impair the fatigue strength of a translucent Y-TZP ceramic.
- In terms of surface characteristics (topography and roughness), glaze application by brush made the surface more homogeneous and smoother than the spray application.

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TABLES

Table 1. Description of materials, their composition, manufacturers and batch number.

Materials	Composition	Manufacturers	Batch number
Yttrium-stabilized tetragonal zirconia polycrystal ceramic	90.4 - 94.5wt% ZrO_2; 4-6 wt% Y_2O_3; 1.5 - 2.5 wt% HfO_2; 0 - 0.3 wt% Al_2O_3; 0 - 0.5 wt% Er_2O_3; 0 - 0.3 wt% Fe_2O_3	Vita YZ-HT Vita Zahnfabrik, Bad Säckingen, Germany	40260
Diamond bur #3101G – grit size 181 μm	Diamond and stainless steel– cylindrical shape with grit size 181 μm	KG Sorensen, Cotia, Brazil	14763
Glassy-based material applied by brush	Body stains - special low fusing glaze material to create a silky matte and sealed surface	Vita Akzent, Vita Zahnfabrik	23750 powder 22601 liquid
Glassy-based material applied by spray		Vita Akzent Plus, Vita Zahnfabrik	E33820

Table 2. Study design.

Groups	Surface treatment	Analysis executed (sample size – n)
Ctrl	Without any treatment – "as-sintered"	Surface topography (n=2) Roughness analyses (n=15) XRD analysis (n=1) Monotonic static biaxial flexural test (n=3) Fatigue test (staircase method, n = 15) Fractography analysis
Gr	Grinding with coarse diamond bur	
Br	Application of glaze powder/liquid	
Sp	Application of glaze spray	
Gr+Br	Grinding with coarse diamond bur + application of glaze powder/liquid	
Gr+Sp	Grinding with coarse diamond bur + application of glaze spray	

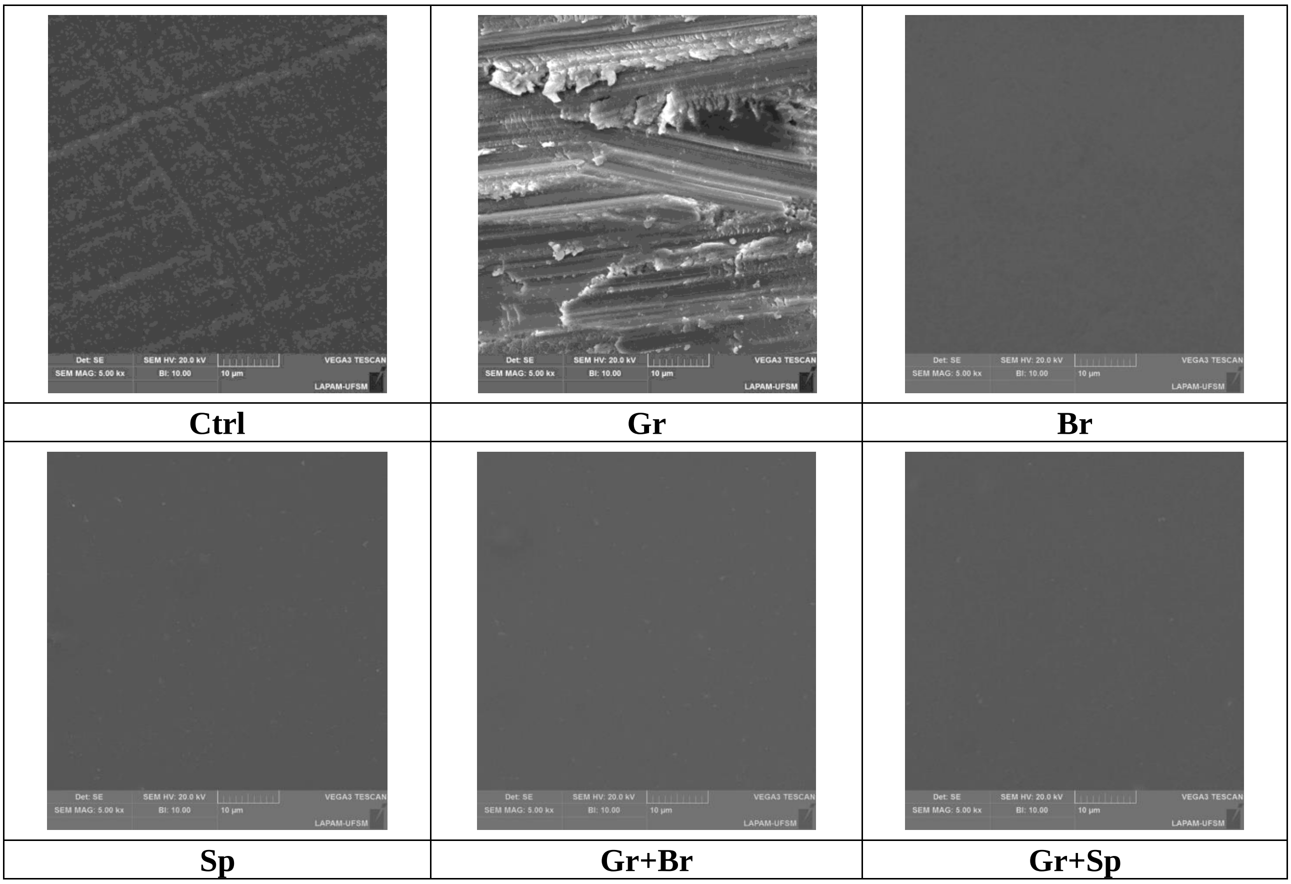
Table 3. Mean of monotonic biaxial strength test (MPa) and standard deviation (SD), initial strength for fatigue test (60% of the monotonic strength), step size (5% of the initial strength), fatigue strength (σf) and respective 95% confidence interval (CI), roughness (Ra and Rz parameters in micrometers) (and SD) and XRD analysis results are showed.

Groups	**Mean monotonic strength test (MPa)**	**Initial strength for fatigue test (MPa)**	**Step size (MPa)**	**Fatigue Strength (MPa)**		**Roughness (μm)**		**XRD Analysis**
				(σf)	**IC (95%)**	**Ra**	**Rz**	
Ctrl	878.53 (114.58)	527.11	26.35	625.49 ^{bc}	612.59-638.38	0.266 (0.06)^{d}	2.201 (2.23)^{c}	0
Gr	1038.35 (45.38)	596.41	29.82	650.08 ^{ab}	624.20-675.97	1.033 (0.18)^{a}	6.474 (1.21)^{a}	13.0
Br	829.07 (125.42)	497.44	24.87	633.39 ^{bc}	612.66-654.13	0.537 (0.07)^{c}	3.607 (0.68)^{b}	0
Sp	874.62 (14.62)	524.77	26.23	673.40 ^{a}	655.47-691.33	0.828 (0.29)^{b}	5.390 (1.90)^{a}	0
Gr+Br	960.02 (208.52)	576.01	28.80	612.49 ^{c}	590.36-634.61	0.615 (0.17)^{c}	3.805 (1.06)^{b}	6.81
Gr+Sp	787.35 (98.65)	472.41	23.62	623.57 ^{bc}	612.74-634.40	1.159 (0.42)^{a}	7.463 (2.51)^{a}	8.13

* Different letters on each column indicate statistical differences depicted by Two-way ANOVA and post-hoc Tukey test for fatigue data and roughness.

FIGURES

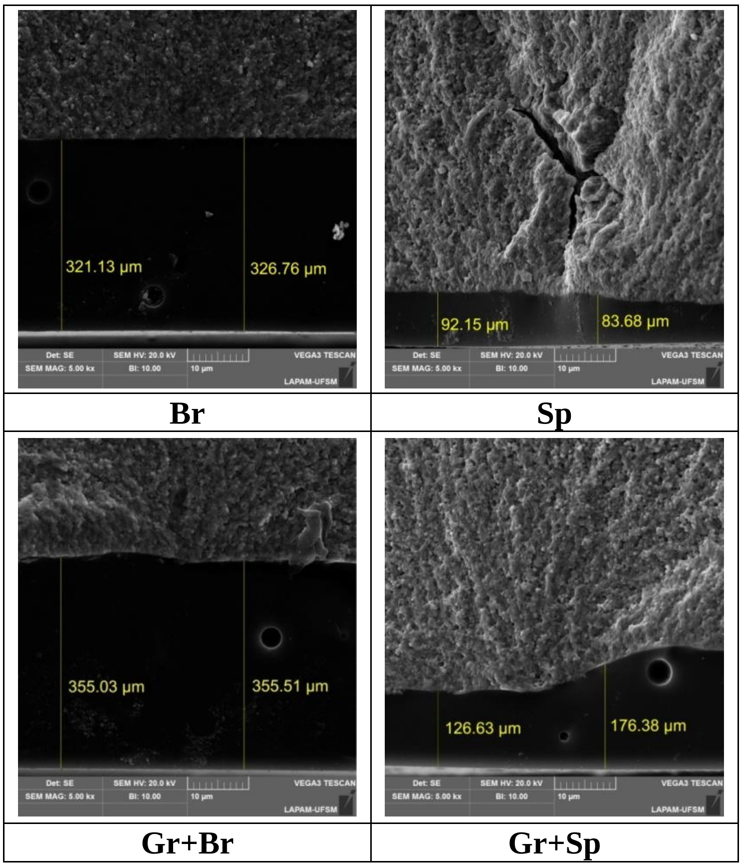
Figure 1. Representative SEM images (5000× magnification) of Y-TZP surface. It shows that grinding (Gr) introduces scratches in the ceramic surface and that the application of a glaze material regardless of technique (brush or spray) produces smoother surfaces.



Ctrl | Gr | Br

Sp | Gr+Br | Gr+Sp

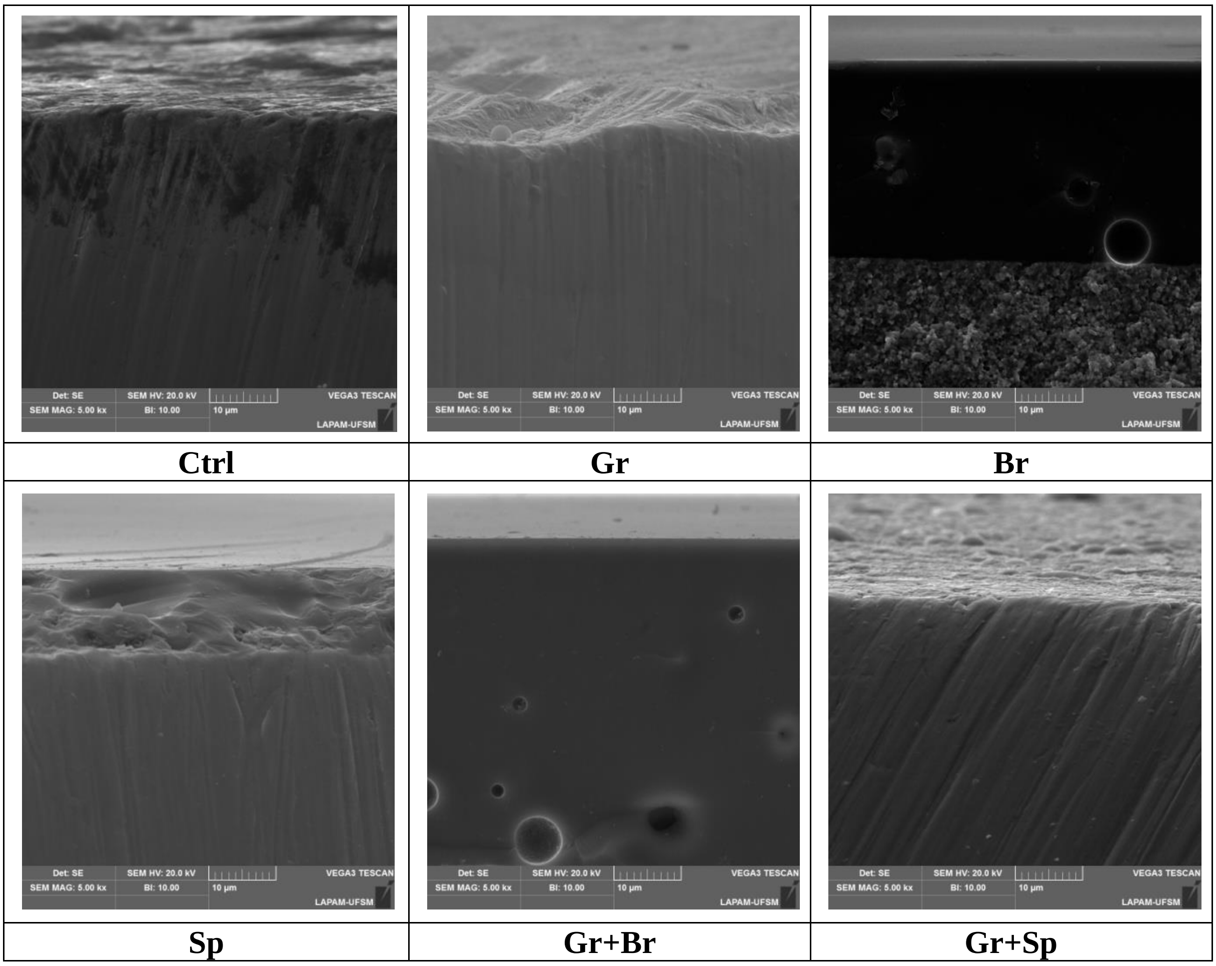
Figure 2. Representative SEM images (5000× magnification) showing that glaze application by brush technique presents higher glaze layer than spray technique.



Br **Sp**

Gr+Br **Gr+Sp**

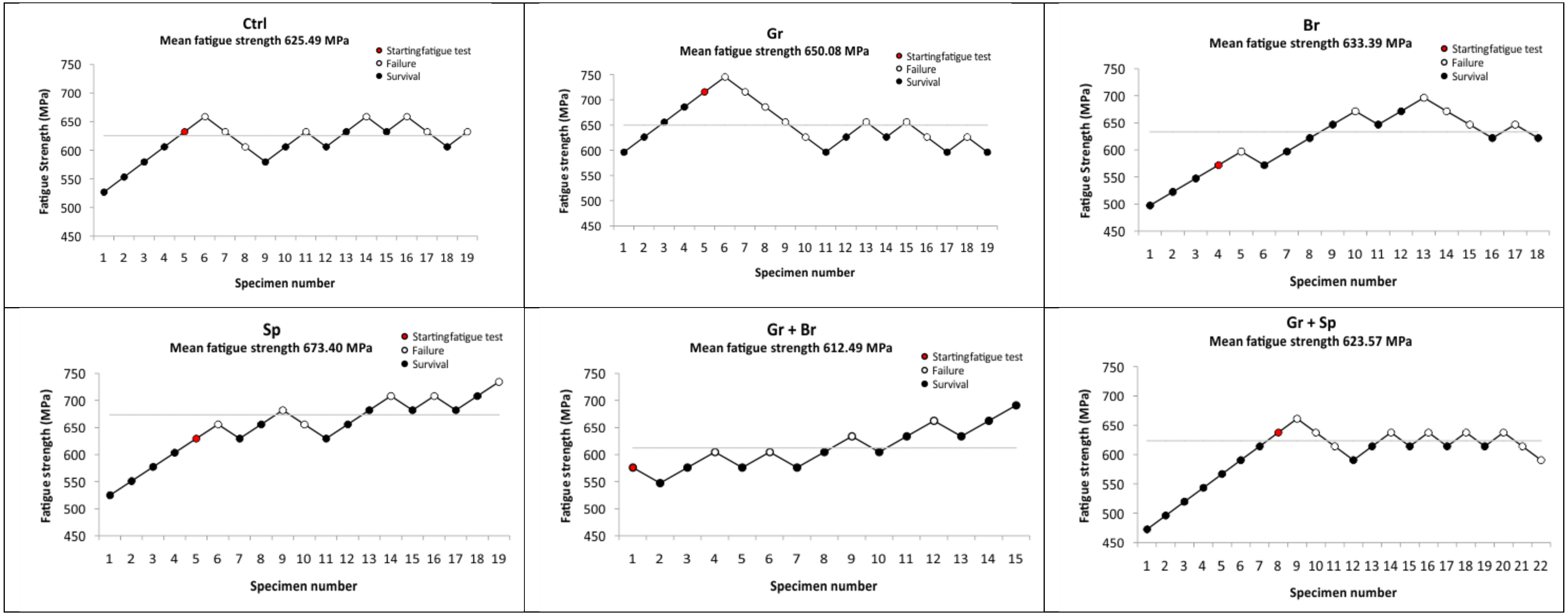
Figure 3. Representative SEM images (5000× magnification) of the cross-section region for all conditions evaluated, showing the presence of surface defects induced by processing on Ctrl group, or by grinding on Gr groups, at groups that received the Br technique, it notices the presence of bubbles inside the glaze layer.



Ctrl **Gr** **Br**

Sp **Gr+Br** **Gr+Sp**

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



Ctrl
Mean fatigue strength 625.49 MPa

Gr
Mean fatigue strength 650.08 MPa

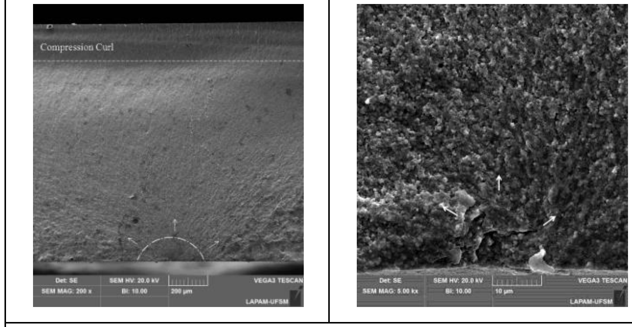
Br
Mean fatigue strength 633.39 MPa

Sp
Mean fatigue strength 673.40 MPa

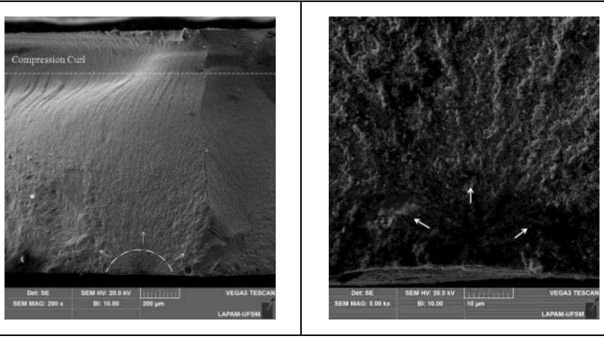
Gr + Br
Mean fatigue strength 612.49 MPa

Gr + Sp
Mean fatigue strength 623.57 MPa

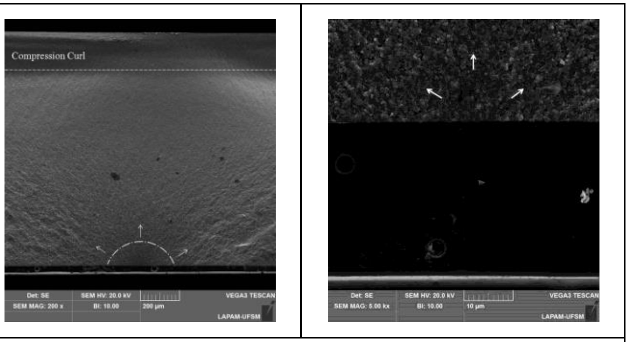
Figure 5. Representative images of the fracture surface for each condition evaluated under SEM (magnification: 200× - left and 5000× - right). It notices that the fractures start (region under the white half-circle) always at the side subjected to tensile stresses during fatigue test (opposite to the load application) on a superficial or sub-superficial defect for Ctrl, Gr and Sp groups, whereas in Br, Gr+Br and Gr+Sp groups, it originates at the interface between Y-TZP and glaze layer. All the cracks propagate (direction pointed by white arrows) to the opposite site where compression stresses are concentrated during the test (compression curl region).



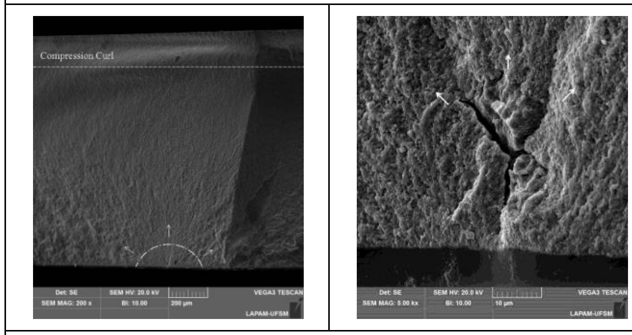
Ctrl



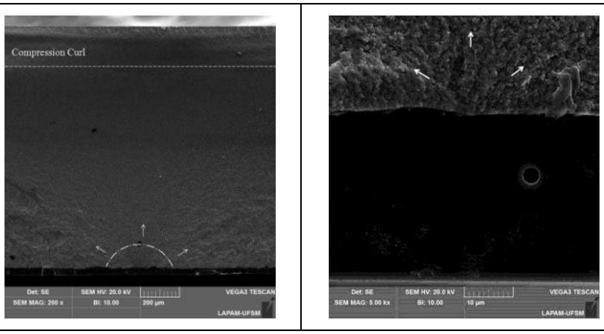
Gr



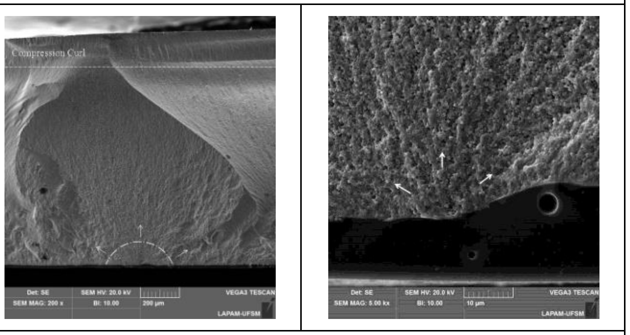
Br



Sp



Gr+Br



Gr+Sp

5. ARTIGO 4- Grinding, polishing and glazing of the occlusal surface do not affect the load-bearing capacity under fatigue and survival rates of bonded monolithic fully-stabilized zirconia simplified restorations

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Running Title: Surface treatments and fatigue of FSZ ceramic.

Abstract

This study aims to evaluate the effect of distinct surface treatments (grinding, polishing and glaze) of the occlusal surface of fully-stabilized zirconia (FSZ) simplified restorations bonded onto epoxy resin on the surface characteristics and fatigue behavior of the restorations. Disc shaped specimens of FSZ (IPS e.max Zircad MT Multi) were produced (Ø = 10 mm and 0.8 mm in thickness) and randomly allocated into 5 groups, considering the factor 'surface treatment' of the occlusal surface: Ctrl – as-sintered; Gr – ground with coarse diamond bur; Gr + Pol– grinding + polishing with two-step polishing system; Gr + Gl – grinding + glaze application; Gr + Pol + Gl – grinding + polishing + glaze application. Next, the FSZ intaglio surface was air-abraded with 45 μm aluminum oxide powder for 10 s at 15 mm of distance under 2 bar pressure and the discs were adhesively cemented (Multilink Automix) onto its dentin analogue pair (Ø = 10 mm; thickness = 2.7 mm). Finally, the step-stress fatigue test was executed (load ranging from 200 to 1300 N; step-size of 100N; 10,000 cycles per step, 20 Hz). In addition, surface topography, roughness, phase transformation and fractography analyses were performed. Grinding altered the topographical pattern introducing defects into the material surface and increasing roughness. Polishing and glaze application led to a smoothening effect, reducing surface defects and statistically decreasing roughness. However, the effect on roughness of polishing and glaze was statistically similar. No phase transformation was observed, thus only cubic and tetragonal phases were detected. No surface treatment had a deleterious effect regarding the fatigue failure load, number of cycles for failure and survival rates. All failures (cracks) started on the bonding surface. Thus, polishing and glaze are indicated to reduce surface roughness, despite not leading to differences in terms of fatigue performance.

Keywords: Fatigue phenomena. Finishing. Polishing. Yttrium-stabilized zirconia. Full-contour restorations.

HIGHLIGHTS

- Grinding introduces surface defects and increases roughness of FSZ ceramics.
- Polishing and glaze lead to the surface smoothening effect (decrease defects and reduce roughness).
- No phase transformation was observed (only cubic and tetragonal phases detected).
- No deleterious effect on the mechanical fatigue performance was observed.

- All cracks started on the bonding surface, regardless of the surface treatment performed on the occlusal surface.

1. Introduction

Recently, the use of a new class of ceramic material, namely 3rd generation zirconia (fully-stabilized zirconia – FSZ), has been proposed for use in restorative dentistry (Stawarczyk et al., 2017). The 3rd generation zirconia presents an increase of yttrium oxide stabilizer content (>5%mol yttrium) (Stawarczyk et al., 2017; Zhang and Lawn, 2018) in comparison to second-generation materials (≈3mol% yttrium), which were partially stabilized ceramics (Y-TZP, yttrium tetragonal zirconia polycrystal) (Stawarczyk et al., 2017). This increase in the amount of oxide stabilizer leads to a higher amount of cubic phase (up to 53%) in their microstructure (Pereira et al., 2018) which enhances the optical properties (Zhang and Lawn, 2018), as cubic crystals allow superior light transmission through the material structure (Stawarczyk et al., 2017).

However, 3rd generation zirconia has been showing inferior mechanical performance in comparison to the 2nd generation zirconia (Sulaiman et al., 2017; Pereira et al., 2018; Zucuni et al., 2019[a]) owing to the absence of the t-m phase transformation mechanism in response to stimuli which would actuate in promoting compression stress concentration around existing defects, preventing subcritical crack growth and consequently crack propagation by defect coalescence, as is already known for 2nd generation zirconia (Stawarczyk et al., 2017; Inokoshi et al., 2018). Thus, the mechanical properties of FSZ may be more influenced by the presence of defects introduced during manufacturing or processing (milling or adjustments executed prior to cementation of the restoration) (Sulaiman et al., 2017).

Clinical adjustments are commonly executed to improve marginal adaptation and occlusal/proximal contact of fixed dental prostheses through grinding with a diamond bur (Preis et al., 2012; Hmaidouch et al., 2014; Mohammadi- Bassir et al., 2017). This procedure may introduce microcracks in the material and increase the surface roughness (Guilardi et al., 2017), which is undesirable because it may predispose the restoration/teeth to plaque formation (Dutra et al., 2018), negatively interfere in aesthetic properties and decrease the long-term success of the rehabilitation (Lee et al., 2016[a]). In this sense, alternatives to counteract the effects of grinding have been indicated, such as glaze application and/or finishing/polishing (Preis et al., 2015; Chun et al., 2017). With regards to glaze application, it can be executed by using a powder material or a spray, where the spray technique results in a thinner and more homogenous layer (fewer internal air bubbles) (Chun et al., 2017; Pozzobon et al., 2017), which may lead to enhanced mechanical properties (Chun et al., 2017). In terms of polishing systems, Zucuni et al., 2019[b] demonstrated that those specifically indicated for zirconia have superior performance for roughness, topography, mechanical and fatigue improvements than the universal polishing systems.

Despite this, existing studies until now have usually evaluated the effects of surface treatments on the mechanical performance of ceramics following the strict guidelines of ISO 6872- 2015 for biaxial flexure test, which demands that the treated surface is positioned to the down-side of the testing assembly where tensile stresses concentrate, meaning that the fracture will be triggered on defects presented in such regions (Chun et al., 2017; Guilardi et al., 2017; Pozzobon et al., 2017; Zucuni et al., 2018; Borba et al., 2011). However, when taking into account the clinical scenario, the polishing/glazing will be executed on the occlusal surface of the restoration, and this surface will be mainly subjected to the compression stress concentration during masticatory stimuli (Bramanti et al., 2017; Monteiro et al., 2018). Hence, studies mimicking such a scenario to understand the influence of surface treatments of the occlusal surface on the mechanical performance of these bonded restorations are still extremely demanded.

Moreover, it is important to emphasize that the failure of ceramic restorations has usually been shown to originate from the cementing/intaglio surface (Kelly, 1999; Prochnow et al., 2018; Venturini et al., 2018), and it has also already been shown that adhesive cementation promotes a reinforcement (increase in mechanical properties) by enhancing the stress distribution throughout the system (Addison et al., 2007). In addition, in clinical scenario, the literature has also demonstrated that the failure may start at the cervical margin (propagating from the cementation interface to occlusal surface) (Oilo et al., 2013). Thus, the issue/question is: can the surface treatments performed on the occlusal surface of 3rd generation zirconia influence the fatigue performance of bonded FSZ restorations?

Finally, based on the aforementioned presuppositions, this study aimed to evaluate the effect of surface treatments (grinding, polishing, glazing) on the fatigue failure load, number of cycles for failure, survival probabilities and surface characteristics of FSZ simplified restorations adhesively cemented on a dentin analogue substrate. The assumed null hypothesis was that the different surface treatment would not influence the fatigue outcomes.

2. Material and Methods

The materials used in this study with their respective composition, manufacturers and batch number are described in Table 1.

2.1 Production of specimens

A simplified tri-layer (zirconia-cement resin-epoxy disc) assembly was used to simulate a molar restoration with a final thickness of 3.5 mm after cementation, because this thickness represents the average distance from the pulp wall to the occlusal surface (Chen et al., 2014).

2.1.1 Manufacturing of zirconia discs

The ceramic discs were manufactured by using the previously performed methodology of Pereira et al. (2014). Basically, FSZ discs (IPS e.max Zircad MT Multi) (98.5 mm in diameter and 20 mm in thickness) were manually cut with diamond discs coupled to an electric motor (W&H Perfecta 300, W&H Dentalwerk, Bürmoos, Austria) into blocks of 20 mm × 20 mm. Metal rings (Ø= 12 mm) were attached at both side of the blocks and a cylinder was obtained through grinding with silica carbide papers (600–1200 grit) in a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA). Afterwards, discs (slices) of 1.15 mm in thickness were cut in a cutting machine (ISOMET 1000, Buehler) with diamond blades under water-cooling. The discs were polished with silica carbide paper (600 and 1200 grit) until 1.0 mm in thickness, and then sintered (1550 °C for 2 h) in a specific furnace (Zyrcomat 6000MS, Vita Zahnfabrik). All the specimens were subsequently inspected with a digital caliper (Mitutoyo Series 209 Caliper Gauge, Takatsu-ku, Kawasaki, Kanagawa, Japan) to make sure that the dimensions were standardized, which must be of 0.8 mm thickness and 10 mm diameter. All samples were randomly allocated into 5 groups (n = 15) considering the study factor ‘surface treatments’.

2.1.1.1 Surface Treatments

All the surface treatments were executed by a single trained operator (CPZ).

Control – Ctrl

Samples did not receive any surface treatment after sintering (as-sintered condition).

Grinding – Gr

To increase the reproducibility and standardization of this procedure, the specimens were attached to a metal base maintaining the specimen surface parallel to the diamond bur. The occlusal (top side) surface of the zirconia specimen was totally marked with a permanent pen (Pilot, São Paulo, Brazil) and then grinding was executed until total removal of the marking. To do so, a coarse diamond bur (#3101G–grit-size 181 µm; KG Sorensen) was coupled to a speed multiplier handpiece (T3 Line E 200 contra-angle handpiece up to 170,000 rpm, Sirona, Bensheim, Germany) attached to a low-speed motor (Kavo Dental, Biberach, Germany). The grinding was executed with oscillatory movement and under constant water-cooling (≈30 mL/min). Moreover, one diamond bur was used for each specimen, according to a methodology previously used by Guilardi et al. (2017).

Polishing – Pol

The *Eve Diacera (Eve Ernst Vetter*) polishing system was used. This system consists of 2 tips (salmon – fine, and green – extra-fine) which were coupled to an electric motor (W&H Perfecta 300, W&H Dentalwerk, Bürmoos, Austria) which allowed speed control (7,000-12,000 rpm) and the polishing was executed as recommended by the manufacturer, following a similar approach described for grinding in which the samples were attached to the metal base maintaining the specimen surface parallel to the polishing tip, and the occlusal surface was polished by 25 s for each specific tip with oscillatory movement (Zucuni et al., 2019[b]).

Glaze – Gl

Previous to the application, the glaze bottle (Vita Akzent Plus, Vita Zahnfabrik) was shaken for about 1 min aiming to mix the material, and the glaze spray was applied according to manufacturer's recommendations: distance of 15 mm between ceramic surface and applicator's tip until completely covering the ceramic surface. Next, glaze firing was performed following the manufacturer's instructions (Vacumat 6000MP furnace, Vita Zahnfabrik): Initial temperature: 500 °C, maintaining it for 4 min for pre-drying, heating rate of 80 °C/min until the final temperature of 950 °C, maintaining it for 1 min.

Polishing plus Glaze – Pol+Gl

The polishing and glazing procedures were performed sequentially, as previously mentioned.

2.1.2 Manufacturing the dentin analogue substrate

Epoxy resin plates (Epoxy Plate 150Plate 150 × 350 × 3 mm, Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany) fiber-glass reinforced were cut in disc shape using a diamond drill (Diamant Boart, Brussels, Belgium) and polished with silica carbide paper (600 grit) until 2.7 mm in thickness under water cooling. Thus, the epoxy discs presented final dimensions of 10 mm diameter and 2.7 mm in thickness.

2.2 Cementation Procedures

Zirconia and epoxy resin discs were cleaned in an ultrasonic bath (1440 D – Odontobras, Ribeirao Preto, Brazil) with 78% isopropyl alcohol for 5 min. The intaglio surface of the zirconia discs was air-abraded with 45 µm aluminum oxide powder (Polidental, Cotia, Sao

Paulo, Brazil) for 10 s at 15 mm of distance under 2 bar of pressure (Aurélio et al., 2016). The silane agent (Monobond Plus, Ivoclar Vivadent, Schaan, Liechtenstein) was then actively applied for 15s, kept to react for 45s, and then gently dried with air spray. Meanwhile, the epoxy resin discs were etched with 10% hydrofluoric acid (CondacPorcelana, FGM, Joinville, Brazil) for 60 s, rinsed for 30s and cleaned in an ultrasonic bath with distilled water for 5 min. Next Multilink Primers A and B (Ivoclar Vivadent) were mixed in a 1:1 ratio, applied with a microbrush on the surfaces (30 s), and gently air-dried until obtaining a thin layer.

Then, the pairs of zirconia/resin epoxy discs were adhesively cemented with a resin cement (Multilink Automix, Ivoclar Vivadent) according to the manufacturer's instructions. The assembly was kept under a constant load of 2.5 N applied in the ceramic surface. The resin cement excesses were removed and light-activation was performed (Radii-cal, SDI, Bayswater, Australia) for 20 s on each side (0°, 90°, 180°, 270°, and occlusal surface). The specimens were stored in distilled water at 37°C (between 2 and 7 days) for the fatigue tests to be performed.

2.3 Roughness analyses

All the specimens from each group (n = 15) were subjected to surface roughness analyses. The measurement was executed using a profilometer (Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan) using the Ra and Rz parameters, which are recommended by ISO 4287-1997, in which Ra is the arithmetical mean of the absolute values of peaks and valleys (mm), and Rz is the average distance between the five highest peaks and five major valleys (mm). For this, three measurements per specimen were carried out with a cut-off (n= 5) of λC 0.8 mm and λS 2.5 µm. After, the mean average value was calculated for each specimen, and then the statistical analysis was carried out.

2.4 Surface topography

One specimen per group was cleaned in an ultrasonic bath (1440 D–Odontobras, Ribeirao Preto, Brazil) with 78% isopropyl alcohol for 5 min. After, the treated surface was gold-sputtered and analyzed by Scanning Electron Microscopy (Se – secondary electron detector, 20Kv; SEM - Vega3, Tescan, Czech Republic) under 100× and 5000× magnifications to assess the surface characteristics. In addition, representative specimens in the glaze group were subjected to SEM (1000× magnification) to evaluate the layer glaze thickness (cross-sectional imaging).

2.5 Fatigue tests

The specimens (n = 15) were tested by the step-stress test method (Collins, 1993; Kelly et al., 2017) using an electric machine (Instron Electro Puls E3000, Instron Corp, Norwood, United States). The load was applied by 40-mm diameter stainless-steel hemispheric piston in the center of the specimens (Kelly et al., 1999). An adhesive tape was placed on the occlusal surface of the ceramic to homogeneously distribute the stress during the test and a polyethylene sheet (0.1 mm thick) was placed between the piston and the specimen to avoid the occurrence of Hertzian cone cracks (Monteiro et al., 2018; Prochnow et al., 2018). Cyclic loads ranging from 10 N up to the maximum load in each step were applied with a frequency of 20 Hz. The specimens were then immersed in water during the fatigue test. An initial maximum load of 100 N was applied for 5,000 cycles to adjust the piston/specimen contact; next, incremental loads of 100 N (step-size) were added to the previously applied load until failure of the specimen (radial crack) with a lifetime of 10,000 cycles/step. The presence/absence of cracks was checked at the end of each step by transillumination (Dibner & Kelly, 2016).

2.6 Fractographic analysis

All specimens were analyzed by light oblique transillumination to identify the occurrence of cracks (radial cracks). Then, one specimen of each evaluated condition was selected and a half (ceramic fragment) was detached of the assembly. The other half of ceramic fragment remained bonded on the epoxy resin. Subsequently, the specimens were ultrasonically cleaned in 78% isopropyl alcohol for 15 min, gold sputtered, and analyzed under scanning electron microscopy (Vega3, Tescan, Czech Republic) at 200× and 3000× magnification (ceramic fragment) and at 1000× magnification (ceramic fragment cemented) to determine fracture origin and fractographic characteristics.

2.7 X-ray Diffraction (XRD analysis)

To identify the crystalline phase content (*m*-, *t*-, and *c*-phases) after surface treatments, one additional specimen of each evaluated condition was analyzed in an X-ray Diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) with CuKα radiation (40kV, 40mA) at a 2θ angular interval of 27 to 76° with a step-size of 0.01° every 3 s, according to a previously-described methodology (Inokoshi et al., 2015; Pereira et al., 2018). Only the firing cycle was performed (without the application of the glaze material) in the Gr+Gl and Gr+Pol+Gl group.

2.8 Data analysis

Statistical analyses were executed by SPSS statistical software (SPSS version 21, IBM, Chicago, IL, USA). The fatigue failure load (N) and the number of cycles for failure data were submitted to survival analysis by Kaplan Meier and Mantel-Cox post-hoc tests (α= 0.05) and after to Weibull analysis (Super SMITH Weibull) to access the structural mechanical reliability of each group (Weibull modulus). Additionally, the survival rates on each load and number of cycles steps were obtained. Finally, roughness data (Ra and Rz parameters) assumed a parametric distribution (Shapiro Wilk test), thus one-way ANOVA and post-hoc Tukey were applied (α= 0.05).

3. Results

The SEM analysis (Figure 1) shows that grinding alters the topographical pattern generating scratches and defects on the material's surface. Otherwise, polishing and glaze show surface smoothening effects. This performance was corroborated by the roughness measurements (Table 2).

None of the surface treatments affected the fatigue failure load, number of cycles for failure and survival rates (Table 2 and 3), and no statistical difference for Weibull modulus could be detected (Figure 2 and Table 2). All cracks started from the cementation surface (radial cracks) (Figure 3 and 4). Figure 5 demonstrates that the glaze layer thickness ranges are between 19.73 and 80.82 µm, and the presence of bubbles inside this layer become clear.

XRD data show that the surface treatments did not trigger phase transformation, thus only cubic (peak 400) and tetragonal phases were detected (characteristic pattern of a 3rd generation zirconia – FSZ) (Figure 6).

4. Discussion

The null hypothesis was accepted since the surface treatments had no effect on the fatigue behavior, which could have probably been the result of the failure (cracks) of ceramic restoration originating at the intaglio surface (bonding surface) (Figure 3 and 4), as also demonstrated by the literature (Kelly, 1999; Kelly et al., 2010; Venturini et al., 2018; Monteiro et al., 2018). The failure occurred in that zone, even though surface treatments were executed on the occlusal surface and distinct roughness patterns and topographical characteristics were noticed.

The characteristics of the intaglio surface of ceramic restorations seem to play an important role in their performance (Pagniano et al., 2005). The presence of defects at the bonding surface may act as a stress concentration factor and lead to failure/fracture of the material (Rodrigues et al., 2018). These defects or cracks are subject to the sub-critical crack growth mechanisms during intermittent cyclic load application in a wet environment (similar to the oral environment) until crown ceramic failure (Kelly, 1999; May et al., 2015). This process is responsible for decreasing the strength of ceramic materials (Anusavice and Hojjatie, 1992).

In this sense, using finite element analysis Monteiro et al. (2018) demonstrated that the tensile stress concentration during the mechanical loading executed in the fatigue test of a glass-ceramic bonded to the dentin analogue material (fiber/epoxy resin material) is mainly concentrated at the intaglio surface of the restoration, the luting material and the substrate (high tensile stresses) (Kelly, 1990), which means on the side opposite to the load application, while the outer surface (occlusal zone) receives compressive stresses (contact with the piston). These findings help to explain our results, as the defects introduced by the surface treatments of the occlusal surface were far from the region with higher tensile stresses (bonding surface) from which the failure/cracks started. Pereira et al. (2019) also confirm these findings. However, as demonstrated by these authors, the fracture origin of FSZs was only on the occlusal surface when the zirconia was bonded into a substrate with high elastic modulus (zirconia).

The literature (Oilo et al., 2013, 2014) has demonstrated that clinically, the failure may start at the cervical margin (propagating from the cementation interface to occlusal surface). Thus, clinical adjustment at this region might induce deleterious effects on fatigue behavior of restorations on such scenario, which is not explored herein. In this sense, it is suggested that more studies are executed in order to evaluated this scenario.

Even if this current study found no effect of the surface treatments on the fatigue outcome, it must be highlighted that rough surface and inherent defects can generate a diffuse light reflection, modifying the optical properties of the material (Obregon et al., 1981; Kim et al., 2016). Moreover, it can induce more plaque accumulation and consequently increase the risk of caries and periodontal diseases (Lee et al., 2019), staining (Lee et al., 2016[b]) and wear of antagonist teeth/restoration (Preis et al., 2015). Thus, a polishing protocol has to be indicated to reduce the surface roughness generated by grinding (Preis et al., 2015; Zucuni et al., 2019[b]).

Still regarding this issue, glaze application is another surface treatment option, which has been recommended after ceramic grinding (Vichi et al., 2018; Carrabba et al., 2017; Bai et al., 2016). The application of glaze spray promoted a thinner and homogeneous layer, which did not generate any negative effect on the fatigue behavior (Chun et al., 2017). At the same time, glazing on the zirconia surfaces has been critically considered due to its incompatibility with zirconia materials (poor adhesion, thermal incompatibility) (Denry & Kelly, 2014; Longhini et al., 2016). The stress generated by differences in CTE (coefficient of thermal expansion) between the zirconia and glaze during the heating/cooling by sintering increases the probability of cracks and material failure (Gostemeyer et al., 2010). Thus, the use of glaze on the zirconia surface and its effects should be investigated overtime.

Regarding the XRD analysis (Figure 6), none of the evaluated surface treatments generated (t-m) phase transformation, with only tetragonal and cubic phases being found. This finding corroborates studies which evaluated the stability of phase transformation over time (Pereira et al., 2018; Zhang & Lawn, 2018). Thus, the structural stability (no phase transformation) of FSZs makes this material more resistant to the degradation mechanisms known as low-temperature degradation (LTD), which impaired the mechanical performance of the previous Y-TZP generations over time (Stawarczyk et al., 2017; Pereira et al., 2018; Zhang & Lawn, 2018).

In conclusion, our data corroborate that the existing post-processing treatments are necessary to reduce/eliminate the defects introduced by grinding, consequently promoting a smoothening effect of the ground zirconia surface. Moreover, no deleterious effect of the treatments (glazing or polishing or associated) on the fatigue performance could be detected. Thus, the clinical adjustment by grinding importantly do not affect the mechanical performance of the FSZ restoration, even though the ground surface has to be polished or receive a glaze application to make the surface smoother, thus preventing other potential negative effects (wear of antagonist teeth, or predisposal to plaque formation and accumulation).

Finally, it is important to highlight that this study presents inherent limitations such as the use of a simplified model to mimic posterior restorations (disc-disc set-up), and the absence of a complete simulation of oral environment stimuli such as cycling pH and temperature, or sliding movements during cyclic loading application, meaning that the current findings should be analyzed critically.

5. Conclusions

- The surface treatments (grinding, polishing, glaze) of the occlusal surface of zirconia restorations do not impair the fatigue behavior of the restorations.

- The FSZ surface subjected to grinding should be polished or glazed for smoothening effect, even though this has no effect on the fatigue behavior.

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TABLES

Table 1. Description of materials used in the study.

Materials	Composition	Manufactures	Batch number
FSZ ceramic	ZrO$_2$ (86.0 – 93.5%); Y$_2$O$_3$ (6.5< - ≤ 8.0 mol %); HfO$_2$ (≤ 5.0%); Al$_2$O$_3$ (≤ 1.0%); Other oxides (≤ 1.0%)	Zircad MT Multi, Ivoclar Vivadent, Schaan, Liechtenstein.	W82689
Diamond bur	Diamond (grit size 181 µm) and stainless steel	#3101G, KG Sorensen, Cotia, Brazil.	14763
Two-step polishing system	Green and salmon tips (coarser and finer grit-sizes, respectively), composed by diamond particles and specifically designed for zirconia polishing.	Eve Diacera, Eve Ernst Vetter, Keltern, Germany.	302410
Glaze (glassy-based material applied by spray)	Body stains - special low fusing glaze material to create a silky matte and sealed surface	Akzent Plus, Vita Zahnfabrik, Bad Sackingen, Germany.	A0764

Table 2. Results (means and respective 95% confidence intervals – IC, or standard deviations - SD) of Survival (Kaplan Meier and Mantel-Cox tests), Weibull analysis and surface roughness.

Groups	Survival analysis		Weibull analysis (Weibull modulus)		Roughness (µm)	
	Fatigue failure load (N) Mean (95% IC)	**Cycles until failure Mean (95% IC)**	**Fatigue failure load (N) Mean (95% CI)**	**Cycles until failure Mean (95% CI)**	**Ra Mean (SD)**	**Rz Mean (SD)**
Ctrl	826.6^a (746.7 – 906.6)	77,666^a (69,672 – 85,660)	8.27 (6.80 – 10.06)^a	7.78 (6.39 – 9.49)^a	0.93 (0.27)^b	4.19 (1.54)^c
Gr	846.6^a (783.6 – 909.7)	79,666^a (73,361 – 85,972)	7.76 (5.14 – 11.7)^a	7.27 (4.81 – 10.98)^a	1.26 (0.28)^a	7.72 (1.52)^a
Gr+Pol	913.3^a (808.8 – 1017.8)	86,333^a (75,880 – 96,786)	5.71 (3.96 – 8.23)^a	5.39 (3.73 – 7.77)^a	0.70 (0.18)^b	4.72 (1.15)^b
Gr+Gl	760.0^a (655.2 – 864.4)	71,000^a (60,558 – 81,441)	5.55 (4.07 – 7.58)^a	5.19 (3.80 – 7 10)^a	0.55 (0.28)^b	3.05 (1.15)^c
Gr+Pol+Gl	920.0^a (806.5 – 1033.4)	87,000^a (75,651 – 98,348)	4.72 (3.13 – 7.13)^a	4.44 (2.94 – 6.71)^a	0.79 (0.26)^b	5.44 (1.66)^b

*Different letters on each column indicate statistical differences for each outcome considered.

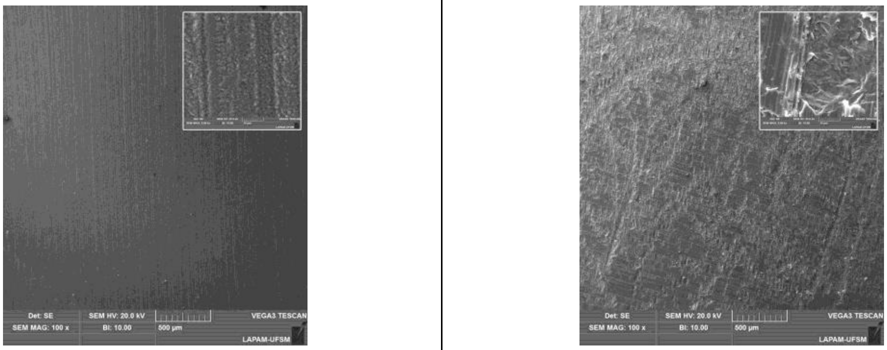
Table 3. Survival rates – specimens' probability to exceed the respective testing step for each group, with their respective standard error measurements.

Groups	Fatigue failure load (N)/ number of cycles until failure											
	200/ 15,000	**300/ 25,000**	**400/ 35,000**	**500/ 45,000**	**600/ 55,000**	**700/ 65,000**	**800/ 75,000**	**900/ 85,000**	**1000/ 95,000**	**1100/ 105,000**	**1200/ 115,00**	**1300/ 125,000**
Ctrl	1	1	1	1	1	0.60 (0.12)	0.40 (0.12)	0.06 (0.06)	0.06 (0.06)	0.06 (0.06)	0.06 (0.06)	0.0
Gr	1	1	1	1	0.93 (0.60)	0.80 (0.10)	0.46 (0,12)	0.26 (0.11)	0.0	-	-	-
Gr+Pol	1	1	1	1	1	0.60 (0.12)	0.60 (0.12)	0.46 (0.12)	0.26 (0.11)	0.13 (0.00)	0.06 (0.06)	0.0
Gr+Gl	1	1	1	1	0.46 (0.12)	0.40 (0.12)	0.33 (0.11)	0.26 (0.11)	0.06 (0.06)	0.06 (0.06)	0.0	-
Gr+Pol+Gl	1	1	1	1	0.80 (0.10)	0.80 (0.10)	0.53 (0.12)	0.53 (0.12)	0.26 (0.11)	0.26 (0.11)	0.0	-

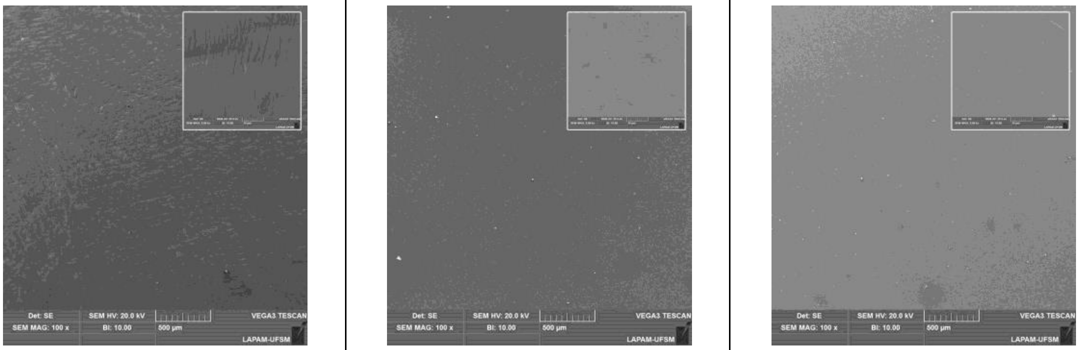
*the sign '-' indicates absence of specimen being tested on the respective step.

FIGURES

Figure 1. Representative SEM images at 100× (main image) and 5000× magnification (upper right corner) of the topography of the treated surfaces, showing that grinding processes introduced scratches and defects on ceramic surface, while the polishing and glaze generated smoother surfaces.



Ctrl **Gr**



Gr+Pol **Gr+Gl** **Gr+Pol+Gl**

Figure 2. Weibull plot for fatigue failure load (N) (left image) and number of cycles for failure (right image).

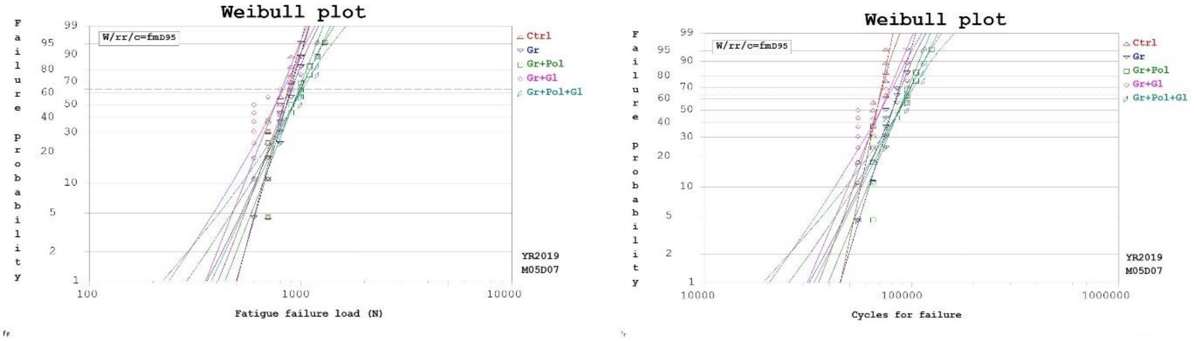
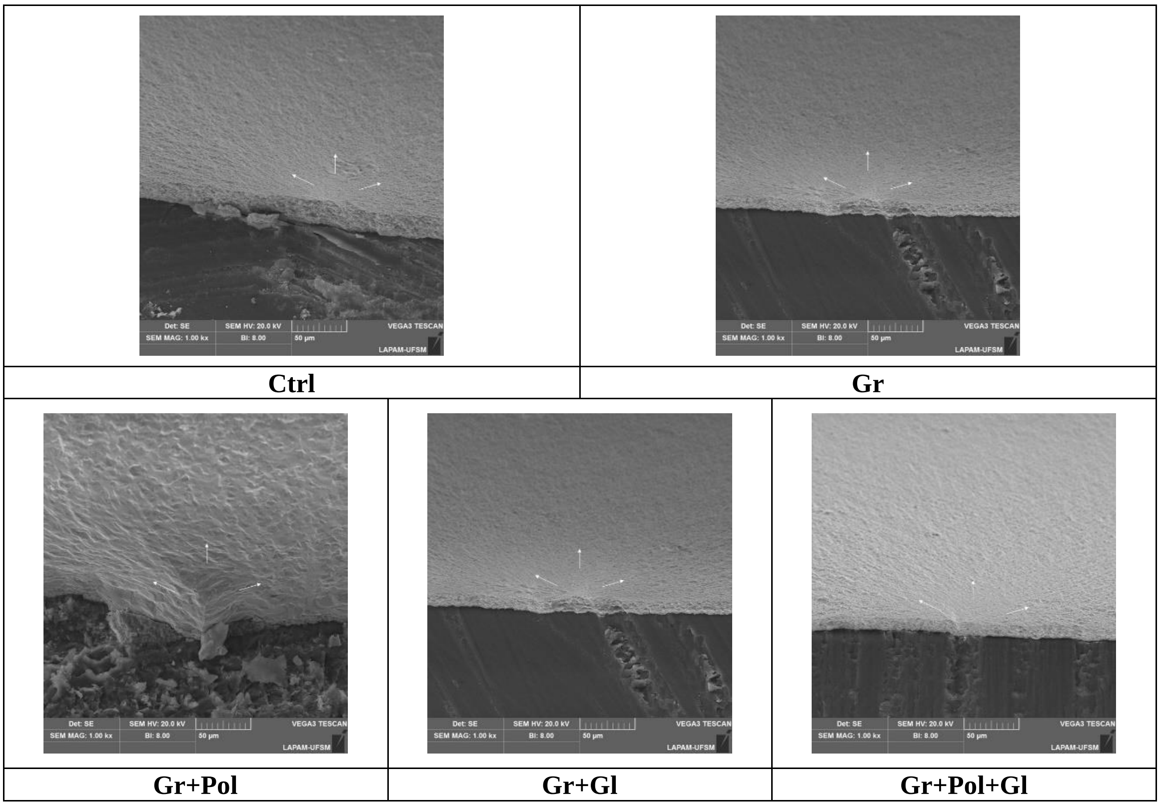


Figure 3. Representative fractography micrographs at 1000× magnification of the assembly (ceramic/resin cement/epoxy resin), showing that all failures start in the interface/bonding zone up to the occlusal surface.



Ctrl

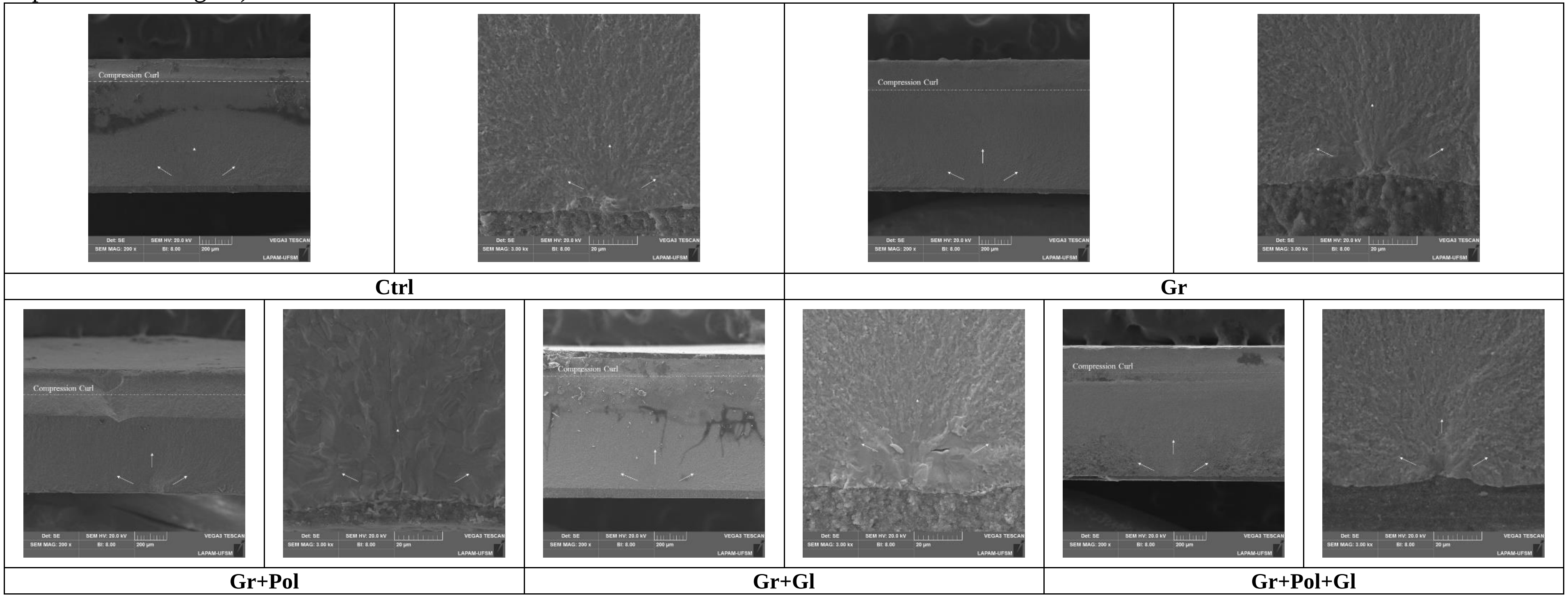
Gr

Gr+Pol

Gr+Gl

Gr+Pol+Gl

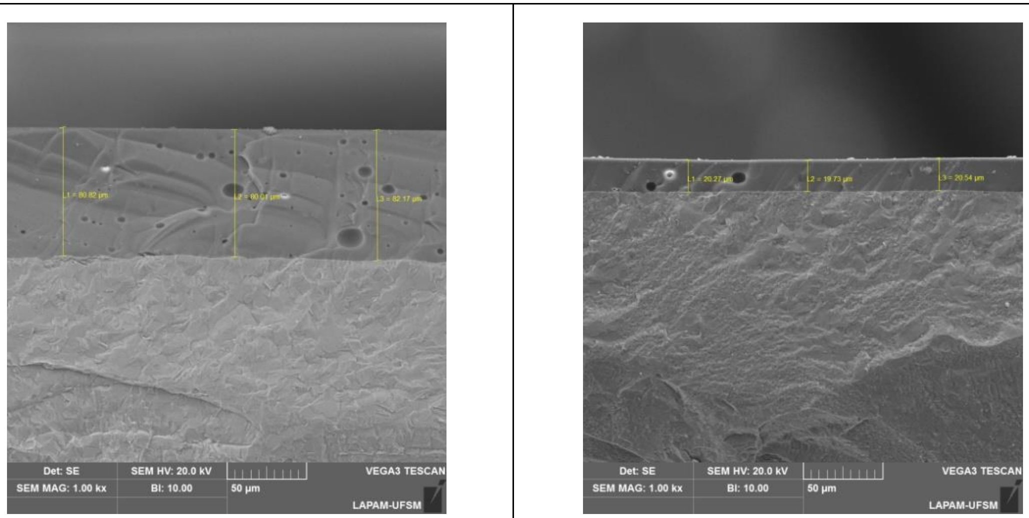
Figure 4. Representative images of the fracture surface for each debonded ceramic fragment from the assembly (ceramic/resin cement/epoxy resin) evaluated under SEM (magnification: 200× - left and 3000× - right). It can be noticed that the fractures start from the cementation surface on all observed specimens. The white arrows show the direction of cracks propagation towards the occlusal surface (region of compression stress concentration – compression curl region).



Ctrl **Gr**

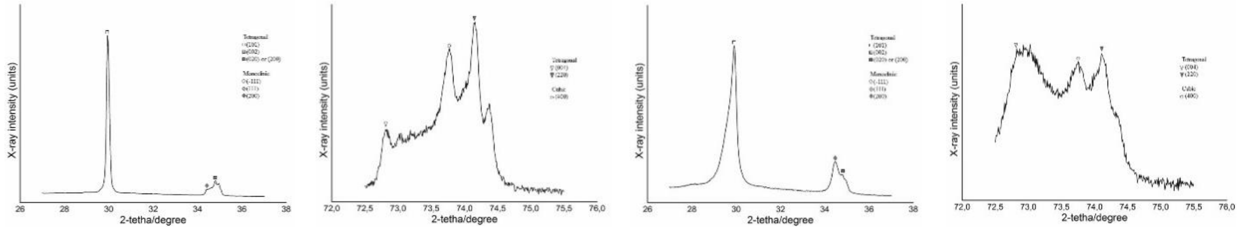
Gr+Pol **Gr+Gl** **Gr+Pol+Gl**

Figure 5. SEM images at 1000× magnification showing the glaze layer thickness ranging between 19.73 and 80.82 µm, and the existence of internal bubbles of varying size.

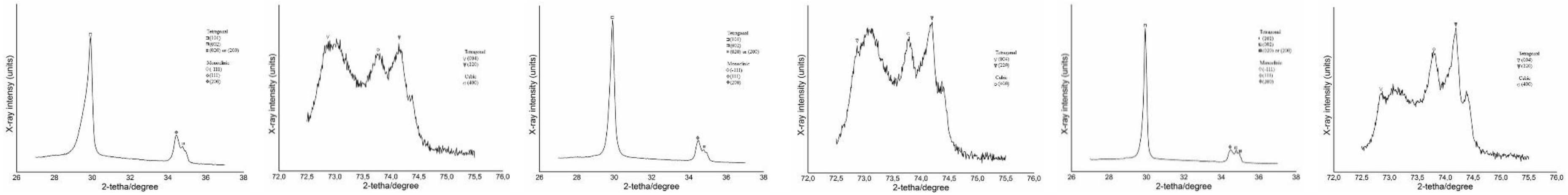


Gr+Gl **Gr+Pol+Gl**

Figure 6. XRD graphs of each evaluated condition showing the peaks of each crystallographic phase, where only tetragonal and cubic phase could be observed.



Ctrl **Gr**



Gr+Pol **Gr+Gl** **Gr+Pol+Gl**

6. DISCUSSÃO GERAL

Com base no estudo 1 desta tese, verificamos que zircônias de segunda geração apresentam melhor comportamento à fadiga que zircônias de terceira geração. Este achado é explicado pela natureza metaestável destas cerâmicas (zircônias de segunda geração) (PICONI & MACCAURO, 1999), ou seja, sofrem transformação de fase (t-m) quando submetidas à estímulos térmicos ou mecânicos (LUCAS *et al*., 2015). A transformação de fase forma uma camada de tensão compressiva em torno de trincas e defeitos, dificultando a propagação destes para o interior do material (DENRY & KELLY, 2014), otimizando a resistência mecânica da cerâmica. Por outro lado, as zircônias de terceira geração são totalmente estabilizadas na fase cúbica (c), e, por apresentarem alta concentração desta, não sofrem transformação de fase (INOKOSHI *et al*., 2018), portanto, não apresentam o mecanismo de tenacificação, de tal forma que, os defeitos introduzidos no material ficam livres para se propagarem quando submetidos à estímulos mecânicos (SULAIMAN *et al*., 2017, ZHANG & LAWN 2018), reduzindo a resistência mecânica do material.

Clinicamente, ajustes com pontas diamantadas são frequentemente realizados a fim de se obter melhor anatomia e relações oclusais/proximais das restaurações protéticas (PREIS *et al.,* 2015). Entretanto, o procedimento de desgaste introduz defeitos que poderão ser danosos ao material, reduzindo sua resistência mecânica e aumentando a rugosidade superficial (GUILARDI *et al*., 2017). Superfícies rugosas geram maior desgaste do dente/restauração antagonista, acúmulo de placa com consequente aumento na susceptibilidade de cáries recorrentes e doença periodontal (DUTRA et al., 2018), além de alterarem as características ópticas das cerâmicas (SARAC *et al*., 2006). Dessa forma, é imprescindível a realização de acabamento/polimento e/ou aplicação de glaze para reduzir os defeitos introduzidos pelo desgaste e tornar a superfície mais lisa (PREIS *et al.*, 2015).

Nesse sentido, o estudo 2 demonstrou que os sistemas de polimento indicados especificamente para zircônia são mais efetivos na redução da rugosidade superficial do que aqueles polidores para cerâmicas em geral. A zircônia é um material duro e difícil de ser polido (KOU *et al.,* 2006), assim, pontas polidoras diamantadas por apresentarem maior poder de corte, são mais indicadas para o polimento desta cerâmica (HUH *et al.,* 2016), enquanto que os polidores não específicos para zircônia podem ser compostos por partículas abrasivas com dureza insuficiente (PARK *et al.,* 2017), sendo ineficientes na redução da rugosidade, como constatado no estudo 2. Assim, quando polidores de uso geral forem utilizados, recomenda-se a realização prévia do acabamento com pontas diamantadas fina e extra-fina, a fim de se ter maior lisura superficial.

No que diz respeito à aplicação de glaze, o estudo 3 observou que a aplicação de glaze na forma spray teve efeito positivo no comportamento à fadiga quando comparado ao glaze aplicado na forma de suspensão pela mistura pó/líquido. O glaze na forma spray produz uma camada mais fina e com menos defeitos/bolhas que o glaze pó/líquido, o qual contrariamente induz a incorporação de muitas bolhas, as quais podem atuar como fator de concentração de tensão, afetando a resistência à fadiga (POZZOBON *et al.*, 2018). Além disso, verificou-se que a interface zircônia/glaze também pode influenciar no comportamento à fadiga do conjunto, visto que os defeitos introduzidos pelo desgaste prévio podem concentrar tensão quando o material é submetido à cargas cíclicas. Assim o risco da falha se originar nessa interface é maior (MECHOLSKY, 1995). Dessa forma, torna-se necessário a realização de um polimento previamente ao glazeamento a fim de se reduzir a rugosidade e os defeitos gerados pelo desgaste, e melhorar o comportamento à fadiga do material.

Interessantemente, como demonstrado no estudo 4, quando se avalia o efeito de tratamentos como desgaste, polimento e aplicação de glaze da superfície oclusal de discos de zirconia cimentados adesivamente em resina epóxi, não se observa efeitos negativos no comportamento à fadiga da cerâmica. Este resultado pode ser explicado majoritariamente pelo fato de as trincas terem se originado na superfície de cimentação da cerâmica (VENTURINI *et al.*, 2017; MONTEIRO *et al.*, 2018), e não na superfície oclusal tratada. Diante de carregamento cíclico mecânico, tensões de tração são induzidas e concentradas na superfície de cimentação, de tal forma que os defeitos presentes nesta superfície funcionam como iniciadores de trincas, que se propagam em direção a superfície externa (oclusal), levando à falha da restauração (KELLY, 1999; MAY *et al.*, 2015). Nossos achados estão de acordo com essas informações. Entretanto, mesmo que os tratamentos da superfície oclusal não tenham tido efeito sob a ótica do comportamento a fadiga, a realização de polimento após o desgaste da superfície cerâmica com pontas diamantadas se torna necessária, à medida que o desgaste gera uma alta rugosidade superficial sendo indesejável por aumentar o risco de doenças periodontais, recidiva de cáries e interferir negativamente nas propriedades ópticas e no comportamento mecânico do material (GUNOLOL *et al.*, 2012; GOO *et al.*, 2016; DUTRA *et al.*, 2018).

Em relação aos ensaios mecânicos, o teste de resistência flexural é amplamente usado para se avaliar a resistência dos materiais cerâmicos (KELLY *et al.*, 2010). Contudo, testes monotônicos não simulam a tensão mecânica cíclica intermitente envolvida nas falhas/fraturas clinicas (KELLY *et al.*, 2010). Em serviço clínico, as restaurações são submetidas à inúmeros ciclos mastigatórios, os quais levam à fadiga do material (ZHANG *et al*., 2013), sendo portanto fundamental e relevante submeter os materiais cerâmicos aos testes de fadiga para que possamos ter maior previsibilidade da performance do material à longo-prazo (SILVA *et al*., 2010; ABOUSHEILIB & ELSAFI, 2016), mesmo reconhecendo que haja limitações nos testes "in vitro", dentre as quais podemos citar: espessura e geometria do espécime, tipo de pistão e seu movimento exercido durante o teste (axial ou em deslizamento), umidade do ambiente e frequência de aplicação da carga (KELLY, 1999; KELLY *et al*., 2010; MIRANDA *et al*., 2019). Nesse sentido, o aprimoramento dos testes de fadiga é necessário para predição com melhor acuidade do comportamento mecânico dos materiais ou restaurações.

7. CONCLUSÕES

A partir dos estudos desenvolvidos na presente tese pode-se concluir que:

- Zircônias de segunda geração apresentam melhor comportamento à fadiga que zircônias de terceira geração, devido às diferenças estruturais e composição de fase inerente de cada material.
- O acabamento/polimento reduzem a rugosidade superficial da Y-TZP gerada pelo desgaste com ponta diamantada;
- O acabamento prévio com pontas diamantadas fina e exta-fina associado ao polimento não apresenta efeitos na redução da rugosidade ou no comportamento à fadiga de discos não cimentados de zircônia;
- Polidores diamantados específicos para zircônia são mais efetivos na redução da rugosidade superficial, aumentando também, a resistência à fadiga.
- A aplicação de glaze independentemente da técnica utilizada (spray ou pó/líquido), não afeta deleteriamente a resistência a fadiga de uma cerâmica Y-TZP;
- Em relação às características superficiais (topografia e rugosidade) a aplicação de glaze pó/líquido produz uma superfície mais lisa que aplicação de glaze na forma spray.
- Tratamentos como desgaste, polimento e glaze realizados na superfície oclusal de discos de zirconia cimentados adesivamente em resina epóxi, não tem efeito deletério no comportamento à fadiga das restaurações.

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