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João Luiz Pozzobon

EFEITO DE DIFERENTES TRATAMENTOS DE SUPERFÍCIE NA RESISTÊNCIA À FLEXÃO BIAXIAL E RESISTÊNCIA DE UNIÃO ENTRE CIMENTO RESINOSO E UMA CERÂMICA Y-TZP

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

Orientador: Prof. Dr. Luiz Felipe Valandro

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Luiz Felipe Valandro
(Presidente/Orientador)

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Marilia Pivetta Rippe (UFSM)

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César Dalmolin Bergoli (UFPEL)

Santa Maria, RS
2016

RESUMO

EFEITO DE DIFERENTES TRATAMENTOS DE SUPERFÍCIE NA RESISTÊNCIA À FLEXÃO BIAXIAL E RESISTÊNCIA DE UNIÃO ENTRE CIMENTO RESINOSO E UMA CERÂMICA Y-TZP

AUTOR: João Luiz Pozzobon
ORIENTADOR: Luiz Felipe Valandro

Este estudo avaliou o efeito de diferentes tratamentos de superfície na resistência à flexão biaxial e confiabilidade da cerâmica Y-TZP usando a análise de Weibull. Além disso, a influência desses métodos na resistência de união entre um cimento resinoso e a cerâmica Y-TZP foi estuda. Os tratamentos de superfície analisados foram: jateamento com partículas de óxido de alumínio revestidas por Sílica por 10 segundos e com 10 mm de distância (TBS); aplicação de glaze na superfície da cerâmica e condicionamento com ácido fluorídrico gel 10% (HF) por 1 min (GLZ_1), 5 min (GLZ_5), 10 min (GLZ_{10}) e 15 min (GLZ_{15}) e deposição de nanofilme de SiO_2 com 5 nm de espessura através da deposição física de vapor (PVD) (SNF). Este último tratamento foi utilizado apenas no trabalho referente a resistência à flexão biaxial. Para a resistência à flexão biaxial e confiabilidade do material 210 discos (15mm x 1.2mm) de Y-TZP foram confeccionados seguindo a ISO 6872-1999 e sinterizados de acordo com instruções do fabricante. Os discos foram divididos (n=30) de acordo com o tratamento de superfície aplicado (6 grupos) além do grupo controle o qual não recebeu tratamento. Através dos dados de resistência à flexão, foi realizada também a análise estatística de Weibull, a qual é utilizada para descrever a confiabilidade de materiais cerâmicos. Para avaliar a resistência de união, 75 blocos de Y-TZP (In-Ceram YZ, Vita) (5×5×4 mm) foram divididos em cinco grupos (n=15) de acordo com o tratamento de superfície. Espécimes de resina composta (diâmetro=3.25mm; altura=3mm) foram cimentados nos blocos de Y-TZP usando um cimento resinoso (Relyx ARC). Todos os espécimes foram submetidos ao envelhecimento (10,000 ciclos e armazenamento por 90 dias), ao teste de cisalhamento, analisados no estereomicroscópio, no microscópio eletrônico de varredura (MEV) e submetidos a análise de transformação de fase (XRD). Com relação à flexão biaxial, pode-se notar que o grupo TBS apresentou o maior valor de resistência e os grupos com aplicação do glaze seguido de condicionamento com ácido fluorídrico mostraram os menores valores independente do tempo de condicionamento. Entretanto nenhum tratamento influenciou de maneira negativa a confiabilidade do material quando comparado com o grupo controle. Para os resultados de adesão, o grupo TBS obteve os maiores valores de resistência de união. É possível concluir que o tratamento com jateamento (grupo TBS) confere uma melhor resistência de união na cerâmica não influenciando de forma negativa na resistência flexural desse material.

Palavras-chave: Flexão Biaxial. Resistência de União. Tratamento de Superfície. Y-TZP.

ABSTRACT

EFFECTS OS DIFFERENTS SURFACE TREATMENT ON BIAXIAL FLEXURE STRENGTH AND BOND STRENGTH OF A RESIN CEMENT AND CERAMIC Y-TZP.

AUTHOR: João Luiz Pozzobon
ADVISOR: Luiz Felipe Valandro

This study evaluated the effect of different surface treatments on the biaxial flexure strength and reliability of a ceramic Y-TZP by using Weibull analysis. In addition, the influence of these methods on the bond strength of a resin cement and ceramic Y-TZP were studied. The surface treatments analyzed were the following: air-borne particle abrasion with silica-coated aluminum oxide particles for 10 s with 10 mm distance (TBS); application of a ceramic overglaze on the surface and etching with 10% hydrofluoric acid gel (HF) for 1 min (GLZ_1), 5 min (GLZ_5), 10 min (GLZ_{10}), and 15 min (GLZ_{15}); and deposition of 5 nm thick SiO_2 nanofilm by physical vapor deposition (PVD) (SNF). This last treatment was performed only to study the biaxial flexural strength. Regarding the biaxial flexural strength and reliability of the material, 210 discs (15mm × 1.2mm) of Y-TZP were made following the ISO 6872-1999 and then sintered according to the manufacturer`s instructions. The discs (n=30) were divided according to the different surface treatments applied (6 groups), apart from the control group which did not receive any treatment. Using the flexural strength data, a Weibull statistical analysis was performed to describe the reliability of the ceramic material. To evaluate the bond strength, 75 Y-TZP (In-Ceram YZ, Vita) blocks (5×5×4 mm) were assigned into five groups (n=15) according to the different surface treatments. Specimens of composite resin (diameter=3.25mm; height=3mm) were cemented to the Y-TZP blocks using resin cement (Relyx ARC). All the specimens were subjected to aging (10,000 thermal cycles and storage for 90 days), tested in shear, analyzed under a stereomicroscope and under a scanning electron microscope (SEM), and measured by phase transformation analysis (XRD). In relation to the biaxial flexure, the TBS group statistically presented the highest strength values, and the groups with glaze application followed by etching with hydrofluoric acid (GLZ_x) showed the lowest values, independent of the HF treatment time. However, no surface treatment negatively influenced the reliability of the material when compared with the control group with no surface treatment, according to the Weibull analysis results. With respect to adhesion, the TBS groups showed the highest bond strength values. It may be concluded that treatment with blasting (TBS group) confers the best bond strength to ceramics out of all the tested methods, without negatively influencing the strength of the material.

Keywords: Biaxial Flexure Strength. Bond Strength. Surface Treatment. Y-TZP.

SUMÁRIO

1 INTRODUÇÃO

A cerâmica Y-TZP (zircônia tetragonal policristalina parcialmente estabilizada por ítrio) devido as suas excelentes propriedades mecânicas como resistência e tenacidade a fratura (KOSMAC et al., 1999), biocompatibilidade (ABOUSHELIB et al., 2008), fraca adesão bacteriana (EGILMEZ et al., 2014) tem sido reconhecida como um material de destaque para a Odontologia nos últimos anos (KOSMAC et al., 1999), sendo freqüentemente utilizada como bráquetes ortodônticos (CONRAD et al., 2007; KIRAN et al., 2013), infraestruturas de próteses unitárias/parciais fixas e, atualmente, na confecção de restaurações monolíticas, via sistemas CAD/CAM (DENRY E KELLY, 2014).

A zircônia pura se apresenta em 3 formas cristalinas, variando de acordo com diferentes intervalos de temperatura (ELGIMEZ et al., 2014). Em temperatura ambiente ela se encontra na fase monoclínica, permanecendo assim até 1170°C, acima deste valor se transforma em tetragonal se mantendo nesta forma até 2370°C e acima deste limite passa para a fase cúbica (PICONI et al., 1999). Na tentativa de estabilizar a zircônia na fase tetragonal em temperatura ambiente é adicionado o óxido de ítrio (Y_2O_3) (PICONI et al., 1999). Mesmo assim, este material apresenta-se em uma forma metaestável, pois fatores externos como jateamento, impacto e envelhecimento térmico (KARAKOCA et al., 2009) podem desencadear uma transformação de fase de tetragonal para monoclínica (t-m), levando a um aumento de volume de aproximadamente 3-5% na região da trinca, induzindo formação de forças compressivas que podem atuar no fechamento de extremidades de algumas falhas prevenindo assim a sua propagação e aumentando a tenacidade a fratura (KOSMAC et al., 1999; PICONI et al., 1999; KARAKOCA et al., 2009).

Apesar de suas excelentes propriedades mecânicas, devido a sua estrutura policristalina e composição química, a Y-TZP não apresenta um bom comportamento com relação a adesão. É classificada como uma cerâmica acidorresistente, pois o ácido fluorídrico não altera sua superfície (OZCAN et al., 2002) por não apresentar fase vítrea (ausência de sílica), impedindo que retenções micromecânicas sejam criadas, assim como, não existindo ativação química da superfície (CURA et al., 2012). Com isso, diversos métodos têm sido investigados e estudados afim de que uma adesão estável e durável com a Y-TZP seja promovida (VALENTINO et al., 2012; ABOUSHELIB et al., 2007; DRUCK et al., 2015).

O tratamento triboquímico é o método mais utilizado e recomendado, consistindo na combinação do jateamento da superfície da zircônia com partícula de óxido de alumínio

revestida com sílica (CoJet-Sand, 3M ESPE AG, Seefeld, Alemanha) seguido da aplicação de silano (PIASCK et al., 2009). Essas partículas jateadas são incorporadas na superfície da cerâmica tornando-a mais reativa, aumentando a rugosidade (KERN et al., 1998) e modificando sua energia superficial. Já o silano, tem como principal função formar as ligações siloxanas entre a sílica depositada e os grupos metacrilatos do cimento resinoso (OZCAN et al., 2002).

Entretanto, não há consenso na literatura a cerca dos efeitos do jateamento sobre as propriedades mecânicas do material. Alguns estudos sugerem que esse método pode causar defeitos na estrutura do material o que aumentaria o risco de falhas catastróficas prematuras, ou seja, influenciariam de maneira negativa nas propriedades mecânicas da Y-TZP reduzindo a sua resistência (ZHANG et al., 2004; ZHANG et al., 2006). Porém, outros pesquisadores (KARAKOCA et al., 2009, KOSMAC et al., 1999) observaram aumento dos valores de resistência do material após o jateamento (SOUZA et al., 2013, SONG et al., 2013). Este efeito positivo seria devido a transformação de fase desencadeada pelo jateamento, a qual leva ao processo de tenacificação, dificultando a propagação de forma catastrófica de trincas. Segundo KOSMAC et al., 1999, a espessura da camada de forças compressivas criadas pelo jateamento é pequena, mas o suficiente para aumentar a resistência, já os defeitos introduzidos por essa técnica, não apresentam comprimento suficiente para influenciar nessa propriedade, pois não excedem essa faixa de compressão.

Outro método de tratamento de superfície que tem sido estudado é a aplicação de uma fina camada de glaze (CATTELL et al., 2009; NTALA et al., 2010). Essa camada rica em sílica torna a superfície capaz de ser condicionada pelo ácido fluorídrico, tornando-a retentiva e quimicamente reativa (VALENTINO et al., 2012; VANDERLEI et al., 2013). Entretanto deve-se lembrar de que através deste método, uma multicamada formada pela cerâmica e o glaze é criada, fazendo assim que o conhecimento da interação desses materiais, principalmente sua interface, seja de fundamental importância (BORBA et al., 2011). Existem diversos fatores que devem ser observados neste tipo de configuração, entre eles a diferença no módulo de elasticidade (BORBA et al., 2011; DELLA BONA et al., 2003), coeficiente de expansão térmica (CET) (BORBA et al., 2011; BENETTI et al., 2010; SWAIN et al., 2009), condutividade e difusividade térmica (SWAIN et al., 2009) dos diferentes materiais.

Nos últimos anos, a deposição de nanofilmes de SiO_2 (através do processamento a plasma) por meio da deposição física de vapor (PVD), com o uso de um sistema *magnetron sputtering* reativo tem sido proposto como método alternativo. Essa técnica tem a capacidade de alterar a propriedades da superfície através da deposição do filme, mas sem alterar a

estrutura do material, pois se baseia apenas na adesão química (QUEIROZ et al., 2013) . Esse método é muito utilizado na indústria, apresentando diversas vantagens como o controle da espessura, rápida deposição, possibilidade de obtenção de filmes com composição pura ou composta (DRUCK et al., 2015). Estudos tem mostrado resultados promissores para esta técnica (DRUCK et al., 2015; QUEIROZ et al., 2011; QUEIROZ et al., 2013), porém uma maior investigação é necessária para comprovar esse beneficio e também para conhecer sua influência no comportamento mecânico do material.

Diante desses fatos, é possível observar que o tratamento triboquímico, devido as suas características, apresenta valores satisfatórios com relação à resistência de união, porém ainda não é bem claro na literatura qual seu real efeito sobre as propriedades mecânicas da Y-TZP. Além disso, novos tratamentos recentemente introduzidos, como a aplicação do glaze sobre a superfície desse material e a deposição de nanofilmes, requerem estudos mais aprofundados para avaliar o verdadeiro efeito na adesão, assim como, sua influência sobre o comportamento mecânico dessas cerâmicas.

Com isso, os objetivos desse trabalho foram investigar os efeitos desses tratamentos de superfície da cerâmica Y-TZP na resistência a flexão biaxial e na adesão entre Y-TZP e cimento resinoso, bem como a transformação de fase.

2 MECHANICAL BEHAVIOR OF Y-TZP CERAMIC AFTER DIFFERENT ZIRCONIA SURFACE TREATMENTS

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MECHANICAL BEHAVIOR OF Y-TZP CERAMIC AFTER DIFFERENT ZIRCONIA SURFACE TREATMENTS

JL Pozzobon, GKR Pereira, V Wandscher, LS Dorneles, LF Valandro

João Luiz Pozzobon. MSc graduate student in Oral Sciences (Prosthodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil
Gabriel Kalil Rocha Pereira. PhD student in Oral Sciences (Prosthodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil
Vinicius Wandscher. PhD student in Oral Sciences (Prosthodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil
Lucio Strazzabosco. PhD, Associate Professor, Physics Department, Federal University of Santa Maria, Santa Maria, Brazil.
Luiz Felipe Valandro. MSD, PhD, Associate Professor, PhD-MS Graduate Program in Oral Science (Prosthodontics Units), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.

Corresponding author:
Luiz Felipe Valandro, D.D.S, M.S.D., Ph.D., Associate Professor,
Federal University of Santa Maria
Faculty of Odontology
PhD-MSc Graduate Program in Oral Science
Prosthodontics Units
Floriano Peixoto, 1184, 97015-372, Santa Maria, RS, Brazil
Phone: +55-55 32209276, Fax: +55-55 32209272
Email: lfvalandro@hotmail.com (Dr LF Valandro)

Authors' emails:

João Luiz Pozzobon (joaoluiz_pozzobon@hotmail.com)
Gabriel Kalil Rocha Pereira (gabrielkrpereira@hotmail.com)
Vinicius Felipe Wandscher (viniwan@hotmail.com)
Lucio Strazzabosco Dorneles (lsdorneles@gmail.com)
Luiz Felipe Valandro (lfvalandro@hotmail.com)

Running title: Effect of surface treatment on Y-TZP.

Mechanical behavior of Y-TZP ceramic after different zirconia surface treatments

ABSTRACT

This study investigated the effects of different zirconia surface conditioning methods on the biaxial flexure strength, surface characteristics and fractographic analysis of a Y-TZP ceramic. Disc-shaped specimens were manufactured according to ISO 6872-2008 for biaxial flexure strength testing, were randomly and divided into the seven groups (n=30). Control (CTRL): without treatment; Tribochemical silica coating (TBS): specimens were sandblasted with silica-coated aluminum oxide particles (CoJet-Sand) for 10 s; Silica nanofilm (SNF): specimens were silica coated with a 5 nm SiO_2 nanofilm; and four protocols of low-fusing porcelain glaze(GLZ): etching with 10% hydrofluoric acid gel (HF) for 1 min (GLZ_1), 5 min (GLZ_5), 10 min (GLZ_{10}) and 15 min (GLZ_{15}). Phase transformation, roughness, micro-morphological, flexural analysis tests, and fractographic analyses were performed. X-ray diffraction (XRD) analysis showed that the TBS promoted the highest *m*-phase content (20.35). However, for the GLZ groups, XRD analysis was not sensitive enough to obtain an accurate reading for phase transformation. The GLZ group had the highest roughness values. The TBS group had the highest characteristic strength (1291.38 ± MPa), followed by SNF (999.26 MPa). These results suggest that (TBS) and (SNF) treatments did not reduce the mechanical properties, while (GLZ) led to a degradation in the mechanical properties.

Key words: Surface treatments, Mechanical properties, Damage, Phase transformation, Zirconium oxide partially stabilized by yttrium

INTRODUCTION

Among all dental ceramics available, the Y-TZP (Yttrium-stabilized Tetragonal Zirconia Polycristal) has been showing superior mechanical properties as high flexural strength, fracture toughness, besides its biocompatibility[1]and appropriate optical properties. Based upon this fact, dental community has shown great interest[1] in their applications, as orthodontic brackets, frameworks for unit/multi-unit fixed dental prostheses (FDPs) and monolithic full-contour restorations[2].

Y-TZP presents a high crystalline content, showing no glassy phase. Zirconia is a polymorphic material that exists in nature in three crystalline forms: monoclinic (*m*), tetragonal (*t*) and cubic (*c*)[3]. Due to the addition of Y_2O_3, ZrO_2 particles are stabilized at *t*-phase on room temperature, although *t-m* phase transformation still may occur when Y-TZP is submitted to distinct stimuli oral mastication forces, exposure to water and different temperatures, pH changes, and oral microorganisms[5–10]

The literature suggests that the effects of *t-m* phase transformation is governed by the transformation toughening mechanism. In this process, *t-m* phase transformation occurs at superficial grains on the ceramic surface, leading to volume increase (~3 to 5%) at a localized area around the superficial defects. This results in compression stress concentration around such flaw sand arresting crack propagation (increase in fracture toughness).[9,10] After that, *t-m* phase transformation spreads throughout the material surface and subsurface, resulting in grain pullout and an increase in roughness,[11] jeopardizing strength, fracture toughness and density of Y-TZP structures,[12] which is known as low-temperature degradation.[13]

In addition, the adhesion to Y-TZP ceramics is challenged.[14] Literature shows that the main reason for poor adhesion is the high crystalline content (no glassy phase) in Y-TZP, classifying it as a non-etchable ceramic.[15,16] In other words, the use of hydrofluoric acid does not create micromechanical retention, neither makes the surface more chemically active.[17] Thus, several methods have been studied and proposed to overcome this problem and promote a stable and durable chemical micromechanical adhesion with zirconia.[14,18–21]

The main zirconia surface treatment recommended for this use is the tribochemical method, which consists of air-abrasion with silica coated alumina particles followed by silanization.[22,23] However, the scientific community is still cautious regarding its use, because this surface treatment may cause surface damage and *t-m* phase transformation,[24] even though controversial results in terms of damage effect have been found.[11,26–32]

An alternative method is the application of a thin layer of porcelain glaze[29] to make the zirconia surface rich in silica, by which the Y-TZP surface becomes etchable by

hydrofluoric acid, improving the micromechanical retention and chemical reaction for resin adhesion.[14,18,30] However, it is important to emphasize that this treatment creates a multi-layer configuration (glass-zirconia layers),[31] which could impact the mechanical behavior of zirconia materials (effect of glass-zirconia interaction on zirconia's strength).

Recently, a new zirconia surface treatment method has been proposed, which consists of deposition of a SiO_2 nanofilm on the Y-TZP surface through a plasma process, followed by silanization. This process allegedly does not damage the surface, and additionally, enhances the bond strength between resin cement and Y-TZP.[20,21] However, there is no study that evaluated if this treatment would influence the mechanical behavior of the Y-TZP ceramics.

Thus, it is very important for the use of Y-TZP as dental ceramics to develop and investigate zirconia treatments for resin bond improvements without compromising its mechanical properties. Literature has been showing promising results on bond strength, especially after tribochemical treatment, glaze application and silica nanofilm deposition,[14,20,32,33] but it lacks data regarding the effects of these treatments on the Y-TZP mechanical properties. Therefore, this *in vitro* experimental study investigated the effects of different zirconia conditioning methods (tribochemical treatment, silica nanofilm, and glaze application) on the biaxial flexure strength and superficial characteristics of Y-TZP ceramics. The null hypothesis was that the surface treatment would have no impact in biaxial flexure strength.

MATERIAL AND METHODS

Sample Preparation

Disc-shaped specimens (N=210) were manufactured according to ISO 6872-2008[34] (ISO 6872) for biaxial flexure strength testing. Pre-sintered blocks of Y-TZP (Vita In-Ceram 2000 YZ, Vita Zahnfabrik, Bad Säckingen, Germany) were molded into cylinders (Ø=18 mm) with 600-1200 grit SiC paper (3M) under water cooling, and then sectioned using a cutting machine (IsoMet® 1000, Buehler, Lake Bluff, Illinois, USA) in slices of 18 mm (Ø) × 1.6 mm (thickness).

To remove irregularities introduced by the process, the slices were finely ground with 1200 grit SiC paper and cleaned in an ultrasonic bath (1440 D – Odontrobras, Ind. E Com. Equip. Méd. Odonto. LTDA, Ribeirão Preto, Brazil) using 78% isopropyl alcohol for 10 min. Then, the specimens were sintered (Zyrcomat T, Vita Zahnfabrik) according to the manufacturer's instructions (1530°C, holding time: 120 min) resulting in pieces with the final dimensions of 15 mm × 1.2 mm approximately.

After sintering, the specimens which presented discrepancies in length above the standard variation recommended by ISO 6872-2008 (1.2±0.2 mm) were discarded and then, samples (n=30) were randomly assigned into seven groups according to the surface treatment conditions executed (Table 1).

Y-TZP surface treatments

Control group (CTRL) remained untouched after the sintering process samples. The other six groups were divided and processed according to the following treatments:

- *Tribochemical treatment (TBS):* For standardization of this procedure, the specimens were attached to a metallic device that ensured that the abraded surface was kept parallel to the air-borne particle device at a distance of 10 mm and at an inclination of 45°. Then, the surface was sandblasted with silica-coated aluminum oxide particles (CoJet-Sand, 3M ESPE AG, Seefeld, Germany) under 2.8 bar pressure for 10 s with circular movements so that the whole disc surface was sandblasted.

- *Silica Nanofilm (SNF):* SiO_2 thin films were deposited at the Magnetism and Magnetic Materials Laboratory, UFSM (Santa Maria, Brazil), using the magnetron sputtering physical vapor deposition (PVD) process. In this process, the Y-TZP blocks and the silicon dioxide target were positioned in a vacuum chamber. The atmosphere inside the chamber was pumped down to 6×10^{-7} Torr. Argon gas was pumped into the chamber at a flow rate of 20 sccm, keeping the pressure at 5.2 mTorr. Pre-sputtering of the target was performed. After pre-sputtering, the substrate holder was placed over the target, initiating the deposition process. By controlling the time of exposure of the substrate to the plasma, the desired film thickness was obtained. The deposition process consisted of accelerating argon ions against the silica target, depositing the ejected material on the substrate (zirconia surface) located in front of the bombarded target. The SiO_2 nanofilm thickness deposited was 5 nm, which corresponded to 90 s of deposition.

-Application of a thin layer of low-fusing porcelain glaze followed by hydrofluoric acid etching: A porcelain overglaze (Vita Akzent, VITA Zahnfabrik) was used. For this process, the liquid was mixed with the powder of the system with a glass spatula, until a creamy consistency was reached. Then, it was applied in a thin coat using a brush and sintered according to the manufacturer's guidelines (starting at 500ºC, then being raised at 80ºC/min to 900ºC, maintaining this temperature for 1 min). After that, four protocols of etching with 10% hydrofluoric acid gel (HF) (Condac Porcelana, FGM - Joinville, Brazil) were investigated by

varying the etching time to 1 min (GLZ_1), 5 min (GLZ_5), 10 min (GLZ_{10}) and 15 min (GLZ_{15}).

After etching, the specimens were washed with air-water spray for 60 s, dried for 30 s, and cleaned in a sonic bath (5 min in distilled H_2O).

Phase transformation analysis by X-ray diffraction (XRD)

Quantitative analysis of phase transformation was conducted (n=2) to determine the relative amount of m-phase and depth of the transformed layer under each condition of surface treatment. The analysis was performed using an X-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany). Spectra were collected in the *2θ*, with a range of 25–35º, at a step interval of 1 s and step size of 0.03º.The amount of m-phase was calculated as[35]

$$X_M = \frac{(-111)_M + (+111)_M}{(-111)_M + (111)_M + (111)_T} \qquad (1)$$

where $(-111)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ=28° and 2θ=31.2°, respectively), and $(111)_T$ indicates the intensity of the respective tetragonal peak (2θ=30°). The volumetric fraction (F_m) of the m-phase was calculated according to Toraya et al.[36] as

$$F_m = \frac{1.311 \cdot X_M}{1 + 0.311 \cdot X_M} \qquad (2)$$

The transformed layer depth (TZD) was calculated on the basis of the amount of *m*-phase, considering that a constant fraction of grains were symmetrically transformed to *m*-phase along the surface, as

$$TZD = \left(\frac{sen\theta}{2\square}\right)\left[ln\left(\frac{1}{1-FM}\right)\right] \qquad (3)$$

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

Micro-Morphological Analysis

Two additional Y-TZP samples from each treatment condition were evaluated under a Field Emission Scanning Electron Microscope (FESEM) (Inspect F50, FEI, USA) with 2000x magnification to observe the surface modification. One more sample was measured to evaluate the thickness of the glaze layer. For this, was used a disc fractured in the biaxial flexure test, thereby being possible to observe the interface between zirconia and glaze.

Roughness analysis

Before flexural testing, the surface roughness of all the specimens was measured using a surface roughness tester (Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan). The reported values were Ra, the arithmetical mean of the absolute values of peaks and valleys measured from a medium plane (mm); and Rz, the average distance between the five highest peaks and five major valleys found in the standard (mm).Three measurements were performed for each specimen parameters with a cut-off (n=5) of λC 0.8 mm and λS 2.5 μm. Arithmetic mean values of all measurements from each specimen were obtained.

Biaxial flexure test

The strength was calculated using the biaxial flexural strength test according to the ISO 6872-2008.[34] Specimens were placed concentrically on the three support balls (ø=3.2mm), which were positioned 10 mm apart from each other in a triangular pattern. The samples were measured with the treated surface turned down (tensile stress). A thin scotch tape was placed on the compression side of the discs to prevent the spread of the fragments and to provide better contact between the piston and the sample.[37] The set was immersed in water, and a flat circular tungsten piston (ø=1.6 mm) was used to apply an increasing load (1mm/min). The point of fracture was recorded in a universal testing machine (EMIC DL 2000, São José dos Pinhais, Brazil).

Flexural strength was calculated according to ISO 6872:[34]

$$\sigma = -0.2387.\frac{P(X-Y)}{b^2} \qquad (4)$$

where σ is the maximum tensile stress on fracture region in MPa, P is the total load causing fracture in N, b is the thickness at the fracture origin in mm, and X and Y are constants calculated as

$$X = (1+v)\ln\left({}^{r_2}/_{r_3}\right)^2 + [\,(1-v)/2]\,(r_2/r_3)^2 \qquad (5)$$

$$Y = (1+v)\,[1+\ln\left({}^{r_1}/_{r_3}\right)^2] + (1-v)(r_1/r_3)^2 \qquad (6)$$

where v is Poisson ratio (according to Borba et al, [31] 0.32), r_1 is the radius of the support circle (5 mm), r_2 is the radius of the loaded area (0.8 mm), and r_3 is the radius of the specimen (7.5 mm).

Data Analysis

Statistical analysis was executed using Statistix 8.0, Analytical Software Inc., Tallahassee, FL, USA. First, a descriptive analysis was executed to determine the mean and

standard deviations of the roughness and the biaxial flexure strength data. Then, roughness data were subjected to a one-way analysis of variance (ANOVA) test and Tukey's test (α=0.05). Weibull statistical analysis[38] was used to describe the reliability of the ceramic material by describing the variation of resistance obtaining the Weibull modulus (m) and the characteristic strength (σ_C) with a confidence interval of 95%, as determined in a diagram according to DIN ENV 843-5, 2007

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

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

Fractographic Analysis

Fractographic analysis was performed using a light microscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany) and a Field Emission Scanning Electron Microscope (FESEM; Inspect F50, FEI, USA) on a representative part of the specimens from each treatment condition. Before analysis, the fracture surfaces were cleaned with distilled water in an ultrasonic bath for 5 min.

RESULTS

X-ray diffraction (XRD) analysis (table 1) showed small phase transformations for the CTRL (2.33) and SNF (3.82) group, while the TBS surface treatment promoted higher *m*-phase content. For the GLZ groups, this analysis was not sensible enough to promote an accurate reading for the *m*-phase.

Roughness data analysis, characterized by *Ra*, showed that the roughness increased as a function of HF etching time (GLZ_{15}>GLZ_{10}>GLZ_5=GLZ_1) for the glaze groups. The lowest roughness values were observed for the CTRL, TBS, and SNF groups, which had statistically similar roughness (CTRL=TBS=SNF<GLZ), as presented in Table 1.

The TBS group statistically presented the highest characteristic strength values, while the groups with a thin layer of porcelain glaze followed by hydrofluoric acid etching (GLZ_x) had the smallest values (table 1). The reliability in all groups, depicted as the *m* modulus, did not decrease after surface treatment, in comparison to the CTRL group (table 1 and figure 4).

Fractographic analysis showed that the origins of fracture were always located on the superficial or subsurface region of the area submitted to tensile stress concentration during the test, as shown in Figure3.

The survival graph (Figure 5), showed that for the groups with glaze, there is a high probability of failure with 800 MPa (GLZ_1-76.6%; GLZ_5-86.7%; GLZ_{10}-90%; GLZ_{15}-86.7%). For the other groups the failure probability is very small, not exceeding 17% (TBS-3.3%; SNF-16.3%; CTRL-13.3%).

DISCUSSION

It is widely known that additional surface treatments are necessary to enhance the adhesion of Y-TZP restorations[18,33]. There are few studies[24,28,39] that evaluate the effect of surface treatment on the mechanical properties, and no study considered the effect of new treatments, such as silica nanofilm deposition and application of a thin layer of low-fusing glaze. Our findings showed that the surface treatments statistically influenced the mechanical properties of Y-TZP, thus the null hypothesis must be rejected.

According to X-ray diffractometric analysis (Table 1), the TBS method induced an increase in the *m*-phase content (20.35%), while the SNF group did not lead to a significantly higher *m*-phase content (3.53%) in comparison to the CTRL group (2.33%). As phase transformation was found even in specimens that were not subjected to surface treatment (CTRL group), it may have been caused by the sample preparation process (cutting, grinding, and sintering).

Regarding the XRD analysis of etched/glazed specimens, Strasberg et al.[44] stated that XRD focuses on analyzing the material surface, where the depth of analysis is dependent on the angle of incidence and the coefficient of linear absorption. As seen in Figure 2, the glaze specimens presented a glaze thickness in the range of 70–110 μm, which seemed to be a thicker layer than the XRD depth readings. Consequently, it has been considered as an inappropriate tool to evaluate phase transformation under these conditions.

In the TBS group, it is possible to observe the higher values for phase transformation (20.35%) and consequently a greater depth (1.15) of transformation. Some authors[45,46] argue that the air-abrasion can cause a negative impact on the zirconia surface. Zhang et al.[45] reported that the particle abrasion could reduce the strength of the Y-TZP ceramic by introducing surface microcracks, which can lead to failure. Other researchers[9,22,24,43,44] showed a positive impact in the zirconia strength due to phase transformation from tetragonal to

monoclinic phase.[24] Surface treatments such as particle air-abrasion are able to trigger this transformation mechanism.[11,31,32,47]

When considering a simulated clinical scenario with conditioning of the inner surface of full-contour all-zirconia crowns, Campos et al.[27] showed that when comparing an adhesive and non-adhesive cement zirconia crown, higher survival rates were found for an adhesive system. When sandblasting is used for treating the inner surface of zirconia crowns, even with larger particles, the system behaved as a bonded crown, promoting a higher fatigue resistance of the cemented crowns. Therefore, the surface treatment did not compromise the structure of the ceramic. Other research[26] also simulated clinical fatigue of Y-TZP crowns coated with porcelain, finding that the treatment did not affect the failure load of the crowns. Similar fatigue resistances were observed for groups with and without treatment of the inner surface of the crowns, both groups with adhesive cementation.

In summary, when the ceramic surface is abraded *t-m* phase transformation occurs, resulting in a 3-5% local volume increase, which promotes compressive stress concentration around superficial defects and increases the fracture toughness.[9] This agrees with our results, which found the highest characteristic strength values (1291.38 MPa) in the TBS groups, which also showed a higher phase transformation. Apparently, the balance between the flaws introduced and the compressive stress surface layer created by *t-m* phase transformation would define the positive or negative impact on the mechanical properties. If the flaws introduced are confined inside this layer, a positive impact is observed.[9] On the other hand, if the flaws introduced were greater than the compressive stress surface layer, it may impact the mechanical properties negatively.[45] However, more studies are still needed to better clarify this effect.

According to literature,[48,49] the acceptable limit of phase transformation with accelerated aging is 25%, and the flexural decrease occurs when more than 50% of *m*-phase is present.[50] In this current study, accelerated aging is not present, since the *m*-phase percentage for all groups did not surpass those values. Although the values of the TBS group are the largest, this group also had the highest characteristic strength values (Table 1). Therefore, it can be observed and confirmed that below the 50% limit, the percentage of phase transformation is not sufficient to degrade the strength of the material.

With SEM analysis (Figure1), it is possible to observe that the surface treatment, except for the SNF group, promoted surface modification. The change in topography for the TBS group was produced by the silica-alumina particle impact. This irregularities created increases the surface area facilitating the creation of micromechanical interlocks at the

ceramic and resin cement interface which are able to enhance the adhesion[49,50]. Even with the visible topographical changes in the SEM analysis, there was no statistical difference related to roughness for this group when compared to the CTRL group. In the GLZ groups, the surface modification is directly related to the increase in HF etching time. Higher acid-etching times resulted in greater topographic alteration, which was also in agreement with the roughness analysis, where higher contact times led to higher roughness (Table 1).

When the stress applied on the material becomes higher than the strength, mechanical failure may occur,[51] being very dependent to the size of preexisting cracks.[52] In these structures with a framework and a porcelain (bilayer), the strength and fracture mode[51,53] are influenced by the material under tension.[31] In particular, Guazzato et al.[54] concluded that the properties of the material under tension stress will dictate the final strength of the structure. This could be the reason why the characteristic strength decreased for the glaze groups independent of the HF etching time (Table 1). In the current study, when bending the zirconia samples, the glazed surface was placed down under tension stress, and this led to a decrease of characteristic strength in comparison to the CTRL group. The fractographic analysis showed that the fracture origins were located in the subsurface region on the surface submitted to tensile stress in the biaxial flexure test. Furthermore, it is possible to observe small bubbles in the glaze layer (Figure 2), which may be considered as flaws. When a load is applied to this structure, these flaws can lead to catastrophic failure propagation, influencing the material strength (Figure 3). The inherent incorporation of these bubbles may be related to the method used for the application of this layer on the zirconia, showing perhaps that the technique used for applying the glaze layer has also influenced the lower characteristic strength values found for the glaze groups.

SNF groups had similar characteristic strength to the CTRL group (Table 1). As expected, silica nanofilms with thickness of 5 nm promoted no zirconia surface alterations,[20] leading to no effect on mechanical properties of the Y-TZP.

Through the Weibull modulus (*m*) (Table 1, Figure 4), it is possible to characterize the reliability and the probability of fracture of brittle materials.[55] It represents the data scatter of a given volume of ceramic under a uniform stress, or the flaw size distribution.[51] A material with a higher Weibull modulus shows a greater structural reliability, a higher level of structural integrity and potentially greater clinical reliability.[26,51] In this current study, the reliability (*m* modulus) did not decrease after surface treatment, having values ranges from 5.07 to 13.44 with statistical similarity, which showed the absence of structural reliability degradation. However, even if the *m*-modulus has not been changed for glaze groups, it is

possible to observe a significant reduction in resistance of the glaze groups, could be depicted due the glazed zirconia surface was tested under tension, and stress concentration may have occurred due to pre-existing flaws from this thin layer application.

Based on the survival graph (Figure5), it may be noted that for the groups with glaze, there is a high probability of failure above 75% with an applied tension of approximately 800 MPa (GLZ_1-76.6%; GLZ_5-86.7%; GLZ_{10}-90%; GLZ_{15}-86.7%), regardless of the acid conditioning time. For the other groups the failure probability is very small, not exceeding 17% (SNF-16.3%; CTRL-13.3%), especially in the TBS group, which has a probability of 3.3%, showing that this type of treatment did not impact deleteriously the strength of the material when compared to the other methods tested.

One limitation of this study was the standardization of the glaze thickness due to the application method used, which is very sensitive to the operator. Analyzing the transversal surface of the glazed surfaces, bubbles and defects incorporated during application of the glaze layer can be noted, even for trained researchers. Furthermore, fatigue tests were not performed in this study, thus future studies using fatigue approaches should be performed.

CONCLUSIONS

- The tribochemical treatment (TBS group) and silica nanofilms deposition (SNF group) did not degrade the mechanical properties of the Y-TZP. Therefore, they could be recommended as zirconia surface treatment.

- The glaze application seems to promote deterioration in the biaxial flexure strength. This deterioration can occur due to factors such as bilayer configuration that is formed but also may have been influenced by the method used.

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TABLE

Table 1: Groups evaluated according to the surface treatment. Flexure strength mean values and standard deviation (SD), Weibull parameters (characteristic strength - σ_c, Weibull modulus - *m* and respective 95% Confidence Intervals (CI - 95%)), XRD analysis (*m*-phase transformation content (%) and depth of the transformed layer - TZD (μm)), and roughness analysis (Ra and Rz parameters) are presented.

Surface Treatment	Group codes	Descriptive Analysis	Weibull Analysis				XRD Analysis		Roughness Analysis	
		Flexure Strength (MPa)	σ_c(MPa)	CI - 95%	M	CI - 95%	*m*-phase (%)	TZD (μm)	Ra (μm)	Rz (μm)
Control	CTRL	889.6^{B}(100)	927.85^{A}	891.29-964.84	10.8^{AB}	7.57-13.91	2.33	0.11	0.19^{D}	1.69^{A}
Tribochemical treatment	TBS	1209.5^{A}(192.7)	1291.38^{B}	1215.04-1370.21	7.13^{A}	5.00-9.18	20.35	1.15	0.21^{D}	1.75^{A}
Silica nanofilm	SNF	910.3^{B}(150.8)	999.26^{AC}	917.19-1086.10	5.07^{A}	3.55-6.52	3.82	0.19	0.18^{D}	1.57^{A}
Glaze + HF 10% 60s	GLZ_1	741.2^{C} (66.6)	772.44^{D}	747.84-797.14	13.44^{B}	9.41-17.28	-	-	0.44^{C}	2.34^{A}
Glaze + HF 10% 5 min	GLZ_5	722.6^{C} (77.8)	756.92^{D}	727.93-786.21	11.14^{AB}	7.80-14.32	-	-	0.51^{C}	4.79^{B}
Glaze + HF 10% 10 min	GLZ_{10}	711.6^{C}(70.2)	746.49^{D}	719.54-773.66	11.83^{AB}	8.28-15.21	-	-	0.98^{B}	9.40^{C}
Glaze + HF 10% 15 min	GLZ_{15}	691.6^{C}(108)	735.79^{D}	695.93-776.73	7.81^{AB}	5.47-10.04	-	-	1.59^{A}	12.75^{D}

*Different letters indicate statistically significant difference (Tukey's, $p<0.05$).

FIGURES

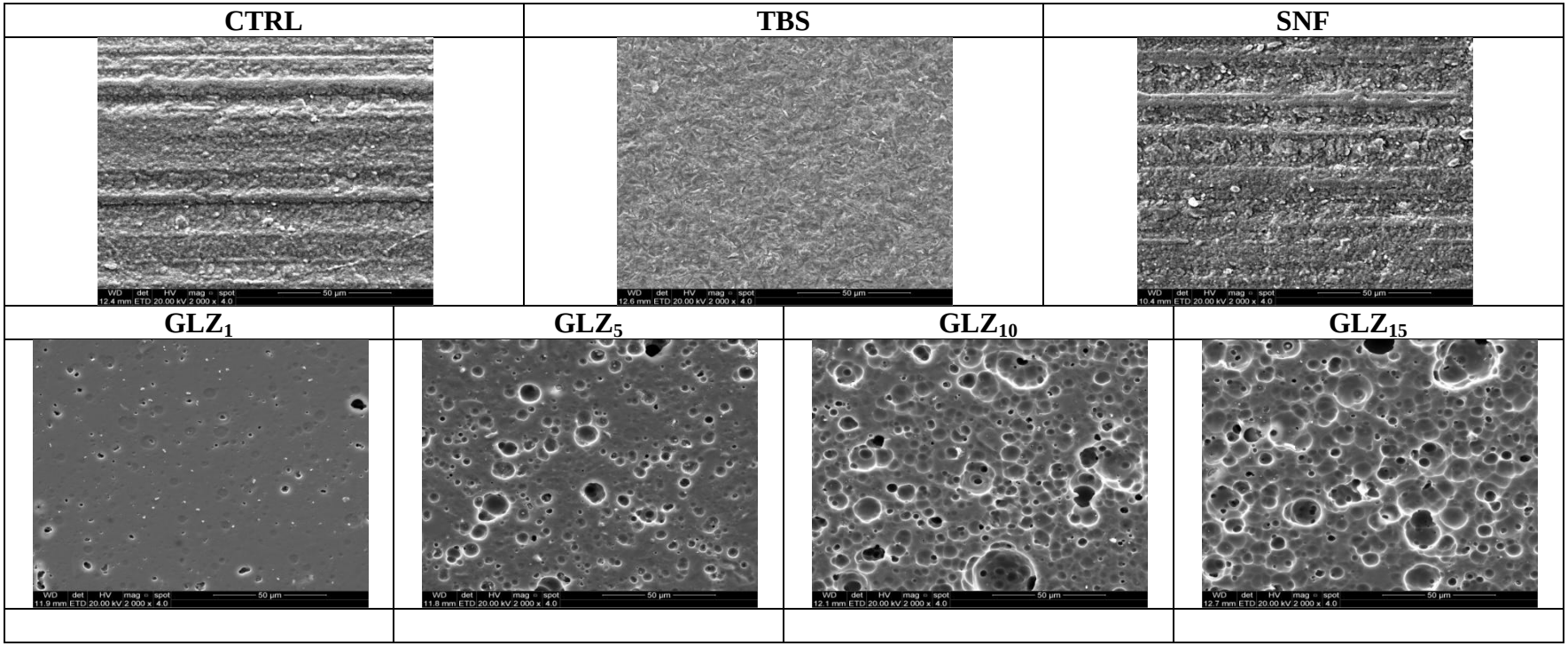
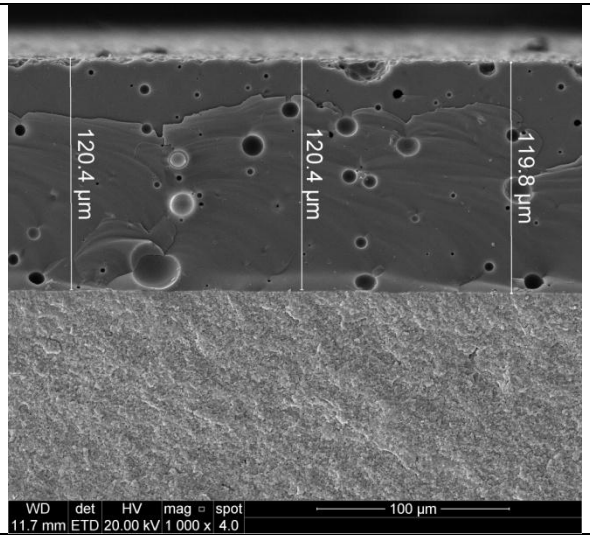
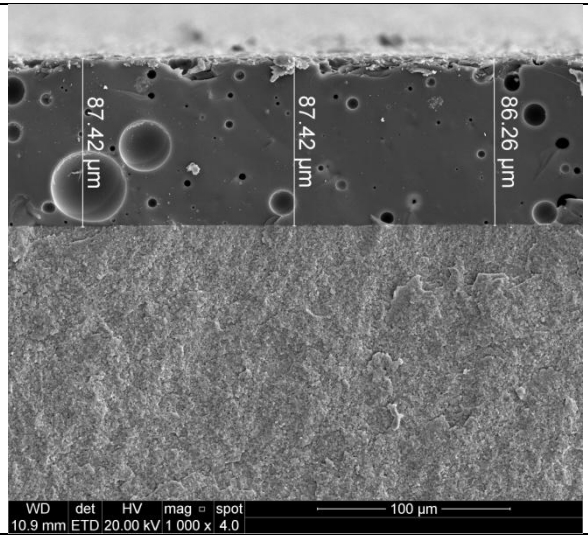


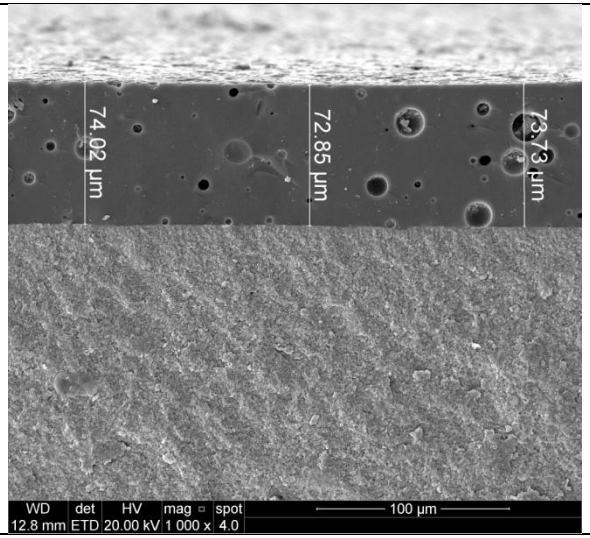
Figure 1. Representative micrographs of Y-TZP surface after different surface treatments with 2000x magnifications. It is possible to observe that the surface treatment, except SNF group, promote surface alteration.



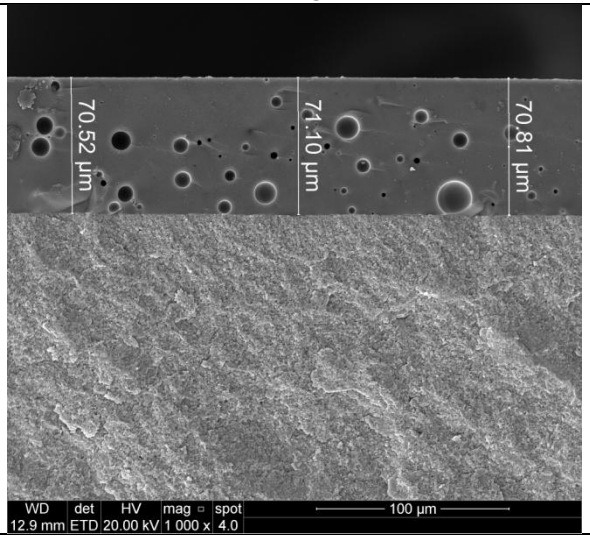
Glz_1



Glz_5



Glz_{10}



Glz_{15}

Figure 2. Representative SEM photomicrographs with 1000x magnifications of Glaze layer thickness. It is possible to observe the difference in the thickness and the presence of bubbles.

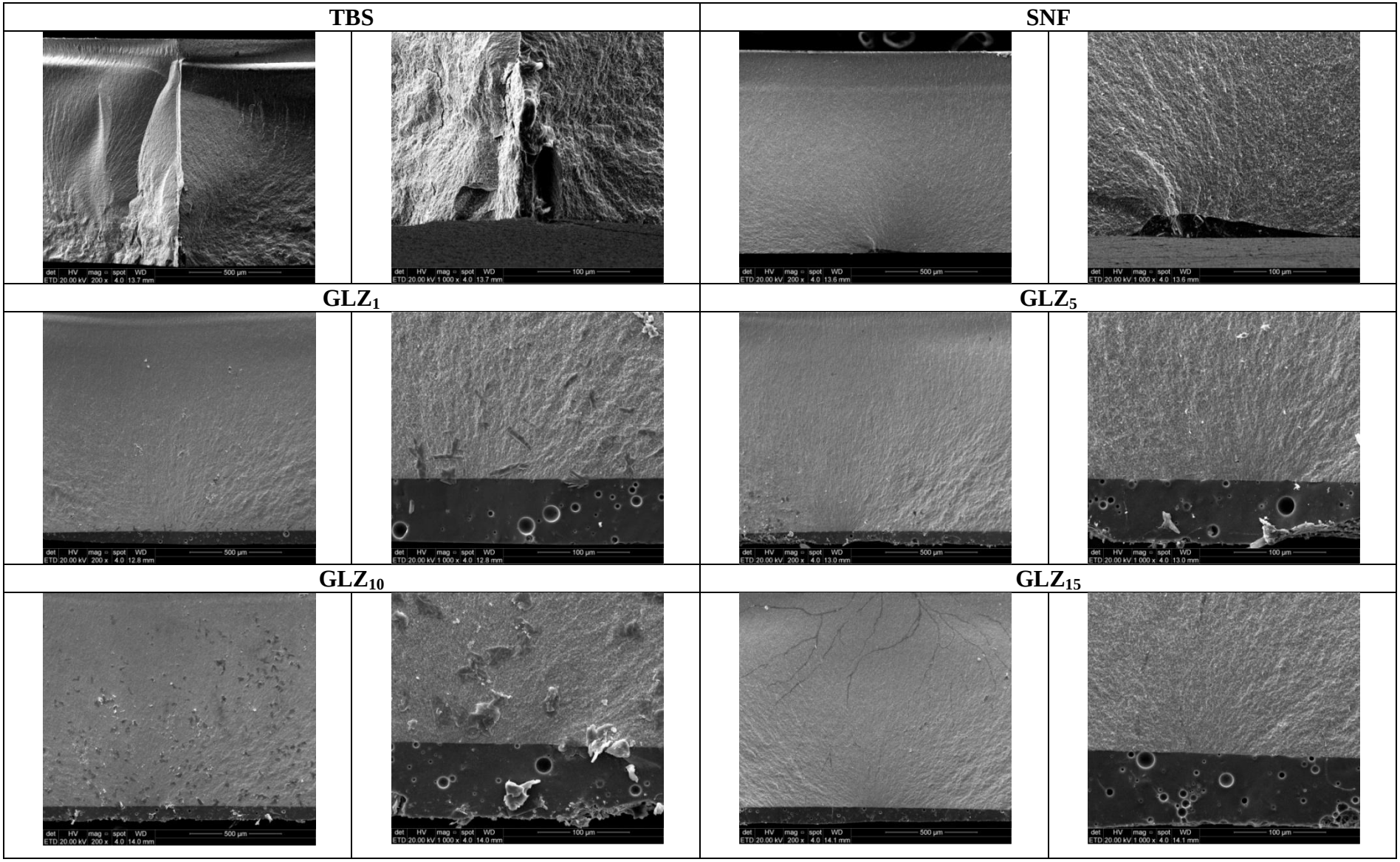


Figure 3. Representative Fractographic analysis after different surface treatments (left to right: 200x and 1000x magnifications. (A-B) TBS group; (C-D) SNF group, (E-F) GLZ_1 group, (G-H) GLZ_5 group, (I-J) GLZ_{10} group, (K-L) GLZ_{15} group.

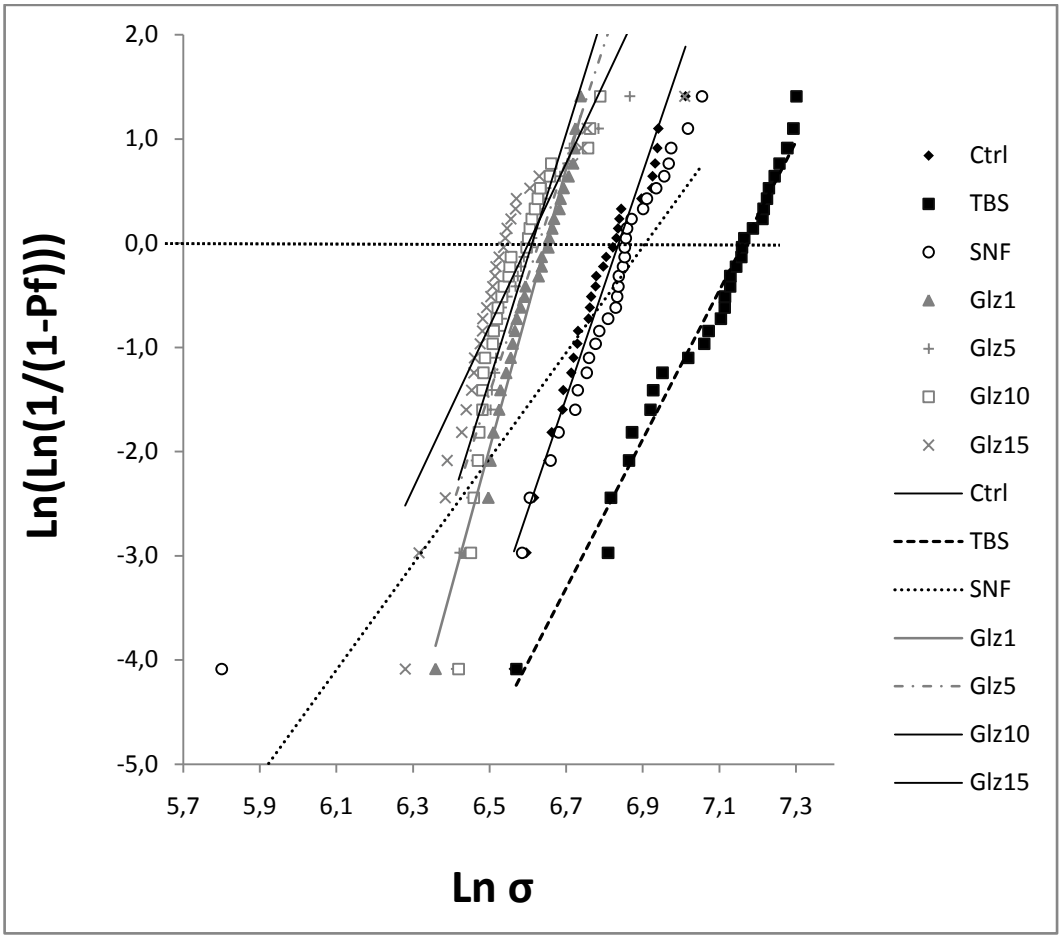


Figure 4. Weibull distribution for flexural strength (MPa) (diamond: Ctrl; black square: TBS; white ball: SNF; triangle: Glz_1; cross: Glz_5; white square: Glz_{10}; xis: Glz_{15}).

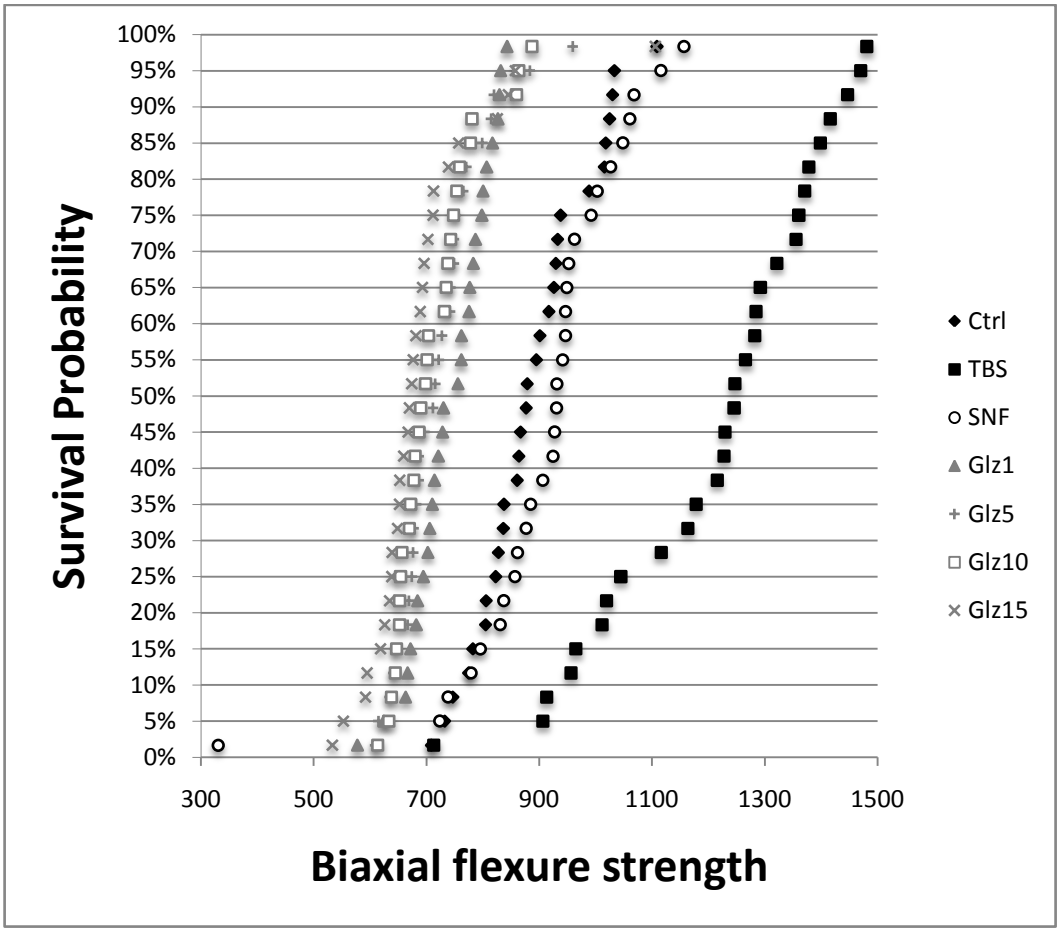


Figure 5. Total Weibull probability for fracture in tested groups.

3 INFLUENCE OF ZIRCONIA SURFACE TREATMENTS ON RESIN CEMENT BONDING AND PHASE TRANSFORMATION

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INFLUENCE OF ZIRCONIA SURFACE TREATMENTS ON RESIN CEMENT BONDING AND PHASE TRANSFORMATION

JL Pozzobon, VF Wandscher, MP Rippe, LF Valandro

João Luiz Pozzobon, MSc graduate student in Oral Sciences (Prosthodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil
Vinicius Felipe Wandscher, PhD student in Oral Sciences (Prosthodontics), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil
Marilia Pivetta Rippe, MSD, PhD, Graduate Program in Oral Science (Prosthodontics Units), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.
Luiz Felipe Valandro, PhD, Associate Professor and Head, Graduate Program in Oral Science (Prosthodontics Units), Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil.

Corresponding author:
Luiz Felipe Valandro, D.D.S, M.S.D., Ph.D., Associate Professor,
Federal University of Santa Maria
Faculty of Odontology
MDS-PhD Graduate Program in Oral Science
Prosthodontics Units
Floriano Peixoto, 1184, 97015-372, Santa Maria, RS, Brazil
Phone: +55-55 32209276, Fax: +55-55 32209272
Email: lfvalandro@hotmail.com (Dr LF Valandro)

Authors' emails:

João Luiz Pozzobon (joaoluiz_pozzobon@hotmail.com)
Vinicius Felipe Wandscher (viniwan@hotmail.com)
Marilia Rippe (mariliarip@hotmail.com)
Luiz Felipe Valandro (lfvalandro@hotmail.com)

Running title: Adhesion to Y-TZP: different surface treatments.

Influence of zirconia surface treatments on resin cement bonding and phase transformation

ABSTRACT

In this study, we have evaluated the effects of different zirconia surface treatments on the bond strength of a resin cement to Y-TZP (yttria-stabilized tetragonal zirconia) ceramics, as well as their phase transformations. 75 blocks (5×5×4 mm) of Y-TZP were assigned into five groups (n=15): specimens coated with tribochemical silica (TBS); specimens subjected to the application of a thin layer of low-fusing porcelain glaze, followed by hydrofluoric acid (HF) etching for four different times: 1, 5, 10, 15 min, and denoted as GLZ_1, GLZ_5, GLZ_{10}, and GLZ_{15}, respectively. After surface conditioning, all the specimens were washed, dried and silanized. Cylinders of composites (diameter: 3.25 mm; height: 3 mm) were cemented to the Y-TZP blocks using a resin cement. All the specimens were subjected to aging (10,000 thermal cycles and 90 days storage), tested under shear conditions, and finally analyzed by a stereomicroscope (failure analysis). In addition, we also performed topographical and phase transformation analyses of the treated zirconia surfaces. The TBS group improved the adhesion and presented the highest bond strength value (23.34 MPa), while the GLZ_1 group presented a high prevalence of pretest failures. X-ray diffraction (XRD) analysis showed a phase transformation for the TBS group (13.14%); however, there was no clear phase change observed for the GLZ groups. From our results, we concluded that air-abrasion with silica-coated aluminum oxide particles improved the resin bonding strength to the Y-TZP ceramics.

Key-words: adhesion, zirconia, surface treatment, phase transformation.

1. Introduction

Owing to their excellent biocompatibility[1] and superior mechanical properties, such as high flexural strength and fracture toughness, Y-TZP (yttria-stabilized tetragonal zirconia) ceramics have been used as the material of choice in the field of dentistry[2]. These ceramics are often used as orthodontic brackets, frameworks, and in monolithic full-contour restorations[3,4].

Y-TZP ceramics exist in three crystalline forms: monoclinic, tetragonal and cubic [5]. This crystalline phase consists of uniform equiaxed grains (0.2-0.5 μm in diameter) prepared from fine ZrO_2 particles[6,7].

When stimuli/stresses, such as mechanical fatigue, water, or grinding, are applied to this material, a phase transformation occurs, mainly from the tetragonal phase to the monoclinic phase. This is followed by a volume increase (3-5%) due to the phase transformation (tetragonal to monoclinic; $t \rightarrow m$) of crystals, generating compressive stresses around the crack end and preventing crack propagation (increase in fracture toughness)[2,5]. In order to control the volumetric alteration during phase transformations and stabilize zirconia in its tetragonal phase at room temperature[3], yttrium-oxide (Y_2O_3) is added at a weight percent of 3.5-8.7.

Although to their excellent mechanical properties, owing to presence of a high crystalline content, Y-TZP ceramics are classified as non-etchable ceramics[8,9]. For this reason, hydrofluoric acid (HF) etching does not cause any changes to the Y-TZP ceramic surface, which is confirmed by the absence of the glassy phase[10]. Therefore, zirconia surface conditioning is a challenging task when applying an adhesive cementation[11]. This demands an alternative surface treatment method, which can promote the surface characteristics and enhance the bond strength. Several methods have been studied and proposed to overcome this challenge and promote a stable and durable chemical micromechanical adhesion with the zirconia; these include the application of a low-fusing porcelain glaze[12], a selective infiltration technique[13], and the deposition of silica nanofilms[14].

Among the aforementioned methods, the tribochemical silica (TBS) coating method is widely used; it involves air-borne aluminum oxide particle abrasion with silica coating, followed by the application of a silane primer[15,16]. In the TBS treatment, the particles under pressure are attached to the ceramic surface, thereby making the surface chemically more reactive; increasing the surface roughness[17]; increasing the available surface area for adhesion, and modifying the surface energy[18]. After silica coating, the silane compound is

applied, which increases the surface wettability[19] and promotes the formation of siloxane bonds between the silica deposited on the surface and the adhesive resin methacrylate groups[10].

Studies have shown that the silica particles not only adhere to the surface, but also interact with zirconia, leading to covalent chemical bonds between the silica particles and the ceramic surface[18]. However, some researchers have reported that the sandblasting method can introduce some defects and micro-cracks on the Y-TZP surface, causing a damage that may have a negative impact on the mechanical properties[20]. Zhang *et al*.[21] found that this negative influence caused by the microcracks could reduce the fracture strength, although it is a controversial issue in the literature[22–28].

An alternative is to apply a thin layer of porcelain glaze, which is rich in silica content. This method provides a surface etchable by HF to yield a retentive and chemically reactive surface[11,12,29,30], allows for a facile interaction with silane[12], and presents favorable micromechanical properties and the formation of chemical bonds[31], as it occurs when adhering to the etchable feldspathic/glass ceramics. Studies have also shown that the bond strengths can be enhanced by the conditioning approach[32]. However, there is no consensus on the method for the surface conditioning of zirconia. Hence, it is important that further studies focus on optimizing this method, especially with regard to the etching time of HF after the application of a thin layer glazing.

Both etching time and concentration of HF had a significant influence on the surface of glass ceramic[33]. The surface roughness increased when the etching time increased from 45 to 90 s, but at 180 s, a reduction in roughness is observed[33]. This treatment could cause a problem, which is the seating of Y-TZP restorations on the prosthetic surfaces, as observed by Vanderlei *et al*.[11]. This study has also shown that the marginal misfit of the Y-TZP frameworks increased when a glassy film was applied.

As a consequence, the aim of this study was to evaluate the effects of different Y-TZP surface treatments on the bond strength between zirconia and resin-based cements, and also the influence of surface modification on the phase transformation. The null hypothesis was that Y-TZP surface treatment would not promote different bond strength.

2. Materials and methods

2.1. Specimen Preparation

Yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramic blocks (VITA In-Ceram 2000 YZ cubes for CEREC In-lab, VITA Zahnfabrik, Bad Säckingen, Germany) were

prepared by using a cutting machine (IsoMet® 1000, Buehler, Lake Bluff, USA), resulting in 75 blocks with final dimensions of 5×5×4 mm. These blocks were finished with Sof-Lex disks (3M ESPE, Seefeld, Germany), polished with 1200-grit sandpaper, sintered (Zyrcomat Oven, VITA) at 1350 °C according to the manufacturer's guidelines and washed with distilled water in an ultrasonic bath (Vitasonic, VITA) for 5 min.

2.2. Y-TZP surface treatments

Seventy five Y-TZP samples were randomly assigned into five groups (n=15). The details of surface treatments are shown in Table 1.

-*Tribochemical silica-coating (TBS)*: Specimens were subjected to airborne particle abrasion for 10 s with silica-coated aluminum oxide particles (CoJet-Sand, 3M ESPE AG, Seefeld, Germany), at a nozzle to surface distance of 10 mm and an inclination angle of 45°.

-*GLZ_1:* A thin layer of low-fusing porcelain glaze (Vita Akzent, VITA) was applied using a clean microbrush and sintered according to the manufacturer's guidelines. Thereafter, the glazed surface was etched with 10% HF for 1 min (FGM - Joinville, Brazil); washed with air-water spray and dried. Finally, all the samples were cleaned with distilled water in an ultrasonic bath for 5 min.

-*GLZ_5:* Glazing procedure was similar to the GLZ_1 group, but with a variation in HF etching time of 5 min.

-*GLZ_{10}:* Glazing was similar to the above procedure, but with a variation in HF etching time of 10 min.

-*GLZ_{15}:* As same as the above glazing procedure, but with HF etching for 15 min.

After the above conditionings, all specimens were silanized (ESPE-SIL silane, 3M/ESPE) and kept aside for 5 min for solvent evaporation.

2.3. Bonding procedure

Each Y-TZP block was embedded in a plastic cylinder (h=14 mm, Ø=25 mm) with chemically activated acrylic resin, which allows for the bonding surface free from contamination.

Before cementation, a bipartite cylindrical metallic template with a diameter of 3.25 mm and a height of 3 mm was used to produce composite resin cylinder specimens (Opallis, FGM, Joinville, Brazil) with a base area of 8.30 mm^2 for adhesion.

Dual-cure resin cement (RelyX ARC, 3M/ESPE, Seefeld, Germany) was applied to the bonding surface of the composite resin cylinder and then cemented to the Y-TZP surface, as recommended by the manufacturer.

The cement was light-cured (Radii-cal, SDI, EUA) for 20 s from the top and the excess cement was removed. Photo-activation was performed at the four lateral marginal regions for 40 s. All specimens were stored in distilled water at 37 °C for 24 h.

2.4. Aging process and shear bond strength (SBS) test

All the specimens were submitted to an extensive thermal cycling (10,000 cycles, 5°C/55°C, 30 s per bath) (Ethic Technology, Vargem Grande Paulista, Brazil) and stored in water at 37 °C for 90 days.

Shear bond strength (SBS) tests were carried out by subjecting the specimens to the universal testing machine (EMIC, São José dos Pinhais, Brazil) using a wire loop (Ø= 0.12 mm) at a cross-head speed of 1 mm/min, and the adhesive interface was loaded until failure occurred. The bond strength, ***R*** (MPa) was calculated according to the formula R=F/A, where ***F*** is the load for specimen failure (N), and ***A*** is the cross-sectional interfacial area (mm^2).

2.5. Failure analysis

All the specimens were observed under a stereomicroscope at 15x (Discovery V20, Carl Zeiss, Gottingen, Germany) to determine the failure type. The failures were classified as follows: (A) adhesive – a failure at the ceramic-cement interface; (B) cohesive – a failure in the resin cement; and (C) mixed failure (A+B). Representative failures were selected and analyzed under a field emission scanning electron microscope (FESEM) (Inspect F50, FEI, USA).

2.6. Phase analysis by X-ray diffraction

X-ray diffraction (XRD) quantitative analysis of phase transformation was acquired (n=2) to determine the relative amount of *m*-phase (monoclinic) and depth of the transformed layer under each experimental condition in the TBS group. The analysis was performed using an X-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany). Spectra were collected at the diffraction angle (2θ) within a range of 25–35º, at a step interval of 1 s and step size of 0.03º. The amount of the *m*-phase was calculated using equation 1[34]:

$$X_M = (-111)_M + (+111)_M / (-111)_M + (111)_M + (111)_T \qquad (1)$$

where $(-111)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ=28° and 2θ=31.2°, respectively) and $(111)_T$ indicates the intensity of the respective tetragonal peak (2θ=30°). The volumetric fraction of the *m*-phase was calculated following equation 2[35]:

$$F_m = 1.331 \, . \, X_M / 1 + 0.311 \, . \, X_M \quad (2)$$

The depth of the transformed layer was calculated using equation 3:

$$PZT = (\sin \theta / 2\mu)[\ln(1/1 - FM)] \quad (3)$$

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

2.7. X-ray energy dispersive spectroscopy

X-ray energy dispersive spectroscopy (EDS) (Quest; Thermo Noran, Middleton, WI) was performed to evaluate the chemical composition of the Y-TZP surfaces after different surface treatments.

2.8. Micro-morphological analysis

Two additional Y-TZP samples from each surface treatment were subjected to the FESEM at 500-1000x magnification to observe the effects of surface modification, while one more sample was used to evaluate the thickness of the glaze layer.

2.9. Data analysis

Kruskal–Wallis and Mann–Whitney multiple comparison tests (5%) were used to analyze the non-parametric data (Statistix 8.0 for Windows, Analytical Software Inc, Tallahassee, FL, USA); $P < 0.05$ was considered to be statistically significant in all tests.

3. Results

Since all the specimens from the groups (GLZ_5 and GLZ_{10}) failed before bond testing (Table 1), they were not considered for the statistical analysis. Besides, some specimens from the GLZ_1 and GLZ_{15} groups (number of preset failure – 7 and 4, respectively) failed before testing. In such cases, the lowest value from the respective group was attributed arbitrarily for a fair comparison, preventing over- or under- estimation for different groups. The determination of those reasonable values for pre-test failures is important to have a more effective comparison between the groups[11,36]. This method of allocation of the lower value of the respective group was accepted by some authors[36,37] as the best alternative, because

it represented the lowest amount required to promote the adhesive failure, showing a more realistic scenario.

The TBS group presented the highest bond strength, while the GLZ_1 and GLZ_{15} groups showed the lowest values, indicating no statistical difference (Table 1). Although we observed a monoclinic phase of 23.34% for the TBS group, we did not see any patterns corresponding to this phase transformation for the glaze groups (Figure 1). As shown in Table 1, the failure types were predominantly adhesive, while three cohesive failures were found in TBS groups and one cohesive failure in GLZ_1 group (Figure 2).

EDS analysis confirmed the presence of silica in the TBS and glaze groups, as evidenced by the major peak (Figure 3). It was also observed that all surface treatments promoted surface alterations and the glaze layer thickness ranged from 70 to 120 μm (Figure 4).

4. Discussion

From our findings, the null hypothesis was rejected, since tribochemical silica-coating increased the bond strength values in contrast with the glazing method.

According to some reports[11,12], glazing after HF etching can improve the bond strength between the Y-TZP surface and the resin cement, as compared to the zirconia surface without treatment. This improvement in glazing groups can be explained by two mechanisms. First, the surface roughness increased upon changes on the surface, which is caused by acid conditioning of the glazed surface. Second, the presence of silica promoted a chemical bond between the glassy film and the resin cement[11]. In our study, the bond strength values (Table 1) showed that at a higher etching time, the adhesive capacity was lowered in the glazing treatment. Probably, it could have occurred due to the absence of one or both characteristics mentioned above, absence of surface roughness or silica. However, the SEM analysis (Fig 4) showed that the increase of the surface topography changes was proportional to the etching time, whilst the EDS analysis showed that the percentage of silica on the YTZP surface did not decrease significantly with 1, 5, 10 or 15 min of etching. Besides the above two mechanisms, an alternative mechanism occurring due to the surface treatment might play a role in influencing the bond strength. Therefore, further studies are needed with other analyses to evaluate other characteristics that may be influencing the bond strength values.

We have found that the TBS surface treatment increased the bond strength values up to 23.34 MPa. Despite some negative aspects suggested by some researchers[21], TBS coating is still considered to be the gold standard in improving the bond strength to

zirconia[10,15]. Higher bond strength values obtained for the TBS group in our study, are in good agreement with other studies in the literature[11,14,38].

The stability and durability of bond strength are important factors in the evaluation of different protocols that are used to improve the adhesion between the resin cement and zirconia. Our current studies focused on the aged specimens through thermal cycling based on the literature reports, which supported the effectiveness of this method for the degradation of the cement/Y-TZP interface[14].

SEM analysis (Fig 4) revealed clearly that different surface treatments changed the surface topography of zirconia. The TBS group allows us to observe the effect of particle impact due to the air-abrasion and silica deposition. The surface topography modification by the silica-coated alumina particles is imperative to improving the bond strength owing to an increase in the surface roughness[39]. In the glaze groups, with an increase in the etching time of HF, the surface topographical changes become more intense. Similar micro-morphological changes were reported by Vanderlei *et al*.[11], wherein the Y-TZP ceramics received a thin layer of glaze with 9 % HF etching for 60 s. EDS analysis (Fig 3) confirmed the presence of silica on the treated surfaces of all groups tested.

According to the XRD (Table 1), the TBS group only showed the phase transformation change (13.14%), which occurred due to the particles impact on zirconia, leading to a volumetric expansion[39]. In literature, some reports showed a positive impact of abrasion on the mechanical properties of zirconia[2,24], due to the volumetric expansion that leads to the formation of toughening mechanism, by protecting the flaw catastrophic propagation. However, other authors[21], reported that the air-abrasion method decreased the bonding strength, as this procedure would be responsible for the introduction of news defects. In terms of bond strength, this transformation can compromise the establishment of reliable micromechanical adhesion due to the surface alterations[40]. However, in our current study, this influence was not detected, although the TBS group showed a higher bond strength value. For glaze groups, the XRD analysis was not sensitive enough to accurately detect peaks associated with phase transformation. According to Strasberg et al.[41], the XRD depth analysis is dependent on the incident angle and the coefficient of linear absorption. In our present study, the glaze layer thickness varied from 70 to 120 μm, which appeared to be greater than the XRD depth reading, thus, proving to be an inappropriate methodology to evaluate the phase transformation. All surface treatments showed values below the acceptable limit of 25% for m-phase transformation, according to the literature[42,43].

The predominant failure type was found to be adhesive (Table 1), but some cohesive failures were found for both TBS and GLZ_1 groups. The presence of pretest failure in some groups can be related to the weak adhesive interaction of the Y-TZP to the resin cement[12]. Although GLZ_1 and GLZ_{15} groups had a lower number of pretest failures compared to GLZ_5 and GLZ_{10}, the bond strength values were very low. Most probably, those premature failures occurred by weak adhesive interactions, the characteristic of which has already been reported[11]. However, further studies are needed to confirm this hypothesis. There is no consensus in the literature regarding the assignment of values for pretest failure. Some authors suggested[11,36] the allocation of reasonable values to those specimens. However, other authors[36,37] proposed that the best solution was to assign a lower value obtained within the group for those failed specimens. This would represent the lowest amount required to promote the adhesion failure. It is also important to show the percentage of premature failures, thus demonstrating a more realistic adhesive performance[11].

In this study, we have chosen a simple and facile shear bond strength mechanical test. A limitation of this test is the non-homogenous stress distribution at the adhesive interface[44]. In order to minimize this, a wire-loop was used[45]. Another limitation of this study lies in the standardization of the coating thickness in the glazing method used, despite the fact that the glazed specimens are properly randomized.

5. Conclusion

We have demonstrated that the tribochemical silica-coating (TBS) surface treatment method enhanced the bond strength in the Y-TZP ceramics, due to greater improvements in adhesion and micromechanical characteristics at the ceramic–silica interface, facilitating stable and durable surfaces.

Acknowledgments

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TABLE

Table 1: Different experimental groups and surface treatment methods with data on mean shear bond strength (standard deviation, SD) (MPa); *m*-phase transformation (%) and transformation depth (μm); failure type and number of pre-test failures in percentage.

Surface Treatment	Groups	n	Pretest Failures		Shear bond strength (MPa) (SD)*	Phase changes (%)	Transformation depth (μm)	Failure Type	
			Number of failures	%				Adhesive (%)	Cohesive (%)
Tribochemical silica	TBS	15	0	0	23.34 (5.8)A	13.14	0.71	80	20
Glaze + HF 10% (1 min)	GLZ_1	8	7	46.6	8.71 (9.95)B	-	-	93.4	6.6
Glaze + HF 10% (5 min)	GLZ_5	0	15	100	-	-	-	100	0
Glaze + HF 10% (10 min)	GLZ_{10}	0	15	100	-	-	-	100	0
Glaze + HF 10% (15 min)	GLZ_{15}	11	4	26.6	3.99 (4.31)B	-	-	100	0

*Different upper-case letters indicate statistically significant difference (Tukey's, $p<0.05$).

FIGURES

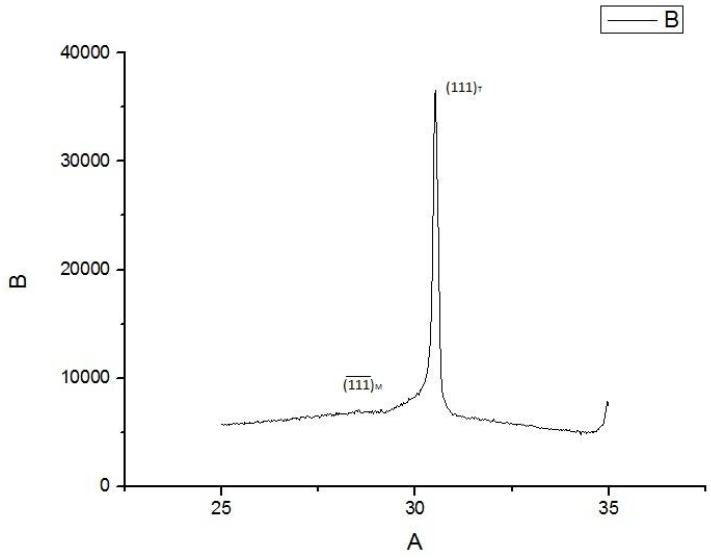

Figure 1. Representative X-ray diffraction spectra for phase transformation analysis of the Y-TZP ceramic material after tribochemical silica (TBS) treatment (A).

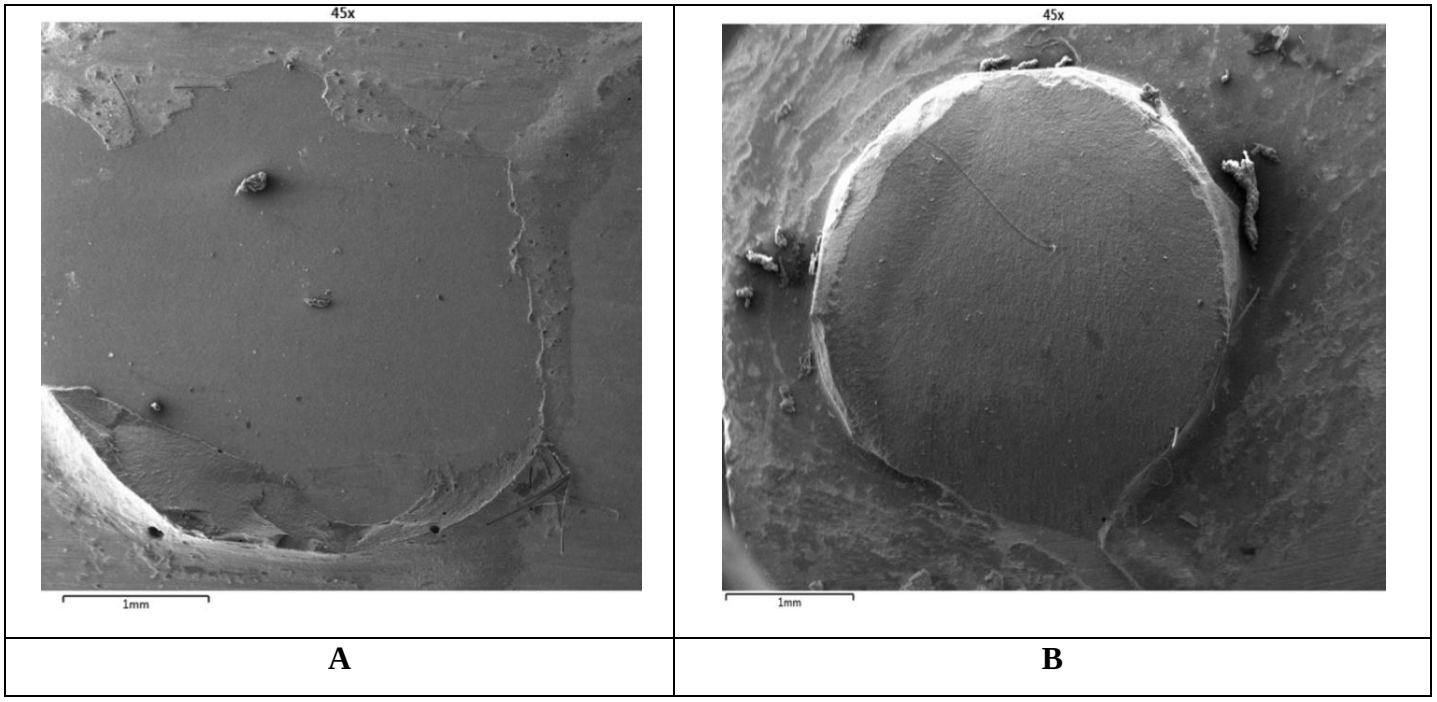

Figure 2. Representative micrographs of the failure modes from the tested samples. (A) Adhesive failure mode at the ceramic–cement interface; (B) Cohesive failure mode.

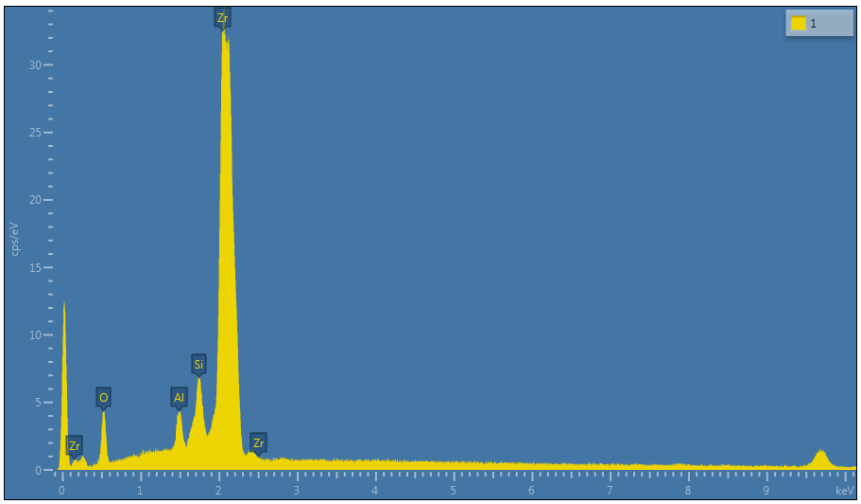

Figure 3: Representative EDS elemental chemical analysis of the Y-TZP surfaces treated by silica particles air-abrasion, wherein the spectrum shows clearly the presence of silicon oxide.

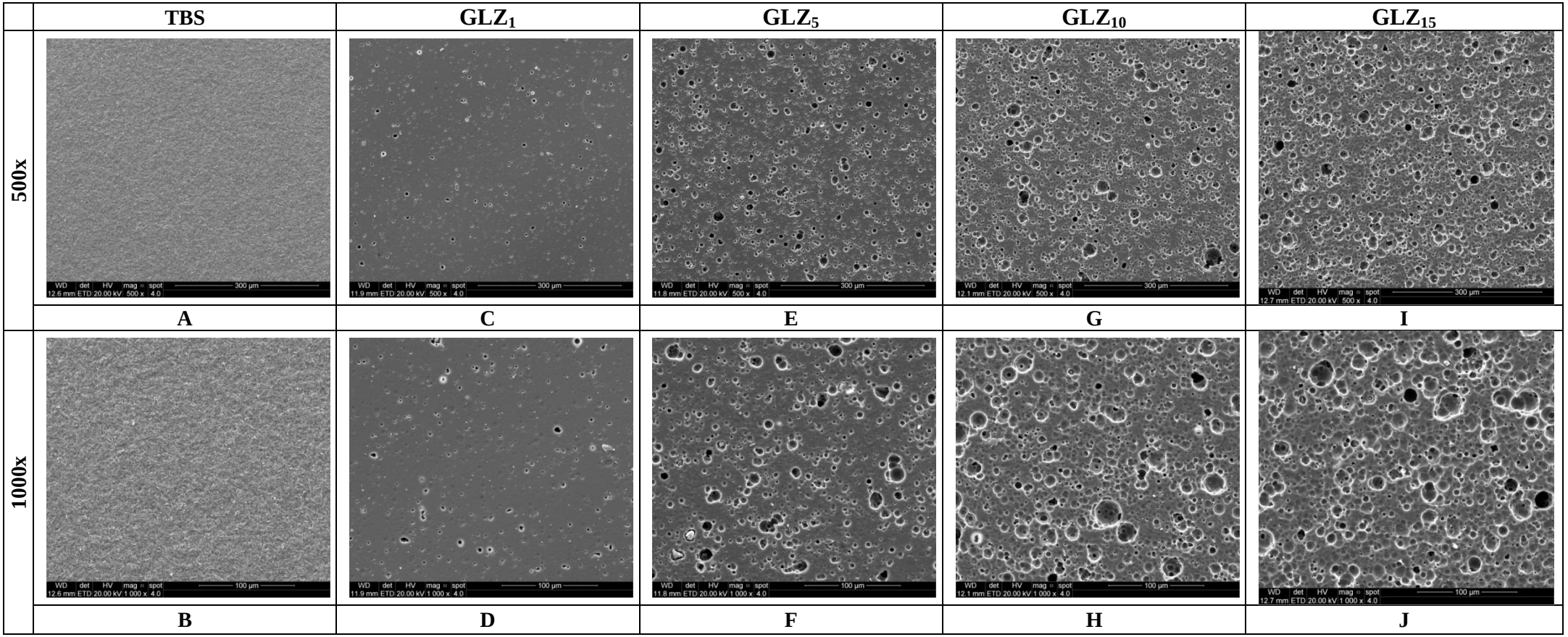

Figure 4. Representative micrographs of the Y-TZP surface alterations after different surface treatments (left to right: 500x, 1000x magnifications. (A, B) TBS group; (C, D) GLZ_1 group; (E, F) GLZ_5 group; (G, H) GLZ_{10} group; (I, J) GLZ_{15} group.

4 DISCUSSÃO

No que diz respeito ao efeito de tratamento de superficial na resistência a flexão de cerâmica Y-TZP, observou-se que grupo TBS apresentou os maiores valores. Já para os grupos com aplicação do glaze, independentemente do tempo de condicionamento, os valores de resistência característica foram menores dentre os grupos avaliados.

Os valores superiores obtidos pelo grupo TBS podem ser explicados através do mecanismo de tenacificação criado pelo impacto das partículas jateadas na superfície da cerâmica o qual leva a uma transformação de fase do material. Junto a essa transformação, um aumento de volume de 3-5% é observado, levando assim a criação de forças compressivas concentradas nas extremidades de trincas presentes (KOSMAC et al., 1999) dificultando a sua propagação. O comportamento inferior dos grupos com aplicação de glaze deve-se principalmente pelo fato da configuração em bicamadas que se forma (GUAZZATO et al., 2005), além das bolhas presentes na região, as quais podem levar a uma concentração de tensão.

Quanto a avaliação da adesão a cerâmica YTZP, notou-se que o grupo TBS também promoveu valores de resistência de união superiores aos outros grupos testados, o que pode ser explicado pela capacidade do jateamento de tornar a superfície dessa cerâmica mais reativa quimicamente, aumentar a rugosidade superficial (KERN et al., 1998), aumentar a área para adesão e modificar a energia de superfície (HALLMAN et al., 2012) além do aumento da molhabilidade e formação das ligações siloxanas criadas pelo uso do agente silano (STANGEL et al., 1987). Nos grupos com glaze, todos os espécimes dos grupos condicionados por 5 e 10 minutos falharam antes do teste. Já nos grupos condicionados por 1 e 15 minutos, os valores de resistência de união foram baixos, mostrando-se esse métodos ineficazes para aumentar os valores de adesão.

Diante dos achados obtidos para os dois parâmetros principais estudados (resistência adesiva e à flexão), o sistema de deposição de sílica por jateamento de partículas de alumina/sílica constitui-se no método mais indicado para materiais a base de Y-TZP, visto que promove adesão mais alta estável entre Y-TZP e cimento resinoso, sem comprometer a resistência do material cerâmico.

5 CONCLUSÃO

- O tratamento triboquímico (grupo TBS) e a deposição de nanofilme de sílica (grupo SNF) não promoveram degradação nas propriedades mecânicas da Y-TZP.

- O grupo TBS apresentou uma maior porcentagem de transformação de fase de tetragonal para monoclínica, contribuindo para o mecanismo de tenacificação do material.

- Foi possível observar uma queda nos valores de resistência à flexão biaxial nos grupos com aplicação do glaze, quando comparada com os outros grupos testados.

- Com relação a adesão, o tratamento triboquímico gerou mais altos e estáveis valores de resistência de união via união micromecânica e adesão química.

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