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Ana Carolina Cadore Rodrigues

EFEITO DO JATEAMENTO COM UM NOVO MATERIAL A BASE DE ALUMINA REVESTIDO POR DIFERENTES CONCENTRAÇÕES DE SILICA NA ADESÃO E NAS PROPRIEDADES MECÂNICAS DA Y-TZP

Santa Maria, RS
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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 Dentária da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para obtenção do grau de **Mestre em Ciências Odontológicas.**

Orientador: Prof. Dr. Luiz Felipe Valandro
Coorientador: Prof. Dr. Gabriel Kalil Rocha Pereira

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DEDICATÓRIA

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"Quem caminha sozinho pode até chegar mais rápido, mas aquele que vai acompanhado, com certeza vai mais longe."

(Clarice Lispector)

RESUMO

EFEITO DO JATEAMENTO COM UM NOVO MATERIAL A BASE DE ALUMINA REVESTIDO POR DIFERENTES CONCENTRAÇÕES DE SILICA NA ADESÃO E NAS PROPRIEDADES MECÂNICAS DA Y-TZP

AUTORA: Ana Carolina Cadore Rodrigues
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Este trabalho está formatado em dois estudos: o objetivo do estudo 1 foi avaliar uma nova abordagem (método Sol-gel) para obtenção de um material alternativo para jateamento (óxido de alumínio revestido por diferentes concentrações de sílica) na resistência de união com e sem envelhecimento entre uma cerâmica Y-TZP e cimento resinoso, considerando diferentes concentrações de sílica. Espécimes de cerâmica Y-TZP ($7 \times 6.3 \times 2$ mm^3) foram confeccionados e embutidos em moldes de PVC com resina acrílica autopolimerizável. Os espécimes foram alocados em 8 grupos (n=20) considerando dois fatores: tratamento de superfície (SiC: partículas de óxido de alumínio revestidas por sílica; AlOx: partículas de óxido de alumínio; 7%Si: material experimental de partículas de óxido de alumínio revestidas por sílica (composto por 93% alumina e 7% sílica); 20%Si: material experimental de partículas de óxido de alumínio revestidas por sílica (composto por 80% alumina e 20% sílica)) e envelhecimento (baseline: 24 horas em estufa a 37°C; ou envelhecido: 90 dias em estufa + 12,000 ciclos, 5-55ºC). O jateamento foi executado com movimentos oscilatórios por 10 segundos a 1 cm de distânica e pressão de 2.8 bar. O silano Monobond Plus foi aplicado na superfície da cerâmica e cilindros de resina composta foram confeccionados e cimentados com Multilink Automix na superfície da Y-TZP e fotopolimerizados. O teste de cisalhamento foi realizado com fio (Ø= 0,5 mm). Na condição baseline todos os grupos apresentaram valores similares de resistência de união. Após envelhecimento, os grupos SiC e 7%Si permaneceram similares aos seus respectivos grupos na condição baseline, enquanto os grupos AlOx e 20%Si apresentaram redução nos valores de resistência de união. O aumento na concentração de sílica de 20% não foi capaz de estabilizar a adesão ao cimento resinoso após envelhecimento e foi similar ao grupo AlOx, entretanto os grupos 7%Si e SiC foram similares e apresentaram melhor desempenho após envelhecimento. O estudo 2 avaliou o efeito do material alternativo para jateamento nas características de superfície (análise topográfica e rugosidade), estabilidade estrutural (transformação de fase $t \rightarrow m$) e comportamento à fadiga (resistência à flexão biaxial) da cerâmica Y-TZP, considerando duas diferentes concentrações de sílica. Discos de cerâmica Y-TZP foram confeccionados conforme a norma ISO 6872:2015 e alocados em 4 grupos (n=30) de acordo com o tratamento de superfície: SiC; AlOx; 7%Si; 20%Si. Os espécimes foram jateados segundo mesmo protocolo descrito para o estudo 1 e o teste de resistência a flexão biaxial foi realizado em fadiga utilizando o método de escada (staircase). O grupo 7%Si apresentou os maiores valores de resistência à fadiga e foi similar ao grupo SiC, enquanto o grupo 20%Si apresentou o pior comportamento a fadiga e foi similar ao grupo AlOx. O aumento na concentração de sílica não promoveu um protocolo mais suave de jateamento, sendo que o grupo 7%Si promoveu um melhor desempenho à fadiga em comparação ao 20%Si.

Palavras-chave: Concentração de sílica. Fadiga. Resistência à Flexão. Resistência de união. Tratamento de superfície. Zircônia policristalina parcialmente estabilizada por óxido de ítrio.

ABSTRACT

EFFECT OF AIR-ABRASION WITH A NEW SILICA-COATED ALUMINA BASED MATERIAL OF DIFFERENT CONCENTRATIONS ON THE ADHESION AND MECHANICAL PROPERTIES OF Y-TZP

AUTHOR: Ana Carolina Cadore Rodrigues
ADVISOR: Luiz Felipe Valandro
CO-ADVISOR: Gabriel Kalil Rocha Pereira

This study is formatted into two parts: the aim of the first study was to evaluate a new approach (Sol-gel approach) to obtain an alternative material for air-abrasion (silica-coated with different concentrations aluminum oxide) on the bond strength with and without long-term aging between resin cement and a Y-TZP ceramic, considering different silica concentrations. Y-TZP ceramic specimens ($7 \times 6.3 \times 2$ mm^3) were made and embedded in PVC molds using chemically activated acrylic resin. The specimens were allocated into 8 groups (n = 20) considering two factors: surface treatment (SiC: silica-coated aluminum oxide particles; AlOx: aluminum oxide particles; 7%Si: experimental material of silica-coated aluminum oxide particles (composed of 93% alumina and 7% silica); 20%Si experimental material of silica-coated aluminum oxide particles (composed of 80% alumina and 20% silica)) and aging (baseline: 24 hours at 37°C in a steam chamber; or aging with 90 days in a steam chamber + 12,000 cycles, 5-55ºC). The air-abrasion was executed for 10 sec at 1 cm distance from the devices tip with oscillatory movements at a pressure of 2.8 bar. The silane coupling agent Monobond Plus was applied on the ceramic surface and resin composite cylinders were made and cemented with Multilink Automix on Y-TZP ceramic and light cured. The shear test was performed using a wire loop (Ø = 0.5 mm). In baseline condition all groups presented similar bond strength values. After aging, the groups SiC and 7%Si remained similar to their counterpart baseline groups, while the groups AlOx and 20% presented a decrease on the bond strength values. A great amount of silica concentration of 20% was not capable to promote stable adhesion to resin cement after aging and it was similar to the AlOx group. However, 7%Si and SiC groups were similar and presented a better performance after aging. The second study evaluated the effect of the alternative material for air-abrasion on the superficial characteristics (topography and roughness), structural stability (t-m phase transformation) and fatigue performance (biaxial flexure fatigue strength) of Y-TZP ceramic, considering two different silica concentrations. Disc-shaped specimens were manufactured according to ISO 6872:2015 and allocated into 4 groups (n = 30) according to the surface treatment: SiC; AlOx; 7%Si; 20%Si. The air-abrasion was executed following the same protocol previously descripted on study 1. The biaxial flexural fatigue strength test was performed using the staircase method. The 7%Si group presented the highest fatigue strength values and it was similar to the SiC group, while the 20%Si group presented the worst fatigue behavior and it was similar to the AlOx group. The increase on silica-coating concentration did not promoted a gentle air-abrasion protocol, and 7%Si group promoted a higher fatigue performance compared to 20%Si.

Keywords: Bond strength. Fatigue. Flexural strength. Silica concentration. Surface treatment. Zirconium oxide partially stabilized by yttrium.

SUMÁRIO

1 INTRODUÇÃO

As cerâmicas à base de zircônia, principalmente a zircônia tetragonal policristalina parcialmente estabilizada com ítrio (Y-TZP), tem sido amplamente estudada e utilizada na odontologia como um material de escolha para infraestrutura de próteses dentais, assim como restaurações monolíticas, devido a sua excelente biocompatibilidade (PICONI; MACCAURO, 1999) e superiores propriedades mecânicas, como alta resistência flexural e tenacidade à fratura (CONRAD; SEONG; PESUN, 2007).

A zircônia é um polimorfo que ocorre na natureza sob três formas cristalinas: monoclínica (m), tetragonal (t) e cúbica (c). A zircônia pura é monoclínica em temperatura ambiente até 1170°C, acima desta temperatura ela se encontra na fase tetragonal e acima de 2370°C ela se encontra na fase cúbica (PICONI; MACCAURO, 1999). Óxidos estabilizadores como cálcio (CaO), magnésio (MgO), céria ($CeO_{2)}$ ou ítria (Y_2O_3) são adicionados a zircônia para que ela se mantenha em fase tetragonal em temperatura ambiente (CHEVALIER et al., 2009).

O notável desempenho da zircônia se deve à transformação da fase tetragonal para a fase monoclínica levando a um aumento de densidade volumétrica da rede cristalina de aproximadamente 3-5% nas regiões de trincas (KOSMAC et al., 1999), devido aos grãos monoclínicos serem mais volumétricos que os tetragonais (HJERPPE et al., 2016). Esse aumento de volume resulta em concentração de tensões compressivas em torno de defeitos superficiais que dificultam a propagação da trinca para o interior do material. Este mecanismo de proteção é conhecido como “tenacificação por transformação” e é responsável por melhorar as propriedades mecânicas da zircônia (HANNINK; KELLY; MUDDLE, 2000) (PICONI; MACCAURO, 1999) (GARVIE; HANNINK; PASCOE, 1975).

Apesar de suas excelentes propriedades mecânicas, a zircônia possui em sua microestrutura alto conteúdo cristalino limitando seu potencial adesivo. A zircônia Y-TZP é classificada como cerâmica ácido-resistente devido à falta de fase vítrea na sua composição (KERN; WEGNER, 1998). Desta forma, a superfície da zircônia não é reativa a ação do ácido fluorídrico, possibilitando limitada união micromecânica (OZCAN; VALLITTU, 2003). Métodos de condicionamento de superfície alternativos foram sugeridos para melhorar a adesão de cimentos resinosos à Y-TZP. O método mais comumente aceito é o uso de jateamento com partículas de óxido de alumínio revestido ou não por sílica (KERN; WEGNER, 1998) que aumenta a rugosidade superficial, modifica a energia de superfície e molhabilidade do material aumentando a retenção mecânica, além de limpar a superfície

cerâmica e remover impurezas (MOON et al., 2016) (PEUTZFELDT; ASMUSSEN, 1988) (OZCAN; VALLITTU, 2003).

As microretenções geradas pelo jateamento fornecem uma união mecânica com os cimentos resinosos e é uma prática clínica comum para o tratamento de restaurações de cerâmica Y-TZP. Este protocolo de jateamento induz um aumento da resistência flexural devido a mudança de fase tetragonal para monoclínica, o que gera um estresse residual compressivo no material (HANNINK; KELLY; MUDDLE, 2000) (PICONI; MACCAURO, 1999) (KARAKOCA; YILMAZ, 2009) (KOSMAC et al., 1999) (GUAZZATO et al., 2005). Entretanto, este efeito gerado pelo tratamento de superfície pode alterar a estabilidade estrutural promovendo danos a superfície da cerâmica Y-TZP (KOSMAC et al., 1999) (ZHANG et al., 2004) (SONG et al., 2013). Estudos propõem que o jateamento introduz falhas superficiais e defeitos que podem agir como concentradores de tensão comprometendo a resistência mecânica dessas restaurações (GUESS et al., 2010) (SONG et al., 2013).

Estudos mostram que um protocolo prejudicial com maior tamanho de partícula associado a maior pressão e menor distância entre o substrato e o dispositivo de jateamento resulta em danos severos na superfície, como trincas, destacamento de grãos e perda de material, além de levar a uma diminuição na resistência mecânica (MOSELE; BORBA, 2014) (GUAZZATO et al., 2005) (WANG; ABOUSHELIB; FEILZER, 2008). De acordo com Zhang e colaboradores (2006) a severidade de abrasão das partículas deve ser minimizada sempre que possível e sugere que, como as partículas de óxido de alumínio são caracterizadas como partículas duras e de formato afiado, o uso de partículas de óxido de alumínio revestido por sílica deve ser explorado devido as suas características de partícula macia e de formato arredondado (ZHANG et al, 2006). Nesse sentido, defeitos profundos podem exercer um efeito negativo na resistência de união entre cerâmica Y-TZP e cimento resinoso devido ao provável aprisionamento de ar e dificuldade no escoamento do cimento, enquanto que defeitos menos profundos e arredondados exercem efeito positivo levando a melhor dispersão do adesivo (HALLMANN et al., 2012).

A energia cinética das partículas no jateamento com óxido de alumínio revestido por sílica produz energia térmica que funde a sílica no substrato cerâmico (OZCAN; PFEIFFER; NERGIZ, 1998) (HALLMANN et al., 2012). A energia cinética das partículas depende da sua massa e velocidade, assim, partículas de menor massa podem não ser capazes de produzir energia térmica suficiente para penetrar sílica na superfície cerâmica (HALLMANN et al., 2012). Desta forma, partículas com maior quantidade de sílica poderiam ser capazes de produzir maior energia cinética penetrando mais sílica no substrato e aumentando a

possibilidade de uniões siloxanas com o agente silano aplicado posteriormente. Além de produzir defeitos menos profundos que possam exercer efeito positivo na resistência de união entre Y-TZP e cimento resinoso. Desta forma, potencialmente o jateamento com óxido de alumínio revestido por sílica pode otimizar retenções micromecânicas para embricamento, assim como aumentar a interação química e maior molhabilidade do substrato pela atuação do silano (OZCAN; BERNASCONI, 2015).

Assim, o principal desafio da aplicação clínica do tratamento de superfície através do jateamento com óxido de alumínio revestido por sílica é encontrar um protocolo capaz de produzir um padrão de microrugosidade superficial que melhore a união com o cimento resinoso sem produzir defeitos que possam comprometer a resistência à fratura da restauração cerâmica. Desta forma, esta Dissertação está apresentada sob a forma de dois artigos:

ARTIGO 1 – "*Air-abrasion using new silica-alumina powders containing different silica concentrations: effect on the bond strength and microstructural characteristics of a Y-TZP ceramic.*" Com o objetivo de avaliar o desempenho de um material alternativo (partículas de óxido de alumínio revestidas por sílica) para jateamento na resistência de união com e sem envelhecimento entre uma cerâmica Y-TZP e um cimento resinoso, considerando diferentes concentrações de sílica.

ARTIGO 2 – *"Air-abrasion using new silica-alumina powders containing different silica concentrations: Effect on the microstructural characteristics and fatigue behavior of a Y-TZP ceramic."* Com o objetivo de avaliar o efeito de um material alternativo (partículas de óxido de alumínio revestidas por sílica) para jateamento nas características superficiais (análise topográfica e rugosidade), estabilidade estrutural (transformação de fase $t \rightarrow m$) e comportamento à fadiga (resistência à fadiga flexural biaxial) da cerâmica Y-TZP, considerando duas diferentes concentrações de sílica.

2. ARTIGO 1 - Air-abrasion using new silica-alumina powders containing different silica concentrations: effect on the bond strength and microstructural characteristics of a Y-TZP ceramic.

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Air-abrasion using new silica-alumina powders containing different silica concentrations: effect on the bond strength and microstructural characteristics of a Y-TZP ceramic

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Running heads: Air-abrasion and adhesion to Y-TZP.

ABSTRACT

This study evaluated the influence of a new air-abrasion material with different silica concentrations (silica-coated aluminum oxide) and aging on the bond strength between resin cement and Y-TZP ceramic. Ceramic slices were randomly allocated into 8 groups (n=20) considering: surface treatment (SiC: silica-coated aluminum oxide particles; AlOx: aluminum oxide particles; 7%Si and 20%Si: experimental materials consisting of 7% and 20% silica-coating of AlOx group material, respectively) and aging (baseline: 24 hours at 37°C in water; aging: 90 days at 37°C in water + 12,000 thermal cycles). A blinded researcher performed the air-abrasion (10 sec). Composite resin cylinders (Ø=3 mm) were cemented onto the silanized ceramic surfaces, light-curing and shear tests were performed (wire loop Ø=0.5 mm). Topography, EDS elemental analysis, contact angle and the powder characteristics were evaluated. Considering baseline condition, all groups presented similar bond strength values. After aging, only SiC and 7%Si remained unaltered and presented lower contact angle values. Surface topography was similar for all air-abraded groups and presented similar silica content, except for AlOx group. 7%Si and SiC were similar and presented better performance after aging, while a greater amount of silica concentration (20%) was not able to promote stable adhesion to resin cement after aging.

Keywords: Silica concentration. Surface treatments. Shear bond strength. Zirconium oxide partially stabilized by yttrium.

INTRODUCTION

Despite their excellent biocompatibility and superior mechanical properties (high flexural strength, fracture toughness and fatigue resistance),[1] Yttrium-stabilized Tetragonal Zirconia Polycrystal (Y-TZP) ceramics have been extensively studied as one of the clinical reasons for failure is debonding.[2] The literature states that it is very difficult to achieve an adequate adhesion to Y-TZP materials[3] since the traditional adhesive technique used for silica-based ceramics (hydrofluoric acid etching) is not effective for Y-TZP ceramics. In addition to the high crystalline content in their composition, Y-TZP ceramics present a high chemical stability which leads to a non-reactive surface.[4,5]

In order to improve adhesion of resin cements to Y-TZP ceramics, alternative surface treatments have been suggested.[4] The most commonly accepted surface treatments are the use of air-abrasion with aluminum oxide or silica-coated aluminum oxide particles; both these methods create a rough surface and increased mechanical retention.[4,5] Due to increased surface energy after these treatments, the Y-TZP ceramic surface becomes more chemically reactive to silane agents,[4,5] consequently increasing the adhesive potential to the Y-TZP surface.[4,5]

Air-abrasion with particles of different sizes and composition may directly affect the surface topography of the Y-TZP ceramic, since the roughness is dependent on the grain size of the abrasive particles (the increase in the roughness is proportional to the increase in particle size).[6,7] Nevertheless, studies evaluating the variation of particle sizes on the bond strength of Y-TZP to resin cement did not find significant differences among the air-abrasion groups with different particle sizes and the control group (no surface treatment), even presenting a significant increase in the surface roughness after air-abrasion.[5,6,8]

The air-abrasion mechanism to promote adhesion varies based on the type of particle employed. The use of aluminum oxide particles results in a micromechanical interlocking based mechanism due to the increased surface roughness.[9] The use of silica-coated aluminum oxide particles generates micro-retentions and leads to a localized temperature increase resulting in silica fusion and its fixation on the Y-TZP surface from the collision of particles to the substrate. In this sense, the substrate becomes more reactive to silane.[10] Until now, it is not known if variations of silica-coating concentrations could interfere in the resin bonding potentiality to Y-TZP ceramics.

In bond strength studies, different aging conditions are performed to estimate the long-term clinical behavior. Thermocycling represents *in vitro* hydrothermal aging and as a

result are expected to have a significant impact on the bond strength, due to the concomitant repetitive contraction expansion stresses that occur inside the specimen or at the interface.[4]

Based on the aforementioned concepts, it becomes relevant to find an adequate silica-coating concentration to optimize the bonding procedure, resulting in a great amount of silica deposition with high kinetic energy to promote the ideal topography pattern and superficial chemical reactivity (by siloxane bonds) without introducing defects that might be deleterious to Y-TZP mechanical properties. Therefore, this study aimed to evaluate an experimental material composed of silica-coated aluminum oxide particles with different silica-coating concentrations (7 and 20%) and compare the performance of the air-abraded Y-TZP ceramics with different powders regarding their bonding to resin cement, with and without long-term aging. The following hypotheses were assumed: 1 - The experimental material with higher silica-coating concentration will lead to higher bond strength results than other available silica-coated aluminum oxide particles after aging; 2 - Air-abrasion with aluminum oxide particles will lead to lower bond strength results than silica-coated aluminum oxide particles after aging; and 3 - The aging will decrease the bond strength of all groups.

MATERIALS AND METHODS

For the present study, the first two groups consisted of commonly available air-abrasion materials in the dental market (Cojet™ System - 3M ESPE; Aluminum Oxide - Polidental). The other two groups consisted of experimental silica-coated aluminum oxide particles produced for this study (Table 1).

Manufacturing of the new silica-coated aluminum oxide particles - Experimental procedure

For manufacturing both experimental silica-coated aluminum oxide particles (7%SiC - 7% silica, 93% aluminum oxide; and 20%SiC - 20% silica, 80% aluminum oxide), the processing conditions were standardized using the same Al_2O_3 particles used in aluminum oxide particles group (AlOx), only modifying the quantity of tetraethyl orthosilicate (Tetraethyl orthosilicate - reagent grade 98%, Aldrich, Wuxi, China). The silica coating on α-Al_2O_3 particles was achieved via the sol-gel approach described by Wang et al. (2005).[11] Aluminum oxide (100 g) was diluted in deionized water (400 mL) and kept under constant agitation for 30 min. The solution remained untouched to hydrate for 24 h. Then, 2 L of ethyl alcohol (95%) were added into the solution and kept under constant agitation for 30 min.

Another solution of 500 mL of anhydrous ethanol (99%) and tetraethyl orthosilicate (100 mL for the 20% experimental material; 35mL for the 7% experimental material) was separately prepared, mixed and carefully added step-by-step to the previous suspension under constant agitation. When all the mixtures were added to the initial solution, ammonia was then carefully added until a pH of 11 was reached. The solution temperature was gradually increased up to 50°C and maintained under constant agitation for a further 6 h. Then, the solution remained in a decantation process in a sealed chamber at room temperature (approximately 27ºC) for 24 h. The liquid was dispensed and the experimental material was washed with alcohol. This decantation process was repeated for 2 consecutive days. The liquid was removed, and the remained material was dried in an oven (80ºC for 4 h).

One hundred grams (100 g) of experimental powder was obtained in each material synthesis process. The process was repeated 3 times for achieving a satisfactory amount of each powder based on the amount of powder used in air-abrading the specimens during a pilot study (3 times in order to obtain 300 g of powder). The powders from all the performed processes were thoroughly mixed with equal percentages to generate a standardized powder mixture.

Sample preparation

Y-TZP ceramic specimens

A hundred and sixty rectangular blocks (≈ 7 × 6.3 × 2 mm^3) were produced from Y-TZP ceramic blocks (IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein). For this, the blocks (B40L – 15.4 x 19.0 x 39.0 mm) were cut into slices (15.4 × 19.0 × 2.5 mm^3) in a precision cutting machine (IsoMet 1000, Buehler, Lake Bluff, USA) under water-cooling. Each slice was cut into four smaller rectangular shaped samples (7.5 × 9 × 2.5 mm^3) using a diamond disc (American Burrs, Palhoça, Brazil) coupled to a handpiece attached to a low-speed micromotor (Kavo Kerr, Joinville, Brazil). The ceramic specimens were manually polished with 1200-grit silicon carbide paper (3M, Sumaré, Brazil) under water to remove any defect introduced by cutting. The specimens were sintered in a furnace (Vita Zyrcomat, Vita Zahnfabrik, Bad Säckingen, Germany) at a temperature of 1530°C for 120 min (as recommended by the manufacturer) and randomly assigned into eight groups (n=20), considering the two study factors “Y-TZP surface treatment” and “aging” (Table 2).

The Y-TZP specimens were embedded in plastic rings with self-curing acrylic resin (JET Clássico, Artigos Odontológicos Clássico, São Paulo, Brazil), keeping the bond surface free from contamination and perpendicular to the vertical plan.

Resin composite cylinders

A stainless-steel piston was used as reference for manufacturing cylindrical matrices of polyvinyl siloxane impression material (Express XT Denso, 3M ESPE, Seefeld, Germany) which guided obtaining standardized resin cylinders (Ø= 3 mm; h= 3 mm). The stainless-steel piston was attached to an adapted surveyor (B2, BioArt) which maintains it in an upright position. Then an impression was taken with the polivinil siloxane material to obtain the cylindrical matrices. Each silicone matrix was used for manufacturing 5 resin cylinders, and then were discarded. The matrix was cleaned with isopropyl alcohol (78%) between each resin cylinder manufacturing.

The resin composite (shade A2, Opallis, FGM) was inserted into the matrix in two increments. Each layer was light-cured (Radii-cal LED curing light, SDI, Bayswater, Australia) for 60 sec. The last layer of resin composite was light-cured with the piston positioned in contact with the resin cylinder and attached to the adapted surveyor; after that, the resin cylinder remained slightly attached to the piston facilitating the cementation procedure.

Air-abrasion protocol and cementing procedure

To perform the surface treatments, the tested powders were placed in recipients by a researcher (G.K.R.P); the vessels were numbered (G1, G2, G3 and G4) containing the materials to be further used for air-abrading the specimens. Thus, the researcher (A.C.C.R) who performed the surface treatment was blinded to the groups.

Prior to air-abrasion, the Y-TZP specimens were ultrasonically cleaned (Vitasonic, Vita Zahnfabrik) with isopropyl alcohol for 5 min. The air-abrasion was executed by the same operator for 10 sec at 1 cm distance from the devices tip with oscillatory movements at a pressure of 2.8 bar.[12] All the specimens were then gentle air-dried to remove any debris.

All the cementation procedures were executed following the manufacturer's guidelines. First, a silane coupling agent (Monobond Plus, Ivoclar Vivadent) was applied over the air-abraded Y-TZP ceramic surfaces for 15 sec and allowed to react for 60 sec; then the excess was removed with a gentle stream of air. Next, the resin cement (Multilink Automix, Ivoclar Vivadent) was mixed, applied onto the resin cylinders (attached to the surveyor) and cemented over the ceramic surfaces. The cement excess was removed, and light curing (Radii-cal LED curing light, SDI) was performed for 20 sec in four directions to the bonded area (0°,

90°, 180° and 270°). The piston was carefully detached from the adapted surveyor with a scalpel to avoid stress on the bond interface.

Storage condition and Shear Bond Strength Tests

For baseline condition (not aged), the specimens were stored in distilled water for 24 h at 37°C prior to testing. The aged samples were stored in distilled water at 37°C for 90 days and subjected to additional thermocycling (12,000 cycles, 5-55°C, 30 s dwel time, 2 s of transfer time from one bath to the other - Nova Etica, Varzea Grande Paulista, Brazil).

The operator for the shear bond strength tests was blinded to the groups. The samples were placed in a jig attached to a universal testing machine (EMIC DL-2000, EMIC, Sao Jose dos Pinhais, Brazil). A wire loop (Ø= 0.5mm) was placed as close as possible to the zirconia ceramic surface for contact with the lower half-circle of the cylinder. The load was applied at a cross-head speed of 1 mm/min. Care was taken to keep the composite cylinder perpendicular to the center of the load cell (50 kgf), in order to maintain the load cell's movement direction perpendicular and as close as possible to the bonding interface (to minimize the risk of resin cohesive fracture). After each group was tested, the wire loop was replaced to standardize its condition (deformation in response to tension) and avoid rupture of the wire loop during the tests. The bond strength *R* (MPa) was calculated according to the formula: $R=F/A$, where "*R*" is the strength (MPa), "*F"* is the load for specimen failure (N) and "*A"* is the cross-sectional interfacial area (A= 7.07 mm^2).

Failure Analysis

The operator for the failure analysis was blinded to the groups. All the tested specimens were firstly analyzed under a stereomicroscope (Discovery V20, Carl Zeiss, Gottingen, Germany) at 15× magnification to categorize the failure type as follows: adhesive (interfacial failure: ceramic/cement interface; or resin/cement interface), predominantly adhesive (more than 50% of the area was interfacial failure, as described by the adhesive pattern) or cohesive (predominantly cohesive failure in resin cement, composite resin or ceramic substrate). Specimens were evaluated under SEM (Vega3, Tescan, Czech Republic) to obtain representative images at 78× magnifications of the failure types.

Topography Analysis

Analysis by SEM (Vega3, Tescan) was performed to determine the topographical pattern of the distinct air-abrasion particles and of the air-abraded Y-TZP surfaces. For this, one

additional specimen of each group and a small portion of each powder were coated with gold-palladium alloy. Images in second electron mode (SE) were obtained at 1000× and 2500× magnification for ceramic specimens, and at 250× and 3500× magnification for the powders.

Elemental composition (EDS)

After performing the previously-described analysis in SE mode, SEM analysis by energy dispersive spectroscopy (EDS) (Vega3, Tescan) was carried out to determine the elemental chemical composition of the air-abrasion powders and on the Y-TZP surfaces after each surface treatment.

Contact angle measurement

Additional Y-TZP specimens (14 × 17 × 2 mm^3) were produced for contact angle analysis (n= 10) and divided into 4 groups according to the surface treatment (Table 2). The air-abrasion was executed as previously described. The contact angle was measured by the sessile drop technique using a goniometer (Drop Shape analysis, model DSA 30S, Krüss GmbH, Hamburg, Germany), which was connected to a computer with a dedicated software program (DSA3, V1 .0.3-08, Kruss GmbH). One drop (11 μl) of distilled water at room temperature (± 24ºC) was placed at the center of each treated ceramic surfaces using a syringe. After 5 sec, 5 contact angle measurements were taken to produce a mean contact angle value for each tested sample.[13,14]

Statistical Analysis

The bond strength data assumed a parametric and homogeneous distribution (assessed by Shapiro Wilk and Levene tests; α=0.05). Data were submitted to Two-way ANOVA and *post-hoc* Bonferroni tests (α=0.05), considering the factors "Y-TZP surface treatment", "aging" and their interaction using the Statistix 8.0 program (Analytical Software, Florida, United States of America).

Contact angle data assumed a parametric and homoscedastic distribution and were submitted to One-way ANOVA and *post-hoc* Tukey's test (α=0.05) using IBM SPSS 21 (IBM Analytics, New York, United States of America).

RESULTS

Two-way ANOVA and Bonferroni tests showed that the type of Y-TZP surface treatment ($p=0.0005$), the aging condition ($p=0.0001$) and the interactions between both parameters ($p=0.0001$) affected the bond strength values (Table 3). All groups in baseline condition were statistically similar. Regarding the comparison between baseline and aged conditions, the SiC and 7%Si aged groups remained similar to their counterpart baseline groups. The AlOx and 20%Si groups presented a decrease in the bond strength values after aging (SiC = SiC aged; 7%Si = 7%Si aged; AlOx > AlOx aged; 20%Si > 20%Si aged).

Only the 'AlOx aged' group presented two pre-test failures during thermocycling (Table 3). In addition, Table 3 shows the distribution of failures: no cohesive failures were found, a higher percentage of adhesive failures for baseline condition and predominantly adhesive failures for aged condition were observed. Figure 1 shows representative images of the adhesive and predominantly adhesive failures.

SEM analysis of the powders (Figure 2) showed a difference in the amount of silica present in the experimental powders, where 7%Si presented a lower concentration of silica clusters than 20%Si, while no clusters were observed for the SiC group. In regards to the air-abraded Y-TZP surfaces, SEM micrographics (Figure 3) showed a uniform air-abrasion pattern and no differences in the topographic pattern were created by any of the air-abrasion powders.

The EDS analysis of the powders confirmed the presence of silica in SiC, 7%Si and 20%Si powders; the absence of silica in the AlOx powder was also confirmed. The 20%Si powder presented a greater amount of silica than the powders SiC and 7%Si. SiC and 7%Si presented the same amount of silica content (Table 4). The amounts of silica deposited on the Y-TZP surface after air-abrasion with the SiC, 7%Si and 20%Si powders seem to be similar (Table 4).

Different air-abrasion powders had a statistically significant influence ($p<0.05$) on the contact angle values (20%Si ≥ AlOx ≥ 7%Si = SiC; Figure 4).

DISCUSSION

Our data support that the air-abrasion with aluminum oxide and high silica concentration particles (20%) presented the lowest bond strength values between Y-TZP ceramic and resin cement after aging. No difference was observed among the air-abrasion powders in the baseline condition, while only the aforementioned groups decreased the bond strength after aging. Thus, the first hypothesis that the experimental material with higher silica-coating

concentration will lead to higher bond strength results than other available silica-coated aluminum oxide particles after aging was rejected; the second hypothesis that the air-abrasion with aluminum oxide particles will lead to lower bond strength results than silica-coated aluminum oxide particles after aging, and the third hypothesis that the aging will decrease the bond strength of all groups were both partially accepted.

The similar bond strength values among the groups in the baseline condition, even with the absence of silica in the AlOx group (Table 4), can be explained by the similar topographic pattern (Figure 3) generated in all the tested groups. Moreover, the resistance to hydrolysis of the bonded surface could not be evaluated in the baseline condition since no significant degradation occurred. Re and collaborators (2008) reported similar bond strength values between Y-TZP and resin cement after air-abrasion with silica-coated aluminum oxide particles and aluminum oxide particles, regardless of particles size, corroborating the findings of our study.[15]

Water storage simulates aging due to water uptake and hydrolytic degradation representing in vitro hydrothermal aging,[4] while the temperature changes simulate the expansion of concomitant repetitive contraction-expansion stresses that occur at the interface. It is expected that both methods have a significant impact on the bond strength values.[4,16] In this sense, it appears that water storage and thermocycling did not similarly influence all the groups, and that silica affinity to silane agent may have influenced those results.

In the SiC and 7%Si groups, the presence of silica on the Y-TZP surface was able to establish a chemical bond via the silane agent that was not broken after aging, presenting similar bond strength values to their counterpart baseline groups (Table 3). Also, SiC and 7%Si groups presented the lowest contact angle values (Figure 4) which should have favored the resin cement wettability because of the enhanced surface area available for adhesion.[4,5] In comparing the bond strength values between groups AlOx and 20%Si in the baseline and its counterpart aged conditions respectively, it was noted that the bond strength was not stable after aging for both air-abrasion treatments.

Despite studies having reported similar bond strength values between Y-TZP and resin cement after air-abrasion with silica-coated aluminum oxide particles and aluminum oxide particles,[15] differences can be observed after 6 months of storage[17] due to the presence of silica in the ceramic surface to stabilize a chemical bond through a silane agent.[5] According to Martins and collaborators (2017), it is likely that roughness is influenced by the particle size and that wettability is determined by particle composition.[18] Thus, particles modified by silica

provide better wettability due to silane, which has a higher chemical affinity to silica than to alumina,[19] explaining the contact angle and bond strength findings.

In relation to the 20%Si group, EDS analysis of the air-abraded Y-TZP surfaces showed a similar amount of silica for SiC, 7%Si and 20%Si experimental materials (Table 4), suggesting that 20%Si was not able to deposit more silica on the Y-TZP surface. Furthermore, the chemical analysis of the powders by energy-dispersive X-ray spectroscopy (EDS) and the micrographics of the powders (SEM) confirmed a greater amount of silica in the experimental material 20%Si than the other silica-coated aluminum oxide powders (Table 4; Figure 2).

During an experimental procedure for obtaining the silica-coated aluminum oxide particles, it is necessary that the mixture remains under constant agitation, allowing the tetraethyl orthosilicate (TEOS) to make contact with the alumina particles in order for the particle coating to occur.[11] It can be hypothesized that the agitation was not enough and/or the TEOS amount was too elevated, resulting in loose silica particles in the material. This fact could explain the high amount of silica in the 20%Si (powder) experimental material and its inability to deposit higher amount of silica on the Y-TZP surface. Loosely adhered particles do not have enough kinetic energy to penetrate the ceramic surface because of their small mass, thereby remaining loosely spread on the ceramic. Since they are easily removed from the surface, these loose particles could be responsible for debonding the polymers from the air-abraded surface substrate.[7]

The present study did not compare the Y-TZP surface treatments with an untreated group because previous studies have already demonstrated the weak adhesion between untreated Y-TZP and resin cements.[20–22] Ceramic restorations are constantly subjected to cyclic loading under wet conditions during chewing functions; however, we did not simulate these conditions in this current investigation.[23,24] In view of the constant challenges that restorative materials are subjected to in the oral environment such as the presence of moisture and masticatory forces, understanding how clinical failures occur is fundamental.[24] However, even with the limitations of an *in vitro* study, our data suggest that the experimental material of aluminum oxide particles coated with 7% silica is a potential alternative for the commercially available silica-coated aluminum oxide particles.

As main limitations of this study, it could be hypothesized that more Y-TZP specimens after air-abrasion should have been subjected to EDS analysis to perform a statistical analysis to corroborate the composition surface after each air-abrasion powders. Regarding the mechanical test, the shear test was selected for its simplicity and facility to evaluate adhesion to Y-TZP substrate. This test method is a quick and repeatable testing

option to rank materials.[4] However, a limitation of the shear test is the non-homogeneous stress distribution, although no cohesive failures were found in the present study.

One of the main existing challenges of the clinical application of this air-abrasion method is to find a protocol to produce a superficial microroughness and chemical reactivity pattern which enhance bonding with the resin cement without producing defects that could compromise the ceramic restoration properties.[5] Thus, future studies to evaluate more rounded particles (with less sharp features) and different silica concentrations and their association with cementation are recommended, as rounded pits with shallow, open and interconnecting structures promoted by air-abrasion have a positive effect leading to better scattering of adhesives on the abraded surface.[7]

In conclusion, a silica concentration of 7% presented better adhesive performance and was similar to the commercially available silica-coated aluminum oxide particles; while air-abrasion of Y-TZP ceramics with aluminum oxide particles and a great amount of silica concentration of 20% was not capable to promote stable adhesion to resin cement after aging.

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TABLES

Table 1 List of materials used: commercial names, manufacturers, batch numbers and composition based on the manufacturer's information.

Material (Commercial name)	**Manufacturer**	**Batch number**	**Composition**
IPS e.max ZirCAD	Ivoclar Vivadent	T42432 V26180	99.5% ZrO_2, Y_2O_3
Aluminum Oxide	Polidental	44493	Aluminum Oxide (45 μm)
Cojet™ System	3M ESPE	566382 411974	Aluminum Oxide (30 μm), Amorphous Silica
Experimental Material 7%	Aluminum Oxide from Polidental modified experimentally	-	Aluminum Oxide (45 μm), Tetraethyl Orthosilicate
Experimental Material 20%	Aluminum Oxide from Polidental modified experimentally	-	Aluminum Oxide (45 μm), Tetraethyl Orthosilicate
Monobond Plus	Ivoclar Vivadent	V12120	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate
Composite Resin (Opallis, shade A2)	FGM Produtos Odontológicos	020317	Bis (GMA), Bis (EMA), UDMA, TEGDMA, Barium-Aluminum, silanized silicate and nanoparticles of silicon dioxide, camphoroquinone, accelerators, stabilizers and pigments
Multilink Automix (Transparent)	Ivoclar Vivadent	V31577 V08514	Dimethacrylate, 2-hydroxyethyl methacrylate, benzoyl peroxide, barium glass, ytterbium trifluoride, spheroid mixed oxide

Table 2 Experimental Design.

Group	Y-TZP Surface Treatment*	Aging**	Particle Size	Silica concentration	N
SiC	Cojet Sand	Without	30 µm	***	20
SiC Aged		With			20
7%Si	Silica-coated aluminum oxide	Without	45 µm	7%	20
7%Si Aged		With			20
AlOx	Aluminum oxide	Without	45 µm	0%	20
AlOx Aged		With			20
20%Si	Silica-coated aluminum oxide	Without	45 µm	20%	20
20%Si Aged		With			20

*Air-abrasion for 10 sec at 1 cm distance with oscillatory movements at 2.8 bar pressure.

**Samples without aging: Baseline condition (24 h of water storage at 37°C before testing); Samples with aging: Aged condition (for 90 days of water storage at 37°C + 12,000 thermocycles 5-55°C before testing).

***Silica concentration do not reported by the manufacturer.

Table 3 Bond strength data (mean ± standard deviation) obtained by the shear bond strength tests and number percentage of each failure type.

Group	Baseline					Aged				
	Bond strength (in MPa)*	Failure Analysis (number of specimens/%)				Bond strength (in MPa)*	Failure analysis (number of specimens/%)			
		ADHES	PRED ADHES	COHE	Pre-test failures		ADHES	PRED ADHES	COHE	Pre-test failures
SiC	27.28 ± 7.35^{A}	16/80%	4/20%	0%	0	31.43 ± 5.08^{A}	1/5%	19/95%	0%	0
7%Si	28.06 ± 8.22^{A}	13/65%	7/35%	0%	0	26.88 ± 5.39^{A}	2/10%	18/90%	0%	0
AlOx	28.15 ± 7.93^{A}	15/75%	5/25%	0%	0	19.69 ± 6.06^{B}	6/33.3%	12/66.7%	0%	2
20%Si	30.13 ±3.55^{A}	2/10%	18/90%	0%	0	19.45 ± 4.42^{B}	11/55%	9/45%	0%	0
Total Failure		46/57.5%	34/42.5%	0/0%			20/25.6%	58/74.4%	0/0%	

Failure analysis: ADHES (interfacial failure: ceramic/cement interface; or resin/cement interface); PRED ADHES (more than 50% of the area was interfacial failure) and COHE (predominantly cohesive failure in resin cement, composite resin or ceramic substrate).

*Different uppercase letters indicate statistical differences by two-way ANOVA and post-hoc Bonferroni tests.

Table 4 Chemical composition (weight in percentage) of the distinct tested powders and of the air-abraded Y-TZP surfaces in each group (obtained by EDS).

Groups	Powder elements (Weight in %)			Air-abraded Y-TZP surface elements (Weight in %)				
	O	**Al**	**Si**	**O**	**Al**	**Si**	**Zr**	**Y**
SiC	54.91	39.27	5.82	37.08	1.78	1.21	59.93	-
7%Si	54.79	38.85	6.36	37.18	2.26	0.97	59.58	-
AlOx	54.11	45.89	-	32.73	1.93	-	64.58	0.76
20%Si	55.96	28.32	15.72	37.67	2.02	1.22	58.73	0.37

FIGURES

Figure 1 Representative images of the failure types under scanning electron microscopy (SEM) at 78× magnifications. A) Adhesive failure B) Predominantly adhesive failure.

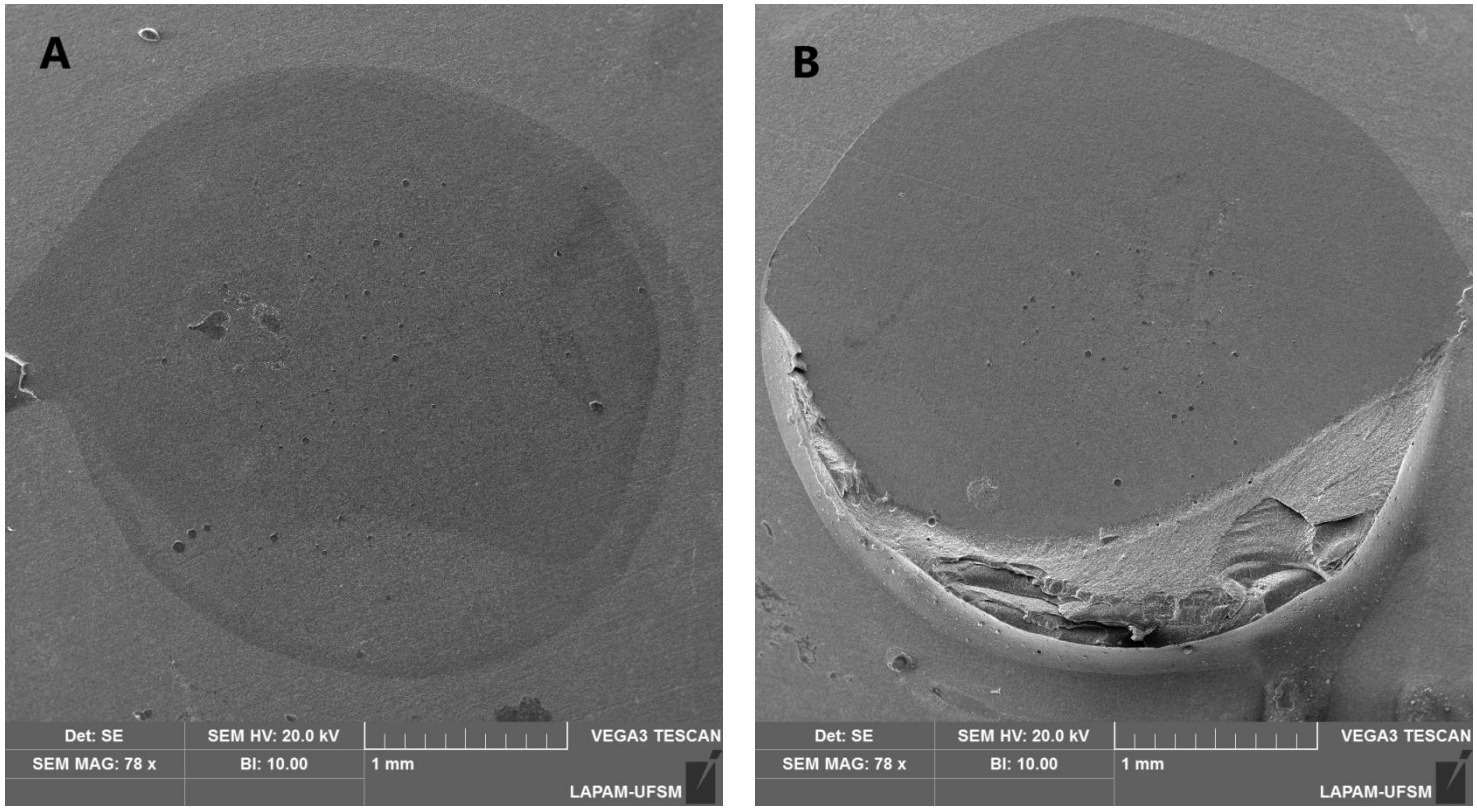


Figure 2 Scanning electron microscopy images of the powders for air-abrasion at 250x and 3500x magnifications.

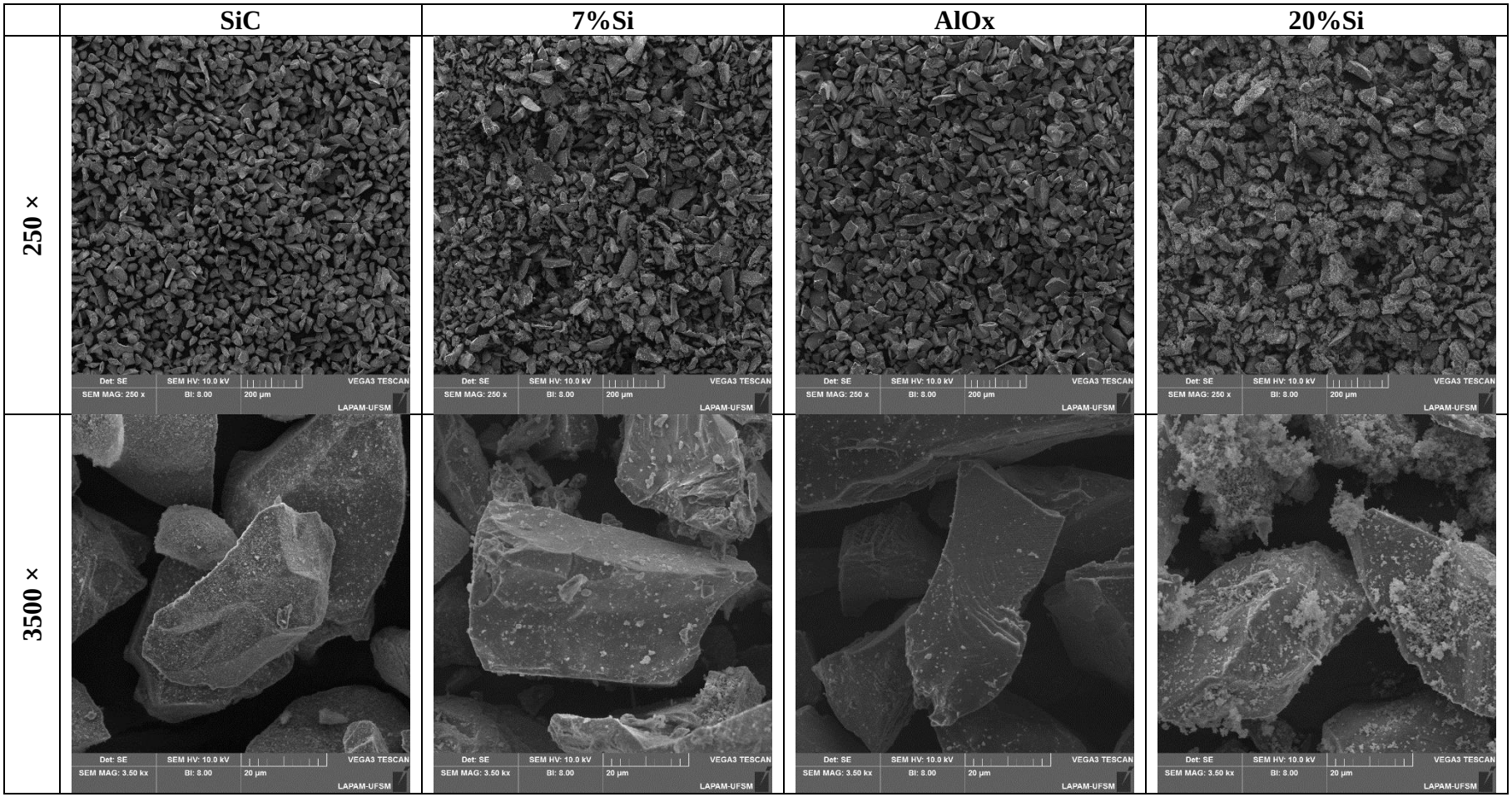


Figure 3 Representative SEM micrographs of the air-abraded Y-TZP ceramic surfaces at 1000× and 2500× magnifications.

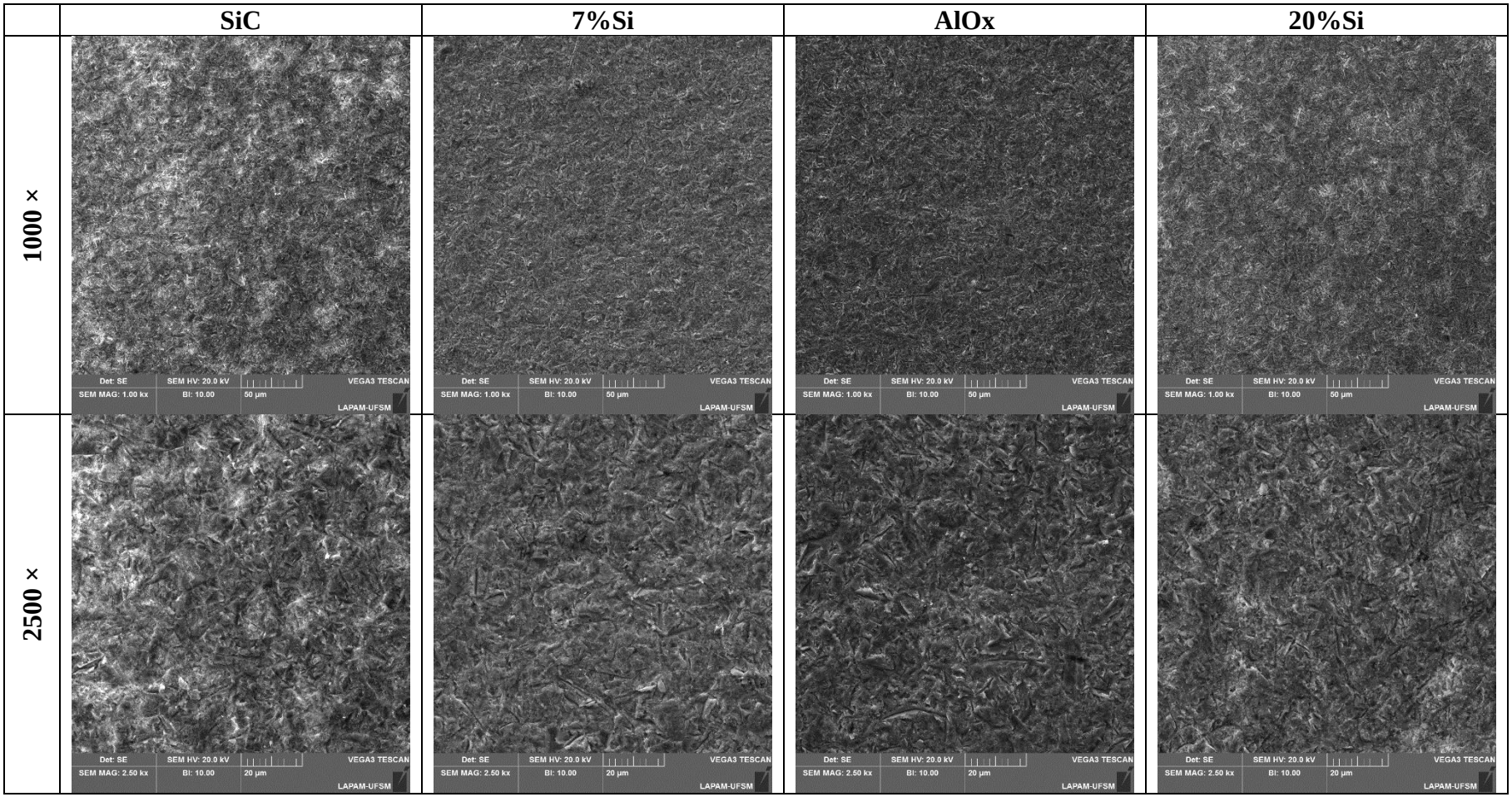
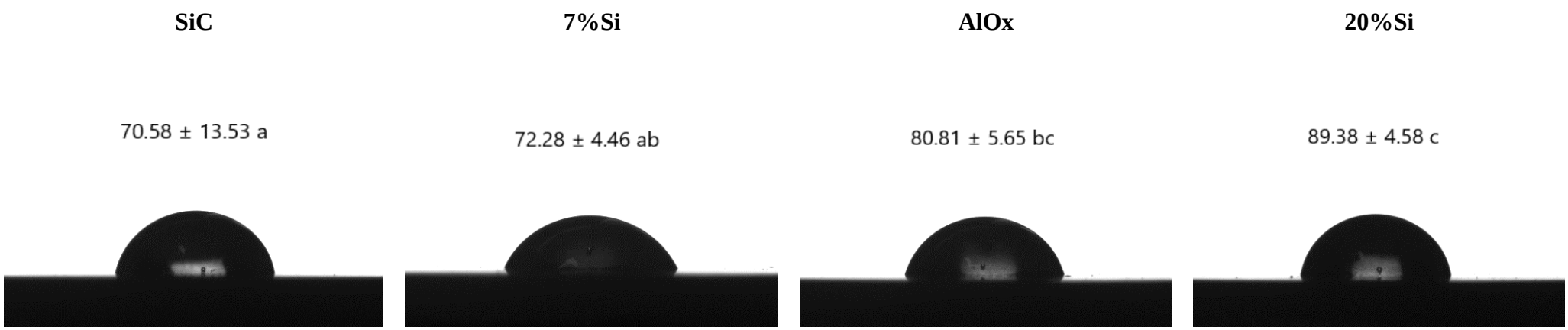


Figure 4 Representative images within mean ± standard deviation of contact angle measurements for the air-abraded Y-TZP surfaces.



*The same letters indicate no significant differences.

3. ARTIGO 2 - Air-abrasion using new silica-alumina powders containing different silica concentrations: Effect on the microstructural characteristics and fatigue behavior of a Y-TZP ceramic.

Este artigo será submetido ao periódico *Dental Materials, Elsevier,* ISSN: 0109-5641, Fator de impacto: 4.039; Qualis A1. As normas para publicação estão descritas no Anexo B.

Air-abrasion using new silica-alumina powders containing different silica concentrations: Effect on the microstructural characteristics and fatigue behavior of a Y-TZP ceramic

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Running title: Air-abrasion and mechanical properties to Y-TZP.

ABSTRACT

Objectives: To assess the fatigue performance (biaxial flexure fatigue strength), surface characteristics (topography and roughness) and structural stability (t-m phase transformation) of an air-abraded Y-TZP ceramic using new powders (7 and 20% silica-coated aluminum oxide particles) in comparison to commercially available powders.

Methods: Disc-shaped specimens were manufactured (ISO 6872-2015) and randomly allocated into four groups considering the air-abrasion materials: SiC: commercially available silica-coated aluminum oxide; AlOx: commercially available aluminum oxide; 7%Si and 20%Si: experimentally produced materials consisting of 7% and 20% silica-coated AlOx, respectively. The air-abrasion was executed for 10 sec by a blinded researcher. The fatigue strength tests (n=15) were performed by staircase method under a piston-on-three-balls assembly. Topography and roughness assessments (n=3) of abraded samples and fractography of failed discs were performed.

Results: The highest fatigue strength (MPa) was observed for 7%Si (887.20±50.54) and SiC (878.16±29.81), while the lowest fatigue strength for 20%Si (773.89±46.44) and AlOx (796.70±46.48). Topography analysis depicted similar surface morphology for all conditions. However, roughness (μm) was only statistically different between 7%Si and SiC, where 7%SiC presented the roughest (*Ra*= 0.30±0.09; *Rz*= 2.31±0.63) surfaces and SiC the smoothest (*Ra*= 0.26±0.04; *Rz*= 1.99±0.34). M-phase grains appeared on Y-TZP surface in a similar content (≈11-12%) for all protocols. Fractography showed all failures starting on air-abraded surface/sub-surface defects from the tensile side.

Significance: In terms of roughness, phase transformation and fatigue, the new 7% silica-coated aluminum oxide presented similar behavior to the commercially available powder. Increasing silica-coating concentration to 20% did not lead to a gentle air-abrasion protocol.

Keywords: Accelerated fatigue. Flexural fatigue strength. Mechanical cycling. Silica concentration. Surface treatment. Zirconium oxide partially stabilized by yttrium.

Highlights

- 7% silica-coating appears to be an adequate alternative to promote a gentle air-abrasion protocol.
- The increase of silica concentration to 20% of aluminum oxide particles did not lead to a gentle protocol.
- Air-abrasion with aluminum oxide and 20% silica-coating led to the worst fatigue performance.

1. Introduction

Yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics are widely used in dentistry for manufacturing framework of metal-free restorations [1] and monolithic *full contour* restorations [2]. This extensive indication is due to their excellent biocompatibility [3] and superior mechanical properties such as high flexural strength, fracture toughness and fatigue resistance [4,5].

Although Y-TZP air-abrasion is related to better results in terms of adhesion, some studies have shown that air-abrasion can lead to superficial defects and t-m phase transformation [6,7]. The impact of air-abrasion particles may lead to significant damage such as extensive erosive wear, lateral cracks and deep defects. If these defects are no longer counteracted by the residual stress introduced by phase transformation mechanism, they may act as stress concentrators, decreasing the final flexural strength of the Y-TZP [8,9]. In this sense, different mechanical tests have been executed to verify the size and severity of defects created by air-abrasion, and also to quantify the cumulative damage to the microstructure and the risk of failure [10,11].

Some studies have depicted that a harmful protocol (bigger grit-sizes in association with higher pressure, closer distances between air-abrading device and ceramic surface, and longer air-abrasion times) leads to a decrease in mechanical strength [8,11]. However, a recent systematic review showed that the particle type, grit-size, pressure and time would generally not deleteriously impact the mechanical properties of Y-TZP ceramic [7]. According to Aurélio and collaborators (2016), the absence of deleterious effects is in response to the toughening mechanism of zirconia by residual stress introduction during t-m phase transformation [7].

Wang and collaborators (2008) showed that the airborne particle abrasion resulted in severe surface damage such as sharp scratches, cracks, grain pull-out, and material loss [12]. The severity of this surface damage was related to the particles size, increasing for 120 μm compared to 50 μm particles size [12]. The depth of the transformed layer mainly depends on the type of air-abrasion particles (size, composition), and it is not related to the air-abrasion time [13]. On the other hand, Guazzato and colleagues (2005) stated that the air-abrasion protocol used for improving bonding between Y-TZP ceramic surface and resin cement may also increase the ceramic's flexural strength since it promotes phase transformation (t- to m-phase) [8]. Despite this, the phase transformation needs to be controlled as it has been

showing that excessive monoclinic phase content decreases the flexural strength of such ceramic material [14].

It has been reported that the alumina air-abrasion introduces deep surface flaws, which can act as stress concentrators [15]. In addition, if a flaw's length extends beyond the surface compressive layer, these stress concentrators will be strength-limiting factors [12]. According to Zhang and collaborators (2006), the severity of particle abrasion should be minimized whenever possible [16]. Therefore, they suggest exploring the use of softer abrasives such as silica-coated aluminum oxide particles (rounded and soft particles), since aluminum oxide particles alone are considered hard and sharp [16]. In this way, the coverage of alumina particles by silica apparently decreases the potential of introducing defects in comparison to its absence, because silica presents a lower hardness than the alumina [17,18].

It is important to highlight that there are not many options of silica-coated aluminum oxide particles for air-abrasion available in the market (e.g. Rocatec Plus – 3M ESPE and Cojet^{TM} System – 3M ESPE). In addition, information addressing the ideal silica content for coating the aluminum oxide particles, as well as the effect of silica coating concentration on the mechanical properties and fatigue strength of Y-TZP ceramics are scarce in literature.

Therefore, to better understand the aforementioned issues, the present study aimed to produce new silica-coated aluminum oxide particles containing different silica concentrations (7% and 20%) through sol-gel approach and to evaluate their effects on the surface characteristics (topography and roughness), structural stability (t-m phase transformation) and fatigue performance (biaxial flexure fatigue strength) of a Y-TZP ceramic. The assumed hypothesis is that an increased silica concentration would generate a gentler and less aggressive air-abrasion protocol and introduce fewer defects onto the Y-TZP ceramic surface, and better fatigue performance would consequently be expected.

2. Materials and methods

For the present study, the first two groups consisted of air-abrasion materials commonly available in the dental market (Cojet^{TM}System – 3M ESPE; Aluminum Oxide – Polidental). The other two groups consisted of experimental silica-coated aluminum oxide particles produced for this study (Table 1).

2.1 Manufacturing of the new silica-coated aluminum oxide particles - Experimental procedure

For manufacturing both experimental silica-coated aluminum oxide particles (7%SiC - 7% silica, 93% aluminum oxide, and 20%SiC - 20% silica, 80% aluminum oxide), the processing conditions were standardized using the same Al_2O_3 particles used in aluminum oxide particles group (AlOx), only modifying the quantity of tetraethyl orthosilicate (Tetraethyl orthosilicate - reagent grade 98%, Aldrich, Wuxi, China). The silica coating on α-Al_2O_3 particles was achieved via the sol-gel approach described by Wang et al. (2005) [19]. Aluminum oxide (100 g) was diluted in deionized water (400 mL) and kept under constant agitation for 30 min. The solution remained untouched to hydrate for 24 h. Then, 2 L of ethyl alcohol (95%) were added into the solution and kept under constant agitation for 30 min.

Another solution of 500 mL of anhydrous ethanol (99%) and tetraethyl orthosilicate (100 mL for the 20% experimental material; 35mL for the 7% experimental material) was separately prepared, mixed and carefully added step-by-step to the previous suspension under constant agitation. When all the mixtures were added to the initial solution, ammonia was then carefully added until a pH of 11 was reached. The solution temperature was gradually increased up to 50°C and maintained under constant agitation for a further 6 h. Then, the solution remained in a decantation process in a sealed chamber at room temperature (approximately 27°C) for 24 h. The liquid was dispensed and the experimental material was washed with alcohol. This decantation process was repeated for 2 consecutive days. The liquid was removed, and the remained material was dried in an oven (80ºC for 4 h).

One hundred grams (100 g) of experimental powder was obtained in each material synthesis process. The process was repeated 3 times for achieving a satisfactory amount of each powder based on the amount of powder used in air-abrading the specimens during a pilot study (3 times in order to obtain 300 g of powder). The powders from all the performed processes were thoroughly mixed of equal percentage to generate a standardized powder mixture.

2.2 Specimen preparation

Disc-shaped specimens were manufactured according to ISO 6872-2015 [20]. Pre-sintered Y-TZP zirconia blocks (IPS e.max ZirCAD B40L, Ivoclar Vivadent, Schaan, Liechtenstein) were shaped into cylinders (Ø= 18 mm; length= 20 mm) using 600 and 1200 grit SiC paper (3M, Sumaré, Brazil) under water-cooling. Next, slices (Ø= 18 mm; thickness= 1.5 mm) were

obtained in a cutting machine under water-cooling (ISOMET 1000, Buehler, Lake Bluff, USA).

The Y-TZP discs were polished with #1200 grit SiC paper to remove any surface irregularities introduced during cutting. Finally, the specimens were sintered in a Vita Zyrcomat furnace (Vita Zahnfabrik, Bad Sackingen, Germany) at a temperature of 1530°C for 120 min. The final dimensions of the disc-shaped specimens were 15 mm in diameter and 1.2 mm (±0,2mm) in thickness (ISO 6872-2015) [20]. They were randomly assigned into four groups according to the air-abrasion protocols (Table 1).

2.3 Surface treatment (Air-abrasion protocol)

The tested powders were placed in recipients by a researcher (G.K.R.P) to perform the surface treatments. The vessels were subsequently numbered (G1, G2, G3 and G4) containing the materials to be further used for air-abrading the discs. Thus, the researcher (A.C.C.R) who performed the surface treatment was blinded to the groups.

Prior to air-abrasion, the Y-TZP discs were ultrasonically cleaned (Vitasonic, Vita Zahnfabrik) with isopropyl alcohol for 5 min. The air-abrasion was executed by the same operator for 10 sec at 1-cm distance from the device's tip with oscillatory movements at a pressure of 2.8 bar [7]. All the specimens were then gentle air-dried to remove any debris.

2.4 Roughness analysis

Before flexural fatigue testing, the surface roughness of all the specimens was measured using a surface roughness tester (n= 30) (Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Japan). The arithmetic mean of two surface roughness measurements was calculated for each specimen according to ISO 4287-1997 [21]. The parameters *Ra* (in μm; arithmetic mean of the absolute roughness values of the peaks and valleys measured from a medium plane) and *Rz* (in μm; average distance between the five highest peaks and five lowest valleys found in the standard) were obtained.

2.5 Topographic analysis

A descriptive analysis on a scanning electron microscope (SEM) (Vega3, Tescan, Brno, Czech Republic) was performed to determine the topographical pattern of the air-abrasion powders and of the abraded Y-TZP surface. For this, additional specimens (n= 3 per condition) were subjected to the particle abrasion, and a small portion of each powder was

coated with gold-palladium alloy. Ceramic surface images were obtained at 5000× and 10000× magnifications, while the powder images were obtained at 500× and 1000× magnifications.

Furthermore, the fractured cross-sectional surface area of representative samples were polished with 800 and 1200 grit SiC paper (3M, Sumaré, Brazil) under water-cooling in a polishing machine (EcoMet 250, Buehler, Germany) and were ultrasonically cleaned (Vitasonic, Vita Zahnfabrik) with isopropyl alcohol for 10 min in order to analyze the effects of the particle abrasion on the zirconia subsurface and surface. The Y-TZP specimens were coated with gold-palladium alloy and images on the second electron mode (SE) were obtained at 1500× and 5000× magnification.

2.6 Phase transformation analysis (X-Ray Diffractometry - XRD)

Quantitative analysis of phase transformation was conducted (n= 2) to determine the relative amount of *m*-phase content generated by each air-abrasion protocol using an X-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany). Spectra were collected in the 2θ range of 25 to 35 degrees (where θ is the angle of incidence relative to the sample surface), at a step interval of 1 s, and step size of 0.03 degrees/step. The monoclinic phase fraction (X_M) was calculated using the method of Garvie & Nicholson (1972) [22]:

$$X_m = \frac{(-111)_M + (111)_M}{(-111)_M + (111)_M + (101)_T} \quad \text{Eq. (1)}$$

where $(-111)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ=28° and 2θ=31.2°, respectively) and $(101)_T$ indicates the intensity of the respective tetragonal peak (2θ=30°). The volumetric fraction of the *m*-phase was calculated according to Toraya et al. (1984) [23]:

$$F_m = \frac{1.311 X_m}{1 + 0.311 X_m} \quad \text{Eq. (2)}$$

2.7 Monotonic biaxial flexural strength tests

Prior to fatigue testing, the mean monotonic biaxial flexural strength (piston-on-three-balls; ISO 6872-2015) [20] was determined from five Y-TZP discs after air-abrasion with each powder in a universal testing machine (EMIC DL 2000, São José dos Pinhais, Brazil). Before testing, an adhesive tape was fixed on the compression side of the discs to avoid spreading the fragments [24], and also to provide better stress distribution between the piston and the sample [25]. Disc-shaped specimens were positioned with the treated surface facing down (tensile side) on three support balls (Ø= 3 mm) under water. A load (1mm/min) was applied

perpendicularly to the center surface of the discs by a flat circular tungsten piston (Ø= 1.6 mm) until catastrophic failure. The monotonic biaxial flexural strength was calculated according to ISO 6872-2015 [20]:

$$\sigma_m = -0.2387P(X-Y)/b^2 \quad \text{Eq. (3)}$$

$$X = (1+\nu)\ln(B/C)^2 + [(1-\nu)/2](B/C)^2 \quad \text{Eq. (4)}$$

$$Y = (1+\nu)\left[1+\ln A/C^2\right] + (1-\nu)(A/C)^2 \quad \text{Eq. (5)}$$

where P is the load at fracture (N), b is the disc thickness (mm), ν is Poisson's ratio (0.25) [26], A is the support ball radius (5 mm), B is the radius of the tip of the piston (0.7 mm), and C is the specimen radius (7.5 mm).

2.8 Biaxial flexural fatigue strength tests

Disc-shaped specimens were subjected to the fatigue tests in an electrical machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, USA) using the same piston-on-three-balls configuration under water, according to ISO 6872-2015 [20], and previously described in section 2.7.

The biaxial flexural fatigue strength was determined after 20,000 cycles using the staircase method described by Collins (1993) [27]. Sinusoidal loading was applied with an amplitude ranging from a minimum of 10 MPa to the maximum tensile for each sample, and a frequency of 20 Hz (20 cycles per second) [28].

The initial stress and step size were determined based on the results of the monotonic biaxial tests for each different test condition (Table 2). The first specimen of each group was tested in this pre-selected stress level, and based on the outcome (survival or failure), while the following specimen was tested with a tensile increment 5% higher (if survived) or lower (if failed) than the initial stress, respectively. This procedure was repeated until at least 15 samples per group were tested (n=15), as previously described by Collins (1993) [27] as the minimum number of tests necessary to obtain a precise estimation using this methodology.

The fatigue tests were controlled by stress, and the load (N) required to achieve the desired stress (MPa) was calculated according to ISO 6872-2015 [20].

2.9 Fractographic analysis

The failed specimens were examined in an optical stereomicroscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany) to determine the fracture origin region. Representative failed samples were evaluated under SEM (Vega3, Tescan) at 200× and 5000× magnifications to

determine the specific crack origin and other fractographical characteristics. Prior to performing this, the ceramic specimens were ultrasonically cleaned (Vitasonic, Vita Zahnfabrik) with isopropyl alcohol for 10 min.

2.10 Statistical Analysis

After assuring a parametric (tested by Shapiro Wilk normality test) and homogenous (tested by Levene homoscedasticity test) distribution of measurements, both the micrometric roughness data (*Ra* and *Rz*) and the fatigue data (all events considered by a staircase approach) were submitted to one-way ANOVA and Tukey's *post-hoc* tests ($\alpha = 0.05$) using IBM SPSS 21 (IBM Analytics, New York, United States of America).

3. Results

3.1 Roughness analysis

Roughness analysis (Table 2) only showed a rougher surface for the 7%Si group than for SiC group. The groups AlOx and 20%Si were statistically similar to 7%Si and SiC groups.

3.2 Topographic analysis

The SEM micrographs of the powders (Figure 1) depicted particles with different sizes and with a sharp edge shape, and the powder particles of the 7%Si and 20%Si groups presented clusters of silica around the alumina particles. Also, the 20%Si group presented higher cluster concentration than the 7%Si group.

The air-abraded Y-TZP surfaces evaluated under SEM (Figure 2) showed an introduction of irregularities due to the impact of the different powder particles during air-abrasion. Quite a similar pattern was also observed for all evaluated conditions.

3.3 Topography of the cross-sectional area

The SEM micrographs of the cross-sectional areas (Figure 3) showed that the powder particles of the 7%Si groups created a regular defects pattern similar to the defects pattern created by the SiC powder particles. The powder particles of the 20%Si groups created more irregular defects which were similar to the AlOx powder particles.

3.4 Phase transformation analysis (X-Ray Diffractometry - XRD)

XRD analysis depicted an m-phase content after air-abrasion similar for all tested groups (Table 2).

3.5 Biaxial flexural fatigue strength tests

The biaxial flexural fatigue strength data (Table 2; Figure 5) showed that the 7%Si and SiC groups presented the highest values. The 20%Si group presented the worst behavior regarding fatigue, which was only statistically equal to the AlOx group.

3.6 Fractographic analysis

Fractographic analysis (Figure 4) showed that all fractures originated from surface/sub-surface defects at the center of the air-abraded specimen side (region of concentrated tensile stresses), in agreement with the ISO 6872-2015 statement [20].

4. Discussion

The tested hypothesis that an increase in silica concentration would generate a gentler protocol of air-abrasion with less defects on the Y-TZP ceramic surface and consequently improved fatigue performance was rejected, as 20% silica-coating concentration led to the worst fatigue performance, and was only similar to the group air-abraded with aluminum oxide (absence of silica).

The importance of air-abrasion on Y-TZP ceramics to promote stable adhesion to tooth substrates is a well-known fact nowadays [6,29,30]. Air-abrasion has been showing the best bonding results [29,30], however particles (powders) with different characteristics (composition, hardness, sizes) are available [31,32]. Also, different adhesive interaction mechanisms to the Y-TZP surface will impact the final bond strength between Y-TZP and resin cements [9]. Until now, there has been no consensus available to guide clinician's choices regarding the best particle and air-abrasion protocol aiming for predictability and adhesion longevity.

Despite promoting bond durability, air-abrasion protocols are related to an introduction of surface defects and the potential to compromise the mechanical properties of Y-TZP ceramics [8,11]. Basically, air-abrasion can trigger superficial Y-TZP grains to transform from tetragonal to monoclinic (t-m) phase, introducing compressive residual stress and defects on the ceramic surface [8,33].

The amount of t-m phase transformation is directly related to the final strength of Y-TZP ceramics [4,33]; the presence of monoclinic phase indicates introducing compressive residual stresses, and the formation of a transformed zone is related to increased flexural strength [34]. However, if the t-m transformation generated during air-abrasion is followed by the occurrence of surface cracks deeper than the compressive residual stress layer, a deleterious impact on the Y-TZP mechanical properties may be observed [10,35].

Additionally, the influence of intrinsic characteristics of the particles used for air-abrasion on the mechanical properties of Y-TZP ceramic is scarce [9]. According to Zhang and collaborators (2006), the severity of hard and sharp air-abrasion particles should be minimized whenever possible [16]. Based on our data, it was observed that the silica concentration used for coating the aluminum oxide particles affected the flexural fatigue strength and the final superficial roughness of the air-abraded Y-TZP ceramics. However, the macroscopic topographical pattern and the amount of t-m phase transformation triggered were not affected.

Previous studies have shown that silica-coated aluminum oxide particles for air-abrasion only led to higher mechanical performance than aluminum oxide particles [17,18]. Our data corroborates that assumption, as the air-abrasion with aluminum oxide particles (AlOx group) introduced surface microcracks, which can act as sources of failure [16]. However, it appears that higher silica concentration also leads to a deleterious effect, as 20%Si silica-coated experimental powder presented statistically similar fatigue strength data to AlOx (Table 2).

The micrographs of the powders (Figure 1) showed particles with different sizes and with a sharp-edge shape for the tested groups. The powder particles of the 7%Si and 20%Si groups also presented clusters of silica particles around the alumina particles, with bigger clusters for 20%Si being detected. Even when depicting different powder patterns, the Y-TZP superficial topography generated in response to air-abrasion by the different tested groups was similar (Figure 2). Thus, a great amount of silica content in the particles was not able to cause lower superficial change.

Air-abrasion is a method based on particles colliding with the substrate creating micro-retentions [36]. It is reported that when air-abrasion is executed with silica-coated particles, the procedure leads to a localized temperature increase that results in the fusion of silica and its fixation on the surface [36]. This procedure makes the zirconia substrate more reactive to a silane coupling agent application [36]. The fixation of the abrasive particles is related to their

kinetic energy, which depends on their mass and velocity [9]. In this study, the same aluminum oxide particles were used to obtain the experimental powders (7%Si and 20%Si groups) and the AlOx group. It was expected that the experimental material particles would present more mass than the original material, therefore it would present less velocity since the air-abrading protocols were executed with the same air pressure, and it was expected that lighter particles (AlOx) assumed higher velocity, and due to hard particles also more defects on the surface. Furthermore, silica particles are known to be softer than aluminum oxide, thereby decreasing the impact on the zirconia surface [17,18]. Hypothetically, lower defects on the Y-TZP surface should be seen using those materials; however, our data do not corroborate that hypothesis. The alterations observed may be so slight that an influence on the defects population, roughness, topography and mechanical properties becomes minimum.

The powder particles of the 7%Si group created a regular defects pattern on the Y-TZP surface (Figure 3) and it was similar to the defects pattern created by the SiC powder particles, which can explain the similar flexural strength. However, the powder particles of the 20%Si groups created more irregular defects, and similar only to the AlOx powder particles. These irregular defects probably acted as stress concentrators and decreased the flexural strength of the tested Y-TZP ceramic [9,15].

In our study, a similar amount of monoclinic phase was found among the tested groups (11.43 up to 12.52%; Table 2). These results are in accordance with the amount reported by other authors who found quantities between 10% and 15% [8,13,37]. According to Chintapalli and collaborators (2013), the air-abrasion process causes material erosion on the surface; meanwhile, part of the material transformed under the first impact is eroded during the subsequent impact [35]. From this, the remaining transformed surface phase is not much different for different particle sizes, explaining the similar phase transformation values regardless of the silica concentration in the particles. In summary, our data point out that more effort should be taken to characterize the ideal silica concentration and shape of the air-abrasion particles, optimizing the silica-coated aluminum oxide particle technique and achieving better mechanical and adhesive performance.

The strength of ceramics depends on the size, number and distribution of defects in the area where the highest tensile stress is concentrated, as well as the capacity of the material to withstand crack propagation [38]. We noted that all fractures originated from surface defects at the center of the tensile side during the tests [24], indicating that the fractures did not occur

due to possible high stresses concentration at the contact surface between the discs and the piston. Also, this failure pattern found was similar for all groups (Figure 4).

Even with the limitations of an *in vitro* study, our data suggest that the experimental silica-coated aluminum oxide particles using the 7% silica approach are a potential alternative for the commercially available silica-coated aluminum oxide particles. However, we did not consider a scenario of associating factors such as air-abrasion and cementation. Only axial loads were applied in conducting the fatigue tests, and some clinical conditions were not simulated such as lateral forces and sliding that may occur clinically during mastication or clenching. Thus, these findings should be considered carefully, and future studies evaluating silica concentration between 7 and 20% associated with adhesive cementation and employing different testing scenarios (which would attempt to mimic different clinical characteristics) are recommended.

5. Conclusions

- Air-abrasion with silica-coated particles of the tested Y-TZP ceramic led to higher fatigue flexural strength, with the exception of 20% silica concentration.
- Among the new silica-coating concentrations tested, 7% promoted better mechanical performance than 20% concentration, although the roughness pattern was similar between them.
- Increasing the silica-coating concentration to 20% of the aluminum oxide particles for air-abrasion did not lead to a gentler protocol.

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Tables

Table 1 Experimental Design

Groups	Y-TZP Surface Treatment	Powder Manufacturer	Particle Size	Silica concentration
AlOx	Aluminum Oxide	Polidental	45 µm	0%
SiC	Cojet Sand	3M Espe	30 µm	*
7%Si	7% Silica-coated aluminum oxide	Aluminum Oxide from Polidental modified experimentally	45 µm	7%
20%Si	20% Silica-coated aluminum oxide		45 µm	20%

*Silica concentration did not reported by the manufacturer

Table 2 Roughness data: mean (standard deviation - SD) of *Ra* and *Rz* parameters; Phase transformation analysis for m-phase content assessment under XRD depicting mean % (standard deviation); Monotonic biaxial flexural strength data: mean (standard deviation); Biaxial flexural fatigue strength findings: initial strength (correspondent to 70% of mean monotonic biaxial strength), step size (correspondent to 5% of mean monotonic biaxial strength) and fatigue strength results (mean and standard deviation).

Groups	Roughness Analysis (µm)		XRD analysis (m-phase %)	Monotonic biaxial flexural strength data (MPa)	Biaxial flexural fatigue strength findings (MPa)		
	Ra (µ*m*)	*Rz* (µ*m*)			Initial strength	Step size	Fatigue strength
AlOx	0.30 (0.05)^{AB}	2.18 (0.37)^{AB}	12.52 (0.30)	1141.91 (83.7)	799.37	39.97	796.70 (46.48)^{B}
SiC	0.26 (0.04)^{A}	1.99 (0.34)^{A}	11.43 (1.72)	1210.15 (93.3)	847.10	42.36	878.16 (29.81)^{A}
7%Si	0.30 (0.09)^{B}	2.31 (0.63)^{B}	11.56 (0.21)	1169.93 (32.0)	818.95	40.95	887.20 (50.54)^{A}
20%Si	0.28 (0.06)^{AB}	2.22 (0.44)^{AB}	12.16 (1.31)	1179.26 (58.5)	825.48	41.27	773.89 (46.44)^{B}

*Different uppercase letters indicate statistical differences.

Figures

Figure 1 Scanning electron microscopy images of the tested powders at 500× and 1,000× magnifications. It can be noted that the groups 7%Si and 20%Si present clusters of silica around the alumina particles, being that the group 20%Si present higher cluster concentration than the group 7%Si.

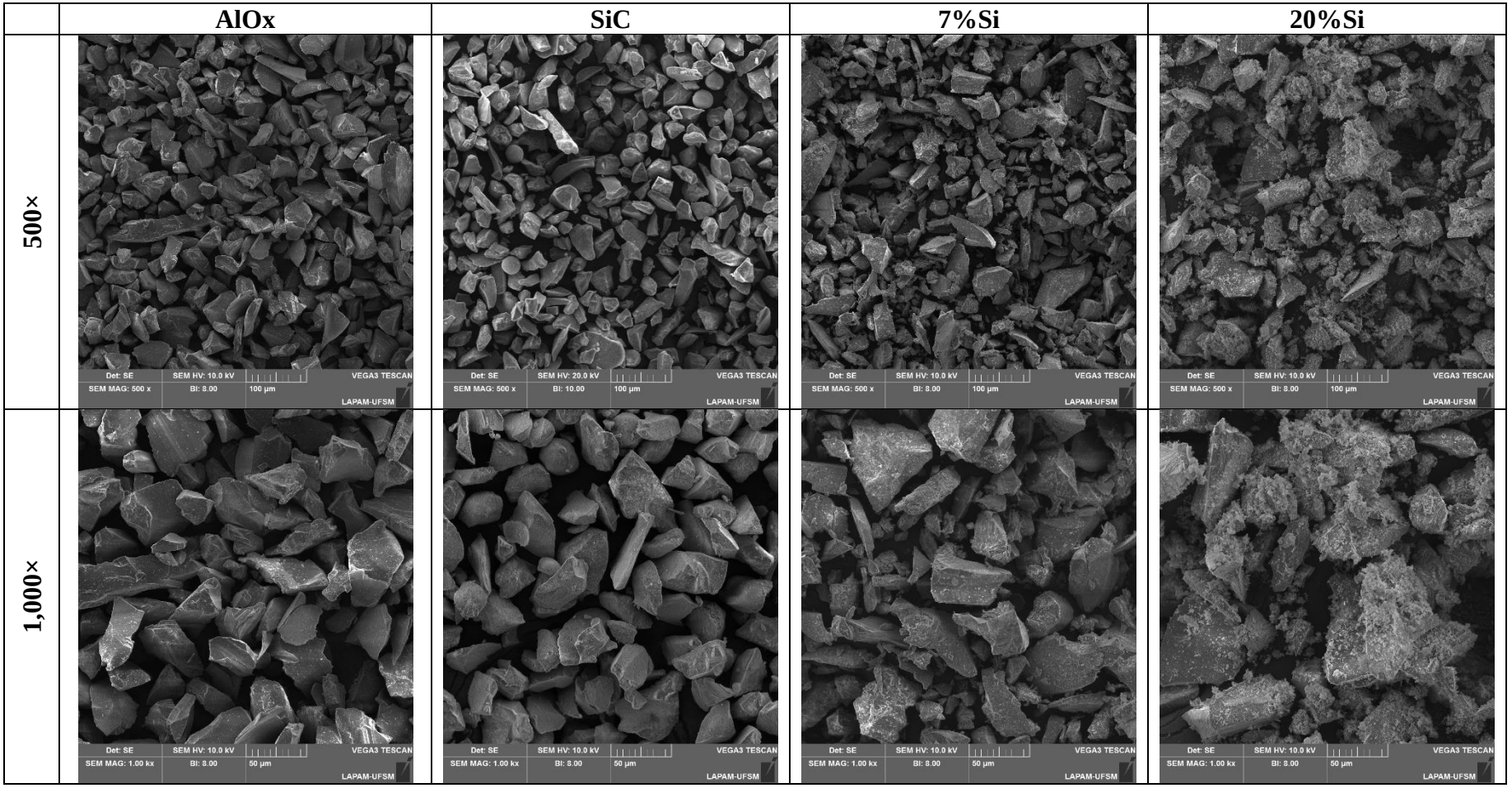


Figure 2 Representative SEM micrographs of the Y-TZP ceramic surfaces after air-abrasion treatments (5,000× and 10,000× of magnification). It can be noted a topographical pattern with similar irregularities for all evaluated conditions.

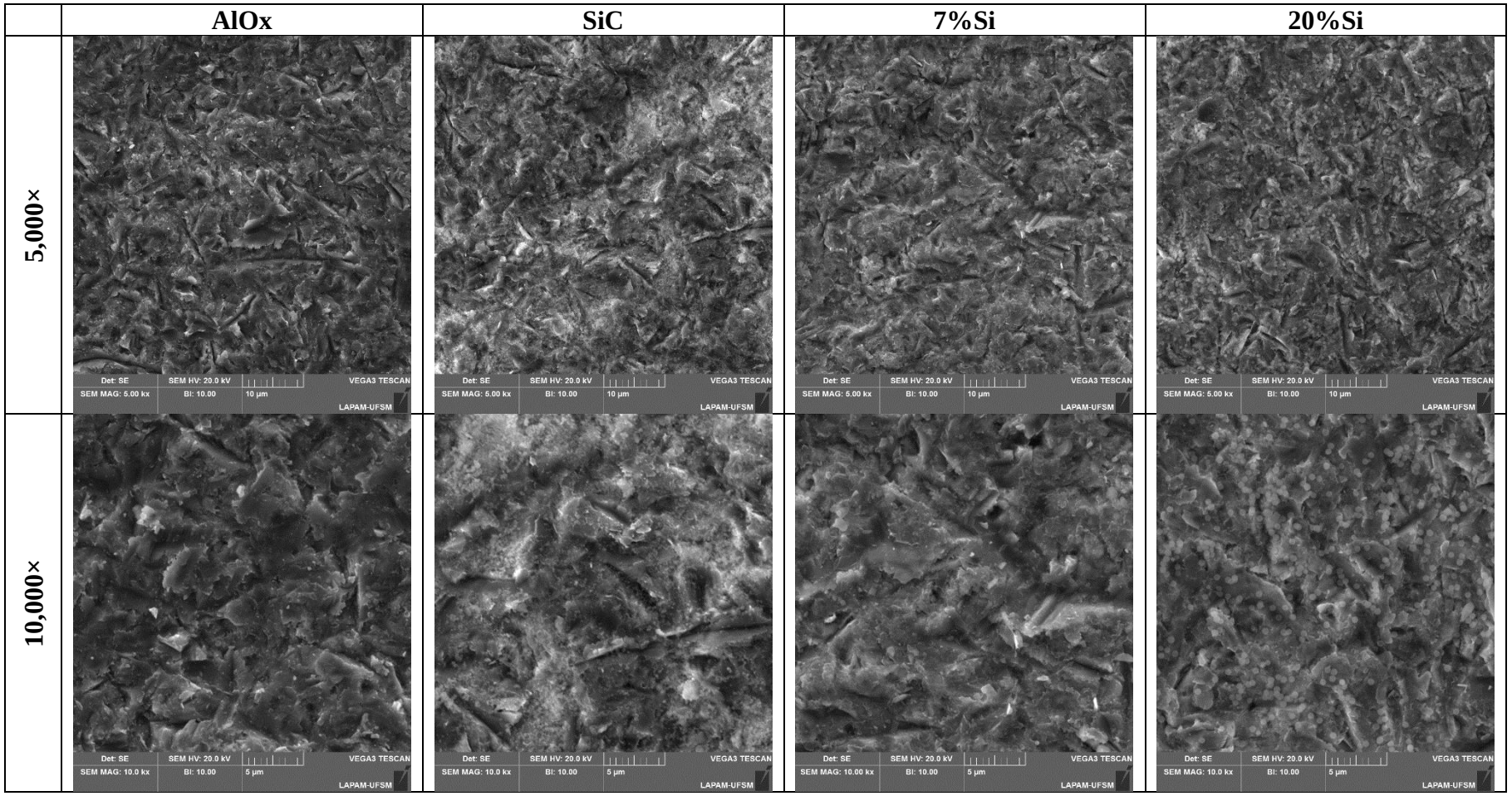


Figure 3 Topographical features of the subsurface (edge zone) of the cross-sectional area of the Y-TZP ceramic surfaces after air-abrasion treatments under 1,500× and 5,000× magnifications. It can be noted slight differences in regards of shape and distribution of defects where the group 7%Si and SiC created a more regular defect distribution, meanwhile the group 20%Si and AlOx introduced areas of almost not alteration (very slight altered surface) and localized areas of deeper influence (deeper defect).

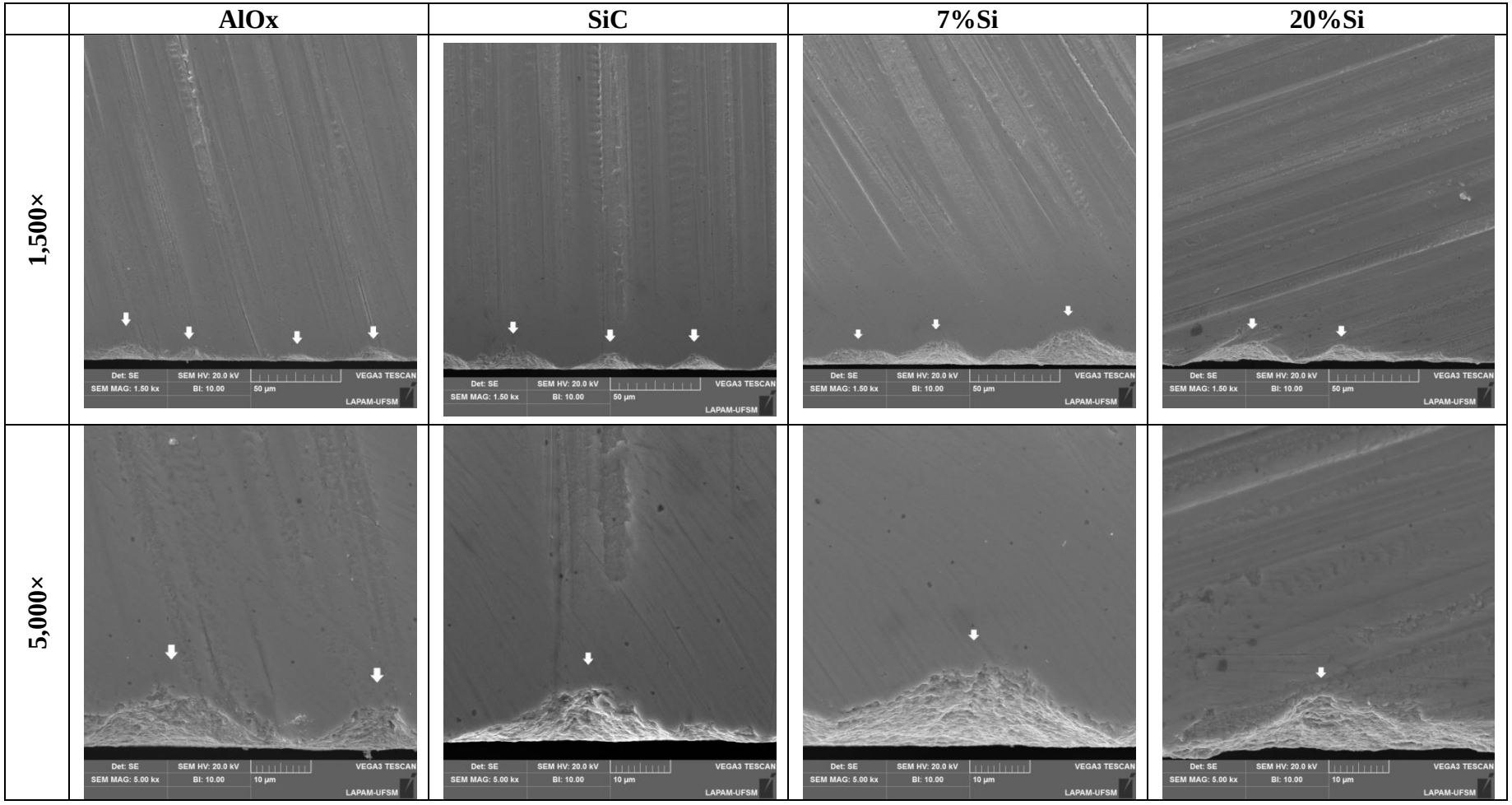


Figure 4 Representative SEM micrographics of fractured surfaces (fractographical examination) at 200× and 5,000× magnifications. The arrows indicate the crack origin at a superficial defect where tensile stress was concentrated. It can be noted that all fractures originated from surface/sub-surface defects at the center of the air-abraded specimen side (tensile side).

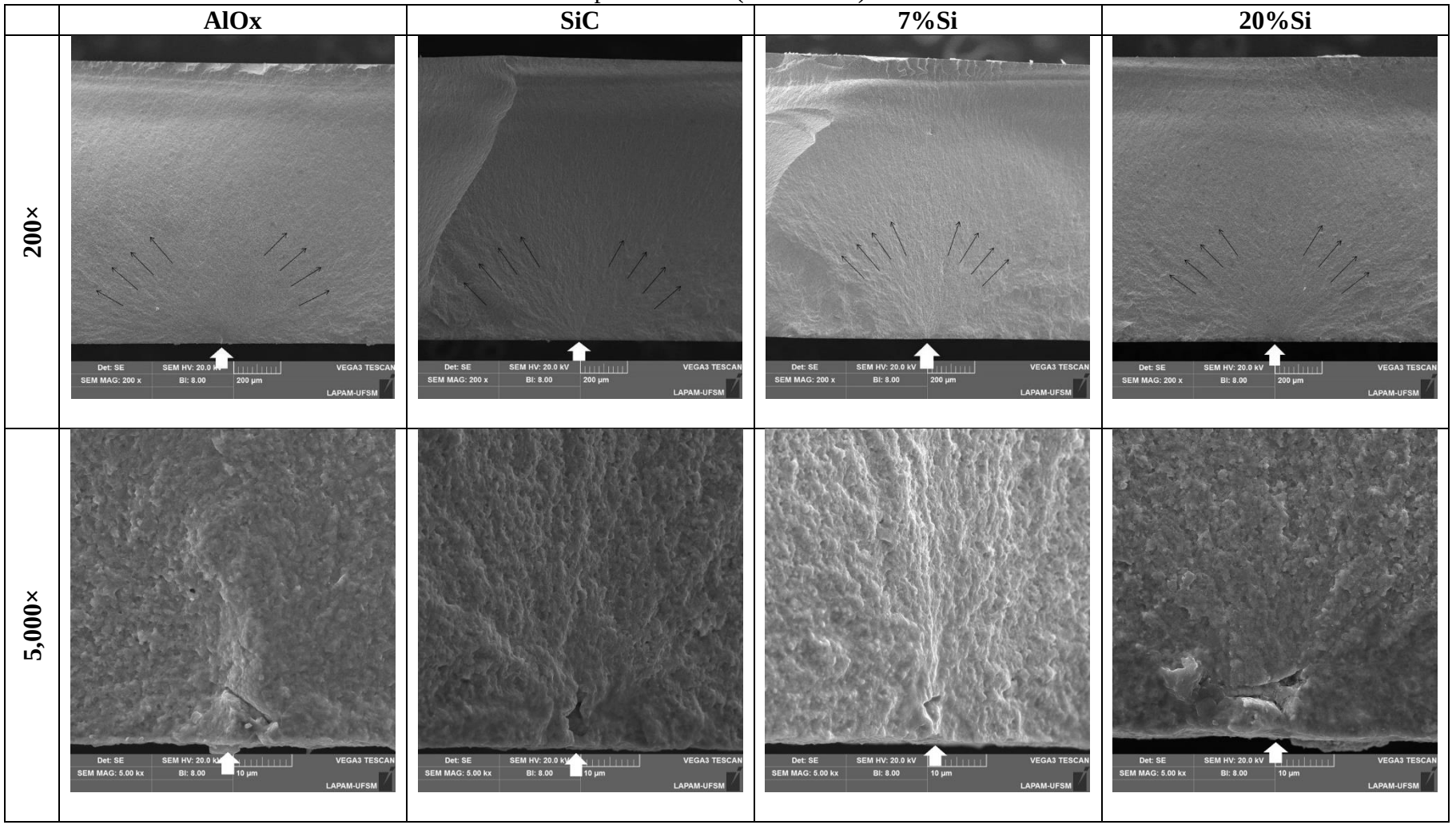
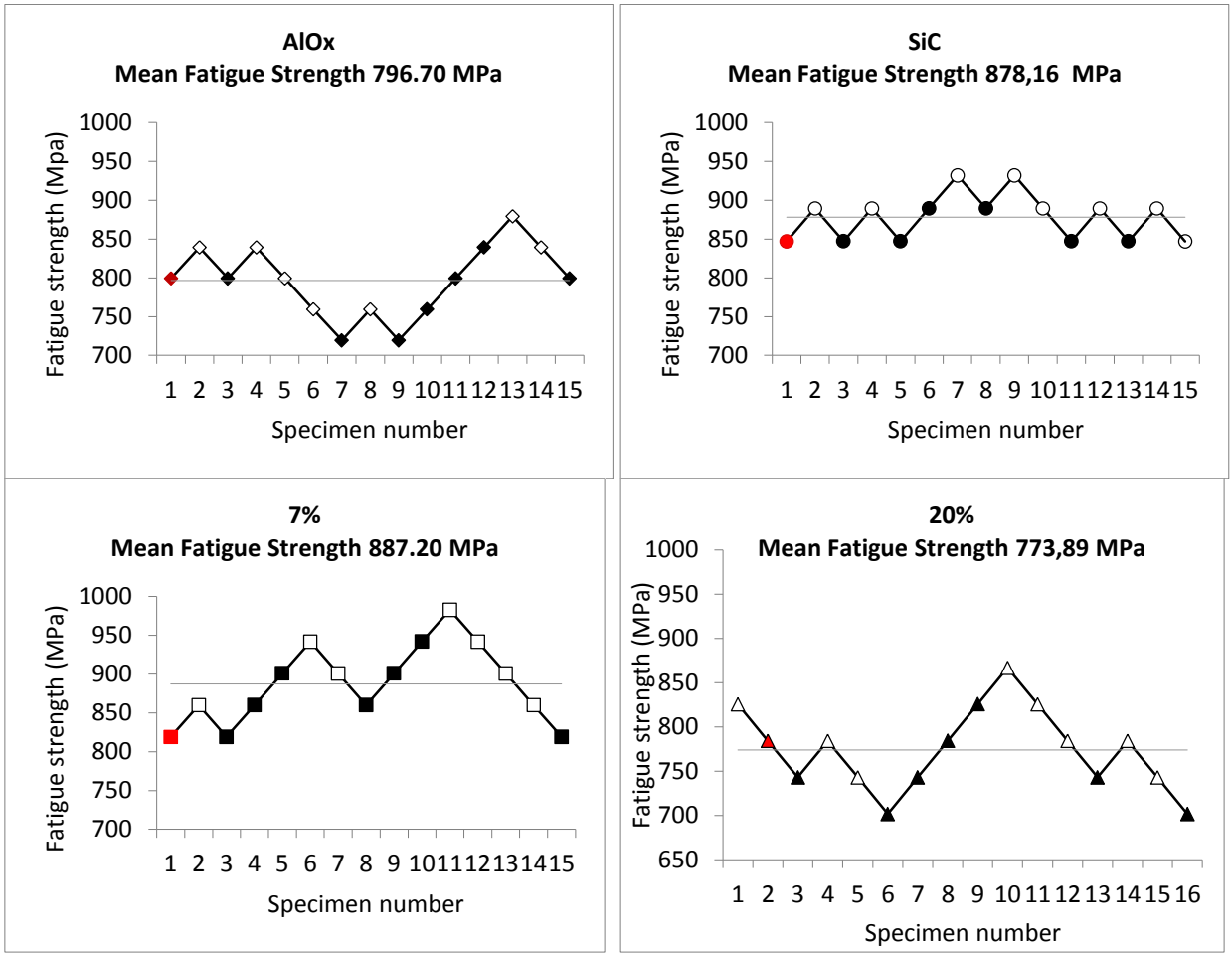


Figure 5 Pattern of survival and failures observed after the fatigue tests: empty marks are failures, filled marks are survivals, the gray lines indicate the mean, and the red mark shows the beginning the staircase test (first stair reversal).



4. DISCUSSÃO

Cerâmicas de zircônia tetragonal policristalina parcialmente estabilizada com ítrio (Y-TZP) têm sido amplamente utilizadas na odontologia devido a sua excelente biocompatibilidade e superiores propriedades mecânicas (PICONI; MACCAURO, 1999; CONRAD; SEONG; PESUN, 2007). Sua microestrutura com alto conteúdo cristalino possibilita limitada união micromecânica devido sua superfície não ser reativa a ação do ácido fluorídrico (OZCAN; VALLITTU, 2003; OZCAN; BERNASCONI, 2015). Assim, o jateamento com partículas de óxido de alumínio revestidas por sílica, além de gerar microrretenções, fixa a sílica na superfície, tornando a cerâmica quimicamente mais reativa ao agente silano (OZCAN; PFEIFFER; NERGIZ, 1998; OZCAN; BERNASCONI, 2015; MOSELE; BORBA, 2014).

Entretanto, o efeito gerado pelo tratamento de superfície pode alterar a estabilidade estrutural promovendo danos a superfície da cerâmica (KOSMAC et al., 1999) (ZHANG et al., 2004) (SONG et al., 2013), como trincas, destacamento de grãos, perda de material, diminuição na resistência mecânica (MOSELE; BORBA, 2014) (GUAZZATO et al., 2005) (WANG; ABOUSHELIB; FEILZER, 2008), além de produzir defeitos profundos que podem exercer efeito negativo na resistência de união (HALLMANN et al., 2012). Desta forma, a presente dissertação buscou avaliar o desempenho de partículas com diferentes concentrações de sílica para aumentar a possibilidade de uniões siloxanas além de produzir defeitos menos profundos que possam comprometer a resistência à fratura da restauração cerâmica.

Os resultados apresentados no artigo 1 da presente dissertação *(Air-abrasion using new silica-alumina powders containing different silica concentrations: effect on the bond strength and microstructural characteristics of a Y-TZP ceramic)* indicam que o jateamento com uma concentração de sílica de 20% não apresenta uma maior estabilidade de união ao cimento resinoso e apresenta desempenho similar as partículas de óxido de alumínio não revestidas por sílica. Entretanto, o jateamento com uma concentração de sílica de 7% apresenta um melhor desempenho adesivo e é similar ao jateamento com as partículas de óxido de alumínio revestidas por sílica comercialmente disponível tanto em resistência de união quanto em estabilidade de adesão.

Após o jateamento, a similaridade na porcentagem de sílica na superfície cerâmica, entre todas as condições baseadas em jateamento com partículas revestidas por sílica indica uma incapacidade do material experimental 20% depositar mais sílica no substrato cerâmico. Este fato pode ser causado por uma deficiência no revestimento da partícula de óxido de

alumínio durante o processo de síntese do material. Para fabricação de ambos os materiais experimentais foram utilizadas as mesmas partículas de óxido de alumínio, dessa forma pode-se notar que as partículas apresentam o mesmo formato e correspondem ao mesmo padrão de alteração superficial criado entre os grupos.

Apesar da similaridade no padrão de alteração superficial e na porcentagem de sílica na superfície cerâmica, o jateamento com o material experimental 7% proporcionou um melhor desempenho na estabilidade de união com o cimento resinoso e parece ser similar ao jateamento com as partículas de óxido de alumínio revestidas por sílica comercialmente disponível. Ambos apresentaram uma maior capacidade de molhamento superficial tornando a superfície cerâmica quimicamente mais reativa ao agente silano e consequentemente aumentando a resistência de união à superfície da Y-TZP (OZCAN; BERNASCONI, 2015; MOSELE; BORBA, 2014).

Tendo-se em vista que defeitos menos profundos podem exercer efeito positivo na resistência de união devido a melhor dispersão do adesivo (HALLMANN et al., 2012), o estudo de arredondamento de partículas para jateamento deve ser considerado. Considerando-se o oposto, de que defeitos superficiais profundos atuam como concentradores de tensão (CURTIS; WRIGHT; FLEMING, 2006), o segundo estudo dessa dissertação foi empregado para avaliar se uma maior concentração de sílica proporcionaria um protocolo menos agressivo, com menor introdução de defeitos e consequentemente um melhor desempenho em fadiga.

Os resultados apresentados no artigo 2 desta dissertação (*Air-abrasion using new silica-alumina powders containing different silica concentrations: Effect on the microstructural characteristics and fatigue behavior of a Y-TZP ceramic*) indicam que uma maior concentração de sílica de 20% desempenha um comportamento mecânico inferior a uma concentração de sílica de 7%, o qual apresenta um superior comportamento em fadiga sendo similar ao desempenho apresentado ao jateamento com as partículas de óxido de alumínio revestidas por sílica comercialmente disponível.

A resistência da cerâmica Y-TZP é diretamente relacionada à quantidade de transformação de fase t-m (KOSMAC et al., 1999; HANNINK; KELLY; MUDDLE, 2000); a presença de fase monoclínica indica uma introdução de estresse residual compressivo e a formação de uma zona transformada correlacionada com o aumento da resistência flexural. Entretanto, uma similaridade na porcentagem de transformação de fase foi encontrada, pois parte do material que é transformado no primeiro impacto do jateamento é erodido durante os

impactos subsequentes (CHINTAPALLI et al., 2013) independente da quantidade de concentração de sílica das partículas.

Considerando o padrão de defeitos criados entre os grupos, nota-se que o material experimental 7% apresentou um padrão de defeitos mais regular e similar as partículas de óxido de alumínio revestidas por sílica comercialmente disponível, enquanto o material experimental 20% criou um padrão irregular de defeitos que agem como concentradores de tensão diminuindo sua resistência flexural (HALLMANN et al., 2012; CURTIS; WRIGHT; FLEMING, 2006) e apresentando um comportamento mecânico similar as partículas de óxido de alumínio não revestidas por sílica.

Na presente dissertação, a resistência de união e o comportamento mecânico da cerâmica Y-TZP foram considerados avaliando o efeito do jateamento com diferentes concentrações de sílica. Apesar de ser um estudo *in vitro*, nossos dados sugerem que o material experimental de partículas de óxido de alumínio revestidas por 7% de sílica é um potencial alternativo as partículas de óxido de alumínio revestidas por sílica comercialmente disponível. Entretanto, um cenário considerando fatores associados como jateamento e cimentação não foram considerados, assim como condições clínicas de forças laterais e deslizamentos que ocorrem durante a mastigação não foram simulados. Assim, os achados deste estudo devem ser considerados com cautela e estudos futuros avaliando o arredondamento de partículas e concentração de sílica e sua associação com a cimentação são recomendados.

5. CONCLUSÃO

Com base nos achados científicos apresentados nessa dissertação, conclui-se que:

- Jateamento de cerâmicas Y-TZP com partículas de óxido de alumínio revestidas com 7% de sílica apresentam um bom desempenho na resistência de união a cimentos resinosos e superior comportamento mecânico, podendo ser um potencial alternativo ao jateamento com partículas de óxido de alumínio revestidas por sílica comercialmente disponível.

- Uma maior concentração de sílica de 20%, apesar de apresentar uma similaridade na quantidade de transformação de fase e no padrão de alteração superficial, não aumenta a resistência de união e diminui a resistência flexural, uma vez que produz defeitos que propiciam a concentração de tensões. Alternativas de aprimoramento de síntese do material são considerados no sentido de aperfeiçoar a qualidade das partículas, assim como sua associação em um cenário de fatores associados.

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ANEXO A - AUTHORS GUIDE FOR PUBLICATION ON JOURNAL OF BIOMEDICAL MATERIALS RESEARCH PART B: APPLIED BIOMATERIALS

GUIDE FOR AUTHORS

Aims and Scope

Journal of Biomedical Materials Research Part B: Applied Biomaterials is an official journal of the Society for Biomaterials, the Japanese Society for Biomaterials, the Australasian Society for Biomaterials, and the Korean Society for Biomaterials. It is a peer-reviewed journals serving the needs of biomaterials professionals who devise, promote, apply, regulate produce, and market new biomaterials and medical devices. Papers are published on device development, implant retrieval and analysis, manufacturing, regulation of devices, liability and legal issues, standards, reviews of different device areas, and clinical applications. Published manuscript fit into one of six categories: original research reports, clinical device-related articles, short research and development reports, review, special report, or columns and editorials. Manuscripts from all countries are invited but must be in English. Authors are not required to be members of a Society for Biomaterials.

Types of Articles Considered for Publication

Original Research Reports: Full-length papers consisting of complete and detailed descriptions of a research problem, the experimental approach, the findings, and appropriate discussion. Findings should represent significant new additions to knowledge.

Clinical Device-Related Articles: Full-length papers addressing such issues as material processing, device construction, regulatory matters, clinical trials, and device retrieval.

Reviews: Scholarly and critical topic-oriented reviews that present a state-of-the-art view. While most reviews are solicited, person interested in contributing may contact the Editor.

Special Reports: Reports of special topic-oriented symposia, device retrieval protocols, or other special reports not described in the above categories, yet of interest to the applied biomaterials research and development community. Potential contributors should contact the Editor before submitting special reports.

Columns and Editorials: While columns and guest editorials are preponderantly solicited, persons interested in becoming columnists or contributing editorials are encouraged to contact the Editor.

Submission of Manuscripts

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