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Mechanical behavior of hybrid zirconia developed through Room Temperature Atomic Layer Deposition (RT-ALD)

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Comportamento mecânico de zircônia híbrida desenvolvida através da Deposição em Camada Atômica à Temperatura Ambiente (RT-ALD)

Tese constituída por artigos apresentada a Faculdade de Odontologia de Bauru - Universidade de São Paulo para obtenção do título de Doutor em Ciências no Programa de Ciências Odontológicas Aplicadas, na área de concentração Dentística.

Orientadora: Profª. Drª. Juliana Fraga Soares Bombonatti

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1Co 2:9

RESUMO

Comportamento mecânico de zircônia híbrida desenvolvida através da Deposição em Camada Atômica à Temperatura Ambiente (RT-ALD)

Objetivos: Avaliar o comportamento mecânico da interface híbrida entre a camada transformada da zirconia e o nanofilme à base de sílica, depositado por meio de Deposição em Camada Atômica à Temperatura Ambiente (RT-ALD) assim como a resistência de união entre a zircônia e o cimento resinoso após a aplicação da técnica de RT-ALD.
Materiais e Métodos: Espécimes Y-PSZ totalmente sinterizados em diferentes translucências (MO, MT, LT) foram distribuídos em 5 grupos: controle (C - sem tratamento); tratamento hidrotérmico (TH- 15h - 134 °C, 2 bar); jateamento de alumina (J - 50 µm Al_2O_3); deposição de sílica RT-ALD (S); TH seguido de deposição de sílica (THS). Os ciclos de RT-ALD consistiram na exposição sequencial das amostras a vapores de ortossilicato de tetrametoxissilano (TMOS - 60s) e hidróxido de amônio (NH_4OH - 10 min) em 40 ciclos. O desempenho mecânico foi analisado pelos testes de resistência à flexão (RF) e carga de ruptura por fadiga. A dureza superficial (D) e o módulo de Young (MY) foram analisados por nanoindentação. Para caracterização química e topográfica de superfície, foram realizadas espectroscopia de fotoelétrons de raios X (XPS) e microscopia eletrônica de varredura (MEV). Para o teste de resistência de união ao cisalhamento (RUC), cilindros de resina composta foram cimentados na superfície da zircônia com cimento resinoso (multilink Automix) e, após o teste, o modo de falha foi avaliado. Os dados de D, MY, RF, RUC e limite de fadiga (LF) foram analisados por ANOVA dois critérios.
Resultados: A topografia de superfície apresentou-se mais áspera para os grupos jateados. Na análise por XPS, um nanofilme de sílica foi observado sobre a superfície da zircônia após RT-ALD. Valores de resistência de união do grupo S e THS foram semelhantes ao grupo J ($p > 0,848$). Ambos os tratamentos S mostraram valores de FS semelhantes aos grupos B ($p > 0,410$). S não afetou LF quando comparado ao grupo C ($p > 0,277$) para todos os materiais avaliados.
Conclusões: A técnica RT-ALD apresentou-se eficaz na deposição de sílica na superfície da zircônia, apresentando resultados de resistência de união semelhantes aos espécimes jateados. Além disso, não apresentou nenhum efeito deletério nas propriedades mecânicas.
Palavras-chave: Cerâmica. Sílica. Microscopia eletrônica de varredura. Resistência ao Cisalhamento

ABSTRACT

Mechanical behavior of hybrid zirconia developed through Room Temperature Atomic Layer Deposition (RT-ALD)

Objectives: The aim Evaluate the mechanical performance of the hybrid interface between the transformed zirconia layer and the silica-based nanofilm, deposited by means of Atomic Layer Deposition at Room Temperature (RT-ALD) as well as the bond strength between the zirconia and the resin cement after RT-ALD technique.

Materials and Methods: Electron Microscopy (TEM)/Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), X-ray Diffraction (XDR) and Fourier Transform Infrared (FTIR). Fully-sintered Y-PSZ specimens in different translucencies (MO, MT, LT) were distributed in 5 groups: control (C - no treatment); hydrothermal treatment (HT- 15h - 134°C, 2 bar); alumina blasting (B - 50 μm Al_2O_3); RT-ALD silica deposition (S); HT followed by silica deposition (HTS). RT-ALD cycles consisted of the sequential exposure of specimens to tetramethoxysilane orthosilicate (TMOS - 60s) and ammonium hydroxide (NH_4OH - 10 min) vapors in 40 cycles. Mechanical performance was analyzed by flexural strength (FS) and fatigue failure load tests. Surface hardness (H) and Young's modulus (YM) were analyzed by nanoindentation. For surface chemical and topographical characterization, X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM) were performed. For the shear bond strength test (SBS), composite resin cylinders were cemented on the zirconia surface with resin cement (Multilink Automix) and, after the test, the failure mode was evaluated. Data from surface H, YM, FS, SBS and fatigue limit (FL) were analyzed by two-way analysis of variance (ANOVA).

Results: The surface topography was rougher for the blasted groups. In the XPS analysis, a silica nanofilm was observed on the zirconia surface after RT-ALD. Bond strength values of group S and HTS were similar to group J ($p > 0.848$). Both S treatments showed values of FS similar to groups B ($p > 0.410$). S did not affect FL when compared to group C ($p > 0.277$) for all evaluated materials.

Conclusions: RT-ALD technique was effective in depositing silica on the zirconia surface, presenting results of bond strength similar to the blasted specimens. In addition, RT-ALD did not have any deleterious effect on the mechanical properties.

Keywords: Ceramics. Microscopy, Electron, Scanning. Silica. Shear Strength.

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1 INTRODUCTION

1 INTRODUCTION

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) has been used in dentistry more than 20 years and considered a versatile material for metal-free prosthodontic treatments [1] due to its optical, physical and mechanical proprieties [2–4]. Their mechanical performance is related with high flexural strength (around 1,000 MPa) [5,6], hardness (19 GPa) [7], and fracture toughness above 7.36 MPa $m^{1/2}$ [8]. Also, Y-PSZ has a wide variety of clinical applications, such as fixed dental prostheses (FDPs), monolithic crowns, implant abutments or screw-retained-protheses [9]. The main failure problem of zirconia rehabilitation systems is the chipping of the veneering porcelain [10,11]. However, the monolithic restorations have become an alternative treatment, since is not necessary the veneering ceramic application and the high translucency [12]. The translucency of zirconia restorations is related to the chemical nature, crystal structure [13], particle and grain size, and the density of the material [14]. These factors determine the amount of light that is reflected, absorbed, and transmitted [15] and, the material indication.

The crystallographic structure of Y-TZP is represented by three crystallographic forms: monoclinic (m), tetragonal (t), and cubic (c), according to the temperature change [7,16]. The t-phase stabilization at room temperature is achieved by the addition of yttria (3 mol%) due to the poor meta-stability of zirconia crystals [17,18]. The best mechanical performance of zirconia-based materials is associated with a mechanism called "transformation toughening" [19], due to the metastability of the tetragonal (t) phase at room temperature. This process occurs when the zirconia is exposed to localized stress fields causing a local transformation of the tetragonal crystals to the monoclinic state (t → m), generating a volume expansion (3-5%) of the 3Y-TZP grains in the region of stress. Also, the process leads to the formation of local compressive stresses which arrest the crack from propagating [20], therefore increasing the material's fracture toughness. However, phase transformation alters the phase integrity and increases the zirconia's susceptibility to aging [21].

The instability of 3Y-TZP is generated by the process called "low-temperature degradation" (LTD) [22]. Zirconia's LTD consists an uncontrolled t→m transformation in the presence of water [23], which affects the 3Y-TZP mechanical properties, leading to fracture [16,24]. Also, studies have been reported that the displacement of the martensitic plates on the surface of the 3Y-TZP transformed layer (t → m) leads to the development of a transitional layer of tetragonal and monoclinic grains surrounded by microcracks [25,26] in addition to a highly uneven monoclinic-saturated transitional layer with maximized grain removal [25]. Therefore, the transformed microcracked layer could become a permeable membrane for the infiltration/deposition of materials.

In addition to the problem of LTD, the literature has been reported several failures related to fracture/chipping of veneering porcelain, marginal discoloration and debonding of the FDPs [27,28]. However, bonding failure is still a challenge between 3Y-TZP and resin-based materials, since the absence of a glass matrix in composition [29]. Therefore, several surface treatments have been developed to increase the bond strength between these two materials [30]. Some chemical and mechanical methods have been used including airborne abrasion with alumina particle (Al_2O_3), silicatization, laser (Nd:YAG, Er:YAG), selective infiltration-etching (SIE), application of low-fused porcelain, primers, and cements with 10-Methacryloxydecyl Dihydrogen Phosphate monomer (MDP) [31–34].

The wide variation of zirconia adhesion protocols reported in in vitro studies interferes in the establishment of the best protocol. However, physicochemical surface methods, increased the bond strength between zirconia and resin cements [30]. Among the chemical conditioning methods, silica (SiO_2) deposition by the atomic layer deposition (ALD) technique has been used in engineering for deposition of monolayers of materials with reactive vapor precursors [35]. Silica deposition at room temperature (RT) was previously reported by the contact and chemical reaction of tetramethoxysilane (TMOS) and ammonia (NH_3) vapors on the polystyrene spheres surface [36].

Considering that the RT-ALD technique can be used in other organic materials such as Al_2O_3, TiO_2, ZnO, and ZnS [36], and the micro-cracks network of the transformed layer can become a permeable membrane for the infiltration/deposition of materials, the deposition of silica via RT-ALD on the zirconia surface is promising. Therefore, the study has two objectives: (1) to evaluate the bond strength at the interface between Y-TZP and resin cement after silica deposition via RT-ALD, and (2) to evaluate the mechanical performance of the hybrid interface between Y-TZP transformed layer and silica-based nanofilm via RT-ALD.

2 ARTICLES

2 ARTICLES

2.1 ARTICLE 1

Silica deposition on zirconia via Room-Temperature Atomic Layer Deposition and bond strength to resin cement

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ABSTRACT

Objectives: This study aimed to evaluate the effect of the controlled growth of yttria-stabilized zirconia (Y-PSZ) transformed layer and silica deposition via Room-Temperature Atomic Layer Deposition (RT-ALD) on bond strength between Y-PSZ and resin cement.

Methods: Y-PSZ specimens (IPS e.max ZirCAD, Ivoclar Vivadent) with different translucencies (MO, MT, LT) were exposed to surface treatments (n=10): control (C - no treatment), aged (A - 15 h, 134°C, 2 bar in autoclave), blasted (B - 50 µm Al2O3), silica deposition via Rt-ALD (R - 40 cycles of sequential exposure to tetramethoxysilane orthosilicate and ammonium hydroxide vapors) and aged followed by silica deposition (AR). Specimens were bonded to resin composite cylinders with a resin cement (Multilink Automix) and shear bond strength (SBS) test was performed. Failure mode and surface chemical and topographical characterization (energy-dispersive spectroscopy, X-ray photoelectron spectroscopy and scanning electron microscopy) were performed. SBS data was analyzed by two-way ANOVA and Tukey test (α=.05).

Results: The results of the two-way ANOVA indicated that surface treatment ($p<.001$) and the zirconia translucency/surface treatment interaction ($p=.001$) had a significant effect on SBS. Silica deposition resulted in bond strength values similar to alumina blasting ($p>.848$), with no significant effect of aging ($p>.664$). Higher incidence of composite resin cohesive failures was observed in the R-treated specimens (MT, LT), with and without aging. SEM images showed a rougher surface for B-treated specimens, while no changes were observed for the other treatments. XPS analysis showed a nanofilm of silica deposited over the zirconia surface after silica deposition.

Significance: RT-ALD was an effective technique to deposit silica on the surface of zirconia, generating bond strength results similar to alumina-blasted specimens.

Keywords: Y-PSZ, Shear Bond Strength, Air Abrasion, Dental materials.

1. INTRODUCTION

The clinical use of yttria-partially stabilized zirconia (Y-PSZ) has increased over the years due to the several indications of zirconia, such as fixed dental prosthesis (FDP)[1], customized abutments[2], single/multiple crowns' frameworks, and monolithic restorations[3]. The clinical longevity of zirconia as a framework has shown high survival rates for single crowns (88.8% and 98.3% at 3 and 5 years, respectively)[4] and FDPs (96.3% up to 3 years)[5], due to its excellent mechanical properties such as high fracture toughness (above 7.36 MPa m1/2)[6], flexural strength (~ 1000 MPa)[7,8], and hardness (19 GPa)[9]. However, different problems have been reported, such as chipping of the veneering ceramic, framework fracture[10,11] and FDP debonding. The debonding of the FDP framework from the abutment teeth is indeed the most frequent problem reported[12] due to the low retention between resin cements and the crystalline zirconia structure[13,14].

The bond between resin-based luting agents and zirconia remains a challenge due to Y-PSZ's lack of glass phase[15]. High bond strength between silica-based ceramics is reported because hydrofluoric acid etching (5% for 30–60s) and silane are combined to maximize chemical and micromechanical bonding[16–18]. To improve zirconia bond strength to resin cements, different surface conditioning methods have been proposed based on mechanical or chemical conditioning properties[14,19]. The surface treatments include airborne-particle abrasion[20–23], which increases surface roughness for micromechanical interlocking, the physicochemical surface modification of zirconia with silica-coated alumina particles followed by silanization[24–28], and the use of adhesives with functional monomers to enhance chemical interactions[29,30].

In addition to the limited bonding capacity, critical structural failures can occur due to low temperature degradation (LTD) of zirconia[31]. LTD is caused by the development of local compressive stresses in the presence of water[32,33] promoting the transformation of the tetragonal grains back to their natural state - monoclinic, a process known as tetragonal-to-monoclinic (t-m) phase transformation. T-m phase transformation is known to increase surface roughness and decrease some of the mechanical properties of zirconia[34]. However, the LTD process starts by a displacement of the martensitic plates on the Y-PSZ surface that results in a transitional layer of tetragonal and monoclinic grains surrounded by microcracks, with some capacity for elastic deformation as indicated by nanoindentation[35,36]. It is possible that this t-m transitional microcracked layer, when developed under controlled conditions, increases the porosity of the Y-PSZ surface, enabling its interaction with chemical agents by effective infiltration of vapors in the nanoscale.

The absence of silica on Y-TZP structure[15] may be overcome by surface silica deposition, enhancing bond strength between zirconia and resin cement. Atomic layer deposition (ALD) is a technique that has been used in engineering for nanoscale layer deposition of inorganic oxides, such as Al2O3, TiO2, ZnO, and ZnS[26,37]. This technique may also be used for the deposition of a nanofilm of silica (SiO2) on different materials' surface through reactive vapor precursors[38,39]. Successful ALD deposition of silica layers was demonstrated at room-temperature conditions (RT-ALD) by using vapors of tetramethoxysilane (TMOS) and ammonia (NH3)[37], a technique that is cheaper than conventional ALD, since it does not demand specialized equipment, while still effective.

Silica deposition on Y-PSZ surface via RT-ALD has the potential to improve its bond strength to resin-based materials. It is also a promising approach to develop zirconia-based hybrid materials in different areas of healthcare and industry. In addition, the development of a microcracked transitional t-m layer under controlled conditions[35,40] may enhance the vapor infiltration capacity of Y-PSZ by acting as a permeable membrane. Therefore, the purpose of this study was to investigate the potential of this t-m microcracked layer to be infiltrated by silica using RT-ALD, through analysis of the bond strength between Y-PSZ and resin cement, and surface characterization. The hypotheses to be tested were: (1) the development of a microcracked layer prior to RT-ALD has an effect on bond strength between Y-PSZ and resin cement; (2) silica infiltration via RT-ALD affects the bond strength between Y-PSZ and resin cement

2. MATERIAL AND METHODS

2.1 Preparation of specimens

Partially-sintered Y-PSZ blocks with three different translucencies (MO, MT, LT - IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein) were cut (Isomet® 1000 Buehler, Lake Bluff, IL, USA) with a diamond blade (15LC, Buehler, Lake Bluff, IL, USA) at low speed under water cooling. Specimens were sintered (Programat® S1, Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions and, after cleaning in ultrasonic bath (QuickClean; Midmark) in acetone for 5 minutes, fifty sintered specimens (5 x 5 x 2 mm) were randomly assigned to five experimental groups for each material (Figure 1): control (C - no treatment), aged (A – controlled artificial aging in autoclave), blasted (B - alumina blasting), silica deposition via RT-ALD (R) and aged followed by silica deposition via RT-ALD (AR). The experimental materials used in this study are described in Table 1.

2.2 Surface treatments

2.2.1 Control – After ultrasonic cleaning, specimens were stored in distilled water at room temperature until the bonding procedures started.

2.2.2 Aged - Twenty specimens from each material were aged in autoclave (Ritter M9 Midmark Sterilizer, Versailles, Ohio, USA) under 2 bars at 134 oC for 15 hours[41,42]. The aging conditions were determined based on findings of a preliminary study (currently under review);

2.2.3 Blasted - Airborne abrasion with 50 µm Al2O3 particle under 2 bar pressure, from a perpendicular distance of 10 mm, for 15s.

2.2.4 Room Temperature Atomic Layer Deposition (RT-ALD) - Silica deposition via RT-ALD - specimens were sequentially exposed to tetramethoxysilane orthosilicate (TMOS – 60 seconds) vapor and ammonium hydroxide (NH4OH - 10 minutes), which represented one cycle[37]. Forty cycles were applied. Vapor exposure was achieved by placing the specimens in a basket suspended 2 cm above the solution. Oxygen plasma cleaning (PDC-001 plasma cleaner, Harrick Plasma, Ithaca, NY, USA) was applied for 10 min every 5 cycles to enhance zirconia surface hydrophilicity. The entire silica deposition process was carried out in a fume hood under ambient pressure conditions. The number of RT-ALD cycles was previously determined in a pilot study after surface characterization by X-ray photoelectron spectroscopy (XPS) (further details provided below).

2.2.5 Hydrothermal aging + RT-ALD - Hydrothermal aging followed by 40 cycles of RT-ALD as previously described.

2.3 Shear bond strength

2.3.1 Bonding

For the preparation of the shear bond strength test specimens, zirconia slabs were embedded in epoxy resin (EpoxyCure 2, Buehler, Lake Bluff, IL, USA). Composite resin cylinders (3.25Ø mm x 3 mm) were prepared by placing 2 mm increments (IPS Empress Direct, Ivoclar Vivadent, Schaan, Liechtenstein) in an acrylic resin mold. Each increment was light-activated for 20 seconds (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein).

For the bonding, Monobond Plus was applied to the Y-PSZ surface and was left to dry for 60 seconds; the composite resin cylinders were treated with the two primer liquids (Multilink Primer A and B) mixed (1:1), applied with a brush (30 seconds) and dried with a gentle air blast. Composite resin cylinders were cemented to zirconia with resin cement (Multilink Automix, Ivoclar Vivadent, Schaan, Liechtenstein) according to manufacturer's instructions, under an axial load of 5 N. Excess was removed and the cement was light-activated for 20 seconds in 4 perpendicular directions. Specimens were stored in distilled water (37°C) for 24 h before the bond strength test.

Shear bond strength (SBS) test of the interface between zirconia and resin cement was performed in a universal testing machine (Instron 8501; Instron, Canton, Mass) with a semicircular knife edge. The load was applied at a crosshead speed of 0.5 mm/min until fracture. The load at failure was recorded and the SBS values (in N) were converted to MPa using the formula P/A, where P is the fracture load (N) and A is the bonded area (mm2)[43].

2.3.2 Failure analysis

All specimens were analyzed for mode of failure in a stereomicroscope (60X magnification, Olympus SZ61, Olympus America Inc., Center Valley, PA, EUA). The failures were classified in adhesive (failure between zirconia and resin cement), cohesive (failure between composite resin and resin cement), or mixed (at least 30% of one of both types of failure).

2.4 SEM/EDS analysis

The morphological characterization of the zirconia surface was performed in 2 specimens per experimental condition, under scanning electron microscope (SEM - JEOL 6610 LV, Jeol USA Inc., Peabody, MA, USA), in high vacuum mode (15Kv), at a working distance of 8 mm and 5,000X magnification. Chemical characterization (Zr, O, Al and Si) of the specimens' surface was performed by Energy-dispersive X-ray spectroscopy analysis (EDS, X-Max, Oxford Instruments, United Kingdom) after sputter coating with gold-palladium.

2.5 X-ray photoelectron spectroscopy analysis

The number of RT-ALD cycles to be performed was defined in a pilot study, based on the generation of a homogeneous layer of silica over Y-PSZ. Medium translucency (MT) zirconia specimens were exposed to different number of RT-ALD cycles, as previously described: 0 (control), 1, 2, 5, 20 and 40; treated specimens were analyzed by XPS using a Thermofisher Scientific K-Alpha system (Thermofisher Scientific—E, Grinstead, UK). The measurements were performed with high resolution spectra (nominal 400 µm spot, 25 eV pass energy) for the observed elemental regions obtained. The scan was performed in wide- and narrow-scan spectra (Y3d5, Si2p, Zr3d, C1s, O1s), using a source gun of Al K Alpha, with standard lens mode, and energy step size of 0.100 eV.

2.6 Statistical analysis

SBS values were analyzed using two-way ANOVA (independent variables: surface treatment and zirconia translucency) followed by one-way ANOVA. The interaction between materials and groups was analyzed using post hoc Tukey test (SPSS 20, SPSS Inc., Chicago, IL, USA). The overall statistical significance was pre-set at 5%.

3. RESULTS

3.1 XPS examination

XPS analysis indicated effective deposition of a nanofilm of silica after 20 and 40 cycles of RT-ALD (Table 2). Narrow-scan XPS spectra of the Si2p region of Y-PSZ are presented in Figure 2. A Si2p peak was observed at an expected binding energy of approximately 154 eV. The atomic ratios of Si were clearly higher after 40 cycles (27.4) than they were at 20 cycles (18.8), and the peaks of zirconia (8.4 and 0) were also the lowest after 40 cycles of silica deposition (Table 2).

3.2 Shear bond strength and failure mode

SBS results are presented in Figure 3. The results of the two-way ANOVA indicated that surface treatment ($p<.001$) and the interaction zirconia translucency and surface treatment ($p=.001$) had a significant effect on SBS. Zirconia translucency was not significant ($p=.512$). The analysis of the interaction indicated significant difference between groups ($p<.000$): bond strength values ranged from 9.83(±2.14) to 26.75(±5.23) MPa. MT/B (26.75±5.23 MPa) and MT/R (26.23±8.27 MPa) presented the highest SBS values, while LT/C (10.09±6.59 MPa) and MT/A (9.83±2.14 MPa) presented the lowest SBS values (Figure 3). There was no effect of hydrothermal aging on bond strength when R and AR groups were compared ($p>.664$), for any of the materials evaluated. Similar SBS values ($p>.848$) were observed between B and R treated specimens, for all the materials analyzed. RT-ALD resulted in significantly higher SBS for MO ($p<.001$) and MT ($p=.025$) in comparison to the control (untreated) specimens.

Failure mode analysis results are presented in Figure 4. Adhesive and mixed failures at the bonding interface between zirconia and resin cement were the most frequent ones observed for all materials. When RT-ALD treatment was applied, a prevalence of cohesive failures was observed for MT (□50%) and LT (60%).

3.3 SEM/EDS analysis

SEM images showed different surface morphologies after surface treatments were applied (Figure 5). When control groups (Figure 5A, F, K) and aged specimens are compared (Figure 5B, G, L), MO shows granular increase as a consequence of aging, while LT shows some slight changes in surface topography. MT specimens did not show surface changes between control and aged conditions. Alumina blasting (Figure 5C, H, M) resulted in superficial and deep notches created by the high impact of alumina particles on zirconia surface. Silica deposition via RT-ALD (Figure 5D, E, I, J, N, O) did not affect surface topography to a level that could be observed under SEM.

The chemical distribution (wt%) of the elements (Zr, O, Al and Si) observed on zirconia surface is presented in Figure 6. Zr and O were identified in all materials and in the different treatments with similar composition. As expected, Al was detected in all alumina-blasted specimens. Si (0.49%) was detected in one LT specimen after RT-ALD treatment for silica deposition.

4. DISCUSSION

The aims of this study were to investigate the use of controlled artificial aging to increase zirconia's wettability by oxide vapors, and to evaluate RT-ALD as a technique for silica deposition on zirconia surface. The effectiveness of the silica nanofilm deposited was characterized by the analysis of bond strength between zirconia and resin-based cement. Under the conditions of the present study, hydrothermal aging combined with RT-ALD did not affect bond strength between zirconia and resin cement in comparison to RT-ALD alone ($p>.664$). RT-ALD resulted in bond strength values that were similar to the ones obtained for alumina-blasted specimens, regardless of zirconia translucency. Therefore, the first hypothesis was rejected and the second hypothesis was accepted.

Long-term clinical success of indirect restorations is achieved, amongst other variables, with predictability of the adhesive cementation[44]. In addition, adhesive cementation improves stress distribution throughout the restorative complex, increasing the restoration's mechanical properties[45]. A dual-cure, self-etching adhesive cement was chosen for the cementation of specimens due to the well-documented bond strength values[46]. The present study used shear bond strength test because it is the technique most commonly used to evaluated bond strength to zirconia[14]. Within the different tests designs available (shear, micro-shear, tensile, and micro-tensile)[14], the choice of SBS test is due to the minimum equipment required and ease of specimen preparation, which makes it a simple and low-cost protocol[47–49]. The degrees of translucency of the zirconia-based materials evaluated (MO, MT and LT) were selected to represent different clinicals scenarios.

In this study, hydrothermal aging was used to induce crystalline changes on zirconia surface, creating a microcracked layer that would be more receptive to the infiltration of a silica nanofilm as per the characteristics of the transitional/transformed layer previously reported in the literature[35,36]. The analysis of the results showed that hydrothermal aging (A) did not positively affect SBS to zirconia when compared to RT-ALD (R) or control (C) specimens, regardless of zirconia translucency. Aging before RT-ALD (AR) resulted in SBS results similar to when RT-ALD was used alone (R). It was expected that the permeability of the Y-PSZ microcracked t-m layer would have an effect on the strength of the silica infiltrated/deposited layer, which was not observed via SBS test. Further investigations into the interface developed are encouraged.

RT-ALD has already shown promising results in terms of controlled growth of a silica nanofilm[26,37]. In the present study, RT-ALD was used to enable the infiltration and deposit of a nanofilm of silica on the surface of zirconia. A clinically acceptable bond strength to zirconia has been reported as approximately 10-13 MPa[50]. From the groups analyzed in this study, only MT/A (9.83±2.14 MPa) and LT/C (10.09±6.59 MPa) were below that range. RT-ALD resulted in bond strength values that were similar to the values presented by alumina blasting, which is considered the standard treatment for proper micromechanical interlocking between zirconia and resin cement (Figure 3). Alternative surface treatments are constantly under scrutiny, since alumina blasting improves bond strength to zirconia[46,51] at the expenses of the restoration's fatigue resistance[52], as demonstrated in a subcritical crack growth study[53]. The roughness reported in alumina particle-abraded specimens can be observed in the SEM images (Figure 5C, H, M), where surface irregularities and sharp indentations are distributed throughout the surface. This finding corroborates other studies[54–56] which reported surface defects that were deeper than the transformation toughening "healing" capacity of the material[52].

Tribochemical silica coating followed by a silane agent application was initially introduced as an alternative to alumina-particle abrasion, but the stability of that bonding[46,57] and the effectiveness of the interaction between silica and zirconia[58] were later questioned. In the present study, surface characterization by SEM showed that RT-ALD resulted in a regular surface (Figure 5D, I, N) that is apparently similar to the control group (Figure 5A, F, K)[56]. No surface damage was expected as a result of the RT-ALD treatment. The analysis of failure suggested that SBS between resin cement and zirconia after RT-ALD was higher than the cohesive strength of the resin cement, as per the high prevalence of cohesive failure in the RT-ALD groups >50% for MT and LT materials (Figure 4). Thus, RT-ALD should be further investigated as an alternative to alumina blasting and silica-deposition methods, as a reliable, simple, damage-free, surface treatment for the deposition of silica on zirconia surface.

The XPS analysis, a highly sensitive technique for surface chemical characterization[59], indicated a nanofilm of silica deposited on Y-PSZ surface during the pilot study, when silica deposition was analyzed after different numbers of cycles (Table 2). However, the EDS analysis of the specimens prepared for bonding only indicated presence of silica in one of the specimens (LT/RT-ALD). These results may be explained by the difference of characterization depth between EDS (□m) and XPS (nm). XPS analyses the specimen in a nanometric scale, being possible to detect chemical elements within that range[59]. EDS, on the other hand, is suitable for the analysis of thicker films. A previous study reported a 20 nm-thick silica growth after 5 cycles[37]. Based on this, a silica film thickness of approximately 160 nm was expected after 40 cycles. In spite of the lack of sensitivity of EDS to detect silica in the R-treated specimens, an effective silica nanofilm is the only possible reason behind the significant difference in bond strength results between RT-ALD-treated and control specimens in all groups. XPS or other sensitive techniques for surface chemical characterization are recommended in future studies to be able to identify the changes in a nanometric scale[59,60].

This study aimed to evaluate the effect of controlled aging and silica deposition on the interfacial bond strength between Y-PSZ and resin cement via RT-ALD. Among the limitations of the present study, the stability of the bonding was not analyzed and the specimens' design and dimensions were not representative of dental restorations. However, the positive outcomes observed in this study indicate that further investigations should be pursued and include more

RT-ALD cycles. The analysis of other surface cleaning methods, more accessible and less expensive than oxygen plasma treatment, is also encouraged.

5. CONCLUSION

Under the limitations of the present study, the following conclusions can be addressed:

1. Controlled aging of zirconia did not positively affect bond strength to resin cement.
2. Bond strength between Y-PSZ and resin cement was positively affected by silica deposition via RT-ALD, with similar results to alumina blasting.
3. Alumina-blasting affected specimens' surface topography in a pattern compatible with surface damage.
4. EDS analysis was not an effective technique to characterize the presence of silica on zirconia surface after RT-ALD.

6. REFERENCES

[1] Tinschert J, Natt G, Mautsch W, Augthun M, Spiekermann H. Fracture resistance of lithium disilicate-, alumina-, and zirconia-based three-unit fixed partial dentures: a laboratory study. Int J Prosthodont 2001;14:231–8.

[2] Bachhav VC, Aras MA. Zirconia-based fixed partial dentures: a clinical review. Quintessence Int 2011;42:173–82.

[3] Stober T, Bermejo JL, Rammelsberg P, Schmitter M. Enamel wear caused by monolithic zirconia crowns after 6 months of clinical use. J Oral Rehabil 2014;41:314–22. https://doi.org/10.1111/joor.12139.

[4] Ortorp A, Kihl ML, Carlsson GE. A 5-year retrospective study of survival of zirconia single crowns fitted in a private clinical setting. J Dent 2012;40:527—530. https://doi.org/10.1016/j.jdent.2012.02.011.

[5] Schmitt J, Holst S, Wichmann M, Reich S, Gollner M, Hamel J. Zirconia posterior fixed partial dentures: a prospective clinical 3-year follow-up. Int J Prosthodont 2009;22:597–603.

[6] Harada K, Shinya A, Gomi H, Hatano Y, Shinya A, Raigrodski AJ. Effect of accelerated aging on the fracture toughness of zirconias. J Prosthet Dent 2016;115:215–23. https://doi.org/10.1016/j.prosdent.2015.08.020.

[7] Kelly JR, Denry I. Stabilized zirconia as a structural ceramic: an overview. Dent Mater 2008;24:289–98. https://doi.org/10.1016/j.dental.2007.05.005.

[8] Borchers L, Stiesch M, Bach F-W, Buhl J-C, Hübsch C, Kellner T, et al. Influence of hydrothermal and mechanical conditions on the strength of zirconia. Acta Biomater 2010;6:4547–52. https://doi.org/https://doi.org/10.1016/j.actbio.2010.07.025.

[9] Catledge SA, Cook M, Vohra YK, Santos EM, McClenny MD, David Moore K. Surface crystalline phases and nanoindentation hardness of explanted zirconia femoral heads. J Mater Sci Mater Med 2003;14:863–7. https://doi.org/10.1023/a:1025678525474.

[10] Schley J-S, Heussen N, Reich S, Fischer J, Haselhuhn K, Wolfart S. Survival probability of zirconia-based fixed dental prostheses up to 5 yr: a systematic review of the literature. Eur J Oral Sci 2010;118:443–50. https://doi.org/10.1111/j.1600-0722.2010.00767.x.

[11] Sola-Ruiz MF, Agustin-Panadero R, Fons-Font A, Labaig-Rueda C. A prospective evaluation of zirconia anterior partial fixed dental prostheses: Clinical results after seven years. J Prosthet Dent 2015;113:578–84. https://doi.org/10.1016/j.prosdent.2014.12.015.

[12] Ohlmann B, Rammelsberg P, Schmitter M, Schwarz S, Gabbert O. All-ceramic inlay-retained fixed partial dentures: preliminary results from a clinical study. J Dent 2008;36:692–6. https://doi.org/10.1016/j.jdent.2008.04.017.

[13] Cavalcanti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, Marchi GM. Bond strength of resin cements to a zirconia ceramic with different surface treatments. Oper Dent 2009;34:280–7. https://doi.org/10.2341/08-80.

[14] Ozcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. J Adhes Dent 2015;17:7–26. https://doi.org/10.3290/j.jad.a33525.

[15] Kim MJ, Kim YK, Kim KH, Kwon TY. Shear bond strengths of various luting cements to zirconia ceramic: Surface chemical aspects. J Dent 2011;39:795–803. https://doi.org/10.1016/j.jdent.2011.08.012.

[16] Chen JH, Matsumura H, Atsuta M. Effect of etchant, etching period, and silane priming on bond strength to porcelain of composite resin. Oper Dent 1998;23:250–7.

[17] Chen JH, Matsumura H, Atsuta M. Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain. J Dent 1998;26:53–8. https://doi.org/10.1016/s0300-5712(96)00078-4.

[18] Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent 2003;89:268–74. https://doi.org/10.1067/mpr.2003.50.

[19] Chai J, Chu FCS, Chow TW. Effect of surface treatment on shear bond strength of zirconia to human dentin. J Prosthodont 2011;20:173–9. https://doi.org/10.1111/j.1532-849X.2011.00695.x.

[20] Atsu SS, Kilicarslan MA, Kucukesmen HC, Aka PS. Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. J Prosthet Dent 2006;95:430–6. https://doi.org/10.1016/j.prosdent.2006.03.016.

[21] Borges GA, Sophr AM, de Goes MF, Sobrinho LC, Chan DCN. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. J Prosthet Dent 2003;89:479–88. https://doi.org/10.1016/s0022-3913(02)52704-9.

[22] Foxton RM, Cavalcanti AN, Nakajima M, Pilecki P, Sherriff M, Melo L, et al. Durability of resin cement bond to aluminium oxide and zirconia ceramics after air abrasion and laser treatment. J Prosthodont 2011;20:84–92. https://doi.org/10.1111/j.1532-849X.2010.00678.x.

[23] Derand T, Molin M, Kvam K. Bond strength of composite luting cement to zirconia ceramic surfaces. Dent Mater 2005;21:1158–62. https://doi.org/10.1016/j.dental.2005.02.005.

[24] Ozcan M, Vallittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. Dent Mater 2003;19:725–31. https://doi.org/10.1016/s0109-5641(03)00019-8.

[25] Ozcan M, Nijhuis H, Valandro LF. Effect of various surface conditioning methods on the adhesion of dual-cure resin cement with MDP functional monomer to zirconia after thermal aging. Dent Mater J 2008;27:99–104.

[26] Yu X, Zhang H, Oliverio JK, Braun P V. Template-assisted three-dimensional nanolithography via geometrically irreversible processing. Nano Lett 2009;9:4424–7. https://doi.org/10.1021/nl9027236.

[27] Tsuo Y, Yoshida K, Atsuta M. Effects of alumina-blasting and adhesive primers on bonding between resin luting agent and zirconia ceramics. Dent Mater J 2006;25:669–74. https://doi.org/10.4012/dmj.25.669.

[28] Valandro LF, Ozcan M, Bottino MC, Bottino MA, Scotti R, Bona A Della. Bond strength of a resin cement to high-alumina and zirconia-reinforced ceramics: the effect of surface conditioning. J Adhes Dent 2006;8:175–81.

[29] White SN, Miklus VG, McLaren EA, Lang LA, Caputo AA. Flexural strength of a layered zirconia and porcelain dental all-ceramic system. J Prosthet Dent 2005;94:125–31. https://doi.org/10.1016/j.prosdent.2005.05.007.

[30] Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. Dent Mater 1998;14:64–71. https://doi.org/10.1016/s0109-5641(98)00011-6.

[31] Kobayashi K, Kuwajima H, Masaki T. Phase change and mechanical properties of ZrO2-Y2O3 solid electrolyte after ageing. Solid State Ionics 1981;3–4:489–93. https://doi.org/https://doi.org/10.1016/0167-2738(81)90138-7.

[32] Amaral M, Valandro LF, Bottino MA, Souza ROA. Low-temperature degradation of a Y-TZP ceramic after surface treatments. J Biomed Mater Res B Appl Biomater 2013;101:1387–92. https://doi.org/10.1002/jbm.b.32957.

[33] Pereira G, Amaral M, Cesar PF, Bottino MC, Kleverlaan CJ, Valandro LF. Effect of low-temperature aging on the mechanical behavior of ground Y-TZP. J Mech Behav Biomed Mater 2015;45:183–92. https://doi.org/10.1016/j.jmbbm.2014.12.009.

[34] Chevalier J, Gremillard L, Deville S. Low-Temperature Degradation of Zirconia and Implications for Biomedical Implants. Annu Rev Mater Res 2007;37:1–32. https://doi.org/10.1146/annurev.matsci.37.052506.084250.

[35] De Souza GM, Zykus A, Ghahnavyeh RR, Lawrence SK, Bahr DF. Effect of accelerated aging on dental zirconia-based materials. J Mech Behav Biomed Mater 2017;65:256–63. https://doi.org/10.1016/j.jmbbm.2016.08.023.

[36] Pinto PA, Colas G, Filleter T, De Souza GM. Surface and Mechanical Characterization of Dental Yttria-Stabilized Tetragonal Zirconia Polycrystals (3Y-TZP) After Different Aging

Processes. Microsc Microanal 2016;22:1179–88. https://doi.org/10.1017/S1431927616011843.

[37] Hatton B, Kitaev V, Perovic D, Ozin G, Aizenberg J. Low-temperature synthesis of nanoscale silica multilayers - Atomic layer deposition in a test tube. J Mater Chem 2010;20:6009–13. https://doi.org/10.1039/c0jm00696c.

[38] Rauwel E, Clavel G, Willinger M-G, Rauwel P, Pinna N. Non-Aqueous Routes to Metal Oxide Thin Films by Atomic Layer Deposition. Angew Chemie Int Ed 2008;47:3592–5. https://doi.org/10.1002/anie.200705550.

[39] Leskelä M, Ritala M. Atomic layer deposition (ALD): From precursors to thin film structures. Thin Solid Films 2002;409:138–46. https://doi.org/10.1016/S0040-6090(02)00117-7.

[40] Pinto PA, Colas G, Filleter T, De Souza GM. Surface and mechanical characterization of dental Yttria-Stabilized Tetragonal Zirconia Polycrystals (3Y-TZP) after different aging processes. Microsc Microanal 2016;22:1179–88. https://doi.org/10.1017/S1431927616011843.

[41] Xiao R, Chu B-F, Zhang L, Cao J-K. Aging performances for resisting low-temperature of three dental Yttria-stabilized zirconia ceramic core materials. Chin Med J (Engl) 2012;125:1999–2003.

[42] Pereira GKR, Venturini AB, Silvestri T, Dapieve KS, Montagner AF, Soares FZM, et al. Low-temperature degradation of Y-TZP ceramics : A systematic review and meta-analysis 2016;55:151–63.

[43] Altintas S, Eldeniz AU, Usumez A. Shear bond strength of four resin cements used to lute ceramic core material to human dentin. J Prosthodont 2008;17:634–40. https://doi.org/10.1111/j.1532-849X.2008.00348.x.

[44] Magne P, Paranhos MPG, Burnett LH. New zirconia primer improves bond strength of resin-based cements. Dent Mater 2010;26:345–52. https://doi.org/https://doi.org/10.1016/j.dental.2009.12.005.

[45] Addison O, Marquis PM, Fleming GJP. Resin Elasticity and the Strengthening of All-ceramic Restorations. J Dent Res 2007;86:519–23. https://doi.org/10.1177/154405910708600606.

[46] Erdem A, Akar GC, Erdem A, Kose T. Effects of Different Surface Treatments on Bond Strength Between Resin Cements and Zirconia Ceramics. Oper Dent 2014;39:E118–27. https://doi.org/10.2341/12-420-L.

[47] Braga RR, Meira JBC, Boaro LCC, Xavier TA. Adhesion to tooth structure: A critical review of “macro” test methods. Dent Mater 2010;26:38–49. https://doi.org/10.1016/j.dental.2009.11.150.

[48] Alves M, Campos F, Bergoli CD, Bottino MA, Özcan M, Souza R. Effect of Adhesive Cementation Strategies on the Bonding of Y-TZP to Human Dentin. Oper Dent 2016;41:276–83. https://doi.org/10.2341/15-052-L.

[49] Tanis MÇ, Akay C, Akçaboy TC, Sen M, Kavakli PA, Sapmaz K. In vitro shear bond strength between fluorinated zirconia ceramic and resin cements. J Adv Prosthodont 2018;10:205–10. https://doi.org/10.4047/jap.2018.10.3.205.

[50] Lüthy H, Loeffel O, Hammerle CHF. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. Dent Mater 2006;22:195–200. https://doi.org/10.1016/j.dental.2005.04.016.

[51] Flores-Ferreyra BI, Cougall-Vilchis RJS, Velazquez-Enriquez U, Garcia-Contreras R, Aguillon-Sol L, Olea-Mejia OF. Effect of airborne-particle abrasion and, acid and alkaline treatments on shear bond strength of dental zirconia. Dent Mater J 2019;38:182–8. https://doi.org/10.4012/dmj.2018-078.

[52] Zhang Y, Lawn BR, Rekow ED, Thompson VP. Effect of sandblasting on the long-term performance of dental ceramics. J Biomed Mater Res B Appl Biomater 2004;71:381–6. https://doi.org/10.1002/jbm.b.30097.

[53] Zhang Y, Pajares A, Lawn BR. Fatigue and damage tolerance of Y-TZP ceramics in layered biomechanical systems. J Biomed Mater Res B Appl Biomater 2004;71:166–71. https://doi.org/10.1002/jbm.b.30083.

[54] Bastos NA, Lisboa-Filho PN, Tabata A, Francisconi PAS, Furuse AY, Honório HM, et al. Effect of alternative sonochemical treatment on zirconia surface and bond strength with veneering ceramic. Mater Sci Eng B Solid-State Mater Adv Technol 2020;252:114473. https://doi.org/10.1016/j.mseb.2019.114473.

[55] Bitencourt SB, Dos Santos DM, da Silva EVF, Barão VAR, Rangel EC, da Cruz NC, et al. Characterisation of a new plasma-enhanced film to improve shear bond strength between zirconia and veneering ceramic. Mater Sci Eng C Mater Biol Appl 2018;92:196–205. https://doi.org/10.1016/j.msec.2018.06.052.

[56] Lima RBW, Barreto SC, Alfrisany NM, Porto TS, De Souza GM, De Goes MF. Effect of silane and MDP-based primers on physico-chemical properties of zirconia and its bond strength to resin cement. Dent Mater 2019;35:1557–67. https://doi.org/10.1016/j.dental.2019.07.008.

[57] Lima RBW, Barreto SC, Alfrisany NM, Porto TS, De Souza GM, De Goes MF. Effect of silane and MDP-based primers on physico-chemical properties of zirconia and its bond strength to resin cement. Dent Mater 2019;35:1557–67. https://doi.org/10.1016/j.dental.2019.07.008.

[58] Lima RBW, Barreto SC, Hajhamid B, de Souza GM, de Goes MF. Effect of cleaning protocol on silica deposition and silica-mediated bonding to Y-TZP. Dent Mater 2019;35:1603–13. https://doi.org/10.1016/j.dental.2019.08.099.

[59] Hantsche H. Comparison of basic principles of the surface-specific analytical methods: AES/SAM, ESCA (XPS), SIMS, and ISS with X-ray microanalysis, and some applications in research and industry. Scanning 1989;11:257–80. https://doi.org/10.1002/sca.4950110602.

[60] Siegbahn K. Electron Spectroscopy for Chemical Analysis BT - Atomic Physics 3. In: Smith SJ, Walters GK, editors., Boston, MA: Springer US; 1973, p. 493–522.

Table 1. Description of the zirconia-based materials employed in this study.

Material	Translucency	Type	Composition - weight percent (wt%)	Manufacturer
IPS e.max ZirCAD	Medium Opacity	3Y-PSZ	ZrO_2 (88-95.5%), Y_2O_3 (> 4.5 % – ≤ 6%), HfO_2 (≤ 5%), Al_2O_3 (≤ 1%), other oxides (≤ 1%)	Ivoclar Vivadent, Liechtenstein
IPS e.max ZirCAD	Medium translucency	4Y-PSZ	ZrO_2 (86-93.5%), Y_2O_3 (> 6.5 % – ≤ 8%), HfO_2 (≤ 5%), Al_2O_3 (≤ 1%), other oxides (≤ 1%)	Ivoclar Vivadent, Liechtenstein
IPS e.max ZirCAD	Low translucency	3Y-PSZ	ZrO_2 (88-95.5%), Y_2O_3 (> 4.5 % – ≤ 6%), HfO_2 (≤ 5%), Al_2O_3 (≤ 1%), other oxides (≤ 1%)	Ivoclar Vivadent, Liechtenstein

Table 2. XPS elemental composition (Rel.At.%) as a function of RT-ALD number of cycles over MT/Y-PSZ.

Cycles	**Peaks**	**Y_3d_5**	**Si_2p**	**Zr_3d**	**C_1s**	**O_1s**
Control		3.7	1.3	17.2	28.4	49.4
1 cycle		3.5	6.1	18	11.7	60.7
2 cycles		3.5	6.7	16.9	22.4	62.8
5 cycles		1.9	16.0	9.7	11.9	63.7
10 cycles		1.9	16.0	9.7	11.9	60.5
20 cycles		1.8	19.8	8.4	3.9	66.1
40 cycles		0.7	27.4	0	10.8	61.5

Figure 1. (A). Schematic representation of the experimental design. (B). Specimens preparation for shear bond strength.

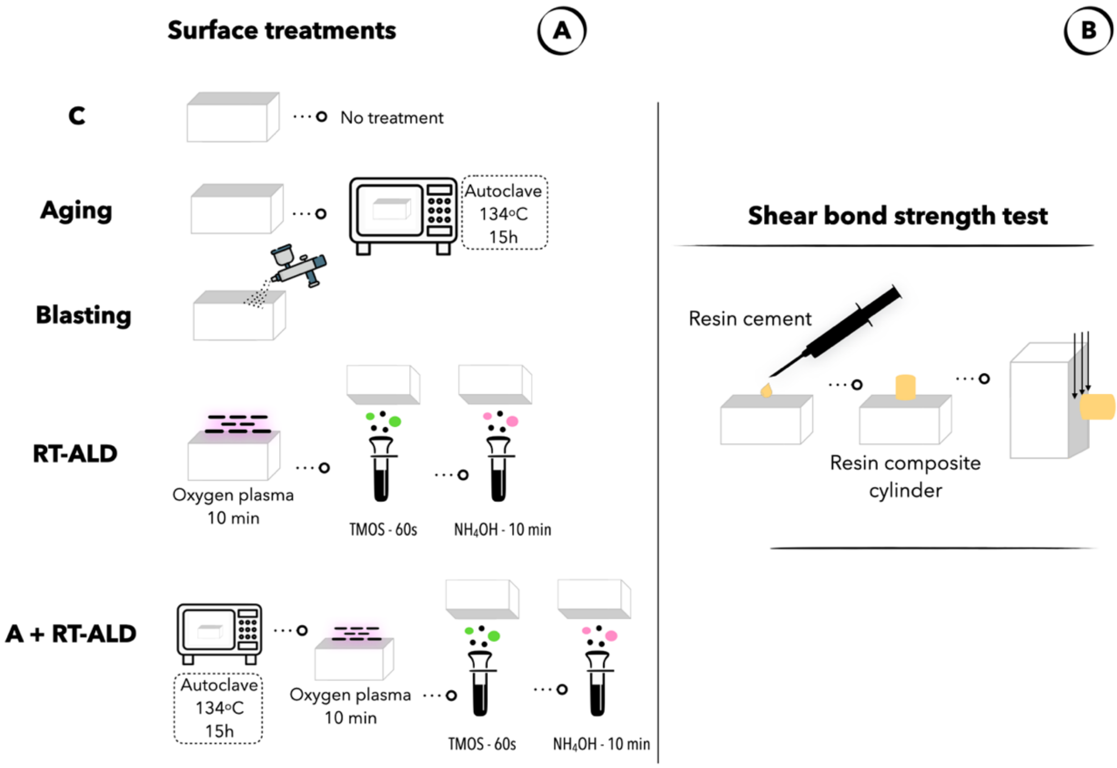
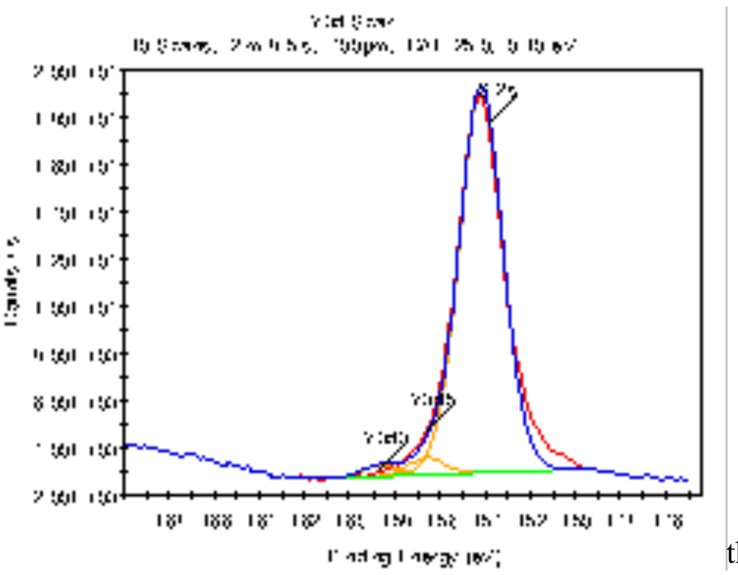
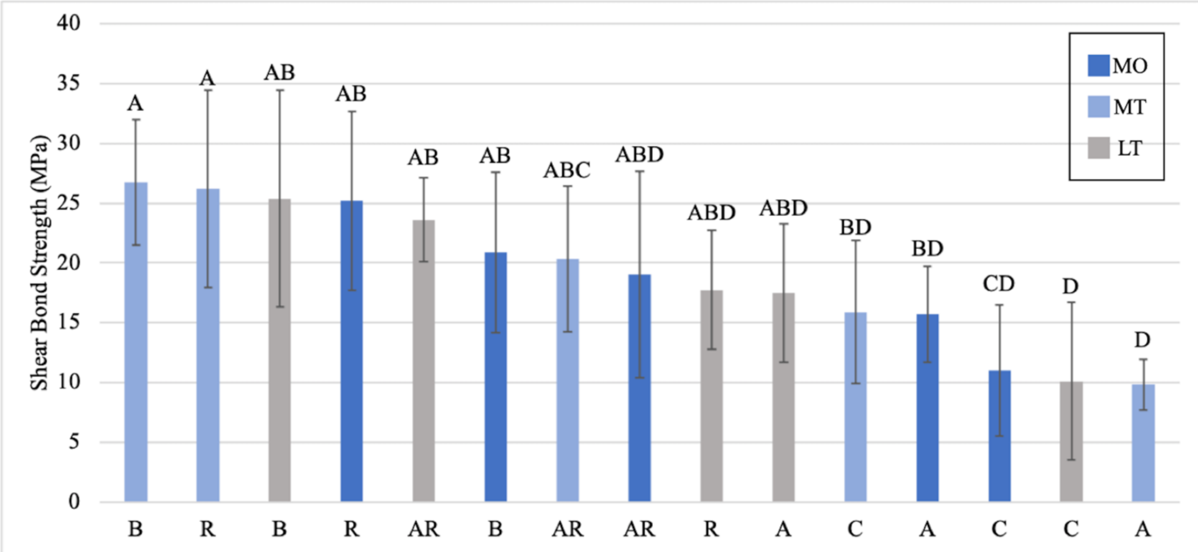


Figure 2. X-ray photoelectron spectroscopy of narrow-scan spectra of the Si2p region of Y-PSZ (medium translucency - MT) after 40 cycles of silica deposition.



F th (MPa) and Tukey test results (p≤0.05).



*Similar uppercase letters within indicate no significant difference at the 5% significance level.

Figure 4. Failure mode analysis (%) of all groups and materials tested.

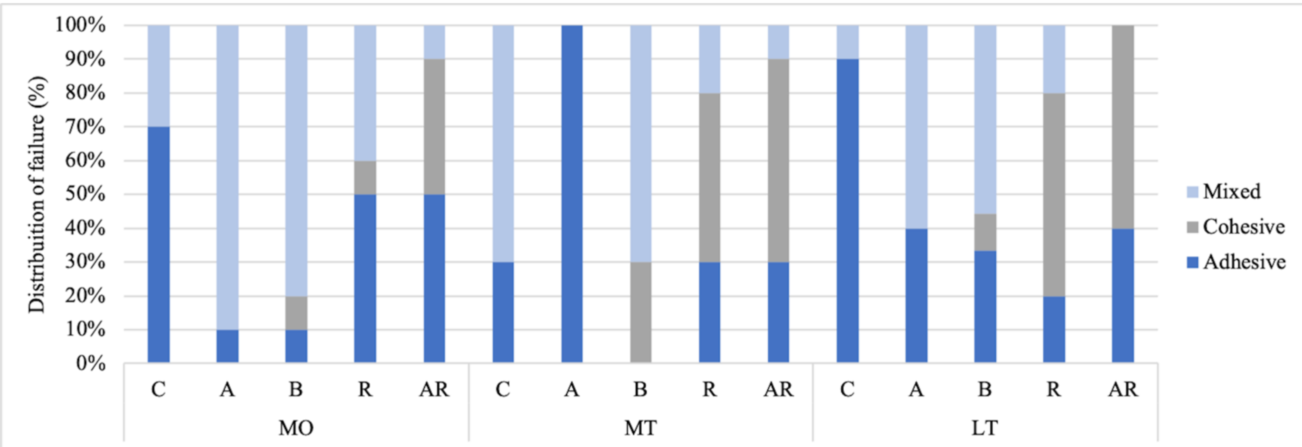


Figure 5. SEM characterization of zirconia specimens after the different surface treatments (X5000). SEM images showed a more regular surface for C (A, F, K) A (B, G, L) and RT-ALD (D, E, I, J, N, O) groups than blasted groups (C, H, M), which presented a more irregular surface.

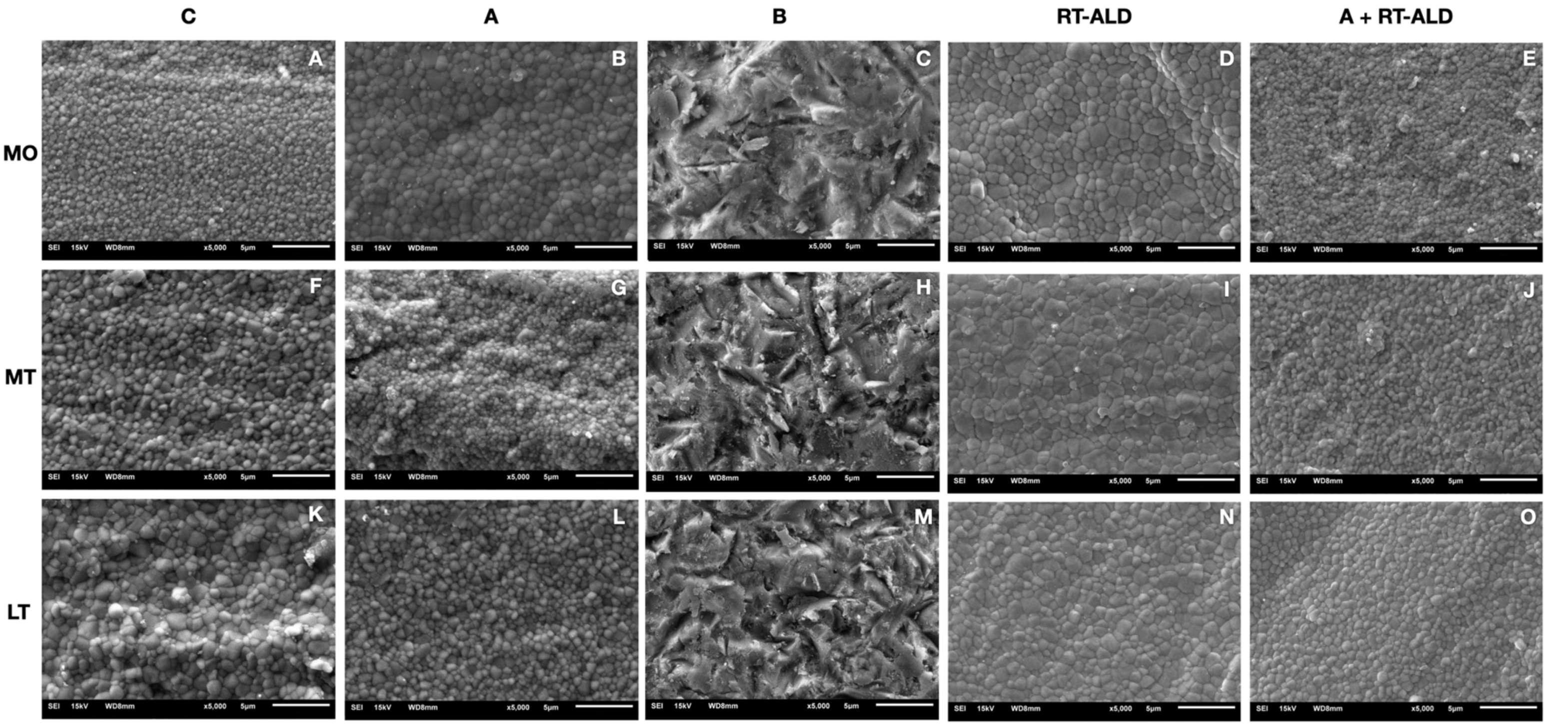
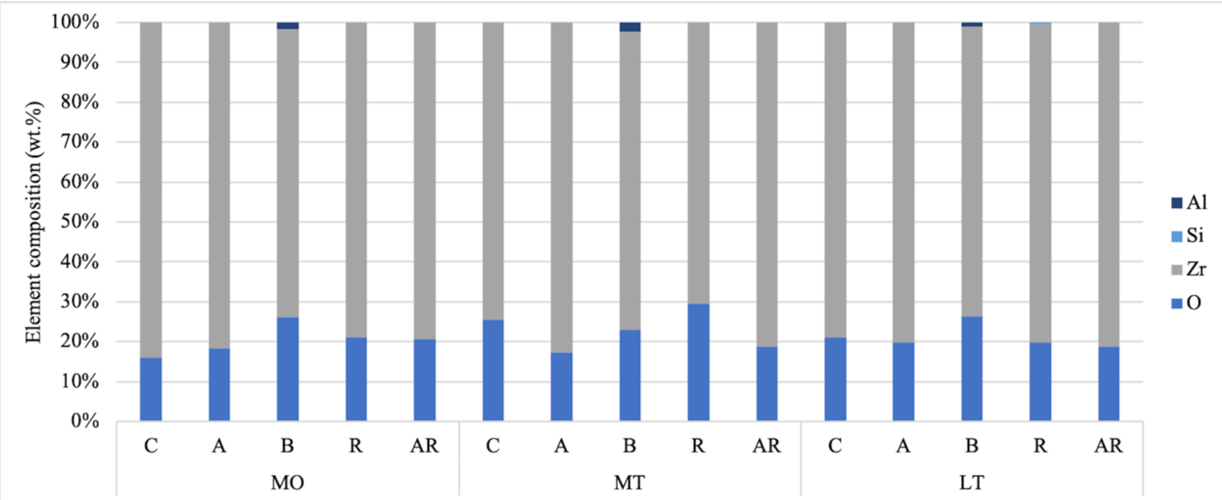


Figure 6. Elemental composition (wt.% - EDS) of all materials and groups evaluated.



2.2 ARTICLE 2

Silica deposition on zirconia via Room-Temperature Atomic Layer Deposition and bond strength to resin cement

This article was submitted to *Journal of the Mechanical Behavior of Biomedical Materials* and was in accordance with this journal.

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ABSTRACT

Objective: Silica-based nanofilm has been successfully deposited via Room-Temperature Atomic Layer Deposition (RT-ALD) on the surface of a glass. The purpose of this study was to evaluate the mechanical performance of the hybrid interface between yttria-stabilized zirconia (Y-PSZ) transformed layer and silica-based nanofilm deposited via RT-ALD.

Material and Methods: Fully-sintered Y-PSZ (14 x 4.0 x 1.5 mm) specimens in different translucencies (MO, MT, LT; IPS e.max Zircad, Ivoclar Vivadent) were distributed in 5 groups: control (C - no treatment); hydrothermal treatment (HT- 15h - 134°C, 2 bar); alumina blasting (B - 50 μm Al_2O_3); RT-ALD silica deposition (S); HT followed by silica deposition (HTS). RT-ALD cycles consisted of the sequential exposure of specimens to tetramethoxysilane orthosilicate (TMOS - 60s) and ammonium hydroxide (NH_4OH - 10 min) vapors in 40 cycles. Mechanical performance was analyzed by flexural strength (FS) ($n = 10$) and fatigue failure load (staircase method; $n = 20$) tests. Surface hardness (H) and Young's modulus (YM) were analyzed by nanoindentation. For surface chemical and topographical characterization, X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM) were performed. Data from surface H, YM, FS, and fatigue limit (FL) were analyzed by two-way analysis of variance (ANOVA).

Results: For all materials, S did not affect FS when compared to C group ($p > 0.269$), except for HTS-MO (807.65 MPa), which presented higher FS than the C-MO (436.23 MPa) ($p < 0.001$). Both S treatments showed similar FS values to B groups ($p > 0.410$). S did not affect FL when compared to the C group ($p > 0.277$) for all materials investigated. HTS resulted in higher FL than S for LT and MO materials ($p < 0.001$). Surface hardness and modulus were similar between control and S-treated specimens for all materials analyzed. XPS analysis showed homogeneous silica content after 20 and 40 RT-ALD cycles, and SEM did not show significant changes in surface morphology between C and S-treated specimens.

Conclusion: RT-ALD resulted in effective silica deposition without any deleterious effect on zirconia-based materials mechanical properties. Alumina blasting promoted higher alteration on surface topography. HT prior to S resulted in superior FL (for MO and MT) and flexural strength (MO) for some of the materials investigated.

Keywords: Dental ceramics, Flexural strength, Phase transformation, Nanoindentation, Hybrid layer.

1. INTRODUCTION

The temperature-dependent crystalline phases of zirconia have been thoroughly investigated and reported in the literature (Catledge et al., 2003; Chevalier, 2006). The tetragonal (*t*) phase is associated with the highest mechanical properties of zirconia, such as flexural strength (FS) (Kim et al., 2009), fracture toughness (Harada et al., 2016), and surface hardness (Catledge et al., 2003). Tetragonal zirconia also results in optimal clinical performance (Miura et al., 2020; Spies et al., 2019) when compared to other dental ceramics. An in vitro study reported that zirconia fixed partial dentures (FPDs) presented a lower failure rate than lithium disilicate FPDs up to 7.5 years (Sulaiman et al., 2020). In addition, high survival rates of zirconia-based posterior single crowns supported by zirconia implants have been reported after 5 years (Spies et al., 2019). Monolithic zirconia crowns in molars have survival rates as high as 90% after 3.5 years (Miura et al., 2020).

The outstanding mechanical properties of zirconia are related to the metastability of the tetragonal phase at room temperature (Shukla and Seal, 2005). Zirconia's capacity to halt a crack from propagating due to local *t-m* transformation triggered by compressive stresses is very unique and increases the work to fracture of the material (Garvie et al., 1975; Hannink et al., 2000). Low temperature degradation (LTD) is, however, an undesired outcome of the metastability of the tetragonal phase (Chevalier et al., 1999; Kobayashi et al., 1981). LTD is characterized by an uncontrolled *t→m* transformation in the presence of water, (Chevalier et al., 2011) that proceeds gradually from the surface into the bulk of the material and micro- and macrocracks develop in the wake of the volume expansion caused by the phase transformation (Chevalier et al., 2007; Sato et al., 1985).

Accelerated hydrothermal treatment (HT) has been used to simulate the long-term aging of zirconia-based materials (Chevalier et al., 1999; De Souza et al., 2017; Kim and Kim, 2019). The 3Y-TZP transformed layer presents a transitional layer of tetragonal and monoclinic grains surrounded by microcracks, which happen due to the dislodgement of the martensitic plates, indicating the porosity of the t-m microcracked layer (Pinto et al., 2016). XRD analysis showed that some grains within the transformed layer do not possess twin lamellae, indicating an incomplete transformation (Keuper et al., 2014). Therefore, the interconnection of the t-m microcracked layer makes it a permeable membrane for infiltration/deposition of nano-scale materials. Consequently, a controlled growth of the t-m transformed layer has the potential to be used for the infiltration of nano-scale materials.

Functionally-graded materials (FGMs) are an advanced class of engineered materials characterized by the presence of two or more materials with spatial variations in their structure and/or chemical composition to improve properties and functionalities of the material (Kawasaki and Watanabe, 1997; Parihar et al., 2018). Based on this concept, FGM technique has been used to develop biomedical zirconia (Volpato et al., 2019). However, fabrication of a biomaterial by FGM technique is challenging task (Parihar et al., 2018). Micro- and macro-structure design of FGMs is a challenge for the modern industry since there is a need for advancement in the processing technique for the production and upgrade of existing FGM processes (Parihar et al., 2018). With these limitations in mind and with the complexity of the FGMs production technique, other simple and viable techniques have been described in the literature by the deposition of oxide nanofilms (Hatton et al., 2010).

Atomic layer deposition (ALD) is generally the elected technique to deposit nano-scale oxides in the surface of materials for engineering applications (Hatton et al., 2010; Yu et al., 2009). ALD allows the sequential deposition of monolayers of materials under controlled thickness with reactive vapor precursors (Leskelä and Ritala, 2002; Rauwel et al., 2008). Organized and controlled silica (SiO_2) deposition can be obtained by ALD in different inorganic materials, including Al_2O_3, TiO_2, ZnO, and ZnS (Hatton et al., 2010). Synthesis of silica at room-temperature (RT) - ALD was successful to enable uniform deposition of 0.2 nm thick layers, after sequential exposure of glass specimens to tetramethoxysilane (TMOS) and ammonia (NH_3) vapors (Hatton et al., 2010).

RT-ALD has not been investigated as a surface treatment for zirconia-based materials. It is possible that the infiltration of silica or other oxides through the microcracked layer of zirconia developed under controlled hydrothermal conditions will result in the successful development of a hybrid surface, improving the chemical and biomechanical performance of the material. The infiltration of metal oxides into the permeable 3Y-TZP *t-m* transformed layer may also seal the surface of Y-PSZ against humidity, controlling LTD in the long-term. Therefore, this study aimed to evaluate the effect of a silica-zirconia hybrid surface, with and without the development of a microcracked layer, on the bulk and surface mechanical properties of dental zirconia. The study null-hypothesis was that surface treatment would have no effect on the mechanical performance of the experimental materials.

2. MATERIALS AND METHODS

2.1 Preparation of specimens

Zirconia pre-sintered blocks (IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein) in three different translucencies (Table 1) were cut using a precision cutter (Isomet® 1000 Buehler, Lake Bluff, IL, USA) with slow speed water-cooled diamond saw (15LC series, Diamond no. 11-4254, Buehler, Lake Bluff, IL, USA). Specimens were sintered in a furnace (Programat® S1, Ivoclar Vivadent, Schaan, Liechtenstein) following manufacturers' instructions and were cleaned in an ultrasonic bath (QuickClean; Midmark) in acetone for 5 minutes. The sintered specimens (15 x 4 x 1.5 mm) were randomly divided into 5 groups according to surface treatment (Table 2).

Mechanical performance was analyzed by flexural strength (n = 10) and fatigue failure load (n = 20) tests (staircase method). Surface hardness (n = 5) and modulus of elasticity (n = 5) were analyzed by nanoindentation. For surface chemical and topographical characterization, X-ray photoelectron spectroscopy (XPS; n = 1) and scanning electron microscopy (SEM; n = 1) were performed.

2.2 Hydrothermal treatment

Specimens were placed in autoclave (Ritter M9 Midmark Sterilizer, Versailles, Ohio, USA) under 2 bar pressure at 134 ºC for 15 hours (Pereira et al., 2016; Xiao et al., 2012).

2.3 Alumina blasting treatment

For alumina blasting, the surface was treated with 50 µm alumina particle abrasion under 2 bar pressure, from a perpendicular distance of 10 mm for 10s. After treatment, the specimens were cleaned in an ultrasonic bath (QuickClean; Midmark) in alcohol for 5 minutes.

2.4 Room-Temperature Atomic Layer Deposition (RT-ALD)

Silica deposition via RT-ALD (S) was performed by exposing specimens to tetramethoxysilane orthosilicate (TMOS - 60s) (Sigma-Aldrich, 98%) and ammonium hydroxide (NH_4OH - 10 min) (Sigma-Aldrich, 30 Wt% solution) vapors for 40 cycles (Hatton et al., 2010). Specimens were exposed to oxygen plasma for 10 min (PDC-001 plasma cleaner, Harrick Plasma) every 5 cycles, to enhance surface hydrophilicity. The exposure cycles were performed in a fume hood. For the vapor deposition, specimens were placed in a basket that was suspended inside a glass tube containing the corresponding liquid, and the basket was placed 2 cm above the liquid (Figure 1). The exposure cycles were performed at room temperature (~22 °C).

2.5 X-ray photoelectron spectroscopy analysis

A pilot study determined the number of RT-ALD cycles based on homogeneous deposition of silica over zirconia. The chemical analysis of the presence of silica was detected by XPS of medium translucency (MT) specimens after 0 (control), 1, 2, 5, 20, and 40 cycles of RT-ALD. Quantitative analysis was conducted using a Thermofisher Scientific K-Alpha system (Thermofisher Scientific—E. Grinstead, UK). The scanning of narrow-scan spectra (Y_3d_5, Si_2p, Zr_3d, C1s, O1s) of each specimen followed the parameters: nominal 400 µm spot, 25 eV pass energy, using a source gun of Al K Alpha, with standard lens mode, and energy step size of 0.100 eV.

2.6 Flexural strength

Three-point-bending test was selected for the analysis of flexural strength (FS) (ISO 6872:2015) (International Organization for Standardization ISO 6872:2015, 2015). The specimens were placed on two supports (10 mm span) and the test was performed (Instron 8501; Instron, Canton, Mass) with a central load at a crosshead speed of 1 mm/min. The load at fracture (σ) was obtained in Newtons (N) and converted to megapascals (MPa) following the equation:

$$\sigma = Pl/2wb^2$$

where σ is the maximum center tensile stress (MPa), P is fracture load (N), l is the span (mm), w is the specimen length (mm), and b is the specimen height (mm).

Flexural strength results were used as reference values for the definition of the initial load and step size in the fatigue failure test.

2.7 Fatigue test

Fatigue testing (n = 20) was performed using the staircase method (J. . Collins, 1993). The test was performed under the same three-point-bending setup (Instron 8501; Instron, Canton, Mass) with each specimen immersed in distilled water at room-temperature (Alshamrani and De Souza, 2020; Kelly et al., 2010). The parameters for the fatigue test were: 100,000 cycles, frequency of 5Hz (Alshamrani and De Souza, 2020).

The fatigue test parameters were: initial load was 70% of the mean flexural strength value for the corresponding group; step size was 5% of the mean flexural strength. If the specimen survived 100,000 cycles, the stress level was increased by the step size. If the specimen failed, the stress level was decreased by the step size. This step was performed for all specimens until all groups were analyzed (Vergani et al., 2010). Data was analyzed for each group and calculated based on the least frequent event data (survival or failure), according to the Dixon and Mood method.(Dixon and Mood, 1948) The mean fatigue limit (XL), standard deviation (S) were calculated according to the following formulas (1 and 2, respectively) (Table 5):(Dixon and Mood, 1948)

$XL = X_0 + d\ (A/N \pm 0.5)$ (1)

$S = 1.62\ d\ (NB - A^2/N^2 + 0.029)$ (2)

Where X_0 is the lowest stress recorded, d is the fixed stress increment, N is the sum of failures (or nonfailures) occurring at the different stress levels, A is the total sum of failures (or nonfailures) multiplied by the stress levels, and B is the total sum of failures (or nonfailures) multiplied by the square of the stress levels. A negative sign was used in formula 1 when the analysis was based on failures.

2.8 Nanoindentation

Surface hardness and modulus of elasticity were analyzed (Hysitron Triboindenter 950 - Minneapolis, MN, USA) on the surface of specimens. A Berkovich indenter (50 nm radius) performed 49 indentations in each specimen in a loading-partial unloading sequence in a grid of 7 x 7 with a spacing of 20 μm between the indentations and, penetration depth of 800 nm. Hardness and modulus were calculated using conventional unloading slope analysis (Oliver and Pharr, 1992).

2.9 SEM characterization

Surface characterization was performed in gold-sputtered specimens in a scanning electron microscope (SEM, JEOL 6610 LV, Jeol USA Inc., Peabody, MA, USA). Specimens were analyzed between 1,000 and 10,000 magnifications.

2.10 Data analysis

Data from flexural strength, fatigue limit (FL), surface hardness, and modulus of elasticity were analyzed by two-way analysis of variance (ANOVA) followed by one-way ANOVA. The comparisons between groups were performed with post-hoc Tukey's test ($\alpha = 0.05$) by software SPSS 20 (SPSS Inc., Chicago, IL, USA).

3. RESULTS

XPS spectra data (Rel.At. %) is shown in Table 5. The narrow-scan XPS spectra of the Si2p region (Figure 4) showed an increase in Si content while Zr decreased (Table 5) after 20 and 40 cycles.

The results of the two-way ANOVA indicated the interaction between material and treatment had a significant effect on FS ($p < 0.001$). The FS values ranged from 436.23 MPa to 856.65 MPa (Table 3). The HT group for LT material presented the highest FS value (856.65 MPa) and the lowest FS value (436.23 MPa) was from the C group for MO material. For all materials, both S treatments showed similar FS values to B groups ($p > 0.410$) and C groups ($p > 0.269$), except for the HT in S treatment (MO – 807.65 MPa) that increased the FS values when compared to C group (MO - 436.23 MPa) ($p = 0.000$). The lowest values for HT (475.62 MPa) ($p < 0.004$) and S treatment with HT (485.97 MPa) ($p < 0.029$) groups were from MT material.

Fatigue results and test parameters are presented in Table 3. The pattern of survival and failure for each group is detailed in Figure 2. The fatigue limits ranged from 296.79 MPa to 678.18 MPa. The lowest FL value (296.79 ± 20.42 MPa) was presented by HTS treatment of MT specimens, while the highest FL (678.18 MPa) was presented by LT specimens after HT. S treatment did not affect FL of any of the materials when compared to the C group ($p > 0.277$). MO and LT materials showed higher FL values after HT (MO - 661.00 MPa; LT - 678.18 MPa) as well as after HTS (MO - 599.01 MPa; LT - 659.49 MPa) in comparison to the C groups (MO – 392.61 MPa; LT - 386.99 MPa) ($p < 0.001$). Only for LT material, the S with HT (659.49 MPa) presented higher FL values compared to B group (496.28 MPa) ($p < 0.001$). When comparing groups of different materials (MO, MT, and LT), B groups from MO presented better performance (582.75 MPa) than the other ($p < 0.001$). Overall, MT showed the lowest FL values after HTS (325.24; 296.79 MPa) in comparison to MO (599. 01 MPa) and LT (659.49 MPa) ($p < 0.001$) exposed to the same treatment.

The results of two-way ANOVA for surface hardness and Young's modulus indicated that the interaction material*treatment was significant ($p < 0.001$) (Table 4). With the exception of MO zirconia, all the other groups showed similar surface hardness between B and S treated specimens ($p > 0.535$). HT affected surface hardness of MT and LT zirconia (6.74 GPa; 11.4GPa, respectively) in comparison to their C groups (12.70 GPa; 14.16 GPa, respectively) ($p < 0.021$). Surface hardness was similar between control and S treated zirconia with and without HT ($p = 0.07$).

The results of Young's modulus are presented in Table 4. According to the Tukey HSD comparison between materials, similar values were observed between the experimental treatments (S and HTS), alumina blasting and control ($p > 0.575$). Only for LT material, the C group (254.34 GPa) presented higher modulus than hydrothermal treatment (141,98 GPa) ($p = 0.035$).

Surface morphology (Figure 3) shows that the C groups (Figure 3A, F, K) presented a more regularity of the grains size than HT for all materials analyzed. All B groups (Figure C, H, M) exhibited similar topography with grooves irregularly generated by the high impact of alumina particles. Similar surface morphology for S groups (Figure 3D, I, N, E, J, O) was also observed, regardless of the material used, and similarities were observed between these specimens and control and hydrothermal-treated zirconia.

4. DISCUSSION

To our knowledge, this is the first study to test the deposition of silica on zirconia via RT-ALD. According to the findings of this study, the fatigue limits of RT-ALD-treated specimens were either equal to or higher than the control group. Therefore, the hypothesis that RT-ALD silica deposition would not have an effect on the mechanical performance of the experimental materials was accepted.

The flexural strength values observed in the present study showed that RT-ALD alone or combined with HT did not compromise FS, since the experimental groups presented higher than or similar results to the control group (Table 5). HT alone also did not affect FS of zirconia specimens. It is possible that the flexural strength test is not sensitive enough to identify the effect of phase transformation on the bulk zirconia flexural strength (De Souza et al., 2017). Similar FS results have been reported when aged and non-aged Y-PSZ were compared (Alghazzawi et al., 2012; Borchers et al., 2010; De Souza et al., 2017; Papanagiotou et al., 2006). When the effect of material and treatment on FS was analyzed, MT zirconia showed lower FS values for the HT-group and the HTS in comparison to the other materials ($p < 0.001$). It can be hypothesized that slight differences in materials composition may have influenced the flexural strength values encountered, since MT (4Y-PSZ) presents a larger cubic phase in proportion to the tetragonal phase (≅ 30%) (Jansen et al., 2019), providing better translucency, however decreasing the FS (Jerman et al., 2020).

The staircase method is considered an effective approach to determine the stress fatigue of a material under a specific lifetime (J. A. Collins, 1993). The high initial load and the load increase by the step size lead to the failure of the specimens within the test parameters previously established, an approach that combines accuracy with time effectiveness. However, it is important to emphasize that a higher number of cycles would be necessary to effectively reproduce the clinical fatigue of a dental material, with one million cycles approximately representing one year in function (Wiskott et al., 1995). RT-ALD did not affect the fatigue limits when compared to the control group for any of the materials tested. This comes as no surprise, since RT-ALD is based on a vapor chemical reaction at room temperature, which does not incur in any surface damage (Hatton et al., 2010). When the fatigue limit of the different materials was compared, the lowest values were presented by MT zirconia (Table 5). Therefore, these results suggest that the composition of MT (4Y-PSZ) influenced the mechanical properties evaluated, and this can be explained by the commitment of the toughening mechanism based on the *t-m* (Bogna Stawarczyk, Christin Keul, Marlis Eichberger, David Figge, Daniel Edelhoff, 2017), making this material more susceptible to less fracture resistance.

The characterization of possible changes in the surface caused by the new treatment proposed in this study was evaluated by the nanoindentation technique, which a sensitive test to capture microcracks on the Y-PSZ surface (Alghazzawi et al., 2012). In this study, S treatment did not affect negatively the hardness and Young's modulus of the materials when compared to the control group (Table 4). According to Hatton et al., 2010 (Hatton et al., 2010), 5 cycles of RT-ALD promoted a SiO_2 layer around 20 nm thick in polystyrene spheres, therefore it is expected a 160 nm layer created after 40 cycles in this study. Hence, considering that the penetration depth of the nanoindentation test is approximately 800 nm and that the silica layer was around 160 nm, the properties of the zirconia were measured and not of the silica layer, explaining the non-change in the hardness and Young's modulus values. Also, RT-ALD presented results comparable to the blasting group. Also, as expected, RT-ALD silica deposition did not affect the mechanical properties of zirconia since this treatment does not promote surface abrasion like the blasting treatment. Therefore, this result implies that the proposed surface treatment performed well and may be an alternative to blasting treatment.

Alumina abrasion has been indicated as a surface treatment to improve bond strength by creating a rougher surface of the zirconia coping, and different application protocols, pressure, distance, and size of particles have been employed and characterized (Kern, 2015; Ozcan and Bernasconi, 2015; Souza et al., 2013). However, alumina abrasion can cause surface defects that act as a source of stress, possibly leading to the failure of the material (Khan et al., 2017; Spazzin et al., 2017). There is also evidence that alumina particle abrasion induces t-m phase transformation (Aurélio et al., 2016; Özcan and Bernasconi, 2015). The SEM micrographs of alumina blasted specimens in this study showed irregularities caused by the impact of the alumina particles on the Y-PSZ surface (Figure 3 C, H, M), similar to images previously reported (Wang et al., 2008). S-treated specimens presented a more homogeneous surface (Figure 3D, I, N, E, J, O). When looking at the results for fatigue limit (Table 3), flexural strength (Table 3), Young's modulus (Table 4), and hardness (Table 4), RT-ALD as a surface treatment resulted in either similar to or superior properties than the alumina-blasted specimens. These indicate that RT-ALD is less damaging to the surface than alumina blasting, and it is worth to further investigate the crystalline stability of the Y-PSZ specimens treated by RT-ALD.

XPS analysis of silica deposition showed an increase of Si content and a decrease of Zr after 20 and 40 cycles of RT-ALD (Table 5). Based on the decrease of Zr content, the potential of RT-ALD for the effective deposition of silica is indicated, which would drive to a chemical bonding to zirconia instead of micromechanical interlocking, as promoted by alumina blasting.

The mechanical properties of a ceramic material have great impact on the prosthesis clinical performance (Guilardi et al., 2019). The findings of this study suggest that the proposed RT-ALD treatment may be an alternative for the deposition of silica on the Y-PSZ surface, since it has not affected the mechanical properties of the materials tested. The mechanical stability of the specimens in this study is reported by the fatigue resistance for up to 100,000 cycles. More studies are encouraged with different fatigue methodologies, considering exposure to water and other clinically-related variables. Also, the clinical reproducibility of the challenges in this study is impaired by specimen geometry, the loading protocols, and the standardized specimen preparations, however it is necessary in the initial *in vitro* studies. Thus, further *in vitro* and clinical studies are necessary to evaluate this subject to corroborate our findings.

5. CONCLUSION

Based on the limitations and conditions of this study, the following conclusions can be drawn:

- RT-ALD, with or without HT, did not affect the mechanical properties evaluated.
- The surface topography of the blasted groups indicated surface damage.
- RT-ALD was shown to be an effective technique for silica deposition on zirconia specimens.
- Zirconia composition had an effect on mechanical performance – flexural strength and fatigue limit.

TABLES

Table 1. Description of the zirconia-based materials employed in this study.

Material	Translucency	Type	Composition - weight percent (wt%)	Manufacturer
IPS e.max Zircad	Medium Opacity	3Y-PSZ	ZrO2 (88-95.5%), Y2O3 (> 4.5 % – ≤ 6%), HfO2 (≤ 5%), Al2O3 (≤ 1%), other oxides (≤ 1%)	Ivoclar Vivadent, Liechtenstein
IPS e.max Zircad	Medium translucency	4Y-PSZ	ZrO_2 (86-93.5%), Y_2O_3 (> 6.5 % – ≤ 8%), HfO_2 (≤ 5%), Al2O3 (≤ 1%), other oxides (≤ 1%)	Ivoclar Vivadent, Liechtenstein
IPS e.max Zircad	Low translucency	3Y-PSZ	ZrO2 (88-95.5%), Y2O3 (> 4.5 % – ≤ 6%), HfO2 (≤ 5%), Al2O3 (≤ 1%), other oxides (≤ 1%)	Ivoclar Vivadent, Liechtenstein

Table 2. Study design according to surface treatment.

Groups/Material	MO	MT	LT
Control - C	No surface treatment		
Alumina blasting - B	Airborne abrasion with 50 µm Al_2O_3 particle under 2 bar pressure, from a distance of 10 mm perpendicularly, for 15s		
Hydrothermal treatment - HT	Hydrothermal treatment in autoclave under 2 bar pressure at 134 ^oC for 15 hours		
RT-ALD - S	Exposure to TMOS for 60s and NH_4OH for 10 min vapors in 40 cycles. Oxygen plasma cleaning for 10 min every 5 cycles.		
Hydrothermal treatment + RT-ALD - HTS	Controlled followed by RT-ALD using the same parameters previously described		

Table 3. Means (standard deviations) of flexural strength, initial fatigue strength, step size (correspondent to 5% of the initial strength), fatigue strength and strength decrease (from flexural strength to fatigue strength - %).

Material	Group	Mean (±SD) of flexural strength (MPa)	Initial strength (70% of FS)	Step size (5% of FS)	Mean (±SD) of fatigue strength (MPa)	Strength decrease (%)
MO	**C**	436.23 (115.69)^{E}	305.36	21.81	392.61 (41.19)^{CDEFGH}	9.99
	HT	852.90 (144.09)^{AB}	597.03	42.64	661.00 (94.11)^{A}	22.4
	B	780.71 (209.75)^{ABD}	546.50	39.03	582.75 (71.52)^{A}	25.3
	S	620.19 (113.78)^{BE}	434.13	31	325.24 (65.84)^{CDEFG}	47.55
	HTS	807.65 (125.26)^{ABC}	565.35	40.38	599.01 (16.43)^{A}	25.83
MT	**C**	451.48 (103.43)^{E}	316.03	22.57	323.56 (30.51)^{DEFG}	28.33
	HT	475.62 (103.54)^{E}	332.93	23.78	321.04 (52.48)^{DEFG}	32.50
	B	637.52 (138.67)^{ABE}	446.26	31.87	345.32 (64.61)^{CDEFGH}	45.83
	S	514.51 (109.33)^{E}	360.15	25.72	325.24 (65.84)^{DEFG}	36.78
	HTS	485.97 (210.33)^{E}	340.18	24.29	296.79 (20.42)^{DEF}	38.92

	C	643.98 (79.20)^{ABE}	450.79	32.19	386.99 (49.32)^{CDEGH}	39.9
	HT	856.65 (148.30)^{A}	599.65	42.83	678.18 (63.69)^{A}	20.83
LT	**B**	759.34 (198.45)^{ABC}	531.54	37.96	496.28 (51.99)^{BCH}	34.64
	S	608.64 (136.36)^{CDE}	426.04	30.44	479.30 (72.29)^{BCDGH}	21.25
	HTS	766.22(224.56)^{ABC}	536.35	38.31	659.49 (32.19)^{A}	13.92

Similar uppercase letter within each column indicates no significant difference at the 5% significance level.

Table 4. Means and standard deviation of hardness and Young's modulus in (GPa) of each material.

Material	Group	Hardness (GPa)	Young's modulus (GPa)
MO	C	12.70 (1.80)^{ABC}	171.56 (41.05)^{AC}
	HT	11.40 (1.46)^{ABD}	210.90 (41.44)^{AC}
	B	9.03 (0.54)^{BD}	190.78 (66.67)^{AC}
	S	14.68 (3.67)^{A}	200.71 (35.58)^{AC}
	HTS	12.70 (1.69)^{ABC}	279.10 (54.58)^{A}
MT	C	14.91 (2.12)^{A}	222.21 (28.54)^{AB}
	HT	6.74 (3.66)^{D}	146.56 (34.41)^{BC}
	B	12.88 (3.9)^{ABC}	242.41 (51.69)^{AC}
	S	13.54 (0.28)^{ABC}	199.29 (26.32)^{AC}
	HTS	13.09 (1.57)^{ABC}	230.05 (42.60)^{AC}
LT	C	14.16 (3.57)^{AB}	254.34 (80.70)^{AB}
	HT	8.23 (1.19)^{CD}	141.98 (22.35)^{C}
	B	10.09 (1.55)^{AD}	172.95 (42.56)^{AC}
	S	12.94 (3.49)^{ABC}	218.84 (66.94)^{AC}
	HTS	9.67 (1.44)^{AD}	194.46 (51.56)^{AC}

Table 5. XPS analysis results (Rel.At. %) as a function of the number of RT-ALD cycles.

Groups \ Peaks	**Y3d5**	**Si2p**	**Zr3d**	**C1s**	**O1s**
ZrO_2-control	3.5	1.2	17.2	28.9	49.2
ZrO_2 1 cycle	2.3	12.0	12.4	12.5	60.8
ZrO_2 2 cycles	3.8	5.9	18.1	10.3	62.0
ZrO_2 5 cycles	3.2	9.1	16.4	9.2	62.2
ZrO_2 10 cycles	2.1	22.9	9.3	5.8	59.9
ZrO_2 20 cycles	0.8	21.9	3.1	11.3	63.0
ZrO_2 40 cycles	0.3	29.4	0.6	4.6	65.1

FIGURES

Figure 1. Schematic representation of 1 cycle of RT-ALD treatment.

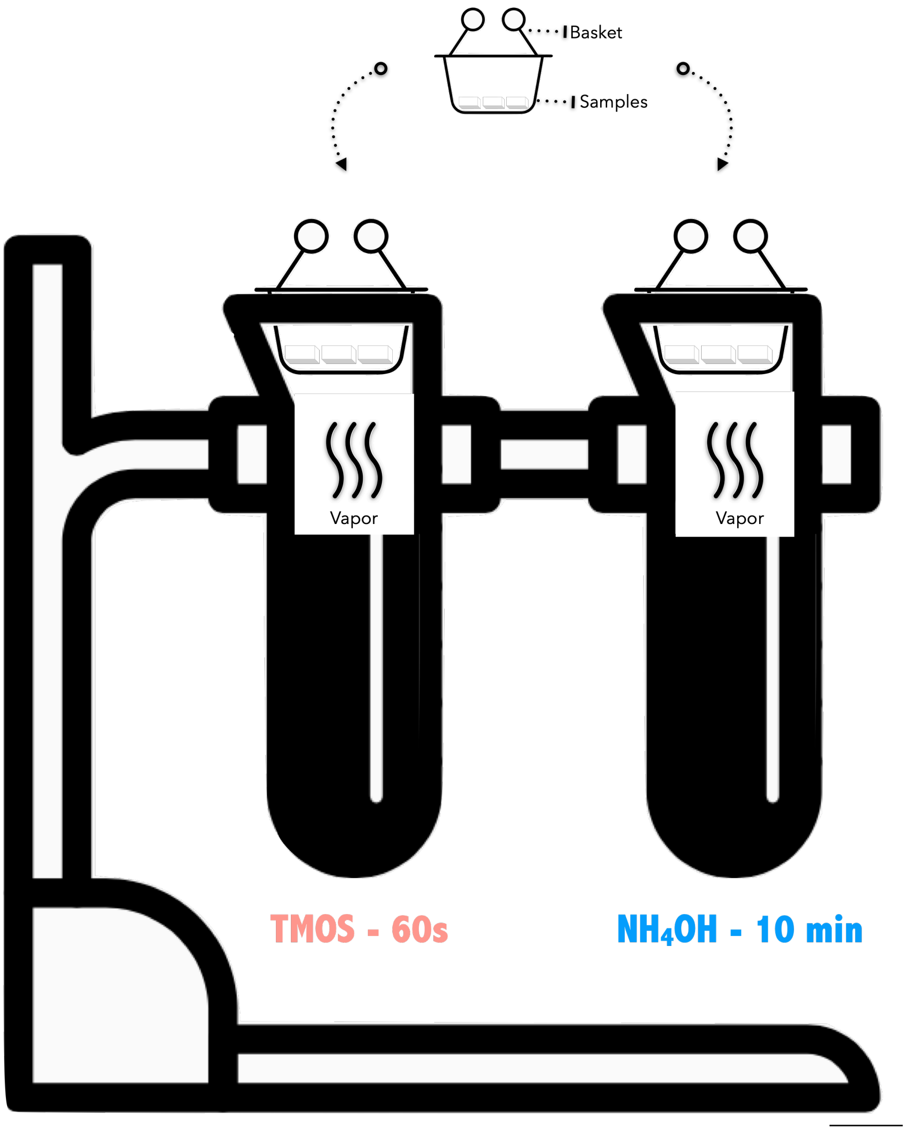


Figure 2. Staircase survival and failure patterns after fatigue tests for each group. A minimum of 15 specimens were tested after the first inversion of outcome (survival or failure) (arrows). The dashed lines indicate the mean load for fatigue strength, black dots indicate survived specimens; white dots indicate failed specimens. (C – control groups, B - blasted groups, HT- aged groups, S - RT-ALD silica deposition, HTS - hydrothermal treatment + silica deposition) and MO, MT, LT refer to different translucencies of the materials used.

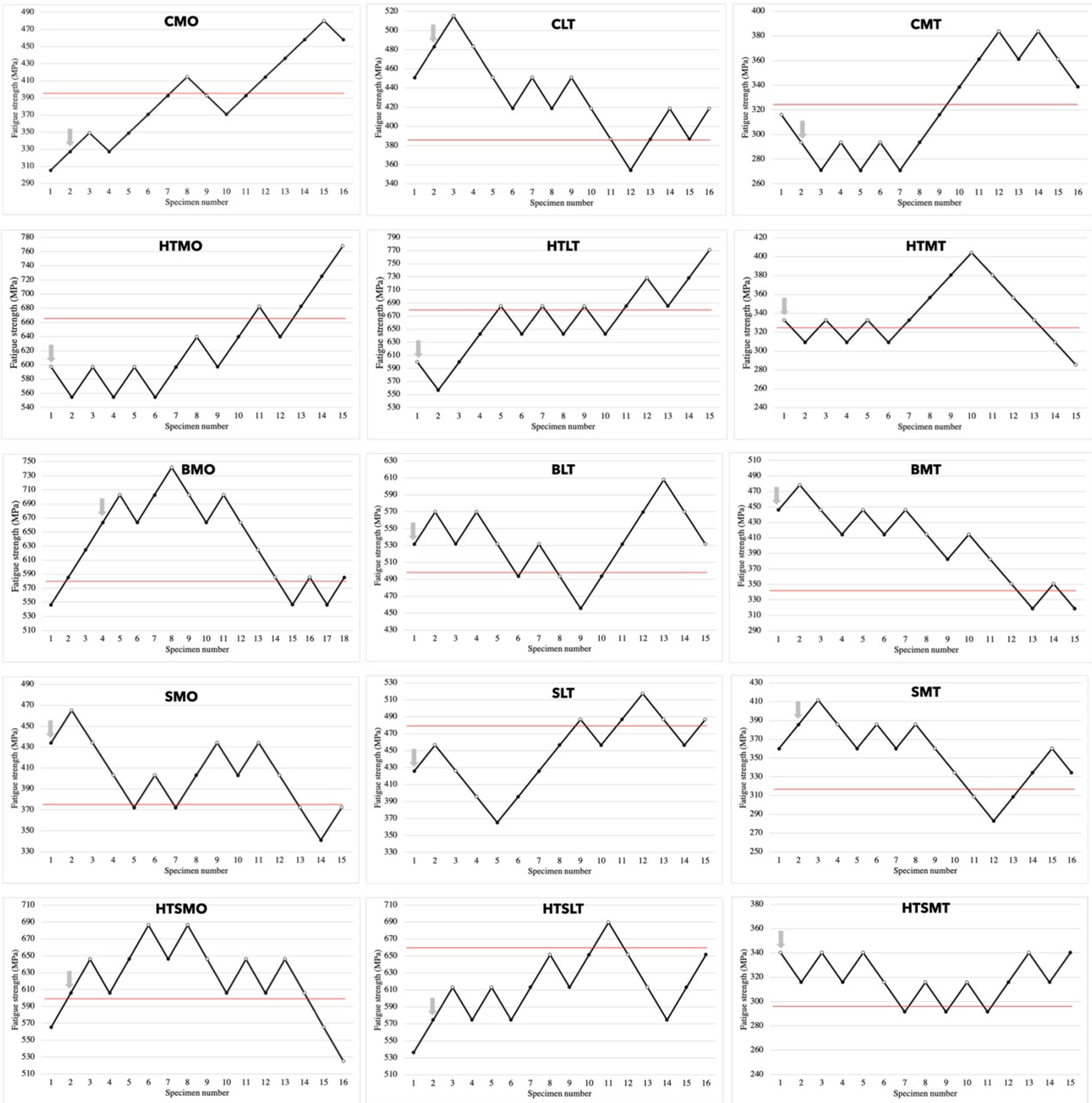


Figure 3. Scanning electron micrographs of zirconia surfaces for each experimental group (x10,000 magnification).

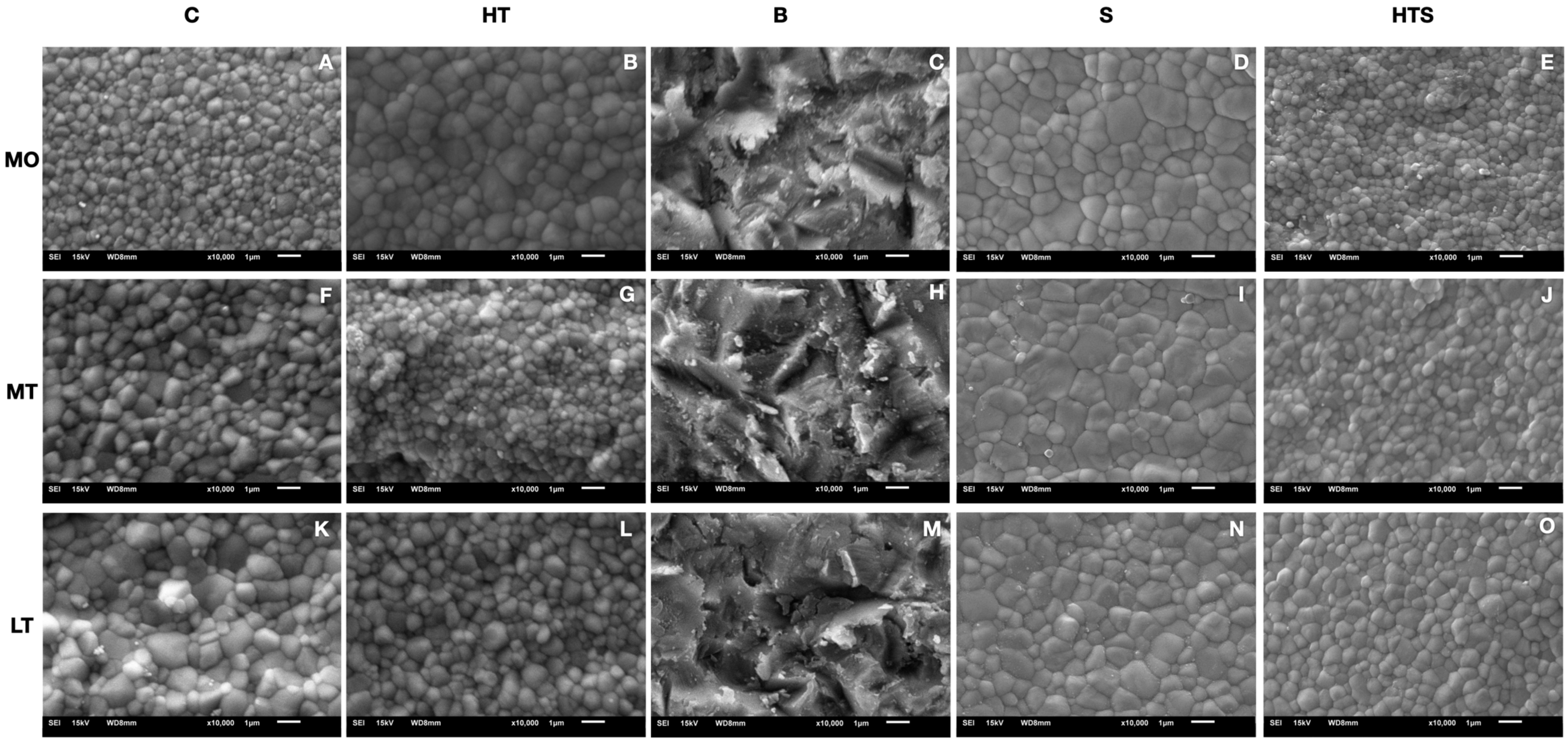
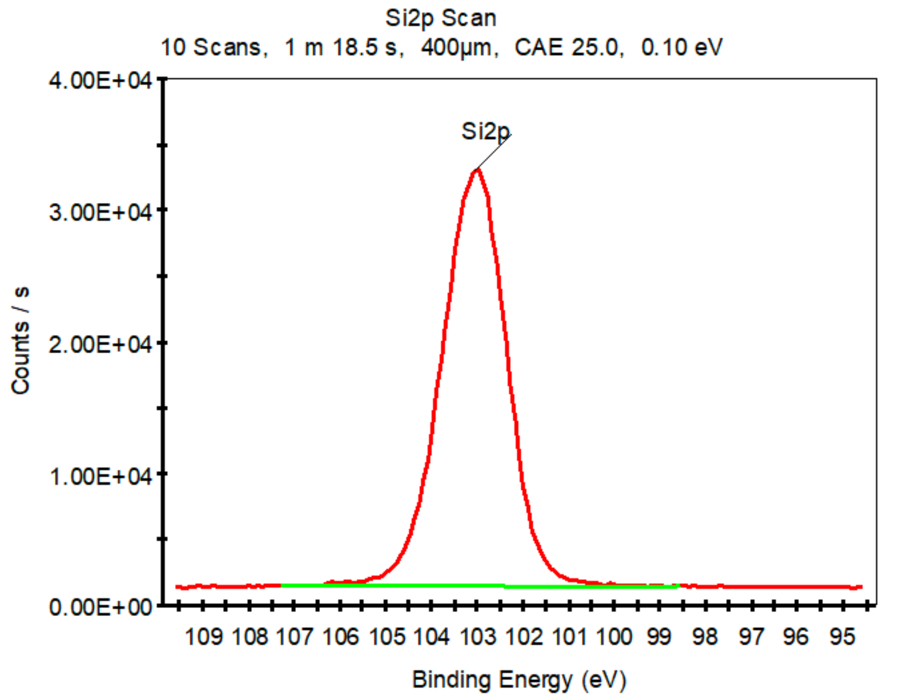


Figure 4. X-ray photoelectron spectroscopy of Y-PSZ Si2p region after 40 cycles.



6. REFERENCES

Alghazzawi, T.F., Lemons, J., Liu, P.-R., Essig, M.E., Bartolucci, A.A., Janowski, G.M., 2012. Influence of low-temperature environmental exposure on the mechanical properties and structural stability of dental zirconia. J. Prosthodont. 21, 363—369. https://doi.org/10.1111/j.1532-849x.2011.00838.x

Alshamrani, A.A., De Souza, G.M., 2020. Effect of ionizing radiation on mechanical properties and translucency of monolithic zirconia. J. Biomed. Mater. Res. - Part B Appl. Biomater. 108, 1068–1076. https://doi.org/10.1002/jbm.b.34458

Aurélio, I.L., Marchionatti, A.M.E., Montagner, A.F., May, L.G., Soares, F.Z.M., 2016. Does air particle abrasion affect the flexural strength and phase transformation of Y-TZP? A systematic review and meta-analysis. Dent. Mater. 32, 827–845. https://doi.org/10.1016/j.dental.2016.03.021

Bogna Stawarczyk, Christin Keul, Marlis Eichberger, David Figge, Daniel Edelhoff, N.L., 2017. Three Generations of Zirconia: From Veneered to Monolithic. Part I. Quintessence Int. 48, 369–380.

Borchers, L., Stiesch, M., Bach, F.-W., Buhl, J.-C., Hübsch, C., Kellner, T., Kohorst, P., Jendras, M., 2010. Influence of hydrothermal and mechanical conditions on the strength of zirconia. Acta Biomater. 6, 4547–4552. https://doi.org/https://doi.org/10.1016/j.actbio.2010.07.025

Catledge, S.A., Cook, M., Vohra, Y.K., Santos, E.M., McClenny, M.D., David Moore, K., 2003. Surface crystalline phases and nanoindentation hardness of explanted zirconia femoral heads. J. Mater. Sci. Mater. Med. 14, 863–867. https://doi.org/10.1023/a:1025678525474

Chevalier, J., 2006. What future for zirconia as a biomaterial? Biomaterials 27, 535–543. https://doi.org/https://doi.org/10.1016/j.biomaterials.2005.07.034

Chevalier, J., Cales, B., Drouin, J.M., 1999. Low-Temperature Aging of Y-TZP Ceramics. J. Am. Ceram. Soc. 82, 2150–2154. https://doi.org/10.1111/j.1151-2916.1999.tb02055.x

Chevalier, J., Gremillard, L., Deville, S., 2007. Low-Temperature Degradation of Zirconia and Implications for Biomedical Implants. Annu. Rev. Mater. Res. 37, 1–32. https://doi.org/10.1146/annurev.matsci.37.052506.084250

Chevalier, J., Loh, J., Gremillard, L., Meille, S., Adolfson, E., 2011. Low-temperature degradation in zirconia with a porous surface. Acta Biomater. 7, 2986–2993. https://doi.org/10.1016/j.actbio.2011.03.006

Collins, J.., 1993. Failure of Materials In Mechanical Design: Analysis, Prediction, Prevention, Second. ed, Failure of Materials In Mechanical Design: Analysis, Prediction, Prevention. New York.

Collins, J.A., 1993. Staircase or up-and-down methods: failure of materials in mechanical design.

De Souza, G.M., Zykus, A., Ghahnavyeh, R.R., Lawrence, S.K., Bahr, D.F., 2017. Effect of accelerated aging on dental zirconia-based materials. J. Mech. Behav. Biomed. Mater. 65, 256–263. https://doi.org/10.1016/j.jmbbm.2016.08.023

Dixon, W.J., Mood, A.M., 1948. A Method for Obtaining and Analyzing Sensitivity Data. J. Am. Stat. Assoc. 43, 109–126. https://doi.org/10.1080/01621459.1948.10483254

Garvie, R.C., Hannink, R.H., Pascoe, R.T., 1975. Ceramic steel? Nature 258, 703–704. https://doi.org/10.1038/258703a0

Guilardi, L.F., Pereira, G.K.R., Giordani, J.C., Kleverlaan, C.J., Valandro, L.F., Rippe, M.P., 2019. Effect of zirconia surface treatment, resin cement and aging on the load-bearing capacity under fatigue of thin simplified full-contour Y-TZP restorations. J. Mech. Behav. Biomed. Mater. 97, 21–29. https://doi.org/10.1016/j.jmbbm.2019.04.050

Hannink, R.H.J., Kelly, P.M., Muddle, B.C., 2000. Transformation toughening in zirconia-containing ceramics. J. Am. Ceram. Soc. 83, 461–487. https://doi.org/10.1111/j.1151-2916.2000.tb01221.x

Harada, K., Shinya, Akikazu, Gomi, H., Hatano, Y., Shinya, Akiyoshi, Raigrodski, A.J., 2016. Effect of accelerated aging on the fracture toughness of zirconias. J. Prosthet. Dent. 115, 215–223. https://doi.org/10.1016/j.prosdent.2015.08.020

Hatton, B., Kitaev, V., Perovic, D., Ozin, G., Aizenberg, J., 2010. Low-temperature synthesis of nanoscale silica multilayers - Atomic layer deposition in a test tube. J. Mater. Chem. 20, 6009–6013. https://doi.org/10.1039/c0jm00696c

International Organization for Standardization ISO 6872:2015, 2015. No Title. Dent. Mater.

Jansen, J.U., Lümkemann, N., Letz, I., Pfefferle, R., Sener, B., Stawarczyk, B., 2019. Impact of high-speed sintering on translucency, phase content, grain sizes, and flexural strength of 3Y-TZP and 4Y-TZP zirconia materials. J. Prosthet. Dent. 122, 396–403. https://doi.org/https://doi.org/10.1016/j.prosdent.2019.02.005

Jerman, E., Lümkemann, N., Eichberger, M., Zoller, C., Nothelfer, S., Kienle, A., Stawarczyk, B., 2020. Evaluation of translucency, Marten's hardness, biaxial flexural strength and fracture toughness of 3Y-TZP, 4Y-TZP and 5Y-TZP materials. Dent. Mater. https://doi.org/10.1016/j.dental.2020.11.007

Kawasaki, A., Watanabe, R., 1997. Concept and P/M fabrication of functionally gradient materials. Ceram. Int. 23, 73–83. https://doi.org/10.1016/0272-8842(95)00143-3

Kelly, J.R., Rungruanganunt, P., Hunter, B., Vailati, F., 2010. Development of a clinically validated bulk failure test for ceramic crowns. J. Prosthet. Dent. 104, 228–238. https://doi.org/10.1016/S0022-3913(10)60129-1

Kern, M., 2015. Bonding to oxide ceramics—laboratory testing versus clinical outcome. Dent. Mater. 31, 8–14. https://doi.org/10.1016/j.dental.2014.06.007

Keuper, M., Berthold, C., Nickel, K.G., 2014. Acta Biomaterialia Long-time aging in 3 mol .% yttria-stabilized tetragonal zirconia polycrystals at human body temperature. Acta Biomater. 10, 951–959. https://doi.org/10.1016/j.actbio.2013.09.033

Khan, A.A., Al Kheraif, A.A.A., Jamaluddin, S., Elsharawy, M., Divakar, D.D., 2017. Recent Trends in Surface Treatment Methods for Bonding Composite Cement to Zirconia: A Reveiw. J. Adhes. Dent. 19, 7–19. https://doi.org/10.3290/j.jad.a37720

Kim, H.-T., Han, J.-S., Yang, J.-H., Lee, J.-B., Kim, S.-H., 2009. The effect of low temperature aging on the mechanical property & phase stability of Y-TZP ceramics. J. Adv. Prosthodont. 1, 113–117. https://doi.org/10.4047/jap.2009.1.3.113

Kim, H.K., Kim, S.H., 2019. Effect of hydrothermal aging on the optical properties of precolored dental monolithic zirconia ceramics. J. Prosthet. Dent. 121, 676–682. https://doi.org/10.1016/j.prosdent.2018.06.021

Kobayashi, K., Kuwajima, H., Masaki, T., 1981. Phase change and mechanical properties of ZrO2-Y2O3 solid electrolyte after ageing. Solid State Ionics 3–4, 489–493. https://doi.org/https://doi.org/10.1016/0167-2738(81)90138-7

Leskelä, M., Ritala, M., 2002. Atomic layer deposition (ALD): From precursors to thin film structures. Thin Solid Films 409, 138–146. https://doi.org/10.1016/S0040-6090(02)00117-7

Miura, S., Yamauchi, S., Kasahara, S., Katsuda, Y., Fujisawa, M., Egusa, H., 2020. Clinical evaluation of monolithic zirconia crowns: a failure analysis of clinically obtained cases from a 3.5-year study. J. Prosthodont. Res. https://doi.org/10.2186/jpr.JPOR_2019_643

Oliver, W.C., Pharr, G.M., 1992. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J. Mater. Res. 7, 1564–1583. https://doi.org/10.1557/JMR.1992.1564

Ozcan, M., Bernasconi, M., 2015. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. J. Adhes. Dent. 17, 7–26. https://doi.org/10.3290/j.jad.a33525

Özcan, M., Bernasconi, M., 2015. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. J. Adhes. Dent. 17, 7–26. https://doi.org/10.3290/j.jad.a33525

Papanagiotou, H.P., Morgano, S.M., Giordano, R.A., Pober, R., 2006. In vitro evaluation of low-temperature aging effects and finishing procedures on the flexural strength and structural stability of Y-TZP dental ceramics. J. Prosthet. Dent. 96, 154–164. https://doi.org/https://doi.org/10.1016/j.prosdent.2006.08.004

Parihar, R.S., Setti, S.G., Sahu, R.K., 2018. Recent advances in the manufacturing processes of functionally graded materials: A review. IEEE J. Sel. Top. Quantum Electron. 25, 309–336. https://doi.org/10.1515/secm-2015-0395

Pereira, G.K.R., Venturini, A.B., Silvestri, T., Dapieve, K.S., Montagner, A.F., Soares, F.Z.M., Valandro, L.F., 2016. Low-temperature degradation of Y-TZP ceramics : A systematic review and meta-analysis 55, 151–163.

Pinto, P.A., Colas, G., Filleter, T., De Souza, G.M., 2016. Surface and Mechanical Characterization of Dental Yttria-Stabilized Tetragonal Zirconia Polycrystals (3Y-TZP) After Different Aging Processes. Microsc. Microanal. 22, 1179–1188. https://doi.org/10.1017/S1431927616011843

Rauwel, E., Clavel, G., Willinger, M.-G., Rauwel, P., Pinna, N., 2008. Non-Aqueous Routes to Metal Oxide Thin Films by Atomic Layer Deposition. Angew. Chemie Int. Ed. 47, 3592–3595. https://doi.org/10.1002/anie.200705550

Sato, T., Ohtaki, S., Endo, T., Shimada, M., 1985. Transformation of Yttria-Doped Tetragonal ZrO2 Poly crystals by Annealing under Controlled Humidity Conditions. J. Am. Ceram. Soc. 68, C-320-C-322. https://doi.org/10.1111/j.1151-2916.1985.tb10133.x

Shukla, S., Seal, S., 2005. Mechanisms of room temperature metastable tetragonal phase stabilisation in zirconia. Int. Mater. Rev. 50, 45–64. https://doi.org/10.1179/174328005X14267

Souza, R.O.A., Valandro, L.F., Melo, R.M., Machado, J.P.B., Bottino, M.A., Özcan, M., 2013. Air–particle abrasion on zirconia ceramic using different protocols: Effects on biaxial flexural strength after cyclic loading, phase transformation and surface topography. J. Mech. Behav. Biomed. Mater. 26, 155–163. https://doi.org/https://doi.org/10.1016/j.jmbbm.2013.04.018

Spazzin, A.O., Bacchi, A., Alessandretti, R., Santos, M.B., Basso, G.R., Griggs, J., Moraes, R.R., 2017. Ceramic strengthening by tuning the elastic moduli of resin-based luting agents. Dent. Mater. 33, 358–366. https://doi.org/10.1016/j.dental.2017.01.002

Spies, B.C., Balmer, M., Jung, R.E., Sailer, I., Vach, K., Kohal, R.J., 2019. All-ceramic single crowns supported by zirconia implants: 5-year results of a prospective multicenter study. Clin. Oral Implants Res. 30, 466–475. https://doi.org/10.1111/clr.13433

Sulaiman, T.A., Abdulmajeed, A.A., Delgado, A., Donovan, T.E., 2020. Fracture rate of 188695 lithium disilicate and zirconia ceramic restorations after up to 7.5 years of clinical service: A dental laboratory survey. J. Prosthet. Dent. 123, 807–810. https://doi.org/10.1016/j.prosdent.2019.06.011

Vergani, C.E., Seó, R.S., Reis, J.M.S.N., Giampaolo, E.T., Pavarina, A.C., Machado, A.L., 2010. Effect of water storage on the shear strength and fatigue limit of the reline resin bond to denture base resins. J. Adhes. Dent. 12, 319–327. https://doi.org/10.3290/j.jad.a17652

Volpato, C.A.M., Carvalho, Ó.S.N., Pereira, M.R. da C., Correia Pereira da Silva, F.S., 2019. Evaluation of the color and translucency of glass-infiltrated zirconia based on the concept of functionally graded materials. J. Prosthet. Dent. 121, 547.e1-547.e7. https://doi.org/10.1016/j.prosdent.2018.09.019

Wang, H., Aboushelib, M.N., Feilzer, A.J., 2008. Strength influencing variables on CAD/CAM zirconia frameworks. Dent. Mater. 24, 633–638. https://doi.org/10.1016/j.dental.2007.06.030

Wiskott, H.W., Nicholls, J.I., Belser, U.C., 1995. Stress fatigue: basic principles and prosthodontic implications. Int. J. Prosthodont. 8, 105–116.

Xiao, R., Chu, B.-F., Zhang, L., Cao, J.-K., 2012. Aging performances for resisting low-temperature of three dental Yttria-stabilized zirconia ceramic core materials. Chin. Med. J. (Engl). 125, 1999–2003.

Yu, X., Zhang, H., Oliverio, J.K., Braun, P. V, 2009. Template-assisted three-dimensional nanolithography via geometrically irreversible processing. Nano Lett. 9, 4424–4427. https://doi.org/10.1021/nl9027236

3 FUNDAMENTED DISCUSSION

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This study evaluated the effect of silica nanofilm coating through RT-ALD treatment. It was believed that RT-ALD treatment would be a potential contributing factor to improve the bonding between Y-TZP and resin cement. Also, the study investigated the mechanical properties of the material would be affected by RT-ALD. In addition, the formation of microcracks in the Y-TZP surface after hydrothermal treatment could promote a better relationship between the silica nanofilm and the Y-TZP surface since silica could penetrate the surface defects, increasing the contact area between the materials.

Zirconia is susceptible to LTD, due to the metastability of the tetragonal phase at room temperature [20,23]. Accelerated aging tests are used to simulate LTD, since it is thermally activated and can be performed at temperatures above 37ºC, with the combination of humidity, intermediate temperatures (134ºC), and pressure [6,27]. The hydrothermal aging in autoclave promotes a phase transformation toughening mechanism initiation, in response to a t–m phase transformation, leading to a temporary improvement of the mechanical properties related to maximized compressive stresses. However, studies have shown that those stresses are overall deleterious for the stability of Y-TZP-based devices [37,38].

In this study, hydrothermal aging was used to create a cracked layer more receptive to the infiltration of a silica nanofilm according to the characteristics of the transformed layer previously reported in the literature [25,39]. Under the conditions of the present study, hydrothermal aging combined with RT-ALD did not affect bond strength between zirconia and resin cement in comparison to RT-ALD alone ($p > .664$). It was expected that the permeability of the Y-TZP microcracked t-m layer would have an effect on the strength of the silica infiltrated/deposited layer, which was not observed via the SBS test. Also, regarding the mechanical properties, for fatigue limit (for MO and MT) and flexural strength (for MO) properties, the hydrothermal aging demonstrated to be positive in RT-ALD treatment when compared without aging and C group, respectively.

Different adhesion techniques have been studied on the zirconia surface, however an adhesive cementation protocol with reliable results has not been identified [30]. Atomic layer deposition is a chemical deposition technique in nanotechnology which allows deposition of solid thin films, such as silica [40]. However, the thickness control of the deposited thin films is obtained by the number of ALD cycles. This study demonstrated that RT-ALD treatment can be easily applied for the controlled deposition of silica layers at the nanoscale, since XPS analysis indicated effective deposition of a nanofilm of silica after 20 and 40 cycles of RT-ALD. Based on a previous study [36], 20 nm-thick silica growth after 5 cycles in polystyrene spheres. Therefore, a silica film thickness of approximately 160 nm was expected after 40 cycles in this study. Also, these results confirmed the better sensitivity of silica identification by XPS analysis, since the characterization depth is given at the nanoscale.

After the blasting treatment, a more damaged surface was observed and the weakening of the bonding interface is due to rougher surfaces that present a higher concentration of stresses [41]. Therefore, the RT-ALD silica deposition may be an alternative surface treatment, since silica deposition via RT-ALD treatment did not affect the mechanical properties evaluated and it has been shown effective in silica deposition on Y-TZP surface. Also, bond strength results between Y-TZP/resin cement was similar to alumina blasting. However, among the limitations of the present study, the experimental treatment has never been reported before in Y-TZP surface. Also, the difficulty in reproducing the clinical situation is represented by design and dimensions of the samples, the loading protocols, and the standardized specimen's preparations Nevertheless, these results should be carefully evaluated and the analysis of other surface cleaning methods is also encouraged.

4 CONCLUSIONS

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Within the limitations of the studies, it can be concluded that RT-ALD was an effective treatment for silica deposition on Y-PSZ and presented similar bond strength results to alumina blasting groups. This new treatment did not affect the mechanical properties, regardless hydrothermal treatment. Also, blasted groups presented damage in surface topography. Therefore, RT-ALD may be an alternative to blasting treatment. Further studies with different fatigue methodologies and loading protocols are encouraged, however it is necessary in the initial in vitro studies.

REFERENCES

REFERENCES

[1] Kelch M, Schulz J, Edelhoff D, Sener B, Stawarczyk B. Impact of different pretreatments and aging procedures on the flexural strength and phase structure of zirconia ceramics. Dent Mater 2019;35:1439–49. https://doi.org/10.1016/j.dental.2019.07.020.

[2] Alaniz JE, Perez-Gutierrez FG, Aguilar G, Garay JE. Optical properties of transparent nanocrystalline yttria stabilized zirconia. Opt Mater (Amst) 2009;32:62–8. https://doi.org/https://doi.org/10.1016/j.optmat.2009.06.004.

[3] Guazzato M, Albakry M, Ringer SP, Swain M V. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part II. Zirconia-based dental ceramics. Dent Mater 2004;20:449–56. https://doi.org/10.1016/j.dental.2003.05.002.

[4] Tinschert J, Zwez D, Marx R, Anusavice KJ. Structural reliability of alumina-, feldspar-, leucite-, mica- and zirconia-based ceramics. J Dent 2000;28:529–35. https://doi.org/https://doi.org/10.1016/S0300-5712(00)00030-0.

[5] Kim H-T, Han J-S, Yang J-H, Lee J-B, Kim S-H. The effect of low temperature aging on the mechanical property & phase stability of Y-TZP ceramics. J Adv Prosthodont 2009;1:113–7. https://doi.org/10.4047/jap.2009.1.3.113.

[6] Borchers L, Stiesch M, Bach F-W, Buhl J-C, Hübsch C, Kellner T, et al. Influence of hydrothermal and mechanical conditions on the strength of zirconia. Acta Biomater 2010;6:4547–52. https://doi.org/https://doi.org/10.1016/j.actbio.2010.07.025.

[7] Catledge SA, Cook M, Vohra YK, Santos EM, McClenny MD, David Moore K. Surface crystalline phases and nanoindentation hardness of explanted zirconia femoral heads. J Mater Sci Mater Med 2003;14:863–7. https://doi.org/10.1023/a:1025678525474.

[8] Harada K, Shinya A, Gomi H, Hatano Y, Shinya A, Raigrodski AJ. Effect of accelerated aging on the fracture toughness of zirconias. J Prosthet Dent 2016;115:215–23. https://doi.org/10.1016/j.prosdent.2015.08.020.

[9] Miyazaki T, Hotta Y, Kunii J, Kuriyama S, Tamaki Y. A review of dental CAD/CAM: current status and future perspectives from 20 years of experience. Dent Mater J 2009;28:44–56. https://doi.org/10.4012/dmj.28.44.

[10] Beuer F, Schweiger J, Eichberger M, Kappert HF, Gernet W, Edelhoff D. High-strength CAD/CAM-fabricated veneering material sintered to zirconia copings — A new fabrication

mode for all-ceramic restorations. Dent Mater 2009;25:121–8. https://doi.org/https://doi.org/10.1016/j.dental.2008.04.019.

[11] Beuer F, Stimmelmayr M, Gueth J-F, Edelhoff D, Naumann M. In vitro performance of full-contour zirconia single crowns. Dent Mater 2012;28:449–56. https://doi.org/https://doi.org/10.1016/j.dental.2011.11.024.

[12] Ioannidis A, Bindl A. Clinical prospective evaluation of zirconia-based three-unit posterior fixed dental prostheses: Up-to ten-year results. J Dent 2016;47:80–5. https://doi.org/10.1016/j.jdent.2016.01.014.

[13] Zhang Y. Making yttria-stabilized tetragonal zirconia translucent. Dent Mater 2014;30:1195–203. https://doi.org/10.1016/j.dental.2014.08.375.

[14] Kim M-J, Ahn J-S, Kim J-H, Kim H-Y, Kim W-C. Effects of the sintering conditions of dental zirconia ceramics on the grain size and translucency. J Adv Prosthodont 2013;5:161–6. https://doi.org/10.4047/jap.2013.5.2.161.

[15] Heffernan MJ, Aquilino SA, Diaz-Arnold AM, Haselton DR, Stanford CM, Vargas MA. Relative translucency of six all-ceramic systems. Part I: Core materials. J Prosthet Dent 2002;88:4–9. https://doi.org/10.1067/mpr.2002.126794.

[16] Chevalier J, Gremillard L, Deville S. Low-Temperature Degradation of Zirconia and Implications for Biomedical Implants. Annu Rev Mater Res 2007;37:1–32. https://doi.org/10.1146/annurev.matsci.37.052506.084250.

[17] Lazar DRR, Bottino MC, Özcan M, Valandro LF, Amaral R, Ussui V, et al. Y-TZP ceramic processing from coprecipitated powders: A comparative study with three commercial dental ceramics. Dent Mater 2008;24:1676–85. https://doi.org/https://doi.org/10.1016/j.dental.2008.04.002.

[18] Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. Biomaterials 1999;20:1–25. https://doi.org/https://doi.org/10.1016/S0142-9612(98)00010-6.

[19] GARVIE RC, NICHOLSON PS. Structure and Thermomechanical Properties of Partially Stabilized Zirconia in the CaO-ZrO2 System. J Am Ceram Soc 1972;55:152–7. https://doi.org/10.1111/j.1151-2916.1972.tb11241.x.

[20] Chevalier J. What future for zirconia as a biomaterial? Biomaterials 2006;27:535–43. https://doi.org/https://doi.org/10.1016/j.biomaterials.2005.07.034.

[21] Deville S, Chevalier J, Gremillard L. Influence of surface finish and residual stresses on the ageing sensitivity of biomedical grade zirconia. Biomaterials 2006;27:2186–92. https://doi.org/https://doi.org/10.1016/j.biomaterials.2005.11.021.

[22] Chevalier J, Gremillard L, Virkar A V, Clarke DR. The Tetragonal-Monoclinic Transformation in Zirconia: Lessons Learned and Future Trends. J Am Ceram Soc 2009;92:1901–20. https://doi.org/10.1111/j.1551-2916.2009.03278.x.
[23] Yoshimura M, Noma T, Kawabata K, S\=omiya S. Role of H2O on the degradation process of Y-TZP. In: S\=omiya S, editor. Hydrothermal React. Mater. Sci. Eng. An Overv. Res. Japan, Dordrecht: Springer Netherlands; 1989, p. 396–8. https://doi.org/10.1007/978-94-009-0743-0_67.
[24] Kim J-W, Covel NS, Guess PC, Rekow ED, Zhang Y. Concerns of hydrothermal degradation in CAD/CAM zirconia. J Dent Res 2010;89:91–5. https://doi.org/10.1177/0022034509354193.
[25] De Souza GM, Zykus A, Ghahnavyeh RR, Lawrence SK, Bahr DF. Effect of accelerated aging on dental zirconia-based materials. J Mech Behav Biomed Mater 2017;65:256–63. https://doi.org/10.1016/j.jmbbm.2016.08.023.
[26] Pinto PA, Colas G, Filleter T, De Souza GM. Surface and mechanical characterization of dental Yttria-Stabilized Tetragonal Zirconia Polycrystals (3Y-TZP) after different aging processes. Microsc Microanal 2016;22:1179–88. https://doi.org/10.1017/S1431927616011843.
[27] Schley J-S, Heussen N, Reich S, Fischer J, Haselhuhn K, Wolfart S. Survival probability of zirconia-based fixed dental prostheses up to 5 yr: a systematic review of the literature. Eur J Oral Sci 2010;118:443–50. https://doi.org/10.1111/j.1600-0722.2010.00767.x.
[28] Sola-Ruiz MF, Agustin-Panadero R, Fons-Font A, Labaig-Rueda C. A prospective evaluation of zirconia anterior partial fixed dental prostheses: Clinical results after seven years. J Prosthet Dent 2015;113:578–84. https://doi.org/10.1016/j.prosdent.2014.12.015.
[29] Kim MJ, Kim YK, Kim KH, Kwon TY. Shear bond strengths of various luting cements to zirconia ceramic: Surface chemical aspects. J Dent 2011;39:795–803. https://doi.org/10.1016/j.jdent.2011.08.012.
[30] Ozcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. J Adhes Dent 2015;17:7–26. https://doi.org/10.3290/j.jad.a33525.
[31] Derand T, Molin M, Kvam K. Bond strength of composite luting cement to zirconia ceramic surfaces. Dent Mater 2005;21:1158–62. https://doi.org/10.1016/j.dental.2005.02.005.
[32] Cavalcanti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, Marchi GM. Bond strength of resin cements to a zirconia ceramic with different surface treatments. Oper Dent 2009;34:280–7. https://doi.org/10.2341/08-80.

[33] Bottino MA, Bergoli C, Lima EG, Marocho SMS, Souza RO, Valandro LF. Bonding of Y-TZP to dentin: effects of Y-TZP surface conditioning, resin cement type, and aging. Oper Dent 2014;39:291–300. https://doi.org/10.2341/12-235-L.

[34] May LG, Salvia ACR, Souza ROA, Michida SMA, Valera MC, Takahashi FE, et al. Effect of sodium ascorbate and the time lapse before cementation after internal bleaching on bond strength between dentin and ceramic. J Prosthodont Off J Am Coll Prosthodont 2010;19:374–80. https://doi.org/10.1111/j.1532-849X.2010.00576.x.

[35] Leskelä M, Ritala M. Atomic layer deposition (ALD): From precursors to thin film structures. Thin Solid Films 2002;409:138–46. https://doi.org/10.1016/S0040-6090(02)00117-7.

[36] Hatton B, Kitaev V, Perovic D, Ozin G, Aizenberg J. Low-temperature synthesis of nanoscale silica multilayers - Atomic layer deposition in a test tube. J Mater Chem 2010;20:6009–13. https://doi.org/10.1039/c0jm00696c.

[37] Pereira GKR, Venturini AB, Silvestri T, Dapieve KS, Montagner AF, Soares FZM, et al. Low-temperature degradation of Y-TZP ceramics: A systematic review and meta-analysis 2016;55:151–63.

[38] Pereira G, Amaral M, Cesar PF, Bottino MC, Kleverlaan CJ, Valandro LF. Effect of low-temperature aging on the mechanical behavior of ground Y-TZP. J Mech Behav Biomed Mater 2015;45:183–92. https://doi.org/10.1016/j.jmbbm.2014.12.009.

[39] Pinto PA, Colas G, Filleter T, De Souza GM. Surface and Mechanical Characterization of Dental Yttria-Stabilized Tetragonal Zirconia Polycrystals (3Y-TZP) After Different Aging Processes. Microsc Microanal 2016;22:1179–88. https://doi.org/10.1017/S1431927616011843.

[40] Bishal AK, Butt A, Selvaraj SK, Joshi B, Patel SB, Huang S, et al. Atomic Layer Deposition in Bio-Nanotechnology: A Brief Overview. Crit Rev Biomed Eng 2015;43:255–76. https://doi.org/10.1615/CritRevBiomedEng.2016016456.

[41] de Jager N, Feilzer AJ, Davidson CL. The influence of surface roughness on porcelain strength. Dent Mater 2000;16:381–8. https://doi.org/https://doi.org/10.1016/S0109-5641(00)00030-0.

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JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS

AUTHOR INFORMATION PACK

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