Original Article

Influence of zirconia surface treatments of a bilayer restorative assembly on the fatigue performance

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Abstract

Purpose: This study evaluated the influence of different surface treatments of zirconia used to enhance bonding with veneering porcelain, and thermocycling on the resistance to porcelain cracking and delamination during fatigue test.

Methods: Bilayer ceramic discs were made from zirconia blocks (IPS e.max Zircad MO, Ivoclar Vivadent – 0.7 mm thickness) and randomized into 8 groups (n=15) according to two factors: ‘zirconia surface treatment’ (Control; Grinding – diamond bur; Air-abrasion – aluminum oxide particles; and Liner – application of a ceramic liner [IPS e.max Zirliner, Ivoclar Vivadent]); and ‘thermocycling’ (presence – 12,000 thermal cycles; 5-55ºC; or absence). The discs were veneered with porcelain (IPS e.max Ceram, Ivoclar Vivadent – 0.7 mm; totaling 1.4 mm thickness) according to ISO 6872:2015 for biaxial flexure strength testing. Fatigue tests (step-stress approach; 20 to 100 MPa; step of 10 MPa; 10,000 cycles per step; 10 Hz frequency) were run, followed by the data analysis (Kaplan-Meier and Mantel-Cox post-hoc tests). Analysis of roughness, topography, crystallographic phase arranges and fractography were also executed.

Results: The surface treatment and thermocycling did not influence the porcelain crack nor delamination resistance. When only comparing the surface treatments for crack resistance outcome, the liner application depicted the worst fatigue performance in comparison to grinding and air-abrasion, while all groups were similar for delamination.

Conclusions: Neither the surface treatment of the zirconia nor the thermocycling influences the porcelain crack resistance or the resistance to delamination of the bilayer porcelain-veneered zirconia specimens.

Keywords: Zirconia-based restoration, Yttria-stabilized tetragonal zirconia polycrystal, Surface modification, Air-abrasion, Ceramic liner, Cyclic loading, Veneer crack, Delamination

1. Introduction

The literature nowadays supports that the use of bilayer restorations with a strong core material covered by an esthetic veneering porcelain is still the best approach to restore teeth which present unfavorable substrate color shades, such as discolored teeth or metal base cores, among others [1,2]. In such a scenario, the use of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) as the infrastructure veneered with a porcelain is commonly employed.

However, several studies [3-5] have reported high failure rates using all-ceramic bilayer systems (13% in 3 years [6]; 21% in 5 years [7]). Among the failure types, delamination and chipping are the most frequent [8-10]. Delamination consists of a fracture which involves the interface between Y-TZP and veneer, while chipping consists in the cohesive failure of the porcelain [3]. The reason for both delamination and chipping occurring has been linked to several factors, such as: repeated occlusal loads during chewing, low thermal diffusivity of the zirconia, residual stress generated by the difference in the thermal expansion coefficient between the infrastructure and the veneering material, as well as the difficulty to enhance bond strength between these materials (zirconia and veneering ceramic) [8,11,12].

According to He et al. [13], some approaches have been applied to enhance the bond strength between the infrastructure and the veneering porcelain. Previous studies evaluated zirconia surface treatments as the particle air-abrasion and grinding with diamond burs that, for instance, may increase the surface roughness, which could generate greater micromechanical interlocking between the core and veneering ceramic.
In addition to the influence on bonding, it is important to consider that these treatments may also lead to a t-m phase transformation, which results in a volumetric expansion of ~4% around the superficial defects, inducing a compressive stress concentration and hindering crack propagation, while also introducing defects in the material’s surface [15-18]. The mechanical properties may be impaired and the risk of failure increased if the treatments are not completely constrained by the transformation mechanism [19]. Thus, an alternative to mechanical surface treatment is to use a liner on the interface between veneering and zirconia, which may enhance the bond strength [20], but it is unknown if it influences the assembly’s mechanical strength. Furthermore, it must be considered that the literature suggests chemical and mechanical approaches to achieve optimal bonding and consequently better mechanical performance [13,14,21]. Therefore, studies considering such scenarios are still encouraged.

It is also important to emphasize that predicting the performance of multilayer ceramic assemblies is a very complex and difficult task, and the interaction between the different materials usually affects the mechanical behavior during the assay [22]. Thus, understanding the adhesion mechanism between both ceramics and its influence over the materials’ strength are not yet fully elucidated and remains contradictory. It is also essential to evaluate the performance of all-ceramic restorations under cyclic loading which occurs daily in the oral environment to better understand the influence of the zirconia surface treatments on the longevity of these restorations [23,24].

Therefore, based on the lack of consensus regarding the best surface treatment to enhance the mechanical behavior of porcelain veneered zirconia and decrease the susceptibility of the bilayer restorative assembly to crack initiation and delamination, the present study aimed to evaluate the influence of four surface treatments (control, grinding, air-abrasion and application of a liner) and aging by thermocycling on fatigue strength, number of cycles for failure and survival rates.

The assumed null hypotheses were that surface treatments (1) and thermocycling (2) will not statistically influence the results of fatigue testing, on fatigue strength, number of cycles for failure and survival rates. The assumed null hypotheses were that surface treatments (1) and thermocycling (2) will not statistically influence the results of fatigue performance for porcelain cracking and delamination occurrence.

2. Materials and methods

2.1. Specimens preparation

The materials used in the study are described in Table 1. Zirconia discs of IPS e.max Zircad MO (Lot W87787; Ivoclar Vivadent, Schaan, Liechtenstein) and yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) were fabricated according to the ISO 6872:2015 [25]. Pre-sintered zirconia blocks were shaped into cylinders by grinding with #60, #200, #400 and #600-grit silica carbide papers (SiC) (3M, Sumare, Sao Paulo, Brazil) in a polishing machine (Ecomet 250 Grinder Polisher, Buehler, Lake Bluff, IL, USA) with constant water irrigation. Next, slices with 18 mm diameter × 1 mm thickness were obtained by using a precision cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA). Then, the discs were ground with #600 and #1200 granulation SiC sandpapers on both sides in order to ensure thickness and surface smoothness standardization, and subsequently cleaned in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ind. And Com. Equip. Odonto. LTDA, Ribeirao Preto, Sao Paulo, Brazil) containing 78% isopropyl alcohol for 5 min.

Finally, the zirconia discs were sintered (VITA furnace Zyrcomat 6000 MS, VITA Zahnfabrik, Bad Sackingen, Germany) according to the manufacturer’s recommendations (Table 1). Thus, disc specimens of 15 mm diameter × 0.7 mm thickness were obtained at the end of the firing cycles. The final dimension was guaranteed using a digital micrometer (Mitutoyo absolute 500-196-20 Digital Caliper; Takatsu-Ku, Kawasaki, Kanagawa, Japan), for which a variation up to 0.03 mm was considered acceptable, while discs with higher measurements were discarded and replaced. Next, all 120 specimens were randomly assigned into 8 groups (n = 15) according to two factors: surface treatment of Y-TZP in 4 levels: control, grinding, air-abrasion, and liner application; and ‘thermocycling’ in 2 levels: presence or absence of thermocycling, as described in Table 2.

2.2. Y-TZP Surface treatments

2.2.1. Control group

The specimens were cleaned with 78% isopropyl alcohol in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras) for 5 min prior to the veneering porcelain application.

2.2.2. Grinding group

A single operator was initially calibrated by means of a pilot test, followed by roughness analysis until a reproducible grinding process was achieved. The grinding was only executed on one side of each specimen using a diamond bur (4219F - 46 μm grain size, KG Sorensen, Cotia, Sao Paulo, Brazil) with a contra-angle coupled to a corresponding low speed motor (Kavo Dental, Biberach, Germany) under constant irrigation (~ 30 mL/min) and with oscillatory movements [26]. One bur was used for each two discs, and the diamond tip was discarded afterwards and replaced with a new one (1 bur/2 specimens). The specimens were previously marked with a permanent marking pen (Pilot, Sao Paulo, Sao Paulo, Brazil) and fixed to a device with a double-sided tape (3M from Brazil, Sumare, Sao Paulo, Brazil) which assured parallelism between the specimen and diamond bur for grinding thickness standardization and to ensure that the entire specimen surface was subjected to grinding, and then grinding procedure was performed until the pen marking was completely eliminated.

2.2.3. Air-abrasion group

The specimens were submitted to air-abrasion using a handheld microblaster (Dento Prep, Ronvig, Daugaard, Denmark) with aluminum oxide particles (Al2O3; 45 μm particles size, Polorid) under oscillatory movements at 2.8 bar pressure and 10mm distance from the device for 10 s [27]. All the specimens were then gentle air-dried to remove any debris.

2.2.4. Liner application group

The IPS e.max Zirller (Lot VI1933; Ivoclar Vivadent, Schaan, Liechtenstein) was manipulated by mixing powder and respective build liquid (Lot W84644) in standardized proportions (1:1) until a creamy consistency mass was achieved according to the manufacturer’s recommendations. The material was subsequently applied with a brush on the zirconia surface until it was homogeneously distributed along the surface. After the application, the specimens were fired in a specific furnace (Vacumat 6000 MP, VITA Zahnfabrik, Bad Sackingen, Germany), following the manufacturer’s instructions (Table 1).

2.3. Roughness and topography analysis

Specimens were cleaned with 78% isopropyl alcohol in ultrasonic bath for 5 min (1440 D, 50/60 Hz, Odontobras) after each treatment. Then, a micrometric analysis was performed with a rugosimeter (n = 15, Mitutuyo SJ-410, Mitutuyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan), and three measurements for each specimen were carried out on three different points considering Ra and Rz parameters (cut-off of 5; λC of 0.8 mm; λS of 2.5 μm), according to ISO 4287:1997 [28]. Ra is the arithmetical mean of the absolute values of peaks and valleys measured from a mean plane (in μm), and Rz is the average distance between the five highest peaks and five major valleys of a surface (in μm).

After the roughness analysis, representative specimens of the 4 surface treatments were cleaned with 78% isopropyl alcohol in an ultrasonic bath for 5 min (1440 D, 50/60 Hz, Odontobras), coated by a nanofilm of gold and inspected by scanning electron microscope.
Table 1. Description of the materials used in the study, their composition and firing cycle.

| Material (manufacturer) | Composition | Firing cycle |
|-------------------------|-------------|--------------|
| IPS e. max Zircad MC (IVOCLAR) | ZrO2, 3 mol% Y2O3, HO3, Al2O3, SiO2, Na2O | Temperature increases 5°C/min until 700°C (±130 min); remains at 700°C for 5 min; temperature increases 5°C/min until 1,000°C (±60 min); remains at 1,000°C for 5 min; temperature increases 5°C/min until 1,600°C (±120 min); remains at 1,600°C for 5 min; cooling until 1,000; 500 and 200°C. |
| IPS e. max Ceram (A2) (IVOCLAR) | SiO2, Al2O3, ZrO2, Na2O, K2O, ZrO2, CaO, P2O5, fluoride and pigments | Pre-drying 403°C for 4 min; temperature increases 40°C/min (±9 min); remains at 750°C for 1 min; cooling until 200°C; vacuum for ±7 min. |
| IPS e. max Zirliner (IVOCLAR) | SiO2, Al2O3, ZrO2, Na2O, K2O, ZrO2, CaO, P2O5, fluoride and pigments | Pre-drying 403°C for 4 min; temperature increases 40°C/min (±14 min); remains at 960°C for 1 min; cooling until 200°C; vacuum for ±14 min. |

Table 2. Study design.

| Group Code | Surface Treatment | Thermocycling | Analysis |
|------------|------------------|---------------|----------|
| Control    | Just cleaned with isopropyl alcohol | Without 5-55°C, 12,000 cycles | Roughness analysis (n=15) |
| Control Aged | Grinding with 4219F diamond bur | With | Fatigue test for crack and delamination (n=15) |
| Grinding   | Blasting with 45 μm aluminum oxide particles for 10s | Without | Survival rate (n=15) |
| Air-abrasion Aged | Treated with IPS e. max Zirliner (Ivoclar Vivadent) | With | X-Ray Diffractometry (n=1) |
| Liner      | With | | |
| Liner Aged | With | | |

(SEM - Vega3, Tescan, Brno, Czech Republic) at 500× and 10,000× of magnification, under a high vacuum with 20.00 kV at a working distance of approximately 13.5 mm for topographic analysis.

2.4. Application of porcelain veneer

The zirconia specimens were cleaned after all surface treatments (as previously described), and positioned inside metal templates (18 mm internal diameter and 2 mm of thickness) which guided the thickness of the porcelain layer application. The ceramic IPS e.max Ceram (Lot W42786; Ivoclar Vivadent, Schaan, Liechtenstein) was manipulated by mixing ceramic powder and build liquid in standard proportions (1:1) according to the manufacturer’s instructions (Lot W44495; Ivoclar Vivadent, Schaan, Liechtenstein) until a slurry solution was obtained. Then, the porcelain was condensed over and around the surface treated zirconia with the aid of a metal spatula. The veneer was applied around the zirconia to compensate the porcelain contraction during the firing cycle and to avoid the discovery of the infrastructure. For each ceramic layer applied inside the template, the excess liquid was gently removed with smooth absorbent paper (Softy’s, Elite, Brazil) with the aid of a flat metal of the same diameter as the internal space of the template. The bilayer specimens were later removed from the template and put on the refractory to be fired in a specific furnace (Vacumat 6000MP; VITA, Zahnfabrik), following the manufacturer’s instructions (Table 1).

After firing, the porcelain layer was polished with SiC sandpapers (#200, #400, #600 and #1200 grit-size) under constant irrigation in a polishing machine (Ecomet 250 Grinder Polisher, Buehler) to achieve a smooth and shining surface. The set thickness was constantly inspected to respect the final dimension of the bilayer set as 1.4 mm (Y-TZP = 0.7 mm in thickness + veneer layer = 0.7 mm), as recommended by the manufacturer. This dimension is also in accordance with ISO 6872:2015 guidelines for biaxial flexure strength testing [25]. Prior to the fatigue test, specimens were analyzed under optical microscopy (Stereo Discovery V20; Carl Zeiss, Göttingen, Germany) and all specimens showing bubbles or irregularities were discarded and replaced.

2.5. Thermocycling

Half of the bilayer specimens were aged in a thermocycler machine (Model 521-6D, Ethik Technology, Nova Etica, Sao Paulo, Brazil) for 12,000 thermal cycles, with temperature ranging from 5 to 55 ºC, with a dwell time of 30 s in each bath, and 2 s of transfer time between baths.

2.6. Fatigue test (piston-on-three-balls biaxial flexure assembly)

The specimens were submitted to a biaxial fatigue strength test, according to ISO 6872:2015 guidelines [25], in an electrodynamic machine (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, United States) by the application of cyclic loading. Each bilayer set was positioned in the device on a metal base composed of three equidistant balls. An adhesive tape was glued on the compression side of the specimen prior to starting the test for better piston contact and tension distribution [29]. This also prevents the spreading of fragments at the moment of fracture which would difficult fractography analysis [30]. In addition, a piece of cellophane paper (2.5 μm) was positioned on the tensile side between the specimen and the three supporting balls to improve the contact pressure [25]. The observance of such aspects is important to avoid Hertzian cone cracks [31]. The set was submerged in distilled water with the porcelain side facing down (tensile tension force), which means steps of 30 MPa, 40 MPa, 50 MPa and so on, up to 100 MPa. The specimens were tested until the first crack and then until the delamination of the porcelain or until 85,000 cycles were reached.
completed, and the step and number of cycles for both outcomes were registered. Cracks were searched by transillumination following each step of the test [23]. The number of cycles and strength data of the test was collected for both cracking and delamination.

2.7. Failure analysis

Failed specimens were analyzed in a stereomicroscope (Stereo Discovery V20, Carl Zeiss, Gottingen, Germany) to identify the failure characteristics and to select representative specimens to be analyzed in the SEM. Representative specimens selected for the scanning electron microscope (SEM - Vega3, Tescan, Brno, Czech Republic) were previously metallized and then analyzed in 32, 100 and 200x of magnification under high vacuum with 20.00 kV at a working distance of approximately 13.5 mm, to detect the crack origin and the direction of crack propagation.

2.8. Phase transformation analysis (X-Ray Diffractometry - XRD)

Additional Y-TZP zirconia discs without veneering were fabricated and submitted to the four surface treatments considered herein as previously described, and taken to the furnace for simulation of porcelain firing (without porcelain). Then, a quantitative descriptive analysis of phase transformation using an X-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) was performed (n= 1) to determine the relative amount of monoclinic phase content generated by each surface treatment and any potential alteration of such composition triggered by porcelain fire cycle. The porcelain veneer was not applied due to the possibility of interference in the phase transformation measurement, since the test is restricted to a superficial or near surface region, typically no more than the top few microns [33,34].

Spectra were collected in the 2θ range of 27 to 37 and 72 to 76 degrees (where θ is the angle of incidence relative to the sample surface), with tension of 40 kV and 40 mA at a step interval of 2 s, and step size of 0.01 degrees/step. The m-phase fraction (XM) was calculated using the Garvie & Nicholson [35] method:

\[
Xm= \frac{(-111)M+(111)M+(101)T}{(-111)M+(111)M+(101)T} \quad \text{Eq. (1)}
\]

where \((-111)\text{M}\) and \((111)\text{M}\) represent the intensity of the monoclinic peaks (2θ = 28° and 2θ = 31.2°, respectively) and \((101)\text{T}\) indicates the intensity of the respective tetragonal peak (2θ = 30°). The volumetric fraction of the m-phase was calculated according to Toraya et al. [36]:

\[
Fm= \frac{1.311Xm}{(1+0.311Xm)} \quad \text{Eq. (2)}
\]

2.9. Statistical analysis

A statistical software program was used to evaluate the roughness (Ra and Rz parameters) and the fatigue data (SPSS version 21, IBM, Chicago, IL, USA) using α= 0.05. As the roughness data assumed a non-parametric distribution (Shapiro-Wilk test), the data was submitted to the Kruskal-Wallis and Dunn’s post-hoc tests. The fatigue data were analyzed by the Kaplan-Meier and Mantel-Cox post-hoc tests, considering both the crack initiation and the delamination as failure outcomes. Moreover, the survival probabilities on each step-stress were calculated, considering the both outcomes (crack initiation and the delamination).

3. Results

3.1. Roughness and surface topography

The roughness analysis showed that control and air-abrasion groups were statistically similar depicting the smoother surfaces, while the grinding and liner groups were statistically similar to each other and showed rougher surfaces (Table 3). Regarding surface topography, the SEM analysis showed completely different surface patterns. Moreover, the control group clearly depicts the presence of the zirconia grains, and all surface treatments alter this surface pattern. Despite having a similar roughness to the control condition, the air-abrasion group depicts a completely deformed surface with the presence of a more irregular surface introduced by the impact of the abrading particle. A deformed surface is also observed after grinding, but with scratches being introduced in relation to the movement of the grinding tool. Liner application clearly leads to the presence of inconsistent layers of material as it seems that the liner concentrates in some areas and depletes in others, creating an aspect of the presence of pores and high surface irregularity (Fig. 1).

3.2. Fatigue results

The survival analysis (Kaplan-Meier and Mantel-Cox post-hoc tests) pointed out that both factors considered in this study (surface treatment and aging by thermocycling) did not statistically influence the strength and number of cycles for cracking or for delamination of bilayer zirconia veneered specimens, since all conditions were statistically similar to the control group (Tables 3-5). However, when only comparing the surface treatments, at the no-aged baseline condition, it notes that the liner application had a worse performance than grinding and air-abrasion for the crack occurrence outcome (Table 3). Besides, the survival analysis showed some slight differences that can be noticed in some steps. For instance, at 50 MPa / 35,000 cycles for the crack initiation outcome (Table 4; Fig. 2), the liner group presented 20% probability of survival, while the grinding group 73% and the air-abrasion group presented 47%. Even so, all treatments were similar to the control group for crack occurrence outcome. For delamination outcome, all groups were statistically similar (Tables 3 and 5; Fig. 2).

3.3. Fractography analysis

The fractography analysis evidenced that the failure originated from surface or sub-surface defects at the outer surface of the porcelain layer (tensile side during fatigue test) for all tested conditions. After initiation, the crack propagated towards the zirconia core until delamination occurred (Fig. 3).

3.4. XRD analysis

The XRD analysis showed the presence of monoclinic phase in the grinding (7.74%) and air-abrasion (8.91%) groups. No m-phase was observed for the control or liner groups (Table 3).

4. Discussion

These current findings showed no influence of different surface treatments or thermocycling on fatigue strength, number of cycles for failure, or survival rates for crack and delamination occurrence of the porcelain-veneered zirconia when the surface treated groups were compared with the control group; thus, the null hypotheses were accepted. Even with the differences in topography, roughness and the presence of monoclinic phase between groups, no surface treatment was superior to the absence of treatment (control group) when considering the crack outcome (Fig. 1, Table 3). It has been reported that the bilayer ceramic
Table 3. Fatigue results (strength in MPa and number of cycles for failure for crack and for delamination occurrence, mean and 95% confidence intervals), roughness results (μm, mean and Standard Deviation), and content of monoclinic phase (%).

| Groups        | Crack occurrence* | Delamination occurrence* | Roughness analysis** | XRD analysis |
|---------------|-------------------|--------------------------|----------------------|--------------|
|               | Strength Mean (95%CI) | Cycles Mean (95%CI) | Strength Mean (95%CI) | Cycles Mean (95%CI) | Ra       | Rz       | m-phase (%) |
| Control       | 53.33 (48.78 – 57.89) | 38.33 (33.78 – 42.88) | 74.00 (70.27 – 77.73) | 57.345 (53.852 – 60.838) | 0.26 (0.06) | 2.37 (0.79) | 0          |
| Control Aged  | 52.67 (48.19 – 57.14) | 37.66 (33.194 – 42.138) | 75.33 (72.10 – 78.57) | 59.676 (56.441 – 62.911) | 0.27 (1.12) | 2.20 (0.49) | -          |
| Grinding      | 58.67 (53.30 – 64.03) | 42.48 (37.907 – 47.061) | 74.67 (70.03 – 79.30) | 58.369 (53.098 – 63.641) | 1.22 (0.26) | 7.03 (1.72) | 7.74        |
| Grinding Aged | 54.00 (50.80 – 57.20) | 39.00 (35.799 – 42.209) | 74.00 (69.39 – 78.61) | 59.000 (54.393 – 63.606) | 1.26 (1.14) | 7.00 (1.07) | -          |
| Air-abrasion   | 54.67 (50.91 – 58.43) | 39.023 (35.809 – 42.233) | 71.00 (68.58 – 75.42) | 55.769 (51.796 – 59.741) | 0.27 (0.04) | 2.14 (0.39) | 8.91        |
| Air-abrasion Aged | 56.67 (51.73 – 61.61) | 40.443 (35.826 – 45.061) | 72.67 (67.80 – 77.53) | 57.139 (52.090 – 62.189) | 0.30 (0.03) | 2.27 (0.22) | -          |
| Liner         | 48.00 (44.08 – 51.92) | 32.344 (28.306 – 36.381) | 71.33 (66.70 – 75.97) | 55.773 (51.300 – 60.246) | 1.58 (1.85) | 7.61 (2.07) | 0          |
| Liner Aged    | 52.67 (50.35 – 54.98) | 37.666 (35.350 – 39.983) | 73.33 (69.20 – 77.47) | 57.036 (53.602 – 60.470) | 1.23 (0.38) | 7.09 (1.08) | -          |

- Distinct letters show significant statistical differences (p < 0.05) depicted by *Kaplan-Meier and Mantel-Cox (Log-rank) tests and by **Kruskal-Wallis and Dunn’s post-hoc tests.

Fig. 1. Scanning electron microscopy (SEM) of the zirconia surface after the 4 treatments, analyzed at 500 and 10,000× of magnification. Grinding, Air-abrasion and Liner application treatments created a more irregular topography compared to the control group. Also, the liner group showed a presence of some porosities after firing.

Behavior under flexural strength test depends on the characteristics of the material positioned as the bottom surface, so if the porcelain takes place under tensile stress, the set strength could be even similar to a monolithic veneering ceramic [22,37]. Thus, the ultimate strength and failure mode is mainly dictated by the material properties on the bottom, which in this case is the porcelain, and the contribution of the Y-TZP core results in just a modest increase in the strength [38].

Regarding the delamination outcome, all Y-TZP surface treatments groups (with or without thermocycling) were statistically similar. The chipping and delamination of veneering ceramics are some of the most frequent reasons for the failure of metal-free restorations [8-10]. The literature points out that different zirconia surface treatments lead to different bond strengths and chipping/delamination occurrence on veneering ceramics [9,14,39]. However, the findings of the present study show that such bonding provided by the grinding, air-abrasion and liner application surface treatments were not sufficient to generate better fatigue performance of the bilayer ceramic system than the control group, and even the survival rate for all the groups were similar (Table 5). For better performance regarding cracking and delamination resistance under repeated load cycles, which is the most frequent mechanical stimulus in a clinical scenario [23,24,40], the adhesion...
Table 4. Survival rates, i.e. specimens' probability to exceed the respective cycles and fatigue strength for crack occurrence, and their respective standard error measurements.

| Groups         | Number of cycles / Fatigue strength (MPa) for crack occurrence |
|----------------|---------------------------------------------------------------|
|                | 5,000 / 20 15,000 / 30 25,000 / 40 35,000 / 50 45,000 / 60 55,000 / 70 65,000 / 80 75,000 / 90 |
| Control        | 1 1 0.80 (0.10) 0.47 (0.13) 0.07 (0.06) 0 - - |
| Control Aged   | 1 1 0.80 (0.10) 0.40 (0.13) 0.07 (0.06) 0 - - |
| Grinding       | 1 1 0.87 (0.09) 0.73 (0.11) 0.20 (0.10) 0.13 (0.09) 0 - |
| Grinding Aged  | 1 1 0.95 (0.06) 0.47 (0.13) 0 - - - |
| Air-abrasion    | 1 1 0.93 (0.06) 0.47 (0.13) 0.07 (0.06) 0 - - |
| Air-abrasion Aged | 1 1 0.87 (0.09) 0.60 (0.13) 0.20 (0.10) 0 - - |
| Liner          | 1 1 0.60 (0.13) 0.20 (0.10) 0 - - - |
| Liner Aged     | 1 1 0.27 (0.11) 0 - - - - |

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

Table 5. Survival rates, i.e. the specimens' probability to exceed the respective cycles and fatigue strength for delamination, and their respective standard error measurements.

| Groups         | Number of cycles / Fatigue strength (MPa) for delamination |
|----------------|-----------------------------------------------------------|
|                | 5,000 / 20 15,000 / 30 25,000 / 40 35,000 / 50 45,000 / 60 55,000 / 70 65,000 / 80 75,000 / 90 85,000 / 100 |
| Control        | 1 1 1 1 0.93 (0.06) 0.40 (0.13) 0.07 (0.06) 0 - - |
| Control Aged   | 1 1 1 1 0.87 (0.09) 0.47 (0.13) 0.13 (0.09) 0 - - |
| Grinding       | 1 1 1 1 0.87 (0.09) 0.47 (0.13) 0.13 (0.09) 0 - - |
| Grinding Aged  | 1 1 1 1 0.93 (0.06) 0.33 (0.12) 0.07 (0.06) 0.07 (0.06) 0 - |
| Air-abrasion    | 1 1 1 1 0.87 (0.09) 0.33 (0.12) 0 - - - |
| Air-abrasion Aged | 1 1 1 1 0.80 (0.10) 0.33 (0.12) 0.13 (0.09) 0 - - |
| Liner          | 1 1 1 1 0.93 (0.06) 0.87 (0.09) 0.27 (0.11) 0.07 (0.06) 0 - |
| Liner Aged     | 1 1 1 1 0.87 (0.09) 0.40 (0.13) 0.07 (0.06) 0 - - |

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

Fig. 2. Survival graphs obtained by Kaplan-Meier test for fatigue strength (A and B) and number of cycles for failure (C and D) considering the two outcomes: crack (left) and delamination occurrence (right).
between porcelain and framework layers should perhaps be even greater, since the bonding achieved between the zirconia core and ceramic veneer is still limited and controversial [12,41]. Even so, from a clinical viewpoint, literature shows that maximum masticatory forces may easily achieve 300–400 N with lower average chewing forces of approx. 220 N in the molar region [42,43]. Assigning those forces to a contact area of 7–8 mm² results in an average chewing pressure of 27–31 MPa [44]. Thus, all the groups of the present study achieved strength superior to 30 MPa, which is also in accordance with the ISO 6872:2015 recommendations for use as a veneer of bilayer crowns [25].

Moreover, thermocycling also did not significantly influence the tested outcomes, which could be an indication that it also did not influence the adhesion between layers or the defects present in the bilayer set. This may have occurred due to the aging protocol used in the present study not being sufficiently deleterious to cause thermal expansion of the ceramics, which have different thermal coefficients [11]. Thus, it might be interesting to use other temperatures or the combination of mechanical and thermal stimulus at the same time in future studies.

However, when comparing the surface treatment groups with each other, a slightly worse performance was observed for the liner application in relation to the grinding and air-abrasion groups when considering the porcelain cracking outcome. This can be explained due to the tetragonal to monoclinic phase transformation of the Y-TZP in the grinding (7.74%) and air-abrasion (8.91%) groups showed by the XRD analysis (Table 3). The mechanism of zirconia phase transformation, which occurs in the presence of a mechanical stimulus [15], is responsible for the increase in the material toughness [45] through the volume increase of the monoclinic grains by 3–4%, thereby generating a compression stress concentration that slows/avoids the crack propagation into the zirconia [16].

Guazzato et al. [38] related two stress concentration peaks during flexural testing for bilayer systems, both on the bottom surface of the core and the porcelain when the specimens were tested with the porcelain turning down (tensile side). Thus, the transformation toughening mechanism may have generated a slight decrease in the zirconia’s deflection during the flexural fatigue test, thereby generating lower stress concentration at the bottom surface of the porcelain [46], which did not happen for the liner application group. Furthermore, the surface topography analysis showed that the liner application leads to the presence of pores and high surface irregularities near the interface which could contribute to the mechanism of crack propagation, as the pores are small in dimension and may have had problems to be filled by the porcelain layer (Fig. 1).

It is important to consider that the presence of pores may facilitate crack propagation towards to the interface [37,47,48]. Thus, if a surface treatment, as grinding, generates more homogeneous surfaces with defects that are easier to be filled by the porcelain (Fig. 1), it may enhance the performance of the system by reducing the presence of bubbles during porcelain application. On the other hand, if a surface topography becomes more complex after the treatment, the presence of bubbles and defects is intensified, thus the risk of premature failure may be increased [48].

In addition, there is a lack of consensus in the literature, but the ceramic liner does not seem to enhance the adhesion of the Y-TZP with porcelain. The manufacturers’ instructions show that the use of liner is dispensed for pre-pigmented zirconia infrastructures. Thus, the liner is basically applied to mask the opacity of the infrastructure for esthetic reasons. Also, Fischer et al. [49] reported that the application of a liner to Y-TZP had no significant effect on its shear bond strength with veneering ceramics, while Wang et al. [50] reported that the use of a liner reduced the bond strength of the zirconia-veneer interface, so a liner should be applied with caution.

Regarding the failure pattern, the fractography analysis showed that the crack initiation for all groups was at the bottom surface of the ceramic assembly (porcelain), which was under tensile stress during the flexural fatigue test. The crack towards to the interface was hindered by the tougher core material and propagates within the interface until the delamination of the porcelain veneer (Fig. 3), which is in accordance with previous studies using such a test assembly [22,38].

Despite the relevance of the present study findings, it is important to consider its limitations. Under the conditions explored herein, the strong control and methodological standards induces a reduced presence of internal pores and defects on porcelain layer. Previous studies have reported that the hand-layered technique generates more porosities at vitreous ceramics [37,47], and these defects may decrease the mechanical performance of the material, according to the Griffith’s law [48]. Thus, on a clinical setup if during porcelain application higher bubbles and defects are introduced, the performance of the set may be influenced. In addition, a simply test (biaxial) was used for fatigue behavior assessment herein, which does not reproduce the clinical loading/stressing when a crown receives the masticatory

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**Fig. 3.** Scanning electron microscopies of a representative delaminated specimen selected from the grinding group and analyzed at 32 (left), 100 (middle) and 200× (right) magnification. The crack propagated from the outer surface of the porcelain which was on the bottom/tensile side during the fatigue test. The filled arrows inside the porcelain point to the wake hackles, which show the direction of crack propagation (dcp; dashed arrows).
intermittent loading, such as, when sliding movements on the occlusal inclinations occur. Thus, our finding should be carefully analyzed and new test approaches should be demanded to evaluate the clinical effects of zirconia surface treatments on the fatigue behavior of restorations. Finally, the study of the fatigue behavior of a ceramic bilayer system may be considered still incipient in literature, encouraging new studies.

5. Conclusion

No zirconia surface treatment (grinding, air-abrasion and liner application) was superior to the absence of treatment (control) when the final crack resistance and delamination was considered.

Aging by thermocycling did not influence resistance to cracking or delamination of bilayer zirconia veneered specimens.

Conflict of Interest Statement

The authors deny the presence of any conflict of interest.

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