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Retentive Strength of Y-TZP Crowns: Comparison of Different Silica Coating Methods on the Intaglio Surfaces

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Clinical Relevance

Tribochemical silica coating and 5-nm thick silica nanofilm conditioning methods used on intaglio surfaces of zirconia crowns improves retention.

SUMMARY

Objective: To evaluate the effect of different methods of silica deposition on the intaglio surface of yttrium oxide stabilized zirconia polycrystal (Y-TZP) crowns on the retentive strength of the crowns.

Methods: One hundred simplified full-crown preparations produced from fiber-reinforced polymer material were scanned, and 100 Y-TZP crowns with occlusal retentions were milled. Crown/preparation assemblies were randomly allocated into five groups (n=20) according to the treatment of the intaglio surfaces: TBS = tribochemical silica coating via air-abrasion with 30-µm silica-coated alumina particles; GHF1 = application of thin glaze layer + hydrofluoric acid (HF) etching for 1 minute; GHF5 = glaze application + HF for 5 minutes;

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DOI: 10.2341/16-090-L

GHF15 = glaze application + HF for 15 minutes; NANO = silica nanofilm deposition (5 nm) via magnetron sputtering. All groups received a silane application. The surfaces of the preparations (polymer) were conditioned with 10% HF for 30 seconds and silanized. The crowns were cemented with resin cement, thermocycled (12,000 cycles; 5°C/55°C), stored for 60 days, and subjected to a retentive strength test (0.5 mm/min until failure). The retention data (MPa) were analyzed using one-way analysis of variance, Tukey tests, and Weibull analysis. Failures were classified as 50C (above 50% of cement in the crown) and 50S (above 50% of cement on the substrate).

Results: The TBS (5.6±1.7 MPa) and NANO groups (5.5±1 MPa) had higher retentive strength than the other groups ($p<0.0001$) and had the highest values of characteristic strength. There was no difference in Weibull modulus, except for the GHF1 group (lower values). The TBS and GHF15 groups, respectively, had 60% and 70% of their failures classified as 50C, while most of the other groups had 50S failures.

Conclusion: Tribochemical silica coating and silica nanofilm deposition on the inner surface of zirconia crowns promoted a higher retentive strength.

INTRODUCTION

Yttrium oxide stabilized zirconia polycrystal (Y-TZP) ceramics have been widely used in dentistry owing to their superior mechanical strength (high fracture toughness)[1,2] and biocompatibility.[3] However, due to their physical, chemical, and microstructural features, the adhesion of Y-TZP ceramics with resin cements is difficult.[4-7] Hydrofluoric acid (HF) etching and silane application can increase the bond strength between vitreous/glass ceramics (based on silica) and composite resins by increasing the surface free energy and wettability[8-13]; in addition, the ceramic primer heat treatment can promote bond improvements.[14-16] However, as zirconia ceramics are dense polycrystalline materials and lack a silicon dioxide (silica) phase, HF treatment fails to produce a microporous surface for bonding.[9,17,18] Thus, surface pretreatment is necessary to modify the topography to increase the mechanical retention and chemical adhesion, thereby enhancing the retention of the crowns to the prosthetic preparation.[19,20] Recently, systematic reviews showed that loss of retention is significantly higher for densely sintered zirconia, compared with all others types of ceramics and metal-ceramics, for both single and multiple crowns.[21,22]

The most commonly used treatment method is air abrasion with silicon oxide particles. This method involves the inclusion/incrustation of silica-coated alumina particles on the cementation surface by air abrasion, followed by application of a silane primer bonding agent.[23-27] These particles increase the roughness of the zirconia surface, while the silane bonding agent promotes adhesion between the abraded surface and the resin matrix of the cement.[28-30] However, studies have shown that air abrasion might create superficial defects and cracks that may initiate fractures.[31,32] Nonetheless, other studies did not find any negative effects when this method was used on Y-TZP ceramics.[33,34] Additionally, clinical failures in Y-TZP crowns (chippings) seem to have no association with the roughness created by the air abrasion of the intaglio surface.[35-38]

Another recently developed technique involves the application of a thin layer of vitreous porcelain (low-fusing porcelain glaze, vitrification or glaze-on) on the ceramic surface. Basically, the glaze is composed by vitreous porcelain (high silica content, amorphous matrix or SiO_2) and pigments (metallic oxides), making a zirconia glazed surface etchable by HF,[39,40] and that can be subjected to silanization or air-particle abrasion.[41] Vitrification is very effective in promoting the adhesion between the Y-TZP ceramic surface and resin cements.[39,42-46] For glass ceramics,[47-49] HF reacts with the silica phase of the porcelain, creating retentive microchannels. Therefore, the effect of longer etching times on the creation of surface irregularities for bonding depends on the microstructure of the ceramic.[8,50,51] Etching increases the contact area between the adhesive agent and the ceramic,[52,53] and the number and size of the irregularities created are associated with the duration of the etching process.[47,54] However, when low-fusing porcelain glazes on zirconia surfaces are treated with HF for too long, the acid etching might completely remove the glaze from the surface. The impact of etching time of porcelain glaze applied on zirconia influences resin bonding strength and crown retention rate has yet to be investigated.

Recently, the deposition of silica nanofilms on the zirconia surface has been studied.[26,55] For this method, a silica nanofilm is deposited on the zirconia surface by plasma processing (reactive magnetron sputtering), making it more chemically reactive. Following up the nanofilm deposition with silanization results in an increase in adhesive strength

Table 1: *Testing Groups and Respective Descriptions of the Surface Conditioning Protocols on the Intaglio Surface*

Group[a] (n=20)	Protocols
TBS[a]	*Tribochemical silica coating:* airborne particle abrasion with silica-coated aluminum oxide particles (30 μm) (Cojet Sand – 3M ESPE), 15 mm distant from Y-TZP crown for 10 seconds at a pressure of 2.8 bar. An adapted device[26] was used to standardize the procedure.
GHF1[b]	*Porcelain glaze + hydrofluoric acid (1 minute):* a single thin layer of low-fusing porcelain glaze (Glaze VITA Akzent, VITA Zahnfabrik, Bad Säckingen, Germany) was applied with a brush (equal proportions of powder and liquid low-fusion porcelains for each five crowns). The crowns were submitted to glaze sintering cycle according to the manufacturer. After, the intaglio surface was conditioned with hydrofluoric acid 10% for 1 minute, washed with air-water spray for 1 minute and dried. The crowns were cleaned via ultrasound (5 minutes in distilled water) and dried.
GHF5 [b]	*Porcelain glaze + hydrofluoric acid (5 minutes):* The procedures were the same as for GHF1, but the surfaces were conditioned for 5 minutes.
GHF15 [b]	*Procelain glaze + hydrofluoric acid (15 minutes):* The procedures were the same that of GHF1, but the surfaces were conditioned for 15 minutes.
NANO	*Silica nanofilm deposition:* A 5 nm SiO_2 nanofilm was deposited by magnetron sputtering. Prior to deposition, the atmosphere inside the chamber was pumped down to $\sim 10^{-7}$ Torr base pressure. During deposition, pressure was kept at 5.2 mTorr using a 20 sccm argon flow rate. The nanofilm thickness was determined by the exposure time of the crowns to the deposition plasma, as the deposition rate is known.[55]

[a] *TBS = tribochemical silica coating via air-abrasion with 30-μm silica-coated alumina particles; GHF1 = application of thin glaze layer + hydrofluoric acid (HF) etching for 1 minute; GHF5 = glaze application + HF for 5 minutes; GHF15 = glaze application + HF for 15 minutes; NANO = silica nanofilm deposition (5 nm) via magnetron sputtering.*
[b] *The groups with glaze application were all made at the same time (n=60). As the technique is inherent to difficulty of standardization, the 60 crowns were randomized in the three glaze groups after the glaze application and sinterization.*

without damaging the Y-TZP surface.[26,55,56] Moreover, this technique forms a homogeneous film, improves chemical adhesion to the substratum,[57] and does not promote *m*-phase transformation after the film is applied.[26]

Considering the aforementioned silica deposition methods (low-fusing porcelain glaze application, or silica nanofilm deposition) for improving bonding to zirconia, a question not yet addressed is this: Do these treatments of the intaglio surface of zirconia crowns improve crown retention compared with tribochemical silica coating via air abrasion? Thus, the objectives of this *in vitro* study were: 1) to evaluate the effects of different silica-based coatings of the intaglio surface of zirconia crowns on retentive strength, 2) to compare three different HF etching times for the groups undergoing the vitrification technique, and 3) to evaluate the reliability of the different treatment methods by Weibull modulus. The null hypothesis tested was that there would be no difference among the zirconia surface conditionings.

METHODS AND MATERIALS

Sample-Size Calculation

To determine the number of specimens for group, a sample-size calculation was performed, based on a pilot study, with software from the site "Java applets for power and sample size" (www.stat.uiowa.edu/~rlenth/Power/). With a statistical power of 80%, a standard deviation of the mean of 1.15 MPa, and a detectable difference of 1.38 MPa, it was established that the number of specimens per group should be 20, for a total of 100 specimens divided among five experimental groups (see Table 1).

Thus, the experimental design was based on one factor (zirconia surface treatment) divided into five levels (testing groups, n=20) according to the treatment of the intaglio surface of the Y-TZP crowns (Table 1).

This study was blinded for: crown cementation, aging procedure, retention test, and failure analysis.

Prosthetic Preparation and Crown Production

G10 rods (G10, FR4 Laminate Round Rods Epoxyglass; NEMA grade FR4, Accurate Plastics, Inc, New York, NY, USA) measuring 11 mm in diameter and 1.2 m in length were sectioned in small cylinders of 16 mm height used to obtain 100 identical, simplified, full-crown preparations by computer-aided machining (6 mm in height and a total occlusal convergence angle of 12° with rounded corners; Figure 1a,b).[37,38] Taking into account the fact that all the preparations in G10 had identical dimensions/geometry, just one preparation was impressed using a vinyl polysiloxane impression material (Elite HD + Putty and Light Body Normal Setting, Zhermack, Badia Polesine, Italy), followed by obtaining a model in special plaster (CAM-base, type 4, Dentona AG, Dortmund, Germany).

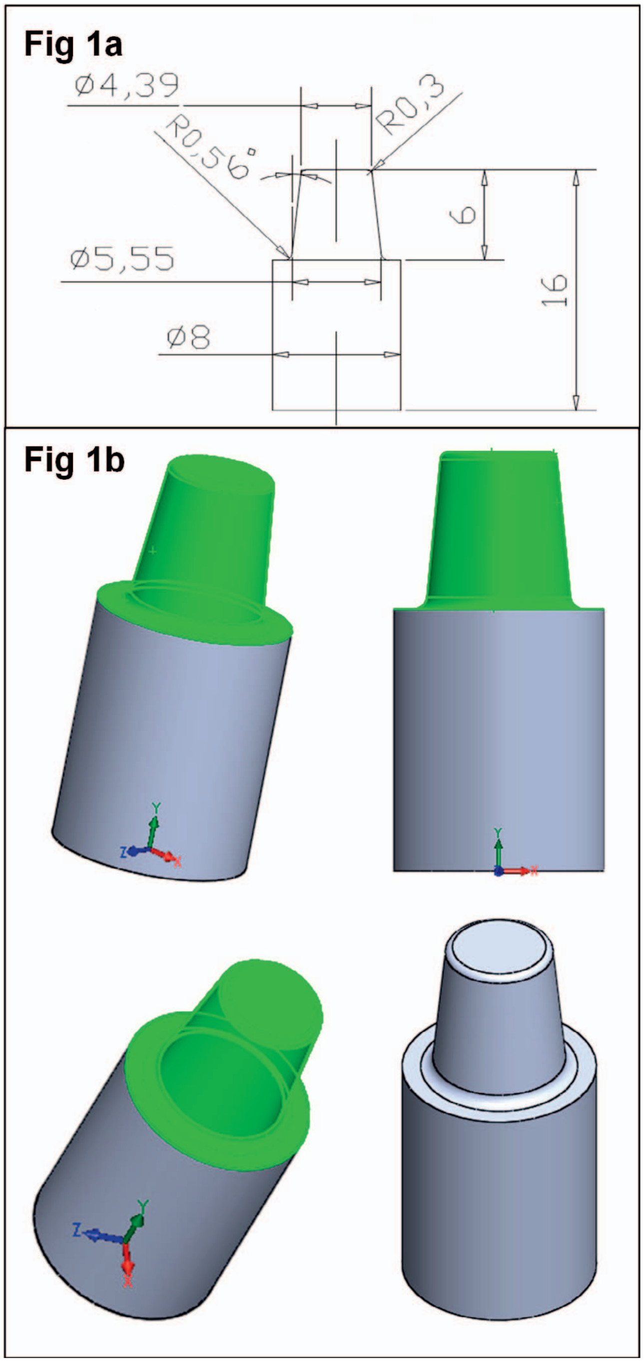

Figure 1. *(a): Schematic drawing of the G10 prosthetic preparations (ø = diameter and R = radius) as a trunked cone. (b): Lateral and frontal views of the G10 preparation in Solid Works software. The green area corresponds to the adhesive area.*

The master die was then scanned (inEos Blue, Sirona, Bensheim, Germany), and the image was transferred to the inLab software (version 3.60, Sirona). Equal crowns (N=100) with occlusal retentions were designed considering a resin cement space of 30 μm, followed by milling the Y-TZP crowns (Cerec InLab MC XL, Sirona; Figure 2a; VITA In-Ceram YZ, YZ-40/19 cubes with dimension of 15.5 × 19 × 40 mm^3, VITA, Bad Säckingen, Germany).

Sintering was performed according to the manufacturer's instructions (Zircomat oven, VITA). The crowns were checked on their preparations for adaptation (Carbono Arti-Spray, Bausch, Bausch Articulating Papers, Inc, Nashua, NH, USA) and cleaned with an ultrasonic device (1440 D – Odontrobras, Ribeirao Preto, Brazil) with distilled water for 10 minutes.

Crown Cementation

After each treatment of the inner surfaces of the Y-TZP crowns (Table 1), methacryloxypropyltrimethoxy-silane (ESPE-Sil Silane, 3M ESPE, Seefeld, Germany) was applied with a microbrush, followed by a 5-minute wait for solvent evaporation.

The preparation surfaces (G10) were conditioned with 10% HF for 30 seconds, cleaned for the same amount of time, and then received an application of silane-based primer (ESPE-Sil, 3M-ESPE), with a 5-minute wait time.[59] The resin cement (RelyX ARC, 3M ESPE) was manipulated according to the manufacturer's instructions and applied to the intaglio surface of the crowns, which were positioned on the preparation. With an adapted surveyor (B2, BioArt, Sao Carlos, Brazil), a load of 750 g was applied to the crowns, the cement excess was removed, and photo activation was performed for 20 seconds on each surface (1200 mW/cm^2, Radii-Cal, SDI, Bayswater, Australia). The specimens were stored in distilled water (37°C) for 24 hours before aging.

Aging: Thermocycling + Storage

All specimens were subjected to thermocycling (12,000 cycles; 5°C to 55°C; 30 seconds per bath and 5 seconds between baths; Ethik Technology, Vargem Grande Paulista, Brazil)[19,20,60,61] followed by storage for 60 days in a wet environment at 37°C.

Retentive Strength Test

After thermocycling, part of the cemented crown was embedded in self-curing acrylic resin (VIPI Flash, VIPI, Pirassununga, Brazil) until total coverage of the retentive part of the crowns was achieved. This process was carried out with the aid of an adapted surveyor (B2, BioArt), which maintained a vertical embedding axis. Retention areas were made apical to the preparations, and the same embedding proce-

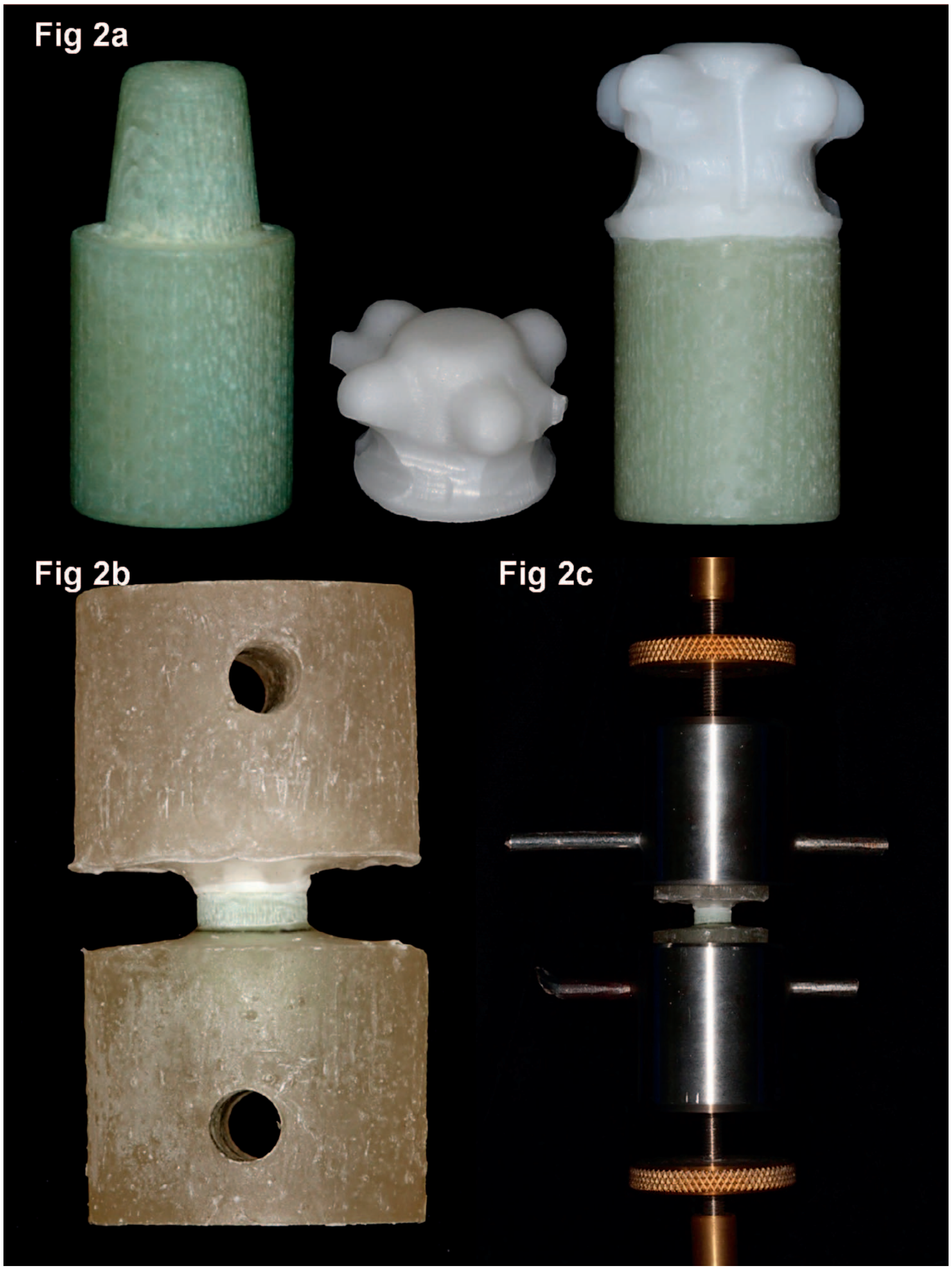

Figure 2. *(a): G10 preparation after milling, zirconia crown with occlusal retention and cemented crown on the G10 preparation. (b): Specimen embedded for the tensile test. (c): Specimen attached on the universal machine test.*

dure was performed, keeping the adhesive interface free to the test. The embedding was necessary to make possible the retention test (Figure 2b).

For the retentive strength test, the superior part of the assembly (the crown) was fixed to the movable axle of a universal testing machine (DL-1000, Emic, São José dos Pinhais, Brazil), which was attached to the load cell (1000 N), while the inferior part (the preparation) was positioned at the fixed base of the testing machine (Figure 2c). The retentive strength test was performed until fracture (decementation) at a speed of 0.5 mm/min.[19,20]

Adhesive Area Calculation

The cementation area was calculated by the SolidWorks software (DS SolidWorks Corporation, Waltham, MA, USA) according to the dimensions presented in Figure 1a, resulting in an adhesive area of 130 mm^2 (Figure 1b). The retentive strength was calculated using the formula: $R = F_{max}/A$, where

Table 2: *One-Way Analysis of Variance Results for Retentive Strength Data*

	Sum of Squares	*df*	Mean Square	F	Significance
Between groups	168.816	4	42.204	30.402	.000
Within groups	131.876	95	1.388		
Total	300.692	99			

Abbreviation: df, degrees of freedom.

R = retentive strength, F_{max} = maximum force for failure (decementation), and A = adhesive area.

Failure Analysis

The tested assemblies were analyzed under a stereomicroscope (Discovery V20, Carl-Zeiss, Gottingen, Germany) to evaluate the type of fracture. The fractures were classified according to the localization of the largest portion of cement: 50C (more than 50% of the cement on the crown), or 50S (more than 50% of the cement on the preparation). Representative images were taken with a scanning electronic microscope (SEM; JSM 5400, Jeol Ltd, Tokyo, Japan). This classification was adapted from Amaral and others[19] and Rippe and others.[20]

Micromorphologic Analysis (Zirconia Blocks and G10 Polymer Surfaces)

Extra zirconia samples (small blocks) were produced from VITA In-Ceram YZ blocks (VITA) in a cutting machine (IsoMet 1000; Buehler, Lake Bluff, IL, USA). The standardization of the analysis surface was performed with Sof-Lex disks (3M ESPE) and polishing with 1200-grit sandpaper. After sintering, the blocks presented an analysis area of 5 × 5 mm, which was conditioned with the aforementioned surface methods.

Etched and non-etched axial surfaces of the G10 preparations (10% HF for 30 seconds) were also analysed.

Surface analyses of both tested strategies and G10 surfaces were performed under an SEM (JSM 5400, Jeol Ltd) to verify the topography and superficial alterations of the Y-TZP ceramic and G10 preparations.

Data Analysis

The nominal values of retentive strength were tabulated and statistical analysis was performed with the SPSS software (version 21, IBM, Chicago, IL, USA). The normality and homoscedasticity were verified and the data were subjected to one-way analysis of variance (ANOVA) and post hoc Tukey test. A Weibull analysis was also performed (Weibull modulus [m]: reliability of retention values represents the variation of strength data and expresses the size distribution of the flaw population in a structure; characteristic strength [σ_0] indicates the strength value at which 63.2% of the specimens survive).

RESULTS

One-way ANOVA (Table 2) showed a statistical difference between the retention values of the groups ($p<0.0001$). Tukey's test showed that TBS and NANO groups presented the highest strength values in relation to the other groups. The GHF15 and GHF5 groups presented intermediate values, and the GHF1 group had the lowest values (Table 3). The Weibull modulus was lower for the GHF1 group, and

Table 3: *Mean ± Standard Deviation Retentive Strength (MPa), Weibull Parameters and Failure Modes of the Zirconia Crowns[a]*

Group[b]	Retentive Strength	Weibull Parameters[c]				Failures[d]	
		σ_0	CI	*m*	CI	50S (%)	50C (%)
GHF1	2.1±0.95 Z	2.55 y	1.76-3.66	1.49 Y	0.94-2	20 (100)	—
GHF5	3.75±1.06 Y	4.14 y	3.59-4.76	3.86 X	2.44-5.19	19 (95)	1 (5)
GHF15	3.78±1.06 Y	4.22 y	3.6-4.9	3.61 X	2.28-4.85	6 (30)	14 (70)
TBS	5.6±1.68 X	6.18 x	5.36-7.1	3.9 X	2.46-5.24	8 (40)	12 (60)
NANO	5.5±0.98 X	5.91 x	5.43-6.41	6.55 X	4.14-8.81	16 (80)	4 (20)

[a] *Different letters in the same column indicate a significant difference.*
[b] *GHF1 = application of thin glaze layer + hydrofluoric acid (HF) etching for 1 minute; GHF5 = glaze application + HF for 5 minutes; GHF15 = glaze application + HF for 15 minutes; TBS = tribochemical silica coating via air-abrasion with 30-μm silica-coated alumina particles; NANO = silica nanofilm deposition (5 nm) via magnetron sputtering.*
[c] *Weibull analysis for retention values: σ_0 = characteristic resistance in MPa, and m = Weibull modulus (95% confidence intervals [Cia]). Lowercase letters were used for values and capital letters for m values.*
[dc] *50S = more than 50% of cement adhered on the substratum; 50C = more than 50% of cement adhered on the crown.*

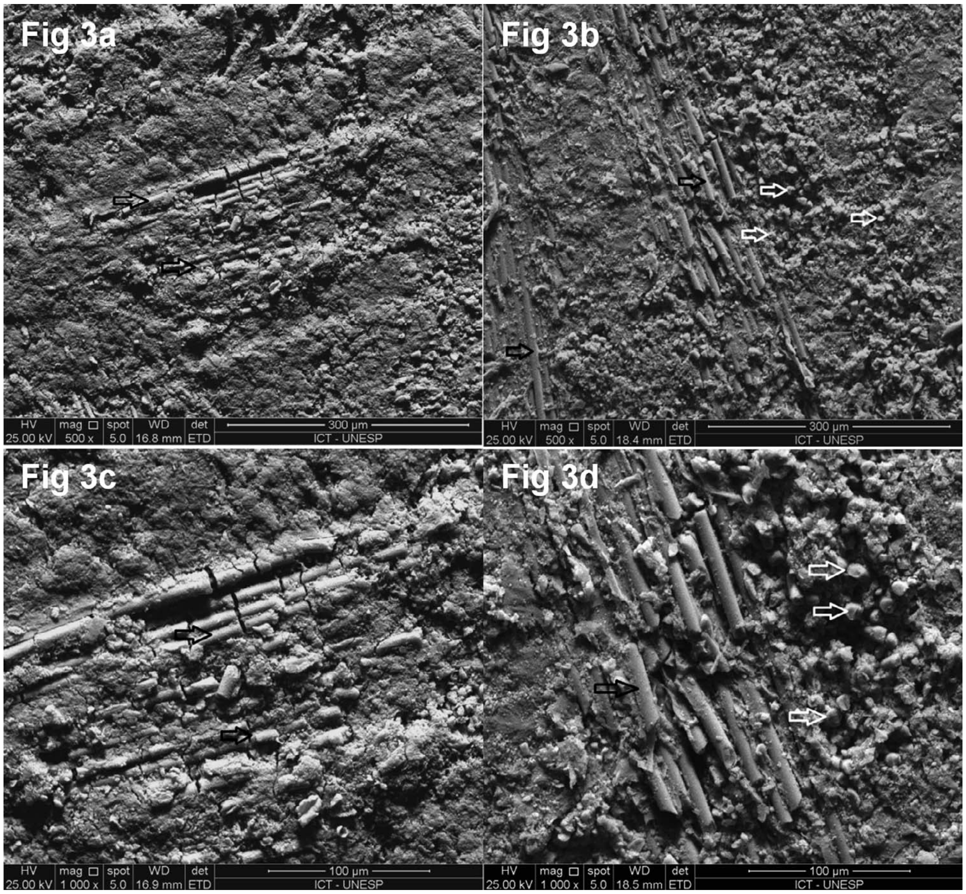

Figure 3. *Representative micrographics of the G10 surfaces with and without HF etching. (a and b): G10 axial surface nonetched and etched, respectively (×500). (c and d): G10 axial surface nonetched and etched (×1000), respectively. G10 analog material presents glass fiber in different senses; it is possible to observe in the nonetched surfaces (a and c) little exposed fiber in only one horizontal direction (black arrow), while the etched surface presents more exposed glass fiber in different directions (black and white arrows).*

the characteristic strength was increased for the TBS and NANO groups.

Figure 3a through d shows the micromorphologic differences between nonetched and etched surfaces of the polymeric material (G10).

We observed the differences between the surface morphologic patterns of the zirconia blocks treated with the different strategies (Figure 4A through M). Compared with the untreated surface (Figure 4L,M), all the groups presented topographic alterations, except the NANO group (Figure 4I,J). The etching time affected the topographic pattern of the glaze groups (the longer the etching time, the greater the presence of pores) (GHF1: Figure 4A,B; GHF5: Figure 4C,D; and GHF15: Figure 4E,F). The TBS group presented irregularities promoted by air abrasion (Figure 4G,H).

In terms of fracture type, the TBS and GHF15 groups presented a higher percentage of 50C failures compared with the other groups (Table 3, Figure 5a, 5b through e).

DISCUSSION

The null hypothesis was rejected: the TBS and NANO groups presented the highest retentive strength values, followed by the GHF15, GHF5, and GHF1 groups.

The silica nanofilm application and tribochemical silica coating presented the highest retention and characteristic strength values (Table 3) compared with other silica deposition methods. It has been shown that silica coating via air abrasion followed by silanization improves the bond strength for silica-based,[9,62] glass-infiltrated alumina,[9,63] and zirconium ceramics.[9,23,61,64-66] Using a shear bond strength test, some authors showed that silica nanofilms deposited by magnetron sputtering reached values of adhesion similar to air abrasion with silica[26] or alumina particles.[56] Despite a similar Weibull modulus (m), the NANO group had a 40% higher value of m than the TBS group, which suggests a high reliability of the silica deposition technique (Table 3). Compared with the vitrification method, deposition of the silica nanofilm via sputtering is

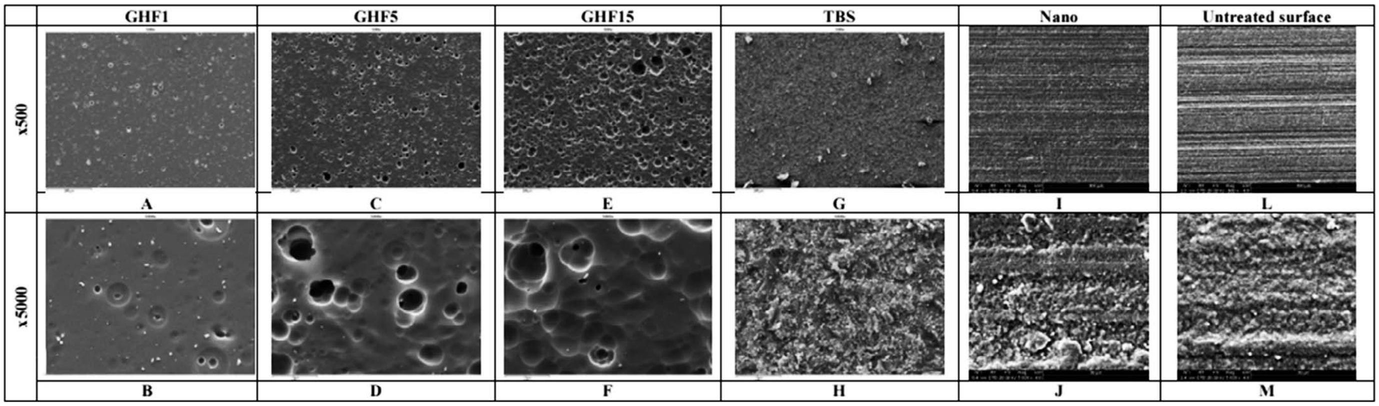

Figure 4. *Representative scanning electron micrographs of the zirconia blocks treated with the same treatments utilized in the intaglio surface of zirconia crowns. (A): GHF1 ×500. (B): GHF1 ×5000). (C): GHF5 ×500. (D): GHF5 ×5000; (E): GHF15 ×500. (F): GHF15 ×5000. (G): TBS ×500 (H): TBS ×5000. (I): NANO ×500. (J): NANO ×5000. (L): Zirconia surface without treatment ×500. (M): Zirconia surface without treatment ×5000. It is possible to observe in glaze groups the greater etching time and the greater quantity and size of pores compared with the air-abraded surface, which presents irregularities promoted by impact of silica-coated alumina particles, and the NANO group, which presents characteristic of the untreated surface (Sof-Lex disks and 1200-grit sandpaper), possibly because the nanofilm presents a thickness of 5 nm.*

rapid, and the thickness and chemical composition of the film can be controlled. Also, it does not subject the ceramic to high temperatures, and avoids the damage associated with sandblasting. However, it requires costly, specialized equipment, and specific training for use.[26]

The NANO group generated mostly 50S (more than 50% of the cement on the G10 substratum surface) failures (80%) compared with the TBS group, which presented 60% 50C (more than 50% of the cement in the crown) failures. According to studies conducted on the magnetron sputtering methods of silica deposition, the coating layer is homogeneous and shows a controlled thickness.[57,58] While the magnetron sputtering creates a 5-nm thick homogeneous silica deposition film (NANO group), the 30-μm diameter silica-coated alumina particles (TBS group) penetrate approximately 15 μm into the Y-TZP crowns.[67] Therefore, despite the fact that both methods promote silica deposition and make the surface more reactive, air abrasion caused more irregularities, thus favoring micro-mechanical retention between the zirconia and cement. This can be seen on the micrographs (Figure 4G through J)[28-30] and it is the cause of the 50C failure mode. In terms of damage effect of the particle air abrasion on zirconia materials, the literature is controversial.[31-34,68]

Among the studied silica deposition methods, the vitrification method (or glaze-on)[42] seems to be a promising technique. *In vitro* shear bond strength studies showed that the application of a thin layer of low-fusing porcelain glaze on the zirconia surface followed by HF etching generated similar or higher adhesion values compared with conventional surface-treatment methods. However, these studies conditioned the glazed surface for different durations and used different acid concentrations.[39,42-46]

In the current study, we aimed to determine the best acid-etching time of the glazed zirconia surface by means of a crown pull-out test, which evaluates not only the bond strength but also all the complex forces involved, including shear and friction. Our results partially agree with other studies that used a similar methodology (vitrification technique + pull-out crown test).[19,20] Rippe and others[20] verified the effect of the surface treatment and type of cement on the retention of Y-TZP crowns on composite cores. They also showed that the vitrification technique presented higher retention values than the group without treatment but similar to those of a tribosilicatization group using a 2-hydroxylethyl methacrylate (HEMA)-based resin cement. However, when a bisphenol A-glycidyl methacrylate (BIS-GMA)-based cement was used, there was no difference between the groups. In addition, Amaral and others[19] evaluated the retention of Y-TZP crowns with different inner treatments and different substrate types and found no difference among the treatments when a dentin substrate was used; however, when the crowns were cemented on composite cores, tribosilicatization and vitrification significantly increased the retention force in comparison with the control group (no treatment). In the current study, the GHF1 group (Figure 4A,B) presented lower values of retention than the silicatized group (TBS) (Figure 4G,H), possibly because the conditioning time (1 minute) was not enough to

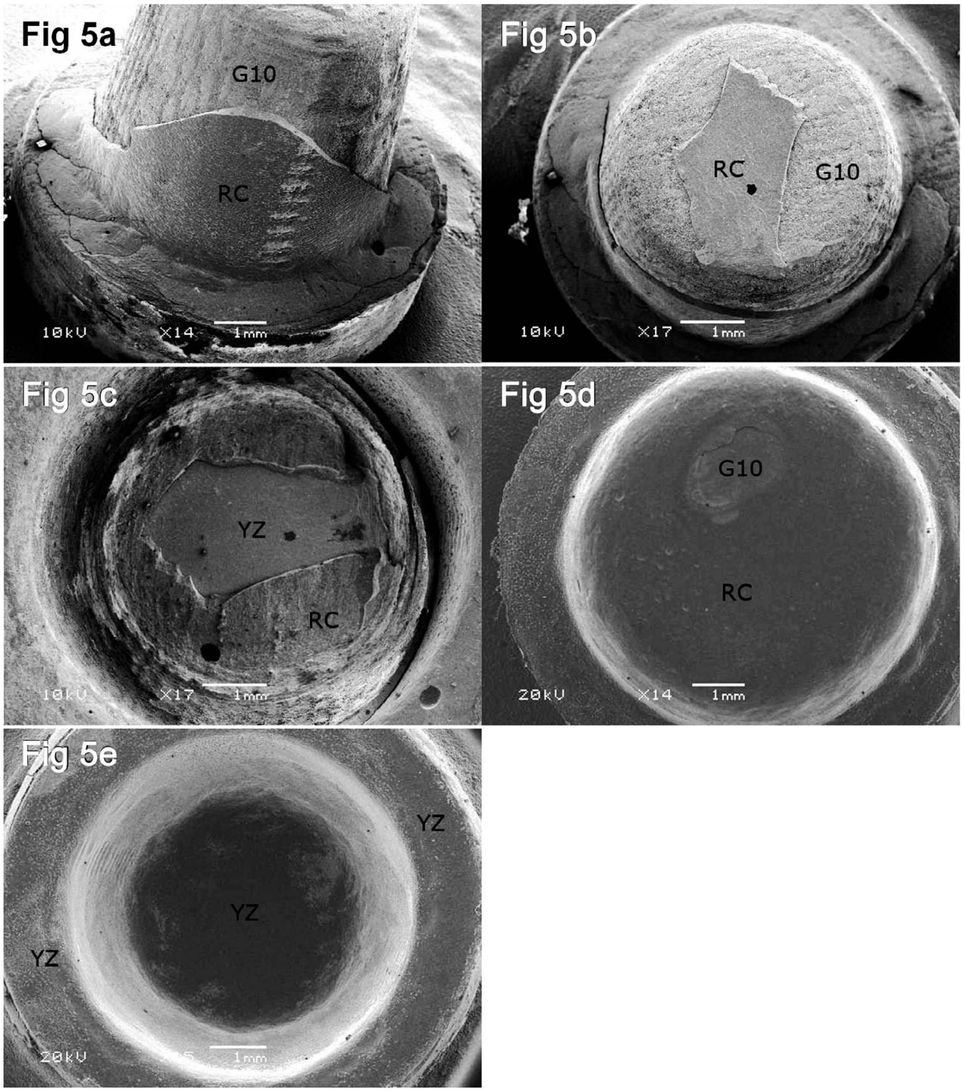

Figure 5. *(A, B, and C): 50C failure of the GH15 group specimen (A and B correspond to G10 preparation and C to intaglio surface of zirconia crown). (D and E): 50S failure of the GH1 group specimen (D corresponds to G10 peparation, and E corresponds to the intaglio surface of the zirconia crown). G10, G10 analog material; RC, resin cement; YZ, zirconia crown.*

generate significant morphologic changes on the glazed surface compared with the air-abraded surface.

Among the studied strategies, the GHF5 and GHF15 groups presented statistically similar and intermediate values of retentive strength (Table 3), as well as characteristic strength. An increase in acid-etching time improved the retention values (GHF5 and GHF15 groups) compared with the GHF1 group. However, the GHF15 group showed a higher number of 50C failures, indicating a greater adhesive interaction with the crown, similar only to the TBS group. This could have been caused by the topographic alterations (Figure 4E,F) promoted by the increased etching time[49] of the glazed surface and consequently a greater surface area for bonding, which facilitates resin cement penetration into the microretentive ceramic surface.[69] The glazing procedure is quick, is performed after the clinical verification of the fit of the crown, being applied by the dental technician. Thus, the clinician receives a zirconia crown with an etchable intaglio surface. But, standardization of the thickness of the low-fusing porcelain glaze is difficult, thus it might increase the marginal gap.[45] At the same time, space for the glaze and cement layer could be produced by computer-aided design/computer-sided manufacturing systems, thus ensuring an adequate fit.[39] Since the thickness of the glass film was approximately 31.8 μm for Ntala and others[39] and 12 μm for Bottino and others,[25] the effect on the marginal fit could be not negligible, considering that the clinically recommended maximum misfit is around 120 μm.[45,70,71] Even though the vitrification/glaze-on technique has its advantages, we believe technical improvements are needed, primarily in the standardization of the thickness of the glaze layer, in order to prevent any negative effects on the marginal fit.

It is important to highlight that the glaze composition (monophase) may have affected the retention values for glaze groups. If a multiphasic glaze material had been used, maybe better results of crown retention could have been obtained.[39,40] In this sense, the development of multiphasic glaze materials might achieve improvements.

In the current study, we utilized a woven glass-fiber-filled epoxy material (G10) as a dentin analog, in order to standardize the substrate and the preparation. This material showed excellent mechanical and adhesive properties,[59,72,73] and we understand that standardization of the substrate is crucial to verify the pretreatment factor alone in zirconia crowns. Nevertheless, Kelly and others[59] showed that resin cement bond strength to dentin was slightly lower than to the dry and wet analog material (40%-50%), which shows that caution is needed when using the G10 in adhesion tests. Up to now, we know of no other studies that compared the bond strength of dentin and G10 to demonstrate adhesion similarity of these substrates. In this sense, when adhesion studies using G10 are planned, it is very important to evaluate if the G10 as a dentin analog influences the results and interpretation. In the current study, we evaluated the zirconia/resin cement interface and most of the failures were 50S; thus, we believe that it was possible to observe the effect of the zirconia surface treatments. Figure 3a through d shows that etched G10 surfaces present more glass fiber exposed for bonding with the silane couple agent. Additionally, the use of copings in the complex adhesion/retention trial (retentive strength of crowns) reflects the clinical reality more properly, especially due to the axial loads exerted during the test.

The present study had some limitations. It is difficult to compare our results with the current literature because most studies did not present similar geometries of the preparation.[19,20,60,61,70] Furthermore, those studies that utilized zirconia crowns had a large number of variables: the substrate, preparation angle, cement type (with or without monomer phosphate), and application of primers. Mechanical cycling or fatigue experiments were not carried out in these studies either, and such test conditions should be employed in the future.[72,74-76] These new silica deposition methods should be included in prospective clinical trials to evaluate the bond effectiveness and the effect on retention of zirconia crowns under clinical situations.

CONCLUSIONS

1. Tribochemical silica coating (via particle air abrasion) and silica nanofilm deposition (via magnetron sputtering) as pretreatments of zirconia crowns in combination with RelyX ARC luting cement promote higher crown retention compared with the low-fusing porcelain glaze applications (apart from acid etching time).
2. Application of a thin layer of low-fusing porcelain glaze showed variable retention results depending on the etching time; thus, further studies about glaze coated polycrystalline zirconium oxide ceramics should be performed.

Acknowledgements

This article is based on a doctoral thesis submitted to the Graduate Program in Oral Sciences (Prosthetic Dentistry Unit), Faculty of Odontology, Federal University of Santa Maria (Santa Maria, Brazil) as part of the requirements for the PhD degree (Dr Vinicius F. Wandscher). We thank CAPES (Agency for the High-Standard Promotion of Graduate Courses, Brazil; scholarship process number 99999.005177/2014-06) for supporting this study. We also thank VIPI dental products for the acrylic resin donation.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 25 February 2017)

REFERENCES

1. Christel P, Meunier A, Heller M, Torre JP, & Peille CN (1989) Mechanical properties and short-term in-vivo evaluation of yttrium-oxide-partially-stabilized zirconia *Journal of Biomedical Materials Research* **23(1)** 45-61.
2. Luthardt RG, Sandkuhl O, & Reitz B (1999) Zirconia-TZP and alumina—Advanced technologies for the manufacturing of single crowns *European Journal of Prosthodontics and Restorative Dentistry* **7(4)** 113-119.
3. Piconi C, & Maccauro G (1999) Zirconia as a ceramic biomaterial *Biomaterials* **20(1)** 1-25.
4. Lüthy H, Loeffel O, & Hammerle CHF (2006) Effect of thermocycling on bond strength of luting cements to zirconia ceramic *Dental Materials* **22(2)** 195-200.
5. Özcan M, Kerdijk S, & Valandro LF (2008) Comparison of resin cement adhesion to Y-TZP ceramic following manufacturers' instructions of the cements only *Clinical Oral Investigations* **12(3)** 279-282.
6. Passos SP, May LG, Barca DC, Ozcan M, Bottino MA, & Valandro LF (2010) Adhesive quality of self-adhesive and conventional adhesive resin cement to Y-TZP ceramic

before and after aging conditions *Operative Dentistry* **35(6)** 689-696.

7. Melo RM, Souza R, Dursun E, Monteiro E, Valandro LF, & Bottino MA (2015) Surface treatments of zirconia to enhance bonding durability *Operative Dentistry* **40(6)** 636-643.

8. Della Bona A, Anusavice KJ, & Hood JA (2002) Effect of ceramic surface treatment on tensile bond strength to a resin cement *International Journal of Prosthodontics* **15(3)** 248-253.

9. Ozcan M, & Vallittu PK (2003) Effect of surface conditioning methods on the bond strength of luting cement to ceramics *Dental Materials* **19(8)** 725-731.

10. Brentel AS, Ozcan M, Valandro LF, Alarça LG, Amaral R, & Bottino MA (2007) Microtensile bond strength of a resin cement to feldpathic ceramic after different etching and silanization regimens in dry and aged conditions *Dental Materials* **23(11)** 1323-1331.

11. Prochnow C, Venturini AB, Grasel R, Bottino MC, & Valandro LF (2017) Effect of etching with distinct hydrofluoric acid concentrations on the flexural strength of a lithium disilicate-based glass ceramic *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **105(4)** 885-891.

12. Venturini AB, Prochnow C, Rambo D, Gundel A, & Valandro LF (2015) Effect of hydrofluoric acid concentration on resin adhesion to a feldspathic ceramic *Journal of Adhesive Dentistry* **17(4)** 313-320.

13. Amaral R, Ozcan M, Bottino MA, & Valandro LF (2011) Resin bonding to a feldspar ceramic after different ceramic surface conditioning methods: Evaluation of contact angle, surface pH, and microtensile bond strength durability *Journal of Adhesive Dentistry* **13(6)** 551-560.

14. Fabianelli A, Pollington S, Papacchini F, Goracci C, Cantoro A, Ferrari M, & van Noort R (2010) The effect of different surface treatments on bond strength between leucite reinforced feldspathic ceramic and composite resin *Journal of Dentistry* **38(1)** 39-43.

15. Corazza PH, Cavalcanti SC, Queiroz JR, Bottino MA, & Valandro LF (2013) Effect of post-silanization heat treatments of silanized feldspathic ceramic on adhesion to resin cement *Journal of Adhesive Dentistry* **15(5)** 473-479.

16. de Figueiredo VMG, Corazza PH, Lepesqueur LSS, Miranda GM; Pagani C, de Melo RM, & Valandro LF (2015) Heat treatment of silanized feldspathic ceramic: Effect on the bond strength to resin after thermocycling *International Journal of Adhesion and Adhesives* **63(December)** 96-101.

17. Derand P, & Derand T (2000) Bond strength of luting cements to zirconium oxide ceramics *International Journal of Prosthodontics* **13(2)** 131-135.

18. Yoshida K, Yamashita M, & Atsuta M (2004). Zirconate coupling agent for bonding resin luting cement to pure zirconium *American Journal of Dentistry* **17(4)** 249-252.

19. Amaral R, Rippe M, Oliveira BG, Cesar PF, Bottino MA, & Valandro LF (2014) Evaluation of tensile retention of Y-TZP crowns after long-term aging: Effect of the core substrate and crown surface conditioning *Operative Dentistry* **39(6)** 619-626.

20. Rippe MP, Amaral R, Oliveira FS, Cesar PF, Scotti R, Valandro LF, & Bottino MA (2015) Evaluation of tensile retention of Y-TZP crowns cemented on resin composite cores: effect of the cement and Y-TZP surface conditioning *Operative Dentistry* **40(1)** E1-E10.

21. Sailer I, Makarov NA, Thoma DS, Zwahlen M, & Pjetursson BE (2015) All-ceramic or metal-ceramic tooth-supported fixed dental prostheses (FDPs)? A systematic review of the survival and complication rates. Part I: Single crowns (SCs) *Dental Materials* **31(6)** 603-623.

22. Pjetursson BE, Sailer I, Makarov NA, Zwahlen M, & Thoma DS (2015) All-ceramic or metal-ceramic tooth-supported fixed dental prostheses (FDPs)? A systematic review of the survival and complication rates. Part II: Multiple-unit FDPs *Dental Materials* **31(6)** 624-639.

23. Bottino MA, Valandro LF, Scotti R, & Buso L (2005) Effect of surface treatments on the resin bond to zirconium-based ceramic *International Journal of Prosthodontics* **18(1)** 50-65.

24. Valandro LF, Ozcan M, Bottino MC, Bottino MA, Scotti R, & Bona AD (2006) Bond strength of a resin cement to high-alumina and zirconia-reinforced ceramics: The effect of surface conditioning *Journal of Adhesive Dentistry* **8(3)** 175-181.

25. Bottino MA, Bergoli C, Lima EG, Marocho SM, Souza RO, & Valandro LF (2014) Bonding of Y-TZP to dentin: effects of Y-TZP surface conditioning, resin cement type, and aging *Operative Dentistry* **39(3)** 291-300.

26. Druck CC, Pozzobon JL, Callegari GL, Dorneles LS, & Valandro LF (2015) Adhesion to Y-TZP ceramic: Study of silica nanofilm coating on the surface of Y-TZP *Journal of Biomedical Materials Research. Part B, Applied Biomaterials* **103(1)** 143-150.

27. Pozzobon JL, Missau T, Druck CC, Özcan M, & Valandro LF (2016) Effects of different particle deposition parameters on adhesion of resin cement to zirconium dioxide and phase transformation *Journal of Adhesion Science and Technology* **30(4)** 412-421.

28. Kern M, & Wegner SM (1998) Bonding to zirconia ceramic: Adhesion methods and their durability *Dental Materials* **14(1)** 64-71.

29. Della Bona A, Donassollo TA, Demarco FF, Barrett AA, & Mecholsky JJ (2007) Characterization and surface treatment effects on topography of a glass-infiltrated alumina/zirconia-reinforced ceramic. *Dental Materials* **23(6)** 769-775.

30. Ozcan M, Cura C, & Valandro LF (2011) Early bond strength of two resin cements to Y-TZP ceramic using MPS or MPS/4-META silanes *Odontology* **99(1)** 62-67.

31. Wang H, Aboushelib MN, & Feilzer AJ (2007) Strength influencing variables on CAD/CAM zirconia frameworks. *Dental Materials* **24(5)** 633-638.

32. Studart AR, Filser F, Kocher P, & Gauckler LJ (2007) In vitro lifetime of dental ceramics under cyclic loading in water *Biomaterials* **28(17)** 2695-2705.

33. Scherrer SS, Cattani-Lorente M, Vittecoq E, de Mestral F, Griggs JA, & Wiskott HW (2011) Fatigue behavior in water of Y-TZP zirconia ceramics after abrasion with 30 μm silica-coated alumina particles *Dental Materials* **27(2)** 28-42.

34. Souza RO, Valandro LF, Melo RM, Machado JP, Bottino MA, & Ozcan M (2013) Air-particle abrasion on zirconia ceramic using different protocols: Effects on biaxial flexural strength after cyclic loading, phase transformation and surface topography *Journal of the Mechanical Behavior of Biomedical Materials* **26(October)** 155-163.

35. Monaco C, Caldari M, & Scotti R (2013) Clinical evaluation of 1,132 zirconia-based single crowns: A retrospective cohort study from the AIOP clinical research group *International Journal of Prosthodontics* **26(5)** 435-442.

36. Koenig V, Vanheusden AJ, Le Goff SO, & Mainjot AK (2013) Clinical risk factors related to failures with zirconia-based restorations: An up to 9-year retrospective study *Journal of Dentistry* **41(12)** 1164-1174.

37. Anami LC, Lima J, Valandro LF, Kleverlaan CJ, Feilzer AJ, & Bottino MA (2016) Fatigue resistance of Y-TZP/porcelain crowns is not influenced by the conditioning of the intaglio surface *Operative Dentistry* **41(1)** E1-E12.

38. Campos F, Valandro LF, Feitosa SA, Kleverlaan CJ, Feilzer AJ, Jager N, & Bottino MA (2017) Adhesive cementation promotes higher fatigue resistance to zirconia crowns *Operative Dentistry* **42(2)** 215-224.

39. Ntala P, Chen X, Niggli J, & Cattell M (2010) Development and testing of multiphase glazes for adhesive bonding to zirconia substrates *Journal of Dentistry* **38(10)** 773-781.

40. Cattell MJ, Chadwick TC, Knowles JC, & Clarke RL (2009) The development and testing of glaze materials for application to the fit surface of dental ceramic restorations *Dental Materials* **25(4)** 431-441.

41. Kitayama S, Nikaido T, Maruoka R, Zhu L, Ikeda M, Watanabe A, Foxton RM, Miura H, & Tagami J (2009) Effect of an internal coating technique on tensile bond strengths of resin cements to zirconia ceramics *Dental Materials Journal* **28(4)** 446-453.

42. Cura C, Özcan M, Isik G, & Saracoglu A (2012) Comparison of alternative adhesive cementation concepts for zirconia ceramic: Glaze layer vs zirconia primer *Journal of Adhesive Dentistry* **14(1)** 75-82.

43. Everson PI, Addison O, Palin WM, & Burke FJ (2012) Improved bonding of zirconia substructures to resin using a "glaze-on" technique *Journal of Dentistry* **40(4)** 347-351.

44. Valentino TA, Borges GA, Borges LH, Platt JA, & Correr-Sobrinho L (2012) Influence of glazed zirconia on dual-cure luting agent bond strength *Operative Dentistry* **37(2)** 181-187.

45. Vanderlei A, Bottino M, & Valandro LF (2013) Evaluation of resin bond strength to yttria-stabilized tetragonal zirconia and framework marginal fit: Comparison of different surface conditionings. *Operative Dentistry* **39(1)** 50-63.

46. Bottino MA, Bergoli C, Lima EG, Marocho SM, Souza RO, & Valandro LF (2014) Bonding of Y-TZP to dentin: Effects of Y-TZP surface conditioning, resin cement type, and aging *Operative Dentistry* **39(3)** 291-300.

47. Barghi N, Fischer DE, & Vatani L (2006) Effects of porcelain leucite content, types of etchants, and etching time on porcelain-composite bond *Journal of Esthetic and Restorative Dentistry* **18(1)** 47-52.

48. Neto SD, Naves LZ, Costa AR, Correr AB, Consani S, Borges GA, & Correr-Sobrinho L (2015) The effect of hydrofluoric acid concentration on the bond strength and morphology of the surface and interface of glass ceramics to a resin cement *Operative Dentistry* **40(5)** 470-479.

49. Naves LZ, Soares CJ, Moraes RR, Gonçalves LS, Sinhoreti MA, & Correr-Sobrinho L (2010) Surface/interface morphology and bond strength to glass ceramic etched for different periods *Operative Dentistry* **35(4)** 420-427.

50. Yen TW, Blackman RB, & Baez RJ (1993) Effect of acid etching on the flexural strength of a feldspathic porcelain and a castable glass ceramic *Journal of Prosthetic Dentistry* **70(3)** 224-233.

51. Della Bona A, Anusavice KJ, & Hood JA (2002) Effect of ceramic surface treatment on tensile bond strength to a resin cement *International Journal of Prosthodontics* **15(3)** 248-253.

52. Hussain MA, Bradford EW, & Charlton G (1979) Effect of etching on the strength of aluminous porcelain jacket crowns *British Dental Journal* **147(4)** 89-90.

53. Kukiattrakoon B, & Thammasitboon K (2007) The effect of different etching times of acidulated phosphate fluoride gel on the shear bond strength of high-leucite ceramics bonded to composite resin *Journal of Prosthetic Dentistry* **98(1)** 17-23.

54. Guler AU, Yilmaz F, Yenisey M, Guler E, & Ural C (2006) Effect of acid etching time and a self-etching adhesive on the shear bond strength of composite resin to porcelain *Journal of Adhesive Dentistry* **8(1)** 21-25.

55. de Queiroz JRC, Duarte DA, de Souza ROA, Fissmer SF, Massi M, & Bottino MA (2011) Deposition of SiO_x thin films on Y-TZP by reactive magnetron sputtering: influence of plasma parameters on the adhesion properties between Y-TZP and resin cement for application in dental prosthesis *Materials Research* **14(2)** 212-216.

56. de Queiroz JRC, Massi M, Nogueira L Jr, Sobrinho AS, Bottino MA, & Ozcan M (2013) Silica-based nano-coating on zirconia surfaces using reactive magnetron sputtering: Effect on chemical adhesion of resin cements *Journal of Adhesive Dentistry* **15(2)** 151-159.

57. Ohring M (1992) *The Materials Science of Thin Films* Academic Press, London.

58. Denardin JC, Knobel M, Dorneles LS, & Schelp LF (2005) Structural, magnetic and transport properties of discontinuous granular multi-layers *Journal of Magnetism and Magnetic Materials* **294(2)** 206-212.

59. Kelly JR, Rungruanganunt P, Hunter B, & Vailati F (2010) Development of a clinically validated bulk failure test for ceramic crowns *Journal of Prosthetic Dentistry* **104(4)** 228-238.

60. Palacios RP, Johnson GH, Phillips KM, & Raigrodski AJ (2006) Retention of zirconium oxide ceramic crowns with three types of cement *Journal of Prosthetic Dentistry* **96(2)** 104-114.

61. Ernst CP, Cohnen U, Stender E, & Willershausen B (2005) In vitro retentive strength of zirconium oxide ceramic crowns using different luting agents *Journal of Prosthetic Dentistry* **93(6)** 551-558.

62. Sun R, Suansuwan N, Kilpatrick N, & Swain M (2000) Characterisation of tribochemically assisted bonding of composite resin to porcelain and metal *Journal of Dentistry* **28(6)** 441-445.

63. Valandro LF, Della Bona A, Antonio Bottino M, & Neisser MP (2005) The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic *Journal of Prosthetic Dentistry* **93(3)** 253-259.

64. Inokoshi M, Kameyama A, De Munck J, Minakuchi S, & Van Meerbeek B (2013) Durable bonding to mechanically and/or chemically pre-treated dental zirconia *Journal of Dentistry* **41(2)** 170-179.

65. Inokoshi M, Poitevin A, De Munck J, Minakuchi S, & Van Meerbeek B (2014) Bonding effectiveness to different chemically pre-treated dental zirconia *Clinical Oral Investigations* **18(7)** 1803-1812.

66. Bielen V, Inokoshi M, Munck JD, Zhang F, Vanmeensel K, Minakuchi S, Vleugels J, Naert I, & Van Meerbeek B (2015) Bonding effectiveness to differently sandblasted dental zirconia *Journal of Adhesive Dentistry* **17(3)** 235-242.

67. 3M ESPE (1998) Cojet System: Product Dossier *Clinical Research* 3/98.

68. Ozcan M, Melo RM, Souza ROA, Machado PB, Valandro LF, & Botttino MA (2013) Effect of air-particle abrasion protocols on the biaxial flexural strength, surface characteristics and phase transformation of zirconia after cyclic loading *Journal of Biomedical Materials Research. Part B, Applied Biomaterials* **20(April)** 19-28.

69. Stangel I, Nathanson D, & Hsu CS (1987) Shear strength of the composite bond to etched porcelain *Journal of Dental Research* **66(9)** 1460-1465.

70. Yeo IS, Yang JH, & Lee JB (2003) In vitro marginal fit of three all-ceramic crown systems *Journal of Prosthetic Dentistry* **90(5)** 459-464.

71. Gonzalo E, Suarez MJ, Serrano B, & Lozano JF (2009) A comparison of the marginal vertical discrepancies of zirconium and metal ceramic posterior fixed dental prostheses before and after cementation *Journal of Prosthetic Dentistry* **102(6)** 378-384.

72. May LG, Kelly JR, Bottino MA, & Hill T (2012) Effects of cement thickness and bonding on the failure loads of CAD/CAM ceramic crowns: Multi-physics FEA modeling and monotonic testing *Dental Materials* **28(8)** e99-e109.

73. Corazza PH, Feitosa SA, Borges AL, & Della Bona A (2013) Influence of convergence angle of tooth preparation on the fracture resistance of Y-TZP-based all-ceramic restorations *Dental Materials* **29(3)** 339-347.

74. Venturini AB, Prochnow C, May LG, Kleverlaan CJ, & Valandro LF (2017) Fatigue failure load of feldspathic ceramic crowns after hydrofluoric acid etching at different concentrations *Journal of Prosthetic Dentistry* May 26 [Epub ahead of print] doi: 10.1016/j.prosdent.2017.03.021.

75. Pereira GK, Silvestri T, Amaral M, Rippe MP, Kleverlaan CJ, & Valandro LF (2016) Fatigue limit of polycrystalline zirconium oxide ceramics: Effect of grinding and low-temperature aging *Journal of the Mechanical Behavior of Biomedical Materials* **61(August)** 45-54.

76. Fraga S, Pereira GK, Freitas M, Kleverlaan CJ, Valandro LF, & May LG (2016) Loading frequencies up to 20Hz as an alternative to accelerate fatigue strength tests in a Y-TZP ceramic *Journal of the Mechanical Behavior of Biomedical Materials* **61(August)** 79-86.