Effect of Conditioning Methods on the Microtensile Bond Strength of Phosphate Monomer-Based Cement on Zirconia Ceramic in Dry and Aged Conditions

Regina Amaral,[1] Mutlu Özcan,[2] Luiz Felipe Valandro,[1,3] Ivan Balducci,[1] Marco Antonio Bottino[1]

[1] Department of Dental Materials and Prosthodontics, São José dos Campos Dental School, São Paulo State University (UNESP), São José dos Campos, Brazil

[2] Department of Dentistry and Dental Hygiene, Clinical Dental Biomaterials, University Medical Center Groningen, University of Groningen, Groningen, The Netherlands

[3] Department of Restorative Dentistry, Division of Prosthodontics, School of Dentistry, Federal University of Santa Maria, Santa Maria, Brazil

Abstract: The objective of this study was to evaluate the durability of bond strength between a resin cement and aluminous ceramic submitted to various surface conditioning methods. Twenty-four blocks ($5 \times 5 \times 4$ mm^3) of a glass-infiltrated zirconia–alumina ceramic (In-Ceram Zirconia Classic) were randomly divided into three surface treatment groups: ST1—Air-abrasion with 110-μm Al$_2$O$_3$ particles + silanization; ST2—Laboratory tribochemical silica coating method (110-μm Al$_2$O$_3$, 110-μm silica) (Rocatec) + silanization; ST3—Chairside tribochemical silica coating method (30-μm SiO$_x$) (CoJet) + silanization. Each treated ceramic block was placed in its silicone mold with the treated surface exposed. The resin cement (Panavia F) was prepared and injected into the mold over the treated surface. Specimens were sectioned to achieve nontrimmed bar specimens (14 sp/block) that were randomly divided into two conditions: (a) Dry—microtensile test after sectioning; (b) Thermocycling (TC)—(6,000×, 5–55°C) and water storage (150 days). Thus, six experimental groups were obtained ($n = 50$): Gr1—ST1 + dry; Gr2—ST1 + TC; Gr3—ST2 + dry; Gr4—ST2 + TC; Gr5—ST3 + dry; Gr6—ST3 + TC. After microtensile testing, the failure types were noted. ST2 (25.1 ± 11) and ST3 (24.1 ± 7.4) presented statistically higher bond strength (MPa) than that of ST1 (17.5 ± 8) regardless of aging conditions ($p < 0.0001$). While Gr2 revealed the lowest results (13.3 ± 6.4), the other groups (21.7 ± 7.4–25.9 ± 9.1) showed statistically no significant differences (two-way ANOVA and Tukey's test, $\alpha = 0.05$). The majority of the failures were mixed (82%) followed by adhesive failures (18%). Gr2 presented significantly higher incidence of ADHESIVE failures (54%) than those of other groups ($p = 0.0001$). Both laboratory and chairside silica coating plus silanization showed durable bond strength. After aging, air-abrasion with 110-μm Al$_2$O$_3$ + silanization showed the largest decrease indicating that aging is fundamental for bond strength testing for acid-resistant zirconia ceramics in order to estimate their long-term performance in the mouth. © 2007 Wiley Periodicals, Inc. J Biomed Mater Res Part B: Appl Biomater 85B: 1–9, 2008

Keywords: bond strength; durability; microtensile test; Panavia; resin cement; surface conditioning; tribochemical silica coating; zirconia

INTRODUCTION

Ceramics with high crystalline content (aluminum and zirconia oxides) are reported to present more favorable clinical results than feldspathic, leucite, and lithium disilicate ceramics.[1–4] The increased content of alumina (Al$_2$O$_3$) in feldspathic ceramics led to a significant increase in mechanical properties of these materials, allowing indication for more predictable metal-free restorations in regions where high mechanical strength is needed.[5–8] On the other hand, the increase in mechanical resistance by the increase in high strength crystalline content and decrease of the glass phase led to an acid-resistant ceramic. In such ceramics, acid etching does not produce significant topographic change to achieve proper micromechanical bonding of resin materi-

als.[9–13] The ceramic In-Ceram Zirconia presents high crystalline content (67% aluminum oxide and 13% tetragonal zirconia crystals), with only 20% glass phase (lanthanum silicate aluminate),[14] thus yielding a ceramic substrate resistant to the action of acids.[15–17]

Ceramics may be classified as acid-sensitive and acid-resistant according to their sensitivity to hydrofluoric (HF) acid gel.[16,17] Within this concept, bonding to the ceramic substrate may be established according to the presence of silica on the surface that presents chemical compatibility with the silane agent, as previously described.[15] The surface of glassy ceramics is degraded by etching with HF acid gel, further exposing silica oxides and increasing the surface roughness for micromechanical bonding.[13,15,18] Because of the microstructure with high crystalline content (aluminum and zirconia oxides), these ceramics could not be etched by HF gel since this agent does not etch the high strength crystalline phase but the glass phase.[9–12,15,18] Only silicon oxide-based ceramics could undergo dissolution of the glass matrix by acid etching.[18–23] Within this context, the so-called acid-sensitive ceramics or glass ceramics (feldspathic, leucite, and lithium disilicate-based ceramics) present surface degradation by the HF gel, resulting in a topographic pattern that favors micromechanical bonding.[17] Also, silane coupling agents applied on the surface may yield chemical adhesion between the ceramic and resin materials, due to their bifunctional characteristics.[15,18–24] On the other hand, acid-resistant ceramics (glass-infiltrated alumina or alumina/zirconia ceramics, yttrium oxide-stabilized tetragonal zirconia ceramics [Y-TZP], and densely sintered alumina ceramics) are not etched by HF gel and are usually submitted to airborne particle abrasion in order to optimize bonding of the resin cement.[9–12,25,26,28–32]

Laboratory or chairside air-abrasion with 110- and 30-μm silica-coated aluminum particles (tribochemical silica coating) provide surface conditioning for acid-resistant ceramics.[12,15,17,33,34] These air-abrasion systems allow the ceramic surface to be embedded by silica.[35] Silica-modified surfaces are chemically more reactive to the resin via silane coupling agents.

Silane molecules react with water to form three silanol groups (—Si—OH) from the corresponding methoxy groups (—Si—O—CH$_3$).[15,36–39] The silanol groups then react further to form a siloxane (—Si—O—Si—O—) network with the silica surface. Methacrylate end groups of the silane molecules react with the methacrylate groups of adhesive resins in a free radical polymerization process. In summary, the tribochemically assisted systems allow for chemical bond via the silica layer on the surface, silane coupling agent and resin cement. The performance of resin cements that contain 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomers has been studied previously.[25,27–29,32,40,41] The results revealed high bonding durability of this cement to acid-resistant ceramics because the phosphate ester group of MDP bonds directly to the metal oxides.[25,27,42] It is observed in these studies that the combination of air-abrasion with aluminum oxide particles (Al$_2$O$_3$) and an MDP-based resin cement was able to generate stable bond to dense ceramics without glass phase (Y-TZP ceramics and densely sinterized alumina) while silica coating and silanization in combination with bis-GMA-based resin cements, without MDP monomer, did not yield stable bonds.[25,27–29,32,40] However, controversial reports exist in the dental literature particularly after aging conditions.[31,34,43]

Therefore, the objectives of this study were to evaluate the durability of bond strength between resin cement containing phosphate monomer and a glass-infiltrated zirconia–alumina ceramic submitted to three different surface treatments before and after aging. The study hypotheses were that bond strength may be affected depending on the surface treatment and storage condition and durability of bonding may be improved by silica coating and silanization.

MATERIALS AND METHODS

For this study, acrylic resin templates were machined with dimensions of $5 \times 5 \times 4$ mm^3 that were employed for fabrication of 24 glass-infiltrated zirconia ceramic blocks (In-Ceram Zirconia Classic, Vita Zahnfabrik, Bad Sackingen, Germany) with the same dimensions. The technical procedures for fabrication of these blocks followed the manufacturer's recommendations: The acrylic resin templates were fixed on flat surface and the special plaster (Sprint, Vita Zahnfabrik, Bad Sackingen, Germany) was poured onto the acrylic block. Later, these blocks were removed; the In-Ceram Zirconia Classic powder was prepared and applied inside the plaster molds. The zirconia blocks were then removed and sintered (Inceramat, Vita). The infiltration glass for In-Ceram Zirconia was applied on each block and submitted to sintering and the zirconia blocks were air-abraded with aluminum oxide to remove the glass excess.

The cementation surface (5×5 mm^2) was leveled and polished in a machine using silicone carbide papers in sequence (600, 800, and 1200) under water cooling (3M, St. Paul, USA). Impressions were made from each ceramic block with addition silicone putty (Express, 3M ESPE, St. Paul, USA; batch # 7312). The block was pulled inside the silicone in order to achieve a 3-mm distance between the upper portion of the mold and the surface of the block. Thereafter, the cement was injected into this space. The specimen preparation has previously been described in detail elsewhere.[34]

Surface Treatment Methods

Twenty-four ceramic blocks were randomly divided into three groups, according to the ceramic surface treatment (ST):

ST1: Airborne particle abrasion (Micro-Etcher, Danville Inc., San Ramon, CA, USA) with 110-μm Al$_2$O$_3$ particles was performed perpendicular to the surface from a distance of approximately 10 mm for a period of 15 s at 2.8 bar pres-

sure. After air blasting the remnants of the sand particles gently, silane coupling agent (ESPE-Sil, 3M ESPE AG, Seefeld, Germany; Batch # 116459) was applied on the surface and allowed to stand for 5 min for its evaporation.

ST2: In this group, tribochemical silica coating system (Rocatec system, 3M ESPE AG; batch # 142820) was used. The specimens were first air-abraded with 110-μm Al_2O_3 particles (Rocatec-Pre) followed by silica-coated Al_2O_3 particles (110 μm, Rocatec-Plus, 3M ESPE AG) under the conditions as described in ST1 for 15 s and then silanized.

ST3: Chairside tribochemical silica-coating system was used, using an intraoral chairside air-abrasion device (Micro-Etcher) filled with CoJet®-Sand (30-μm Al_2O_3 particles coated with silica) (30-μm CoJet-Sand, CoJet system, 3M ESPE AG; Batch # 142820) perpendicular to the surface from a distance of approximately 10 mm for a period of 15 s at 2.8 bar pressure. The specimens were silanized according to the aforementioned protocol in ST1.

Additional ceramic specimens (1 specimen/group) were prepared and submitted to the three types of air abrasion procedures corresponding to the experimental groups and analyzed using Scanning Electron Microscope (SEM) (Jeol JSM T330A, Jeol Ltd., Tokyo, Japan) in order to observe the topographic patterns achieved by the treatment methods proposed.

Application of Cement

Each treated ceramic block was placed in its silicone mold. The resin cement (Panavia F, Kuraray, Okayama, Japan; Batch # 00022E) was mixed according the manufacturer's instructions and injected into the mold on the treated surface of the ceramic block, using a centrix syringe (DFL, Rio de Janeiro, Brazil).

The cement in the mold was photoactivated (XL 3000, 3M ESPE, MN, USA; light output: 500 mW/cm^2) for 40 s at each side of the specimen.

The intensity of the light was verified not to be lower than 500 mW/cm^2 using a radiometer (Demetron LC, Kerr) before starting the polymerization application per group. Oxygen inhibiting gel (Oxyguard II, Kuraray, Okayama, Japan; Batch # 00482A) was applied on the free surfaces. After 10 min, the ceramic block–resin cement assembly was removed from the mold and the cement was once again submitted to light polymerization from the five aspects of the block (upper and lateral) for 40 s per side.[44] The blocks were rinsed with water and stored in distilled water at 37°C for 24 h, until preparation of the specimens for the microtensile test.

Specimen Preparation for the Microtensile Test

Ceramic–cement blocks were sectioned using a diamond disc (Microdont, São Paulo, Brazil, n. 34570) at low-speed, under water cooling, in a sectioning machine (LabCut 1010, Extec, Enfield, CT, USA)[45] Initially, the cemented blocks were fixated with cyanoacrylate adhesive gel (Super Bonder Gel, Loctite Ltd., São Paulo, Brazil) on a metallic base that
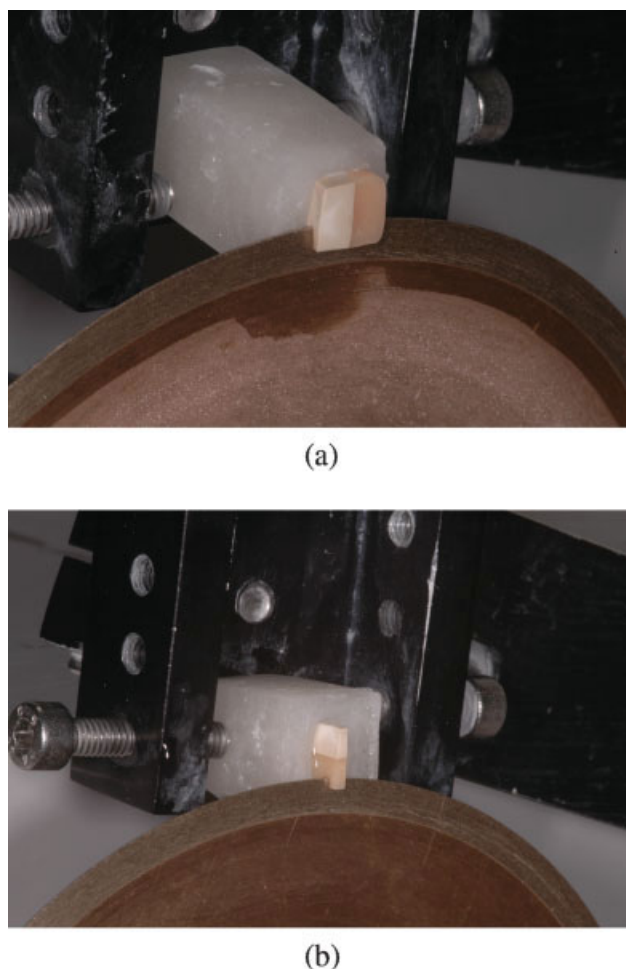

Figure 1. (a) Resin cement applied on ceramic block and attached to the metal base and (b) production of nontrimmed of bar specimens. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

was attached to the sectioning machine. The blocks were positioned as perpendicular as possible in relation to the diamond disc of the machine. The first section, measuring approximately 1 mm was discarded in case of the possibility of excess or absence of cement at the interface that might alter the results. Thereafter, two sections measuring 0.8 $\pm$ 0.1 mm in thickness were obtained. Each section was rotated in 90° and once again fixated to the metallic base. The first section was discarded (1 $\pm$ 0.1 mm) because of the aforementioned reasons. Subsequently, three to four other sections were obtained, also measuring 0.8 $\pm$ 0.1 mm in thickness. This process was followed for the other two sections and thus only the central specimens were used for the experiments.[34] Fourteen specimens were obtained from each block. The beam specimens had nonmachined (nontrimmed) bonding areas with a bonded area measuring approximately 0.6 mm^2 and 10-mm length [Figure 1(a,b)].

The 14 specimens obtained from each ceramic block were randomly divided into two testing conditions. In dry conditions (dry), the specimens were submitted to microtensile test immediately after sectioning. In aged conditions

(TC), specimens were submitted to thermal cycling (6 cycles; 5–55°C, dwelling time: 30 s, transfer time: 2 s) and stored in distilled water at 37°C for 150 days and then submitted to testing. Thus, six groups were obtained, considering the "surface treatment" (3 levels) and "storage condition" (2 levels) factors yielding to 50 specimens each (Table I).

Microtensile Bond Strength Test

Each specimen was fixated with cyanoacrylate gel (Super Bonder Gel, Loctite Ltd., São Paulo, Brazil), keeping the adhesive zone free, to the rods of a device adapted for this test. The specimens were positioned parallel to the long axis of the device in order to reduce the bending stresses. The device was fixated in the universal testing machine (EMIC DL-1000, EMIC, São José dos Pinhais, Brazil), as parallel as possible in relation to application of the tensile load, and testing was performed at a crosshead speed of 1 mm/min.[34]

The bond strength was calculated according to the formula $R = F/A$, where R is the strength (MPa), F is the load required for rupture of the specimen (N), and A is the interface area of the specimen (mm^2), measured with a digital caliper before the test.

Failure Type Analysis

All specimens (300) submitted to the microtensile test were analyzed using light microscopy (MP 320, Carl Zeiss, Jena, Germany) at 50× magnification and some specimens were selected for analysis in scanning electron microscopy (SEM) at 100× and 2000× magnification for observation of the type of failure, namely between ceramic and cement (ADHES); cohesive failure of the cement (COHES-cem); cohesive failure of ceramic (COHES-cer); cohesive failure of cement and ceramic (MIX).

Statistical Analysis

The means of each group were analyzed using two-way analysis of variance (ANOVA), with microtensile bond strength as the dependent variable and the surface conditioning methods and the storage conditions as the independent factors (Statistix 8.0 for Windows, Analytical Software Inc, Tallahassee, FL, USA). P values less than 0.05 are consid-

ered to be statistically significant in all tests. Multiple comparisons were made by Tukey's adjustment test. Furthermore, one-way ANOVA was used to determine the significant differences between surface conditioning methods. The distribution of the failure types were statistically analyzed using χ^2 analysis. The beam was used as the experimental unit since the aging affect was tested on the beams instead of the blocks.

RESULTS

Microtensile Bond Strength Results

Means and standard deviations are described in Table II.

The two-way ANOVA revealed significant influence of the storage condition ($p < 0.0001$), surface treatment method ($p < 0.0001$). Interaction between factors was also significant ($p = 0.010$). The Tukey test for multiple comparisons showed that tribochemical silica coating systems ST2 (25.1 ± 11 MPa) and ST3 (24.1 ± 7 MPa) produced statistically higher bond strengths compared to air abrasion with Al_2O_3 (ST1) (17.5 ± 8 MPa).

Gr2 (13.3 ± 6.4 MPa) showed significantly lower results than those of Gr1 (21.7 ± 7 MPa), Gr3 (25.9 ± 9.1 MPa), Gr4 (24.3 ± 12 MPa), Gr5 (25.7 ± 7.4 MPa), and Gr6 (22.5 ± 5.9 MPa) ($p < 0.0001$).

Gr2 presented significantly higher incidence of ADHESIVE failures (54%) than those of other groups ($p = 0.0001$). There was however no significant difference between the groups for MIXED failures ($p = 0.0965$).

Topographic Analysis and Failure Types

Throughout the experiments no failures of the cyanoacrylate occurred. Even though the silica coating groups showed higher bond strength values compared to the group air-abraded with Al_2O_3, the topographic patterns of the specimen surfaces after three treatment methods appeared to be similar with sand particles attached to the surface [Figure 2 (a–c)].

Light microscopy analysis demonstrated that 82% of specimens presented MIX failure type whereas 18% of specimens displayed ADHES type of failures. Specimens treated with the silica coating systems (Gr3–Gr6), as well as the group treated with Al_2O_3 (Gr1) without water storage

TABLE II. Mean (±SD) of Microtensile Bond Strength Data (MPa)

Surface Treatment	Storage	
	Dry	TC
Al_2O_3	Gr1: 21.7 ± 7.4[a]	Gr2: 13.3 ± 6.4[b]
SiO_x (Rocatec)	Gr3: 25.9 ± 9.1[a]	Gr4: 24.3 ± 12[a]
SiO_x (Cojet)	Gr5: 25.7 ± 6.8[a]	Gr6: 22.5 ± 5.9[a]

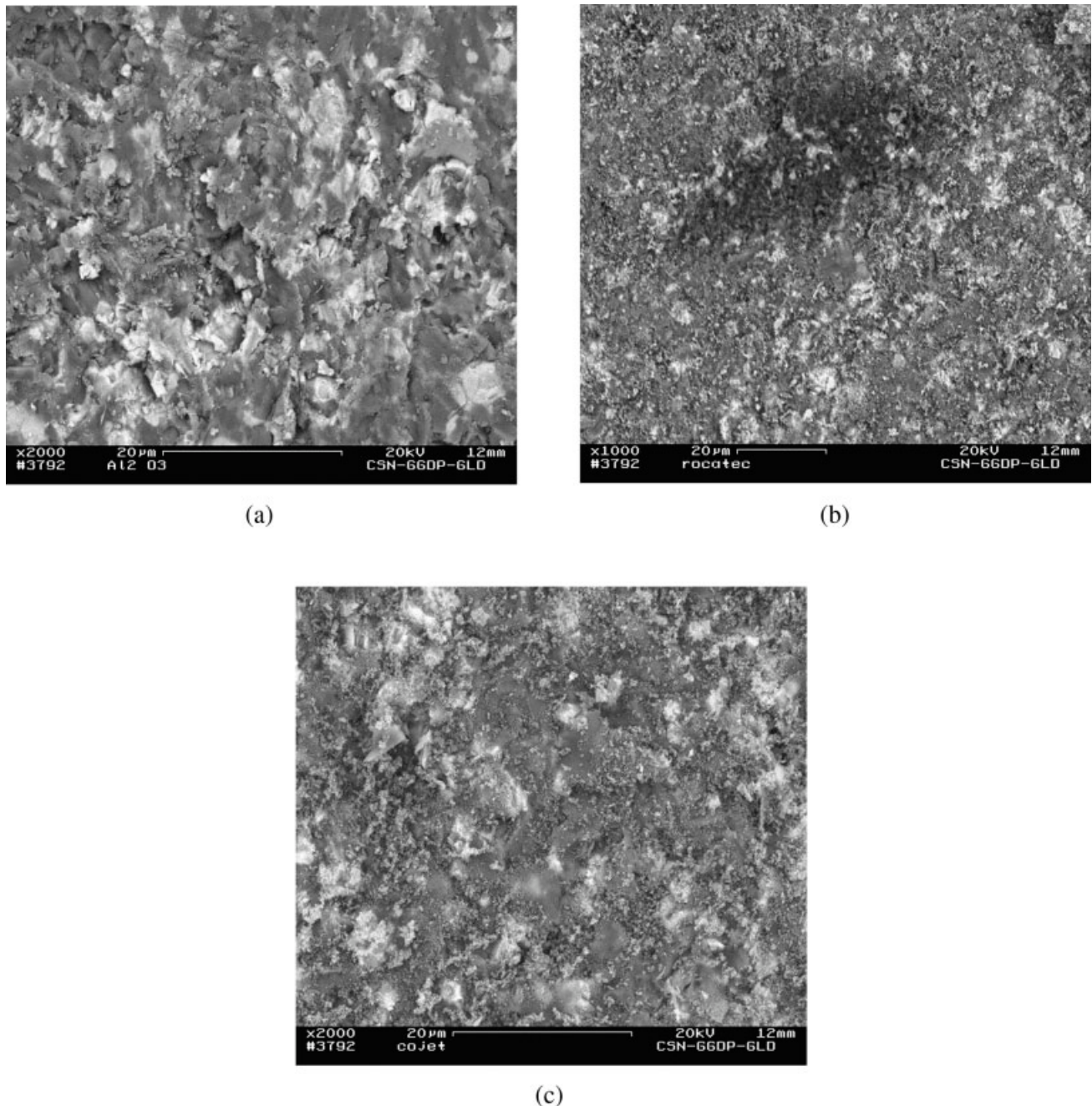Different superscript letters indicate significant differences.

Figure 2. Typical SEM view of the conditioned ceramic surface for (a) air-abrasion with 110-μm Al$_2$O$_3$ particles (ST1), (b) air-abrasion with 110-μm Al$_2$O$_3$ and SiO$_x$ (ST2) and (c) air-abrasion with 30-μm silica coated Al$_2$O$_3$ (ST3) (original magnification ×2000). Note that the sand particles penetrated the substrate surfaces and covered them with abundant sand particles.

presented predominantly MIX failure type (86–90%) and the incidence of ADHES failures were 54% in the group treated with Al$_2$O$_3$ (Gr2) (Table III). Micrograph representing the failure types of the specimens are presented in Figure 3.

DISCUSSION

The present study revealed statistically significant interaction. The microtensile bond strength of resin cement, Pana-

via F, to In-Ceram Zirconia ceramic may vary depending on the surface treatment methods employed and the aging conditions (thermal cycling/water storage). Significant difference was observed between the group treated with Al$_2$O$_3$ particles submitted to thermal cycling and water storage (Gr2) compared to that of the group tested immediately after sectioning (Gr1). On the other hand, storage and thermal cycling did not significantly affect bond strength of the groups conditioned using tribochemical silica coating (Gr4 and Gr6). These results are in compliance with some previous
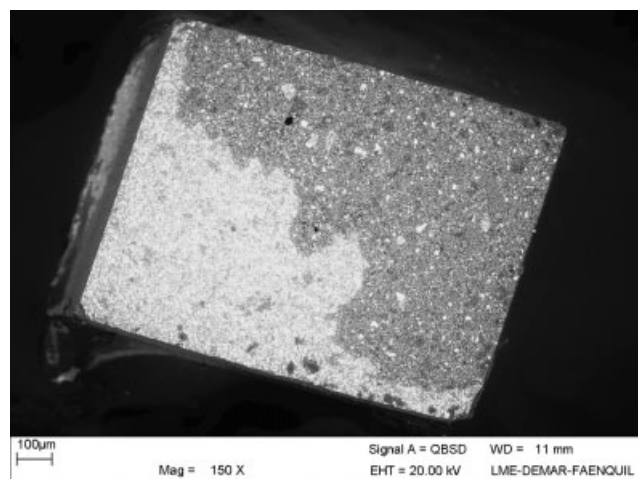
Figure 3. Representative micrographics of the mixed failures.

reports where this finding was attributed to the chemical bonding between the deposited silica, silane coupling agent, and the resin material[12,15,31,38,46,47] as well as the chemical adhesion of the MDP monomer-based cement to zirconia, aluminum, and silica oxides.[16,17,25–29,30–32,34] Even though previous studies suggested surface treatment with Al_2O_3 and the use of resin cement with MDP as the most favorable method to obtain durable bond to reinforced ceramics,[25,27,29,32,40] the present study demonstrated the silica coating being more effective to achieve durable bonding of the resin cement to glass-infiltrated zirconia ceramic.

In laboratory and chairside tribochemical silica coating systems (Rocatec and CoJet), the surface is initially covered with silica particles followed by the application of an MPS silane coupling agent (3-methacryloxypropyltrimethoxy silane in ethanol). The silane molecules react with water forming silanol groups ($-Si-OH$) from methacryloxy groups ($-Si-O-CH_3$). Silanol groups react with the silica deposited on the material surface to form a siloxane network ($-Si-O-Si-O-$). The monomeric ends of the silane react with the methacrylate groups of the resin material.[36,38] Thus, the bonding process between the silica-coated ceramic surface and resin cement occurs through a chemical process between the silica on the surface and the silane coupling agent (tribochemical silica coating).

Stability of the bond strength achieved by tribochemical silica coating compared to the treatment with Al_2O_3 is related to the fact that chemical adhesion between silica and silane created after silica coating is chemically more stable and undergo less hydrolysis compared to bonding between alumina and silane.[12,15,38] Another explanation might be the fact that the silica-coated surface is chemically more reactive to phosphate monomers of the resin cement than the surface conditioned with Al_2O_3.

The process of silica coating on the surface of acid-resistant ceramics is based on the chemical concept experienced in acid-sensitive ceramics. The 110- (Rocatec) and 30-μm (CoJet) Al_2O_3 particles covered with silica are blasted on the surface with which the surface becomes artificially silicatized.[35] After this process, the mechanism of chemical adhesion is similar to that of acid-sensitive ceramics. In fact the ceramic In-Ceram Zirconia is a two phase material with the high crystalline content and 20% glass phase. When alumina or zirconia ceramics are glass infiltrated, they are melted together at high temperatures to form a ceramic matrix. The chemical components of the ceramics (traces such as Li_2O, Na_2O, K_2O, CaO, MgO) are then bound to each other by strong covalent bonds with hydroxyl groups at the surface of the ceramic material. When the surface is air abraded, this would generate more hydroxyl groups on the surface and also enhance the micromechanical retention. Furthermore, the methoxy groups of silane would react with water to form silanol groups that in turn will react with the surface hydroxyl groups to form siloxane network. Amphoteric alumina in the ceramic matrix could form chemical adhesion, covalent bridges, through its surface hydroxyl groups with hydrolyzed silanol groups of the silane: $-Al-O-Si-$.[15] Even under controlled conditions, however, there could be islands of the substrate exposed after air-abrasion. Energy dispersive X-ray spectroscopy (EDS) analysis results also revealed increase in silica content but also showed traces of the substrate material.[48] In this study, no EDS analysis was performed therefore the amount of silica available on the substrate after air abrasion is not known. Our ongoing studies (unpublished data) always showed some amount of silica on two or three phase ceramics. In principle, the presence of the glassy phase in ceramics favors better siloxane bonds

TABLE III. Number of Specimens and Percentage (%) of Failure Types

Groups	Total Number of Specimens	Type of Failure[a]			
		ADHES	Mix	COHES-cem	COHES-cer
Gr1	50 (100%)	10 (20%)	40 (80%)	0	0
Gr2	50 (100%)	27 (54%)	23 (46%)	0	0
Gr3	50 (100%)	5 (10%)	45 (90%)	0	0
Gr4	50 (100%)	4 (8%)	46 (92%)	0	0
Gr5	50 (100%)	7 (14%)	43 (86%)	0	0
Gr6	50 (100%)	6 (12%)	44 (88%)	0	0
Total	300 (100%)	55 (18%)	245 (82%)	0	0

[a] Failure between ceramic and cement (ADHES); cohesive failure of the cement (COHES-cem); cohesive failure of ceramic (COHES-cer); cohesive failure of cement and ceramic (MIX).

and possible amounts of silica available in the glassy matrix of the ceramic could have contributed to bonding mechanism. One other assumption could be that the particle deposition and thereby their retention on the glass surface could be more favorable compared to their impaction on the harder ceramic surfaces such as alumina or zirconia.

Adhesion of resin cements containing MDP phosphate monomers to acid-resistant ceramics has been observed in several studies with high and durable bonding to glass-infiltrated alumina–zirconia ceramics,[33,12] Y-TZP ceramics[25,27,32,40,49] and densely sintered alumina ceramics.[28,29,50] The present study partially agrees with these previous studies, since it was observed that combination of cement containing MDP monomer and air-abrasion with Al_2O_3 did not yield stable bond strength compared to silica coating. Similar to the present study, Kern and Thompson[33] demonstrated stable bonding to a glass-infiltrated alumina ceramic by silica coating in combination with bis-GMA cement. When Al_2O_3 was combined with MDP-based resin cement, the resins obtained were less stable as in this present study (Gr2).[12] Özcan and Vallittu[15] also observed stable results with surface silica coating of a glass-infiltrated alumina–zirconia ceramic luted with the cement without MDP monomer.

Besides the investigation of different acid-resistant ceramics, there is an important difference between the present study and some previous reports.[26–29,32,40] In these previous studies, authors observed that stable bonding after long periods of water storage and thermal cycling may only be achieved by combining Al_2O_3 air-abrasion and resin cements that contain MDP, while in these studies, combination of silica coating and bis-GMA-based resin cements without MDP monomer did not provide durable bond strength. Within this context, the authors concluded that the durability of bonding of resin cements with MDP occurred due to the chemical interaction of ester phosphate groups to zirconia and aluminum oxides in the ceramic. On the other hand, the present study evaluated and confirmed the assumption that combined use of a resin cement containing MDP and silica coating may generate more stable bonding than utilization of the same cement and air abrasion with Al_2O_3. Obviously, it should be considered that these previous studies[25,27–29,32,40] evaluated bonding to compact ceramics without glass stage, whereas the present study was conducted on an alumina–zirconia ceramic with glass stage. In other studies[25,27,40] durability of bonding to compact acid-resistant ceramics without glass phase (Y-TZP ceramics) was evaluated and it was observed that silica coating followed by the use of a resin cement without MDP did not provide stable bonding most probably due to the water sorption of the hydrophobic radical groups of the functional monomers in MDP.[32] It was observed that under the same experimental condition, bond strength was less durable and lower than the combination of Al_2O_3 surface treatment and the use of cement with MDP monomer.[28,29] Durable bonding was obtained by this treatment for glass-infiltrated alumina–zirconia ceramics (containing 20% of glass phase). Thus, despite utilization of acid-resistant ceramics, possibly the presence of a glass phase in acid-resistant ceramics may be a determining factor on the stability of bonding.

With regard to analysis of the fracture mode in the present study, it was observed that silica-coated specimens presented predominantly mixed failures, even in those submitted to long water storage and thermal cycling. The aged specimens air-abraded with Al_2O_3 on the other hand, displayed more frequently adhesive failures between ceramic and cement. This finding possibly represents the strong adhesion of cement to the silica-coated ceramic surface while adhesive failures in Gr2 may indicate higher susceptibility to hydrolytic degradation.[41] Comparison of results at the initial period (dry testing) did not reveal any significant difference between groups submitted to silica coating (Gr3 and Gr5) and the group abraded with Al_2O_3 (Gr1). It should be considered that the lack of significant difference between the three groups at dry conditions (groups 1, 3, and 5) reinforces the need to evaluate the durability of bonding to acid-resistant ceramics, since one of the groups after Al_2O_3 treatment, in the aged condition, presented significant reduction in bond strength compared to the other two treatments (Rocatec and CoJet).

From the clinical standpoint, no longitudinal clinical study has established the best surface treatment of acid-resistant ceramics so far. Considering the results of *in vitro* studies, silica coating on the surface of these ceramics seems to be the treatment of choice. This is particularly true for glass-infiltrated alumina–zirconia ceramics. However, bonding seems to be more unstable for compact acid-resistant ceramics without glass stage, such as densely sinterized alumina or zirconia ceramics and Y-TZP ceramics. Currently, there is a tendency toward utilization of compact Y-TZP ceramics industrially processed and prepared in CAD–CAM equipments, due to their mechanical, chemical and physical properties. However, as previously mentioned, the bonding process is still critical to Y-TZP ceramics and further studies should be conducted for optimization of bonding.

Clinical studies should be performed to establish the best surface treatment protocol for acid-resistant ceramics, especially for clinical situations where adhesion plays a fundamental role in retention of all-ceramic restorations.[51–53] such as resin-bonded fixed-partial-dentures in the anterior regions and inlay-retained fixed-partial-dentures or overlays in the posterior area of the mouth.

CONCLUSIONS

Both laboratory and chairside silica coating followed by silanization showed durable bond strength of the resin cement to glass-infiltrated zirconia–alumina ceramic. After aging, air abrasion with 110-μm Al_2O_3 particles and silanization showed the largest decrease.

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