

Bond Strength of a Resin Cement to High-alumina and Zirconia-reinforced Ceramics: The Effect of Surface Conditioning

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Purpose: The aim of this study was to evaluate the effect of two surface conditioning methods on the microtensile bond strength of a resin cement to three high-strength core ceramics: high alumina-based (In-Ceram Alumina, Procera AllCeram) and zirconia-reinforced alumina-based (In-Ceram Zirconia) ceramics.

Materials and Methods: Ten blocks (5 x 6 x 8 mm) of In-Ceram Alumina (AL), In-Ceram Zirconia (ZR), and Procera (PR) ceramics were fabricated according to each manufacturer's instructions and duplicated in composite. The specimens were assigned to one of the two following treatment conditions: (1) airborne particle abrasion with 110- μm Al_2O_3 particles + silanization, (2) silica coating with 30 μm SiO_x particles (CoJet, 3M ESPE) + silanization. Each ceramic block was duplicated in composite resin (W3D-Master, Wilcos, Petrópolis, RJ, Brazil) using a mold made out of silicon impression material. Composite resin layers were incrementally condensed into the mold to fill up the mold and each layer was light polymerized for 40 s. The composite blocks were bonded to the surface-conditioned ceramic blocks using a resin cement system (Panavia F, Kuraray, Okayama, Japan). One composite resin block was fabricated for each ceramic block. The ceramic-composite was stored at 37°C in distilled water for 7 days prior to bond tests. The blocks were cut under water cooling to produce bar specimens ($n = 30$) with a bonding area of approximately 0.6 mm². The bond strength tests were performed in a universal testing machine (crosshead speed: 1 mm/min). Bond strength values were statistically analyzed using two-way ANOVA and Tukey's test (≤ 0.05).

Results: Silica coating with silanization increased the bond strength significantly for all three high-strength ceramics (18.5 to 31.2 MPa) compared to that of airborne particle abrasion with 110- μm Al_2O_3 (12.7-17.3 MPa) (ANOVA, $p < 0.05$). PR exhibited the lowest bond strengths after both Al_2O_3 and silica coating (12.7 and 18.5 MPa, respectively).

Conclusion: Conditioning the high-strength ceramic surfaces with silica coating and silanization provided higher bond strengths of the resin cement than with airborne particle abrasion with 110- μm Al_2O_3 and silanization.

Keywords: bond strength, ceramics, microtensile test, silica coating, surface conditioning methods.

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Early types of metal-free ceramics did not enjoy success in dentistry, especially in the posterior region.³³ However, with the introduction of high-strength ceramic systems to the dental market almost two decades ago, ceramic restorations became viable options for posterior teeth.

High-strength ceramic systems are expected to provide restorations with sufficient mechanical strength to resist occlusal forces as well as deliver more esthetic results than conventional metal-fused-to-ceramic restorations. New ceramic systems include ceramic cores reinforced through dispersion with leucite,^{10,16,27,29,32} glass-infiltrated sintered alumina,^{5,28} and the use of high-purity alumina²¹ or zirconium oxide.³⁰

Bonding of ceramic to dental tissue is based on the adhesion of luting cement to the ceramic substrate, together

with the adhesion of luting cement to enamel and/or dentin. Previous investigations revealed that most clinical failures initiate from the cementation or internal surfaces. Failure rates due to high-strength ceramic fractures have been reported to range between 2.3% and 8%.^{2,20,25} Therefore, the integrity of the luting cement to ceramic surfaces plays a major role in the longevity of the restoration; the failures originating from cementation surfaces identified the need for a reliable conditioning method to strengthen this critical area.

In order to enhance the bond strength of luting cement to the ceramic surface, a number of techniques have been reported that mechanically facilitate resin-ceramic bonding.^{13,17} Etching the inner surface of a restoration with hydrofluoric acid followed by the application of a silane coupling agent is a well-known and recommended method to increase bond strength. Although hydrofluoric acid is effective in roughening feldspathic ceramic for bonding composite resin, neither etching with these solutions nor adding silane resulted in an adequate resin bond, particularly to high-alumina^{1,6,7} or zirconia-reinforced ceramics,^{1,18,34} since such ceramics do not contain a silicon oxide phase. For this reason, special conditioning systems are indicated for these ceramics.

Modern surface conditioning methods require airborne particle abrasion of the non-silica based ceramic surfaces before bonding in order to achieve high bond strength. One such system is silica coating. In this technique, the surfaces are air abraded with alumina particles coated with silica.^{15,19} The blasting pressure results in the embedding of silica particles in the ceramic surface, rendering the silica-modified surface chemically more reactive to the resin through silane coupling agents. Silane molecules, after being hydrolyzed to silanol, can form a polysiloxane network or hydroxyl groups covering the silica surface. Monomeric ends of the silane molecules react with the methacrylate groups of the adhesive resins by the free-radical polymerization process.

The air abrasion systems are based on air-particle abrasion with different particle sizes (250, 110 and 30 μm).³¹ The abrasive process removes loose contaminated layers, and the roughened surface provides some degree of mechanical interlocking or "keying" with the adhesive. It can be argued that the increased roughness also forms a larger surface area for the bond. While these mechanisms explain some of the general characteristics of adhesion to roughened surfaces, they may also introduce physicochemical changes that affect surface energy and wettability. However, there is limited knowledge as to whether micromechanical retention using large or small particle size increases resin bond to high-strength ceramics of different microstructures and chemical compositions.

The aim of this study, therefore, was to evaluate the effect of two surface conditioning methods based on airborne particle abrasion, employing two types of sand particles, on the microtensile bond strength of the resin cement to three high-strength core ceramics: two high-alumina-based ceramics and one zirconia-reinforced alumina-based ceramic.

MATERIALS AND METHODS

Ten blocks (5 x 6 x 8 mm) of high-alumina-based (In-Ceram Alumina [AL; Vita Zahnfabrik, Bad Säckingen, Germany], Procera AllCeram [PR; Nobel Biocare, Stockholm, Sweden]) and zirconia-reinforced alumina-based (In-Ceram Zirconia [ZR; Vita Zahnfabrik]) ceramics were fabricated according to each manufacturer's instructions. Ceramic surfaces were finished using a silicon carbide abrasive up to 1200 grit (3M, St Paul, MN, USA) in a polishing machine (Labpol 8-12, Ex-tec, Enfield, CT, USA), cleaned for 10 min in an ultrasonic bath (Quantrex 90, L&R Ultrasonics, Kearny, NJ, USA) containing ethyl acetate, and air dried. Each ceramic block was duplicated in composite resin (W3D Master, Wilcos, Petrópolis, Brazil) using a mold made out of silicon impression material (Elite HD, Zhermack, Badia Polesine, Italy). Composite resin layers were incrementally condensed into the mold to fill up the mold, and each layer was light polymerized for 40 s (XL 3000, 3M/ESPE, St Paul, MN, USA; light output: 500 mW/cm²). One composite resin block was fabricated for each ceramic block.

Surface Conditioning Methods

The specimens were assigned to one of the two following treatment conditions:

- Grit blasting (GB): In this group, airborne particle abrasion was performed using 110- μm grain sized Al_2O_3 particles using an intraoral air abrasion device (Micro-Etcher, Danville, San Ramon, CA, USA) at a pressure of 2.8 bars from a distance of ca 10 mm for 15 s.
- Silica coating (SC): Silica coating was achieved using an intraoral air abrasion device (Dento-Prep, RØNVIG A/S, Daugaard, Denmark) filled with CoJet-Sand (30- μm SiO_x particles) (3M/ESPE, St Paul, MN, USA) under the same conditions as for GB.

Following both surface conditioning methods, the remaining sand particles were gently blown off with air, and silane coupling agent (ESPE-Sil, 3M/ESPE, Seefeld, Germany) was applied and allowed to evaporate for 5 min.

Bonding Procedure and Specimen Preparation

Each conditioned ceramic block was bonded to a composite block under a load of 750 g using a resin cement system (Panavia F, Kuraray, Okayama, Japan) according to the manufacturer's instructions. The excess resin cement was removed by means of a brush. The resin cement was then light polymerized (XL 3000) for 40 s from each direction. Oxy-guard was applied on the cement layer for 10 min. The blocks (Fig 1a) were washed with air-water spray and stored in distilled water at 37°C for 7 days prior to bond testing.

The blocks were then bonded with cyanoacrylate glue (Super Bonder Gel, Loctite Ltd, São Paulo, Brazil) to a metal base that was coupled to a cutting machine (Fig 1b). Slices were obtained using a slow-speed diamond wheel saw (KG

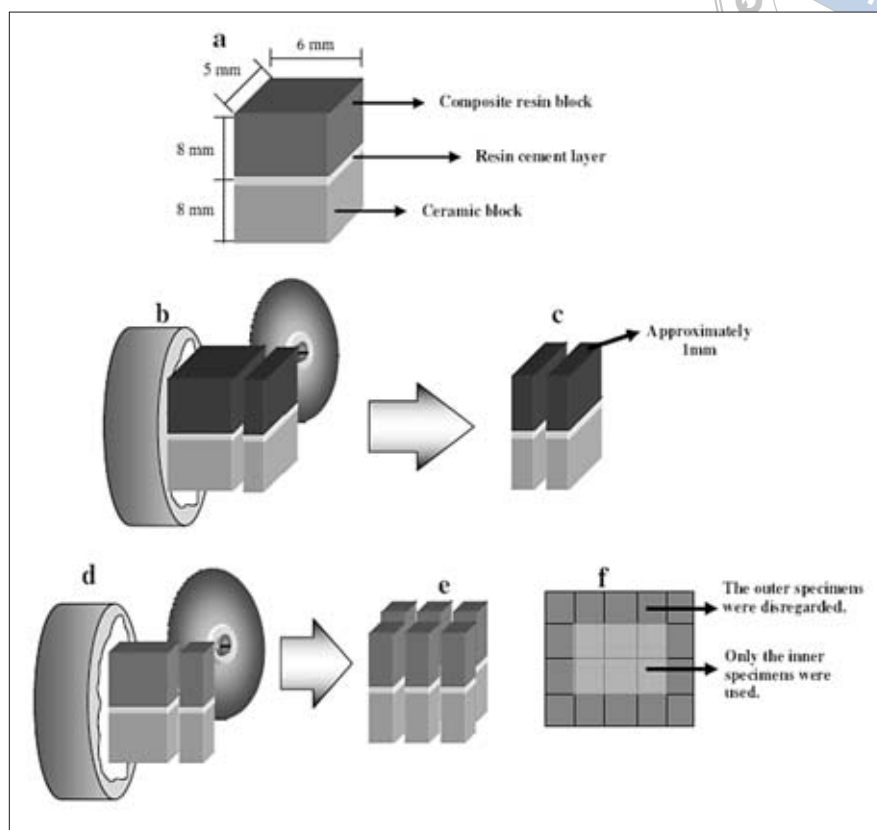


Fig 1a-f

a) Cemented ceramic and composite blocks. b) The blocks bonded to a metal base that was coupled to a cutting machine. c) Two slices were obtained per block. d) The slices were rotated 90 degrees and bonded onto the metal base again for further cutting. e) Six non-trimmed bar specimens (approximately 0.6 x 0.6 x 16 mm) were obtained per block. f) Note that the outer bar specimens were disregarded.

Sorensen, Barueri, Brazil) under water cooling. The outer slices were discarded to prevent the results being influenced by either an excess or insufficient amount of resin cement at the interface. Two slices (0.8 ± 0.1 mm thick) were obtained per block (Fig 1c). The slices were rotated 90 degrees and bonded onto the metal base again (Fig 1d). The outer bar specimens were also discarded for the same reasons described above. Six nontrimmed bar specimens with dimensions of approximately 0.6 x 0.6 x 16 mm were obtained per block ($n = 30$) (Figs 1e and 1f).

Microtensile Bond Strength Test

The bar specimens were glued parallel to the long axis of an adapted caliper using cyanoacrylate glue (Figs 2a and 2b). This apparatus was coupled to the universal testing machine (EMIC DL-1000, EMIC, São José dos Pinhais, Brazil), and the specimens were loaded in tension to failure at a crosshead speed of 1 mm/min.

Bond strength values were calculated using the formula, $\sigma = L/A$, where "L" is the load at failure and "A" is the adhesive area measured with a digital caliper (Mitutoyo, Tokyo, Japan) prior to the tests.

Additional ceramic specimens were conditioned using the two surface conditioning methods in order to observe

the topographic surface changes under SEM (JEOL JSM-T330A, JEOL, Tokyo, Japan).

Statistical Analysis

Statistical analysis was performed using SAS System for Windows, version 8.02/2001 (Cary, NC, USA). The means of each group were analyzed by 2-way ANOVA, with microtensile bond strength as the dependent variable, and the surface conditioning methods and the ceramic types as the independent factors. P values less than 0.05 were considered to be statistically significant in all tests. Multiple comparisons were made with Tukey's adjustment test. Furthermore, one-way ANOVA was used to determine the significant differences between surface conditioning methods.

RESULTS

The results of the microtensile bond strength tests for two surface conditioning methods and three ceramics are presented in Table 1.

ANOVA showed a significant influence of the surface conditioning methods and the ceramic type ($p < 0.05$) (Tables 2 and 3). No significant interactions were found ($p > 0.05$).

Table 1 Microtensile bond strength (MPa) of the resin luting cement to In-Ceram Alumina (AL), Procera AllCeram (PR), and In-Ceram Zirconia (ZR) after grit blasting (GB) or silica coating (SC)

Groups	σ^* (MPa) (SD)
1. AL-GB	17.3 ^d (2.6)
2. AL-SC	31.2 ^a (4.3)
3. ZR-GB	15.1 ^{de} (5.3)
4. ZR-SC	26.8 ^b (7.4)
5. PR-GB	12.7 ^e (2.6)
6. PR-SC	18.5 ^c (4.7)

*The same superscript letters indicate no significant differences between the ceramic/surface conditioning combinations ($p > 0.05$). SD= standard deviation.

Table 2 Microtensile bond strength (MPa) of the resin luting cement by surface conditioning factor

Conditioning	σ^* (MPa) (SD)
GB	15.1 ^b (4.2)
SC	25.5 ^a (7.7)

*Different superscript letters indicate significant differences between the ceramic/surface conditioning combinations ($p < 0.05$). SD= standard deviation.

Table 3 Microtensile bond strength (MPa) of the resin luting cement according to ceramic material

Ceramic	σ^* (MPa) (SD)
AL	24.3 ^a (7.9)
ZR	21.0 ^a (8.7)
PR	15.6 ^b (4.8)

*The same superscript letter indicates no significant differences between the ceramic-surface conditioning combinations ($p > 0.05$). SD= standard deviation.

The highest microtensile bond strength was obtained with AL ceramic after silica coating (31.2 MPa). The lowest bond strengths were found with PR ceramic after grit blasting (12.7 MPa). While no significant difference in bond strength ($p > 0.05$) was observed between AL and ZR (24.3 and 21.0 MPa, respectively) regardless of the conditioning method, the lowest bond strengths were found with PR (15.6 MPa) ($p < 0.05$).

Complementary to the bond strength tests, SEM analysis at 2000X magnification revealed that 110- μm Al_2O_3 particles partially penetrated the substrate surfaces (Figs 3a to 3e), and after silica coating, the ceramic surfaces were covered with abundant sand particles (Figs 3b to 3f). The observed failures were exclusively at the adhesive zone.

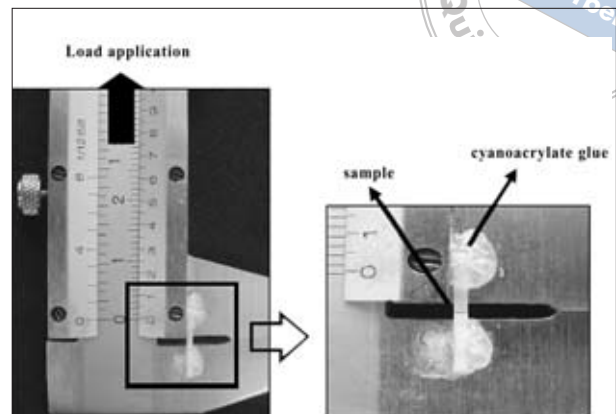


Fig 2 The bar specimens were glued parallel to the long axis of an adapted caliper using cyanoacrylate prior to microtensile bond testing.

DISCUSSION

Air-particle abrasion is a prerequisite for achieving sufficient bond strength between the resins and high-strength ceramics that are reinforced either with alumina or zirconia.^{17,18} In this study, roughening high-alumina and zirconia-reinforced ceramic surfaces with air particle abrasion and applying silanes prior to cementation provided high bond strengths, and silica coating followed by silanization evidently enhanced the bond between the ceramic surfaces and the luting cement. A particular increase was observed for AL and ZR ceramics.

The silica layer left by silica coating on the ceramic surface provides a basis for silane to react. In the ceramic-resin bond, silane functions as a coupling agent, which adsorbs onto and alters the surface of the ceramic, thereby facilitating interaction.^{7,31} When a ceramic chemically exhibits silicon and oxygen, then a siloxane bond will be achieved, as these represent the binding sites for the coupling agent to the ceramic surface. Since silane coupling agents do not bond well to alumina, the bond strengths of resin composite to the ceramic could be affected.²⁴ However, 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 of compounds such as Li_2O , Na_2O , K_2O , CaO , MgO) are then bonded to each other by strong covalent bonds with hydroxyl groups at the surface of the ceramic material.²³ 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, which in turn will react with the surface hydroxyl groups to form a 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: $-\text{Al}-\text{O}-\text{Si}-$.¹

In principle, the presence of the glassy phase in ceramics facilitates better siloxane bonds. The silanol groups then react further to form a siloxane ($-\text{Si}-\text{O}-\text{Si}-\text{O}-$) network with the silica on the surface. Both In-Ceram ceramic systems tested in this study – In-Ceram Alumina (AL) and In-Ceram Zirconia

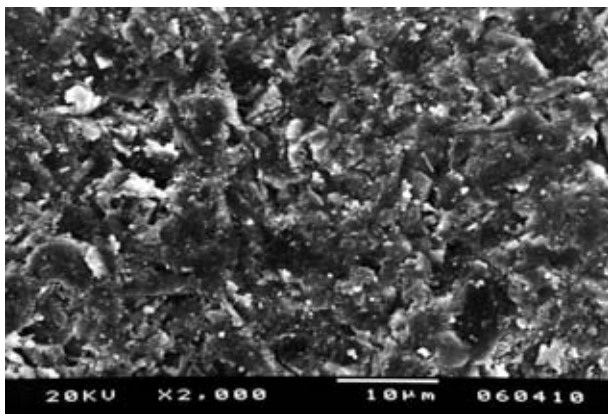


Fig 3a SEM image of the conditioned ceramic surface for AL-GB. Note that 110- μm Al_2O_3 particles penetrated the substrate surfaces partially.

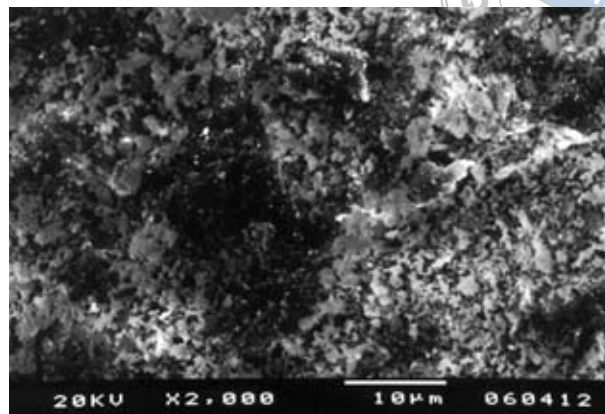


Fig 3b SEM image of the conditioned ceramic surface for AL-SC. Note that after silica coating, the ceramic surfaces were covered with abundant sand particles.

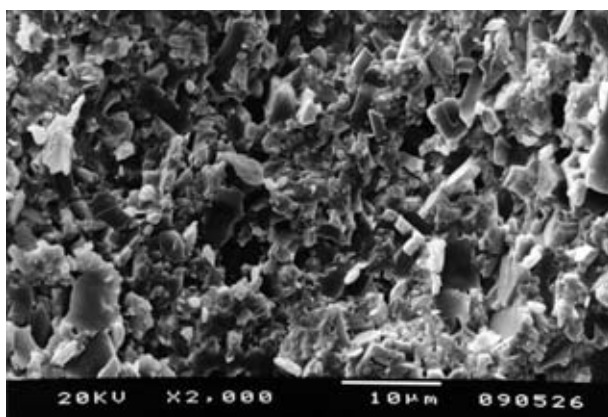


Fig 3c SEM image of the conditioned ceramic surface for ZR-GB. Note that 110- μm Al_2O_3 particles penetrated the substrate surfaces partially.

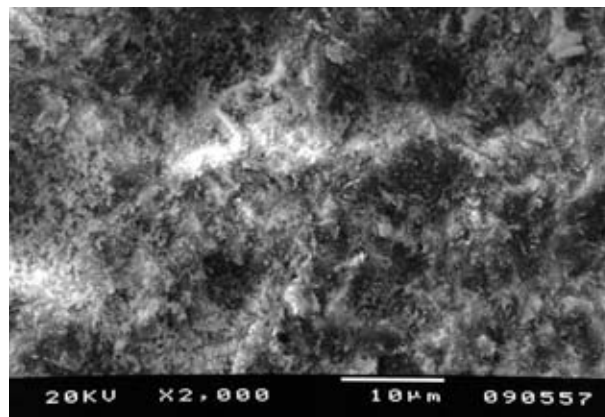


Fig 3d SEM image of the conditioned ceramic surface for ZR-SC. Note that after silica coating, the ceramic surfaces were covered with abundant sand particles.

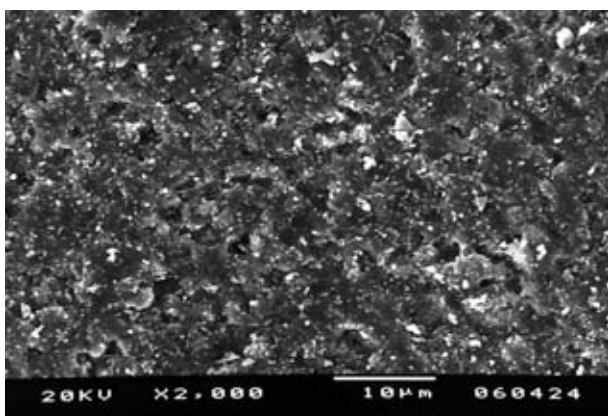


Fig 3e SEM image of the conditioned ceramic surface for PR-GB. Note that 110- μm grain sized Al_2O_3 particles penetrated the substrate surfaces partially.

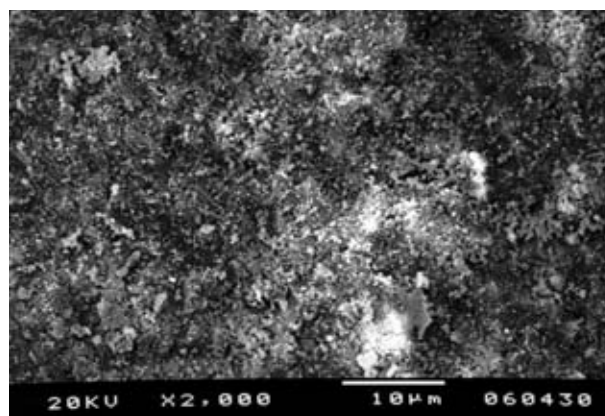


Fig 3f SEM image of the conditioned ceramic surface for PR-SC (original magnification x 2000). Note that after silica coating, the ceramic surfaces were covered with abundant sand particles.



(ZR) – are glass infiltrated. Most probably, the glass infiltration facilitated better silane bonding, and therefore, higher bond strength values were obtained for these ceramics. Despite the fact that both In-Ceram Alumina (AL) and Procera AllCeram (PR) are considered aluminous ceramics,²⁴ Procera (PR) showed significantly lower values than those of the other two ceramics. This could be attributed to its 99.9% alumina content, whereas the alumina content of In-Ceram Alumina (AL) is 80%.^{21,22} These findings are in agreement with the study of Özcan and Vallittu,²³ although a different experimental setup was used in which a bis-GMA-based resin cement and shear bond test were employed.

Material selection and clinical recommendations on resin bonding are based on mechanical laboratory tests that show great variability in materials and methods. One of the most common testing methods is the shear bond test; however, the specific force application in shear testing may cause cohesive failure in the substrate, possibly leading to erroneous interpretation of the data, while stress distribution in microtensile tests was reported to be more homogenous.^{4,8,9,11} For this reason, the microtensile test was employed in this study; nevertheless, the ranking of ceramic-cement performance was comparable with the study of Özcan and Vallittu.²³ Because the microtensile test is performed by cementing a block of composite to the conditioned ceramic substrates^{18,24} and not – as in vivo – by cementing the ceramic to dental tissue, the application of its results to the clinical situation may be limited. This issue is under investigation in our laboratories.

Some studies, on the other hand, have evaluated ceramics with different microstructures, reporting that densely sintered alumina ceramics, such as PR, are compact materials, difficult to grit blast.^{13,15} However, regardless of the ceramic studied, the silica coating system with 30- μm SiO_x particles produced statistically higher mean bond strength values than with grit blasting using 110- μm Al_2O_3 particles. One would expect higher surface roughness created with larger particle size and thus higher micromechanical retention, but this was not achieved in that study. One reason for this could be associated with the phenomenon of less wettability and contact angle between the silane coupling agent and the deep grooves on the ceramic surfaces caused by grit blasting.^{3,15,26} One other reason may be that particle deposition mechanisms differ depending on the substrate characteristics, particle composition, size distribution, quantity and morphology.

The results of this study together with some other studies reveal good adhesion of silica particles in the vitreous phases of the glass-infiltrated zirconia ceramics.²⁸ In a previous study,²¹ a significant increase of silica on the surface of the In-Ceram ceramic (15.8 wt% to 19.7 wt%) was detected after blasting with Rocatec-Plus (SiO_x), compared with the samples blasted only with Al_2O_3 particles, suggesting better bond strength between the In-Ceram ceramic and the resin cements due to the increase of silica content and the interaction with the silane agent. Energy Dispersive X-ray Spectroscopy (EDS) analysis results also revealed an increase in both silica and alumina content on the zirconia substrates.¹⁴ Tribochemical coating on glass-infiltrated alumina is surely more effective than on dense alumina ceramic, and it is likely that particle deposition and thereby embedding of silica

is easier on the glass with lower hardness. In contrast, alumina crystals of the dense alumina ceramic present higher hardness, impairing the silica particle penetration.^{14,18}

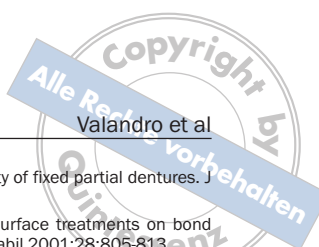
The adhesion of resin cement to ceramic is susceptible to chemical, thermal, and mechanical influences under intraoral conditions. One limitation of this study is the lack of thermocycling or any other long-term aging procedure; for instance, hydrolytic degradation of the cementation interface was not examined after one week of water storage. Decreased bond strength of the cement onto zirconia or high alumina ceramics has been previously reported after water storage.^{1,18} In this study, however, the objective was only to compare the effect of two types of conditioning methods, simulating the possible early failures of the cementation interface of such reinforced ceramics, and to compare solely the cement bond strength to three types of reinforced ceramics. The results obtained in this study agree with those found by Bottino et al,¹² in which the specimens were also stored for 1 week in water. The influence of thermal cycling and long-term water storage are considered in our ongoing studies, in order to find out more about the durability of resin cement-ceramic adhesion.

Although satisfactory bond strength values of resin cement to high-strength ceramics have yet to be determined for clinically successful performance, the bond values obtained for all high-strength ceramics tested in this study could be sufficient with both conditioning methods. In clinical chairside applications, however, air abrasion may have a considerable impact on the marginal areas of the restorations. Clinicians should also consider the possible material loss²⁴ especially at the margins of the restorations, which may lead to ditching when high grain size particles are used during airborne particle abrasion. This finding was also confirmed in the SEM images of ceramic surfaces after conditioning, where 110- μm grain sized Al_2O_3 particles penetrated the substrate surfaces, leaving some areas with no particles evident; in contrast, after silica coating, the ceramic surfaces were covered with abundant sand particles even after air blowing.

The general outcome of this study suggests that relatively recent surface conditioning techniques based on a combination of micromechanical and chemical conditioning should be considered for improved adhesion of cements to high-strength ceramics. More importantly, these methods seem to offset the importance of the varieties of the substrates and therefore may be applicable for a wide range of high-strength ceramics. In the past, the equipment needed to apply these techniques was rather complicated and expensive, but it has recently been simplified for chairside use. Until conditioning methods and the resin composite cements are optimized, developments in the ceramic field are expected to continue to experience failures.

CONCLUSIONS

1. Microtensile bond strengths of phosphate monomer-based resin cement to glass-infiltrated high-alumina and zirconia-reinforced ceramics were significantly higher than to densely sintered high-alumina ceramic with no glass infiltration.



2. Chairside silica coating with 30- μm SiO_x particles followed by silanization increased the bond strengths significantly when compared with 110- μm Al_2O_3 particle grit blasting and silanization.

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Clinical relevance: Chairside silica coating and silanization improve the adhesion of phosphate monomer-based resin cement to reinforced ceramics.

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