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Effect of Surface Treatment on Shear Bond Strength of Zirconia to Human Dentin

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Keywords
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Abstract

Purpose: The effect of surface treatment using tribochemical silica coating/silane coupling on the shear bond strengths of (1) a glass-infiltrated, zirconia-reinforced alumina (In-Ceram Zirconia) and (2) a yttria-stabilized zirconia ceramic (YZ Zirconia) to human dentin was studied.
Materials and Methods: Twelve specimens of each ceramic were randomly assigned to one of three surface treatments: (1) no surface treatment (control group); (2) a chairside tribochemical silica coating/silane coupling system (CoJet group); and (3) a laboratory tribochemical silica coating/silane coupling system (Rocatac group). The mode of failure of each specimen was determined under magnification.
Results: The shear bond strengths (mean $\pm$ SD) of In-Ceram Zirconia of the control, CoJet and Rocatec groups were 5.7 $\pm$ 4.3 MPa, 11.4 $\pm$ 5.4 MPa, and 6.5 $\pm$ 4.8 MPa, respectively. The corresponding figures for YZ Zirconia were 8.2 $\pm$ 5.4 MPa, 9.8 $\pm$ 5.4 MPa, and 7.8 $\pm$ 4.7 MPa. Two-way ANOVA revealed significant differences in bond strength due to the difference in surface treatment ($p = 0.02$), but the bond strengths between the two ceramics were not significantly different ($p = 0.56$). *Post hoc* tests showed that In-Ceram Zirconia treated with CoJet had significantly higher shear bond strengths than those untreated ($p < 0.05$) or treated with Rocatec ($p < 0.05$). Surface treatment did not affect the shear bond strength of YZ Zirconia significantly ($p > 0.05$).
Conclusion: The bonding of In-Ceram Zirconia can be improved by the chairside surface treatment system.

The superiority in mechanical strength of zirconia-reinforced dental ceramics over conventional all-ceramic materials prompted a wider availability of such ceramics on the market.[1-4] The more popular of such zirconia-reinforced dental ceramics include a glass-infiltrated, zirconia-reinforced alumina (In-Ceram Zirconia, VITA Zahnfabrik, Bad Sackingen, Germany) and machined-milled, yttria-stabilized zirconia ceramics (Y-TZP) such as Cercon (Cercon Smart Ceramic System, Dentsply Intl, York, PA), LAVA (LAVA system, 3M ESPE, St. Paul, MN), DC-Zirkon (Precident-DCS system, Allschwil, Switzerland), and YZ Zirconia (Vita Zahnfabrik).

The clinical use of zirconia-reinforced dental ceramics poses a different challenge when it relates to the adhesive bonding of such materials. Traditional methods of prebonding surface treatments such as hydrofluoric acid (HF) etching or airborne-particle-abrasion (airbrasion) have only limited success. HF preferentially etches the glassy phase and phase boundaries, thereby promoting the micromechanical retention of luting resin onto the ceramic surface with surface irregularities created;[5-8] however, HF is largely ineffective against ceramics reinforced with alumina or zirconia, which are chemically resistant to HF etching.[8-10] The surface topography of a zirconia-reinforced alumina treated with 9.5% HF for 90 seconds remains unchanged.[10]

Airbrasion with alumina particles can alter the surface topography of zirconia-reinforced ceramics.[10] There are reports of improved bond strength of airbraded machinable feldspathic ceramics or glass-infiltrated alumina ceramics[11,12]; however, this procedure is usually ineffective in improving the bond strength of resin to ceramics with high alumina or zirconia content.[13-17]

Bonding between a ceramic surface and a resin luting agent may be facilitated by a silane coupling agent. The alkoxy groups (RO_3Si-) of the silane molecule react with water to form silanol groups (SiOH). The silanol groups further react with hydroxyl (OH) groups on a ceramic surface with available Si and O to form siloxane (-Si-O-Si-O-) covalent bonds. The monomeric end of the silane molecules reacts with the methacrylate groups of resin luting agent. Thus, a strong network of siloxane covalent bonds is formed between the ceramic surface and methacrylate resin.[18,19] The silane coupling agent in itself does not improve bonding of high-purity alumina or zirconia because such ceramics do not have a silica base.[8,13,20-22] Alternatively, the glassy phase of a glass-infiltrated alumina (In-Ceram Alumina) or zirconia-reinforced alumina (In-Ceram Zirconia) provides the necessary silicate groups for interaction with silane coupling agents and thus improves their bonding.[14] It is also evident that airbrasion with Al_2O_3 enhances the action of silane by generating more hydroxyl groups on the ceramic surface to react with the silanol groups of the silane. This mode of bonding enhancement is in addition to the micromechanical retention that airborne-particle abrasion provides.[18,19]

Another method of improving silane coupling is tribochemical surface conditioning of the ceramics by silica coating. One such system uses a three-step laboratory procedure (Rocatec system, 3M ESPE, Seefeld, Germany). The ceramic surface is first airbraded with Al_2O_3 particles to remove contaminants and provide microroughness. Then, the surface is airbraded with a silica-modified Al_2O_3 particle to establish the tribochemical coating of silicon-dioxide. Finally, the ceramic surface is silanized and is ready for bonding with resin luting agent. Another such system employs a two-step chairside procedure that tribochemically coats the ceramic surface by airbrading it with a silica-modified Al_2O_3 particle before silanization. Tribochemical coating, together with silanization, has been shown to be effective in improving the bond strength of glass-infiltrated alumina, glass-infiltrated zirconia-reinforced alumina, a high purity alumina (Procera, Nobel Biocare, Stockholm, Sweden) and a yttria-stabilized zirconia ceramic (Cercon).[14,18] The improved chemical and micromechanical surface of the coated ceramics was evident from an electron dispersive spectroscopic analysis showing that the silicon surface content of a glass-infiltrated, zirconia-reinforced alumina (In-Ceram Zirconia) increased from 1.25% to 2.2% together with a quantifiable increase in microroughness.[10] Conversely, at least two studies showed that tribochemical silica coating did not improve the bonding of a yttria-stabilized zirconia ceramic (Cercon) to composite blocks.[23,24]

To the authors' knowledge, published studies on the bonding characteristics of zirconia-reinforced dental ceramics used composite resin block[10,18,19,22-24] or stainless steel[25] as the bonding substrate. The relevance of these studies should be limited to the bonding of such ceramics to a composite resin core or stainless steel. This contrasts with the more usual clinical application when all-ceramic crowns or veneers are bonded to enamel or dentin. Thus, the purpose of the present study was to test the hypothesis that surface treatments do not affect the shear bond strength of a glass-infiltrated, zirconia-reinforced alumina (In-Ceram Zirconia) and a machined-milled, yttria-stabilized zirconia ceramic (YZ Zirconia) to human dentin using a resin luting agent.

Materials and methods

The shear bond strength test was carried out following the guidelines of ISO Technical Specification 11405 (2003) with the use of cementation and shear test jigs.[26]

Preparation of specimen before surface treatment and bonding

A glass-infiltrated, zirconia-reinforced alumina (In-Ceram Zirconia) and a machined milled, yttria-stabilized zirconia ceramic (YZ Zirconia) were selected for the study. Thirty-six ceramic rods (3 mm diameter, 10 mm length) of each material were fabricated and supplied by the manufacturer (Vita Zahnfabrik). All-ceramic specimens were supplied dry and were stored in an enclosed environment. Twelve specimens of each material were randomly assigned to one of three surface treatments: (1) no surface treatment (control group); (2) a chairside tribochemical silica coating/silane coupling system (3M ESPE CoJet System Set) (CoJet group); and (3) a laboratory tribochemical silica coating/silane coupling system (3M ESPE Rocatec System) (Rocatac group).

Human permanent molar teeth with post-extraction time between 1 and 6 months were selected and stored in distilled water. Each tooth was partially embedded in an autopolymerizing acrylic resin (ProBase cold-curing denture base material, Ivoclar Vivadent AG, Schaan, Liechtenstein) using a powder:liquid mixing ratio of 10 g:10 ml. The blocks were immersed in water to dissipate the heat generated during polymerization and were then milled to allow secure mounting on the cementation and shear test jigs. The dentin surfaces for bonding were exposed with a tungsten carbide cutting blade (Micro 100) without perforation into the pulp chamber. The bonding surfaces were finished by hand lapping with 600-grit silicon carbide abrasive paper under running water. The finished dentin surfaces were examined under a 2.5× magnification to ensure that a uniform surface finish was achieved. Care was taken not to contaminate the dentin surface with acrylic resin residues during cutting and polishing. The bonding surface at one end of each ceramic rod was finished by hand on 600-grit silicon carbide abrasive paper under running water and dried with an air syringe for approximately 15 seconds.

Surface treatment and bonding of the ceramic and dentin specimens

The bonding surface of each CoJet specimen was airbraded with silicatized aluminum oxide particles (30 μm) (CoJet Sand) at a pressure of 2.5 bars at a distance of 2 mm to 10 mm for 15 seconds and then air dried. A silane coupling agent (ESPE-SIL, 3M ESPE) was applied onto the surface and allowed to dry for 5 minutes. A resin-bonding agent (Visio Bond, 3M ESPE, St Paul, MN) was then applied and light polymerized for 20 seconds as part of the CoJet manufacturer's protocol.

The bonding surface of each Rocatec specimen was airbraded with 110 μm Al_2O_3 particles (ROCATEC-PRE) at 2.8-bar

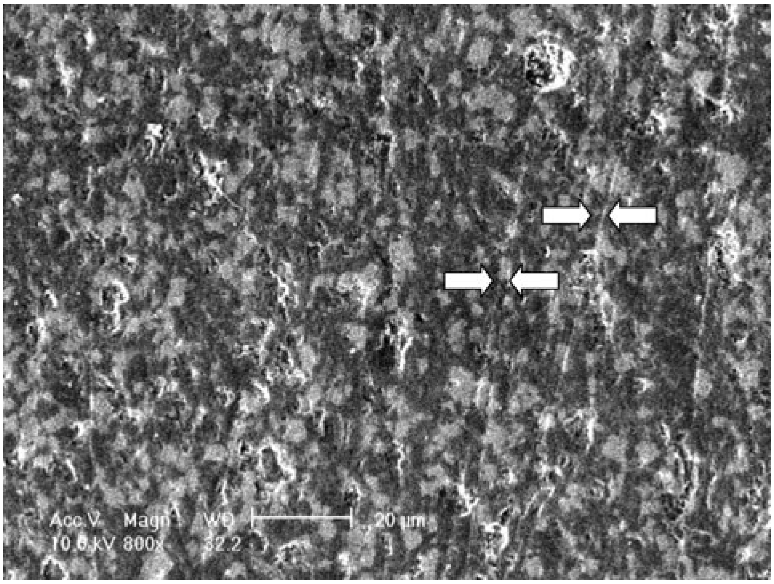

Figure 1 Scanning electron micrograph (800×) of untreated In-Ceram Zirconia. Alumina and Zirconia (lighter color) platelets embedded in a glass matrix (darker color); surface abrasion due to polishing is evident (arrows).

pressure for 10 seconds. Then, 30 μm silicatized Al_2O_3 particles (ROCATEC-PLUS) were applied at 2.8-bar pressure for 13 seconds at a distance of about 10 mm, in accordance with the manufacturer's recommendation. A silane coupling agent (ESPE-SIL) was then applied onto the surface and allowed to dry for 5 minutes. The bonding surface of control specimens did not receive any additional surface treatment.

A phosphate monomer (MDP) containing a resin luting agent (Panavia F 2.0, Kuraray, Co. Ltd., Osaka, Japan) was applied according to the manufacturer's instruction. Each ceramic rod was cemented onto the dentin bonding surface with a cementation jig. A 10 N load was applied and maintained for 3 minutes. The bonded specimens were light-cured for 20 seconds each from two opposite sides.

Shear bond strength testing

All bonded specimens were immersed in distilled water (37 ± 2°C) for 24 hours before being tested for shear bond strength. Each specimen was fixed onto the shear test jig, and the load required to break the bond at a crosshead speed of 0.5 mm/min was measured on a universal testing machine (Instron, Model 1185; Bucks, UK) with a 1000 N load cell. The shear bond strength of each specimen was calculated by dividing the load at failure by the area of bonding surface. The mode of failure of each specimen was determined by inspecting the bonding surfaces of each specimen under optical magnification (9× Handheld loupe, Carl Zeiss AG, Oberkochen, Germany). Failure mode was classified into four types: (1) adhesive failure between resin luting agent and dentin; (2) cohesive failure within resin luting agent; (3) adhesive failure between resin luting agent and ceramics; and (4) mixed mode of failure including (1) and (3) with or without (2). Photographs of representative specimens were taken.

Statistical analysis

Statistical software (SPSS, Version 11.5; SPSS Inc, Chicago, IL) was used for data analysis. The differences in shear bond strength among the six groups of specimens were analyzed with two-way ANOVA according to surface treatments and ceramic materials and Bonferroni *post hoc* tests ($p = 0.05$).

Results

The shear bond strengths (mean ± SD) of In-Ceram Zirconia of the control, CoJet, and Rocatec groups were 5.7 ± 4.3 MPa, 11.4 ± 5.4 MPa, and 6.5 ± 4.8 MPa, respectively. The corresponding figures for YZ Zirconia were 8.2 ± 5.4 MPa, 9.8 ± 5.4 MPa, and 7.8 ± 4.7 MPa. Two-way ANOVA revealed significant differences in bond strength due to the difference in surface treatment ($p = 0.02$), but the bond strengths between the two ceramic types were not significantly different ($p = 0.56$). *Post hoc* tests showed that In-Ceram Zirconia treated with CoJet had significantly higher shear bond strengths than those untreated ($p < 0.05$) or treated with Rocatec ($p < 0.05$), while the control and Rocatec group were not significantly different ($p > 0.05$). For YZ Zirconia, *post hoc* tests showed no significant difference between the CoJet and control ($p > 0.05$), Rocatec and control ($p > 0.05$), and CoJet and Rocatec groups ($p > 0.05$).

Scanning electronic micrographs of untreated In-Ceram Zirconia showed alumina and zirconia platelets embedded in a glass matrix. Surface abrasion created during polishing in the form of linear streaks was evident (Fig 1). The untreated YZ Zirconia, being a pure zirconia, showed a uniformly structured surface. Abundance of surface abrasion due to polishing can be observed (Fig 2). Figure 3 shows both ceramics treated with silicatized alumina (CoJet) and coated with a silane coupling agent and a resin bonding agent. The resin-bonding agent covers the entire surface of the ceramics. Loose particles of

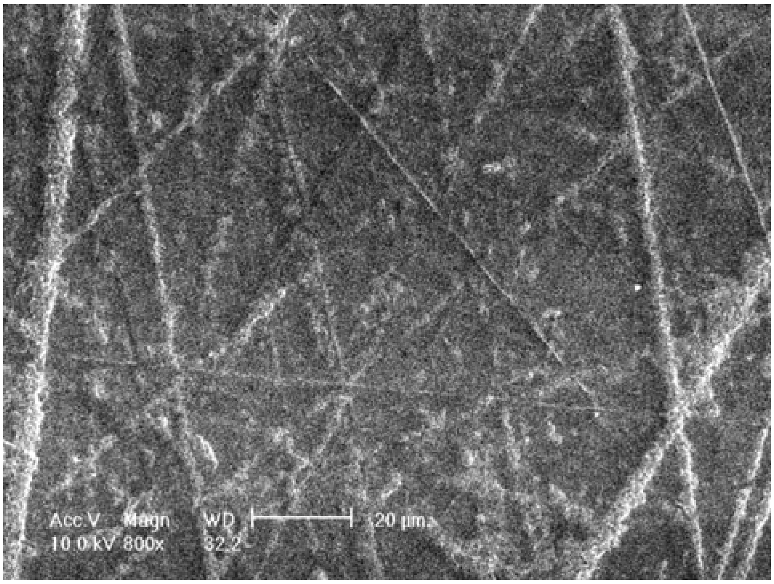

Figure 2 Scanning electron micrograph (800×) of untreated YZ Zirconia. Uniform ceramic surface with abundance of surface abrasion from polishing.

Table 1 Number of specimens classified according to modes of failure

		Adhesive failure between resin luting agent and dentin	Mixed mode of failure	Total
In-Ceram Zirconia	Control	5	7	12
	CoJet	8	4	12
	Rocatec	12	0	12
YZ Zirconia	Control	3	9	12
	CoJet	9	3	12
	Rocatec	12	0	12

alumina or ceramics can be observed scattered on their surfaces. Surfaces of both ceramics treated with Rocatec showed a coarse surface topography (Fig 4). As a result of airbrading with 110 μm Al_2O_3 and 30 μm silicatized Al_2O_3 particles and the subsequent silane coupling agent treatment, features of the original ceramics were less identifiable. Superimposed on the original features are loose particles of alumina and ceramics, giving the surface a coarser appearance.

A summary of the modes of specimen failures is presented in Table 1. Specimens either failed at the interface between dentin and resin-luting agent or exhibited a mixed mode of failure. Thus, all failures involved the dentin surface.

Discussion

The present study investigated the bonding characteristics of zirconia-reinforced dental ceramics to dentin, which to the authors' knowledge is not available in the literature. Reports of their bonding characteristics to composite resin or stainless steel blocks are nevertheless available.[14,18,23,24]

Failure mode analysis revealed that both types of zirconia-reinforced ceramics treated with Rocatec failed adhesively between dentin and resin luting agent (Table 1). The control and CoJet specimens failed either adhesively between dentin and resin luting agent or exhibited a mixed mode of failure. It is important to note that failure involved the dentin surface irrespective of surface treatment and the type of zirconia-reinforced dental ceramics. This directly implies that the dentin surface plays an important role in determining the bond strength of zirconia-reinforced ceramics. Thus, it is unlikely that bond strength studies of zirconia-reinforced ceramics using substrates other than dentin [14,18,23,24] are directly relevant to predicting the clinical characteristics of bonding between such ceramics and dentin.

Based on using dentin as the bonding substrate, the present study showed that surface treatment significantly affected the shear bond strength of zirconia-reinforced ceramics in general, in accordance with statistical analysis using two-way ANOVA. Further analyses showed that the shear bond strength of the glass-infiltrated, zirconia-reinforced alumina to human dentin using a phosphate monomer (MDF) containing resin-luting agent was improved with the chairside tribochemical silica coating/silane coupling system (CoJet group). Thus, the hypothesis that "surface treatments do not affect the shear bond strength of a glass-infiltrated, zirconia-reinforced alumina (In-Ceram Zirconia) to human dentin using a resin luting agent" is rejected.

The results of this study further showed that neither the chairside or laboratory tribochemical silica coating/silane coupling system improved the shear bond strength of a machined-milled, yttria-stabilized zirconia ceramic (YZ Zirconia). Thus, the hypothesis that "surface treatments do not affect the shear bond strength of a machined-milled, yttria-stabilized zirconia ceramic (YZ Zirconia) to human dentin using a resin luting agent" is accepted.

The effectiveness of the chairside system in improving the bond strength of one zirconia-reinforced ceramic but not the other can be discussed in the context of three published

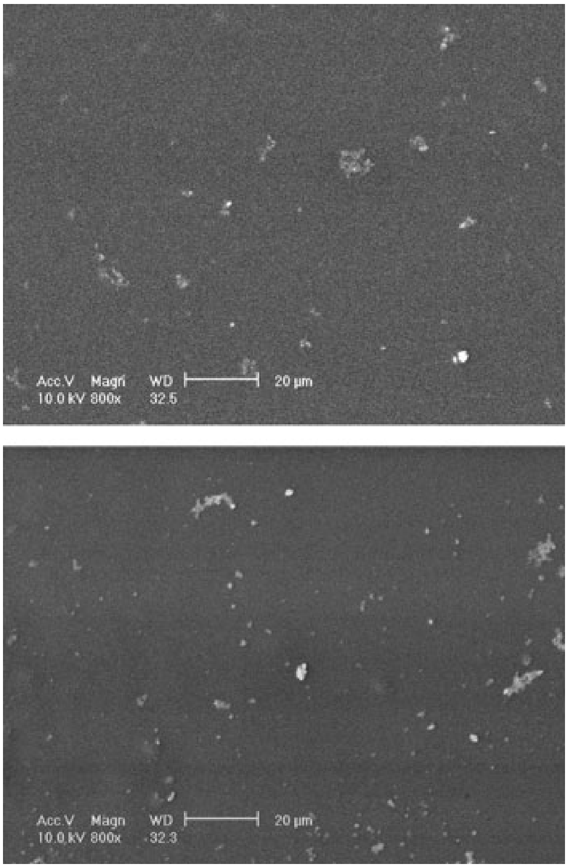

Figure 3 Scanning electron micrographs (800×) of In-Ceram Zirconia specimen (top) and YZ Zirconia (bottom) airbraded with silicatized Al_2O_3 particles (CoJet) and coated with a silane coupling agent and a resin bonding agent (dark background).

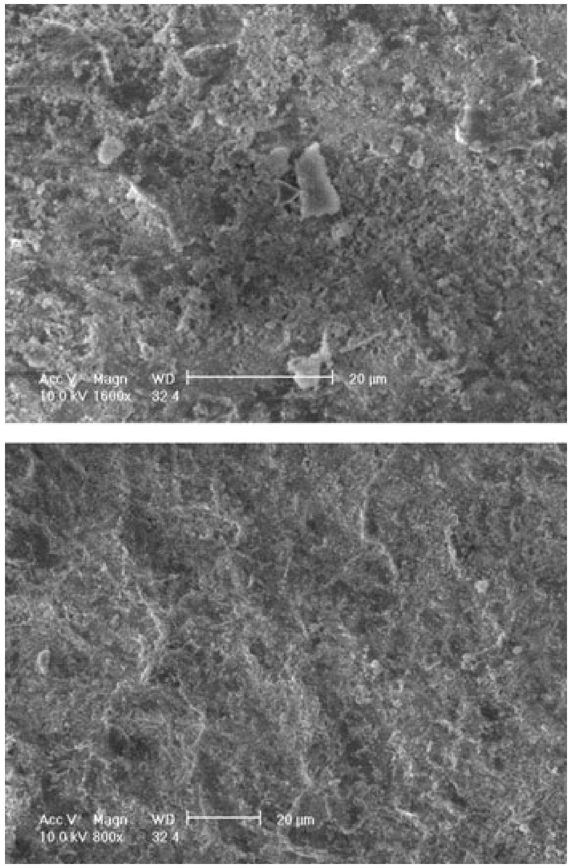

Figure 4 Scanning electron micrographs (1600×) of In-Ceram Zirconia specimen (top) and YZ Zirconia (bottom) airbraded with 110 μm Al_2O_3 particles and 30 μm silicatized Al_2O_3 particles, and then coated with a silane coupling agent.

studies.[18,19,22] The microtensile bond strength between a glass-infiltrated, zirconia-reinforced ceramic, and composite resin blocks using an MDP containing resin luting agent significantly increased with ceramic surface treatment using a chairside tribochemical silica coating system.[18,19] The shear bond strength between a zirconium oxide ceramic and composite resin using an MDP containing resin luting agent (Panavia F) significantly increased with the chairside tribochemical silica coating surface treatment in one study[22] but had no significant effect in another study using stainless steel as the bonding substrate.[25] Yet the shear bond strength of another MDP containing resin luting agent (Panavia 21) between the same zirconium oxide ceramics and stainless steel was significantly improved with the chairside system.[26]

Conversely, the present study did not show the laboratory tribochemical silica coating system significantly affecting the bond strength of the two zirconia-reinforced ceramics to dentin. This is in contrast to an earlier study showing the effectiveness of the laboratory system in improving the bond strength between a glass-infiltrated, zirconia-reinforced ceramic, and composite resin blocks using an MDF containing resin luting agent.[19] Two other microtensile bond studies of zirconium-oxide ceramics using composite resin block as substrate showed that tribochemical silica coating with 50 μm Al_2O_3 particles modified by silica was ineffective in improving the bond strength of three luting agents, including an MDP containing resin luting agent (Clearfil Esthetic Cement, Kuraray Co., Ltd.). The result was the same with or without the effect of 6 months of aging.[23,24] The discrepancy in findings between previous and present results is likely due differences in bonding substrate, the methods of bond strength test, cements used, and experimental protocol.

The improvement of dentinal bond strength by at least one method of tribochemical silica coating/silane coupling for In-Ceram Zirconia but not for YZ Zirconia may be explained by the difference in microstructure of the two ceramics. SEM observations made in the present study confirm previous findings.[18,19,22] The topography of both glass-infiltrated, zirconia-reinforced ceramics and zirconium oxide ceramics conform to their typical structure. Surface abrasion as a result of polishing is evident (Figs 1 and 2). Airbrasion with silicatized Al_2O_3 particles with (laboratory system) or without (chairside system) pretreatment airbrasion with 110 μm Al_2O_3 particles produced ceramic surfaces with an abundance of Al_2O_3 particles (Fig 4). Theoretically, air-particle abrasion could improve bond strength by enhancing surface microroughness and generating more hydroxyl groups on the ceramic surface to react with the silanol groups of the silane. The silane molecules alternatively chemically bond to the methacrylate groups of the luting resin.[18,19] It has also been suggested that the silane agent also promotes resin bonding by increasing surface energy and thus wettability of the bonding substrate.[22]

What favors In-Ceram Zirconia over YZ Zirconia in surface bonding enhancement with tribochemical silica coating/silane coupling may be explained by the presence of its vitreous glass phase. As discussed earlier, the glassy phase of In-Ceram Zirconia provides the necessary silicate groups for interaction with silane coupling agents and thus improves their bonding.[14] An electron dispersive spectroscopic analysis also showed a quantifiable increase in the silicon surface content of In-Ceram Zirconia after silica coating.[10] The extreme surface resistance of the yttria-stabilized zirconia ceramics (YZ Zirconia) to tribochemical etching may explain the lack of bonding enhancement by both chairside and laboratory surface treatments found in the present study, thus confirming similar findings published earlier.[23,24]

The chairside tribochemical silica coating/silane coupling system improved bonding of In-Ceram Zirconia while the laboratory system did not. The difference in materials and procedures of the two systems should be analyzed. The laboratory system requires airbrasion of the ceramic surface with 110 μm Al_2O_3 particles for 10 seconds prior to silicatization with 30 μm silicatized Al_2O_3 particles, while the chairside system does not. The chairside system calls for the application of a resin-bonding agent after coating the ceramic surface with the silane-coupling agent, while the laboratory system does not (resin to be applied by clinician after the "restoration" is delivered after surface treatment in the laboratory). Abrasion with 110 μm Al_2O_3 particles according to the laboratory system may promote bonding by increasing microroughness; however, the impact of 110 μm Al_2O_3 particles on the surface integrity of the ceramics or on the adhesion of silicatized Al_2O_3 particles is unknown. The application of a resin-bonding agent according to the chairside system may enhance bonding by improving the wettability of the resin luting agent to the ceramic surface. Further study is necessary to confirm this hypothesis.

Conclusions

Within the limitations of this study, the following conclusions are drawn:

1. The type of surface treatment using tribochemical silica coating/silane coupling significantly affected the shear bond strength of zirconia-reinforced alumina to human dentin while the type of ceramics did not.
2. A chairside tribochemical silica coating/silane coupling system, which used a resin bonding agent, is effective in improving the shear bond strength between a glass-infiltrated, zirconia-reinforced alumina (In-Ceram Zirconia) and human dentin using an MDF containing resin luting agent. The same system was not effective in improving the shear bond strength of a machined-milled, yttria-stabilized zirconia ceramic (YZ Zirconia).
3. A laboratory tribochemical silica coating/silane coupling system, which does not use any resin bonding agent, did not significantly change the shear bond strength between two zirconia-reinforced ceramics and human dentin using an MDF containing resin-luting agent.

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