High shear bond strength between zirconia ceramic and resin cement via surface treatment and cleaning

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Abstract
The bonding strength between ceramic and resin is an important property for all-ceramic dental restoration. However, the chemically inert surface of the ceramic generally limits the interaction with the resin and surface contamination, resulting in reduced bonding strength. Therefore, several methods have been used to physically and chemically treat and clean the surface of the ceramic. However, few reports exist regarding long-term bond durability. Herein, we investigate the effect of surface cleaning and multiple treatments on the long-term bonding strength between zirconia and resin cement before and after thermal cycling. The shear bond strength in every group was characterized using a universal mechanical testing machine and averaged from 16 measurements. Results show that after cleaning using Ivoclean with a microbrush, the zirconia surface treated by a mixed silane showed a maximum shear bonding strength of up to 32.5 MPa and retained 29.7 MPa after 5000 thermal cycles. This result indicates that the silanized surface shows excellent bond durability by resisting the damage induced by cyclic expansion and contraction. Long-term high bonding strength arises from combined cleaning and treatment, removing saliva or other organic impurities and forming Si–O–Si and –C–C– at the interface and micromechanical interlocking. This result is promising for future applications in high-strength all-ceramic restoration by optimizing the surface treatment.

1. Introduction
Given the continuous requirements for quality and aesthetics of restorations, common restorative materials are gradually developed from metal–ceramic to all-ceramic matrix materials [1]. Yttrium stabilized tetragonal zirconia (Y-TZP) is formed by injecting a small amount of yttrium (Y2O3) into zirconia. Compared with several other dental restoration materials, Y-TZP has excellent physical and chemical properties. Hence, zirconia ceramics are widely used as the base material in all-ceramic restorations[1, 2]. Because of its good biocompatibility and appropriate mechanical strength, it is also widely used in post and core, orthodontic brackets, implants, and other oral repair or reconstruction treatments.

The high crystalline content and low glass phase of zirconia make it resistant to dissolution and corrosion from numerous strong acids and alkalis and organic or inorganic treatment agents [3]. However, the strengthened inertia on the zirconia surface considerably affects the zirconia surface modification with commonly used bonding agents. The difficulty of bonding ability also considerably reduces the applications of zirconia ceramic as an excellent restoration material, such as inlay, Maryland bridge, and veneer [4, 5]. Therefore, the bonding strength improvement of zirconia ceramic has attracted significant research attention in the past decade.

Numerous studies [6–8] suggest that air particle abrasion (APA) is an effective surface modification method to improve the bonding strength between zirconia and resin cement. APA improves the surface energy and
wettability of the zirconia surface. Moreover, the created roughened micron-scale surface can improve the bonding area and form a micromechanical interlocking structure with the resin cement. However, sandblasting may lead to the transformation of the surface or lower-layer crystals from the tetragonal to monoclinic phase\cite{9}, which may degrade the mechanical properties of the materials\cite{10–13}. Therefore, some manufacturers suggest using alumina particles with particle sizes <50 μm to sandblast the zirconia surface for minimizing the crystal phase transformation\cite{14}. The use of a silane-coupling agent can increase the wettability of the zirconia ceramic surface. A commercial silane-coupling agent is commonly used as a single liquid type, an aqueous solution mixture of 3-methacryloxypropyl trimethoxysilane (γ-mps) and acidic organic monomer. The silane molecule contains an organic functional group and three hydrolyzable methoxyl groups\cite{15–20}. Silane should be hydrolyzed in an acidic ethanol–water solution to catalyze the formation of siloxane. Finally, hydrolyzed silane can be chemically or physically adsorbed on the porcelain surface. The three methacrylate groups can copolymerize with the unreacted C=C of the resin cement monomer via the light-curing procedure\cite{21}. Previous studies\cite{22, 23} showed that using an experimental primer which inorganic acid (hydrochloric acid (HCl)) as hydrolysis catalyst and adding nonfunctional silane 1, 2-bis trimethoxysilyl ethane (BTS) can effectively improve the shear strength between ceramic and resin cement.

In clinical prosthetic work, the modified zirconia ceramic surface can be easily contaminated by saliva or other organic impurities, reducing bonding strength. Unlike silicon-based ceramics, cleaning zirconia with phosphoric acid is ineffective. Theoretically, organic molecules in the oral saliva such as phosphate group might react with the monomer of the resin to form hydroxyl groups on the zirconia surface. Acidic organic compounds in the cement are of great importance of high bonding strength between resin and modified zirconia ceramic, however organic molecules to some degree inhibit the formation of chemical bonds between monomer and modified zirconia ceramic.

Therefore, the author considered using commercial cleaning solutions (Ivoclean, Ivoclar, Vivadent, Schaan, Lichtenstein). According to the manufacturer’s instructions, after using the Ivoclean solution on the tissue surface of the restorations, washing and drying can effectively clean the bonding surface of the restorations, which are contaminated by saliva or impurities\cite{24–26}. However, only a few studies concerning the effect of this cleaning solution on the long-term storage periods or thermocycling regimens between zirconia and resin cement exist\cite{27}. Our study assesses the effect of different pretreatment methods on the bond durability of zirconia ceramic to resin cement. The null hypotheses are the following: (a) the cleansing methods would not negatively affect bond durability; (b) the surface pretreatment of zirconia ceramic with experimental primer can effectively improve the shear bonding strength between the zirconia ceramic and resin cement.

2. Materials and method

2.1. Preparation of samples

Two hundred and fifty-six zirconia block specimens were cut using a precision saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to a square of 6-mm length and 2-mm thickness (ZS-B70/20, KaVo Arctica, Kavo Dental, Germany) (table 1). After being fully sintered according to the manufacturer’s instructions, the specimens were embedded in acrylic resin (GC, Tokyo, Japan). The embedded zirconia ceramics were exposed to a square
Table 1. Details of materials used in this study.

| Materials             | Composition                                      | Manufacture                          | Lot number |
|-----------------------|--------------------------------------------------|--------------------------------------|------------|
| Zirconia              | ZrO$_2$ 87%, and Y$_2$O$_3$                     | ZS-B70, KaVo Artica, Germany         | 102548460  |
| Monobond-s            | Ethanol: 50%−100%, 3-methacryloxypropylmethacrylate-silane < 2.5% | Ivoclar-Vivadent, Liechtenstein      | Z000FZ     |
| Experimental silane   | 3-methacryloxypropyl- trimethoxysilane           | ShinEtsu Chemical, Industry, Tokyo, Japan | 901770 / HH35E |
| 1,2-Bis(trimethoxysilyl)−Ethane | decyldihydrogenphosphate                             |                                       |            |
| Panavia F 2.0 Ivoclean | 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate silica, dl-camphorquinone, catalysts Zirconium oxide, water, polyethylene glycol, sodium hydroxide, pigments | Kuray, Tokyo, Japan Ivoclar-Vivadent, Schaan, Liechtenstein | 071200 Y49151 |

Bis-GMA: Bisphenol A Diglycidil Metacrilate, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate, 10-MDP,10-methacryloyloxy decyldihydrogenphosphate.

surface on one side, serving as the bonding adherents. The exposed ceramic surfaces were continuously polished using 320-, 600-, and 1000-grit rotating silicon carbide paper (Labopo-5, Struers, Ballerup, Denmark) in sequence under running water. The samples were ultrasonically cleaned using 95% ethanol for 2 min to remove any loose and contaminated impurities.

2.2. Preparation of experimental zirconia primer
The experimental zirconia primer used in this study was designed as a two-liquid type: Primers A and B. Primer A comprised a mixture of two types of 50-mg silanes dissolved in 1-ml ethanol. The produced mixed silane was composed of γ-tnps and BTS (Tokyo Chemical Industry, Tokyo, Japan) at 70 and 30 mol%, respectively.

For Primer B, hydrochloric acid was selected as the initiator to accelerate the hydrolysis of the methoxy groups of silane molecules. The hydrochloric acid solution (0.1 mol l$^{-1}$, Wako Pure Chemical Industries, Osaka, Japan) was diluted to 0.05 mol l$^{-1}$ (pH = 1.64) using distilled and deionized water with pH = 6.0. Then, the diluted hydrochloric acid solution was dissolved in ethanol (50% by volume).

2.3. Pretreatment of adherent samples
According to different surface treatment methods, 256 specimens were randomly divided into 8 groups (32 samples in each group; figure 1).

For groups 1–4, the zirconia surface was treated via airborne-particle abrasion using silica-modified 50-μm Al$_2$O$_3$ particles for 20 s at 40-psi pressure and a distance of approximately 40 mm (Cobra, Renfert, Hilzingen, Germany). Subsequently, the specimen was ultrasonically cleaned in isopropyl alcohol for 2 min and then rinsed with water and finally air-dried using oil-free compressed air to remove residual particles. The remaining four experimental groups 5–6 did not require surface-sandblasting pretreatment. Then, each surface of the eight groups was contaminated with human saliva for 3 min. The saliva used in this study was collected from a healthy, nonsmoking male donor who had refrained from eating and drinking 2 h prior to collection. This procedure was performed following the ethical regulations of the Stomatological Hospital of Tianjin Medical University.

The cleaning procedures were then applied to the zirconia surface of groups 3, 4, 7, and 8 with Ivoclean using a microbrush with agitation for 20 s, followed by rinsing with water for 20 s, and drying for another 15 s, according to the manufacturer’s instructions.

The commercial ceramic primer (Monobond-s) was used to silanize the zirconia surface in groups 1, 3, 5, and 7 for 60 s, according to the manufacturer’s instruction. The designed zirconia ceramic primer was used to pretreat the zirconia surface in groups 2, 4, 6, and 8. The amount of primer applied each time was 0.3 mg, comprising equal amounts of Primers A and B and 60-s actuation duration. This duration was selected based on preliminary experimental research and the highest index of silanization efficiency on the ceramic surface.

2.4. Shear bonding strength test
After gentle air drying, the silicone rubber ring mold was placed on the pretreated zirconia ceramic surface. The inner diameter of the circular hole of the silicone ring was 3 mm, and the thickness was 2 mm. Filled the resin cement Panavia F2.0 into the silicone ring and pressed with 50 g weight. Then, the sample was irradiated with visible light (Coltolux LED, Coltene, OH, USA) for 30 s. The power density of the light source was 350–600 mW cm$^{-2}$. After irradiation, the remaining resin cement and ring mold were carefully removed.
The above-mentioned eight experimental groups were randomly divided into two subgroups \((n = 16)\): one subgroup was stored in water at 37 °C for 24 h (not thermocycled), and the other subgroup was subjected to thermocycling treatment. The samples were circulated 5000 times between two water baths at 5 °C and 55 °C (Thermal Shock Tester, Thomas Kagaku, Japan), the residence time in each water bath was 60 s, and the transfer process was 7 s.

The shear bond strength of the resin cement used on the zirconia surface was tested using a universal testing machine (Instron, 3367, Canton, MA, USA) with a 1.0-mm min \(^{-1}\) crosshead speed until failure. The shear load was measured in megapascals by dividing the load measured at the failure instant by the bonding area of each sample (in mm\(^2\)). If debonding occurred before the shearing tool touched the cement, the bonding strength was defined as 0 MPa. The cross section of the sample-bonding process and the schematic of the shear bond test are shown in figure 2.
2.5. SEM observation
The surface morphology of the zirconia sample before and after sandblasting and their bonding performance between the resin cement were observed by SEM (Hitachi S-4800, figure 3) using the backscatter detector. The surface was highly polished before the observation. All samples were observed after being coated with gold (figures 3 and 4).

2.6. Determination of the failure mode
After SBS testing, the fracture was observed using a digital microscope (MSV 330, Anyty, 3R, Japan) and SEM (figure 4). Figure 4 illustrates the fracture mode of all samples.

The results show that the fracture modes of the bonding interface can be divided into three types: 1) cohesive failure of zirconia ceramic (figures 4(a) and (b)). It can be seen that the failure is mainly dominated by the broken surface of ceramic; 2) mixed failure, including cohesive failure of ceramic and interfacial failure (figures 4(c) and (d)). The failure surface consists of the broken surface of the ceramic and the remains of the resin. 3) interfacial failure between the zirconia ceramic and resin cement (figures 4(e) and 4(f)). There are no visible remains of either the ceramic or the resin.

2.7. Statistical analysis
The mean shear bonding strength and standard deviation (SD) of each group before and after thermocycling were calculated. Statistical analysis included a one-way analysis of variance (ANOVA) along with Tukey’s post hoc test for multiple comparisons (SPSS Software, San Diego. CA, USA) with a significance level of $P < 0.05$. The significance of the differences between the groups was determined using the student’s test of each independent variable. Moreover, ANOVA tests were performed on all samples.
3. Results

3.1. Bonding performance

The shear bond strength (Table 2) between zirconia ceramic and resin cement is determined by whether the surface is sandblasted or the surface cleaner is used, and different ceramic primers are used. Without thermocycling, the lower shear bonding strengths of 6.5, 7.3, 11.2, and 17.6 MPa were measured for the NM, NE, NIM, and NIE test groups, respectively. Correspondingly, the AM, AE, AIM, and AIE test groups treated with air abrasion exhibited relatively higher bonding strength of 22.5, 30.7, 23.7MPa, and 32.5 MPa, respectively. This shows that air abrasion of zirconia ceramic can effectively improve the initial bonding strength between the ceramic and resin cement. Compared with the treatment with commercial ceramic primer (groups 1 and 3), treatment with experimental zirconia primer (groups 2 and 4) could lead to a significant increase (P < 0.05) in the initial bonding strength.

We also found that using Ivoclean to clean the ceramic bonding surface under the same processing conditions slightly improves the bonding effect, but there is almost no significant difference. The initial bonding strength was considerably improved only between groups 6 and 8.

The bonding effect between the zirconia ceramic resin and cement can also be revealed by the failure mode of different front processing methods. The air-abrasion effect was glaringly obvious via the fracture mode analysis. All sandblasted experimental groups (1–4) exhibited cohesive failure, which was different from the mixed failure or interfacial failure of unsandblasted groups (5–8). By evaluating the Ivoclean effect on the fracture mode, in the unsandblasted experimental groups (5–8), the specimens treated with Ivoclean (groups 7 and 8) showed more mixed failures than those untreated with Ivoclean (groups 5 and 6). However, no significant difference existed between groups 5, 6, 7, and 8. Notably, a little cohesive failure existed in experimental groups 7 and 8. Figures 3(c) and (d) show that the air abrasion efficiently increased the surface roughness of the zirconia ceramic, created an irregular surface morphology, and increased the bonding reliability surface area, resulting in a micro-interlocking of the bonding structure.

To evaluate the effects of pretreatment methods and two types of ceramic primers on the bond durability, the shear strength between the resin cement and zirconia ceramic surfaces after 3000 cycles is given in Table 2.

The bonding strength of test groups 5–8 considerably decreased after thermocycling, and the fracture surface mode changed from mixed and cohesive to mostly interfacial failures. The lowest bonding strength of 0 MPa appeared in test group 5 (no sandblasting + no Ivoclean + commercial silane). Group 8 exhibited a significantly higher adhesion (5.8 MPa) (Ivoclean + experimental treatment agent) than groups 5–7. Although no significant difference exists in the fracture mode of this group compared with the other three groups, more mixed failures can be observed.

Using only air abrasion as a variable, the bonding strength of experimental groups 1–4 after thermocycling is much higher than that of experimental groups 5–8. To match with it, a significant difference also exists in the fracture mode. Experimental group 4 (AIE) showed the highest bonding strength (29.7 MPa), which did not decrease considerably compared with that before thermocycling. Notably, all cohesive failures still occurred.

### Table 2. Mean and standard deviations of shear bonding strength values of all groups.

| Group (n = 32) | Mean ± SD (MPa) | Failure modes [C/M/I] |
|---------------|-----------------|-----------------------|
|               | Before TC       | After TC              | Before TC       | After TC       |
| Group 1 (AM)  | 22.5(3.4)       | 10.7(1.5)             | [16/0/0]        | [4/4/8]        |
| Group 2 (AE)  | 30.7(4.1)       | 25.3(3.2)             | [16/0/0]        | [13/3/0]       |
| Group 3 (AIM) | 23.7(3.3)       | 16.6(3.5)             | [16/0/0]        | [7/5/4]        |
| Group 4 (AIE) | 32.5(3.8)       | 29.7(4.1)             | [16/0/0]        | [16/0/0]       |
| Group 5 (NM)  | 6.5(1.4)        | 0.0(0.0)              | [0/3/13]        | [0/0/16]       |
| Group 6 (NE)  | 7.3(0.9)        | 1.2(0.4)              | [0/3/13]        | [0/0/16]       |
| Group 7 (NIM) | 11.2(1.7)       | 2.1(0.7)              | [2/8/6]         | [0/1/15]       |
| Group 8 (NIE) | 17.6(2.0)       | 5.8(1.3)              | [4/5/7]         | [0/3/12]       |

The different superscript numbers for each horizontal row: significant difference (P < 0.05). The different subscripts characters (a)–(d) for each vertical column: significant difference (P < 0.05). [C/M/I]: Cohesive failure/Mixed failure consisting of interfacial failure and cohesive failure/interfacial failure.
The shear strength of the AIE group was considerably higher than that of all other experimental groups except group 2 (25.3 MPa). The bonding strength of the experimental groups (2 and 4) treated using the experimental primer was approximately twice of those treated with commercial silane (1 and 3). The fracture mode of groups AM and AIM changed from all cohesive failure to and mixed or interfacial failure. Under the same conditions, using Ivoclean can effectively increase the bonding strength, especially when combined with commercial silane.

3.2. Discussion

This research project demonstrates the effect of different zirconia surface preconditioning methods and different zirconia ceramic primers on the bonding strength of resin cement. From the analysis of the initial shear strength, it can be found that surface sandblasting is critical for enhancing the bonding strength between the zirconia ceramic surface and resin cement. SEM observation can reveal the apparent effect of surface-sandblasting on surface roughness increase (figure 3). The observation shows that the sandblasting results in the rough surface of zirconia with more trench and punctate imprints. Such rough surface is beneficial to be infiltrated by resin cement in order to form micro-undertag in the adhesive layer, which contributes to high bonding strength.

In clinical practice, surface sandblasting is routinely performed to increase the bonding effect. This operation can increase adhesion because air abrasion can effectively increase the roughness of the bonding surface and wettability. The most efficient is that due to the wettability increase, the resin cement penetrates the rough surface or the undercut area of the adherent. After the light-curing polymerization reaction, a micromechanical interlocking force is formed, providing more resistance to microtensile or microshear strength to considerably enhance the adhesive retention.

The initial bonding strength of the experimental group using an experimental ceramic primer is generally higher than that of the commercial primer group. This difference in the condition effect is because the organic acid monomer (methacryloyl group) of the commercial ceramic primer cannot efficiently catalyze the hydrolysis of silane molecules [28, 29]. A part of the unhydrolyzed silane \( \gamma \)-MPS molecules results in a physical adsorption effect on the zirconia surface, competitively occupying the limited Zr–O– on the zirconia surface and hindering the formation of beneficial Zr–O–Si bonds. The bonding strength increase in the experimental ceramic primer group may be due to the increase in the number of silane species chemisorbed onto the ceramic surface, changing the adsorption characteristics of the silane species.

However, the carbonyl group in 4-methacryloyloxyethyltrimethacrylate (4-META) in the commercial ceramic primer forms a hydrogen bond with zirconium oxide. This reaction hinders the formation of chemisorption of the hydrolyzed \( \gamma \)-MPS molecules and zirconia, and the reduction in the effect of condensation efficiency will eventually lead to weak bonding strength between the ceramic surface and resin cement. When the hydrochloric acid solution in the experimental ceramic primer initiates the hydrolysis of \( \equiv Si–O–CH_3 \) (or \( \equiv Si–O–CH_2CH_3 \), the \( \equiv Si–OH \) group becomes stronger and finally forms a strong \( =Si–O–Zr \) Group [30–33].

In this study, the zirconia ceramic samples were contaminated using saliva, causing contamination of the bonding surface by organic substance residues. Some studies have found that phosphoric acid or exclusive cleaning agents reduce the concentration of carbon oxides on the adherent surface compared with the uncleaned surface. Recently, some studies have investigated the use of cleaning agents to rinse the ceramic surface before coating the adhesive. A relative novelty commercial cleaning solution (Ivoclean) is generally employed in clinical processes. This technology’s cleansing agents rely on an alkaline zirconia particle suspension, which likely adsorbs phosphorus on the adherent surface contaminated by saliva to form a hypersaturated zirconia particles solution. Compared with the NM and NE of the uncleaned group, NIM and NIE in the Ivoclean cleaning group achieved better bonding performance, and the morphology of the failure mode also showed more adhesive failure.

After performing the fatigue test (thermocycling), the data of all test groups except the AIE group showed a significant decline. Most of the samples in groups 5 and 6 were debonded before the shear test. Groups 7 and 8 also showed extremely low bonding strength, and most of the fracture modes showed interfacial failure, indicating that surface sandblasting is crucial for improving the bonding durability. Unlike the initial adhesion, the experimental and commercial ceramic primer groups showed a significant difference after thermocycling. This reason has been discussed in previous studies. The experimental ceramic primer is developed using two types of silanes. As a nonfunctional silane, BTS can increase the elasticity of the silane layer and better resist the thermal expansion and contraction stress of the bonding layer caused by the thermal cycle fatigue test. Moreover, owing to the presence of certain nonfunctional silanes, an interpenetrating polymer network (IPN) can be formed inside the silane layer. The formation of IPN effectively improves the toughness and intensity of the silane layer and the ability to resist external forces. Using the cleaning agent as a separate variable to examine the data after thermocycling showed no significant difference in the data, indicating that the ceramic cleaning agent is inconsequential in increasing the bonding durability.
Hence, our two hypotheses are correct: the cleansing methods would not negatively affect the bond durability, and the surface pretreatment of zirconia ceramic with experimental primer can effectively improve the shear bonding strength between the zirconia ceramic and resin cement.

4. Conclusion

We investigated the effect of surface cleaning and multiple treatments on the long-term bonding strength between zirconia and resin cement before and after thermal cycling. The maximum shear bonding strength of up to 32.5 MPa in the zirconia surface treated using a mixed silane was obtained after cleaning with Ivoclean using a microbrush, retaining 29.7 MPa after 5000 thermal cycles. This result indicates that the silanized surface shows high resistance to the damage induced by cyclic expansion and contraction, leading to excellent bond durability. Furthermore, the long-term high bonding strength derived from combined cleaning and treatment removes saliva or other organic impurities and forms an IPN crosslinked by Si–O–Si–C–C– at the interface as well as micromechanical interlocking. This result holds great potential for future applications in high-strength all-ceramic restoration via surface treatment optimization.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Conflicting interests

Rui Li is an employee of Stomatological Hospital, Tianjin Medical University. No authors have received grants from anywhere.

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Contributorship

Rui Li and Ying Chun Sun researched literature and conceived the study. Rui Li was involved in protocol development, gaining ethical approval, and data analysis. Rui Li wrote the first draft of the manuscript. All authors reviewed and edited the manuscript and approved the final version of the manuscript.

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