Evaluation of mold-enclosed shear bond strength between zirconia core and porcelain veneer

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This study aimed to evaluate the mold-enclosed shear bond strength (ME-SBS) of zirconia to veneering porcelain with different surface treatments. Colored or uncolored zirconia coupons were either highly polished or airborne-particle abraded. The specimens were divided into groups with/without application of liner. Veneering porcelain was fired into an alumina ring mold on the zirconia coupons. The assembled specimens were subjected to the ME-SBS test. The mean ME-SBS for groups ranged from 7–10 MPa with no significant difference ($p>0.05$). A three-way ANOVA showed that coloring and surface roughening of the zirconia specimen had no significant influence on the ME-SBS value, but liner application exhibited a significant effect with a minor decrease in the ME-SBS ($p=0.049$). Surface treatments (coloring, airborne-particle abrasion, and liner application) were found to not cause a significant increase to the zirconia-porcelain bond strength. The application of zirconia liner had a slight negative influence on the ME-SBS results.

Keywords: Zirconia, Coloring, Airborne-particle abrasion, Liner application, Mold-enclosed shear bond strength

INTRODUCTION

With advances in the field of dental materials and technology, all-ceramic or metal-free tooth-colored restorations have become a routine option in contemporary prosthodontic treatments. Among such restorative systems, zirconia ceramics are central to all-ceramic restorative systems, including multiunit fixed partial dentures, owing to their outstanding mechanical properties and dental CAD-CAM technologies¹⁻³. However, zirconia ceramics are pure white and highly opacity, making it difficult to mimic a natural tooth with an optimal shade using this material alone. Thus, to fabricate zirconia restorations with highly esthetics, it is necessary to apply veneering porcelains onto zirconia substrate by means of powder-slurry build-up or heat-pressing⁴. An extrinsic or intrinsic coloring of zirconia core with a shade similar to dentin would possibly be help to improve the esthetics of zirconia restorations⁵.

However, chipping of the porcelain veneer from the zirconia core are often occurred in clinical applications and the most common failure in zirconia restoration when compared to metal-ceramic restorations. The initial incidence rate of chipping in zirconia restorations has been reported as generally 15–25% after 24–31 month⁶⁻⁸. The incidence rate was decreased to 6% for 37 months lately, chipping was main hurdle for clinical success of zirconia-porcelain restorations⁹. There are many factors contributing to this chipping¹⁰. Porcelain

chipping in all-ceramic restorations, including zirconia-porcelain systems, is caused by excessive tensile stress due to a thermal mismatch between the porcelain veneer and the zirconia core, inadequacies in the geometry of the framework design and/or core/veneer thickness ratio, an insufficient bond strength of the core-veneer combination, and so on^{6,11}. To overcome these chipping-induced failures, a higher bonding strength between the zirconia core and veneer materials is essential because this bond constitutes the weakest part of a zirconia prostheses veneered with porcelain¹².

Researchers have determined that the factors influencing the bond strength between zirconia and porcelain are chemical bonding, mechanical bonding relying on the surface roughness of zirconia, the wetting properties of porcelains, and the compressive stress acting on veneer layer due to difference in the thermal expansion coefficient^{13,14}. Among the various factors affecting the bond strength, airborne-particle abrasion and the application of a zirconia liner has been the subject of many studies with the goal of increasing the zirconia-porcelain bond strength, as these surface treatments are strongly recommended by manufacturers. However, studies addressing the effects of surface treatments on the bond strength have often produced conflicting results¹³⁻¹⁸. For example, it has been indicated that roughening of the zirconia can increase the shear bond strength of veneering ceramic to zirconia¹⁶⁻¹⁸. However, other studies have concluded that neither particle abrasion nor liner application increases the bond strength significantly¹³⁻¹⁵. Therefore,

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Table 1 Materials used in this study

Materials	Brand	Manufacturer	Lot No.
Zirconia	IPS e.max ZirCAD		N78026
Coloring liquid	CL1	Ivoclar Vivadent, Schaan, Liechtenstein	R60559
Liner	IPS e.max Ceram Zirliner		T42562
Porcelain	IPS e.max Ceram Dentin (A2)		S20293

the influence of those surface treatments on the bond strength between zirconia core and veneering porcelain remains uncertain.

Although various test methods have been proposed to evaluate the bond strength between the core and veneer materials, micro- or macro-shear bond strength (SBS) tests have been most widely adopted by previous studies due to the simplicity of the sample preparation and the testing procedure. However, the conventional SBS test protocol has been criticized due to its stress localization and the non-uniform distribution of stresses in the interface between the adhesive and adherend surfaces^{19–22}. As an alternative, the mold-enclosed shear bond strength (ME-SBS) test has been proposed as a means of eliminating the uneven stress issue of the conventional SBS test^{23–25}. Previous studies have adopted the ME-SBS test to compare the bond strength between composite resin/cement materials and dental alloys^{24,26}. However, to the best of our knowledge, no studies have used the ME-SBS test method for investigating the ceramic-ceramic bond strength.

The goal of the present study is to evaluate the influences of the coloring, liner application, and airborne-particle abrasion of zirconia core on the bond strength between the zirconia ceramic and the veneering porcelain, using the ME-SBS test. In particular, the bond strength between the veneering porcelain and a highly polished zirconia surface was compared with that between a veneering porcelain and particle abraded zirconia. The null hypothesis of this study was that the surface treatments of zirconia, including coloring, roughening by particle abrasion, or the application of a zirconia liner have no influence on the ME-SBS between zirconia and porcelain ceramic.

MATERIALS AND METHODS

Preparation of zirconia specimen and coloring

A total of 80 presintered zirconia specimen coupons measuring 19 mm (W)×15 mm (L)×3 mm (H) were made by cutting commercial zirconia blanks intended for CAD-CAM (IPS e. max ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein) using a dental cast-cutting machine with a diamond disk (Cutter Plus, High Dental, Seoul, Korea). To establish the baseline condition, the surfaces of all the sample coupons were wet-ground with #180 SiC paper (Struers, Ballerup, Denmark), ultrasonicated in distilled water for 10 min, and then dried. The prepared zirconia coupons were divided into colored and

uncolored groups ($n=40$). Forty specimens of the colored group were immersed in coloring liquid (CL1, Ivoclar Vivadent) for 2 min, washed in water, and then dried to make the colored specimens. Then, all the specimen coupons were subjected to a final sintering process in the furnace intended for ZirCAD (Programat S1, Ivoclar Vivadent) according to the manufacturer's instructions. Table 1 shows the materials used in the present study.

Surface treatments and zirconia liner application

All the zirconia specimens (both colored and uncolored) were again separated into two groups, namely, high-polished and airborne-particle abraded groups ($n=20$). To produce a roughened zirconia surface, a particle blaster (Basic, Renfert, Hilzingen, Germany) and 110- μ m Al_2O_3 particles (Renfert) were used, with the blaster set to a distance of 10 mm, an angle of 45°, and a pressure of 4 bar, for 10 s. For the highly polished group, the zirconia coupons were ground using a horizontal grinding machine (HRG 150, AM Technology, Ansan, Korea) and then polished sequentially using a lapping machine (Okamoto, Annaka, Japan) with 6, 3, and then 1- μ m diamond polishing slurries. Then, ultrasound cleansing was performed with acetone for 10 min. After the surface treatments, the surface roughness (R_a) of each group was determined using a surface roughness tester (SJ-400, Mitutoyo, Tokyo, Japan) operating at a speed of 0.5 mm/s with a 4 mm scan. Three tests were performed on each surface ($n=6$).

Each of the surface treatment groups were further divided into 2 groups, that is, with or without the application of a zirconia liner (IPS e.max Ceram Zirliner, Ivoclar Vivadent) ($n=10$). A thin silicone index with a hole (4 mm diameter) was used to position the Zirliner in the center of the specimens. The liner was first applied to the indexed area using a brush and then fired independently according to the manufacturer's instructions. After the first firing, the Zirliner was re-applied to compensate for the shrinkage that occurs in sintering and then fired again.

Preparation of zirconia-porcelain mold-enclosed specimens and the shear bond test

To prepare zirconia-porcelain ME-SBS specimen, an alumina ring mold (with a material thickness of 1.0 mm, a height of 5 mm, and an inner diameter of 4.0 mm) was used to enclose the veneering porcelain (IPS e.max Ceram Dentin A2, Ivoclar Vivadent), bonded to the zirconia core ceramic. First, porcelain mixed with

the manufacturer's liquid was built-up into the alumina ring mold to a depth of about 1 mm using an ultrasonic vibrator (Ceramasonic, Shofu, Kyoto, Japan) and then fired without the ring mold. After the first firing, the alumina ring was repositioned on the specimen disks and veneer porcelain was added to compensate for the porcelain shrinkage, up to a depth of 2 mm. Then, the specimens were fired twice with the alumina ring. A total of five firing procedures (two liner and three dentin porcelain firings) were performed according to the manufacturer's instructions to simulate the routine laboratory process for fabricating dental zirconia prosthesis. After firing, a shiny, glassy surface was observed on the surface of the porcelain, indicating that the firing was satisfactory (Fig. 1). The assembled zirconia-porcelain ME-SBS specimens were embedded into a self-cured acrylic resin (Quickcure, Alliedtech, Rancho Dominguez, CA, USA) using a cylindrical mold (diameter 30 mm and height 20 mm). All specimens were stored in distilled water at a temperature of 37°C for 24 prior to the test.

The SBS test was performed using a metal apparatus conforming to ISO/TS 11405 (Dental Materials-Testing of adhesive to tooth structure) mounted in a universal test machine (Instron 3344, Instron, Norwood, MA, USA). The specimens were debonded using a hardened steel shear blade with an 8-mm diameter hole with a blunt end (1 mm) at a crosshead speed of 1 mm/min (Fig. 1). The ME-SBS value (MPa) was calculated by dividing the maximum debonding load (N) with the cross-section area (12.6 mm²) of the alumina ring mold. The debonded surfaces of specimens were analyzed using a stereomicroscope to identify the failure mode (adhesive or cohesive) and selected samples are observed using scanning electron microscope (S-3000H, Hitachi, Tokyo, Japan).

Statistical analysis

The mean SBS values were obtained by testing 10 specimens from each group. Three-way ANOVA was performed to examine the effect of coloring, surface treatment, liner application, and their interactions, on the SBS. The mean SBS values were statistically analyzed using one-way analysis of variance (ANOVA) with Tukey's *post hoc* test (SPSS 22.0; SPSS, Chicago, IL, USA). The surface roughness was analyzed by an independent *t*-test ($\alpha=0.05$). A value of $p<0.05$ was

considered to be significant.

RESULTS

The values obtained for the surface roughness of the zirconia specimen are shown in Fig. 2. The machine polishing procedure of zirconia produced a mirror surface with a very low mean R_a ($\sim 0.02 \mu\text{m}$), compared to the alumina-particle blasting treatment ($0.61\text{--}0.64 \mu\text{m}$). However, there were no differences in the mean R_a between colored and uncolored specimens ($p=0.81$).

An ME-SBS test of the zirconia-porcelain system was successfully conducted in the present study. All the specimens ultimately failed with the detachment of the porcelain-filled alumina ring mold. SEM images of the debonded area are shown in Fig. 3. Those specimens without the zirconia liner exhibited interface (adhesive) failures between the zirconia and porcelain, but some porcelain components remained on both the alumina blasted (Fig. 3A) and highly polished zirconia surfaces (Fig. 3B). However, the particle abrasion specimens with the zirconia liner exhibited a cohesive failure in the veneer porcelain (Fig. 3C) while the polished specimens with the liner exhibited a combination of adhesive and cohesive failures (Fig. 3D).

Figure 4 shows the mean ME-SBS values between a zirconia core and a porcelain veneer, depending on the surface treatments (coloring and air abrasion) and whether a liner is applied. The mean ME-SBS value was highest in that group which was colored and alumina-blasted but which had no zirconia liner. In this case, the value was 9.6 ± 2.0 MPa. Meanwhile, group with liner, which was also uncolored and polished, marked the lowest bond strength value 7.0 ± 1.9 MPa. However, the one-way ANOVA results showed no statistically significant difference for the mean ME-SBS value ($p=0.130$). A three-way ANOVA showed that coloring of the zirconia core ($p=0.058$) and surface roughening ($p=0.584$) had no statistically significant influence on

Fig. 1 Mold-enclosed zirconia-porcelain SBS specimen (A). Schematic diagram of test setup (B).

Fig. 2 Surface roughness (R_a) values of zirconia specimens.

Fig. 3 Typical SEM images of debonded surface of zirconia-porcelain specimens: A, airborne-particle abrasion with no liner; B, high-polishing with no liner; C, airborne-particle abrasion with liner; D, high-polishing with liner. Z (zirconia), P (porcelain), and L (liner).

Fig. 4 Comparison of ME-SBS of groups according to surface treatments.

Table 2 Results of three-way ANOVA for comparison of mold-enclosed shear bond strength with coloring, roughening, application of liner, and their interactions

Source	Sum of Squares	df	Mean Square	F	p
Coloring	16.038	1	16.038	3.697	0.058
Roughening	1.312	1	1.312	0.303	0.584
Application of liner	17.392	1	17.392	4.009	0.049
Coloring×Roughening	6.084	1	6.084	1.403	0.240
Coloring×Liner	3.639	1	3.639	0.839	0.363
Roughening×Liner	5.352	1	5.352	1.234	0.270
Coloring×Roughening×Liner	5.510	1	5.510	1.270	0.263
Error	312.320	72	4.338	—	—
Total	6,145.348	80	—	—	—

the SBS of the zirconia core and porcelain veneer (Table 2). However, the application of a liner had a statistically significant influence on the ME-SBS value ($p=0.049$). All the interactions were insignificant ($p>0.05$) (Table 2).

DISCUSSION

The use of the ME-SBS test was suggested to provide a more evenly distributed interfacial stress by enclosing the adherent in a mold²³. Jin *et al.* demonstrated, using 3D finite element analysis, that the ME-SBS test setup was suitable than the conventional SBS test method for evaluating SBS in adherend-composite resin bond models²⁵. It was previously demonstrated that the ME-SBS test could provide a more valid result in metal-

cement bonding joints, relative to non-ME SBS tests²⁴. In the present study, a pure alumina ceramic ring with a significantly higher elastic modulus (~370 GPa) than that of the porcelain veneer (~70 GPa)²⁷, was used as the mold material so that the exerted shear force could be distributed over the entire zirconia-porcelain interface during the shear bond test.

Based on the three-way ANOVA test results, it can be said that the null hypothesis in that the surface treatments (coloring and air abrasion) applied to the zirconia core do not influence the ME-SBS values with a porcelain veneer was proven (Table 2). Only the application of a liner to the Zirconia resulted a slight decrease in the mean ME-SBS values with statistical significance ($p=0.049$).

The use of a zirconia liner is recommended by manufacturers for masking the innate white color and opaqueness of a zirconia core while increasing the bond strength of a porcelain veneer. A positive effect of using a liner on the bond strength can be expected in a limited range of combinations of zirconia and layered porcelain. However, the results of most previous studies on the use of a liner have actually exhibited a negative or no effect on the zirconia-porcelain bond strength^{13,15,16,28-30}, as shown in this study. In the present study, liner layers were observed to remain on the zirconia surface after the debonding test (Fig. 3C). This indicates that a debonding failure could be initiated in the liner-porcelain interface, rather than in the zirconia-liner interface²⁹.

The coloring of the zirconia ceramic may affect the strength of the bond with a porcelain veneer due to the effects of the coloring agents (which are metallic oxides). The present study found that there is little difference in the SBS value of colored and uncolored zirconia, with only small variations arising from the surface coloring, which is in agreement with the results of previous studies^{11,31}. However, the interpretation of these results should be cautious because coloring treatments of some zirconia ceramics can compromise their mechanical properties depending on the composition of the coloring oxides³²⁻³⁴, possibly resulting in a decrease in the bond strength with the veneering porcelain.

On the other hand, previous studies have produced inconsistent results regarding the effect of surface roughening on the zirconia-porcelain bond strength. Several studies reported that surface roughening of the zirconia by airborne-particle abrasion had a positive effect on the conventional SBS value^{16-18,35}. However, other studies found that the air abrasion had no effect on the SBS value^{6,13,30}, in line with the results of the present study. Moreover, the present study found that even those zirconia surfaces that had been polished to a mirror finish (R_a , 0.02 μm) produced a zirconia-porcelain bond strength that was comparable to that with a particle-abraded zirconia surface (R_a , 0.64 μm).

It is notable that the bond strengths between zirconia and porcelain can differ depending on the test method employed⁶. For example, those previous studies reporting a positive effect in the zirconia-porcelain bond strength by an air abrasion treatment have used the conventional SBS test¹⁶⁻¹⁸, with no mold-enclosing of the porcelain. However, other studies used either the Schmitz-Schulmeyer test^{6,13,36} or Schwickerath crack-initiation test³⁰ have concluded that airborne-particle abrasion had little effect on increasing the SBS between zirconia and the veneer porcelain.

The mean conventional SBS values reported for zirconia-porcelain combinations in the literature covered a range of 17–55 MPa^{4,16,37-44}. However, the mean ME-SBS values obtained in the present study ranged from 7–10 MPa, which are much lower than those for conventional SBS. The relatively wide variation in the conventional SBS values could be attributed to the effects of stress concentration and non-uniform stress resulting from a conventional shear test without mold

enclosing. The ME-SBS value determined by the present study exhibited a similar result to that of the Schmitz-Schulmeyer SBS (9–12 MPa) obtained for several zirconia-porcelain systems in a previous study⁶. The Schmitz-Schulmeyer method has been assumed to be a reliable test setup for metal-ceramic systems⁴⁵. Even with this test setup, however, a large difference in the Schmitz-Schulmeyer SBS values has been found for zirconia-porcelain systems^{36,46}.

Currently, no single bond strength test has been identified as being a de-facto standard for ceramic-ceramic systems. Although the Schwickerath crack-initiation test was adopted by the ISO 9693-2:2016 standard for zirconia-porcelain systems⁴⁷, very few studies have applied this standard to bond characterization. In the present study, the ME-SBS test was first applied to evaluate the effect of various surface treatments on the SBS of a zirconia-porcelain combination. Other variables should be further studied to demonstrate the validity of this test setup for dental ceramic-ceramic systems.

CONCLUSION

Within the design of this study, no surface treatment produced a significant increase in the zirconia-porcelain bond strength. It was found that coloring treatment and different surface roughness (R_a , 0.02 μm vs. 0.64 μm .) of the zirconia ceramic do not have a significant effect on the zirconia-porcelain ME-SBS. The application of a zirconia liner has a slightly negative effect on the ME-SBS value.

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