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Comparison of shear test methods for evaluating the bond strength of resin cement to zirconia ceramic

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Abstract

Objective. This study compared the sensitivity of three shear test methods for measuring the shear bond strength (SBS) of resin cement to zirconia ceramic and evaluated the effects of surface treatment methods on the bonding. ***Materials and methods.*** Polished zirconia ceramic (Cercon® base, DeguDent) discs were randomly divided into four surface treatment groups: no treatment (C), airborne-particle abrasion (A), conditioning with Alloy primer (Kuraray Medical Co.) (P) and conditioning with Alloy primer after airborne-particle abrasion (AP). The bond strengths of the resin cement (Multilink N, Ivoclar Vivadent) to the zirconia specimens of each surface treatment group were determined by three SBS test methods: the conventional SBS test with direct filling of the mold (Ø 4 mm × 3 mm) with resin cement (Method 1), the conventional SBS test with cementation of composite cylinders (Ø 4 mm × 3 mm) using resin cement (Method 2) and the microshear bond strength (μSBS) test with cementation of composite cylinders (Ø 0.8 mm × 1 mm) using resin cement (Method 3). ***Results.*** Both the test method and the surface treatment significantly influenced the SBS values. In Method 3, as the SBS values increased, the coefficients of variation decreased and the Weibull parameters increased. The AP groups showed the highest SBS in all of the test methods. Only in Method 3 did the P group show a higher SBS than the A group. ***Conclusions.*** The μSBS test was more sensitive to differentiating the effects of surface treatment methods than the conventional SBS tests. Primer conditioning was a stronger contributing factor for the resin bond to zirconia ceramic than was airborne-particle abrasion.

Key Words: *adhesion, airborne-particle abrasion, metal primer, microshear test, surface treatment*

Introduction

Zirconia ceramics are widely used as frameworks for all-ceramic restorations due to their good esthetics and superior mechanical properties [1]. Zirconia crowns and fixed partial dentures can be cemented with conventional cements. However, adhesion between the tooth substrates and the restoration is advocated for improving retention, the marginal seal and fracture resistance [2]. Reliable adhesion can expand the clinical applications of zirconia restorations. Hydrofluoric acid etching and silanization, which have been used successfully on silica-based ceramics, are not applicable to zirconia ceramics because zirconia ceramics do not have a glass phase or silicon dioxide [3–7]. Various surface treatment methods have been suggested to improve the bond strength of resin-based materials to zirconia [3,6,8–11]. Although there is no consensus on the most effective surface treatment for zirconia bonding, the combination of airborne-particle abrasion for micromechanical interlocking and conditioning with phosphate monomer-containing luting agents for chemical bonding has been recommended [3,4,12–14].

Surface treatment methods for enhancing the resin bond to zirconia ceramics have been evaluated through bond strength tests, in shear [4,5,8,11,14], tensile [3,12,13,15], microshear [16,17] and microtensile [6,10] modes. These test methods are based on the application of shear or tensile stresses to the bonded interface until failure occurs. The failure

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load (N) is divided by the bonded area (mm^2) to give the bond strength in MPa. Direct comparison of the data obtained in different studies is impractical due to the differences in the test methods including test protocols, specimen geometry and loading conditions. Although a superior *in vitro* bond strength value may not ensure a successful clinical result, the bond strength value is a parameter that can predict the performance of a bonding technique or system in the clinical environment.

Most studies on zirconia bonding have used shear bond strength (SBS) tests [4,5,8,11,14]. The advantages of SBS tests include the ease of specimen preparation and simplicity of the test protocol. SBS tests more closely simulate the clinical situation compared to tensile bond strength tests [18]. On the other hand, SBS tests have been criticized for non-homogeneous stress distributions at the bonded interface, inducing cohesive failures within the substrates and a misinterpretation of results [19–21]. However, cohesive failures within zirconia have rarely been reported due to the superior mechanical properties of the material [4,5].

SBS tests conducted in previous studies on zirconia bonding can be classified into three types. In the first method (Method 1), a luting cement was filled directly into a mold positioned on a zirconia specimen [14]. In the second method (Method 2), a prefabricated composite cylinder was cemented to a zirconia specimen using luting cement [4,5,8,11]. Both of these two methods used a bonding area of ~4 mm in diameter and are referred to as the conventional SBS tests. The third method (Method 3) was the microshear bond strength (μSBS) test that uses small-diameter (~1 mm) tubing as a mold [16,17]. A small testing area allows the regional mapping and many tests to be performed on the same substrate. According to Shimada and his research group [22–24], the μSBS test maximizes shear forces at the bonded interface and gives precise results with relatively small standard deviations.

To date, there is no consensus with regard to an appropriate test method for evaluating the bond strength of resin cements to zirconia ceramics. Lack of standardization in the SBS testing methods makes it difficult to compare the results of different studies. Therefore, the purpose of this study was to determine the bond strength of resin cement to zirconia ceramic after four different surface treatments by means of three SBS test methods and to compare the sensitivity of the shear test methods. The null hypotheses tested were that there would be no difference between bond strengths obtained from three SBS test methods and between them after different surface treatments of zirconia.

Materials and methods

The materials used in the present study are shown in Table I.

Zirconia disc preparation

A total of 120 zirconia ceramic discs (10 mm × 10 mm × 3 mm) were made from a partially sintered milling block (Cercon® base, DeguDent, Hanau, Germany) and then sintered according to the manufacturer's instructions. The fully sintered zirconia specimens were embedded into acrylic resin blocks and sequentially polished with up to 600-grit silicon carbide paper using a polishing machine (Rotopol-V, Struers, Ballerup, Denmark) under water cooling, followed by ultrasonic cleaning in isopropyl alcohol for 3 min. The specimens were randomly divided into four groups of 30 specimens, each according to their surface treatments (Table II). The specimens of each surface treatment group were further divided according to the three different SBS test methods (n = 10).

Conventional shear bond strength (SBS) test

The specimens for the conventional SBS tests were prepared by two different procedures. In Method 1, a polytetrafluoroethylene (PTFE) mold (4 mm in inner diameter and 3 mm in height) was placed on the zirconia disc. Resin cement (Multilink N, Ivoclar Vivadent, Schaan, Liechtenstein) was injected directly into the mold through an automix tip of the resin cement syringe. The cement was light-polymerized from four directions onto the mold for 20 s per side with an LED curing unit (Elipar FreeLight 2, 3M ESPE, St. Paul, MN). The light intensity

Table I. Materials used in this study.

Material	Composition	Manufacturer	Batch no.
Cercon® base	Zirconium oxide, yttrium trioxide, hafnium dioxide	DeguDent, Hanau, Germany	18009687
Multilink N	DMA, HEMA, barium glass, ytterbium trifluoride, spheroid mixed oxide	Ivoclar Vivadent, Schaan, Liechtenstein	R86339
Alloy primer	VBATDT, MDP, acetone	Kuraray Medical Co., Osaka, Japan	00442B

DMA, dimethacrylate; HEMA, hydroxyethly methacrylate; VBATDT, 6-(4-vinylbenzyl-*n*-propyl amino)-1,3,5-triazine-2,4-dithione; MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

Table II. Surface treatment group codes and the corresponding procedures for zirconia.

Group	Surface treatment procedure
C	No further treatment (control)
A	Airborne-particle abrasion with 50 μm aluminum-oxide (Al_2O_3) particles at 0.28 MPa for 20 s at a distance of 10 mm, followed by ultrasonic cleaning in isopropyl alcohol for 3 min
P	Conditioning with Alloy primer according to the manufacturer's instructions
AP	Conditioning with Alloy primer after airborne-particle abrasion

of 800 mW/cm^2 was frequently monitored with a radiometer (Demetron 100, Demetron Research Co., Danbury, CT). After 30 min at room temperature, the mold was carefully removed from the bonded specimen.

In Method 2, the same PTFE molds were used to fabricate composite cylinders. The mold was filled with composite resin (Filtek Z-250, Shade A3, 3M ESPE) and it was light-polymerized from four directions for 20 s per side. After polymerization, the composite cylinder was removed from the mold. Resin cement mixed through an automix tip was applied onto the composite cylinder, which was then placed on the zirconia disc under a fixed load of 10 N. Excess resin cement was removed with a microbrush and a dental explorer. After applying an oxygen-inhibiting gel (Liquistrip, Ivoclar Vivadent) around the bonded interface, the cement was light-polymerized from four directions for 20 s per side.

The bonded specimens were stored in distilled water at 37°C for 24 h before testing. Shear bond strengths were measured with a universal testing machine (LF Plus, Lloyd Instruments, Fareham, UK). A mono-angled chisel was placed as close as practically possible to the bonded interface. The shear force was applied at a crosshead speed of 0.5 mm/min until failure occurred.

Microshear bond strength (μSBS) test

In Method 3, composite resin cylinders (0.8 mm in diameter and 1 mm in height) were fabricated by filling polyethylene tubing (Tygon® R-3603 tubing, Saint-Gobain Co., Courbevoie, France) with composite resin (Filtek Z-250, Shade A3, 3M ESPE). The composite resin was light-polymerized from four directions for 20 s per side and then removed from the tubing. Mixed resin cement was applied onto the composite cylinder, which was then placed on the zirconia disc under a fixed load of 0.4 N. The luting procedure including light-polymerization was performed in the same manner described in Method 2. The bonded specimens were stored in distilled water at 37°C for 24 h before testing. For measuring bond strengths, a stainless steel orthodontic wire (diameter 0.2 mm) was used to apply a shear force to the bonded interface. The wire, which was attached to the universal testing machine, was looped around the composite cylinder as close as possible to the bonded interface. The shear force was applied at a crosshead speed of 0.5 mm/min until failure occurred.

Analysis of failure mode

The fractured interfaces of the specimens were examined with a stereomicroscope (SZ4045, Olympus Optical Co. Ltd., Tokyo, Japan) at 40× magnification to determine the failure mode. The failure mode was classified as 'adhesive failure' when it occurred at the zirconia surface. On the other hand, it was classified as 'mixed failure' when adhesive fracture and cohesive fracture within the resin cement occurred simultaneously and as a result the zirconia surface was partly covered by the remaining resin cement. Representative fractured zirconia specimens were examined using a field-emission scanning electron microscope (FE-SEM; S-4700, Hitachi High Techonologies Co., Tokyo, Japan) with an acceleration voltage of 15 kV.

Statistical analysis

The bond strength data were analyzed using statistical software (SPSS 18.0, SPSS Inc., Chicago, IL). Two-way analysis of variance (ANOVA), with one within-subject factor (surface treatment, 4 levels) and one between-subject factor (test method, 3 levels), was used to analyze the effects of the independent factors and the interaction. To interpret the main effects, one-way ANOVA was performed for the surface treatment factor within each test method and for the test method factor within each surface treatment. The Dunnett T3 test was selected for *post hoc* pairwise comparisons. The analyses were performed at a significance level of $\alpha = 0.05$.

To compare the sensitivity of the three test methods, the coefficient of variation (CV) and the Weibull parameters were used. The CV was calculated by dividing the standard deviation by the mean SBS value [25,26]. Using the Weibull distribution of the bond strength values of each test group, the Weibull modulus (m) and the characteristic strength (σ_θ) were obtained to compare the three test methods [26–28]. The σ_θ is the stress level at which 63% of the specimens have failed.

Results

The means and standard deviations of bond strength values are summarized in Table III. The two-way ANOVA showed that both the surface treatment ($F = 471.8$, $p < 0.001$) and the test method

Table III. Comparison of bond strength values (in MPa) after four different surface treatments measured with three different shear bond strength test methods.

Surface treatment	Shear bond strength test method		
	Method 1	Method 2	Method 3
C	0.5 (0.2)$^{A}_{a}$	1.3 (0.4)$^{A}_{b}$	2.3 (0.5)$^{A}_{c}$
A	3.5 (0.7)$^{B}_{a}$	4.7 (0.7)$^{B}_{b}$	5.9 (1.0)$^{B}_{c}$
P	4.5 (0.7)$^{B}_{a}$	4.9 (0.6)$^{B}_{a}$	24.6 (3.5)$^{C}_{b}$
AP	8.1 (1.6)$^{C}_{a}$	9.8 (1.4)$^{C}_{a}$	29.7 (3.5)$^{D}_{b}$

The numbers in the parentheses are standard deviations.
Uppercase letters with same superscript show no significant differences between the surface treatments for the same test method. Lowercase letters with the same subscript show no significant differences between the test methods for the same surface treatment.
Group codes: C, control; A, airborne-particle abrasion; P, conditioning with Alloy primer; Method 1, conventional shear bond strength test with direct filling of the mold with resin cement; Method 2, conventional shear bond strength test with cementation of the cured composite cylinder using resin cement; Method 3, microshear bond strength test with cementation of the cured composite cylinder using resin cement.

(F = 600.1, p < 0.001) significantly influenced the bond strength values (Table IV). There was also a significant interaction between the surface treatment and the test method (F = 145.1, p < 0.001). As shown in Table III, Method 3 resulted in significantly higher bond strengths than Methods 1 and 2 for each surface treatment (p < 0.05). The AP groups exhibited the highest bond strengths regardless of the test methods. There was no significant difference in mean SBS values between the P group and the A group in Methods 1 and 2. In contrast, the P group exhibited significantly higher bond strength than the A group in Method 3 (p < 0.05).

The distribution of failure modes in the test groups is presented in Figure 1. With surface treatments C and A, all of the specimens failed adhesively at the zirconia surface regardless of the test methods. With surface treatment AP, 40%, 30% and 30% of the specimens were classified as mixed failure with Methods 1, 2 and 3, respectively. Figures 2 and 3 show representative SEM images of the zirconia surface after bond strength tests.

Table IV. Two-way ANOVA results of bond strength data after four different surface treatments measured with three different shear bond strength test methods.

Source	Sum of squares	df	Mean squares	F	p-value
Surface treatment	3818.0	3	1272.7	471.8	< 0.001
Test method	3237.8	2	1618.9	600.1	< 0.001
Between	2348.3	6	391.4	145.1	< 0.001
Error	291.4	108	2.7		
Total	18020.5	120			

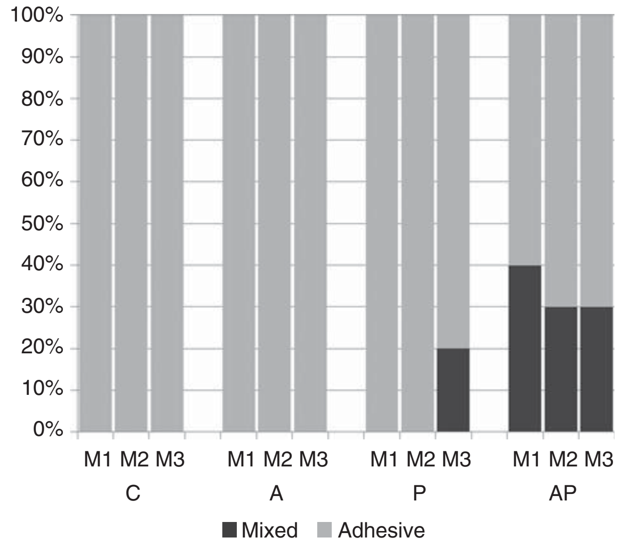

Figure 1. Failure mode distribution after three different shear bond strength tests. Group codes: C, control; A, airborne-particle abrasion; P, conditioning with Alloy primer; M1, conventional shear bond strength test with direct filling of the mold with resin cement; M2, conventional shear bond strength test with cementation of the cured composite cylinder using resin cement; M3, microshear bond strength test with cementation of the cured composite cylinder using resin cement.

In each test method, the AP group exhibited the highest σ_θ, followed in order by the P group, the A group and the C group (Table V). These results were in line with the distribution of mean SBS values. For each surface treatment, Method 1 resulted in a higher CV and lower m than Methods 2 and 3. Methods 2 and 3 showed very similar CV values. In Methods 1 and 2, the AP groups showed a higher CV and lower m than the P groups, although the AP group showed the highest mean SBS and σ_θ. In contrast, in Method 3, the AP group showed the lowest CV and the highest m, which was in accordance with the highest mean SBS and σ_θ.

Discussion

A variety of surface treatment methods, such as a vapor phase deposition technique [6], glass micro-pearls [8], selective infiltration etching [10] and plasma spraying [11], have been proposed to improve the resin bond to zirconia. However, for practical use of these methods, improvement of equipment and simplification of the process are required. Tribochemical silica coating has been widely used to produce a silica layer on non-silica based materials and allows silane coupling agents to be employed [7]. Some studies have presented favorable results on initial bond strength to silica-coated zirconia, but the bond strength was significantly reduced after artificial aging [3,29,30]. It has been assumed that tribochemical silica coating may not produce a uniform silica layer firmly attached to the

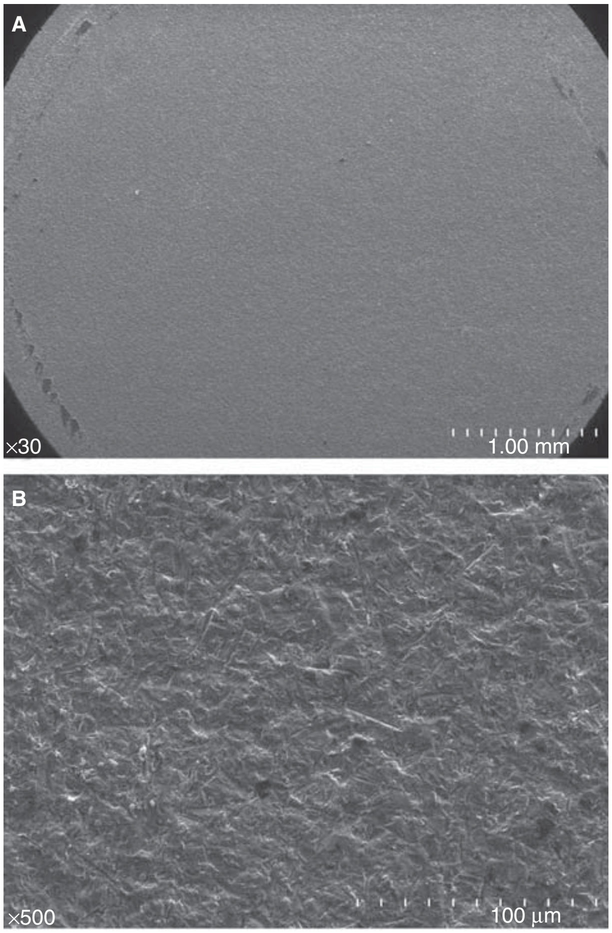

Figure 2. Representative scanning electron microscopic image of adhesive failure. This specimen was selected from the A group tested with Method 1. (A) The zirconia surface was exposed without any resin cement remnants after fracture. (B) The high magnification image of the specimen showing the typical topography of air-abraded zirconia after adhesive failure. Group codes: A, airborne-particle abrasion; Method 1, conventional shear bond strength test with direct filling of the mold with resin cement.

zirconia surface. In addition, the siloxane bond is sensitive to hydrolytic degradation [7]. In contrast, resin cements and primers containing phosphate monomers have been shown to provide a long-term durable bond to air-abraded zirconia [3,4,12–14]. For this reason, airborne-particle abrasion and an MDP-containing primer were selected as surface treatment methods in the present study.

Alloy primer (Kuraray Medical Co., Osaka, Japan) was originally designed for enhancing the bond between resin-based materials and dental metal alloys. This primer contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and 6-(4-vinylbenzyl-*n*-propyl amino)-1,3,5-triazine-2,4-dithione (VBATDT). MDP chemically bonds to non-precious metals and helps the reaction of VBATDT with precious metals [31]. Alloy primer was selected for use in the present study because it had exhibited superior results with regard to zirconia bonding compared to other phosphate monomer-containing primers [13–15].

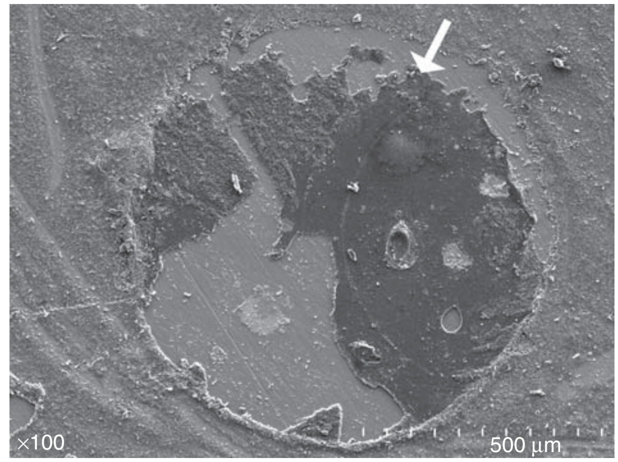

Figure 3. Representative scanning electron microscopic image of mixed failure. This specimen was selected from the P group tested with Method 3. The arrow shows the direction of shear force. The resin cement remained on the side of the loading point. At the opposite side of loading, the fracture did not propagate into the zirconia substrate because of its superior mechanical properties. Group codes: P, conditioning with Alloy primer; Method 3, microshear bond strength test with cementation of the cured composite cylinder using resin cement.

In the present study, both the test method and the surface treatment influenced the bond strength values. Therefore, the null hypotheses were rejected. Method 2 presented significantly higher SBS values than Method 1 for surface treatments C and A. The bonded areas in Methods 1 and 2 were identical. However, the resin cement was passively applied on the zirconia specimen in Method 1, whereas it was under even pressure during the luting procedure in Method 2. The pressure may have promoted the adaptation of the resin cement to the zirconia ceramic, resulting in higher SBS values in Method 2 [32]. The difference in the results of the two methods can also be attributed to the thickness of the resin cement. The entire mold was filled with resin cement in Method 1, whereas in Method 2 it formed a layer between the composite cylinder and the zirconia disc. The thickness of the resin cement can be considered infinite in Method 1. The excessive thickness of a luting agent has an unfavorable influence on the bond strength between a restoration and the substrate due to its inferior mechanical properties and high polymerization shrinkage [33]. Method 2 is considered to be more representative of the clinical situation in which resin cement exists in a thin layer between a restoration and tooth substrate.

Method 1 presented higher CV and lower *m* values than Methods 2 and 3 for each surface treatment. The passive application of resin cement in Method 1 may have caused uneven adaptation of the resin cement to the zirconia specimen, thus resulting in relatively high variations as well as lower bond strength values. In addition, luting the prefabricated composite cylinders with resin cement was easier to control, which may

Table V. Coefficient of variation (CV), Weibull modulus (m) and characteristic strength (σ_θ in MPa) of the shear bond strength values of four different surface treatment groups measured with three different shear bond strength test methods.

	Shear bond strength test method								
	Method 1			Method 2			Method 3		
Surface treatment	CV	m	σ_θ	CV	m	σ_θ	CV	m	σ_θ
C	0.40	2.89	0.59	0.26	4.38	1.47	0.22	5.26	2.53
A	0.21	5.59	3.81	0.15	8.19	4.95	0.16	7.10	6.32
P	0.16	6.99	4.76	0.12	9.91	5.19	0.14	8.22	26.07
AP	0.20	5.59	8.74	0.14	7.95	10.44	0.12	10.07	31.21

Group codes: C, control; A, airborne-particle abrasion; P, conditioning with Alloy primer; Method 1, conventional shear bond strength test with direct filling of the mold with resin cement; Method 2, conventional shear bond strength test with cementation of the cured composite cylinder using resin cement; Method 3, microshear bond strength test with cementation of the cured composite cylinder using resin cement.

have contributed to the more consistent results obtained from Method 2. Therefore, the SBS test methods with the luting procedures, in which pre-fabricated composite cylinders are cemented to zirconia specimens using resin cement, are considered more reproducible and reliable.

Method 3, the μSBS test, resulted in significantly higher bond strength values than Methods 1 and 2 for each surface treatment. The main characteristic of the μSBS test is the testing of a smaller area compared to the conventional SBS test. According to the study on the microtensile bond strength (μTBS) test, the bond strength was inversely related to the bonded area [34]. Brittle materials, such as ceramics and composite resins, fail due to the progression of existing flaws when subjected to stresses above a critical level [35]. The small bonded area contains fewer defects and, thus, results in a higher bond strength value. This principle also explains why the μSBS test resulted in higher bond strength values than the conventional SBS tests.

Adhesive failures were primarily observed in all test groups, which was in accordance with previous studies on zirconia bonding [4,5]. Cohesive failures within zirconia were not observed in the present study. These findings implied that the bonded interface between the resin cement and zirconia ceramic was the weakest link. The P group presented 20% mixed failure in Method 3. The AP groups presented an average of 33% mixed failure in accordance with their highest mean SBS. The mixed failures can be attributed to the resulting deviation of stress propagation. The resin cement remained mainly on the side of the loading point in the mixed failure specimens (Figure 3). Under shear loading, tensile stress occurred at the loading side, whereas compressive stress occurred at the opposite side of the loading point [19–21]. At the tension side, the fracture could start within the resin rather than between the resin cement and zirconia ceramic. At the opposite side from where it was loaded, the change in stress from tension to compression guided the progress of the crack toward the zirconia ceramic. However, the crack did not propagate into the zirconia ceramic because of its superior mechanical properties. When SBS tests are conducted on zirconia ceramics, problems related to cohesive failures within weak substrates such as dentin can be avoided.

According to the Weibull statistics, a high m indicates that the flaw population is consistent and the bonding procedure is reliable [27,28]. If the flaw population is consistent in the specimens within a group, m (reliability) will increase with the increase of σ_θ (probability of failure for a given stress level), reflecting a relative decrease in the variation within the group, that is, low variability and low spread in bond strength with high reliability of σ_θ. Therefore, in addition to the CV and mean bond strength, both of the Weibull parameters m and σ_θ should be considered in evaluating the sensitivity and reliability of the test methods. The scales of the Weibull parameters are wider than those of the CV and mean bond strength and, thus, the parameters are more discernible. In this study, comparing the CV and m of the four surface treatment groups, Method 3 showed the lowest CV with the highest mean SBS in accordance with the highest m and σ_θ (Table V). Therefore, Method 3 is considered the most sensitive and reliable test method in terms of the consistency of specimens. This assumption is supported by previous studies conducted on enamel, where the μSBS test showed advantages in differentiating the performance of dental adhesive systems and providing consistent results without the premature failures that frequently occurred in the μTBS test [36,37]. However, it was difficult to fix the small-diameter tubing firmly on the specimen without disrupting the subsequent bonding procedure. This was why the pre-fabricated composite cylinders were used in the present study instead of directly filling the tubing with resin cement.

Although we focused on the SBS test methods, the μTBS tests have been employed to evaluate the bond strength of resin cements to zirconia ceramics in some studies [6,10]. The μTBS tests allow for a more homogeneous distribution of stress and evaluation of the bond strength of a small region of interest within the substrates. The tests have been widely used to evaluate the adhesion to tooth substrates [38]. However, premature failures frequently occur during the fabrication procedures of the μTBS test specimens. Cutting the specimens into microbeams is a technically sensitive and labor-intensive step. The cutting procedure can cause microcracks in brittle materials like ceramics [39]. It is very difficult to cut zirconia ceramics into microbeams without damaging the specimens because of the superior mechanical properties of zirconia ceramics. In a previous study [40] that compared the conventional SBS test with the μTBS test, the test method did not significantly affect the bond strength results of resin cement to glass-infiltrated alumina-zirconia ceramic. In the present study, the μSBS test allowed for the differentiation between the effects of surface treatment methods with low standard deviations and primarily adhesive failures at the bonded interface. Therefore, the μSBS test seems to be appropriate for screening the performance of surface treatment methods with simple and reproducible procedures.

Regardless of the test methods, the AP groups exhibited the highest bond strength values in accordance with previous studies [3,4,12–14]. Airborne-particle abrasion increases the surface roughness, thereby improving micromechanical retention to zirconia ceramics. The application of phosphate monomer-containing primers improves the wettability of the surface and produces a chemical bond between resin cement and zirconia ceramic [9]. On the other hand, there has been controversy over which one of these two surface treatment methods contributes more to the resin bond to zirconia [13,14,16]. The P group exhibited significantly higher bond strength than the A group in Method 3, which was more sensitive to the effects of surface treatments. Based on this finding, the resin bond to zirconia may rely mainly on chemical bonds rather than micromechanical retention. This assumption is supported by previous studies that showed no correlation between surface roughness and bond strength [13,41]. Airborne-particle abrasion may not make the zirconia surface rough enough to provide strong micromechanical retention because of the superior physical properties and surface hardness of zirconia ceramics. However, the results should be interpreted with caution. Commercial zirconia ceramics can differ in physical properties and surface hardness and the effect of the surface treatments can be dependent on the materials. In addition, long-term studies have shown that the application of a primer was not sufficient to be used alone and the combination with airborne-particle abrasion was required for a durable bond to zirconia [13,14].

Within the limitations of the present study, the SBS test methods with the luting procedure, in which pre-fabricated composite cylinders were cemented using resin cement, were more reliable and reproducible for evaluating the bond strength of resin cement to zirconia ceramic. The μSBS test was more sensitive and, thus, better able to differentiate the effects of surface treatment methods than the conventional SBS tests. Although it seems that the resin bond to zirconia relies mainly on chemical bonds, the combination of airborne-particle abrasion and phosphate monomer-containing primers is recommended for improving the bond strength of resin cements to zirconia ceramics.

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