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Determination of Resin Bond Strength to Zirconia Ceramic Surface Using Different Primers

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Abstract

Objective. To evaluate the effect of different primers on the bond strength of adhesive resin cement to zirconia ceramic surface *in vitro*. ***Material and methods.*** Thirty zirconia disk-shaped ceramic specimens (10×2 mm^2) were divided into three equal groups for the application of different primers: Monobons-S, Clearfil Ceramic Primer and Signum Zirconia Bond. A further 10 specimens served as a control group. Forty composite resin blocks (6×2 mm^2) were cemented (Panavia F 2.0) to zirconia ceramic specimens. Shear bond strengths were measured. Treated zirconia ceramic surfaces were examined using scanning electron microscopy. ***Results.*** One-way ANOVA showed that the application of different primers affected the shear bond strength of adhesive resin cement to zirconia ceramic. The highest test values were obtained with application of Signum Zirconia Bond, and the lowest values were obtained in the control group. ***Conclusion.*** Signum Zirconia Bond is effective for increasing the bond strength of adhesive resin cement to zirconia ceramic.

Key Words: *Air abrasion, bond strength, silane, surface treatment, zirconia ceramic*

Introduction

In clinical situations that require highly demanding esthetic restorations, all-ceramic restorations have the potential to be a more effective selection compared to metal–ceramic restorations [1]. Thus there is growing interest in all-ceramic restorations and materials. One of the most commonly used all-ceramic core materials is yttrium tetragonal zirconia polycrystal (Y-TZP; zirconia) [2], because of its favorable esthetic characteristics, mechanical properties, and biocompatibility [3]. The introduction of zirconia frameworks extended the design and application limits of all-ceramic restorations, leading to improved success and reliability [4]. With the development of computer-aided design–computer-aided manufacturing (CAD-CAM) technology, the design and production of zirconia frameworks could be achieved using a digital process [3,5]. Therefore, restorations using a zirconia framework became more practical [5].

Zirconia ceramics can be cemented with conventional (zinc phosphate or glass ionomer) or adhesive resin cements [6–11]. Improvement of marginal adaptation, prevention of microleakage, and increased fracture resistance have made resin cements more popular in dental practice [12]. Previous studies showed that conventional bisphenol A glycidyl methacrylate (bis-GMA)-based composite resins without adhesive monomers were unable to form long-term bonds to zirconia ceramic [13–15]. However, 10-methacryloyloxy-decyl-dihydrogenphosphate (MDP)-containing composite resins showed a long-term durable bond to zirconia ceramic after airborne particle abrasion [4,16].

A strong, durable resin bond to ceramics is established by the formation of chemical bonds and by micromechanical interlocking [3]. In order to increase the bond strength of resin materials to ceramic restorations, many surface treatment methods and cementation procedures with different resin cements have been evaluated. These surface treatment methods comprise grinding, abrasion with diamond rotary instruments, airborne particle abrasion with aluminum oxide (Al_2O_3), silica-coating (CoJet, Rocatec), acid etching with hydrofluoric acid, coupling with silane and combinations of any of these methods [6,11,17]. Etching with hydrofluoric acid is

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recommended only for surfaces with a glassy component, but it has no influence on zirconia ceramics, where no micro-grooves will be created [18]. Airborne particle abrasion with Al_2O_3 abrasive particles has been identified as an effective factor in achieving a stable, durable bond for alumina and zirconia-based ceramics [3]. However, airborne particle abrasion may have a deleterious effect on the treated surface by creating microcracks, which may reduce the fracture strength of the ceramics [3].

Silanes have been used in recent years in dental practice in order to form a siloxane network between the resin-luting cement and ceramic surface to improve bonding strength [19]. Although the most frequently used silane-coupling agents represent an effective conditioning method for bonding resin composite to conventional silica-based dental ceramics, these agents showed an unsatisfactory resin bond to oxide-based dental ceramics. It has also been suggested that silanization may increase the wetting ability of the oxide ceramic surface, thereby resulting in small increases in bond strength values [20,21]. The strength of the resin–zirconia bond achieved using selective infiltration etching could be further improved with specially engineered zirconia primers. In addition to methacrylatesilane, some other reactive functional silanes have recently been evaluated for bonding resins and luting cements to silica-coated titanium and zirconia [21–23].

There have been reports of bond strengths of various types of ceramics and composites that have been tested with various adhesive systems. However, information is lacking on shear bond strengths of adhesive resin cements to zirconia ceramics and, in particular, few primer comparisons have been reported [13,22,24].

The aim of this study was to evaluate the effect of different primers on the bond strength of adhesive resin cement to a zirconia ceramic surface *in vitro*. The proposed hypothesis was that different primers may produce different bond strength values.

Material and methods

The materials used and their corresponding batch numbers, compositions and manufacturing information are presented in Table I.

Forty zirconia core specimens (10-mm diameter, 2-mm thickness) were produced by a copy-milling system (Zirconzahn, Bruneck, Italy) using prefabricated blanks of zirconia (ICE Zircon Translucent; Zirconzahn) and then sintered according to the manufacturer's instructions. The surfaces were cleaned with ethanol and carefully air-dried before surface treatment (Branson 2210; Branson Ultrasonics Corporation, Danbury, CT). Zirconia cores were embedded in the centers of autopolymerizing acrylic resin blocks (Meliodent; Heraeus Kulzer, Armonk, NY). Zirconia core surfaces were ground-finished with a 600-grit silicon carbide abrasive (3M ESPE, St. Paul, MN) under running water on a polishing machine (Buehler Metaserv, Buehler, Germany), ultrasonically cleaned (Bransonic B221; Branson Ultrasonics Corporation) for 3 min in deionized water and then wiped with 95 vol.% ethanol. Airborne particle abrasion was applied to all specimens using 110-µm Al_2O_3 particles (Korox 110 µm; BEGO, Bremen, Germany) at an air pressure of 2.8 bar using an air abrasion device (BEGO TopTec 4) for 15 s at a distance of 10 mm.

Subsequently, specimens were randomly divided into four groups, each containing 10 specimens, for the following different surface treatment methods. One of the groups (Group C) served as a control and no surface treatment was applied. The other groups were as follows. In Group MS, specimens were treated with methacyloxypropyltrimethoxysilane (MPS) containing a silane-coupling agent (Monobond-S) and then dried with oil-free air for 60 s. In Group CP, specimens were treated with MPS- and MDP-containing ceramic primer (Clearfil Ceramic Primer) and dried with oil-free air. In Group ZB, specimens were treated with a bifunctional

Table I. Materials used in the study.

Material	Type of material	Batch no.	Manufacturer
Ice Zircon	Yttrium partially stabilized with tetragonal polycrystalline structure (Y-TZP)	ZA9110M	Zirconzahn SRL, Bruneck, Italy
Korox 110	99.6% 110-µm alumina oxide particles	681128	BEGO, Bremen, Germany
Monobond S	1% 3-MPS, ethanol-/water-based solution	G15672	Ivoclar-Vivadent, Schaan, Liechtenstein
Clearfil Ceramic Primer	3-MPS, 10-MDP, ethanol	005BA	Kuraray, Okayama, Japan
Signum Zirconia Bond	Bond I: Acetone, 10-MDP, acetic acid	010106	Heraeus Kulzer, Milan, Italy
	Bond II: Methyl methacrylate, diphenyl(2,4,6- trimethylbenzoyl)phosphinoxide	010101	
Panavia F 2.0	BPEDMA, MDP, DMA, Ba–B–Si glass, silica, chemical and photo-initiators	41233	Kuraray, Okayama, Japan

MPS = methacyloxypropyltrimethoxysilane; BPEDMA = bisphenol-A-polyethoxy dimethacrylate; DMA = aliphatic dimethacrylate; MDP = 10-methacryloyloxy-decyl-dihydrogenphosphate.

methacrylate-based component containing acetone and MDP (Signum Zirconia Bond I) and dried with oil-free air. A bonding agent containing methyl methacrylate and diphenyl (2,4,6-trimethylbenzoyl) phosphinoxide (Signum Zirconia Bond II) was then applied to the ceramic surface with a disposable brush and light-cured for 90 s in a polymerization unit (Heraflash; Heraeus Kulzer, Germany).

Forty composite resin cylinders (Filtek Z250; 3M ESPE, Germany) were made using a polytetrafluoroethylene mold (Isoflon; Diemoz, France) with a hole in the center (6-mm diameter, 2-mm thickness). Composite resin was placed into the mold and incrementally condensed to fill it up and each layer was light-polymerized for 40 s at a distance of 1 mm using a light-polymerizing unit (Astralis 3; Ivoclar Vivadent, Schaan, Liechtenstein) with an output power of 600 mW/cm^2. After removing the composite cylinders from the mold, an additional 40 s of polymerization was performed on the portions that were previously in contact with the mold.

Composite cylinders were bonded to treated ceramic surfaces using a dual-polymerized adhesive resin cement (Panavia F 2.0; Kuraray Co Ltd., Osaka, Japan) containing the adhesive phosphate monomer (APM) according to the manufacturer's recommendations. Each composite cylinder was bonded to zirconia core specimens under a load of 500 g. Foam pellets and microbrushes were used to remove excess resin. The specimens were light-cured for 40 s from all sides with a curing light (Astralis 3). A glycerin gel (Oxyguard; Kuraray Co Ltd) was applied to the cement layer for 10 min. Specimens were washed with an air–water spray and then stored in distilled water at 37°C for 24 h before testing.

The specimens were mounted in the jig of a universal test machine (Lloyd LRX; Lloyd Instruments PLC., Fareham, UK) and a load was applied to the adhesive interface until failure occurred (crosshead speed 1.0 mm/min). The shear bond strength values were calculated in megapascals (MPa) by dividing the failure load (N) by the area of the composite resin ($N/\pi r^2$).

Debonded specimen surfaces were examined by the same observer using a stereomicroscope (Leica MZ12; Leica Microsystems, Bensheim, Germany) at a magnification of ×40 to assess the mode of failure. Adhesive and/or cohesive failure of bonding could occur at three locations: (1) adhesive failure at the interface between the ceramic and resin-luting agent or between the resin-luting cement and composite interface; (2) cohesive failure within the ceramic, within the resin-luting cement or within the composite resin only; and (3) adhesive and cohesive failure at the same site, or mixed failure [6].

Data were statistically analyzed using the SPSS 12.0 software program (SPSS Inc., Chicago, IL). The Kolmogorov–Simirnov test showed that the data followed a normal distribution ($P > 0.05$). A homogeneity of variance test was done using Levene's test ($F = 2.869$, $P < 0.05$). Means and standard deviations (SDs) of bond strengths were calculated and mean values were compared by one-way ANOVA (SPSS 12.0; SPSS Inc., Chicago, IL), followed by a multiple-comparisons test performed using a post-hoc Tukey test ($\alpha = 0.05$).

To evaluate the effect of surface treatment methods, five additional samples were prepared. Four specimens were treated with the same experimental protocol as described above. The other specimen was ground-finished with 600-grit silicon carbide abrasive. All specimens were coated with gold using a sputter coater (S150B; Edwards, Crawley, UK) and examined under a field emission scanning electron microscope (JSM-6335F; JEOL, Tokyo, Japan) at 20 kV. The scanning electron microscopy (SEM) images were developed at a magnification of ×500 for visual inspection.

Results

The results of the one-way ANOVA revealed a significant difference between the groups ($P < 0.001$) (Table II). The shear bond strength depended on the type of primer. The mean shear bond strength values and the differences between the groups are shown in Table III.

The highest shear bond strength values were obtained in Group ZB (54.9 ± 9.4 MPa). No significant differences were found between Groups MS (40.6 ± 7.4 MPa) and CP (43.5 ± 5.0 MPa). The lowest shear bond strength values were obtained in Group C (30.1 ± 4.0 MPa).

SEM images of treated zirconia core surfaces are shown in Figure 1. When the SEM images were evaluated, the sandblasted surface of the zirconia core showed irregularities (Figure 1A). Treatment with Monobond-S did not change the superfacial structure when compared with the sandblasted surface (Figure 1B). Application of Clearfil Ceramic Primer showed a similar surface structure when compared with the Monobond-S application (Figure 1C). No surface irregularities were observed in the zirconia core surface treated with Zirconia Bond (Figure 1D). The surface of the zirconia core treated with Zirconia Bond was completely covered with irregularities.

Table II. Result of one-way ANOVA test.

	Sum of squares	df	Mean square	*F*	*P*
Between groups	2728.70	3	909.57	19.63	0.001
Within groups	1482.63	32	46.33		
Total	4211.33	35			

Table III. Mean and standard deviation (SD) of shear bond strength values[a].

		Shear bond strength (MPa)	
Group	Surface treatment	Mean	SD
C	Control	30.5^a	3.9
MS	Monobond-S	40.6^b	7.4
CP	Clearfill Ceramic Primer	43.5^b	5.0
ZB	Signum Zirconia Bond	54.9^c	9.4

[a,b,c]Identical letters indicate no statistically significant difference ($P > 0.05$).

The greatest number of adhesive failures occurred in Group C and the greatest number of cohesive failures in Group ZB. The percentage failure rates of the test specimens are listed in Table IV.

Discussion

The results of this study confirm our hypothesis that different primers produce different shear bond strength values. The bond strength was found to be significantly higher in Group ZB when compared with Groups C, MS, and CP, indicating that application of an MDP-containing silane will improve the bonding on zirconium dioxide ceramic restorations.

Resin-luting cements containing MDP can bond strongly to sandblasted zirconia [2,8,13]. Reactions may occur between hydroxyl groups in the MDP and on the zirconia ceramic surface, similar to the chemical effects reported as the result of bonding of a phosphate ester monomer to metal oxides such as chromium, nickel, aluminum, and zirconium dioxides [2,8].

Silane-coupling agents, or more precisely trialkoxysilanes, are hybrid inorganic–organic bifunctional molecules and they play a recognized (and remarkable) role by way of their use as adhesion-promoting agents. Typical silane-coupling agents contain an organofunctional part and three hydrolyzable alkoxy groups. Before becoming adhesion promoters, and in order to be activated, trialkoxysilanes must undergo a hydrolyzation reaction in a slightly acidic ethanol–water solvent to form silanols from trialkoxy groups. The organofunctional part, most often a methacrylate group, can then be polymerized with the monomers of a resin composite system. Silane-coupling agents lower the surface tension of a substrate, wet it and make its surface energy higher, making it accessible for effective bonding. Thus a hydrophobic matrix (resin composite) can adhere to hydrophilic surfaces, such as silica, glass, and glass-ceramics [22].

The monomeric ends of the silane molecules react with the methacrylate groups of the adhesive resins by means of a free radical polymerization process. Conventional silanes are not as effective with high-strength ceramics such as alumina and zirconia, as these ceramics are more chemically stable than silica-containing glasses and ceramics and not as easily hydrolyzed [9,22].

Although higher bond strengths were obtained in Group CP than Group MS for 3-MPS-containing silanes in this study, no significant differences were found. Matinlinna et al. [22] evaluated the effects of three different silane solutions on zirconia surfaces

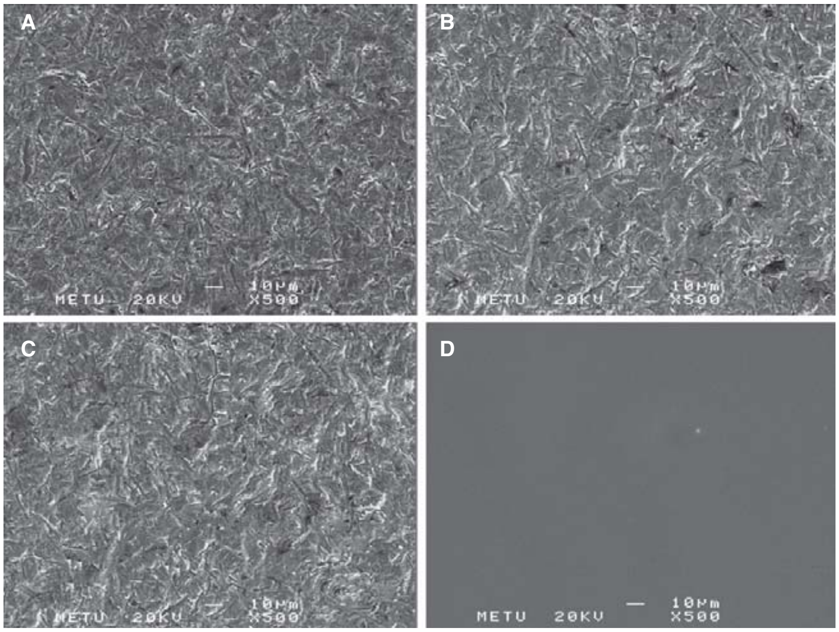

Figure 1. SEM images of the zirconia core surface after application of different primers: (A) control (110-µm Al_2O_3); (B) Monobond-S; (C) Clearfill ceramic primer; (D) Signum Zirconia Bond.

Table IV. Failure rate (%) of tested specimens.

Group	Adhesive	Cohesive	Adhesive + cohesive
C	60	30	10
MS	40	40	20
CP	30	50	20
ZB	20	60	20

and concluded that bonding of a resin cement to a silica-coated zirconia is effective with 3-MPS or 3-acryloyloxypropyltrimethoxysilane, but not with 3-isocyanatopropyltriethoxysilane. Aboushelib et al. [5] also evaluated the effects of five different silane solutions on zirconia surfaces and concluded that significant differences between the primers were observed, indicating that these primers react differently to the same zirconia surface.

The results of the present study are consistent with those of Blatz et al. [18] and Atsu et al. [6], who demonstrated that application of an MDP-containing bonding/silane-coupling agent mixture to zirconium dioxide ceramic restorations abraded with airborne Al_2O_3 particles can yield a superior shear bond strength. These results may be explained by the improved wettability of a rough zirconia surface due to the mixture of the bonding/silane agent and the presence of chemical bonding. Moreover, some authors reported that an MDP-containing resin-luting agent and an MDP-containing bonding/silane-coupling agent mixture provided a strong resin bond to airborne particle-abraded zirconia and aluminum oxide ceramic restorations [6]. However, a strong resin–ceramic bond achieved in strictly controlled, clean *in vitro* tests might be compromised in clinical situations, leading to significantly reduced bond strength. During clinical test procedures, contamination of restorative luting surfaces by saliva, blood or silicon–silicon indicators cannot be avoided. Saliva contamination is frequently the main reason for reduced resin bond strength. Also, Yang et al. [16] reported that the activated ceramic surface is sensitive to the environment and will partially lose its wettability because of air contamination. In dental textbooks, organic solutions are recommended for the removal of saliva contamination of luting surfaces of restorations before cementation [25]. In the instructions for modern adhesive composite resins, phosphoric acid gel is sometimes recommended for the removal of contaminants. In addition, application of acetone and alcohol to the ceramic surface is generally available in dental offices [16].

In the present study, the highest bond strength values were obtained in Group ZB. This can be related to differences in the chemical composition of the primers. Signum Zirconia Bond I contains acetone and MDP and Signum Zirconia Bond II contains 50–75% methyl methacrylate. As previously mentioned, application of acetone has a positive effect on the air contamination of a ceramic surface [16]. Yang et al. [16] reported that immersion in acetone for 15 s increased the strength of the bond between the resin cement and the zirconia ceramic surface. Our research confirmed these results. The acetone content of Signum Zirconia Bond I may increased the surface wettability of the ceramic surface. In addition, methyl methacrylate in the primer and methacrylates in the resin cement can be polymerized with the monomers of a resin composite system, so that bonding between the resin and the ceramic surface might have been increased.

In several studies of zirconia ceramic bonding, air abrasion with Al_2O_3 particles is used to condition the ceramic surface to increase the surface roughness [3,7,26–28]. The methods used to increase micro-mechanical retention to silica-based ceramics, such as application of phosphoric or hydrofluoric acid, do not produce an acceptable surface roughness for high-strength ceramics [4]. Different sizes of abrasive particles between 50 and 110 μm are usually used [3,6,18]. In the present study, 110-μm Al_2O_3 particles were used to provide micro-mechanical retention.

Several test methodologies, namely shear, tensile, and microtensile tests, have been suggested for evaluation of the bond strength of resin-based materials to dental ceramics [29]. Each test method has its suite of advantages and disadvantages [29]. Some advantages of the shear bond strength test method include ease of specimen preparation, the clear test protocol, and rapid production of test results. On the other hand, microtensile tests allow better alignment of the specimens and a more homogenous distribution of stress, in addition to a more sensitive comparison or evaluation of bond strengths [30]. However, Valandro et al. [29] reported that the cutting procedure of high-strength ceramic–resin assemblies is much more complicated and time-consuming than cutting a glass ceramic bonded to resin materials. The stress generated during cutting can affect the adhesive interface and consequently reduce the microtensile bond strength.

In the present study, shear bond tests were used. It can be questioned whether tension tests are more appropriate for evaluating the adhesive capabilities of resin agents to ceramics [17]. However, the study did not intend to determine absolute bond strength values but to evaluate whether the pre-treatments used produced dramatic differences in bond strength.

The effects of thermal cycling and long-term storage on bond strength were not evaluated and may be considered limitations of this study. However, in a previous study, Lindgren et al. [27] reported that the shear bond strengths of RelyX Unicem or Panavia F resin composite cements to a zirconia ceramic surface were not significantly affected by storage in water for

1 week, thermocycling after 24 h of aging in water, or air abrasion.

The clinical implication of this study is that the bond strength between zirconia ceramic and a resin-luting agent is affected by the ceramic surface treatment, which is dependent on the chemical nature of the primers. Further research is needed with other commercial silanes and metal primers, with and without surface conditioning. Also, the durability of these ceramic-to-composite chemical bonds should be further evaluated.

Conclusions

(1) The difference in chemical structure of each primer has an effect on the bonding between the zirconia ceramic surface and resin cement.
(2) High bond strength values were obtained with MDP-containing primers.
(3) The highest bond strength values were obtained with the application of Signum Zirconia Bond. The presence of methyl methacrylate in the primer may affect the bond strength.

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