

Influence of Glazed Zirconia on Dual-Cure Luting Agent Bond Strength

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Clinical Relevance

Treatment of yttrium-stabilized tetragonal zirconia ceramic surfaces with a low-fusing porcelain layer as a glaze significantly increased the bond strength of dual-cure resin cement to the ceramic surface.

SUMMARY

The current study evaluated the influence of a novel surface treatment that uses a low-fusing porcelain glaze for promoting a bond between zirconia-based ceramic and a dual-cure resin luting agent. Bond strengths were compared with those from airborne particle abrasion,

hydrofluoric acid etching, and silanization-treated surfaces. Twenty-four yttrium-stabilized tetragonal zirconia (Cercon Smart Ceramics, Degudent, Hanau, Germany) discs were fabricated and received eight surface treatments: group 1: 110 μm aluminum oxide airborne particle abrasion; group 2: 110 μm aluminum oxide airborne particle abrasion and silane; group 3: 50 μm aluminum oxide airborne particle abrasion and silane; group 4: 50 μm aluminum oxide airborne particle abrasion and silane; group 5: glaze and hydrofluoric acid; group 6: glaze, hydrofluoric acid, and silane; group 7: glaze and 50 μm aluminum oxide airborne particle abrasion; and group 8: glaze, 50 μm aluminum oxide airborne particle abrasion and silane. After treatment, Enforce resin cement (Dentsply, Caulk, Milford, DE, USA) was used to fill an iris cut from microbore Tygon tubing that was put on the ceramic surface to create 30 cylinders of resin cement in each treatment group ($n=30$). Microshear bond testing was performed at a crosshead speed of 0.5 mm/min. One-way analysis of variance, and multiple comparisons were made using Tukey's test ($p<0.5$). The bond strength was affected

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only by surface treatments other than silanization. The groups that utilized the low-fusing porcelain glaze with airborne particle abrasion or hydrofluoric acid showed bond strength values statistically superior to groups that utilized conventional airborne particle abrasion treatments with 50 or 110 μm aluminum oxide ($p < 0.001$). The treatment that utilized low-fusing porcelain glaze and hydrofluoric acid showed bond strength values statistically superior to remaining groups ($p < 0.001$). Treatment of zirconia ceramic surfaces with a glaze of low-fusing porcelain significantly increased the bond strength of a dual-cure resin luting agent to the ceramic surface.

INTRODUCTION

Unique mechanical properties, chemical stability, and biocompatibility make zirconia-based ceramic an attractive core material for fabrication of all-ceramic restorations.¹ Combined with CAD/CAM technology, the fabrication of complex restorations incorporating zirconia cores has become a completely digitized process and a relatively simple procedure.^{2,3} The flexural strength and fracture toughness are considerably higher than those of other dental ceramics,⁴ and the zirconia-based ceramic shows a distinct mechanism of stress-induced transformation toughening, meaning that the material undergoes microstructural changes when submitted to stress.⁴⁻⁶ Zirconia-based ceramic can actively resist crack propagation through a transformation from a tetragonal to a monoclinic phase at the tip of a crack, which is accompanied by a 3% to 4% volume increase.⁵ Based on these characteristics, zirconia-based ceramic has been used as a prosthetic implant for medical and dental applications, posts, implant abutments, orthodontic brackets, and frameworks for crowns and bridges.^{2,7-10}

Long-term durable bond strength to ceramic surfaces is the aim for dental clinical applications and is dependent on the micromechanical and chemical interaction between luting agent and ceramic surface. The retention and the stability of the ceramic restorations are enhanced by the adhesive bond strength, which must be strong enough to resist the expected functional loads¹¹ and hydrolytic degradation.¹² The luting of a zirconia restoration can be done with zinc phosphate or with modified glass ionomer cements.¹³ However, the advantages of resin luting agents, such as marginal seal, good retention, and improvement of fracture resistance, have made them popular for use

even with high-strength ceramics.^{13,14} Several studies have investigated the bond strength between resin luting agent and zirconia-based ceramic, and several methods have been proposed to promote a durable chemical and micromechanical bond with zirconia. The conventional treatments for ceramic surfaces, such as oxide airborne particle abrasion and hydrofluoric acid etching, are not able to promote a strong and stable bond with zirconia.¹⁵ Air abrasion might affect the ceramic surface by creating microcracks that could reduce the fracture strength of the ceramic,¹⁶ and the hydrofluoric acid etching combined with silanization, which is used with other glass and disilicate-based ceramics, has not been successful with acid-resistant and glass-free zirconia ceramics.^{7,15}

In recent years, the literature has shown new treatments aimed at optimizing the bond strength to zirconia-based ceramic. However, chemical bonding to zirconia is limited by the inertness of the ceramic composition and has led to the investigation of various surface-roughening methods, such as silica coating followed by silanation,¹⁷ plasma spraying,¹³ airborne particle abrasion combined with the application of phosphate ester monomer (MDP),^{8,18} ceramic primers,⁷ Er:YAG laser,¹⁹ a selective infiltration etching technique,⁷ and heat-induced maturation.¹¹ All these treatments provide an immediate increase in bond strength. However, the association between the increased bond strength, durability, and clinical performance, as well as the development of a simple surface treatment protocol for clinicians, is still not fully defined and needs more clinical and longitudinal laboratory investigations to elucidate an optimum protocol for zirconia ceramic.

The purpose of this study was to evaluate a novel surface treatment that uses a low-fusing porcelain glaze for promoting a bond between zirconia-based ceramic and a dual-cure resin luting agent, and to analyze the association of this surface treatment combined with conventional airborne particle abrasion, hydrofluoric acid etching and silanization treatments. The null hypothesis was that there would be no difference between dual-cured resin luting agent bond strength to zirconia following conventional ceramic treatments and a novel glaze surface treatment.

MATERIALS AND METHODS

Ceramic Surface Treatments

Twenty-four ceramic discs 94% ZrO_2 stabilized by 5% Y_2O_3 (Cercon Smart Ceramics, Degudent, Ha-

nau, Germany) were fabricated to be 16 mm in diameter and 1 mm thickness. The ceramic discs were randomly assigned to eight treatment sequences and then received one of the following surface treatments:

Group 1: 110 μm aluminum oxide particle abrasion (Renfert GbmH, Hilzingen, Germany) for 15 seconds at four-bar pressure and a distance of 4 mm from the ceramic surface. No additional treatment was applied, but the ceramic surface was washed with tap water for one minute, ultrasonically cleaned in a water bath for 10 minutes, and air-dried.

Group 2: The same treatment performed in group 1 and a silane agent (Scotchbond Ceramic Primer, 3M ESPE, Seefeld, Germany) was applied on the ceramic surface and allowed to dry for five minutes.

Group 3: The ceramic surface received airborne particle abrasion with 50 μm aluminum oxide for 15 seconds at four-bar pressure (Renfert, Hilzingen, Germany). The distance of the tip from the ceramic surface was approximately 4 mm, and the tip was moved over the entire ceramic surface. The disc was washed with tap water for one minute, ultrasonically cleaned in water bath for 10 minutes, and air-dried.

Group 4: The same treatment performed in group 3, and a silane agent (Scotchbond Ceramic Primer, 3M ESPE) was applied on the ceramic surface and allowed to dry for five minutes.

Group 5: A liner ceramic (Cercon Ceram Liner, Degudent) was applied and sintered on the ceramic surface followed by a low-fusing porcelain glaze (Cercon Ceram Glaze, Degudent) with a no. 1 brush (Ney, Hanau, Germany) and sintered following the ceramic manufacturer's instructions. After that, the glaze was acid etched with 10% hydrofluoric acid (Dentsply, Milford, DE, USA) for 20 seconds, washed with tap water for one minute, and finally ultrasonically cleaned in a water bath for 10 minutes and air-dried.

Group 6: The liner, glaze application, and sintering was the same as in group 5. A silane agent (Scotchbond Ceramic Primer, 3M ESPE) was applied on the ceramic surface and allowed to dry for five minutes.

Group 7: The liner, glaze application, and sintering was the same as in group 5. After that, the glazed surface received airborne particle abrasion with 50 μm aluminum oxide for five seconds at four-bar pressure (Renfert, Hilzingen, Germany). The distance of the tip from the ceramic surface was approximately 4 mm. The disc was washed with tap water for one minute, ultrasonically cleaned in a water bath for 10 minutes, and air-dried.

Group 8: Surface preparation was as described for group 7. In addition, a silane agent (Scotchbond Ceramic Primer, 3M ESPE) was applied on the ceramic surface and allowed to dry for five minutes.

Bonding Procedure

The materials used in this study are listed in Table I. After surface preparation, in order to prepare the resin cement cylinder for cementation, equal lengths of Enforce resin cement (Dentsply) base and catalyst pastes were mixed for 20 seconds and then used to fill an iris that was cut from microbore Tygon tubing (TYG-030; Small Parts Inc, Miami Lakes, FL, USA) with an internal diameter and height of approximately 0.75 and 0.50 mm, respectively. The Tygon tubing was cut in a lathe using a stainless steel no. 11 scalpel blade (Scalpel SS, Miltex Instruments Co, Rietheim-Weilheim, Germany). The Tygon tubing containing resin luting agent was put on the ceramic surface and photocured for 40 seconds with 800 mW/cm² from a halogen light curing unit (Optilux Demetron 501, Demetron Kerr, Danbury, CT, USA). In this manner, each ceramic surface was bonded at 10 different locations with the resin cylinders. The assembly of ceramic/resin luting agent was stored at room temperature (23°C \pm 2°C) for one hour prior to removal of the Tygon tubing, then the specimens were immersed in distilled water at 37°C for 24 hours before microshear bond testing.

Microshear Bond Test

Before the test, all the ceramic/resin cylinder interfaces were analyzed with a light microscope (Nikon Measurescope UM-2, Tokyo, Japan) for bonding defects. The cylinders with apparent interfacial gap formation, bubble inclusion, or any other defects were excluded and replaced by another one. Three sets of ceramic/resin luting agents (30 cylinders of resin cement in each treatment group) were used for each test group.

The assembly of the ceramic plate and the resin cement was adhered to the testing device using cyanoacrylate adhesive (Loctite UltraGel super glue, Loctite, São Paulo, Brazil), which in turn was placed in a universal testing machine (Instron 4411, Instron, Canton, MA, USA) for microshear bond testing. An edge of stainless steel with a thickness of 0.5 mm was fixed on the superior part of a universal testing machine and was gently adapted against the ceramic/resin luting agent interface. A microshear bond test was applied to each specimen at a crosshead speed of 0.5 mm/min until failure.

Table 1: Material Type, Brand Name, Manufacturer, and Composition			
Material Type	Brand Name	Manufacturer	Composition ^a
Zirconia ceramic	Cercon	Degudent	ZrO ₂ stabilized by Y ₂ O ₃
Low-fusion porcelain glaze	Cercon Ceram Kiss	Degudent	Vitreous porcelain and pigments
Resin cement	Enforce	Dentsply	BisGMA, BHT, EDAB, TEGDMA, fumed silica, silanized barium, aluminum borosilicate glass (66% wt)
Ceramic primer	Scotchbond Ceramic Primer	3M ESPE	Bisphenol A polyethoxy dimethacrylate 3-methacryloyloxypropyl trimethoxysilane
Ceramic liner	Cercon Ceram Liner	Degudent	Metallic oxide combination
^a Manufacturer's information.			

Statistical Analysis

The data were statistically analyzed using one-way analysis of variance, and multiple comparisons were made using Tukey's test. The statistical significance level was set at $\alpha = 0.05$.

RESULTS

The bond strength between the dual-cure resin cement and zirconia-based ceramic was affected by the ceramic surface treatments, and the silane application did not increase the bond strength values ($p < 0.001$; Table 2). The means and standard deviations of microshear bond strength values for the groups tested are shown in Table 3.

The groups that utilized the low-fusing porcelain glaze in association with hydrofluoric acid (Figures 1 and 2) or airborne particle abrasion (Figures 3 and 4) showed bond strength values statistically superior to the groups that utilized the conventional airborne particle abrasion treatments with 50 and 110 μm aluminum oxide particles (Figures 5 and 6, respectively), which is the treatment recommended by the zirconia manufacturer ($p < 0.001$).

The low-fusing porcelain glaze treatment in association with hydrofluoric acid (Figures 1 and 2), independent of the silanization process, showed bond strength values statistically superior to the others groups tested ($p < 0.001$). The groups that associated low-fusing porcelain glaze and 50 μm aluminum oxide airborne particle abrasion (Figures 3 and 4) showed statistically intermediate bond strength values ($p < 0.001$). The airborne particle abrasion groups (1 through 4) showed bond strength values statistically inferior to the glaze groups (5 through 8). Different oxide granulations tested did not improve bond strength values. The silane application did not increase the bond strength values within each treatment group ($p < 0.001$; Table 3).

DISCUSSION

The surface roughening methods for densely sintered zirconia ceramics are limited by the inertness and hardness of this ceramic.¹¹ These characteristics inhibit the creation of grooves for microretention and chemical bond formation for optimal interaction with luting agents. The evaluated novel surface treatment that used a low-fusing porcelain glaze was able to

Table 2: Results of One-Way Analysis of Variance for Microshear Bond Test of Enforce Dual Resin Cement Bonded to Zirconia-Based Ceramic					
Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F	p-Value
Treatment	7	15367.386	5122.462	96.781	<0.001
Error	90	1026.5252220	11.4058358	—	—

Table 3: Means (SD) of Microshear Bond Strength in MPa

110 μ m Airborne Particle Abrasion		50 μ m Airborne Particle Abrasion		Glaze + 50 μ m Airborne Particle Abrasion		Glaze + Hydrofluoric Acid	
No silane ^a	Silane	No silane	Silane	No silane	Silane	No silane	Silane
4.06 (1.36) c ^b	5.33 (1.58) c	3.95 (1.24) c	6.02 (1.61) c	17.45 (8.55) B	18.41 (7.47) B	20.75 (8.29) A	25.17 (8.37) A

^a Treatment recommended by the zirconia-based ceramic manufacturer.
^b Letters denote significant differences among the zirconia-based surface treatments ($p < 0.001$; Tukey's test).

promote superior bond strength values between zirconia-based ceramic and a dual-cure resin luting agent in comparison with the conventional surface treatment methods that utilized airborne particle abrasion (Figures 5 and 6) as recommended by the zirconia manufacturer (Table 3). Based on the results obtained, the proposed null hypothesis that the airborne particle abrasion treatments and a novel glaze surface treatment would not influence the bond strength of a dual-cured luting agent was not accepted.

In attempts to promote optimal bond strength to zirconia-based ceramics, several roughening methods have been investigated.^{7,8,11-13,17-19} The low-fusing porcelain glaze treatment was able to promote an increase in bond strength and appears to be a simple treatment that enhances traditional treatments like hydrofluoric acid or airborne particle abrasion (Figures 1 through 4).

The ceramic surface treatments should meet certain criteria, such as not compromising the integrity of the ceramic, not promoting additional problems with crown adaptation, and good interac-

tion with the luting agent.¹³ Surface roughening methods can result in structural damage, material loss, grain pullout, and creation of sharp crack tips,²⁰ causing bonded restorations to become more susceptible to radial cracking under functional loads.¹¹ The low-fusion porcelain glaze forms a penetrating vitreous layer into the zirconia-based ceramic surface (Figures 1 through 4). This glaze-treated surface seems to be similar to a glass-based ceramic surface that is susceptible to air abrasion and hydrofluoric acid treatments and provides an interaction with the silane agent for promoting chemical reactivity.

The highest bond strengths were observed with the low-fusion porcelain glaze groups, and the association with hydrofluoric acid conditioning showed superior statistical bond strength values and promoted a better interaction within the cementation process (Table 3). The use of a silane agent did not improve the bond strength to zirconia-based ceramic for the groups tested, and these results are consistent with other studies that have observed that silane utilization does not enhance

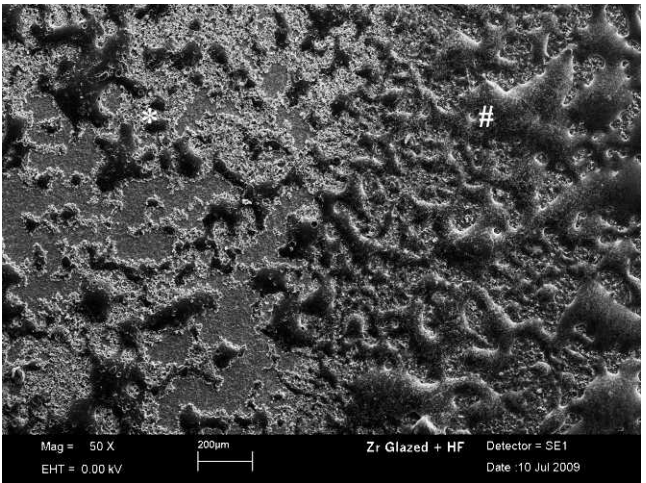


Figure 1. Zirconia-base ceramic treated with low-fusing porcelain glaze and 10% hydrofluoric acid conditioning $\times 50$ (# glazed area without etching and * glazed area with etching).

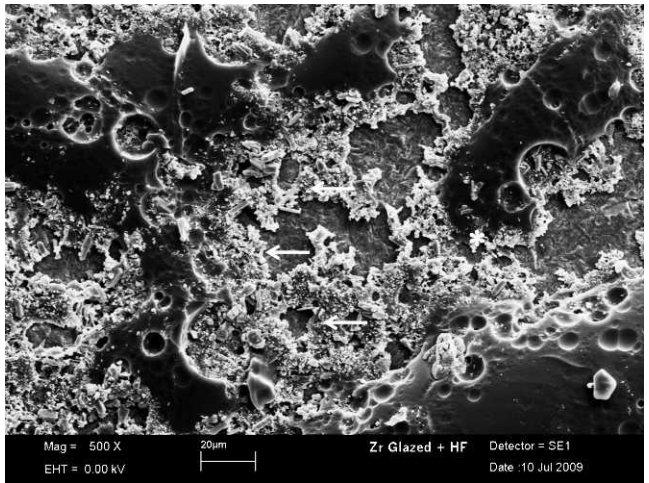


Figure 2. Zirconia-base ceramic treated with low-fusing porcelain glaze and 10% hydrofluoric acid conditioning $\times 500$ (← glazed zirconia treated with 10% hydrofluoric acid and * glazed zirconia).

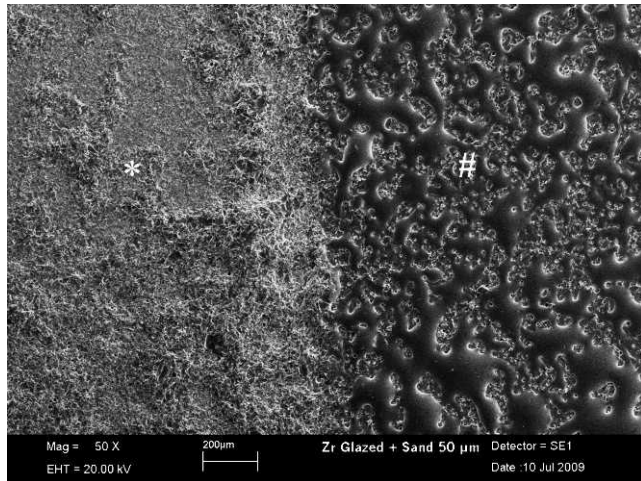


Figure 3. Zirconia-base ceramic treated with low-fusing porcelain glaze and treated with 50 μm aluminum oxide airborne particle abrasion $\times 50$ (# glazed area and * area glazed with sandblasted).

performance for high-crystalline ceramics.^{6,10,13,21,22} In addition, another reason that the silane agent did not improve the bond strength to the vitreous layer created by the glaze application can be attributed to the application of a small layer of glaze, necessary to promote an acceptable clinical marginal adaptation, and by the subsequent use of airborne particle abrasion or hydrofluoric acid etching that was responsible for partial removal of the vitreous layer (Figures 1 through 4). The zirconia airborne particle abrasion, with 50 or 110 μm aluminum oxide, is not able to promote high bond strengths to zirconia surfaces because of the ceramic composition. Other pretreatments that aim to increase roughness or chemical bond interaction are necessary for achiev-

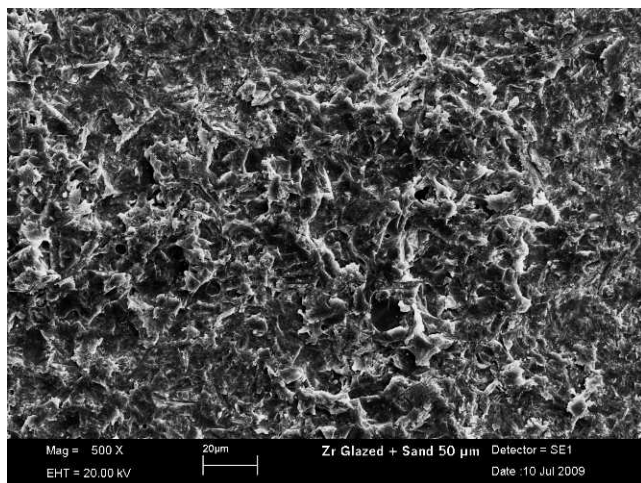


Figure 4. Zirconia-base ceramic treated with low-fusing porcelain glaze and treated with 50 μm aluminum oxides airborne particle abrasion $\times 500$.

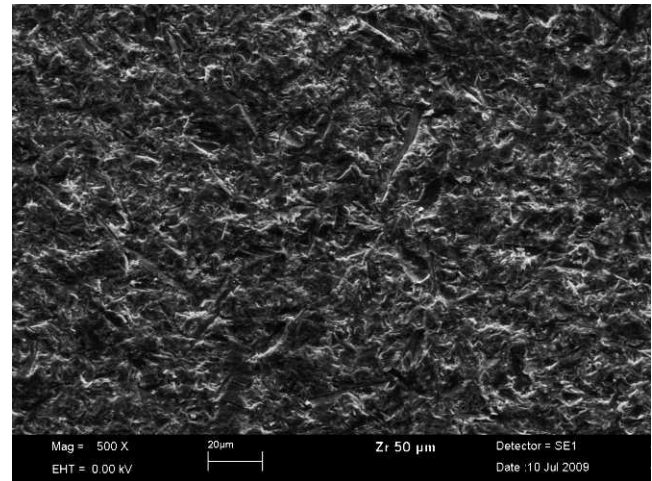


Figure 5. Zirconia-base ceramic treated with 50 μm aluminum oxide airborne particle abrasion $\times 500$.

ing high immediate bond strengths and long-term clinical performance.

Optimal interaction between the ceramic surface and luting agent are necessary for the success and long-term durability of ceramic restoration.²² Superb mechanical properties make zirconia-based ceramic a promising core material for fabrication of all-ceramic restorations and other dental applications.^{1,2,7-10} A low-fusing porcelain glaze treatment seems to be a promising enhancement for resin bonding to zirconium oxide. Future investigations should assess the influence of this treatment on the overall adaptation of restorations to tooth preparations as well as degradation tests and glaze delamination of the zirconia ceramic surface.

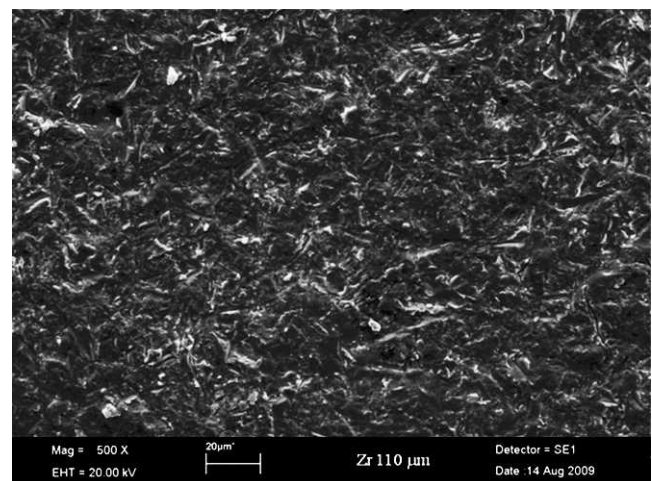


Figure 6. Zirconia-base ceramic treated with 110 μm aluminum oxide airborne particle abrasion $\times 500$.

CONCLUSIONS

Within the limitations of the present study, the following conclusions may be drawn:

- 1) Airborne particle abrasion with 50 or 110 μm aluminum oxide promotes a similar effect on the bond strength of a dual-cure resin luting agent to a zirconia-based ceramic surface.
- 2) The addition of a low-fusing porcelain glaze to a zirconia-based ceramic significantly increases the bond strength to a dual-cure luting agent.
- 3) The use of a silane coupling agent does not influence the resin bond strength to zirconia-based ceramic.

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Conflict of Interest Declaration

The Authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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