

Effect of Different Glaze Coating of Zirconia Based Ceramics on Surface Roughness and Shear Bond Strength

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ABSTRACT

Objectives: The aim of the study was to assess the effect of different glaze coating on micro-shear bond strength (μ SBS) between zirconia based ceramic and two adhesive resin cements.

Materials and Methods: Twenty four zirconium oxide disc specimens were milled using CAD/CAM. The specimens were divided into three equal groups according to the type of the surface treatment: group I: Air borne particle abrasion $50 \leq \mu\text{m}$ Al₂O₃ particles, group II: VM9 glaze material was coated to one side of the zirconia core discs (Vita Zahnfabrik, Bad Sackingen, Germany), group III: IPS e.max glaze material was coated to one side of the zirconia core discs (IPS e.max, Ivoclar Vivadent, Liechtenstein, Germany). The discs of each group were further subdivided into two equal subgroups according to the adhesive resin cement: subgroup A: Total-etch adhesive resin cement, subgroup B: Self-adhesive resin cement was used. After different surface treatments, surface roughness (Ra) in μm was analyzed using digital microscope. The two types of resin cement were applied to the pretreated ceramic discs and μ SBS was measured using a universal testing machine. The mode of failure of the debonded specimens was determined by SEM.

Results: There was no statistically significant difference between the mean surface roughness values of the three tested groups. Disc glazed with VM9 material showed statistically significant highest mean μ SBS values than discs glazed with e-max material, while sandblasted zirconia discs showed the statistically significantly lowest mean μ SBS.

Conclusion: Silica based ceramic coating to zirconia restoration significantly improved the μ SBS to adhesive resin cements

Keywords: zirconia, Glaze-on, Adhesive resin cement, Surface treatment, microshear bond strength, surface roughness.

INTRODUCTION

The use of zirconia all-ceramic fixed dental prostheses (FDPs) provides tooth-colored restorations with high flexural strength. ⁽¹⁾ Nowadays, construction of zirconia based restoration with computer-aided design/computer-aided manufacturing (CAD/CAM) system that promises to transform everyday dentistry. ⁽²⁾ The three-dimensional design of Y-TZP frameworks requires a computer software (CAD)

provided by the manufacturer. After scanning procedure of the designed work, data were transferred to a computerized manufacturing unit (CAM) that performs a production of the zirconia framework. CAD/CAM technology relies on the exact dimensional predictions to compensate for sintering shrinkage, which is an economical and reproducible method. ^(3,4) With the introduction and advancement in adhesive promoters, adhesively bonded restorations

can be considered an integral part of minimally invasive dentistry. Not only the strength of the restoration but also the adhesion of cements to dental tissues and to a particular restorative material, which is an important issue for long-term clinical success of the restoration. (5,6)

This aspect becomes more important when retention of FDPs does not rely on macro-mechanical principles, as in the case of resin-bonded FDPs or cantilever restorations. (7) Although etching of the fitting surface with hydrofluoric acid (HF) and subsequent silanization of the glassy matrix of ceramic restoration was an effective method to achieve durable adhesion to resin adhesive cements. (8,9) Neither etching with these solutions nor adding silane coupling agents resulted in adequate resin bond to high alumina (10,11) or zirconia ceramics, (12,13) since such ceramics do not contain a silicon dioxide (silica) phase. For this reason, in order to enhance the bond strength of luting cements to oxide-based ceramics, a number of surface conditioning methods have been suggested during the last two decades. (5,14) While some of these methods micromechanically facilitate resin-ceramic bonding by employing air-borne particle abrasion with alumina particles, (5,13,15) others are based on physicochemical activation of the ceramic surfaces using silica-coated alumina particles followed by silanization, (5,13) or chemical activation with cements containing functional monomers (12) In addition to these methods, flame treatment/silane deposition, (16) selective infiltration etching (17) of the surface, and the use of cements containing the phosphate ester monomer 10-methacryloyloxydecyl di-hydrogen phosphate(MDP) have been proposed. (18) Durable adhesion of bis-GMA resin to zirconia ceramics was not achieved using some of these methods and roughness created by air abrasion was thought to be the main bonding mechanism for MDP monomers. The selective infiltration etching technique and the use of MDP monomers have also been combined with novel

reactive silane monomers to yield initial high bond strengths that decreased after thermocycling. (19)

Adhesion of resin-based materials to yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramic is currently a topic of great interest, due to the expanded use of this ceramic material as a framework for fixed dental prosthesis (FDP) in combination with CAD/CAM procedures. For clinical purposes, conventional bonding procedures used for etchable glassy matrix ceramic systems are not effective on Y-TZP, since hydrofluoric acid produces insufficient roughness for resin cement bonding. (20, 21) Surface changes and adequate cementation procedures seem to be necessary to achieve a stable bond to Y-TZP frameworks especially for resin-bonded surface-retained FDPs. (22,23) A reliable cementation to Y-TZP ceramics improves the retention, prevents microleakage and increases fracture and fatigue resistance of the FDPs. (24) Therefore, the objectives of this study were to chemically create a silica-based coating on the Y-TZP ceramic surfaces using two glaze -on coatings, test the adhesive bond strength of resin cements using microshear bond strength test, and assess the failure types after debonding by scanning electron microscope (SEM). The tested hypothesis was that silica-based coating followed by silane coupling agent application would not improve chemical adhesion between Y-TZP ceramic with different resin cements compared to conventional air abrasion technique.

MATERIALS AND METHODS

A total of twenty four all-ceramic disc specimens with 2 mm height and 1cm diameter were constructed. The disc samples were divided into three equal groups according to the type of the surface treatment used (n=8): group I: Air borne particle abrasion using $50 \leq \mu\text{m}$ Al₂O₃ particles (served as control), group II :IPS e.max glaze material was coated to one side of the zirconia core discs(IPS e.max, Ivoclar Vivadent, Schaan, Liechtenstein), group

III:VM9 glaze material was coated to one side of the zirconia core discs .The flat round discs of each group were randomly further subdivided into two equal subgroups(n=4) according to the adhesive resin cement used to: subgroup A:Variolink total-etch adhesive resin cement. Subgroup B: Rely x U200 self -adhesive resin cement was used

A specially designed circular Teflon mold was used for construction of the disc specimens. The mold consists of an outer cylinder and inner cylinder .The outer cylinder contains an inner hole with an internal diameter of 1 cm. The inner cylinder is constructed to fit within the hole. The difference in height between the two cylinders is 2mm. Therefore, a mold space of 2mm in height and 12mm in diameter was formed.

Construction of the zirconia core disc samples

Construction of the zirconia core samples was achieved through laser scanning of the mold cavity (1cm x 2 mm),

then the zirconia disc samples were constructed using a computer-aided design/computer aided milling (CAD/CAM) process (Laser dentaGmbH, Bergheim, Germany). Sintering of the zirconia discs was carried out following the manufacturer’s instructions in high temperature furnace (VITA ZYrcomat-Germany). The zirconia discs were ultrasonically cleaned in distilled water for 10 minutes and then dried with compressed air. The thickness of the discs was measured using a digital micrometer, with accuracy of $\pm 10 \mu\text{m}$. For group I: Zirconia samples were sandblasted with aluminum oxide $\leq 50 \mu\text{m}$, cleaning with alcohol and drying with air (served as a control), for group II: VM9 glaze ceramic powder was applied in a thin single coat using a ceramic brush and for group III: IPS e.max glaze ceramic powder was applied in a thin single coat using a ceramic brush. The heat temperature program for the two glaze materials were done following the manufacturer's instructions. Table1.

Table1: Chemical composition of experimental materials.

MATERIALS	CHEMICAL COMPOSITION	MANUFACTURER
Vita In-ceram YZ	prefabricated pre-sintered ceramic blanks: ZrO ₂ <95%wt, Y ₂ O ₃ 35%wt, HfO ₂ <3%wt, Al ₂ O ₃ <1%wt, SiO ₂ <1%wt	Vita Zahnfabrik Bad Sackingen ,Germany
Vita VM9	SiO ₂ : 60-64 wt.%,Al ₂ O ₃ : 13-15wt%,K ₂ O: 7-10 wt.%,Na ₂ O : 4-6 wt.%,TiO ₂ :<0.5 wt.% CeO ₂ :<0.5wt.%,ZrO ₂ : 0-1wt.%,CaO: 1-2wt.% B ₂ O ₃ : 3-5wt.%,BaO: 1-3 wt.%,SnO ₂ :<0.5wt.%	Vita Zahnfabrik Bad Sackingen, Germany.
IPS e.max ceram	SiO ₂ : 61-68 wt% Al ₂ O ₃ : 5.0-8.0 wt% N ₂ O: 5.0-8.0 wt% K ₂ O: 5.0-8.0 wt% Other oxides 2.0-4.0 wt%	Ivoclar,vivadent.,
Variolink II adhesive resin cement.(total-etch)	Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments	Ivoclar, Vivadent.
Rely- X unicem resin cement.(self-adhesive)	Powder: - Alkaline (basic) fillers, Silanated fillers Initiator components, Pigments Liquid: -Methacrylate monomers containing phosphoric acid groups ,Methacrylate monomers Initiator components,Stabilizers	3M ESPE, Germany

Surface treatment for the samples:

The glazed surface for each disc sample was then conditioned with 9.5% hydrofluoric acid gels. (Ultradent; south Jordan, UT, USA) for 90 seconds. After

acid etching, the specimens were washed with tap water for one minute and air dried.

Surface Roughness evaluation:

Quantitative characterization of surface topography was carried out using an optical method without contact. Specimens

were photographed using USB Digital microscope with a built-in camera (Scope Capture Digital Microscope, Guangdong, China) connected with an IBM compatible personal computer using a fixed magnification of 50X. The images were recorded with a resolution of 1280×1024 pixels per image. Digital microscope images were cropped to 350×400 pixels using Microsoft office picture manager to specify/standardize area of roughness measurement. The cropped images were analyzed using WSxM software (5 develop 4.1, Nanotec, Electronica, SL). Within the software, all limits, sizes, frames and measured parameters are expressed in pixels. Therefore, system calibration was done to convert the pixels into absolute real world units. Calibration was made by comparing an object of known size (a ruler in this study) with a scale generated by the software. Subsequently, a 3D image of the surface profile of the specimens was created. Three 3D images were collected for each specimen, both in the central area and in the sides at area of $10 \mu\text{m} \times 10 \mu\text{m}$. This software was used to calculate the average surface roughness values (R_a) expressed in μm .

Bonding procedures:

The glazed ceramic disc surface for subgroup A and sub group B were coated with silane coupling agent (Monobond-S, Ivoclar Vivadent) using a brush, the silane coupling agent was left to react for 60s, then air dried. Subsequently for subgroup A, a thin layer of adhesive resin (Heliobond, Ivoclar Vivadent) was applied to the glazed surface of ceramic discs for 1 minute using a brush, gently air thinned, light cured for 10s and air dried.

Small transparent microtubules were cut from polyvinyl tube with internal diameter of 0.9 mm and a height of 0.5mm. Five microtubules were mounted over each ceramic disc to restrict the bonding area. (Fig.1) For Variolink II adhesive resin cement (Ivoclar, Vivadent), base and catalyst were mixed according to manufacturer's instructions and packed into the

microtubules lumen and a plastic matrix strip was placed over the resin cement and gently pressed flat and light-cured for 40 seconds using light curing unit. Following the manufacturer's recommendations, RelyX Unicem (3M ESPE, Seefeld, Germany) was mixed for 10 seconds in an automatic mixer and packed into the cylinder lumen and a plastic matrix strip was placed over the resin cement and gently pressed flat and light-cured for 40 seconds using light curing unit.

Micro shear bond testing (μSBS)

Micro-shear bond strengths (μSBS) were measured using a universal testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK). A 0.014 mm diameter wire was looped around the bonded micro-cylinder assembly as close as possible to the base of the microcylinder and aligned with the loading axis of the upper movable compartment of the testing machine (fig.2). A shearing load was applied at a crosshead speed of 0.5 mm/min. until failure occurred. The load required to cause debonding was recorded in newton using computer software (Nexygen-MT, Lloyd Instruments Ltd., Fareham, UK). Micro-Shear bond strength was calculated according to the formula $T = P / \pi r^2$ where; T =bond strength (MPa), P =load at failure (N) and r = radius of micro- cylinder (mm).

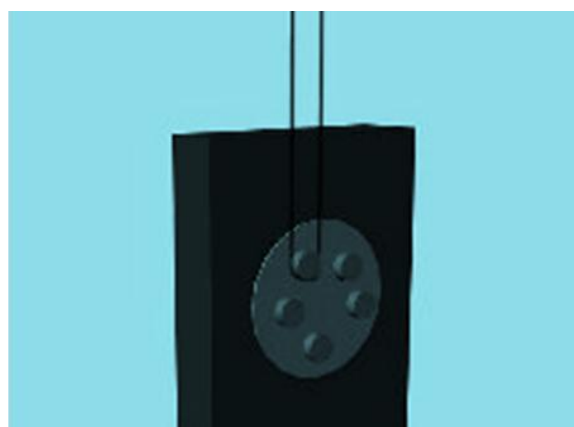


Figure (1): Schematic diagram of micro shear bond testing.

Scanning electron microscopic evaluation

To determine the mode of failure after microshear bond strength test, one sample from each group was coated with

gold-palladium alloy using a sputter-coating technique (Ladd sputter Coater, USA) and examined by scanning electron microscope (SEM) (Scanning electron microscope, Philips, XL, 30) at X1000 to observe the mode of failure at de bonded zirconia-cement interfaces.

Statistical analysis:

Data were presented as mean and standard deviation (SD) values. Regression model using Two-way Analysis of Variance (ANOVA) was used for testing the effect of material, surface pretreatment protocols and their interactions on micro-shear bond strength and surface roughness. Tukey's post-hoc test was used for pair-wise comparison between the mean values when ANOVA test is significant.

The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM SPSS Statistics Version 20 for Windows.

RESULTS:

I. Surface roughness (Ra) .

Means and standard deviations (SD) of surface roughness (Ra) mean values in μm of all tested groups are presented in

table (2).Results revealed that there was no statistically significant difference between the tested groups

Table 2: Descriptive statistics and results of comparison between surface roughness (Ra) of the three materials.

VM9 glaze material		e-max glaze material		Sandblasted zirconia		P-value
Mean	SD	Mean	SD	Mean	SD	
0.239	0.001	0.241	0.003	0.238	0.001	0.096

*: Significant at $P \leq 0.05$.

Digital microscope images of the ceramic surfaces following different surface treatments showed similar degrees of roughness and irregularities in the surface. (Fig 2) After air borne particle abrasion (group I), the digital microscopic images showed a deep irregular, wide valleys and troughs due to the mechanical action of air borne particles abrasion (Fig.2a).Following hydrofluoric acid etching for both type of glaze materials applied on zirconia samples, digital microscopic images showed a homogenous meshwork and this might be attributed to the dissolution of the glassy matrix by the effect of hydrofluoric acid, resulted in a uniform distribution of pores all over the etched surface (fig.2 b, and 2c).



Fig.2: 3D image of the surface profile of the yz ceramic samples after different surface treatments; a. air borne particle abrasion, b. VM9 glaze coating with hydrofluoric acid etching, c. e.max glaze coating with hydrofluoric acid etching. (Magnification x50)

Micro-shear bond strength

The results of Two-way ANOVA showed that the material type had no statistically significant effect on μSBS mean values while surface treatment and the interaction between the two variables had statistically significant effect on μSBS mean values. Table 2.

Means and standard deviations (SD) of μSBS mean values in MPa were

displayed in Table 3; Samples glazed with VM9 (group II) material showed the statistically significant highest mean micro-shear bond strength values(8.63 ± 1.40) than samples glazed with e-max (group III) material,(7.76 ± 0.74) ,while sandblasted zirconia samples(group I) showed the statistically significant lowest mean micro-shear bond strength(6.34 ± 0.36).

For sandblasted zirconia samples (group I); there was no statistically significant difference between mean micro-shear bond strength values with variolinkII and Rely X Unicem. For both VM9 glaze material(group II) and e.max glaze

material(group III), Rely X unicem adhesive resin cement showed statistically significantly higher mean micro-shear bond strength values than Variolink resin cement. Table 4

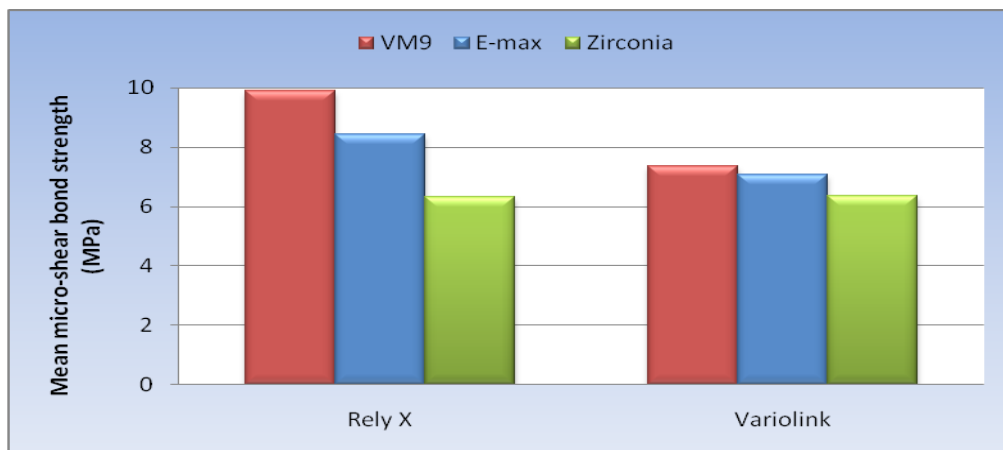


Figure (3): Bar chart representing mean micro-shear bond strengths of the two adhesive resin cements with each ceramic material.

Table 3: Descriptive statistics and results of comparison between micro-shear bond strength of the three materials regardless of cement type.

VM9 glaze material		E-max glaze material		Sandblasted zirconia		P-value
Mean	SD	Mean	SD	Mean	SD	
8.63 ^a	1.40	7.76 ^b	0.74	6.34 ^c	0.36	<0.001*

*. Significant at $P \leq 0.05$, Different superscripts are statistically significantly different.

Table 4: Descriptive statistics and results of comparison between micro-shear bond strength with different interactions.

Material	Cement type		Rely-X unicem		Variolink II	
	Mean	SD	Mean	SD	Mean	SD
VM9 glaze material	9.92 ^{A,C}	0.05	7.35 ^{B,C}	0.43		
e-max glaze material	8.44 ^{A,D}	0.17	7.08 ^{B,D}	0.10		
Sandblasted Zirconia	6.31 ^E	0.13	6.37 ^E	0.55		

A,B.Superscripts indicate statistically significant difference between cements.

C,D,E.Superscripts indicate statistically significant difference between materials.

Scanning electron microscopic evaluation of debonded ceramic surfaces

SEM photos representing the resin-ceramic interface of the debonded specimens of the tested groups (fig. 4). For group I for both subgroups, adhesive mode of failure was predominating at resin/ceramic interface. Meanwhile, for group II subgroup A, the mode of failure was a cohesive mode of failure with resin cement particles appearing stuck to the ceramic surface. For group II, subgroup B, the mode of failure was a mixed shown at resin /ceramic interface with areas of scattered

resin cement. Whereas; for group III, subgroup B, the mode of failure was a mixed type of failure with resin cement particles appearing stuck to the ceramic surface. While; for group III subgroup A, the mode of failure was a cohesive mode of failure within the cement layer.

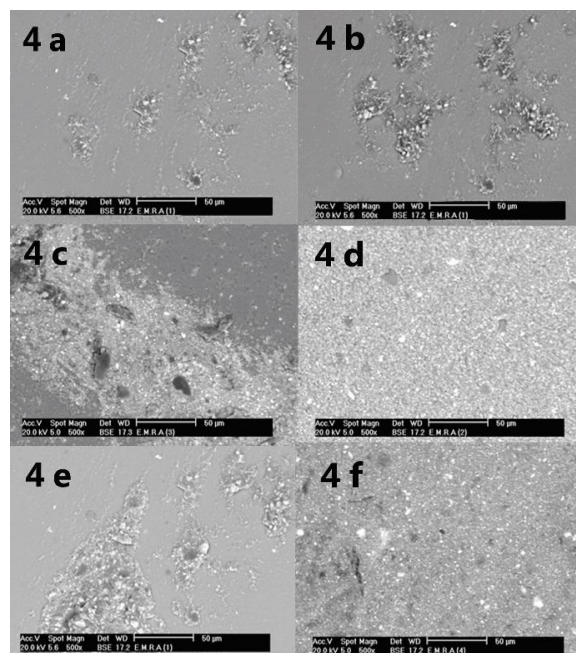


Fig.4: SEM photos for YZ ceramic/resin interface: 4a, sandblasted zirconia surface with variolinkII resin cement .4b, Sandblasted zirconia surface with rely X unicem.4c, VM9 coated zirconia disc with variolink II resin cement. 4d, Vm9 coated zirconia disc with RelyX unicem resin cement .4e, e.max zirconia coated disc with variolink II resin cement. 4f, e.max coated zirconia disc with Rely X resin cement.

DISCUSSION

The superiority in mechanical strength of zirconia-reinforced dental ceramics over conventional all-ceramic materials prompted a wider availability of such ceramics on the market. (25, 26) the more popular of such zirconia-reinforced dental ceramics include a glass-infiltrated, zirconia-reinforced alumina (In-Ceram Zirconia, VITA Zahnfabrik, Bad Sackingen, Germany) and machined-milled, yttria-stabilized zirconia ceramics (Y-TZP) such as Cercon (Cercon Smart Ceramic System, Dentsply Intl, York, PA), LAVA (LAVA system, 3M ESPE, St. Paul, MN), DC-Zirkon (Precident-DCS, Switzerland), and YZ Zirconia (Vita Zahnfabrik).

Bonding to silica-free oxide ceramics required other methods than those used for silica based ceramics, such as HF acid etching and silanization. Recommended methods often include the use of silica coating and silanization, (13) or phosphate monomer-containing MDP composite resins. (18,27) Both methods required surface cleaning or surface activation by air-borne particle abrasion prior to cementation that not only required additional equipment in the laboratory or chairside, but also may detrimentally affect the fatigue properties of zirconia restoration. (28) For these reasons, the purpose of this study was to examine the effect of the application of an etchable, thin glaze layer on the bond strength with two adhesive resin cements.

In this study, the application of a glaze layer and subsequent HF etching and silanization significantly improved the microshear bond strength as opposed to the non-glazed group in the case of Rely X-Unicem adhesive resin cement followed by Variolink II adhesive resin cement. While for the non-glazed samples (group I) showed the lowest microshear bond strength than the two other glazed groups, this may be attributed to the HF acid etching which was capable of creating numerous undercuts and surface pits by preferential dissolution of the glass phase of the ceramic

matrix, which leads to increase in the surface area to be silanized and to the possibility of micromechanical attachment. (29, 30) For Rely X unicem, showed the highest μ SBS mean values for group II and group III respectively (9.92 ± 0.05), (8.44 ± 0.17) than Variolink II resin cement for group II and group III respectively (7.35 ± 0.43), (7.08 ± 0.10) this might be due to the standardized manipulation of the cement by the auto mixing, easier application through the tip of the capsule into the microtubules and the superior performance of MPD functional monomer, which adsorbs onto and alters the surface of the ceramic facilitating chemical interaction. (31-33) While for the variolink II the results may be related to manual mixing and application of the cement into the microtubules in addition to the higher viscosity of the cement which might result in deficiency of the cement in the microtubules leading to uncontrolled polymerization shrinkage during light curing.

The obtained results of this study were partially in accordance with Amaral et al (34) and Blatz et al (35) who reported that, when pure zirconium and zirconia ceramic were treated with silane coupling agent the shear bond strength was found to be improved compared with the untreated group. In contrast Derand et al (36) reported that silane treatment did not positively affect the bond strength, on the contrary, it was reduced and that was explained by that silane did not react with the surface at room temperature.

The bond strength results should always be coupled with the failure type analysis. (Fig.4) Regardless of the cement type in non-glazed groups, adhesive failures were noted. In contrast, in the glazed groups, the incidence of cohesive failure type was more frequent. This supports the hypothesis that the application of a glaze layer gives more reliable results. So, the hypothesis of this study could be accepted. Since cohesive failures within the cement substrate were observed, further investigation should be done in order to

evaluate the durability of the bond, the effect of the glaze layer on the marginal fit and adaptation of zirconia restorations.

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