

Effect of Different Surface Treatments of Tetragonal Zirconia Polycrystal on Surface characteristics and Shear Bond Strength to Resin Composite as an Attempt for Repair.

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Abstract

Statement of problem: can the composite resin repairs of yttrium-tetragonal zirconia polycrystal (YTZP) be an effective, rapid and cost effective method. Purpose: The purpose of this in vitro study was to evaluate the shear bond strength (SBS) and scanning the failed adhesive surfaces to detect the mode of failure of zirconia repaired with composite resin with different surface pretreatments (without surface treatment, airborne-particle abrasion and wheel stone abrasion) and each group divided into two sub groups according to adding or not zirconia primer plus before the all bond adhesive. Material and methods: Sixty yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP, or zirconia) blocks with standard dimensions (10x10x5 mm) were prepared from green-state zirconia. The specimens were divided into three main groups (n=20) according to surface roughening treatment (As fired no ttt, air born particles abrasion and stone roughened). The Zirconia blocks were further subdivided into two subgroups whether treated with zirconia primer or not, thus there were 6 experimental groups according to the surface modification done and adhesive/primer application protocols used. Shear Bond Strength was measured with a single-plane lap device in a universal testing machine. For mode of failure analysis all specimens were scanned using scanning electron microscope. Results: The two- way ANOVA for comparing the two factors affecting SBS (surface treatment and Zirconia primer) showed that surface treatment of zirconia had a significant effect on SBS where air born abrasion group was significantly higher than stone grinding group

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and no treatment group respectively. On the other hand Z primer groups were significantly higher than no primer groups. The one way ANOVA showed that there is a significant difference between the tested subgroups in μ SBS. The post hoc Tukey test showed that the air born abrasion subgroups were statistically significantly higher than all other subgroups. Furthermore the two air born abrasion subgroups were not statistically significantly different. Conclusions: Within the limitations of this in vitro study, the following conclusions were drawn: 1. Preparation of zirconia surface with air born abrasion is the most effective and essential step before bonding repair material to zirconia. 2. Zirconia primer plus application before applying the bonding agent would improve the adhesive bond strength of repair material regardless of the type of surface treatment.

Introduction

Nowadays, many patients are demanding for metal free restorations to achieve better esthetic outcome, as these restorations permit light interaction properties similar to that of natural dentition. ¹Also, being biologically well tolerated by tissues is an important issue. However, the esthetics and the biocompatibility are not the only factors for selecting the materials to be used. But being mechanically superior to withstand occlusal stresses is another factor governing the selection of materials; especially when a long span prosthetic framework is advised.² Zirconia is a dental ceramic with high flexural strength and fracture toughness, excellent biocompatibility and esthetically accepted that has been widely used in the dental laboratory for many years; whether as a framework material or a fully anatomical alternative, enabling its use for fixed dental or implant supported restorations either cemented or screw retained. ^{3, 4} Chipping has been considered the most frequent technical failure in yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic restorations.⁵⁻⁷ Despite the recent technology to create more translucent monolithic systems; zirconia is still

more opaque than traditional ceramics. That is why zirconia is still widely used as a core material veneered by feldspathic ceramic to gain superior esthetic.^{8,9} The interface between the zirconia core and the feldspathic veneer is considered the weakest area liable for fracture or chipping; known as delamination, exposing the underlying zirconia.^{10, 11} Repairing or changing these restorations in dental laboratory is expensive and time consuming, but many feasible attempts were tried to repair with composite resin intraorally in a single appointment with reasonable cost.^{12, 13} However, Y-TZP ceramic is almost inert glass-free structure, with low surface energy and wettability and resists conventional etching with hydrofluoric acid. ^{14, 15} Furthermore, absence of silica in zirconia makes it difficult to gain adequate bond strength to methacrylate-based composite resins.^{16, 17} Many surface treatments to improve the bond strength at the resin-zirconia interface by providing micromechanical retention and chemical bonding have been proposed.¹⁸ This micromechanical abrasion can be created either by grinding, airborne aluminum oxide particles, tribo chemical silica coating, laser irradiation, or selective infiltration etching.¹⁹⁻²³ Some primers containing 10- methacryloyloxydecyl dihydrogen phosphate (MDP) monomer have been used to promote adhesion between composite resin and zirconia, with promising results. ²⁴⁻²⁷ Recently, Z-Prime Plus (ZPP) is considered the most effective of these MDP containing primers that promote adequate adhesive bond to zirconia.²⁸⁻³⁰ Many studies are needed to investigate the most reliable technique for zirconia repairing. Therefore, the purpose of the present study was to evaluate the shear bond strength (SBS) and scanning the failed adhesive surfaces to detect the mode of failure of zirconia repaired with composite resin with different surface pretreatments (without surface treatment, airborne-particle abrasion and wheel stone abrasion) and each group divided into two sub groups according to adding or not zirconia primer plus before the all bond adhesive. The null hypothesis was that the use of the new multimode adhesive

with or without ZPP application protocols would not influence the SBS of composite resin to zirconia surfaces either mechanically abraded or not.

Materials and Methods:

Sixty yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP, or zirconia) blocks with standard dimensions (10x10x5 mm) were prepared from green-state zirconia. With a diamond saw (Diamond wafering blade, N11-4244; 15HC) in a cutting machine (Isomet 1000; Buehler). These blocks were polished under running water (DAP-U; Struers) with Silicon Carbide abrasive paper with ascending grit (600,800, 1000, 1200). The specimens were then rinsed with distilled water for 10 seconds to remove debris. After sintering according to the manufacturer's instructions (Lava Therm; 3M ESPE). The specimens were divided into three main groups (n=20) according to surface roughening treatment (As fired no tt, air born particles abrasion and stone roughened). For air born particle abrasion one surface of each block was abraded with 50 mm aluminum oxide (Al₂O₃) particles (Microetcher II; Danville Engineering) perpendicular to the zirconia surface (0.25 MPa pressure, 15 seconds, 10-mm distance). For stone roughening, one surface of each block was abraded with a wheel diamond stone (125-150 um) in a circular motion for 5s with gentle hand pressure by one operator. The zirconia blocks were then placed in an ultra-sonic bath (Elmasonic One; Elma) in ethanol for 5 minutes.

The mean surface roughness (Ra) in Um was measured for each group using TR200 surface roughness tester (Times group incorporated, USA)

The Zirconia blocks were further subdivided into two subgroups whether treated with zirconia primer or not, thus there were 6 experimental groups according to the surface modification done and adhesive/primer application protocols used.

The subgroups were as follow:

1-No surface modification/all bond universal adhesive / curing / flowable composite repair material.

2- No surface modification/Zirconia primer plus/ all bond universal adhesive / curing / flowable composite repair material.

3- Air born particle abrasion (50um Alumina)/ all bond universal adhesive / curing / flowable composite repair material.

4-Air born particle abrasion (50um Alumina)/ Zirconia primer plus/ all bond universal adhesive / curing / flowable composite repair material.

5- Wheel stone abrasion (125-150 um)/ all bond universal adhesive / curing / flowable composite repair material.

6- Wheel stone abrasion (125-150 um)/ Zirconia primer plus/ all bond universal adhesive / curing / flowable composite repair material.

The bonding of repair material was applied to an area of 1 mm in diameter standardized with a perforated adhesive tape (Adhesive vinyl, SRA3; Xerox). In group1, 3 and 5: one coat of all bond universal adhesive was applied with an application brush, air thinning for 5s was done followed by light curing for 15s using (Ortholux LED Curing Light; 3M Unitek), then 1mm internal diameter Tygon tube of 2mm height was fitted over the bonded area and filled by flowable composite material (Reveal, Bisco, USA) followed by 15s light curing.

In group2, 4 and 6: The same procedures were performed as in groups 1,3and 5 except that Zirconia primer plus was applied and air thinned for 5s before the step of all bond universal adhesive.

The light curing device has an output of 800 mW/cm² the power was continuously checked with a radiometer (Demetron LED Radiometer; Kerr Corp)

Specimens were stored in distilled water at 37°C for 48 hours before testing. Shear Bond Strength was measured with a single-plane lap device in a universal testing machine (Instron

model 4502, Instron Ltd) with a 1 kN load cell and at a crosshead speed of 1 mm/min.

For mode of failure analysis all specimens were scanned using scanning electron microscope. The specimens were sputter coated with gold in Sputter coater (S150A, Edwards instruments, London, UK). The sputter coated specimens were examined using SEM (Joel, Tokyo, Japan) operated at 30 kv. The failure was classified by 2 in-dependent observers as adhesive if the failure occurred at the adhesive interface and as mixed if a combination of failures such as adhesive and cohesive was observed in the composite resin. Data were statistically analyzed with software (IBM SPSS Statistics 20; SPSS Inc). SBS data were submitted to 1-way ANOVA, followed by Tukey post hoc tests The Kruskal-Wallis and Fisher least significant difference post hoc tests of the ranked data were used to analyze failure mode data. The significance level was set at P-value ≤ 0.05 .

RESULTS

The results are shown in tables (1, 2 and 3) The roughness testing showed that the least Ra value was recorded to the as fired no ttt group(0.05um +0.01um)followed by air born particles alumina abraded Zirconia (0.09um + 0.02um) whereas the highest roughness value was recorded to diamond stone roughened Zirconia surfaces that recorded mean Ra value (0.14um +0.06). The two way ANOVA(table 1) for comparing the two factors affecting SBS (surface treatment and Zirconia primer) showed that surface treatment of zirconia had a significant effect on SBS where air born abrasion group was significantly higher than no treatment group and stone grinding group. On the other hand Z primer group was significantly higher than no primer group.

Table 1) Two way ANOVA showing the effect of test factors and their interaction on SBS

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Surface ttt	588.829	2	294.414	43.807	.000
Z primer	86.339	1	86.339	12.847	.001
Surface ttt * Z primer	7.472	2	3.736	.556	.577

The one way ANOVA showed that there is a significant difference between the tested subgroups in μ SBS (table 2).

Table (2) one way ANOVA for SBS showing a P value less than 0.05 indicating significant differences between subgroups.

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	682.639	5	136.528	20.315	.000
Within Groups	362.915	54	6.721		
Total	1045.554	59			

The post hoc Tukey test showed that the air born abrasion subgroups were statistically significantly higher than all other subgroups. Furthermore the two air born abrasion subgroups were not statistically significantly different.

Table (3) Means ± SD for Experimental subgroups

Experimental subgroups	Mean ± SD
No TTT No Zprimer	19.5 ^b ± 2
No TTT Zprimer	17.4 ^b ± 1.24
ABA no Zprimer	23.8 ^a ± 2.7
ABA Zprimer	27.2 ^a ± 4.2
S G no Zprimer	18.6 ^b ± 2.7
S G Zprimer	20.3 ^b ± 1.7

Means with the same superscript letter are not statistically significantly different
 No TTT= no treatment, ABA= Airborn abrasion, SG= Stone grinding.

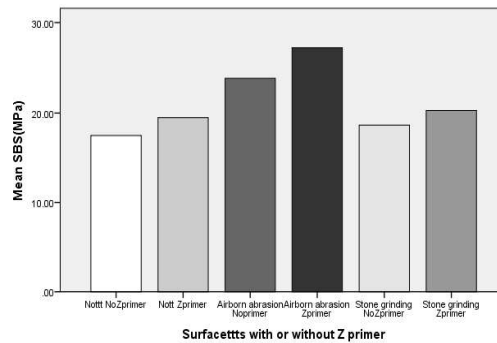


Fig (1) Bar chart representing mean SBS in MPa for different subgroups.

The fracture mode analysis using SEM analysis (fig. 2) and table

(4) Revealed that the most failure mode was adhesive 63.3% then mixed failure mode 30% and the least recorded failure mode was cohesive 6.6%.

Table (4) Failure mode recorded in each subgroup

	Adhesive	Mixed	Cohesive	Total
No TTT no Zprimer	8	2	0	10
No TTT Zprimer	7	3		10
Air born abrasion No Zprimer	6	3	1	10
Air born abrasion Zprimer	5	3	2	10
Stone grinding No Zprimer	7	2	1	10
Stone grinding Zprimer	5	5	0	10
total	38	18	4	60

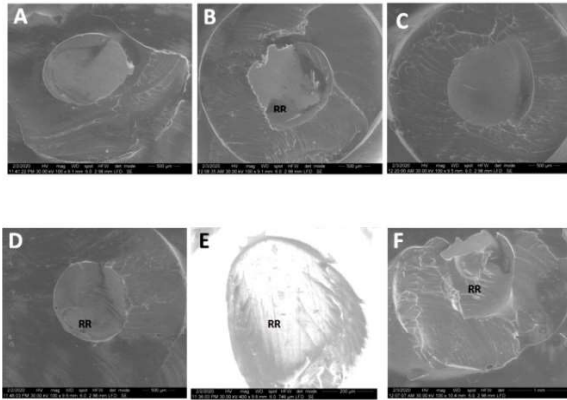


Fig. (2) Representative SEM photomicrographs for different tested subgroups showing failure modes. Adhesive failure was prominent in groups without Z primer pretreatment (A and C). However mixed failure was prominent with Z primer pre treatment (B and D). In case of air born particle abrasion group mixed and cohesive failures were prominent regardless of Z primer pretreatment(E and F) as indicated by repair material remnants (RR).

DISCUSSION

Veneer chipping and delamination is a common complication in zirconia crowns.

The combination of veneering ceramics and zirconia as framework material is one consisting exclusively of brittle materials. Both partners have no ductility and therefore are not able to compensate the overall tensions. Since the veneering ceramic has significantly lower strengths than the zirconia, it fractures more rapidly. This occurs because of the difference in coefficient of thermal expansion between both partners.

An extremely thin layer of veneering ceramic remains on the zirconia framework, and this is defined as a cohesive fracture. Because the cohesive fracture occurs in the veneering ceramic, and this can reasonably be concluded that the bonding strength between zirconia framework and veneering ceramic are good.

The null hypothesis that surface treatment and zirconia primer would not influence the shear bond strength at the zirconia-composite resin interface was rejected.

The present study was based on the expectancy that changing the morphology, roughness, and texture of the zirconia surface could influence the adhesive bonding at the zirconia-composite resin interface. In addition the application of zirconia primer plus would affect the bond strength or not. The interaction between surface treatment and zirconia primer was significant.

The importance of micromechanical retention on the adhesive bonding at the zirconia-composite resin interface was shown in this study when the specimens that were not airborne-particle abraded, showed less shear bond strength than that roughened either by stone grinding or air born abraded.

It indicates that the bond strength between the restorative material and bonding agents is highly dependent on micromechanical retention.

Once micro mechanical retention is present, the chemical bond can improve the bond strength between the zirconia and composite repair material.

Since the use of the new multimode adhesives and the ZPP application protocols influenced the SBS of composite resin to zirconia, Even though recently an increase has been observed in the use of anatomically contoured zirconia, porcelain-fused-to zirconia is still widely used, and the intraoral repair of fractured veneered zirconia restorations with composite resin is an important challenge for a clinician. Achieving high bond strength is central to the clinical success of any restoration.³¹

In previous studies, the mechanical conditioning of the zirconia surface

has proven efficient in creating micro retentions and improving adhesion.³²

Increasing zirconia roughness not only led to a higher surface area for

Micro mechanical retention, but also increased the surface energy and therefore wettability and adhesion.³³

Zirconia airborne-particle abrasion with 50 μm Al_2O_3 under a pressure of 0.25 MPa has proved to be effective for that purpose.³⁴

Therefore, in the present study, some specimens were abraded by the air born particles, using stone grinding or without any surface treatment.

According to the results the micro mechanical roughing using the stone grinding doesn't produce the favorable surface roughness for bonding as that of air born abrasion. That can explain our results as the air born specimens gives higher results than that of stone abrasion

Meanwhile, the specimens without surface treatment showed the least SBS results. and this indicate the importance of creating surface micro roughness as an essential step for zirconia repair.

However, micro retention has not been sufficient to achieve clinically acceptable bonds, and so different adhesive systems have been introduced to establish a chemical union to zirconia.

Therefore, The MDP monomer has 2 functional groups, a phosphoric acid group that is responsible for bonding to hydroxyl groups present at the zirconia surface and a carboxylic acid group (methacrylate) that can be light polymerized and will bond to composite resin.³⁵

New multimode 1-bottle adhesives, for which manufacturers claim a zirconia bonding ability, incorporate the MDP molecule in their composition.

Despite the presence of MDP in the composition of all bond universal, the use of zirconia primer in a separate step before adding the adhesive is an essential step to increase the SBS as in the obtained results.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Preparation of zirconia surface with air born abrasion is the most effective and

essential step before bonding repair material to zirconia.

2. Zirconia primer plus application before applying the bonding agent would improve the adhesive bond strength of repair material regardless of the type of surface treatment.

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