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Effect of Different Surface Treatments on Bond Strength to Tetragonal and Cubic Zirconia

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Abstract

Background: Dental zirconia has been widely used due to its superior mechanical properties. However, traditional etching techniques and surface treatments are generally ineffective on zirconia surfaces due to its inertness and lack of a silica phase. Hence, various surface treatments are applied to improve bonding to zirconia.

Objectives: The aim of this study was to evaluate the effect of three different surface treatments: low pressure air borne particle abrasion, selective infiltration etching and fusion sputtering on bond strength to both Tetragonal zirconia and Cubic zirconia.

Materials and Methods: Ninety two specimens of zirconia were used in this study. Two of the specimens were investigated for surface analysis and ninety specimens was divided into 2 groups according to type of zirconia used cubic zirconia (Bruxir anterior) and tetragonal zirconia (Cercon), specimens of each group will be divided into 3 subgroups according to type of surface treatments low pressure airborne-particle abrasion, selective infiltration etching and fusion sputtering, then will be divided into 3 divisions according to time interval as immediate, thermocycling and water storage.

Results: Statistical analysis of data revealed significant differences in surface treatments on shear bond strength ($F=124$, $P<0.001$) between the three types of surface treatments, with SIE associated with the highest bond strengths and particle abrasion with the lowest. The mean of shear bond strength of tetragonal zirconia specimens as immediate ranged from 26 to 22.80 MPa, thermocycling specimens ranged from 21.10 to 16.30 MPa, specimens stored for12 months ranged from 21.40 to 13.90 MPa. While the mean shear bond strength of cubic zirconia as immediate ranged from 20.70-16.20 MPa, thermocycled specimens ranged 15-5.20 MPa and specimens stored for 12 months ranged from 14.80- 3.30 MPa. Tetragonal zirconia showed higher bond strength values than cubic zirconia. There were significant differences between the three tested time intervals ($F=88$, $P<0.02$), the immediate group showed the highest bond strength while the other two groups (15000 cycle and 12 month) showed reduced bond strength.

Conclusion: Within limitations of this study, Selective infiltration etching produced the highest shear bond strength compared to other surface treatment. Selective infiltration etching is a promising surface treatment for both cubic and tetragonal zirconia. Both thermocycling and water storage significantly affected the shear bond strength of both cubic and tetragonal zirconia.

Keywords: Bond Strength, Tetragonal zirconia, Cubic Zirconia, Selective infiltration etching, Fusion sputtering, Low pressure airborne-particle abrasion.

INTRODUCTION

Zirconium oxide (ZrO_2), is a bioceramic that was first identified by the Chemist Martin Heinrich Klaproth in 1789 [1,2] . Zirconium oxide has been used in dental restoration applications since 1998 [3,4]. In vivo studies have showed a great biocompatibility of zirconia [5,6].

Yttria partially-stabilized tetragonal zirconia polycrystals 3Y-TZPs contained 0.25 wt% alumina (Al_2O_3) sintering aid and exhibited more than 1 GPa in flexure strength [7,8]. However, those types of zirconia ceramics had high opacity because of the inherent birefringence of non-cubic zirconia phases, which results in light scattering from grain boundaries, pores, and additive inclusions. They were indicated as fixed dental prostheses in posterior and anterior regions and framework materials in porcelain veneered crowns. [9, 10]

Recent development of monolithic zirconia (cubic zirconia) includes transparent phase in the final product to decrease opacity. This was achieved by increasing yttria content to produce partially stabilized zirconias, 4 mol% (4YPSZ) or 5 mol% (5Y-PSZ), with increased amounts of nonbirefringent cubic phase. This markedly improved translucency, but with decrease of both flexural strength and fracture toughness because cubic zirconia does not undergo stress-induced transformation [11].

Traditional adhesives are not effective on zirconia ceramics surfaces, since they are essentially inert and non-polar. moreover, acid etchants like hydrofluoric acid do not sufficiently roughen the surface for micromechanical retention. Air abrasion with Al_2O_3 particles and use of a tribochemical silica coating allows for chemical bonding to a silane coupling agent and to resin cement. [12] This procedure that does not produce bond strengths coperable to those reported for silane bonded porcelain [13,14]

Bond strength of zirconia to resin cements has been improved by conditioning the zirconia surface with chemical and mechanical pre-treatment techniques [15,16] Such as alumina air abrasion, laser irradiation, tribochemical silica coating, ceramic coating and chemical etching can improve the bond strength of zirconia to resin cements due to an increase of surface roughness and micro-mechanical interlocking [17,18,19,20,21]. Recently newer surface treatments were introduced such as Selective infiltration etching, Low pressure airborne-particle abrasion and Fusion Sputtering.

The aim of this study was to evaluate the effect of three different surface treatments: low pressure air borne particle abrasion, selective infiltration etching and fusion sputtering on bond strength to both Tetragonal zirconia and Cubic zirconia. The null hypothesis of this study was that various surface treatments have the same bond strength on cubic and tetragonal zirconia.

MATERIALS AND METHODS

Table 1: Materials used in the study

Material	Manufacturer	Composition
BruxZir Anterior Solid Zirconia	Glidewell Dental Laboratory	ZrO_2>89 wt%, Y_2O_3<12 wt%, HfO_2<4 wt%, Al_2O_3<0.05 wt%
Cercon base zirconia	Degudent GmbH, Hanau-Wolfgang, Germany	Zirconium oxide (92%vol), yttrium oxide (5%vol), hafnium oxide (2%vol), alumina and silica (<1%vol)
Panavia V5	Kuraray Co Ltd, Tokyo, Japan	Paste A & B Clearfil ceramic primer plus
Clearfil AP-X Esthetics	Kuraray Co Ltd, Tokyo, Japan	Silanated barium glass filler, Pre-polymerized organic filler, Bis-GMA, Hydrophobic aromatic dimethacrylate, dl-Camphorquinone

Ninety two bar shaped samples of zirconia size (19 x 10.22 x 1 mm) were used in this study. Specimens were divided into two equal groups according to type of zirconia used Cubic Zirconia (Bruxzir anterior) and Tetragonal Zirconia (Cercon

Base). Specimens of each group were further subdivided into 3 subgroups (15 samples each) according to type of surface treatments Low Pressure Airborne-particle Abrasion, Selective Infiltration Etching and Fusion Sputtering, each group of surface treatment were also divided into three divisions according to time interval between cementation and shear bond testing as Immediate, Thermocycling and Water Storage. One sample from each zirconia group was used for surface analysis.

Table 2: Samples groups

Surface Treatment / Material	Cubic Zirconia	Tetragonal zirconia
Low pressure airborne-particle abrasion	15	15
Selective infiltration etching	15	15
Fusion sputtering	15	15
Surface Analysis	1	1

Preparation of zirconia blocks:

Zirconia samples were prepared using precision cutter* with a diamond coated cutting disc** under water for cooling. All samples were manually polished on all sides using #2000, #1200, #1000, #800 and #800 Al_2O_3 polishing papers for 1 minute under water to produce smooth surface. All specimens were cleaned ultrasonically in 90% ethanol for 20 min to remove any contamination. Low pressure airborne-particle abrasion and selective infiltration etching specimens undergo sintering using sintering furnace*** according to manufacturer instructions (Table 3), fusion sputtering samples were sintered after surface treatment. The size of Ninety two zirconia samples was checked by using digital caliper after sintering (19 x 10.22 x 1 mm).

Table 3: Sintering Protocol for both types of zirconia

Bruxzer sintering	Cercon base sintering
1st Heating Rate – 15°C/min to 1200°C	1st Heating Rate – 22°C/min to 900°C
1st Holding Time at 1200°C – 60 minutes	1st Holding Time at 900°C – 0 minutes
2nd Heating Rate – 2°C/min to 1300°C	2nd Heating Rate – 11°C/min to 1500°C
3rd Heating Rate at 10°C/min to 1530°C	
Sintering Temperature – 1530°C	Sintering Temperature – 1500°C
2nd Holding Time at 1530°C – 150 minutes	2nd Holding Time at 1500°C – 145 minutes
Cooling Rate – 15°C	Cooling Rate – With closed furnace cooling down to 200 °C

Surface analysis:

One sample from each type of zirconia was used for surface analysis. Surface roughness parameters (R_a, R_p, and R_v) values were recorded using a non-contact laser surface profilometer****1 where R_a is the mean surface roughness, R_p is the peak surface roughness, and R_v is the valley surface roughness**.** Surface hardness was recorded using Vickers micro-indentation tester***** using 2 kg load for 30 sec contact time. Zirconia specimens were prepared for scanning electron microscopy******, gold sputter coated and examined to study the internal structure, grain size and grain boundary regions of both cubic and tetragonal zirconia.

Surface treatments:

A- Low pressure airborne particle abrasion:

The samples were mounted in a holder at a distance of 10 mm from tip of the sandblaster machine*. Specimens were abraded with 50 µm alumina particles for 15 s, 1 bar air pressure. The incidence angle of particle delivery was maintained at 90° with nozzle diameter 0.8 mm.

* MICRACUT®150
** Diamond wafering blade, No 11-4276; Buehler
*** TABEO-1/M/ZIRKON-100,MIHM-VOGT GmbH & Co. KG, Germany

**** Profilm 3D, Filmetrics Inc
***** HM-210/220 Series 810- Micro Vickers Hardness Testing Machines, Mitutoyo Inc
****** Jeol 126, Jeol ltd, Tokyo, Japan
* Basic eco fine sandblasting unit, Renfert GmbH Untere Giesswiesen 2 78247 Hilzingen, Germany

B- Selective infiltration etching:

A glass conditioning agent composed of silica (65%wt), alumina (15%wt), sodium oxide (10%wt), potassium oxide (5%wt), and titanium oxide (5%wt) were applied in a thin layer on the samples. The glass powder was mixed with 70% ethanol to achieve a creamy mix, it was evenly sprayed on the surface using compressed air. The specimens were then heated in open air to 750°C for 3 min and cooled to room temperature. The heating and cooling rates (90°C/min) were programmed in an electrical furnace**. All traces of the glass conditioning agent were completely removed by subjecting the specimens to 15 min of ultrasonic cleaning in 5% hydrofluoric acid followed by washing the specimens under running water for 15 min. [27]

C- Fusion Sputtering:

Five grams of unsintered zirconia powder** * and a 1mm zirconia ball were placed in a plastic capsule. Placing sealed capsule in an electric mixer for 45 min to achieve fine powder of zirconia. Only particles of size 7-µm to 12-µm were selected by shaking the zirconia powder through fine stainless steel meshes. Repeating the process several times until 50 g was obtained of the required powder. Adding 10 grams of the selected powder to a glass jar filled with 10 ml of 50% ethyl alcohol and the mixture was placed in an ultrasonic shaker to allow even distribution of the particles. Immediately after mixing add dye material to color the powder to be distinguished then, the suspension was transferred to a compression glass container used to spray paint (Figure 1), and the air pressure was adjusted to 0.3 MPa. The ceramic spraying nozzle**** was adjusted and kept at a constant distance from the zirconia specimens (20 mm) using a plastic rod attached to the nozzle. A manual flow controller was used to maintain a constant spray*****. First, two short jets were released from the nozzle onto black paper until a constant mixture was observed, then the surface of the zirconia disks was sprayed for five seconds. The surface-sputtered zirconia disks were stored at 60°C for 2h to allow proper drying of the surface before sintering according to the manufacturer's instructions (Table 3). [24]

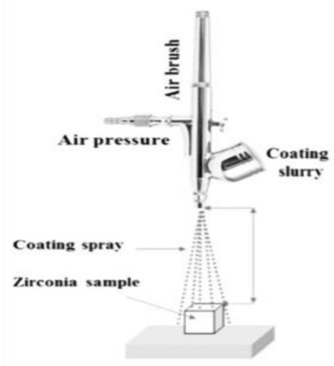

Figure 1: Compression glass container

Bonding procedure:

Specimens of zirconia were cleaned ultrasonically with 90% ethanol for 20 minutes to remove any contaminations on the surface. Composite discs (CLEARFIL AP-X Esthetics) were prepared using metal mold diameter (3.25 mm x 1.55 mm), which held between two glass slides followed by light curing for 20 sec (according to manufacture instructions) for each surface using elipar s10 light cure* . Six of composite discs were cemented on each specimen of zirconia. The composite discs were cemented on zirconia specimen using resin cement kit (PANAVIA V5). Constant load (3 kg) was maintained during cementation using cementing device. Removal of residual cement using micro brush then light cured for 10 sec. according to manufacture instructions.

** Austromat 3001; Dekema Dental-Keramiköfen; Freilassing, Germany
*** SNE-SS6-CER 08, Spray nozzle engineering; Melbourne, Australia
**** Brooks Model FC8744 NRS, Brooks Instruments; Hat- field, PA, USA
***** E-grade zirconia, Tosoh; Tokyo, Japan

* Elipar S10/3M ESPE

Time Intervals:

Immediate Group:

Thirty samples were subjected to shear bond strength test after 24h of cementation to ensure complete polymerization.

Thermocycling Group:

Thirty samples were stored in distilled water 37°c for 24 hours, then thermocycled in water for 15,000 cycles, 2 min immersion time between 5°C and 55°C using an automated custom-made device, then undergo the shear bond strength test.

Water storage Group:

Thirty samples were stored in distilled water for 12 months, storage media was refreshed every 2 weeks and specimens were kept at 37°C using incubator** then undergo the shear bod strength test

Shear bond strength test:

The shear bond strength was measured by applying an axial load on the bonded interface using a universal testing machine***. Loading was performed at a crosshead speed of 0.1mm/min until failure occurred. Bonded specimens were fixed to a special attachment unit that ensured that the bonded interface was parallel to the loading blade of the universal testing machine. The loading blade had a pre-fabricated circular notch that precisely fitted the diameter of the composite disk to ensure even stress distribution, while the rest of the loading blade was aligned parallel to the zirconia specimen. Axial force was applied by the universal testing machine till failure occurred. Load to failure values was extracted from computer generated file and shear bond strength was calculated by dividing failure load in Newton by surface area of the composite disk. Load cell was calibrated after 10 repetitive measures. (Figures 2)

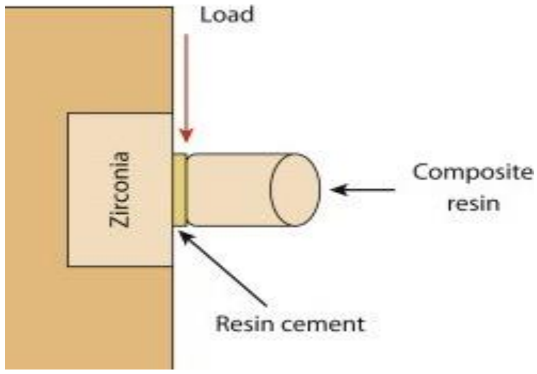

Figure 2: Demonistration of shear bond strength test

Analysis of failure pattern after debonding:

The fractured zirconia specimens were prepared for scanning electron microscopy, gold sputter coated and examined at different magnifications to study fractured surfaces. Failure mode was classified either as interfacial failure were the crack traveled at the zirconia-resin cement interface (considering area of crack origin) or a cohesive failure in the resin cement where the crack originated outside the bonded interface in the resin cement.

Statistical analysis:

Levene's test of equality of error variances was performed to test the null hypothesis that error variance in SBS was similar in tested groups. One way and three-way analysis of variance (ANOVA) were selected to analyze the data with 3 within-group factor (zirconia type, surface treatment, time). Bonferroni post hoc test was selected for pair-wise comparisons (α=.05, n=5). Data were analyzed using computer software*.

** BST 50 20, VEB MLW Dentalfabrik leipzing, Germany

*** Accuforce Elite Test Stand, Ametek, Mansfield & Green Division 8600 Somerset Drive Largo, Florida, USA

* SPSS 14.0; SPSS, Inc, Chicago, Ill

RESULTS

Surface Analysis:

SEM examination of cubic zirconia revealed a much larger grain size in range of (1.2-1.6µm) with more refined grain boundary regions (Figures 3,4), while tetragonal zirconia was composed of smaller round grains with average size of (0.2-0.3µm) demonstrating homogenous and thick grain boundary regions (figures 5,6). These differences were associated with higher surface hardness and lower surface roughness parameters for high translucency zirconia as shown in (Table 4) (Figures 5,6).

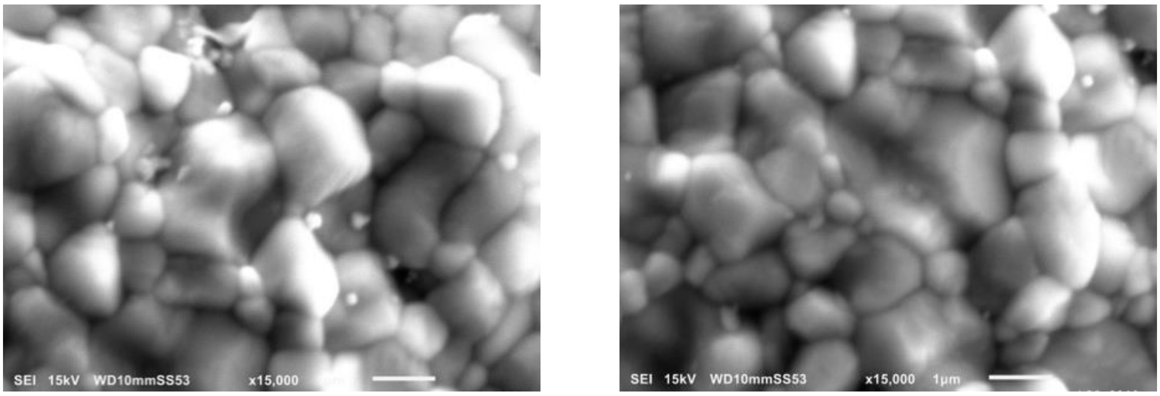

Figure 3,4: SEM image, ×15,000, showing internal cubic grain size ranging 0.9-1.9µm in size

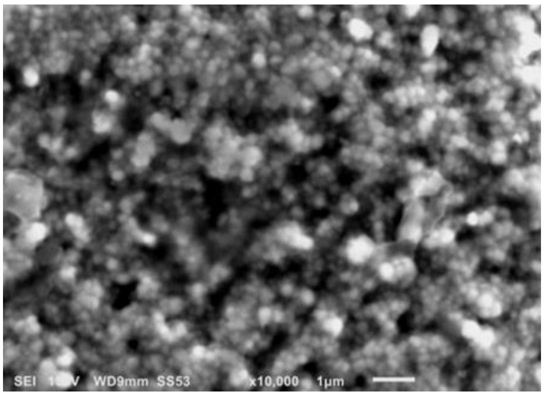

Figure 5: SEM image, x10,000 showing smaller homogenous rounded grains of tetragonal zirconia.

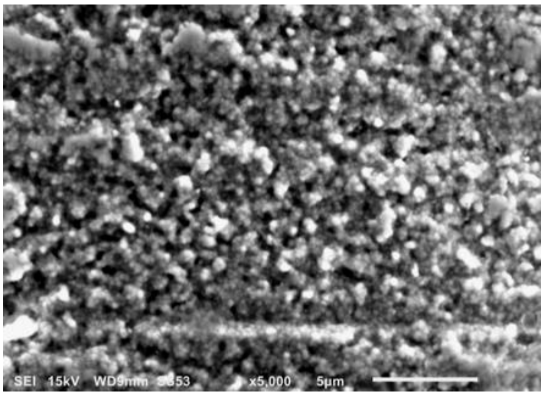

Figure 6: SEM image, x5,000, showing smaller homogenous rounded grains of tetragonal zirconia.

Table 4: Surface properties of the two types of zirconia as-sintered

Materials	Surface roughness			Surface Hardness (VHN)
	R_a	R_v	R_p	
Tetragonal	1.98	9.2	7.2	1290
Cubic	1.46	8.6	6.9	1470

SEM examination revealed that airborne particle abrasion increased the surface roughness of **both types of zirconia** by producing surface scratches and indentations. Selective infiltration etching was associated with the creation of surface and subsurface porosities, forming a three-dimensional network of interconnected channels. Fusion sputtering was associated with characteristically fused surface beads, creating areas of micromechanical retention and interlocking. (Figures 7,8,9,10,11,12)

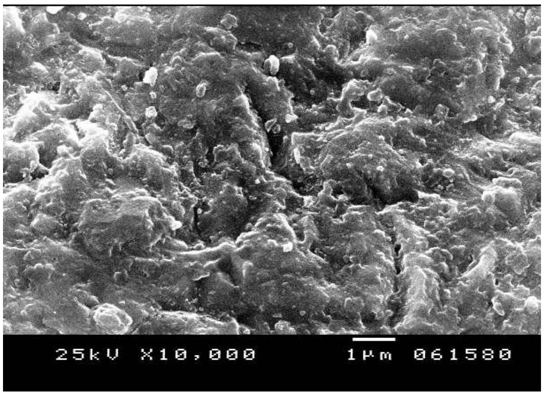

Figure 7: SEM image of airborne-particle– abraded cubic zirconia showing scratches and abrasions on the surface (x10,000)

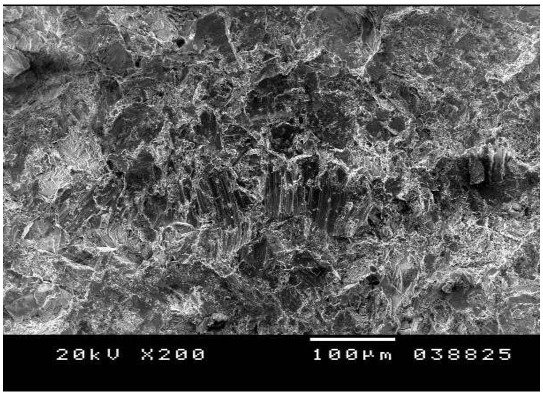

Figure 8: SEM image of airborne-particle– abraded tetragonal zirconia showing scratches and abrasions on the surface (x200)

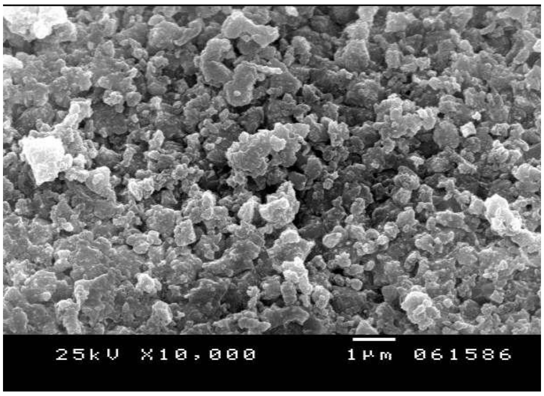

Figure 9: SEM image of selective infiltration etched tetragonal zirconia surface showing nanoporosities and subsurface network of nanospaces where adhesive can infiltrate (x10,000).

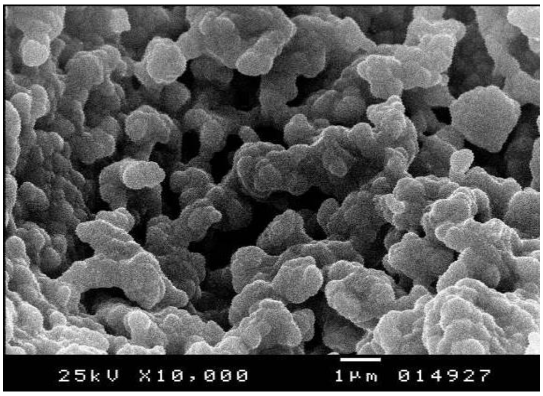

Figure 10: SEM image of selective infiltration etched tetragonal zirconia surface showing nanoporosities and subsurface network of nanospaces where adhesive can infiltrate (x10,000).

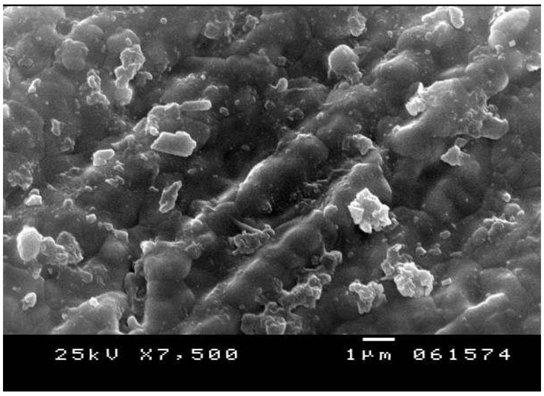

Figure 11: SEM image showing fusion sputtering of cubic zirconia characterized by presence of fused beads on the surface of zirconia (x7,500).

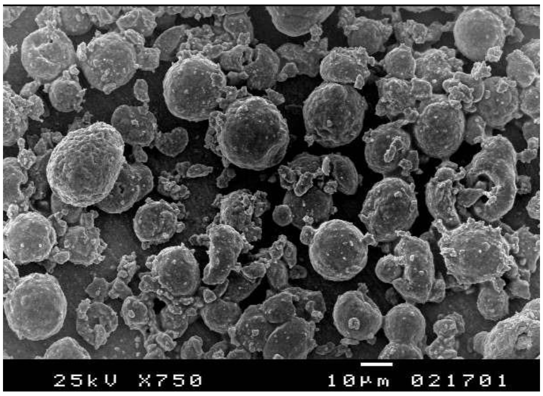

Figure 12: SEM image showing fusion sputtering of tetragonal zirconia base characterized by presence of fused beads on the surface of zirconia (x750).

Shear Bond Strength Test:

Type of Surface Treatment:

Analysis of data revealed significant differences in shear bond strength ($F = 124$, $p < 0.001$) between the three types of surface treatments, with SIE associated with the highest bond strengths and particle abrasion with the lowest.

Zirconia Type:

Analysis of data revealed significant differences between the materials used (F =112, p <0.001) between the two types of material (tetragonal cercon / cubic bruxzir anterior), tetragonal zirconia associated with high bond strength and cubic zirconia with lower bond strength.

Time Interval:

There were significant differences between the three tested time intervals (F =88, p <0.02), immediate group showed the highest bond strength and the other two groups (15000 cycle and 12 month) reduction in bond strength.

In this study the mean of shear bond strength of tetragonal zirconia specimens as immediate ranged from 26 to 22.80 MPa, thermocycling specimens ranged from 21.10 to 16.30 MPa, specimens stored for 12 months ranged from 21.40 to 13.90 MPa. While the mean of shear bond strength test of cubic zirconia specimens as immediate ranged from 20.70 to 16.20 MPa, thermocycling specimens ranged 15 to 5.20 MPa, specimens stored for 12 months ranged from 14.80 to 3.30 MPa.(Table 5) (Figure 13,14,15)

The highest bond strength is the selective infiltration etching over all surface treatments on the three time intervals to tetragonal and cubic specimens, while the lowest is low pressure particle abrasion on the three time intervals to tetragonal and cubic specimens, also tetragonal specimens showed high bond strength over cubic specimens in all surface treatments and all time intervals. (Figure 16)

Table 5: Mean, std. deviation of shear bond strength test on all groups.

Surface treatment	Time interval	Mean/Std. Deviation Tetragonal	Mean/Std. Deviation Cubic
Low pressure particle abrasion	Immediate	22.80 (8.149)	16.20 (1.033)
	15000 cycle	16.30 (2.111)	5.20 (3.795)
	12 month	13.90 (0.994)	3.30 (3.561)
SIE	Immediate	26.00 (1.333)	20.70 (1.160)
	15000 cycle	21.10 (1.101)	15.00 (1.054)
	12 month	21.40 (1.713)	14.80 (0.789)
Fusion sputtering	Immediate	23.00 (1.247)	16.40 (1.265)
	15000 cycle	20.20 (1.317)	8.90 (6.173)
	12 month	17.50 (0.850)	8.90 (6.208)

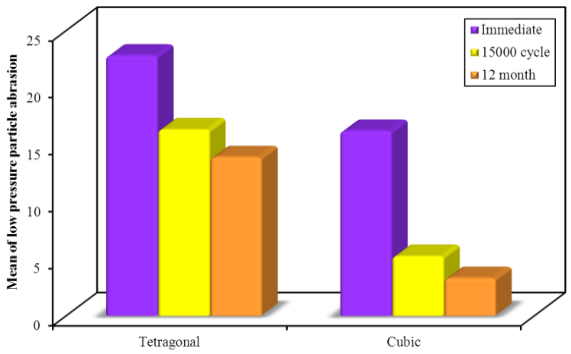

Figure 13: Mean of low pressure particle abrasion surface treatment between tetragonal and cubic zirconia on different time intervals.

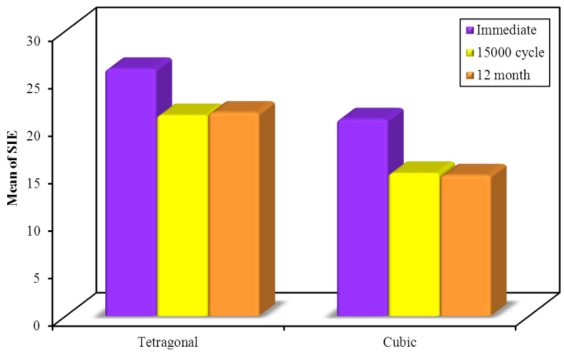

Figure 14: Mean of selective infiltration etching surface treatment between tetragonal and cubic zirconia on different time intervals

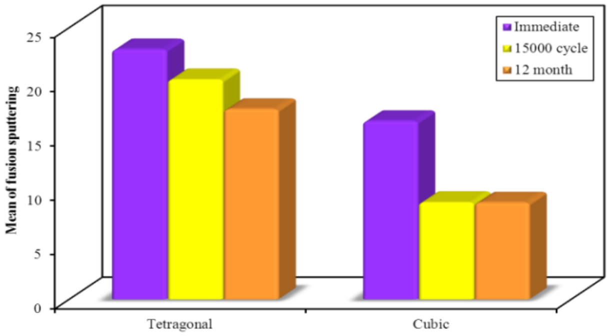

Figure 15: Mean of fusion sputtering surface treatment between tetragonal and cubic zirconia on different time intervals.

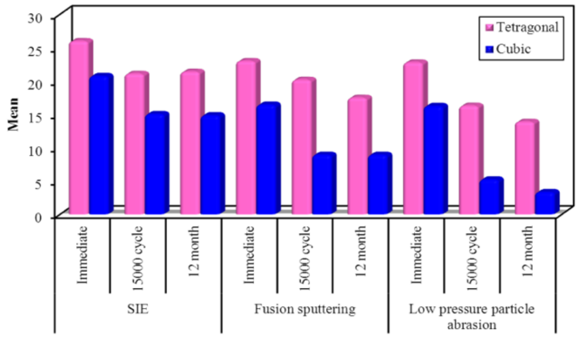

Figure 16: Mean of selective infiltration etching, fusion sputtering and low pressure particle abrasion surface treatments between tetragonal and cubic zirconia on different time intervals.

Failure Pattern:

After debonding pattern of failure were analyzed (Table 6), showed that selective infiltration etching, fusion sputtering and low pressure particle abrasion was predominantly cohesive failure within composite discs. Some examples of cohesive and adhesive failures (Figures 17,18,19).

Table 6: Failure pattern of the specimens after debonding.

Material	Surface Treatment	Cohesive Failure			Adhesive Failure		
		Immediate	Thermocycling 15,000 Cycle	12 Months water storage	Immediate	Thermocycling 15,000 Cycle	12 Months water storage
Tetragonal Zirconia	Selective Infiltration Etching	100%	95%	90%	0%	5%	10%
	Fusion Sputtering	95%	80%	80%	5%	20%	20%
	Low Pressure Particle Abrasion	80%	70%	60%	20%	30%	40%
Cubic Zirconia	Selective Infiltration Etching	95%	90%	90%	5%	10%	10%
	Fusion Sputtering	90%	80%	75%	10%	20%	25%
	Low Pressure Particle Abrasion	80%	75%	70%	20%	25%	30%

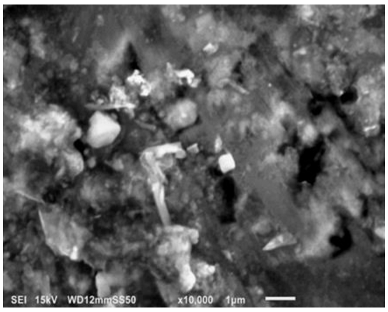

Figure 17: SEM x10,000 shows cohesive failure within composite disc of specimen received Selective infiltration etching.

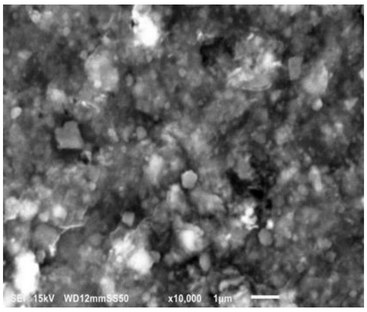

Figure 18: SEM x10,000 shows cohesive failure within the composite disc of specimen received Fusion sputtering.

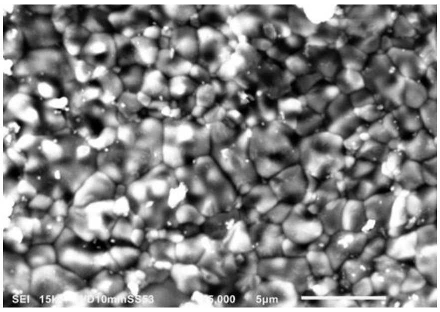

Figure 19: SEM x5,000 shows adhesive failure of specimen received Low pressure particle abrasion of cubic zirconia

DISCUSSION

Although ZrO_2 provide exceptional high flexural strength, bonding on ZrO_2 surface poses an obstacle due to its natural characteristics. The choice of surface treatment, mechanical and chemical, thus plays a key role in the overall stability and effectiveness of bond strength.

There are several methods for mechanical pretreatments such as tribochemical silica coating, airborne partial abrasion, laser irradiation, ceramic coating, chemical etching fusion sputtering and selective infiltration etching can boost the bond strength of zirconia to resin cements due to an increase of micro-mechanical interlocking and surface roughness. [17][18][19][20][21][24][38][31][32-36]

In the present study, shear bond strength was used by applying force in direction perpendicular to bonding plane. In vitro bond strength tests, such as shear, tensile, microshear, microtensile are based on the application of a load to generate stress in the specimens until fracture occurs. The shear bond strength test in one of the most commonly used bond strength tests, because of being fast, easy to preform and also reflecting the clinical situation, and was used to evaluate the bond strength of resin to the ceramic.[15][39][30]

Surface analysis:

The internal structure of the tested zirconia revealed that tetragonal zirconia was composed of submicroscopic round grains with average size of (0.2-0.3µm) and thicker grain boundary regions, Cubic zirconia revealed a much larger grain size in range of 0.9-1.9 µm with more refined grain boundary regions.(Figures 28,29,30,31) The larger gain size of cubic zirconia resulted in higher Vickers hardness number compared to tetragonal zirconia as the loading indenter fell on higher percentage of grains compared to the weaker grain boundaries. Higher hardness was associated with lower surface roughness parameters of cubic zirconia, (Table 4, Figures 32,33). [40][41] Full anatomical cubic zirconia must receive a layer of glazing material in order to seal surface defects and reduce wear of opposing natural teeth [42][43].

The Hall-Petch relation (Grain boundary strengthening method) stated that decrease in the grain size increase the surface hardness of the material. [44] This was not consistent with the current study findings as cubic zirconia showed more surface hardness while it was composed of larger grains compared to tetragonal zirconia which means that the indenter falls all the time on gains not on the gain boundaries. [35] Candido et al 2018 [46], concluded that monolithic zirconia have similar hardness and roughness compared to the tetragonal zirconia, This was not consistent with the current study.

To date, the most highly recommended method and one of the most commonly tested surface treatments used for bonding zirconia restorations is the combination of airborne particle abrasion as a surface treatment with application of a phosphate-based monomer as an adhesion promoter.[47][37][29] This combination was found to resist hydrolysis under water storage for several months.[48]

Fusion sputtering is a simple surface treatment method that does not require any special equipment to perform, and can be simply conducted during preparation of the zirconia framework either chairside or in the lab. The created surface beads become a part of the framework that creates Three-dimensional undercuts that enhance micromechanical retention with the resin cement. Additionally, fusion sputtering increased the surface area of the bonding surface, which explains the high shear bond strength values between resin cement and fusion sputtered specimens and thus the associated cohesive failure. [24][31][49]

The advantage of selective infiltration etching surface treatment is that it only involves surface grains that are exposed to the molten glass, allowing control of the area to be selectively etched, which create 3-dimensional network of intergranular porosity lead the resin cement to penetrate more into zircona. [(27)(26)(23)]

The three surface treatments produced three different types of surface architecture, each with its unique interaction with adhesive.[(22)(50)] Airborne particle abrasion produced microscratches and pits on the surface of zirconia, which increased its average surface roughness. (Figures 34,37) [(51)] However, this surface architecture does not provide sufficient mechanical interlocking with the resin adhesive. [(19)(52)] Selective infiltration etching created a three dimensional network of nanoporosities on the surface of zirconia into which the adhesive can penetrate and interlock, creating a hybrid layer of composite infiltrated ceramic (Figures 35,38).[(27)(28)] In contrast, fusion sputtering created a layer of fused zirconia beads which became a part of the zirconia surface into which the adhesive penetrated more deeply, providing micromechanical retention (Figures 36,39).[(49)]

In addition to mechanical retention, selection of the proper adhesive that will interact properly with the mechanically roughened surface is of equal importance. The role of phosphate monomer as an adhesion promoter is well known when bonding to zirconia frameworks.[(53)] Phosphate monomer is known to facilitate bonding to zirconia by establishing a covalent bond with the terminal phosphate. Moreover, it is known to protect the bonded surface from the hydrolytic effect associated with microleakage.[(19)(55)] Additionally, adhesives containing MDP are hydrophilic, designed to enhance wetting of ceramic surfaces. Compared to hydrophobic bis-GMA–based adhesives, this increased hydrophilicity could result in compromised bonded interfaces, especially during long-term storage.

The null hypothesis was rejected because the results of this study demonstrate different bond strength values on cubic and tetragonal zirconia with the three surface treatments.

In this study, the highest bond strength was obtained with the selective infiltration etching compared with other types of surface treatments of both tetragonal and cubic specimens, while the lowest was low pressure particle abrasion for tetragonal and cubic specimens, also tetragonal specimens showed high bond strength over cubic specimens in all surface treatments. (Table 5) (Figure 16)

Kern M et al. 2009 [(19)] stated that air abrasion at low pressures with suitable adhesive primers is an effective way to form strong, long-lasting bonds between resin composites and zirconia to minimize possible surface damaging effects from air abrasions at relatively high pressures. A less aggressive air-abrasion pressure (1 bar/0.1 MPa) proved to be as effective as the conventional treatment at (2.8 bar /0.28 MPa). Aggressive techniques of surface roughening resulted in the creation of surface defects ending in marked deterioration of the mechanical properties of the restoration. [(23)] The surface defects caused by air abrasion with 110 µm Al_2O_3 at 4 bar were 12 µm deep in the ceramic and gradually declined to 4 µm using a less aggressive sandblasting (50 µm, 2.8 bar) [(56)]**.**

Aboushelib et al 2018 [(31)], Found significant differences in bond strength of the three types of surface treatments (particle abrasion, fusion sputtering and selective infiltration etching), Selective infiltration etching associatesad with the highest bond strengths and particle abrasion with the lowest. Regardless of the surface treatment used, all aging protocols used in that study was significantly decreased the bond strength over 5 years.

Effect of aging and thermocycling on bond strength & Pattern of failure:

The conditions most common for testing the durability of resin bonds are long-range storing water and thermal cycling. Thermocycling in an aqueous environment commonly used in the moist oral environment to simulate mechanical fatigue. Thermocycling temperature changes use mechanical stress on the interfaces of differential expansion and contraction between dissimilar materials. [57]

In this study two protocols for aging were used. Thermocycling was performed for 15,000 cycles [31][26][58] and water storage for 12 months [31][29][49].

Most adhesives experienced a degree of dissolution in water which increased when exposed to other media, eg, acids and bases. [59] Assisted by the hydrolytic pumping action associated with thermocycling, the bonding interface could be considered as a wet environment. Water has a well-known catalytic power that degrades covalent bonds, in addition to its ability to chemically attack polymeric chains and their interstitial spaces. [60][61]

In the early stage of the reaction there must be sufficient wettability to use the hydrophilic property, but excessive hydrophilicity can cause swelling that can adversely affect dimensional stability and mechanical strength, thus increasing hydrophobicity need after the initial reaction. [62] As-sintered zirconia specimens from previous trials reported early adhesive failure during water storage.[26] Surface treatment had a great influence on failure mode.

Christine Keul et al. [25], Seto et al. [57] and Si-Eun Lee et al. [63], studied the shear bond strength of self-adhesive resin cement to zirconia before and after thermocycling. Christine Keul et al. [25]and Seto KB. et al. [57] stated a decrease in shear bond strength of self-adhesive resin cement to zirconia after thermocycling compared to without thermocycling. The authors concluded that thermocycling significantly affects the bond strength of cement; this explains the observed reduction in bond strength in the present study.

In this study, the analysis of the failure patterns, showed that selective infiltration etching, fusion sputtering and low pressure particle abrasion demonstrated predominantly cohesive failure within composite disks (Table 6) (Figures 17,18,19)

These results were inconsistent with Minh et al [34], who tested the bond strength of cubic and tetragonal zirconia as sintered , hydrofluoric acid etching and air abrasion groups to composite discs with adhesive resin cement. They found that the pattern of failure was predominantly adhesive for both cubic and tetragonal zirconia.

While Aboushelib (2012) [24], found that predominant failure type for fusion sputtering and air abrasion groups for tetragonal zirconia was cohesive and the predominant failure type for control as sintered group for tetragonal zirconia was adhesive. In 2018, Aboushelib et al [31], also demonstrated predominantly cohesive failures with the selective infiltration etching, fusion sputtering and air abrasion groups of zirconia.

CONCLUSION

From the results of the present study and within limitations, the following conclusion can be obtained,

1- Selective infiltration etching produced the highest shear bond strength compared with other surface treatment.

2- Selective infiltration etching is a promising surface treatment for both cubic and tetragonal zirconia.

3- Both thermocycling and water storage significantly affected the shear bond strength of both cubic and tetragonal zirconia.

RECOMMENDATIONS

In-vivo studies are needed to validate the in-vitro results and to understand what is the real performance of the bond strength of cubic and tetragonal zirconia in the oral environment.

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