

Evaluation of shear bond strength of zirconia to tooth structure after different zirconia surface treatment techniques

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ABSTRACT

Background: This study aimed to evaluate the effect of zirconia different surface treatments (primer, sandblast with $50\mu\text{mAl}_2\text{O}_3$, Er,Cr:YSGG laser) on shear bond strength between zirconia surface and resin cement.

Material and methods: Sixty presintered Y-TZP zirconia cylinder specimens (IPS e.max ZirCAD, Ivoclar vivadent) will be fabricated and sintered in high temperature furnace of (1500 C for 8 hours) according to manufacturer's instructions to the selected size and shape of (5mm. in diameter and 6mm in height). All specimens were ground flat using 600.800.1000.1200, aluminum oxide abrasive paper to obtain a standardized surface roughness. Surface roughness values were then recorded in μm using surface roughness tester (profilometer) to obtain a standardized data base line for all samples. The specimens were then randomly divided into three main groups (n=20); group A: no surface treatment (control group), group B: specimens in this group treated with $50\mu\text{m Al}_2\text{O}_3$ and group C: specimens in this group treated with Er,Cr:YSGG laser.

Sixty sound human premolars were used in this study, after construction of acrylic blocks, the occlusal surface of the teeth were ground flat, with diamond cutting disk to obtain a flat dentine surface .

Prior to cementation of zirconia cylinders to tooth specimens subgroups (A1,B1,C1) will receive a coat of metal/zirconia primer and left to react for three minutes, while the subgroups (A2,B2,C2) were left undisturbed.

Bonding surface of zirconia cylinder was then luted with SpeedCEM self adhesive resin cement under a static load of 2Kg. placed on the vertical arm of the surveyor and allowed to auto cure for 4minutes.The final cemented specimens were then stored in distilled water at room temperature for 24hours.

All specimens were subjected to shear loading force in a universal testing machine at crosshead speed of 1mm/min. The shear bond strength values were analyzed statistically with one-way ANOVA; the fractured surfaces of zirconia cylinders were examined with a stereo-microscope to observe the failure mode.

Results: The air borne-particle of $50\mu\text{m}$ followed by primer application showed significantly the higher bond strength than other groups.

Conclusion: Within the limitation of this study, the results showed that sandblasting the bonding surface of zirconium cylinders with $50\mu\text{mAl}_2\text{O}_3$ produced the highest values of shear bond strength , also the use of primer enhanced shear bond strength as well.

Keyword: Zirconia surface treatments, shear bond strength. (J Bagh Coll Dentistry 2015; 27(1):77-85).

INTRODUCTION

In recent years, there is increasing demand for metal free restoration due to the increasing interest in aesthetic. Both patients and clinicians have been seeking superior aesthetic metal free tooth colored restorations ⁽¹⁾.

All ceramic restorations provide the most aesthetic pleasing restorations; significant effort has been made over the years to improve their brittleness and low tensile strength. Zirconium oxide-based materials, especially yttria-tetragonal zirconia polycrystals (Y-TZP), were introduced for prosthetic rehabilitations as a core material for single crowns, conventional and resin-bonded fixed partial dentures (FPDs) ⁽²⁾ and, in dental implantology, as abutments or implants Furthermore the combination of Y-TZP and computer-aided design/computer-aided manufacture (CAD/CAM) systems reduces the number of steps in prosthetic manufacturing and eliminates the variables introduced by the manual procedures of the dental technician. ⁽³⁾

Y-TZP exhibits exceptional physical and mechanical properties, such as high flexural strength, fracture toughness, hardness, wear and corrosion resistance in acidic and basic ambient conditions, translucency colour stability, greater effectiveness of diagnostic radiographs ^(4, 5, 6) and high biocompatibility.

Moreover, the polycrystalline structure, which lacks a glass matrix, makes zirconia ceramic more resistant to hydrofluoric acid etching and, as a consequence, resistant to chemical roughening ⁽⁷⁾.

Reliable cementation of fixed prosthetic restorations represents one of the most sensitive and crucial tasks during the dental treatment with desirable long term clinical success. Because of its particular structure, zirconia restorations require a special conditioning before cementation in order to achieve a strong bond to dentine, as the clinical success of ceramic restorations depends on the cementation process ⁽⁸⁾.

For this reason, different approaches have been used to enhance the bond between the zirconia and resin cements, such as coating methods ⁽⁹⁾, a selective infiltration-etching technique ⁽¹⁰⁾. Phosphate ester monomer, 10-methacryloyloxydecyl dihydrogen phosphate

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(MDP) based materials (4), Surface roughening by airborne-particle abrasion, and surface roughening by the use of laser.

MATERIALS AND METHODS

Fabrication of zirconia samples:

Sixty zirconia cylinders were milled from Pre-sintered Y-TZP zirconium oxide blocks (IPS e.max ZirCAD, Ivocalr Vivadent), with the dimensions of (19 mm, 15.5mm, 55mm), each zirconia blank was detached from its fitting pin, each blank was divided into two halves with a cutting saw, each half was divided longitudinally into four equal parallel sided shaped blank by a cutting saw as well, each part was then glued into the fitting pin Figure (1), the fitting pin was then placed into the designated place in the milling machine. A straight hand piece with a carbide round bur operating at high speed was fixed to the movable member of the milling machine in a way allowing back and forth free movement of it along the zirconia parallel sided blank figure (2), in this way each parallel sided blank was cut into a cylinder shaped blank of 6.25mm in diameter figure (3), Each cylindrical blank was cut by a diamond cutting disk into 3 cylinders, each cylinder was measured 7.5 mm in height figure (4).



Figure 1: Parallel sided blank glued on the fitting pin



Figure 2: Milling and cutting the zirconium parallel sided blank



Figure 3: Cylindrical zirconia blank

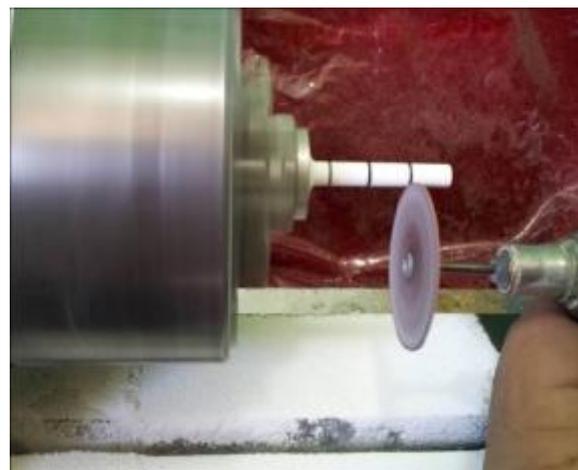


Figure 4: Cutting cylindrical blank with diamond disk

The obtained cylinders were then sintered in tube furnace (Infire HTC speed sintering furnace, Sirona) at 1500 °C for 8 hours including cooling, according to manufacturer's instructions. During this process a 3-dimensional volumetric

shrinkage of the milled cylinder of approximately 20% took place that is why the cylinders were milled approximately 20% larger.

Following sintering each zirconia cylinder was measured approximately (5mm in diameter, 6mm in height) figure (5)



Figure 5: Final shape and size of the zirconia cylinders

The bonding surfaces of zirconia cylinders were then ground flat using a grinding machine and polished consecutively with 600, 800, 1000 and 1200-grit silicon carbide abrasive papers under water cooling to obtain standardized surface roughness⁽¹¹⁾, to facilitate handling the zirconia cylinder during the polishing process a custom made holder was fabricated figure (6).



Figure 6: Custom made holder

Surface roughness for each sample was then confirmed by the use of the profilometer to ensure standardization.

Before the surface treatments, all specimens were ultrasonically cleaned in distilled water for 3 minutes to remove contaminants. Then, the specimens were randomly assigned into three groups of equal size, (n = 20), according to the surface treatments were used.

Specimens grouping

Sixty zirconia cylinders were divided into 3 main groups (n=20) according to the surface treatment that had been applied:

Group A: 20 zirconia cylinders were left without treatment (control group)

Group B: 20 zirconia cylinders were treated with Er,Cr:YSGG laser.

Group C: 20 zirconia cylinders were treated with sand blast (50 μm Al_2O_3).

Prior to cementation each group was subdivided into two subgroups one of them received a coat of primer while the other was without treatment

Treatment of the bonding surface of the zirconia cylinder:

Preparation of zirconia samples before surface treatment:

Prior to surface treatment the specimens were cleaned with 70% ethanol by wiping their surfaces with cotton and subsequently cleaning them for five minutes in an ultrasonic bath with ethanol⁽¹²⁾.

Group A (control group):

No treatment was performed to this group and it is considered as the control group.

Prior to cementation the specimens were cleaned with 70% ethanol by wiping their surfaces with cotton and subsequently cleaning them for five minutes in an ultrasonic bath with ethanol⁽¹²⁾.

Group B (Er,Cr:YSGG Laser treatment)

An Er, Cr: YSGG laser system (Waterlase MD, Biolase) was used on the bonding surface of each zirconia cylinder. A custom made holder was made especially to keep the distance between the tip of the device and the zirconia bonding surface fixed at 1 mm, Er,Cr: YSGG laser, $\lambda = 2780$ nm, pulsed laser-powered hydrokinetics, and the power was 2.5 W with a 6mm quartz core tip (G4, Biolase Technologies Inc., Irvine, CA, Germany) positioned at 1 mm (90°) from the bonding surface of each zirconia cylinder (focused mode)⁽¹³⁾. Repetition rate was fixed on 20 Hz., the air and water were adjusted to 50% of the laser unit. Irradiation was done under the supervision of a laser specialist. Each sample was irradiated in a circular motion for 30 sec. to promote homogeneous irradiation and cover the entire sample area figure (7).⁽¹⁴⁾

Then, the specimens were cleaned with 70% ethanol by wiping their surfaces with cotton and subsequently cleaning them for five minutes in an ultrasonic bath with ethanol⁽¹²⁾.

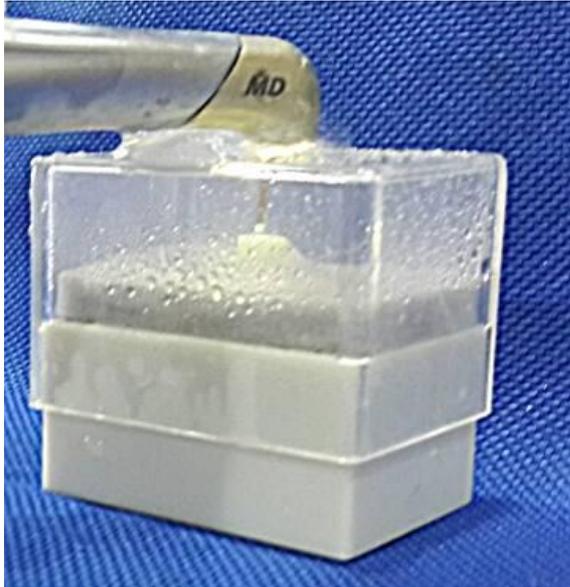


Figure 7: Er,Cr:YSGG, laser irradiating Zirconia bonding surface

Group C (Sand blast treatment)

Zirconia cylinders were mounted in a special holder so that the blasting tip is in a straight line with the sample at a distance of 10mm between the surface of the zirconia cylinder and the blasting tip of the airborne-particle hand-piece. The surfaces of the specimens were air particle abraded for 15 s with 50 μm Al_2O_3 particles at 2.5 bars. Then, the specimens were cleaned with 70% ethanol by wiping their surfaces with cotton and subsequently cleaning them for five minutes in an ultrasonic bath with ethanol⁽¹²⁾.



Figure 7: Placing the blasting tip in a straight line with the zirconium surface

Preparation of teeth samples:

Teeth selection:

Non-carious, un-restored sixty, freshly extracted, upper first human premolars, for orthodontic purposes (the patient age range from 13-20years), of comparable size and shape were selected and collected for this study. All teeth were examined under a magnifying eye lens & light from a light curing unit to check for the absence of caries, cracks, fractures, and

restoration. Only intact teeth free of defect were selected. The teeth were then cleaned from debris by using slurry of pumice in a rubber cup used with a low speed hand piece, then, washed with distilled water. The teeth were stored in normal saline at room temperature until sample preparation.

Construction of acrylic blocks:

A custom made square rubber mold of (1cm x 1cm x 2cm) was used for construction of acrylic blocks for this study. The root of each tooth was embedded along their long axes in mixed cold cure acrylic to about 3mm. Occlusal to the cemento-enamel junction. A dental surveyor was used to position the clinical crown parallel to that of the acrylic resin block.

Preparation of teeth

A standardized occlusal surface reduction was obtained for all samples by using a surveyor; straight hand piece with a diamond cutting disc operating at high speed was adapted to the horizontal arm of the surveyor in such a way so that the long axis of the disc was kept parallel to the long axis of the tooth. The movable table of the surveyor was adapted by using a special mold to hold each sample during cutting procedure to secure each specimen in such way so that the long axis of each clinical crown was parallel to the shaft of the cutting disk. Thus, the long axis of the bur will be kept parallel to the long axis of the tooth sample all the way during occlusal surface reduction.

For each tooth sample the occlusal surface was reduced to the depth of the central groove with diamond disc using high speed hand piece, with copious water cooling, to expose the upper part of the peripheral dentine surface, a new cutting disc was used every (5) teeth.

Then the dentin surface was prepared with 240, 400, and 600 grit aluminum oxide abrasive papers, respectively under running tap water for 10 seconds each to obtain a polished surface⁽¹⁵⁾. The teeth were kept hydrated in distilled water as this storage solution will not alter the permeability of dentine⁽¹⁶⁾. The storage solution was stored in the refrigerator⁽¹⁷⁾. After that the teeth specimens were distributed evenly among the six subgroups.

Bonding of zirconia cylinder to tooth surface

In order to have a standardized bonding procedure, an adhesive tape with a 5mm hole in diameter was fixed on the dentine surface of each prepared tooth to restrict the bonding area to a diameter of 5 mm.

Prior to cementation a coat of Metal zirconia primer was applied to the bonding surface of the subgroups (A2,B2,C2) of zirconia cylinder⁽¹⁸⁾ with a microbrush and left to react for 180

seconds according to manufacturer's instructions, and dried with water and oil free air.

Speed CEM (Ivoclar Vivadent) self curing resin cement was used as a luting agent in this study, it was automixed and dispensed using disposable mixing tips supplied with the cement kit and applied directly as a thin, even layer onto the zirconia cylinder bonding side.

The bonding side of zirconia was then seated onto its respective area on the bonding surface of the prepared tooth with finger pressure. For standardization, a dental surveyor was used during cementation procedure, tooth sample was secured by using a special mold to hold each tooth sample to the movable table of the surveyor, the upper part of the vertical arm of the surveyor was used to apply a static load of 2 Kg. to the zirconium block during bonding procedure to the tooth, this load is used to avoid any internal cement gaps⁽¹⁹⁾ and to standardize the cementation process.

Excess cement was then removed using cotton pellet, light polymerization was carried out for 20 sec. per surface at 1200mW/cm² (following the manufacturer's instructions. Then each cemented specimen was kept under the load for 4 minutes according to the manufacturer's instructions, and kept for one hour to bench set for complete curing.

One hour after cementation, specimens were stored in distilled water in a dark container at room temperature for 24hours⁽²⁰⁾.

Shear bond strength test

The specimens were attached to a universal testing machine, (Tinius Olsen, H50KT, UK).and subjected to a shear force using a stainless steel chiseled-shaped rod with across head at a crosshead speed of 1mm/min until failure occurred. The tested specimens were placed in the lower part (jaw) of the testing machine. While the acrylic block was held in a horizontal position in such a way that the long axis of the chisel shaped rod is placed parallel to the occlusal surface of the tooth, The chisel end of the rod was positioned at the interface between the tooth surface and the zirconia cylinder interface, so that distance between chisel and the interface was 0.1 mm to avoid a cantilever effect on the adhesive surface⁽²¹⁾. The specimen was secured tightly in place so that to ensure that the zirconia cylinder was always at 90 degree to the vertical plane, the specimens were stressed to failure. The Shear bond test values were calculated from this measurement and expressed in MPa. Fig. (2-26).

Shear strength [MPa] = maximum force [N] / bonding area [mm²].

Light Microscope Examination

The fractured specimens were examined after debonding to determine the mode of failure. The specimens were examined under light microscope (BioVision line, Italy) at magnification of 40 X to evaluate the fracture pattern. Failure modes were classified into:

- 1. Adhesive failure:** If more than 75% of the zirconia cylinder surface was visible.
- 2. Cohesive failure:** If more than 75% of the zirconia cylinder surface was covered with resin. All other cases were classified as mixed failures.

RESULTS

Descriptive statistics

A total of 60 measurements of shear bond strength from six groups were recorded for six different surface treatments (Appendix I). The means and standard deviations of shear bond strength with minimum and maximum values for each group are shown in Table (1).

Table 1: Descriptive statistics of the shear bond strength of different zirconia surface treatment.

Groups	Descriptive statistics			
	Mean	S.D.	Min.	Max.
No treatment without primer A1	1.66	0.29	1.12	2.06
No treatment with primer A2	2.77	0.23	2.45	3.06
Laser without primer B1	4.68	0.32	4.13	5.07
Laser with primer B2	5.72	0.28	5.2	6.04
Sand blast without primer C1	7.25	0.38	6.63	7.79
Sand blast with primer C2	8.40	0.52	7.64	9.08

Table (1) shows the lowest mean of shear bond strength was detected in group A1 (1.66±0.29), while the highest mean (8.40±0.52) was detected in group C2.

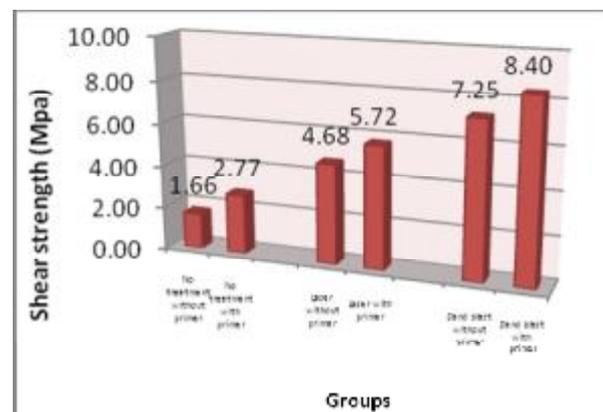


Figure 8: Bar-chart showing the mean values of the shear bond strength of the six groups.

Inferential statistics

Within the single group (Effect of primer)

To see whether there is statistically significant difference within the group (between the subgroups), Student's t-test was applied Table (2).

Table 2: Student's t -test.

Tested groups	Comparison d.f.=18	
	t-test	p-value
A1 vs. A2	9.57	0.000(HS)
B1 vs .B2	7.74	0.000(HS)
C1 vs.C2	5.6	0.000(HS)

*P≤0.05 Significant(S),

P≤0.01 Highly significant (HS),

P≤0.001 very Highly significant (VHS)

t-test showed that group A1 that there is statistically high significant difference between the subgroups according to the application of primer.

Among the groups (Effect of the surface treatment)

To see whether the difference in the mean value for the subgroups (A1,B1,C1) were statistically significant or not, one way (ANOVA) test was applied in Table (3).

Table 3: One way-ANOVA between and within groups (A1,B1,C1).

	Sum of Squares	d.f	Mean Square	F-test	p-value
Between Groups	156.40	2	78.20	708.09	0.000 (HS)
Within Groups	2.98	27	0.11		
Total	159.38	29			

Table (3) shows that the difference in shear bond strength for the groups (A1,B1,C1) were statistically highly significant.

To examine the source of the difference Among the subgroups (A1, B1,C1). Further analysis of these subgroups was performed using LSD test table (4).

Table 4: LSD test among (A1,B1,C1) subgroups

Groups	Mean Difference	p-value
A1 B1	-3.02	0.000 (HS)
A1 C1	-5.59	0.000 (HS)
B1 C1	-2.57	0.000 (HS)

Table (4) shows that there is highly significant difference between subgroups.

And to see whether the difference in the mean value for the groups (A2,B2,C2) were statistically significant or not, one way (ANOVA) test was applied in Table (5).

Table 5: One way-ANOVA between and within groups(A2,B2,C2)

	Sum of Squares	d.f.	Mean Square	F-test	p-value
Between Groups	158.21	2	79.10	584.69	0.000 (HS)
Within Groups	3.65	27	0.14		
Total	161.86	29			

Table (5) shows that the difference in shear bond strength for the groups (A2, B2, C2) was statistically highly significant.

To examine the source of the difference among these groups (A2, B2, C2) Further analysis of these subgroups was performed using LSD test, Table (6).

Table 6: LSD test among (A2,B2,C2) subgroups

Groups	Mean Difference	p-value
A2 B2	-2.95	0.000 (HS)
A2 C2	-5.62	0.000 (HS)
B2 C2	-2.68	0.000 (HS)

Table (6) shows that there is highly significant difference between the subgroups

Mode of failures

The results of failure mode after shear bond testing as observed with a stereomicroscope are summarized in table (7).

Table 7: Modes of failures

Groups	Subgroups	Adhesive failure		Cohesive failure		Mixed Failure	
		No.	%	No.	%	No.	%
Group A	A1	2	20	4	40	4	40
	A2	1	10	5	50	4	40
Group B	B1	1	10	6	60	3	30
	B2	-		8	80	2	20
Group C	C1	-		8	80	2	20
	C2	-		9	90	1	10

This table shows that the predominant mode of failure for the sub groups (C2, C1, B2, B1) was cohesive failure as shown in the Fig (9)

Adhesive failure was observed in the subgroups (A1, A2, B1) as shown in Fig. (10)

Mixed failure occurred in all subgroups in low percentage as shown in fig(11)

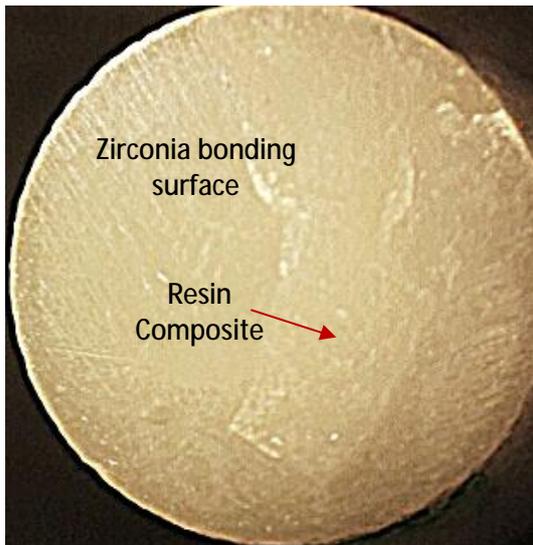


Figure 9: Adhesive failure

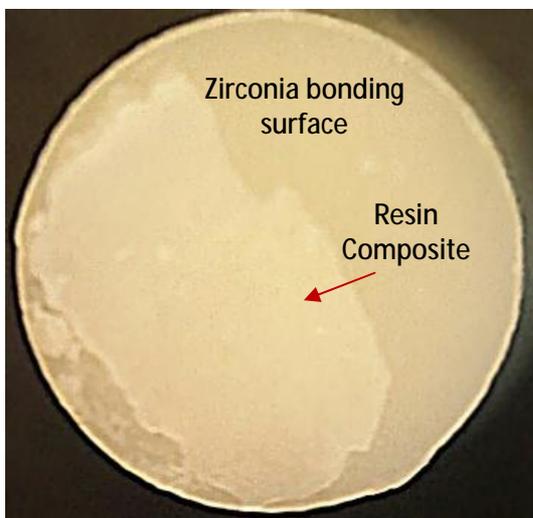


Figure 10: Mixed failure

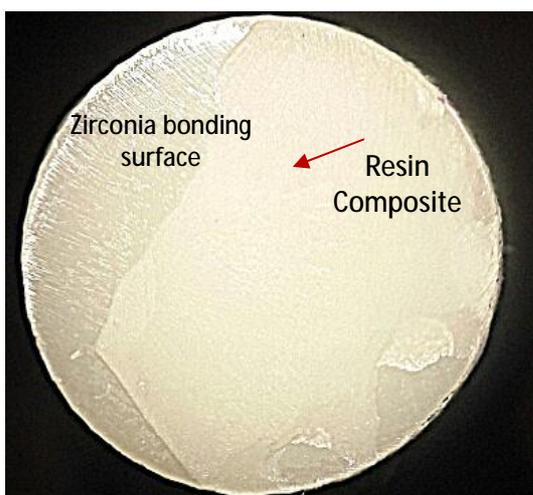


Figure 11: Cohesive failure

DISCUSSION

Effect of primer

The results of this study showed that treatment of the bonding surface of zirconia with metal zirconia surface prior to cementation to tooth surface significantly improve bond strength and this is clearly shown when comparing subgroups (A2,B2,C2) with the subgroups (A1,B1,C1). This can be explained by the fact that phosphonate or phosphate monomers which are the main constituents in metal/zirconia primer, Phosphate monomers bond to zirconia by forming covalent bonds (Zr-O-P covalent bond) with its surface and have polymerizable resin terminal ends that copolymerize with the resin cements.

Furthermore, the surface wetting theory recognizes a key role to the wetting capacity of the primer for improved adhesion. According to this theory the viscosity of the metal/zirconia primer would assist zirconia surface wetting, thus promoting physical adhesion. That could be attributed to phosphonate or phosphate monomers that is the main element in metal/zirconia primer, Phosphate monomers bond to zirconia by forming covalent bonds (Zr-O-P covalent bond) with zirconia bonding surface and have polymerizable resin terminal ends that copolymerize with the resin cements.

This was clearly shown in this study when there was increased bonding strength of zirconia surface to dentine surface when using SpeedCEM self-adhesive luting resin cement containing a functional phosphate monomer with metal/zirconia primer that contains phosphate monomer as well

The result of this study agrees with others⁽²²⁻²⁹⁾ whom stated that the metal primes that contain phosphate monomers, are effective for improving bond strengths between zirconia and resin cements.

The effect Er,Cr:YSGG laser treatment

The result of this study shows that using of Er,Cr:YSGG laser on the bonding surface of zirconia surface resulted in significantly enhanced shear bond strengths compared with the control group, probably because of the surface roughness and irregularities on the zirconia bonding surface that enhance the interlocking with the resin cements.

This result is in agreement with Cavalcanti *et al.*⁽³⁰⁾, whom concluded that laser irradiation on zirconia bonding surface significantly increase shear bond strength due to surface roughness.

However the result disagrees with Ersu *et al.*,⁽³¹⁾ and Aboushelib *et al.*⁽³²⁾. Whom concluded that lasers are not effective to improve the bond strength between ZrO₂ and resin cement, this can

be explained by the fact that the authors in their study used different laser parameters.

However the result of this study showed lower bond strength values accompanied with sandblast treatment.

Effect of sandblast treatment

Treating the bonding surface of zirconia with 50 $\mu\text{mAl}_2\text{O}_3$ Resulted in high values of shear bond strength when comparing it to laser treatment, that may be attributed to the fact that treating zirconia bonding surface with sandblast increases surface roughness and undercuts.

The result of this study agrees with Cavalcanti *et al.* 2009⁽³⁰⁾ who showed an increase in bond strength after air-abrasion with 50 $\mu\text{mAl}_2\text{O}_3$

And disagrees with de Oyague *et al.*⁽³³⁾ Who concluded that air-abrasion on the bonding surface of zirconia substrate did not produce higher bond strength, even though the substrate surface became rougher than the control group, probably because of different grain size, or different pressure used in the study.

Furthermore, the result of this study shows that treating zirconia bonding surface with 50 $\mu\text{mAl}_2\text{O}_3$ produced significant enhancement in bonding strength when comparing it with other treatment subgroups.

Effect of primer with other surface treatments

The result of this study has showed that using sandblast with primer give us the highest mean of shear bond strength when comparing it with other subgroups, this could be explained by the fact that using multifunctional methods ,which mix the ability to create a rough surface for micromechanical interlocking and increase the surface area to establish chemical bond with reactive substances .This was clearly shown in this study when metal/zirconia primer was applied to the zirconia bonding surface of the subgroups (A2,B2,C2) after air abrasion and Er,Cr:YSGG laser treatment, and high significant difference was noticed among these subgroups, when comparing them with subgroups (A1,B1,C1).

The result is an agreement with Yang *et al.*⁽³⁴⁾ , whom stated that the combination of primers and air-abrasion methods tend to produce better bond strength, especially in long term durations.

Mode of failure

Studying the results of examining of bonding surface of zirconia, by using stereomicroscope at 40X magnification, table (7) highly support the result of this study. Studying table (7) shows that mode of failures when using sandblasting treatment was mostly cohesive failure and this

indicates that sandblasting the bonding surface of zirconia creates high bonding to resin cement.

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