

# Comparison of Shear Bond Strength of Three Different Brackets Bonded on Zirconium Surfaces (*In Vitro* Study)

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## ABSTRACT

**Background:** With the increased in the demands of adult orthodontics, the challenge of direct bonding to non-enamel surface (zirconium) had been increased. The present study was carried out to compare the shear bond strength of three different brackets (stainless steel, sapphire and composite) bonded to zirconium surface and study the mode of bond failure.

**Materials and methods:** The sample was comprised of 30 models (8mm \*6mm\*1.5mm) of full contour zirconium veneers. They were divided into three groups according to the brackets type; all samples were treated first by sandblast with aluminum oxide particle 50 µm then coated by z-prime plus primer. A central incisor bracket of each group was bonded to the prepared zirconium surface with light cure adhesive resin (Transbond TM XT, 3M Unitek, USA). Shear bond strength was measured by using Tinius Olsen universal testing machine at crosshead speed of 0.5\min. After debonding, each bracket and zirconium surface were examined using magnifying lens and adhesive remnant index was recorded. The difference in shear bond strength between main groups was analyzed by using ANOVA at  $p \leq 0.05$ .

**Results:** The results revealed high significant difference among all tested groups and the highest value was for sapphire brackets (7.49±1.45 Mpa) of all groups followed by stainless steel brackets (6.46±1.43Mpa) and composite brackets had the least value (4.35±0.72). Non-significant difference in the site of bond failure among all groups of brackets and zirconium-adhesive interface failure (score III) was the predominant.

**Conclusion:** the new zirconium prime plus primer can be successfully used in bonding stainless steel and sapphire brackets to zirconium surface.

**Keywords:** Zirconium, zirconium prime plus primer, shear strength. (J Bagh Coll Dentistry 2016; 28(3):142-148).

## INTRODUCTION

Zirconia is polycrystalline ceramic and it is silica free, acid-resistant material. It does not incorporate amorphous silica glass (such as, leucite-reinforced ceramics, Feld spathic porcelain, and lithium disilicate ceramics); therefore, the traditional surface treatments of ceramic such as hydrofluoric acid etching followed by silane application are ineffective <sup>(1,2)</sup>.

Zirconium materials have been used in clinical dentistry for many years with great success. Making adhesion to non-silica-based oxide ceramic materials like zirconia, metal and alumina was the challenge that limited their use <sup>(1,3)</sup>. There are dilemmas in bonding of zirconium; the well-known methods of mechanical and chemical bonding that used on glass-ceramics cannot be applicable for use with zirconia, due to important fact which is the absence of silica in the microstructure of zirconia and this ignores the viability of roughening the etching material which is an essential method for mechanical bonding as well as restricted the use of silanes for forming surfaces hydroxyls and developing the essential chemical bond <sup>(1)</sup>.

The approaches suggested improving bond strength to zirconium surfaces can be grouped into three broad categories, namely mechanical, chemical, or combination.

The purpose of mechanical alteration of the zirconium surface is to remove the glaze and roughen the surface to provide sufficient mechanical retention for the adhesive, allowing for the successful placement and retention of the orthodontic bracket. This alteration of the zirconium surface has been achieved by surface abrasion or roughening (grinding, air borne particle abrasion using Al<sub>2</sub>O<sub>3</sub> (50-110) µm, rotary abrasion by using diamond burs) creates adhesion only through micro-mechanical retention <sup>(4)</sup>.

The glaze of zirconium is translucent, low-fusing, porcelain which may be applied to the surface as the final stage in the firing cycle and has the effect of filling surface defects <sup>(5)</sup>. Mechanical adhesion alone is not enough for providing the optimal bond strength so; they promote the chemical adhesion in zirconia bonding. However, roughness of the surface is a key factor for adhesion to zirconia and the elimination of these particles abrasion for surface treatment could result in great reduction in bond strength <sup>(6,7)</sup>.

Chemical bonding to zirconium can be done by adhesive functional monomers, which are supposed to have the capability to form chemical hydrogen bonds with metal oxides at the resin/zirconia interface and improving the wettability <sup>(8)</sup>.

Phosphate monomers are proven to be effective in bonding to non-silica-based polycrystalline materials of zirconia, metal and

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alumina<sup>(9)</sup>. Numerous studies have shown that phosphate /phosphonate monomers are very effective in improving zirconia bonding. In theory, phosphate monomers form chemical bonds with the zirconia, alumina, and metal oxide surfaces<sup>(10)</sup>.

Z-PRIME PLUS is a phosphate monomer and it contains a propriety formula of concentrated methacryloxydecyl dihydrogen phosphate (MDP) and carboxylic monomers formulated specific to zirconia, alumina, and metal. The versatility of these primers is a compelling feature for use on many different indirect substrates<sup>(11)</sup>.

Recently, the use of zirconium in cosmetic dentistry was expanded obviously coincided with a new trend of adult orthodontics; therefore it is very important nowadays to find an accepted method and material of bonding orthodontic appliances effectively to zirconium surface with subsequent removal of these appliances without any damage to these restorations. There was no any previous study in Iraq regarding this important subject so; it is intended to implement the current study to provide a base line data regarding that.

## MATERIALS AND METHODS

### Sample

Thirty zirconium veneers of upper right central incisor of identical size & shape of the same company (Zolid, Amann Girrbach GmbH) would be made by using CAD/CAM machine (ceramill motion2). Each surface had been examined by using a 10X magnifying eye lens to see if there is any manufacturer defect including cracks, roughness or irregularities on the labial surface of the veneer<sup>(12)</sup>.

The zirconium veneers would be divided according to the bonded brackets into three groups:

- 1) Ten stainless steel brackets would be bonded on the labial surface of ten zirconium veneers (Orthotechnology, U.S.A), surface area 9.6 mm<sup>2</sup>.
- 2) Ten composite brackets would be bonded on the labial surface of ten zirconium veneers (Orthotechnology, U.S.A), surface area 21mm<sup>2</sup>.
- 3) Ten sapphire brackets would be bonded on the labial surface of ten zirconium veneers (Orthotechnology, U.S.A), surface area 12 mm<sup>2</sup>.

### Construction of zirconium veneers

Well prepared tooth of upper right central incisor had been done by professional dentist to form zirconium veneer for this tooth, after that the prepared tooth had been scanned by the CAD/CAM machine to design the veneer using

specific software by well-trained technician. The same model could be duplicated by definite software to form thirty veneers of identical size and shape. The dimensions of these veneers were 8mm in length, 6mm in width and 1.5 mm in depth.

Since these zirconium veneers were partially sintered, sintering at 1450°C for 4 hr. was necessary to achieve the required hardness. Then, the outer surface of each veneers would be covered with glaze and stain liquid (IPS e.max Ceram Glaze and Stain all round, Ivoclar Vivadent) and fired at 930°C.

### Construction of the Acrylic Blocks

A square of stainless steel tube would be cut into slices of identical cubes. The dimensions of these cubes would be 2.5cm in width and 1cm in depth, after that, each cube would be drilled in both sides in order to remove the excess of the acrylic that had been passed through the holes. Then cold cure acrylic would be mixed according to manufacture instruction and poured into each cube for 1 cm height.

Each zirconium veneer was placed in the middle of each acrylic cube. Then, glass slide was fitted against the veneer and pressed by the vertical arm of the surveyor by applying 100 gm. on the top of this arm. Finally, the excess of acrylic that had been passed through the holes could be removed by sharp scaler. All samples were hydrated in deionized distilled water at 37°C in incubator for 1-week before bonding to simulate the oral condition<sup>(13)</sup>.

The labial surface of all veneers were polished using a non-fluoridated pumice (for standardization one rubber cap used for each subgroup) attached to a low speed hand piece for 10 seconds<sup>(14,15)</sup>, then each surface was washed with water spray for 10 seconds, and dried with oil-free air for 10 seconds<sup>(16-18)</sup>. A distance of 1cm that used as standardization to hold the air water syringe away from veneer surface kept fixed throughout this study<sup>(19,20)</sup>.

### Bonding procedures

The zirconium surfaces would be sandblasted by 50µm Aluminum Oxide powder for 5 sec. at 10 mm distance with 2.5 bars<sup>(21)</sup>. The bonding could be done by applying a thin layer of primer on the outer surface of zirconium veneer and on the mesh of the brackets by using a disposable brush and wait for 10 sec. according to the manufacture instruction, and then suitable amount of light cure composite would be applied on the bracket base according to the manufacturer instructions, which would then position in the

middle third of the outer surface and parallel to the long axis of the veneer using a clamping tweezers. Then, a constant load would be applied by vertical arm of the surveyor by weight fixation of 200 gm. on the top of this arm, which would be placed on the bracket at 90 ° for 10 sec. to ensure that each bracket would seat under equal force (21,22).

Any excess bonding material could be carefully removed from around the bracket base with a sharp hand scaler without disturbing the seated bracket (23-25), then the brackets would be cured for 40 sec (20 sec on the mesial and 20 sec. on the distal of the brackets (26) by using LED Light cure (High intensity 1500mW/cm<sup>2</sup>, 6mm depth of cure, SDI, China), at a distance of 5 mm (27) (for standardization we fixed a ruler at the tip of the light probe) and an angle of 45° to the proximal surface of the bracket (28).

After the completion of the bonding procedure, the specimens would be allowed to bench cure for 30 minutes, then would be immersed in deionized distilled water and could be stored in the incubator at 37° C for 24 hours (29).

### Shear Bond Strength Test

Shear test was accomplished using Tinius Olsen universal testing machine, with loading cell 50 kilogram & a crosshead speed of 0.5 mm/min (25, 30). Each sample was seated in the mounting metal vice and placed on the base of the testing machine (which was parallel with the horizontal plane). The chisel end rod was fitted inside the upper arm of the testing machine with its chisel end downward parallel to the bonded zirconium labial surface to apply a force in an gingivo-incisal direction of the bracket that produce a shear force at the bracket base/ zirconium surface interface, until debonding occurs.

When the bracket was debonded from the zirconium labial surface by the force applied from the testing machine, the ultimate magnitude of the reading was taken; this force was measured in kilograms and converted into Newtons according to the following equation: Force (N) = Load (kg) X Ground acceleration (9.8 m/sec.). Then the force was divided by bracket base surface area to get the strength value in Mega Pascal (MPa) units. Each debonded bracket was kept with its corresponding zirconium veneer to estimate the adhesive remnant index.

### Estimation of the Adhesive Remnant Index

The debonded bracket and zirconium surface of each tooth were inspected using a 10X

magnifying lens to determine the predominant site of bond failure (24,31).

The site of bond failure is scored according to Wang et al. Index (32) that had been modified to the zirconium surfaces, as follow:

Score I: Failure between the bracket base and adhesive.

Score II: Cohesive failure within the adhesive itself, with some of the adhesive remained on the zirconium surface and some remained on the bracket base.

Score III: Failure between adhesive and zirconium surface.

Score IV: Zirconium detachment.

### Statistical Analyses

Data were collected and analyzed using SPSS (statistical package of social science) software version 15 for windows XP Chicago, USA. In this study the following statistics were used:

A. Descriptive statistics: including mean, standard deviation, minimum, maximum, percentage, frequency and statistical tables.

B. Inferential statistics: including;

1. One way analysis of variance (ANOVA):

To test any statistically significant difference among the tested groups.

2. Least significant difference (LSD): To test any statistically significant differences between each 2 groups when ANOVA showed a statistical significant difference.

3. Chi-Square: To test the non-parametric data for Adhesive remnant index.

\*P level of 0.05 was accepted as statistically significant at the following levels:

p > 0.05 **NS** Non significant

0.05 > p > 0.01 **S** Significant

p < 0.01 **HS** Highly significant

## RESULTS

The descriptive statistics (means, standard deviations, minimum and maximum values) of the shear bond strength of each group were presented in Table (1).

It was clearly obvious that sapphire brackets group had the highest mean value of shear bond strength (7.49±1.45 Mpa) of all groups followed by stainless steel brackets group (6.46±1.43Mpa) while composite brackets group had the least value (4.35±0.72 Mpa).

ANOVA showed that there was statistically highly significant difference (P ≤ 0.01) among the mean values of the shear bond strength of the three types of brackets. LSD test showed that, there was non-significant difference between stainless steel and sapphire brackets groups (P-value > 0.05) but there was highly significant

difference between stainless steel and composite brackets and between sapphire and composite brackets groups ( $P \leq 0.01$ ).

**Table 1: Descriptive statistics of the shear bond strength (MPa) in different groups.**

Groups	N	Mean	S.D.	Min.	Max.
SS	10	6.46	1.43	4.9	9
Sapphire	10	7.49	1.45	5.83	10.80
Composite	10	4.35	0.72	3	5.4

**Adhesive remnant index “ARI”**

The sites of bond failure of all tested groups were shown in Table (2). The highest percentage of bond failure was seen at zirconium-surface interface (score III) and associated with the use of both sapphire (90%) and stainless steel (90%) bracket groups while the composite bracket group was the least (60%). Regarding (score I) and (score IV) there were no any value registered among all three groups.

However for the cohesive failure (score II) it was less in sapphire (10%) and stainless steel (10%) bracket groups than composite bracket group (40%). Statistically, Chi-square test showed non-significant difference in the site of bond failure among all groups of brackets. Yate's correction test was used to compare the site of bond failure between each groups and showed non-significant difference.

**Table2: Frequency and distribution of the ARI scores in different groups.**

Groups		ARI Scores				Total
		I	II	III	IV	
Stainless steel	No	0	1	9	0	10
	%	0	10	90	0	100
Sapphire	No	0	1	9	0	10
	%	0	10	90	0	100
Composite	No	0	4	6	0	10
	%	0	40	60	0	100
Total	No	0	6	24	0	30
	%	0	20	80	0	100

( $X^2=3.750$ , d.f. =2, p-value=0.169 NS).

**DISCUSSION**

With the exception of composite bracket, sapphire and stainless steel brackets had shear bond strength exceed or within the normal limits that suggested by Reynolds<sup>(33)</sup> which is (6-8) MPa; to be able to withstand masticatory and orthodontic forces in different clinical conditions. The highest shear bond strength value was demonstrated in sapphire brackets on zirconium surfaces and this could be attributed to:

(1) The presence of zirconia particles coating the bracket base that creates millions of undercuts<sup>(34)</sup>, those secure the bracket in place, due to the micro mechanical retention means, and this revealed difference in shear bond strength when compared to other bracket types. In more practical words, it was greater by 1.16 times than stainless steel brackets and 1.72 times than composite brackets.

(2)The translucency of sapphire brackets gave them a better chance for a more complete polymerization with light curing as compared to other bracket types. This gives the operator more confidence to use sapphire brackets keeping in mind that it has a lower possibility of failure as compared to other bracket types<sup>(35-37)</sup>.

(3) Sapphire brackets are single-crystalline brackets so, they are hard and offer great strength that prevents or reduces the peeling effects that may occur during brackets debonding thus gave them high SBS values<sup>(37)</sup>.

(4)The fine mesh of sapphire brackets provides a good mechanical interlock into which the resin adhesive with low viscosity can penetrate and engage the retentive mesh and fill the undercuts with good air evacuation and without air entrapment<sup>(38)</sup>. Stainless-steel brackets arranged secondly regarding SBS value on zirconium surface and had good mean of shear bond strength 6.46 MPa and this could be due to:

(1)The type of retention means on the base of the bracket which equipped with (80 Gauge Foil Mesh Bonding Base) so, the composite resin adhesive can penetrate easily between these projections and fill the undercuts and provide a mechanical interlock and prevent air entrapment as the air can escape easily from the peripheries of the base of the bracket and there would be a good retention of the adhesive into the bracket base<sup>(38,39)</sup>.

(2)The compound contour of bracket base provide superior fit and greater contact surface area for improved retention which provide a good seating and adaptation to the surface of the tooth which result in a thin layer of adhesive between the bonded bracket base and the tooth surface and this could increase the bond strength<sup>(40)</sup>.

However, There was no significant difference in shear bond strength between stainless steel and sapphire brackets in LSD test but there was little difference in mean value of bond strength between them which could be due to the dimness color of steel brackets as compared with translucency of sapphire brackets and thus would affect the intensity of light and the polymerization of the adhesive<sup>(35, 36)</sup>. On the other hand composite brackets had the lowest shear bond

strength. Its shear bond strength had a significant difference when compared to stainless steel and sapphire brackets and this could be attributed to:

(1)The difference in surface area of the brackets (composite bracket 21 mm<sup>2</sup>, sapphire bracket 12 mm<sup>2</sup>, stainless steel brackets 9.6 mm<sup>2</sup>) though, enlarging the surface area of brackets increase the load carrying capacity, that means there is an inverse relationship between bond strength and bonded surface area, the smaller the surface area, the greater the bond strength<sup>(41)</sup>.

(2)The larger the size of the examined specimen (composite bracket) leading to presence of a greater number of defects and vice versa, therefore when the specimen is loaded, stress concentration will be expected at the defects and initiates crack formation<sup>(42)</sup>.

(3)Bracket base morphology could influence the bond strength of the bracket resin interface by determining the geometry (depth, size, and distribution) of the resin tags and stress distribution within the cement bracket interface. Moreover, the penetration of the light and polymerization of light activated materials could be influenced by base morphology. Mechanically retained composite bracket that has base design characterized by undercut channels open horizontally at the medial and distal extremities on the surface of the base<sup>(34)</sup>. Therefore, this result could be due to different base design which lead to different mechanical interlock between the adhesive and bracket base and could influence the light penetration and polymerization<sup>(43)</sup>.

(4)The lowest shear bond strength of composite brackets may be due to the fact that the retentive groove bracket base will form an edge angle of 90 degree this leading to high localized stress concentration area around the sharp edge, and this may lead to brittle failure of the adhesive<sup>(44)</sup>.

Concerning the adhesive remnant index scores which gave the indication about the type of bond failure for each group, it appeared that, there was no significant difference in ARI among all groups. The occurrence of ARI score (III) which indicate failure at adhesive-zirconium interface were the predominant and represented 80% (24 specimens) of all tested samples, and the highest percentage occurred both in stainless steel and sapphire brackets groups (90%) while the least percentage occurred in composite bracket (60%) and this might be due to:

(1)The bond failure occurs usually at the area of least resistance which means that the bond strength between the adhesive-bracket interface and the cohesive bond strength of the adhesive itself were stronger than the bond strength

between the adhesive and zirconium. This could be attributed to the hardness glossy surface of zirconia, so the mechanical retention might not be sufficient enough.

(2)Air abrasion of zirconia, with alumina or other particles produces lower bond strength compared to other surfaces like enamel and porcelain therefore surface scratching by these particles might be not sufficient enough to produce optimal mechanical retention between the adhesive and zirconium surface<sup>(45)</sup>.

(3)Adhesive failure at the zirconium surface might be the result of reduced depth of adhesive penetration because the resin tags were thin, and less uniform, which was conducive to weaker bond, hence less adhesive would remain on the tooth at the time of debonding. Furthermore, bracket failure typically occurs at the weakest link in the adhesive junction and the weakest link appeared to be at the surface/adhesive interface<sup>(46)</sup>. The ARI score (II) indicate cohesive failure within the adhesive itself, with some of the adhesive remained on the zirconium surface and some remained on the bracket base and occurred in (20%) (6 specimens) of all tested samples collectively. On other hand the occurrence of score (II) in a low percentage specially in sapphire and stainless steel samples (10% for each) and this could be negligible while in composite bracket score (II) occurred in a higher percentage (40%) and this could be due to the presence of three dove tail in the bracket base that may act as a stress concentration area since, the adhesive penetration in these grooves produced weaker link than that between bracket/adhesive interface or than that between surface/adhesive interface. None of the tested samples showed score (I) which indicates failure that usually occurred between brackets and adhesive, this might be due to high mechanical interlock provided with each bracket base without any weak point between bracket-adhesive links. The sapphire bonding base is coated with powder of zirconium that create millions of undercuts which mechanically lock with the bracket adhesive, while stainless steel bracket is equipped with (80 Gauge Foil Mesh Bonding Base) and composite bracket has three dove tail grooves<sup>(34)</sup>.

Also none of the tested samples showed score (IV) which usually indicates surface detachment, this may attributed to excellent strength of the zirconia surface which could reach to (1000) MPa in addition to that, most of the values of the shear bond strength were within or below the normal range (6-8) MPa of safe debonding as suggested by Reynolds<sup>(33)</sup>.

The conclusions that could be drawn from this study were:

1. Bonding with z-prime plus primer provides optimum value of shear bond strength for sapphire and stainless steel brackets, while regarding the composite bracket the shear bond strength was insufficient.

2. Adhesive-zirconium interface failure (score III) was the predominant mode of bond failure in all groups which is considered as the most preferable one and none of the samples showed detachment between the composite and the bracket(score I) or fractures within the zirconium itself during debonding (score IV).

3. The site of bond failure is influenced not only by the value of the shear bond strength, but also by the design of the retention means on the attachment base of the bracket.

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