Comparison of Shear Bond Strength of Sapphire Bracket Bonded to Zirconium Surface after Using Different Surface Conditioning Methods (*In Vitro* Study)

Hawraa Ihsan, B.D.S. [(a)]
Dhiaa Jaafar Al-Dabagh, B.D.S, M.Sc. [(b)]

ABSTRACT

Background: The present study was carried out to compare shear bond strength of sapphire bracket bonded to zirconium surface after using different methods of surface conditioning and assessment of the adhesive remnant index.

Materials and methods: The sample composed of 40 zirconium specimens divided into four groups; the first group was the control, the second group was conditioned by sandblast with aluminum oxide particle 50 μm, the third and fourth group was treated by (Nd: YAG) laser (1064nm)(0.888 Watt for 5 seconds) for the 1st laser group and (0.444 Watt for 10 seconds) for the 2nd laser group. All samples were coated by z-prime plus primer. A central incisor sapphire bracket was bonded to all samples with light cure adhesive resin. Shear bond strength was measured by using Tinius Olsen universal testing machine. After debonding, each bracket and zirconium surface were examined and adhesive remnant index was registered. The difference in shear bond strength among groups was analyzed by using ANOVA test. The adhesive remnant index was assessed using Chi-square test.

Results: The 2nd laser group had the highest mean value of shear bond strength then the 1st laser group followed by the sandblasting group, while the control group had the least value, non-significant difference in the shear bond strength was found between the laser groups and highly significant difference was found between all other comparable groups. Non-significant difference in the site of bond failure was found between the laser groups and sandblasting group, and between the two laser groups.

Conclusion: The laser conditioning method showed higher value of shear bond strength than the sandblasting conditioning method.

Keywords: Zirconium, zirconium prime plus primer, laser, shear strength.**(J Bagh Coll Dentistry 2017; 29(3):86-92)**

INTRODUCTION

At present, the number of adults seeking orthodontic treatment is increasing. Many of them present to orthodontic clinics with restorations such as crowns and bridges in their mouth, made of yttrium-stabilized tetragonal zirconia (Y-TZP) ceramics or in short, zirconium crowns. These crowns are widely used and favored for their advantages including biocompatibility, aesthetics, cost effectiveness, fracture resistance, and accurate fabrication. Zirconia crowns are used to restore posterior teeth and occasionally anterior teeth when the focus is more on strength rather than aesthetics [(1)].

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 [(2)].

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 [(3,4)].

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 [(5)]. Phosphate monomers are proven to be effective in bonding to non-silica based polycrystalline materials of zirconia, metal and alumina [(6)]. Numerous research studies have shown that phosphate/phosphonate monomers are very effective in improving zirconia bonding [(7)].

Z-PRIME PLUS is a phosphate monomer and it contains a propriety formula of concentrated methacryl oxydecyl 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 [(8)].

There was no any known previous study that conducted to compare between shear bond strength using laser and sandblasting method of sapphire bracket bonded on zirconium surface, so

[(a)] Master Student. Department of Orthodontics, College of Dentistry, University of Baghdad.
[(b)] Professor. Department of Orthodontics, College of Dentistry, University of Baghdad.

it is intended to implement this current study to provide baseline data regarding this important subject.

MATERIALS AND METHODS

Sample

Two blocks of zirconium was cut by one dental technician in order to obtain forty cylindrical specimens with a diameter of 8mm and a height of 6mm. The samples were cured in a special oven according to manufacture instruction, after that the samples were painted with glaze layer and fired at 940ºC for 15 minutes.

Each surface had been examined by using a 10X magnifying eye lens to see if there is any manufacture defect including cracks, roughness or irregularities on the labial surface of the veneer [9].

Construction of the acrylic blocks

Silicone mold with a cubic hole (15mm) in dimensions and a circular hole in the bottom of the cube (8mm) in diameter was used so that the glazed surface of the specimen was inserted inside the hole in order that no acrylic material had came in contact with the surface.

Each zirconium specimen was placed in the acrylic mold, after that the acrylic was mixed according to manufacture instructions and poured into each mold, then the acrylic block with the zirconium specimen was taken out of the mold.

Sample grouping

The specimens would be divided according to the surface conditioning into four groups:

1- Group (A) control group (with blue acrylic block)
2- Group (B) sandblasting group (with brown acrylic block)
3- Group (C) and (D) laser groups (with pink and green acrylic blocks respectively)

All samples were ultrasonically cleaned with distilled water for 6 minutes [10] to remove the factors that inhibit adhesion and dried naturally in the atmosphere.

Surface conditioning

A-Sandblasting group

By using Twin-Pen sandblaster machine, the zirconium surfaces would be sandblasted by 50µm Aluminum Oxide powder for 5 sec. at 10mm distance with 2.5 bars [11]. For standardization, a ruler was fixed at the tip of the sanblaster pen).

B-Laser groups

A drill stand had been used so that the hand piece of Nd:YAG laser device was placed on the upper part of the stand and the zirconium samples were placed on a cube table on the base of the stand. The laser groups were treated with Nd:YAG laser (1064 nm). Energy density of 141.54 J/cm^2 was delivered through laser handpiece and kept 1mm from the specimen [12].

According to the calculation, the energy density was set at 4.44J for both groups. The spot size for the laser device is 2mm in diameter so the radius is 1mm which equivalent to 0.1 cm

The surface area = $(0.1)^2 \times 3.14 = 0.0314$ cm

The energy density = ENERGY/ AREA

141.54J/cm^2= Energy / 0.0314 cm^2

E=4.44J The output energy of the laser device for the first group was 888mJ for an irradiation time of 5 seconds result in an accumulated energy of 4.44J

Power=Energy/Time Power=4.44J/5 second= 0.888 Watt. The output energy of the laser device for the second group was 444mJ for an irradiation time of 10 seconds result in an accumulated energy of 4.44J Power=4.44J/10 second = 0.444 Watt

The time was selected according to a pilot study by fabricating a zirconium disks (1mm) in thickness because the Optimal labial and lingual reduction will range from 1.0-1.5 mm for teeth preparation of the crown of the anterior teeth [13,14].

The disk was putted on around table and the end of thermo couple device was in direct contact with the disk, when the output setting of (444mJ) was applied on the disk, the thermo couple readings recorded 1.2 degree temperature elevation after 5 seconds and 3.3 degree temperature elevation after 10 seconds.

When the out put of (888mJ) was applied on the disk, the thermo couple device readings recorded 3.7 temperature elevations after 5 seconds. (5.5) temperature elevation considered as a critical temperature elevation for the pulp [15]. For this reason (5-10) seconds were selected as a safety exposure time for the two laser groups.

Bonding procedures

The bonding was done by applying a thin layer of primer on the outer surface of zirconium sample and on the mesh of the brackets by using a disposable brush (as standardization one rubber cup used for each specimen with single stroke in gingival incisal direction) and wait for 10 seconds according to the manufacture instruction, and then an equal amount of light cure composite would be applied on the bracket base according to the manufacturer instructions, which would then position in the center of the circle surface of the zirconium specimen using a bracket holder.

Then, a constant load would be applied by vertical arm of the surveyor by weight fixation of 200gm. on the top of this arm, which would be placed on the bracket at 90° for 10sec., to ensure that each bracket would seat under equal force [9,16].

Any excess bonding material was carefully removed from around the bracket base with a sharp hand scaler without disturbing the seated bracket [17-19] then, the brackets cured for 40sec. (20sec. on the mesial and 20sec. on the distal of the brackets) [20] at a distance of 5mm [21] (for standardization, a ruler was fixed at the tip of the light probe) and an angle of 45° to the proximal surface of the bracket [22].

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 37ºC for 24 hours to stimulate the oral condition [23].

Shear Bond Strength Test

Shear test was accomplished using Tinius Olsen universal testing machine, with loading cell 50 kilogram and a crosshead speed of 0.5mm/min [19,24]. Each sample was seated in the mounting metal vice and placed on the base of the testing machine.

The chisel end rod was fitted inside the upper arm of the testing machine with its chisel end downward parallel to the zirconium outer 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 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 Newton according to the following equation:

Force (N) =Load (kg) X Ground acceleration (9.8m/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 [18,25]. The site of bond failure is scored as follow [26]:

Score I: 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: 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:

1. Descriptive statistics: including mean, standard deviation, minimum, maximum, percentage, frequency and statistical tables.
2. Inferential statistics: including:
   A. One way analysis of variance (ANOVA): To test any statistically significant difference among the tested groups.
   B. The Tukey HSD test was performed to compare the difference between each two tested groups when ANOVA showed a statistical significant difference.
   C. Chi-Square: To test the non-parametric data for Adhesive remnant index.

P level was set 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). The shear bond strength values of all tested samples were expressed in Mega Pascal (MPa).

Regarding the methods of surface conditioning, the 2nd laser group had the highest mean value of shear bond strength (30.67±2.33Mpa) of all groups followed by 1st laser group (30.25±2.31Mpa) followed by the sandblasting group (22.29 ± 1.18Mpa) while the control group had the least value (11.08±1.96Mpa).

One way analysis of variance (ANOVA) showed that, there was statistically highly significant difference (P≤0.01) among the mean values of the shear bond strength of all tested groups

The Tukey HSD test was performed to compare the difference between each two tested groups (Table 2). It showed the followings:

A- Highly significant difference (P≤0.01) between control and all other comparable groups.

B- Highly significant difference (P≤0.01) between sandblasting and all other comparable groups.

C- Highly significant difference (P≤0.01) between 1st and 2nd laser group respectively with control and sandblasting group.
D- Non-significant difference (P-value>0.05) was found between 1st and 2nd laser groups

Table 1: Descriptive statistics of the shear bond strength (MPa)

Groups	N	Mean	S.D.	Min.	Max.
Control	10	11.08	1.96	8.75	14.58
Sandblasting	10	22.29	1.18	20.41	23.75
1st laser	10	30.25	2.31	27.08	34.58
2nd laser	10	30.67	2.33	27.5	35

Table 2: Comparing the shear bond strength between each two groups using Tukey HSD test

Groups		Mean Difference	p-value
Control	Sandblasting	-11.21	0.000 (HS)
	1st laser	-19.17	0.000 (HS)
	2nd laser	-19.59	0.000 (HS)
Sand blasting	1st laser	-7.96	0.000 (HS)
	2nd laser	-8.38	0.000 (HS)
1st laser	2nd laser	-0.42	0.966 (NS)

Adhesive remnant index "ARI"

The sites of bond failure of all tested groups were shown in Table (3).

The highest percentage of bond failure was seen at zirconium-surface interface (score III) in control group while the cohesive failure (score II) was seen in laser groups in high percentage (90%) with only (10%) score III.

In sandblasting group (score III) was seen in low percentage (20%) while (score II) was seen in high percentage (80%) in this group. Regarding (score I) and (score IV) there were no any value registered among all three groups.

Statistically, Chi-square test showed highly significant difference in the site of bond failure among all tested groups.

Yate's correction test was used to compare the site of bond failure between each two groups and showed highly significant difference between the control group and the other groups while there was no significant difference among the groups other than control.

DISCUSSION

The effects of four surface preparation methods on the SBS values of sapphire brackets to zirconia surfaces were compared. The results of this study revealed that laser conditioning groups specimens

Table 3: Frequency distribution of the ARI scores in different groups

Groups		Scores I	II	III	IV	Total
Control	N	0	0	10	0	10
	%	0	0	100	0	100
Sandblasting	N	0	8	2	0	10
	%	0	80	20	0	100
1st laser	N	0	9	1	0	10
	%	0	90	10	0	100
2nd laser	N	0	9	1	0	10
	%	0	90	10	0	100
Total	N	0	26	14	0	40
	%	0	65	35	0	100

possessed the highest SBS followed by sandblasting group, while the control group possessed the least SBS.

Comparison between air abrasion and laser as a conditioning method to zirconium surface pointed out several advantages and disadvantages as following:

Regarding the advantages of both mentioned methods, it is well known that both of them lead to surface roughness which increases the surface area, improves the wettability by diminishing the surface tension, and produces micromechanical retention. However, the present study found that laser conditioning increases SBS more than sandblasting.

While regarding the disadvantages of both methods, the following were found:

In air abrasion method, sandblasting and mechanical abrasion are capable to create micro cracks in zirconia to supply retention; even though these methods also weakens the mechanical properties of zirconia [27,28], this was overcome in the current study by reducing the pressure during air abrasion and using particles up to 50 µm in size coincided with Piascik [29].

While in laser conditioning method, it was found that heat generation by laser irradiation may cause:

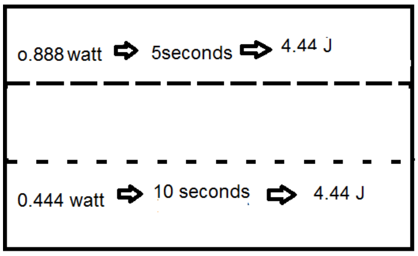
1- Wide-melting areas and big cracks lead to defects and decrease the mechanical properties of zirconia ceramics especially if the used power setting ranged from 3-4 watt [12].
2- Critical temperature elevation for the pulp.

These two mentioned disadvantages were overcome in current study by using power setting (0.888, 0.444) watt.

It is important to mention that power setting of laser irradiation is affected by time and energy according to the equation: Power=Energy/Time. We select the exposure time according to a pilot study to get precise power with least harmful effect on zirconium surface and consequently the tooth health and (5,10) seconds were selected according to this study as a safety exposure time for the two laser groups.

In the current study Nd:YAG laser with different parameters as irradiation time (in seconds) and power (in watt) was used to study the influence of the variation on zirconium surface because Various laser parameters are known to influence ceramic materials differently [30].

From clinical point of view changing the irradiation time and the power can be represented by following diagram:



The variation in the manner of radiation logically affected both the chair time and the risk of laser on tooth health and structure, and there is a difference in the thermal relaxation time. However, statistically no significant difference was found between the two manners and this may be due to the same total accumulated energy in the two groups.

Failure site

Regarding the occurrence of ARI score (II) which indicated cohesive failure within the adhesive itself, it was the predominant one and represented 65% (26 specimens) of all tested samples, and the highest percentage happened both in the laser groups, it was (90%), and it was (80%) in the sandblasting group, while there was no occurrence of ARI score (II) in control group.

Regarding the occurrence of score (III) which indicate failure at adhesive zirconium interface, it represented 35% of all tested samples, more specifically (100%) (10 specimens) in control group, and it was in a low percentage (10%) and (20%) in laser groups and sandblasting group respectively and this could be negligible.

Control group

The occurrence of ARI score (III) represented (100%) in this group and this might be due to:

1- The bond failure occurs generally at the area of least resistance that 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 might be attributed to the hardness glossy surface of zirconia, so the mechanical retention might not be adequate enough.

2-Adhesive failure at the zirconium surface might be the result of reduced depth of adhesive penetration since the resin tags were thin, and less uniform, which was conductive to weaker bond, hence less adhesive would stay on the tooth at the time of de-bonding, in addition, bracket failure typically occurs at the weakest link in the adhesive junction and the weakest link appeared to be at the surface/adhesive interface, agreed with the finding of [31,32].

Sandblasting group and laser groups

The occurrence of ARI score (II) were the predominant in these groups and represented (80%) and (90%) in sandblasting and laser groups respectively and this might be due to:

1- The bond failure happened usually at the area of least resistance which means that the bond strength between the adhesive and zirconium were stronger than the bond strength between the adhesive–bracket interface and the cohesive bond strength of the adhesive itself. This might be attributed to occurrence of sufficient mechanical retention by air abrasion and laser.

2- Aluminum oxide (Al_2O_3) sandblasting has the potential for enhancing surface energy, surface area, and wettability for the proper adhesive procedure [33]. However this result disagreed with Obradović-Djuričić [34] who stated that air abrasion of zirconia, with alumina or other particles produces surface scratching that might be not adequate enough to produce optimal mechanical retention between the adhesive and zirconium surface.

3- The increased bond strength observed in the laser-treated group is related to micromechanical retention that gained by laser conditioning [35], the laser irradiation on zirconia bonding surface considerably increase shear bond strength because of surface roughness [36].

Smith [37] stated that surface-adhesive interface failure score (III) is desirable, since the problem of residual adhesive is not encountered, while Saraç and Harari [11,38] stated that, cohesive failure within the adhesive itself (score II) is preferable to avoid surface damage throughout debodning which clinically leading to the long-term integrity of the restorations, however this type of residual adhesive may need further treatment to remove it from the zirconium surface, a process that might cause additional damage to zirconium restoration surface and this coincided with the finding of [39].

None of the tested samples showed score (1) that indicates that usually failure happened between brackets and adhesive, this might be owing to high mechanical interlock provided with every bracket base without any weak point between bracket adhesive links, the sapphire bonding base is coated with powder of zirconium that produce millions of undercuts which mechanically lock with the bracket adhesive [40].

Also none of the tested samples showed score (IV) which usually indicates surface detachment, this may attributed to exceptional strength of the zirconia surface which might reach to (1000Mpa) in addition to that, the values of the shear bond strength were within (8.75- 35 Mpa).

A direct relation between bond strength and ARI scores was found in the current study, suggesting that greater bond strength was associated with lower ARI scores.

Knox and Wang [41,42] reported that there is no relation between the value of the SBS and the site of bond failure and this seems to be because of different types of bonding materials used, different types of brackets base designs; or due to different testing methods applied, while Coups-smith and Klocke [31,43] stated that, there is a relation between the SBS value and ARI and when the value of the SBS increase the site of failure will move toward the surface of the tooth.

The conclusions that could be drawn from this study were:

1. Zirconium surface conditioning with air abrasion and laser provides good value of shear bond strength for sapphire brackets; however irradiation with the laser was better than air abrasion.

2. No significant statistical difference was found regarding the values of shear bond strength when two different ways for laser application (high power for a short time and low power for a long time) were used.

3. The occurrence of ARI score (II) which indicate cohesive failure within the adhesive itself were the predominant mode of bond failure in surface conditioning groups which is considered as the most preferable to avoid surface damage during debonding which clinically leading to the long-term integrity of the restorations, and none of the samples showed detachment between the bracket base and adhesive (score I) or fractures within the zirconium itself during debonding (score IV).

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الخلاصة

تم اجراء الدراسة الحالية لمقارنة قوة القص للحاصرات التقويمية من نوع الياقوت الملصقة على سطح الزاركون بعد استعمال طرق مختلفة لتكييف السطح وتقدير مشعر الالتصاق المتبقي .

تكونت العينات من اربعين نموذج اسطواني من الزاركون وتم تقسيمها الى اربعة مجاميع , المجموعة الاولى هي مجموعة التحكم , والمجموعة الثانية عولجت برمل اوكسيد الالمنيوم (50 مايكروميتر), والمجموعتين الثالثة والرابعة عولجت بالليزر من نوع نوديميوم ياك (1064نانومتر) (0.888 واط لمدة 5 ثواني) لمجموعة الليزر الاولى و(0.444 واط لمدة 10 ثواني) لمجموعة الليزر الثانية,ومن ثم طليت كل العينات بمادة (primer Z prime plus) ثم اضيفت حاصرة تقويمية للسن القاطع من نوع الياقوت لكل العينات ولصقت بمادة الارتباط الضوئية التصلب .

تم قياس قوةالقص باستخدام الة الفحص العالمية (Tinius-Olsen) وبعد فك الارتباط فحصت قاعدة الحاصرة وسطح الزاركون باستخدام عدسة مكبرة (10x) وتم تسجيل مشعرالالتصاق المتبقي .تم تحليل الفرق في القوة اللاصقة للارتباط بين المجاميع باستعمال تحليل ANOVA.

اظهرت النتائج ان مجموعة الليزر الثانية اظهرت اعلى قيمة لقوة القص تليها مجموعة الليزر الاولى ومن ثم المجموعة المعالجة برمل اوكسيد الالمنيوم ,بينما مجموعة السيطرة اظهرت اقل قيمة, لم تكن هناك فروق ذات دلالة عالية بالنسبة لقوة القص بين مجموعتي الليزرو وكان هناك فروق ذات دلالة عالية بين كل المجاميع الاخرى المقارنة, لاتوجد اختلافات مؤثرة في موضع الفشل بين مجموعتي الليزر والمجموعة المعالجة وبين مجموعتي الليزر فيما بينهم.

من ذلك نستنتج ان طريقة المعالجة بالليزر اظهرت قيمة افضل لقوة القص من طريقة المعالجة برمل اوكسيد الالمنيوم.