



## The Effect of Surface Treatment on Hardness and Shear Bond Strength between Ceramic and 3-Yttrium Zirconia

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### Abstract

**Background:** The veneer chipping from zircon core is one of the elements driving ongoing research. Two types of 3Y zirconia is mostly used as a core in both anterior and posterior region according to their indications, covered by veneering ceramics and need strong bond force.

**Aim:** To reveal effects of the surface treatments on the shear bond strength and hardness of 3Y zirconia to veneering porcelain.

**Materials and methods :** 80 zirconia samples were used in this study, 40 samples of (Dentaldirekt DD Z) 3Y high-alumina zirconium core, another 40 samples were made of (Dentaldirekt DD ZX) 3Y low-alumina. Each type were subdivided into two groups: 20 micro-rods (5\*25\*1.5 mm) in size were created for hardness test, 5 samples for each type of surface treatments (5 samples laser, 5 samples acid etching, 5 samples for sandblasting and 5 samples as a control group). The other subgroup zirconia specimens (diameter of 7 mm\ height: 3 mm) for shear bond strength test, 5 for each type of surface treatments. 6W Er;Cr:YSGG as a laser surface treatment was used and 9.5 % Hydrofluric acid was used as acid etching. 120 µm alumina used for sandblasting.

**Results:** Hardness test was showed no statistical difference between surface treatments groups and control group. While shear bond strength test was showed that laser and acid etching a rise in shear bond strength.

**Conclusion:** Surface treatments did not affect the Hardness while laser and acid etching improve zirconia shear bond strength to veneering ceramic.

## Introduction:

Since the 1980s, all ceramic dental restorations have been on the market. Because of its superior mechanical qualities, yttrium-stabilized tetragonal ZrO<sub>2</sub> (Y-TZP) has grown in popularity in dentistry. Y-TZP, on the other hand, has a low transparency. As a result, a matching porcelain veneer is still necessary to provide a more pleasing visual outcome, this is referred to as multiple layers prosthesis. However, the problem of veneering chipping stay in multiple layers restorations <sup>(1)</sup>. The Chemical bonding with mechanical stability, wetting the behavior, coefficient of expansion thermal, and variances in temperature of glass transition all influence the binding with zirconia and porcelain veneers. According to Kwon et al., the bonding between zirconia and ceramic veneers happens mechanically more than chemically, and rough patches generated on the zirconia surfaces are one of most critical bonding elements. There are other method for increasing roughness of the surface and creating micromechanical confinement, roughening using aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) particles is the most commonly utilized. Porcelain veneer bonding can be increased by increasing surface roughness and establishing lower zones, as well as cleaning the zirconia surface or boosting and wettability and surface energy <sup>(2)</sup>. Lasers are recently developed approach for increasing surface roughness. Lasers have discovered to be a safe and effective method of roughening surface of materials. Er,Cr:YSGG is a powerful laser systems. Water and hydroxyapatite crystals in tooth tissue can effectively absorb this type of laser. Evaporation results from photon energy absorption, which creates huge and microscopic imperfections on the material's surface via microbursts <sup>(3)</sup>. The major effect of the laser irradiation in zirconia may include the melting and re-hardening of surface materials. Surface roughness ratings will fluctuate when the surface topography of zirconia varies <sup>(4)</sup>. It is critical to create an efficient and micromechanical ceramic surface for successful adhesive bonding and/or repair of ceramic restorations. The

most essential adhesion mechanism in ceramic systems is produced by penetration and polymerization at this retention surface. Several approaches for producing a comparable micromechanical cohesive ceramic surface have been developed and used clinically. The most common is hydrofluoric acid etching (HF) and sandblasting <sup>(5)</sup>.

## Materials and Methods

In our study's sections 3Y-HA refer to 3yttrium high alumina content TZP ( DD Z ) , While 3Y-LA refer to 3 yttrium low alumina content TZP (DD ZX).

### 1.Experimental Design:

DD bio ZX<sup>2</sup> (3Y-TZP-LA) and DD bio Z (3Y-TZP-HA) are the two primary zirconia kinds that are used as samples. These are mostly advised for zirconia core.

Eighteen samples were split up based on the surface treatments and the tests that needed to be performed.

For each test (shear bond strength and hardness), fourteen samples of each type of zirconia are divided into twenty samples.

Additionally, the 20 samples are separated into 5 control, 5 laser, 5 acid etching, and 5 sandblasting groups based on the surface treatment that will be applied.

### Procedure:

#### A- Samples Preparation:

1- Five samples of micro-rods measuring (5 \* 25 \* 1.5 ) mm were made for each kind of surface treatment. (ISO specification 6872-2015).

2- Zircon circular samples (diameter: 7 mm; height: 3 mm) were made using unsintered blocks in order to assess the shearing bond strength.5 with the kinds of surface modifications.

Following surface treatments, zirconia samples were covered with a ceramic layer that measured 3mm in diameter and 1 mm in thickness<sup>(3)</sup>.

After designing the sample with EXO CAD software (DentalCAD 3.1 Rijeka version 2015), the designs are transmitted to a CAD/CAM machine for milling. A dry grinding mill was used to grind all

samples. CAD/CAM (CORiTEC 350i pro) as shown in figure (1).

Following the completion of the milling, all of the rollers that contain the sample are cut with a zircon disc and a carbide bur (IMS 303 UL & IMS 302 UL (1- 0.6 mm) cutting burs (one bur used for each zirconium blank) <sup>(6)</sup>.

### **B-Surface Treatments:**

Numerous investigations revealed that pre-sintered zirconia needs to be surface treated using a laser. They suggested that all crystals would maintain their tetragonal shape following the sintering process, preventing undesired phase changes at the material's surface <sup>(7)</sup>. Er,Cr:YSGG laser irradiation (Millenium\Biolase Technology\ San Clemente\ CA) at the wavelength of 2.78  $\mu$ m, pulsed laser of hydrokinetic, a repetition rate for twenty Hz, and a the power of the beam density of six watts was used to treat the surface of the samples. During the 20-second irradiation, water and air fluxes of 55% to 65%, respectively, were administered. At a distance of 10 mm, a laser optical fiber (diameter of 600  $\mu$ m\ length: 6 mm) was put perpendicular to surface as shown in figure (2). The samples of hardness are sectioned into four areas to ensure that all the sample become irradiated with laser. <sup>(8)</sup>. While for acid etching the treating surface of specimen has been etched by 9.5% hydrofluoric acid (Bisco\ USA) for sixty seconds. After that, The samples was washed with water for thirty seconds and then dried by air <sup>(9)</sup>. When blasting the surface of zirconia to produce micro-roughness or micro-irregularities to improve micro-mechanical interlocking with porcelain veneer, aluminum oxide particles are most frequently utilized <sup>(10)</sup>. The specimens were subjected to sandblasting on the test surface for 15 seconds at a distance of 10 mm using 120  $\mu$ m Al<sub>2</sub>O<sub>3</sub> particles (renfert.germany) at a pressure of 2 bar. then, For 30 seconds, specimen cleaned under running water then air dried, Also the samples of hardness are sectioned into 4 areas to ensure that all the surface become sandblasted, Sintering is done according to manufacturer instruction <sup>(11)</sup>. A veneering ceramic cylinder built in prepared surface

of each sample using a custom-made spliced silicone mold. The Porcelain powder (lumex vita; vita Inc) was combined with distilled water before being add and condensed to create the cylinder 3mm diameter and 1mm in height. Tissue paper was used to remove excess water. Firing performed according to manufacturer instructions ( Sintering furnace Pro Dent China) each sample is placed in cold cure acrylic resin molding <sup>(12)</sup>.

### **C-TESTS:**

#### **1- Test Of Shear Bond Strength:**

All samples were embedded in self-polymerizing acrylic resin. so that it could be held in universal testing machine during test.

For this test, a universal testing machine (Gester GT-K03B/China) with 3mm diameter The sample was supported during the test by support rollers. The specimens were put onto a universal testing machine at a transverse speed of 1 mm/min while the base was stabilized <sup>(13)</sup>.

#### **2- Vicker's Micro Hardness Test:**

Each subgroup received 5 specimens, and the value of hardness was evaluated by a Vickers hardness tester (Digital Micro-Hardness Tester/LARYEE). A normal pyramid with a square bottom is utilized as the diamond indenter. Load capacity (for zirconia, 1 kilogram) <sup>(14)</sup>.

For 15 seconds, provide a continuous load. An optical microscope is used to measure the diameters of the indentation once the load is removed, and an average value is determined <sup>(15)</sup>.

## **Results**

### **1. Normality of Data Distribution**

The normality of the data distribution was assessed using the Shapiro-Wilk and Kolmogorov-Smirnov tests, as well as parametric tests, because the data seemed to be regularly distributed at  $p \leq 0.05$ .

### **2. Surface Treatment's Influence on Shear Bond Strength (SBS)**

#### **2.1 Shear Bond Of 3Y-HA ( high-alumina) To Ceramic After Surface Treating:**

At  $p < 0.05$ , the one way variance analysis (ANOVA) test revealed highly significant SBS values as revealed in table (1). While sandblasting did not significantly raise SBS values, both laser and acid etching surface treatments significantly increased SBS values as compared to the control group as in the figure (3) and table (2) in Duncan's test.

**2.2 SBS of 3Y-LA(low-alumina) to Ceramic after Surface Treatments:** As shown in figure (4), the oneway variance analysis (ANOVA) test revealed extremely significant SBS values at  $p < 0.05$ .

According to Duncan's multiple range assessment, sandblasting produced non-significantly different SBS values from the control group, whereas laser and acid etching considerably increased SBS when compared to that group as what is revealed in table (3).

### **3. Effects of Surface Treatments On Hardness of Yttrium Stabilized Zirconia**

#### **3.1 Hardness of 3Y-HA (high-alumina) After Surface Treatments:**

The table (4) was produced by applying descriptive statistics to the four groups (control, laser, sandblasting, and acid etching). At  $p \leq 0.05$  in the statistical analysis, one-way analysis of variance (ANOVA) indicated non-significant test results. Because of this, there was no variation in the Hardness values of 3Y-A following the three surface treatments (laser, sandblasting, and acid etching) listed in table (5).

#### **3.2 Hardness of 3Y-LA(low-alumina) After Surface Treatments :**

The descriptive statistics of 3Y-LA, as presented in table (6), indicate that there was no discernible impact of surface treatments on the hardness of the material. Based on the One Way of Analysis of Variance (ANOVA) test, there was no significant difference found between the control group and the acquired hardness values following surface treatments (see table (7)).

### **4. Modes of Failure**

In this investigation, three forms of bond failure between zirconia cores and veneering ceramic were observed with the bare eye:

1-Adhesive failure: every ceramic veneer separates from the core. This kind of failure was seen in 35% of the study's samples.

2-Cohesive failure: partial veneering separates while the remaining portion is attached to the core. In this study, 48% report having it.

3-Both cohesive and adhesive failures occur in a mixed failure. It has happened in 17% of all samples combined.

### **Discussion**

Metal-ceramic restorations are giving way to all-ceramic prosthetics in prosthetic dentistry, particularly for esthetic and to be biocompatible. Ceramic, also, have brittleness and broke easily. So, it must be developed a strong and aesthetically pleasing porcelain material has recently piqued the interest of researchers <sup>(16)</sup>. Tetragonal zirconia polycrystalline (Y-TZP), which is stabilized with yttria and has excellent mechanical and biocompatibility properties, is the strongest restorative ceramic. There are numerous alternative types of Y-TZP that can be made by combining different chemical components, such as alumina. Development has progressed from a framework material of the monolithic anterior and posterior restorations to fixed anterior and posterior prostheses <sup>(17)</sup>. However, because zirconia is a solid structure of polycrystalline without a glass phase, it has an opaque look that impacts the esthetics. As a result, the zirconia substructure must be cover by an appropriate ceramic veneer, however chipping of the ceramic veneer is the most prevalent cause of zirconia failure. Restorations that are already in place <sup>(18)</sup>. By mechanically or chemically roughening and altering the surface topography of zirconia, one can increase its surface area and create imperfections, which strengthen the micro-mechanical interaction <sup>(19)</sup>.

### 1- Shear Bonding Strength Test:

The value of (SBS) increased significantly for both laser and acid etching surface treatments, according to the results, although sandblasting had less of an impact and did not statistically differ from the control group for either of the two types of zirconium cores (3Y-LA and 3Y-HA). By using vaporization and micro-blasts to remove particles from the surface, erbium lasers can alter surfaces<sup>(20)</sup>. Fisher et al. claim that air abrasion is not a surface treatment required to increase binding strength. Air abrasion, on the other hand, in an disagreement to this study, created higher bond strength values, according to Kim et al. It is commonly known that zirconia may be sandblasted to activate and clean the surface while also increasing its roughness. Nevertheless, even after cleaning by water for ten minutes, the aluminium particles used for sandblast may still be present on the surface of zirconia. After sandblasting, only low amounts of alumina element were found on all of the ultrasonic cleaned zirconia surfaces; however, the amount steadily increased with large abrasive particle size and pressure; this could be the cause of the lack of an increase in the SBS value<sup>(21)</sup>. In an agreement with this study, The close resemblance between polished zirconia surfaces and shell ceramics suggests that fire forms chemical bonds between the two materials. Therefore, it was not necessary to increase bond strength by sandblasting the surface to make it rougher<sup>(22)</sup>. According to Yukiko et al.'s findings, which corroborate this work, zirconia's surface roughness increases steadily following sandblasting with 75-, 100-, and 150- $\mu\text{m}$  aluminum oxide. However, the shear bond strength does not alter significantly<sup>(23)</sup>. When compared to the control groups, the HF acid etching group demonstrated stronger bond strength. Acid etching by HF acid even it does not modify the surface morphology of zirconia, it does enhance wettability and surface energy<sup>(24)</sup>. This study results agree with the study made by Casucci A et al. (2009) who conclude that SEM and AFM studies revealed that acid etching produced a higher Ra value and a better

rough structure than sandblasting. There was also evidence of a loose porous structure, which could account for the increased SBS compared to Group Sand. These findings were in line with earlier research showing that by eliminating the non-organized and higher energy atoms at the prephery from the surface of zirconia, the etching solution greatly increases the intergrain gap and roughens the surface. Temperature, application time, concentration, and solution movement across the ceramic can all have an impact on etching rate<sup>(25)</sup>. These findings agree with those of Attoll et al.<sup>(26)</sup> and Sato et al.<sup>(27)</sup>, who found that the etching with HF acid had the maximum bond strength. These findings are explained by the fact that etching with HF acid improves micromechanics retention by dissolving the glass matrix.

### 2-Hardness Test:

The study's mean hardness for the 3Y-LA (low alumina) control group is (1423.6) Hv, whereas the surface treatment groups' means are as follows: laser, the mean hardness is equivalent to (1380.8), (1374.02) for sandblasting, and (1423.9) for acid etching. With  $p \leq 0.05$  for statistical analysis of variance (ANOVA), there were no statistically significant differences between the control group and the other surface treatment groups, While , the surface treatment groups' mean hardness for 3Y-

HA (high alumina) zirconia was (1393.5) for the control group and the following for the treatment groups:

The average hardness for the laser group was (1428.1), the sandblasting group was (1388.2), and the acid etching group was (1293).

Additionally, at  $p \leq 0.05$  in the statistical analysis of variance (ANOVA) there were no statistically significant differences between the control and other surface treatment groups.

These findings align with the research conducted by (Pittayachawan et al., 2008)<sup>(28)</sup> and (Chen et al., 2020)<sup>(20)</sup>. These outcomes are primarily the result of surface treatments that were carried out before sintering to minimize any

substantial damage to physical qualities. It has been suggested that mechanical surface treatments performed to fully sintered zirconia could result in a phase transition of zirconia from tetragonal to monoclinic, reducing reliability, toughness, and increasing the risk of material fracture. - Preferred is sintered zirconia to prevent phase transition <sup>(29)</sup>. This may be possibly due to a phase transfer tetragonal » monoclinic caused at the time of hardness test, specially at the grooves created by the indenter's edges.

### 3. Failure Mode:

This behavior can be explained by separating the shell from the integral zirconia core structure, which is in line with several research on the breakdown mode of the core-shell link.

This trend was also observed in other laboratory investigations (Stoddart et al., 2007) <sup>(30)</sup>.

Where fracture deflection at the core/shell interface was evaluated. Two ways exist to explain this: First, crack deflection could be caused by YTZP's increased resistance to fracture propagation.

Second, interlaminar fracture deflection is caused by the comparatively weak relationship between the ceramic shell and the zirconia core <sup>(31)</sup>.

### Conclusion

1. sandblasting doesn't effectively affect SBS between zirconium core and veneering porcelain.

2. HF acid etching was more effective than sandblasting in improving SBS.

3. Irradiation using Er,Cr:YSGG lasers effectively enhance SBS at (6W).

4. Hardness of zirconium cores didn't affected significantly by pre-sintering surface treatments.

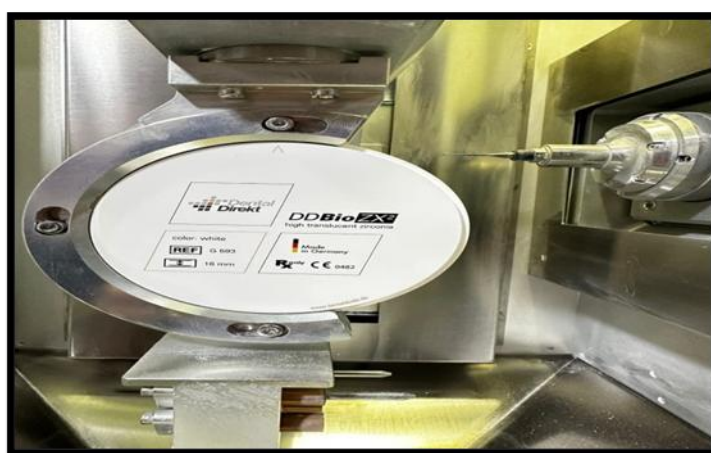


Figure (1): Grinding machine with a zirconia blank



Figure (2): laser surface treatment

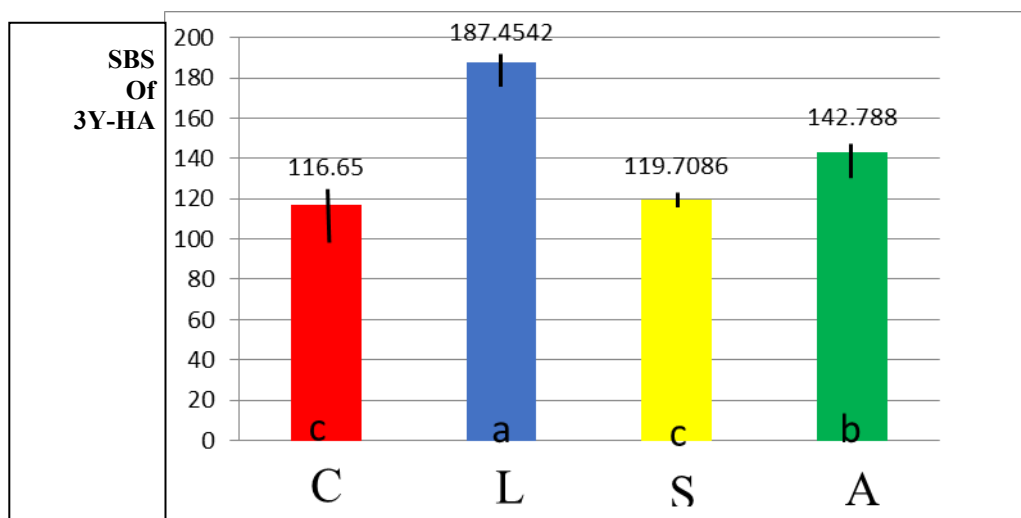


Figure (3): Mean  $\pm$  Std deviation of SBS for 3Y-HA after surface treatments (where S refer to sandblasting , L Laser , C control and A refer to acid etching).

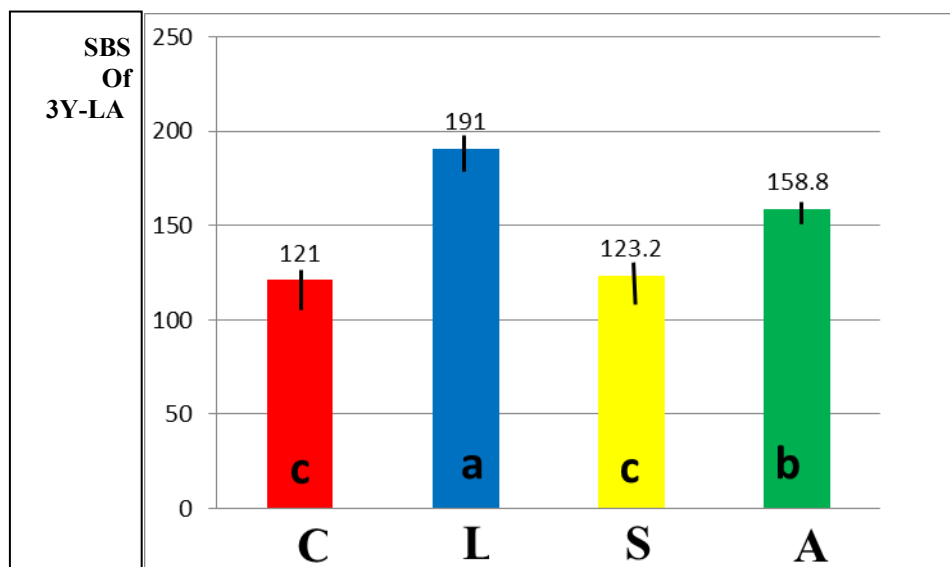


Figure (4): Mean  $\pm$  Std deviation of SBS for 3Y-LA after surface treatment

Table (1) ANOVA of SBS for 3Y-A to ceramic after surface treatments

	Sum. Squares	D.F.	Average Square	F.	Sig.
Among Groups	23182.078	3	7727.359	61.845	.000
Within Groups	1999.146	16	124.947		
Total	25181.224	19			

Table (2) duncan for SBS of 3Y-HA after surface treatment

Duncan				
name	N	Subset for alpha = 0.05		
		1	2	3
3Y-HA control	5	116.6500		
3Y-HA sandblast	5	121.7086		
3Y-HA acid etch	5		142.7880	
3Y-HA laser	5			202.3442
Sig.		.485	1.000	1.000



Table (3): Duncan's multiple range comparison of SBS for 3Y-LA

	N	C	B	A
3Y- LA control	5	121.0000		
3Y-LA sandblast	5	123.2000		
3Y-LA acid etch	5		158.8000	
3Y-LA laser	5			191.0000
Sig.		.746	1.000	1.000

Table (4): descriptive statistics of Hardness for 3Y-HA after surface treatments

	N.	Mean.	Std . Dev.	Min.	Max.
3Y-HA control	5	1393.5200	109.17865	1277.00	1572.40
3Y-HA laser	5	1428.1200	126.13989	1231.00	1566.00
3Y-HA sandblast	5	1388.2800	87.70491	1280.80	1507.60
3Y-HA acid etch	5	1293.0600	139.28423	1126.60	1505.00
Total	20	1375.7450	119.19889	1126.60	1572.40

Table (5): ANOVA for Hardness of 3Y-HA after surface treatment

	Sum. Squares	D.F.	Average Square	F.	Sig.
Among Groups	50265.134	3	16755.045	1.220	.335
Within Groups	219693.996	16	13730.875		
Total	269959.130	19			

Table (6): descriptive statistics for Hardness of 3Y-LA after surface treatments

	Nu.	Me.	Std. Dev.	Min.	Max.
3Y-LA control	5	1423.6000	85.70473	1314.00	1516.00
3Y-LA laser	5	1380.8800	28.13400	1334.10	1402.10
3Y-LA sandblast	5	1374.0200	36.12696	1327.30	1419.40
3Y-LA acid etch	5	1423.9400	113.1453 2	1300.70	1584.00

Table (7): ANOVA for hardness of 3Y-LA after surface treatments

	Sum. Squares	Df	Me. Square	F.	Sign.
Among Groups	10845.650	3	3615.217	.650	.594
Within Groups	88975.368	16	5560.961		
Total	99821.018	19			

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