

Effect of Different Surface Treatments on Shear Bond Strength of Zirconia Based Ceramic and Veneering Porcelain



Sarah El-ayouty1, Mohamed El-layeh2, Manal Abou-Madina.3

Teaching assistant, Mansoura University, Faculty of Dentistry, Fixed Prosthodontics Department, Egypt. Lecturer, Mansoura University, Faculty of Dentistry, Fixed Prosthodontics Department, Egypt. Professor, Mansoura University, Faculty of Dentistry, Fixed Prosthodontics Department, Egypt.

Abstract:

Aims of the study: Aim of this in-vitro study was to evaluate the shear bond strength of veneering ceramic to Y-TZP infrastructures after different surface conditioning techniques.

Material and Methods: 43 Y-TZP blocks were cut from pre-sintered zirconia disks. Specimens were divided into 4 groups (n=10 in each group) according to the surface conditioning technique used (Diamond grinding, Al₂O₃ air-borne particle abrasion, Silica containing glass-ceramic liner application and Nd:YAG Laser irradiation). 3 of the specimens were used for optimization of Nd:YAG Laser surface conditioning. Lithium disilicate veneer layers were pressed on each Y-TZP specimen. Shear bond strength was tested using a universal testing machine. All specimens of each group were examined for failure mode analysis using an optical microscope. Scanning Electron Microscope (SEM) was used to detect the roughness produced over Y-TZP block surface after different surface conditioning techniques and X-ray diffraction test (XRD) for phase transformation determination. Analysis of the data was done using one-way Analysis of variance (ANOVA) test.

Results: Silica-containing glass ceramic liner application and Nd:YAG Laser irradiation resulted in a significantly higher SBS ($p \le 0.05$) than did Diamond grinding and Al₂O₃ air-borne particle abrasion. Diamond grinding and Al₂O₃ air-borne particle abrasion groups showed more adhesive failure than mixed failure in contrast to Silica containing glass ceramic and Nd:YAG Laser irradiation groups which showed more mixed failure than adhesive failure. SEM revealed great surface irregularities at the Nd:YAG laser irradiated group. XRD test revealed that Al₂O₃ was the only conditioning technique which resulted in monoclinic phase transformation in contrast to the other conditioning techniques used.

Conclusion: Silica containing glass ceramic liner application and Nd:YAG Laser surface conditioning could be considered valuable means of shear bond strength improvement between Y-TZP core and lithium disilicate veneering layer.

Key words: Y-TZP, Lithium disilicate, shear bond strength, surface conditioning techniques.

Introduction

irconia stands out as a promising and versatile ceramic owing to its optical, biological and mechanical features. Zirconia has three allotropic forms with different temperature range stability (monoclinic at 1170 °C, tetragonal at 2370 °C, and cubic at 2680 °C). Addition of oxides like magnesia, ceria and yttria can lead to stabilization of tetragonal phase at room temperature. Yttria (Y₂O₃) is the most popularly utilized oxide, which creates yttria- stabilized tetragonal zirconia poly-crystals (Y-TZP). ¹Y-TZP presents modulus of elasticity of about 210 GPa, fracture toughness 10 MPa and flexural strength of more than 900 MPa. ²Yet, a compatible veneering ceramic layer is needed over such a highly crystalline ceramic with high opacity to obtain better esthetics. ³Considering all-ceramic multilayered restorations, Y-TZP -veneering ceramic interface is the most common area of clinical failure. Chipping of the veneering ceramic is the most recorded type of failure noticed in Y-TZP veneered restorations. These failures compromise the restoration both functionally and aesthetically. The veneering ceramic chipping has been caused by variable factors, like: infrastructure design, which should provide support to the veneering ceramic ⁴, thicknesses proportionality of the restoration layers, residual thermal stresses throughout the restoration ⁵ and veneering ceramic

mechanical features. ⁶Therefore, various surface conditioning techniques have been utilized to increase surface roughness and thus enhancing Y-TZP infrastructure -veneering ceramic bond strength. ⁷These surface conditioning techniques include Diamond grinding, Airborne particle abrasion, Liner application, Plasma and Laser surface conditioning. No standard technique for the achievement of optimal shear bond strength between Y-TZP infrastructure and veneering ceramics, favorable failure mode and without causing any phase transformation has been established yet.

Material and Methods:

Specimens preparation:43 Y-TZP blocks (40 will be used for surface conditioning and 3 will be used for optimization of Nd:YAG Laser surface conditioning). Blocks with dimensions of (13.8 mm ×16.1 mm×3.6 mm) were cut from pre-sintered zirconia disks (Ceramill Zolid White, Amman Girrbach AG, Herrschaftswiesen, Austria) using Isomet 4000 linear precision saw with presence of water coolant during the cutting procedure.The Y-TZP blocks were then fully sintered in the sintering furnace (Ceramill Therm, AmmanGirrbach AG, Herrschaftswiesen, Austria) according to manufacture instructions.After sintering, All Y-TZP blocks shrank to dimensions of (11.2 mm × 12.96 × 2.89 mm). All Y-TZP blocks were then cleaned in an ultrasonic bath for 5 mins and air-dried before surface conditioning. Then a circle with 5 mm diameter was drawn using a pencil at the centre of each Y-TZP blocks for marking of the bonding area between Y-TZP block infrastructure and lithium disilicate veneer disc.

Specimen grouping: The 40 Y-TZP intended to be used for the different surface conditioning techniques were divided randomly into four groups (n=10).

Group A (Diamond grinding): The bonding area of the Y-TZP blocks in this group were ground by a standard (106 μm-125 μm) grit size diamond (DIA-BURS BR-31, Mani Inc., Tochigi, Japan) in (Kavo Dental, Biberach, Germany) slow-speed motor coupled to (Sirona T3 mini, Dentsply Sirona Inc., Germany) hand-piece. All Y-TZP blocks were fixed to a surveyor to assure the parallelism between the diamond bur used for grinding and Y-TZP blocks bonding area. To assure that the whole bonding area of each Y-TZP block was ground, the bonding area was marked with a permanent marking pencil. A mild oscillatory movement was employed to prepare the Y-TZP blocks bonding area with minimal force to minimize stress concentration till a point where all the marking of the bonding area was eliminated. The diamonds were commuted after grinding of every five specimen to ensure a symmetrical stone grittiness.Group B (Al₂O₃ air-borne particle abrasion): The bonding area of the Y-TZP blocks in this group were submitted to Al₂O₃air-borne particle abrasion at 10 mm specimen-to nozzle distance with a particle size of 50 µm at 0.4 MPa pressure for about 10 seconds.Group C (Silica containing glass ceramic liner): The bonding areas of Y-TZP blocks in the liner group were submitted to low-fusing flourapatite containing glass-ceramic liner (IPS e.max Ceram Zirliner, Ivoclar Vivadent, Liechtenstein). Mixing of the powder and the liquid of the glass-ceramic liner was done until creamy consistency is achieved and then a brush was used for application of the liner on the bonding area of all Y-TZP blocks. All Y-TZP blocks were then vibrated was till an even, greenish color effect is obtained to create a regular and a well-condensed layer of liner, fired in the furnace (Programat P500, Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacture instructions (at 960°c for 1 hour) and then allowed to cool to room temperature. Group D (Nd:YAG Laser irradiation):For Laser optimization, 3 Y-TZP blocks were used for determination of the condition which will create better surface roughness. Then all Y-TZP block bonding areas were irradiated using Nd:YAG Laser (PowerLite DLS 9000, Amplitude Laser, Boston, USA) with 1064 wave length,7 ns pulse width, 10 hz repetition rate and .8 diameter of beam and 8 w power setting. The light source was perpendicular to the zirconia ceramic surface with a working distance of 10 cm, and the bonding areas were scanned for 30 shot with no cooling. All the 40 specimens of the 4 test groups were cleaned ultrasonically for 5 mins to remove any surface contaminations.

Application of Lithium Disilicate veneer disc:Wax patterns were prepared using a splitted pattern resin mold. Wax was heated to softening point to fill the splitted resin mold and form a wax disc with (5mm diameter and 2 mm height) and were placed over the Y-TZP blocks surface corresponding the bonding area at the centre of each block.

The Y-TZP blocks with the wax patterns were invested using phosphate-bonded universal investment material inside IPS investment silicone ring. The wax patterns were burnt out in a furnace at 850°C for 60 mins. Lithium Disilicate ingots (IPS Emax Press HT; Ivoclar Vivadent, Schaan, Liechtenstein) were automatically pressed into the mold in a furnace (Programat Ep 3010; Schaan, Liechtenstein) at 905°C for 25:50 mins. The IPS silicone investment ring was then allowed to cool to room temperature. Al₂O₃ air-borne particle abrasion at a pressure of 0.4 MPa and particle size of 110 μ m was used for devesting. Eventually, Y-TZP blocks infrastructures with lithium disilicate veneer disc were cleaned in an ultrasonic bath with distilled water for 5 minutes and then air-dried.

Specimens fixation: Cylindrical epoxy resin blocks were fabricated and then (Marathon 103, saeyang, Daegu, Korea) surveyor was used to prepare a cavity within every cylindrical epoxy resin blocks with dimensions slightly larger than that of Y-TZP blocks ($11.2 \text{ mm} \times 12.96 \text{ mm} \times 2.89 \text{ mm}$). All veneered Y-TZP specimens were fixed in their position inside the cylindrical epoxy blocks using a cyanoacrylate based glue so that the interface between Y-TZP blocks infrastructures and Lithium disilicate veneer disc would be flushed with the top surface of the epoxy resin blocks.

Shear Bond Strength Testing: Shear bond strength of all the specimens of each group was evaluated using (Instron 3345, Instron, MA, USA) universal testing machine Shear force was applied at the interface between Y-TZP block infrastructure and Lithium disilicate veneer (Figure 1)disc with cross-head speed of 0.5 mm/min till failure. The load was measured in newtons (N). Shear bond strength was then figured in MPa (Bluehill Lite software, Instron, MA, USA) by dividing load in N/Area in mm².



Y-TZP block infrastructure.

Figure 1:Specimen and test design.

Failure mode Analysis:Optical microscope (Olympus SZ 30, Olympus, Tokyo, Japan) at x20 magnification was used over all 40 specimens of the 4 different groups to recognize the failure mode. Failure mode can be classified as: (A) Adhesive, (B) Cohesive within Y-TZP blocks, Cohesive within lithium disilicate veneer disc and (C) Mixed failure. **Scanning Electron Microscope:**From each group, three fractured specimens were randomly selected and cleaned in an ultrasonic bath for 5 mins, allowed to dry, then ion-sputtered for 60 seconds with Au-Pd to make the specimens

electro-conductive for SEM. Specimens were then observed under SEM (JSM-6510LV, JEOL, Japan), pictured at magnification (X1000, X2000) to detect the roughness produced over the debonded Y-TZP block surface of all different groups.

Phase Distribution Analysis: Y-TZP crystalline phase distribution analysis after debonding of the specimens with different surface conditioning techniques was carried out over 3 randomly selected specimens in each group using X-ray diffraction (Empyrean, PANanalytical, Netherlands) utilizing Cu K α ($\lambda = 1.540598$ Å) radiation source at 40 kV and 40 mA, Scan angle range (2 θ) of 5°-100° and step size (the angle at which the diffractometer moves) of 0.0131°. For the recognition of the crystalline phase distribution the software (X'pert high score plus, PANalytical, Netherlands) was used. The monoclinic weight fraction was measured utilizing the next equation: ⁸

 $Xm = \frac{Im(111) + Im(111)}{Im(111) + Im(111) + It(101)}$

Where Im (111) represents the intensity of monoclinic peaks at $2\theta=28$ and Im (111) represents the intensity of the monoclinic peaks at $2\theta = 31.2$ degrees, and It (101) represents the intensity of the tetragonal peaks ($2\theta = 30$ degrees).

The percentage of monoclinic phase (Fm) was measured utilizing the next equation: $^{9, 10}$

 $Fm = \frac{1.3111 Xm}{1+0.3111 Xm}$

Statistical analysis: One way ANOVA and Fischer's least significant difference (LSD) multiple comparison tests were used to analyze the resultant data using (SAS Statistics, IBM, Chicago, IL) software program at $p \le (0.05)$.

Results:

Shear bond strength evaluation: The mean shear bond strength and standard deviation values of all tested groups are represented in (Table 1). The lowest mean SBS was noticed in the Diamond grinding group (11.303 ± 2.440) MPa), followed by the Al₂O₃ air-borne particle abrasion group (15.144±4.101 MPa), while the highest mean SBS values was the Nd:YAG Laser irradiation group (35.631±4.201 MPa) followed by the Silica containing glass ceramic liner application group (29.334±3.034 MPa).Oneway ANOVA statistical analysis was used to determine the SBS differences of all tested groups which, revealed that there a significant difference among all groups (Diamond grinding, Al₂O₃air-borne particle abrasion, Silica containing glass ceramic liner application and Nd:YAG Laser irradiation) ($p \le 0.05$).

 Table 1: Mean shear bond strength and standard deviation values (±SD) of the groups (MPa).

Group	Ν	Mean	±SD (MPa)
		(MPa)	
Grinding	10	11.303 ^d	±2.440
Airborne	10	15.144 ^c	±4.101
particle			
abrasion			
Liner	10	29.334 ^b	±3.034
Nd:YAG Laser	10	35.631ª	±4.201
P value	0.0001		
LSD	3.1944		

a-c= Means with the same letter in the column are not significantly different at $p \le 0.05$.

Failure mode classification:Optical microscope analysis showed that adhesive type failure (at the LiSi₂- Y-TZP interface)was the most prevalent in the Diamond grinding group and the Al₂O₃ air-borne particle abrasion group), whereas mixed type failure (adhesive and cohesive with very little quantity of LiSi₂ layer left connected to Y-TZP surface) was most frequent in the Silica containing glass-ceramic liner application group and the Nd:YAG Laser irradiation group with no evidence of pure cohesive failure in all tested groups.

Scanning electron microscope imaging:SEM images revealed that, InDiamond ground surface the grooves were observable with a relatively smooth background surface whereas, Al_2O_3 air-borne particle abrasion caused a distinguished rough surface with depressed and elevated areas with dissimilar indentations. SEM images of Y-TZP surface within the Silica containing glass ceramic liner application group revealed crystalline needle like shapes which may suggest remnants of the liner material or LiSi2 which were partly connected to the detached Y-TZP surface and finally, Nd:YAG Laser irradiated surfaces displayed small pits with deep cervices and great surface irregularities. Phase distribution analysis: Analysis of the diffractograms revealed that neither Diamond grinding, Silica containing glass ceramic liner application nor Nd:YAG Laser irradiation caused destabilization of the tetragonal phase of Y-TZP infrastructure without any appearance of the monoclinic phase. Whereas, Al2O3 air-borne particle abrasion caused appearance of some characteristic peaks representing the monoclinic phase at Im(111). Applying the volume fraction equation revealing that the volume fraction was about 0.16 %.

Discussion:

The objective of this prospective study was to evaluate the shear bond strength of veneering porcelain to zirconia infrastructure after different surface conditioning techniques (Diamond grinding, Al₂O₃ air-borne particle abrasion, Silica containing glass ceramic liner application and Nd:YAG Laser irradiation). The use of Nd:YAG Laser irradiation and Silica containing glass ceramic liner application increased the shear bond strength thus, the null hypothesis of the present study was accepted. The use of lithium disilicate glass ceramic for fabrication of veneer layer is because of its good aesthetic properties, as translucency, fluorescence, opacity,

shine, texture and imitating the optical qualities of the tooth structure. ¹¹However, it has a high degree of friability, high modulus of elasticity and low fracture toughness, which can cause internal fractures. ¹²With the aim of controlling these limitations, Yttria-stabilized tetragonal zirconia (Y-TZP) can be used as an infrastructure material owing to its excellent fracture strength, due to the high crystalline content. ¹³Although Y-TZP has high opacity and reduced aesthetic characteristics; it is used wisely for the fabrication of singleunit and multiple fixed dental prostheses. ¹⁴The assembly of these ceramic materials creates a restoration that has both resistance to occlusal forces, owing to its Y-TZP framework, and high aesthetic qualities, due to the fluorapatite glass ceramic used as a veneering material. ¹⁵However, the junction between these two ceramic materials is one of the most vulnerable aspects of the restoration. Therefore, the chipping of the veneering ceramic is blamed in the literature as the major problem of all-ceramic prosthesis with a Y-TZP infrastructure. ¹³SBS test was used in this study as did Korkomaz et al.¹⁶ and Fischer et al.¹⁷ as this test is easily used and the forces are applied vertically to the bonding area. 33 but, the unfavorable stress distribution of the test may cause incorrect data interpretation. 18

In this in vitro study, the 1st surface conditioning technique used was Diamond grinding as grinding with diamond is a common step of fabrication to bring out individual structures on the Y-TZP core. Diamond grinding reported the lowest mean SBS (11.303 MPa) in agreement with Mosharraf R et al. ¹⁹They concluded that, Diamond grinding significantly reduced the SBS of zirconia and veneering porcelain as it showed lower SBS values when compared to the control group (no surface conditioning) and Al₂O₃ air-borne particle abrasion with the use of a liner. These results were also supported by Korkmaz et al.¹⁶ who reported that Diamond grinding did not improve the SBS between core and veneer layers, in contrast to the postulate that a rougher surface area of Y-TZP gives a better bonding. This might be revealed to Diamond grinding may develop high stresses and create severe surface cracks which can decrease the strength and reliability of the material. ²⁰

This in vitro study utilized Al₂O₃ air-borne particle abrasion as one of the most commonly used surface conditioning techniques of Y-TZP. This procedure is used for obtaining a rougher surface, to increase the bonding area either to the inner surface of the restoration or between the core-veneer surfaces. ¹⁷Al₂O₃ air-borne particle abrasion was done at 0.4 MPa pressure and a grain size of 50 µm as recommended by Nakamura et al.²¹ who concluded that 50 µm grain sized and 0.4 MPa pressured Al_2O_3 air-borne particle abrasion will provide a strong adhesive bond between the zirconia infrastructure and the veneering ceramic. In addition, Liu et al. ²² found that 0 .35 MPa pressured and 50 μ m grain sized air-borne particle abrasion led to a significant different improvement of shear bond strength. Surface conditioning with Al₂O₃ air-borne particle abrasion as compared to Silica containing glass ceramic liner application and Nd:YAG Laser irradiation groups showed significantly lower SBS because the differences were significant at ($p \le 0.05$). This was in agreement with Kirmali et al. ²³ who stated that "as compared to untreated zirconia, Pre-sintering air-borne

particle abrasion resulted in higher SBS but the differences were not statistically significant".Elsaka. ²⁴ stated that the surface treatments of different zirconia cores with air-borne particle abrasion did not significantly improve the adhesion between zirconia and veneering ceramic.Savas et al.²⁵ reported that air-borne particle abrasion caused no significant improvement of the bond strength between the lithium disilicate veneer layer and the Y-TZP infrastructure layer.Al₂O₃ air-borne particle abrasion of Y-TZP infrastructure in the current study caused the monoclinic volume fraction increase by about 0.16% which was similar to Kim et al. ²⁶results who also showed monoclinic volume fraction increase of the air-borne particle abrasion group compared to control group with no surface conditioning and liner application group. The tetragonal Y-TZP TEC $(10.8 \times 10 - 6/K)$ is much higher than that of monoclinic Y-TZP (7.5×10–6/K). ²⁷The tetragonal and monoclinic phases' thermal expansion coefficient (TEC) differences have a harmful effect on the shear bond strength of Y-TZP infrastructure and LiSi2 veneering layer.28To avoid these phase transformations, He et al. ⁷ suggested pre-sintering airborne particle abrasion and Çağlar et al.²⁹ suggested heat treatment following the surface conditioning. The transformed Y-TZP monoclinic phase following the airborne particle abrasion procedure would be changed back to the tetragonal Y-TZP phase after the heat treatment. ²⁹If airborne particle abrasion is used after the sintering process, the heat treatment could be a veneering step or re-glazing after clinical adjustments. ³⁰Also, Passos et al. ³⁰ suggested that tetragonal-monoclinic phase change can be reduced by using relatively small-size Al₂O₃ particles. In addition, optimum duration for the application of air-borne particle abrasion procedure is not evident. Sato et al. ³¹ reported that the airborne particle abrasion particles type affect the density of Y-TZP surface transformed layer, not the application duration. Therefore, 10 sec application time was performed in the current study as recommended by Fischer et al. 17 and Qeblawi et al. ³².XRD results revealed that none of the surface conditioning techniques except the Al₂O₃ air-borne particle abrasion method, caused monoclinic phase change which agrees with the findings of He et al. ⁷ and Liu et al. ²² Silica containing glass ceramic liner application in this in vitro study caused a significant enhancement of the interfacial bond strength between Y-TZP infrastructure and LiSi₂ veneer layer (P≤0.05). Silica containing glass ceramic liner application significantly increased the mean SBS between Y-TZP and LiSi2 than did Al2O3 air-borne particle abrasion and Diamond grinding. The wetting action may have been increased by the silica-containing glass ceramic liner application. Interfacial bonding between the lithium disilicate (or liner) and Y-TZP infrastructure is suggested to be chemical or mechanical as, some studies ¹⁷ observed propagation of different components of silica-containing glass ceramic liner into the facing surface of Y-TZP. Energy dispersive x-ray spectroscopy(EDS spectrum) of Kim et al. ²⁶ study showed diffusion of Al, Si, K, Na into Y-TZP infrastructure which supports the evidence of a chemical bond between veneer and infrastructure materials. ³³In agreement with this, SEM images of the current study suggest presence of remnants of the Liner material or LiSi2

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that were left attached to Y-TZP surface. Also analysis of the failure mode revealed that adhesive fracture in liner specimens was noticed less than in other surface conditioning techniques specimens with higher frequency of mixed failure mode. In contrast to current study findings Sinem S et al. ³⁴ reported Liner group bond strength reduction as compared to air-borne particle abrasion group. They explained the bond strength reduction by flatness of the Y-TZP surface at which the liner was applied which caused delamination of the thin veneer layer used. Aboushelib et al. ³⁵ explained the lower bond strength following Liner application by presence of flaws and voids resulted from liner application causing crack propagation because of concentration of the stresses under loading.

In the present study, Nd:YAG Laser surface irradiation produced a significant increase in mean SBS compared to the other surface conditioning techniques used (Diamond grinding, Al_2O_3 air-borne particle abrasion and Silica containing glass ceramic liner application). Nd:YAG irradiation caused a mechanical bonding between Y-TZP infrastructure which is supported by SEM images taken after the conditioning procedure and the high frequency of mixed failure type in the Nd:YAG Laser group. In agreement with Henriques et al. ³⁶ results who, stated that Nd:YAG Laser irradiation of Y-TZP infrastructure significantly increased the SBS by about 75% of that of air-borne particle abrasion. Their ³⁶ results also supported XRD results of the current study. At which, No phase transformation was caused by Nd:YAG Laser irradiation surface conditioning.On the contrary, Noda et al. ³⁷ stated that tetragonal zirconia should not be treated by Nd:YAG Laser irradiation as it causes change of the Y-TZP composition of elements, cracks and blackening effect.

SEM images of the current study suggest that the Nd:YAG Laser irradiation group had the greatest surface roughness with deep cervices and more surface irregularities that any other test group in the current study which may explain SBS results at which Nd:YAG Laser irradiation group had the highest mean SBS.

Conclusions:

Due to restrictions of the present study, the next conclusions were deduced:

- 1. Shear bond strength between Y-TZP core and lithium disilicate veneer layer increased by Nd:YAG laser irradiation or the silica-containing glass-ceramic liner application.
- 2. Al₂O₃air-borne particle abrasion caused monoclinic phase change which, possibly weaken the Y-TZP Lithium disilicate interfacial bond strength due to the difference in the TEC between the monoclinic and tetragonal phases.

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