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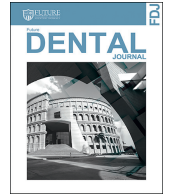
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Effect of different surface treatments on bond strength, surface and microscopic structure of zirconia ceramic

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ABSTRACT

Objectives: To evaluate the effect of different surface treatments; plasma treatment, silica coating using plasma technology and sandblasting, on bond strength, surface roughness and microscopic structure of yttria-stabilized tetragonal zirconia polycrystals(Y-TZP) after thermo-cycling.

Materials and methods: One hundred discs (n = 100) of yttria-stabilized tetragonal zirconia were prepared from (Y-TZP) ceramic blocks using MAD/MAM milling technology, and were divided into four equal groups (n = 25) according to the type of surface treatment. Group (1): control (no surface treatment). Group (2): zirconia discs were sandblasted by alumina particles. Group (3): zirconia discs treated by plasma technology to produce surface roughness. Group (4): zirconia discs coated by silica using plasma technology. Samples of each group were subdivided into four subgroups according to different analytical techniques. Subgroup (A): (n = 10) subjected to testing of bond strength of zirconia discs to adhesive resin cement after thermo-cycling. Subgroup (B): (n = 5), to evaluate the microscopic changes of zirconia discs by scanning electron microscope (SEM). Subgroup(C): (n = 5) to evaluate the crystal structure and phase transformation of YZ ceramic by X-ray diffraction (XRD). Subgroup (D): (n = 5) to measure three dimensional surface roughness of YZ ceramic by optical interference microscope.

Results: Statistical analysis of shear bond strength by ANOVA revealed the presence of no statistically significant difference between group (3) and (4); both showed the statistically highest mean shear bond strength values. Group (2) showed statistically significantly lower mean values followed by group (1). SEM showed that the topographic pattern differed by different surface treatments of samples. XRD revealed that; group (1) showed the statistically significantly highest mean % of zirconium oxide (Tetragonal phase). Group (2) showed the statistically significantly lowest mean % of Zirconium oxide (Tetragonal phase) and highest mean % of Boehmite and Zirconium oxide (Anorthic phase); Group (3) and (4) showed the statistically significantly highest mean % of Zirconium oxide (Monoclinic phase) and low % of zirconium oxide (Tetragonal phase). 3D- optical surface roughness showed that group (3) and (4) had highest mean (Ra) values. Group (2) showed statistically significantly lower mean values. Group (1) showed the statistically significantly lowest mean (Ra) values.

Conclusions: (1) Surface treatments of Y-TZP ceramic together with MDP primer and silane-coupling agent application improve the bond strength to resin cement. (2) Plasma-Silica coating and plasma-oxygen treatment, both are valuable methods that improve the bond strength of resin cement to Y-TZP ceramic. (3) Silica coating by plasma technology provides durable bond strength and can be a promising alternative pretreatment before silane application to enhance bonding with zirconia ceramic. (4) Tetragonal-monoclinic phase transformation had occurred in Y-TZP samples received both types of plasma treatment.

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1. Introduction

New ceramic systems have been developed as attempts to eliminate metal infrastructures and allow optimal distribution of reflected light, providing high quality aesthetic restorations through the use of reinforced ceramic cores either by dispersion of leucite [1], glass infiltration into sintered alumina (Al_2O_3) [2,3], the use of high-purity alumina [4] or zirconium dioxide (zirconia, ZrO_2) [5]. Zirconia has emerged as a versatile and promising ceramic material because of its biological, mechanical and optical properties. With a flexural strength of more than 900 MPa, fracture toughness of up to $10 \text{ MPa/m}^{0.5}$, and an elastic modulus of 210 GPa, they exhibit better mechanical performance, superior strength and fracture resistance than do other ceramic materials [5,6]. Zirconium dioxide as a dental material has a wide range of applications [7], they were initially used for endodontic dowels and implant abutments [8,9]. Its use has been extended to single crowns [10,11] and posterior three-unit fixed partial dentures which can be fabricated with a manual copy-milling machine, or computer aided design/computer aided manufacturing (CAD/CAM) systems [12,13] of either pre-sintered [14] or fully sintered zirconia blocks [15].

Adhesion of resin cement to high-strength zirconia ceramics is not expected to be improved by acid etching and silanization because they are inert acid resistant ceramics [16–21]. For zirconia ceramics, airborne-particle abrasion is an alternative method for roughening the ceramic surface [20,22,23].

However, there are some possibilities for improving bonding to zirconia based ceramics that need to be tested, including modern techniques for surface treatments by plasma technology. Plasma is defined as a gas in which part of the particles that make up the matter are present in ionized form. This is achieved by heating the gas leading to dissociation of the molecular bonds and subsequently ionization of the free atoms. Thereby, plasma consists of positively and negatively charged ions and negatively charged electrons as well as radicals, neutral and excited atoms and molecules [24,25].

In material science, possible applications of plasma include the modification of surface properties like electrochemical charge or amount of oxidation, wettability, hardness, resistance to chemical corrosion, the water absorption capacity as well as the affinity toward specific molecules can be modulated [26], using the common plasma gas sources as oxygen, argon nitrogen or hydrogen.

Some studies have examined the effect of sandblasting on shear bond strength of resin cement to zirconia ceramic, but further data is needed to correlate the effect of sandblasting and different plasma modalities on shear bond strength, microstructure, surface roughness and phase transformation of yttria-stabilized tetragonal zirconia.

2. Materials and methods

To conduct the present study, one hundred discs ($n = 100$) of Yttria-stabilized tetragonal zirconia ceramic (Y-TZP) were fabricated using Manual-aided Design/Manual-aided Manufacturing (MAD/MAM) system (Talent dental, FP50-XP, Korea) referred to as copy milling technique which is based on the pantographic principle.

2.1. Preparation of composite resin pattern

In order to standardize the shape and dimensions of the samples, a specially designed Teflon mold was machine-cut in order to fabricate circular resin discs of 10 mm diameter and 3 mm thickness. The inner walls of the mold were painted with separating medium (*Vaseline petroleum jelly*, Mainland, China) then composite

resin (*Te-Econom Plus*, Ivoclar Vivadent, Liechtenstein) layers were incrementally condensed into the mold and light cured (*XL-3000*, 3M/ESPE, St. Paul, USA) for 40 s on each side, for a total of 120 s. After complete polymerization, the composite resin pattern was removed from mold and inspected for any deficiencies which if found were corrected by addition.

2.2. Milling of Y-TZP samples

The composite resin pattern was placed in the pantographic machine. The copying arm of the machine traced the composite pattern while the cutting arm, which had a carbide cutter (*TF14*, Syandent tools, China) milled the pre-sintered zirconia block. After completion of the milling process, the milled discs were separated and handled with care to avoid damage to their margins or initiation of microscopic cracks leading to subsequent failure.

2.3. Sintering process of zirconia discs

The milled zirconia discs were sintered in high temperature furnace (*Wholesale Sintering Furnace, Ds-1700MX*, Mainland, China) according to the manufacturer's recommendations. The temperature was raised to 1500°C in 2 h then kept at final temperature (1500°C) for 2 h. Samples were slowly cooled to less than 100°C in 1 h.

2.4. Surface treatments of samples

One hundred discs ($n = 100$) of Yttria-stabilized tetragonal zirconia ceramic (Y-TZP) were divided into four equal groups, ($n = 25$ each) according to type of surface treatment; Group (1): control (no surface treatment). Group (2): zirconia discs were sandblasted by alumina particles. Group (3): zirconia discs treated by plasma technology to produce surface roughness. Group (4): zirconia discs coated by silica using plasma technology.

2.4.1. Sandblasting of the samples, group (2)

1. Each sample was individually mounted in a specially constructed holder which aided in standardization of the distance of sample exposure from the sandblasting nozzle (10 mm).
2. The sample was sandblasted with $110 \mu\text{m}$ aluminum oxide, at 2 bar pressure, for 15 s [27], using sandblasting machine (*Sandstorm*, Vaniman manufacturing Co, Fallbrook, California, US).
3. After sandblasting, samples were cleaned using water and air stream to remove any remnants of alumina particles on the surfaces.

2.4.2. Oxygen etching by plasma technology; group (3)

Constructed zirconia samples of group (3) were etched using plasma technology. For this purpose, oxygen gas was used as the working gas in the plasma focus system, Fig. 1, and the condenser bank was charged to 12 kV. The substrate holder that holds the samples was incorporated in the vacuum chamber facing the rim of the anode. The capacitor bank potential was transformed to the plasma focus tube through the air spark gap, in this state the plasma focus was formed after that it broke into ions and electron beams. The energetic oxygen ion beam took the shape of fountain and spread upwards to bombard the facing samples. To enhance the treatment, the process was repeated 15 times.

2.4.3. Silica coating by plasma technology; group (4)

Constructed zirconia samples of group (4) were silica coated using plasma technology. Argon gas was used as working gas; an

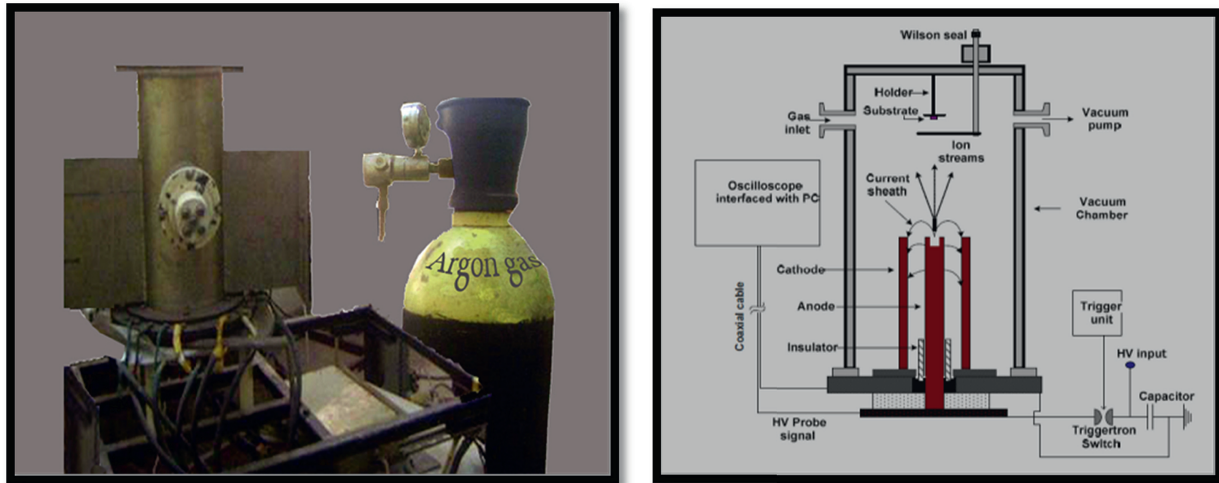


Fig. 1. The plasma focus system and its schematic diagram.

energetic argon ion beam obtained from an ion source was used to deposit thin film of silica on the zirconia substrate. The deposited material originated from a second ion gun that was used to sputter silica onto the substrate. The ion bombardment both; cleans the substrate by sputtering and changes the chemical bonding at the interface, prior to deposition.

2.5. Samples subgrouping

Samples of each group ($n = 25$) were subdivided into 5 sub-groups according to the testing procedure employed.

2.6. Subgroup (A)

10 samples of each group were prepared for shear bond test as follows:

Testing procedures:

1. Shear bond testing:

a. Acrylic block construction:

Previously treated Y-TZP samples were embedded in acrylic resin block (*Acrostone, Industrial area El-Salam City, Egypt*). A specially designed acrylic block former of 10 mm length and 20 mm diameter was painted with Vaseline. Self-cure acrylic resin was mixed according to manufacturer's directions in a glass container and poured into the block former. After complete curing, the acrylic resin block was removed and inspected for any deficiencies which were corrected by addition.

b. Construction of composite discs:

Split metal ring with three central holes of 5 mm internal diameter and 3 mm thickness each, was filled with composite resin (*Te-Econom Plus, Ivoclar Vivadent, Schaan, Liechtenstein*) to fabricate composite resin discs, which were light polymerized (*XL-3000, Curing Light 3M™ ESPE™, Australia*) for 40 s on each side for a total of 120 s.

c. Cementation procedures:

Composite resin discs were cemented to previously treated zirconia discs according to the manufacturer procedure:

1. A mix of silane coupling agent and metal/zirconia primer was applied to the surface of Y-TZP sample with a micro-brush.
2. The material was allowed to react for about 3–5 min, and then it was exposed to a strong stream of air.
3. The base and catalyst pastes of Multilink (*3M ESPE, St Paul, MN, USA*) cement were precisely dispensed in equal amounts, auto-mixed together through the disposable automix tip and applied to the surface of the Y-TZP ceramic disc.
4. Composite disc was then bonded to surface of zirconia ceramic disc; the bonding assembly was kept under a static load of 3 kg using a specially constructed load applicator. Which consists of 5 parts: Base portion with internal central tube of 2 cm in diameter into which the acrylic resin block was placed, two vertical fixed arms connected to the base on each side, one horizontal arm fixed to the two vertical arms, a central movable vertical arm, that has a rounded table in its upper end to accommodate for the load and its lower end is 5 mm diameter to ensure even load distribution, and a load of 5 kg
5. Excess luting cement was removed using disposable micro-brush, then all the margins of the bonding area was covered by a viscous gel (*Oxyguard, Kuraray Medical Inc., 1621 SakaZu, Kurashiki. Okayama 710-8622, Japan*) used to block the oxygen and light cured for 20 s. After complete polymerization the samples were washed with air-water spray.

d. Thermocycling:

The samples were stored in humidior at 37 °C for 48 h then subjected to thermocycling in thermocycling device (*Espec Corp, United States*) for 3000 cycles. Each cycle consisted of 1 min in 5 °C cold bath and 1 min in 55 °C hot bath with a dwell time of 30 s, and then the samples were air- dried.

e. Shear bond strength assessment:

1. A circular interface shear test was designed to evaluate the bond strength between zirconia samples and composite resin discs.
2. Each sample (acrylic embedded zirconia with its bonded composite disc) was secured to the lower fixed compartment of testing machine by tightening screws
3. Shearing test was conducted by compressive mode of load applied at ceramic- composite resin interface using a mono-bevelled chisel shaped metallic rod attached to the upper

movable compartment of testing machine) *Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK*) traveling at cross-head speed of 0.5 mm/min.

4. The load required for debonding was recorded in Newton using computer software (*Nexygen-MT-4.6; Lloyd Instrument*).
5. The load at failure was divided by interfacial bonding area to express the bond strength in MPa: $\tau = P/\pi r^2$, where; τ = shear bond strength (MPa), P = load at failure (N), $\pi = 3.14$, r = radius of composite disc (mm).
6. The data were collected, tabulated and statistically analyzed.

f. Assessment of mode of failure:

The interfaces of the debonded samples were examined with a stereomicroscope microscope (*Olympus SZ-PT-Japan*) at ($X = 60$) magnification to determine the failure pattern, which was assigned to belong to one of the following types:

- Cohesive failure within resin cement or composite resin,
- Adhesive at ceramic/cement interface or
- Mixed adhesive/cohesive modes.

Representative samples for each failure pattern were further examined using a scanning electron microscope (SEM) with an acceleration voltage of 20 kV and a working distance of 10 mm.

2. Scanning Electron Microscopic (SEM) analysis

In order to study the surface morphology of surface treated Y-TZP samples, 5 samples from each group were gold coated with a sputter coater (*K550X sputter coater, England*) then examined using SEM (*Quanta 250-FEG, FEI, Netherlands*) at a magnification X 1000.

3. X-Ray Diffraction (XRD) analysis:

X-ray diffraction (*Panalytical Empyrean, Holand*) was employed for the identification and quantitative determination of the various crystalline phases present in Y-TZP samples after various surface treatments with angular range 2θ : from 10° to 90° , at scan speed 19 s/step, using Cu K α radiation at 30 mA, 45 KV and measurement temperature was 25 °C.

4. Optical surface roughness measurements:

The evaluation of the surface roughness was carried out using profilometer (*ZYGO Maxim-GP 200*), which is a general purpose surface optical profiler that measures the microstructure and topography of surfaces in three dimensions.

3. Results

3.1. Results of shear-bond strength determination

3.1.1. Statistical analysis of shear bond strength

One-way ANOVA test was used for comparison between shear bond strength (MPa) after different surface treatments. There was no statistically significant difference between oxygen plasma treated and silica plasma coated treatments; both showed the statistically significantly highest mean shear bond strength values. Sandblasted group showed statistically significantly lower mean value. Control group showed the statistically significantly lowest mean shear bond strength (Table 1 and Fig. 2).

3.1.2. Failure modes

Three modes of failure were detected after the samples have

been subjected to shear bond test (Figs. 3–5). The prevalence of each type of failure in each group and the statistical analysis is shown in Table 2 (Fig. 6).

3.2. Results of scanning electron microscopic (SEM) examination

SEM analysis at 1000 \times magnification showed that the topographic pattern differed by different surface treatments of samples, Figs. 7–10. Untreated Y-TZP ceramic surface (group (1)) showed a series of parallel cuts after milling of the zirconia block and multiple-sized debris covering almost all the surface with slight roughness and shallow porosities, Fig. 7.

In samples sandblasted by 110 μm Al_2O_3 (group (2)) impurities were removed, surface roughness and irregularities were increased showing grooves and sharp edges with extensive exposure of zirconia granules and wider inter-granular spaces, Fig. 8.

Samples roughened by oxygen gas using plasma (group (3)), showed a series of parallel cuts after milling as untreated samples, with more roughness, irregularities, and both irregular and rounded pits and porosities, Fig. 9.

Silica coated samples using plasma technology show atypical honeycomb pattern and clusters of silica particles covered loosely the surface, porosities, and almost all the surface showed flaws and micro-cracks, Fig. 10.

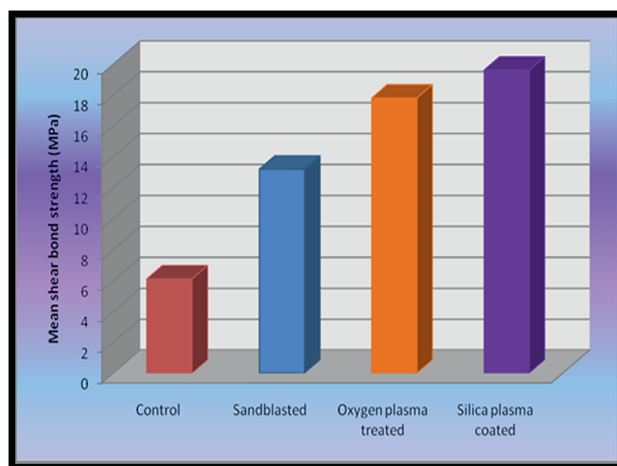


Fig. 2. Bar chart representing mean values for comparison between shear bond strength (MPa) of different groups.

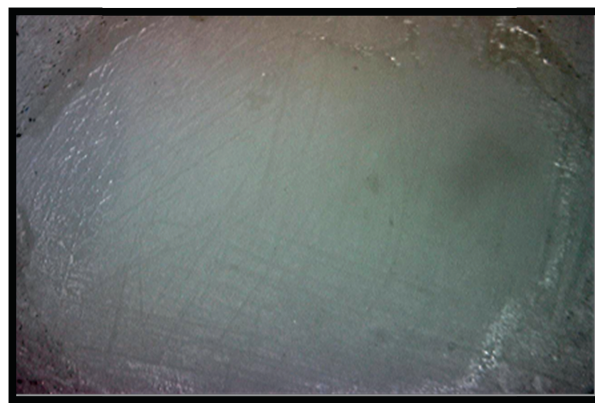


Fig. 3. Stereomicroscopic image of the zirconia fractured sample demonstrating an adhesive failure mode ($X = 60$).

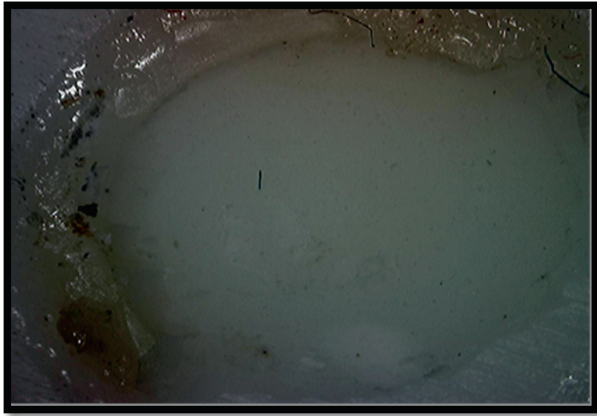


Fig. 4. Stereomicroscopic image of the zirconia fractured sample demonstrating a cohesive failure mode (X = 60).

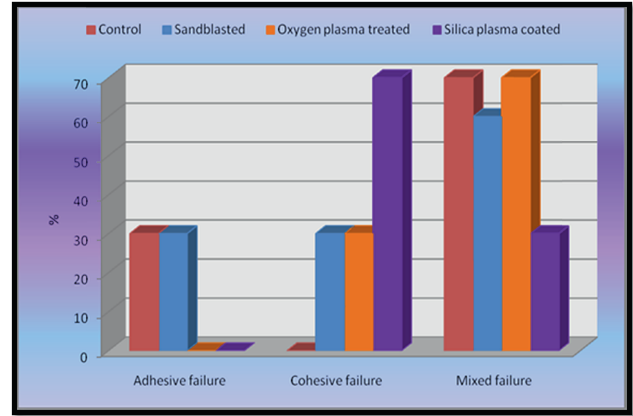


Fig. 6. Bar chart representing prevalence of failure modes after different surface treatments.



Fig. 5. Stereomicroscopic image of the zirconia fractured sample demonstrating combined adhesive/cohesive failure mode (X = 60).

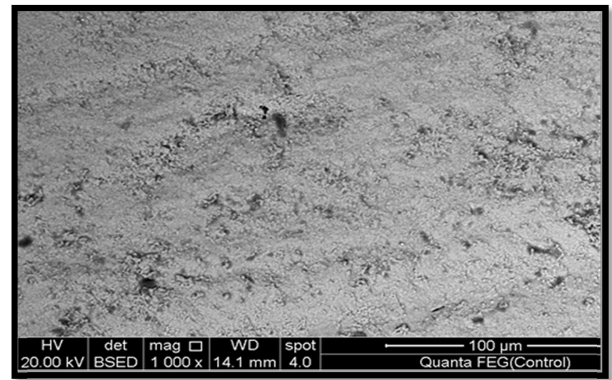


Fig. 7. SEM (1000x) image of untreated Y-TZP sample.

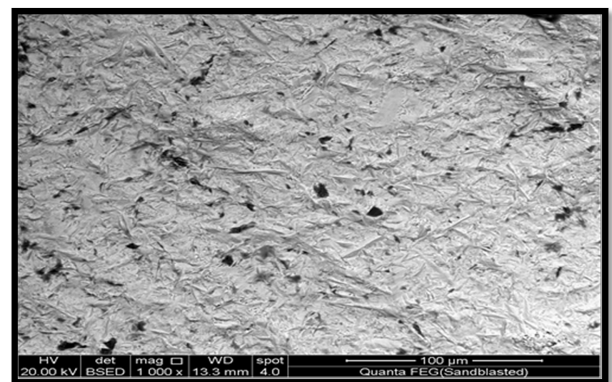


Fig. 8. SEM (1000x) image of sandblasted Y-TZP sample.

Table 1
Mean, standard deviation (SD) values of shear bond strength (MPa) measurements.

Surface treatment	Mean	SD	P-value
Control	6.1 ^c	2	<0.001*
Sandblasted	13.2 ^b	2.5	
Oxygen plasma treated	17.8 ^a	2.1	
Silica plasma coated	19.6 ^a	2.6	

Significant at $P \leq 0.05$. Different letters are statistically significantly different according to Tukey's test.

Table 2
Frequencies (N), percentages (%) and results of Chi-square test for comparison between failure modes after different surface treatments.

Surface treatment	Control		Sandblasted		Oxygen plasma treated		Silica plasma coated		P-value
	N	%	N	%	N	%	N	%	
Adhesive	3	30	3	30	0	0	0	0	0.014
Cohesive	0	0	3	30	3	30	7	70	
Mixed	7	70	4	40	7	70	3	30	

*: Significant at $P \leq 0.05$.

3.3. X-ray diffraction analysis

The phases and mineralogical composition of samples after different surface treatments are represented in Tables 3–6. Figs. 11–14 show their XRD analysis.

3.4. Statistical analysis of X-Ray diffraction

Zirconium oxide (Tetragonal phase): Control group showed the statistically significantly highest mean percentage. Oxygen plasma

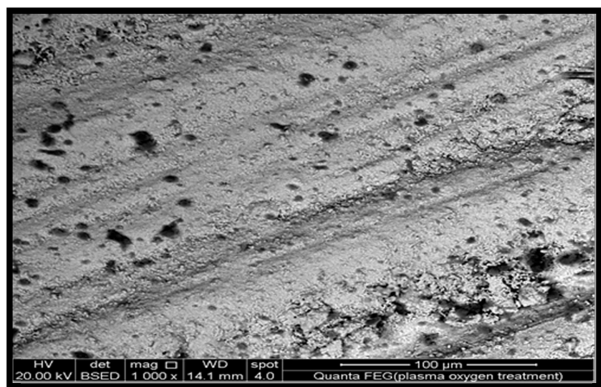


Fig. 9. SEM (1000×) image of Y-TZP sample after oxygen plasma treatment.

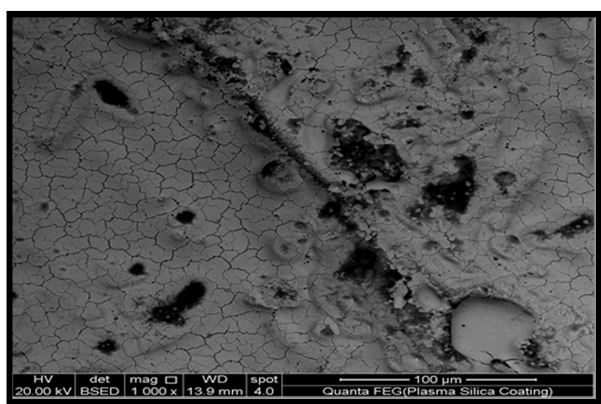


Fig. 10. SEM (1000×) image of Y-TZP sample after silica plasma coating.

Table 3
Phases and mineralogical composition of untreated Y-TZP ceramic samples.

Crystalline chemical formula	Compound name	Crystal system (phase)	%
O ₂ Zr ₁	Zirconium oxide	Tetragonal	100%

Table 4
Phases and mineralogical composition of Y-TZP ceramic samples after sandblasting.

Crystalline chemical formula	Compound name	Crystal system (phase)	%
O _{1.99} Zr ₁	Zirconium Oxide	Tetragonal	29%
H ₁ Al ₁ O ₂	Boehmite	Orthorhombic	23%
O ₂ Zr ₁	Zirconium Oxide	Anorthic	48%

Table 5
Phases and mineralogical composition of Y-TZP ceramic samples after O₂- plasma treatment.

Crystalline chemical formula	Compound name	Crystal system (phase)	%
O ₂ Zr ₁	Zirconium Oxide	Tetragonal	81.2%
O ₂ Zr ₁	Zirconium Dioxide	Monoclinic	18.8%

Table 6
Phases and mineralogical composition of Y-TZP ceramic samples after silica plasma coating.

Crystalline chemical formula	Compound name	Crystal system (phase)	%
O ₂ Zr ₁	Zirconium Oxide	Tetragonal	63.7%
O ₂ Zr ₁	Zirconium Oxide	Monoclinic	36.3%

treated showed statistically significantly lower mean percentage followed by silica plasma coated. Sandblasted group showed the statistically significantly lowest mean percentage.

Zirconium oxide (Monoclinic phase): Silica plasma coated group showed the statistically significantly highest mean percentage. Oxygen plasma treated group showed statistically significantly lower mean value. There was no statistically significant difference between control and sandblasted groups; both showed no presence of Zirconium oxide (Monoclinic phase).

Table 7 shows the statistical analysis of presence of different phases in tested groups.

3.5. 3D- optical surface roughness determination

3.5.1. Statistical analysis of surface roughness (Ra)

There was no statistically significant difference between mean (Ra) in oxygen plasma treated and silica plasma treated groups; both showed the statistically significantly highest mean (Ra) values. Sandblasted group showed statistically significantly lower mean value. Control group showed the statistically significantly lowest mean (Ra) value, Table 8, Fig. 16. 3D optical images are presented in Figs. 17–20.

4. Discussion

Y-TZP ceramics have superior strength, toughness, fatigue resistance and potentially, enhanced long-term viability than other ceramics [28,29]. MAD/MAM systems bear the advantages of reduced cost, less sophistication, easy and fast technique compared to other types of milling machines [30,31]. Samples were milled from “green” pre-sintered zirconia blocks at a larger dimension to compensate for 20%–25% shrinkage during the sintering stage [14,32].

High crystalline content of zirconia ceramics renders them

resistant to acid etching [33,34] therefore, alternative surface treatment techniques for long-term durable bonding are required [35–37]; such as sandblasting [38–43], plasma etching and silica ceramic coating [44] used in the present study.

As the material and fabrication of test discs may have an influence on bond strength values to ceramic, composite resin discs

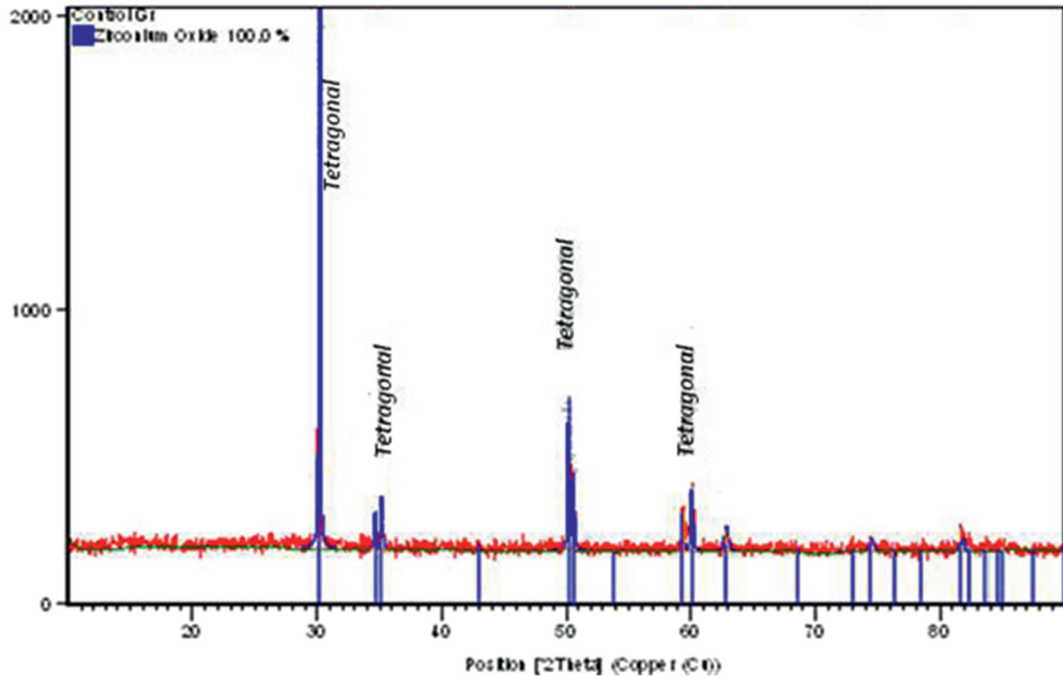


Fig. 11. XRD analysis of untreated Y-TZP ceramic samples.

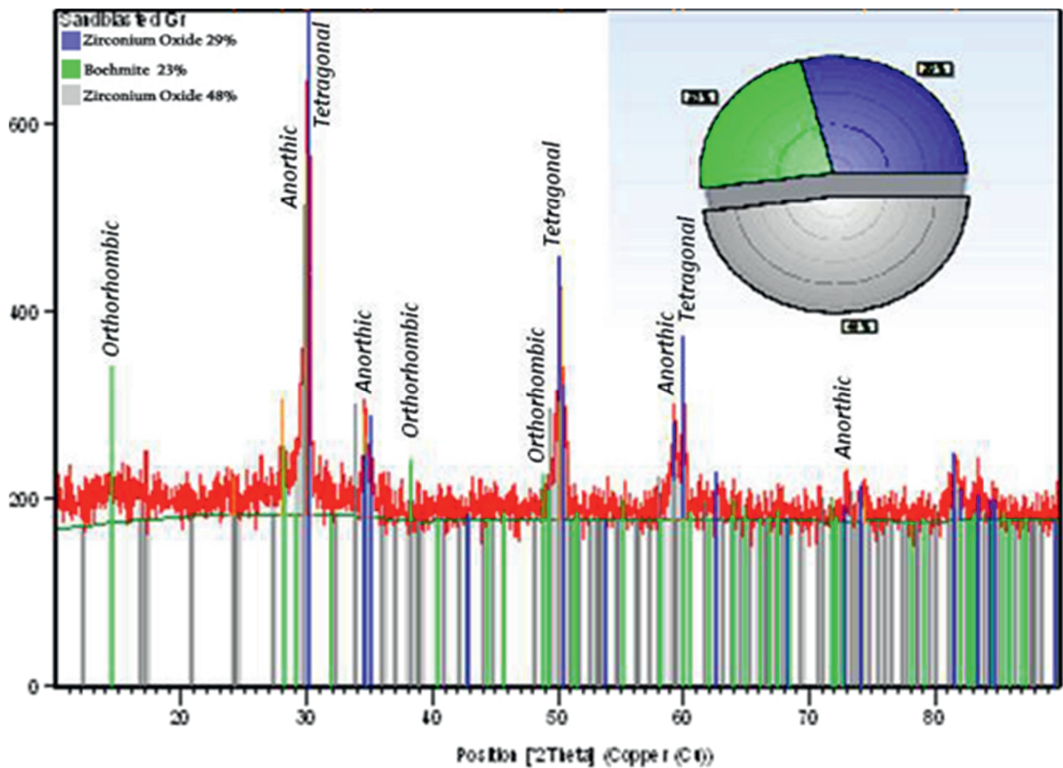


Fig. 12. XRD analysis of Y-TZP ceramic after sandblasting.

were cemented in this study to zirconia samples as most investigations [45]. This will provide a uniform rather than heterogeneous structure of tooth enamel and dentin, allowing for more precise interpretation of bond strength values [40]. Also the present study aimed at evaluating the bond strength at cement/ceramic interface only after variable surface treatments of YZ-zirconia.

Although there is an increase in clinical use of zirconia ceramics due to improved mechanical properties, further evidence regarding the adhesive cementation of Y-TZP restorations is necessary for the clinical success and the long-term performance of such restorations through establishing the most reliable bonding technique [46]. Bond strengths are influenced by several factors one of which is the

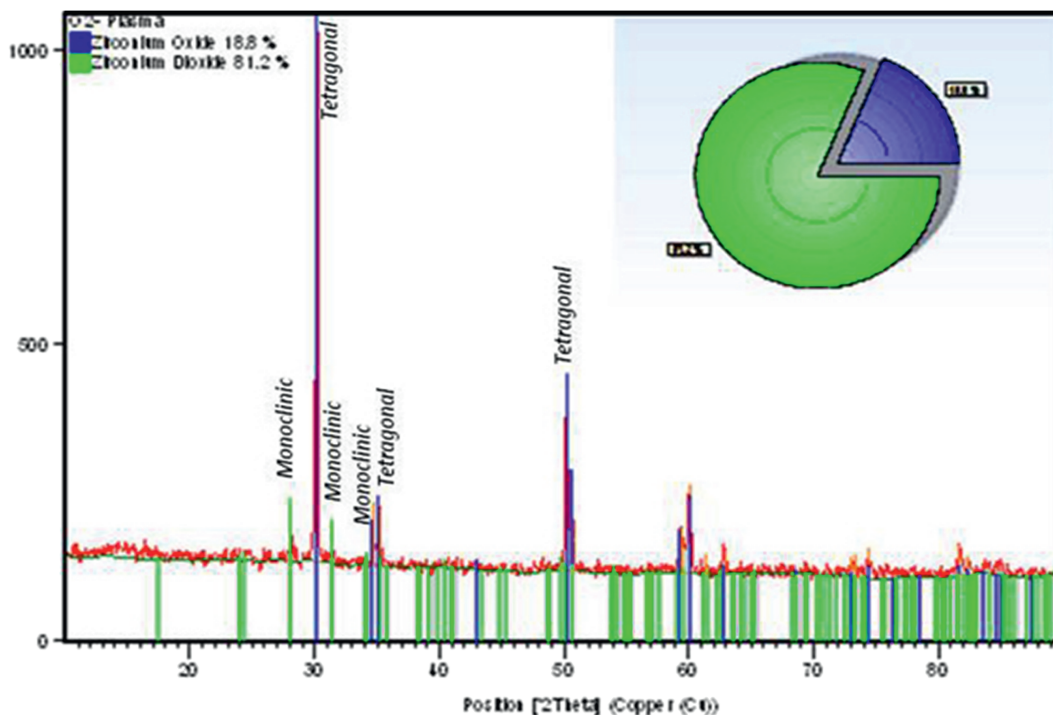


Fig. 13. XRD analysis of Y-TZP ceramic after oxygen plasma treatment.

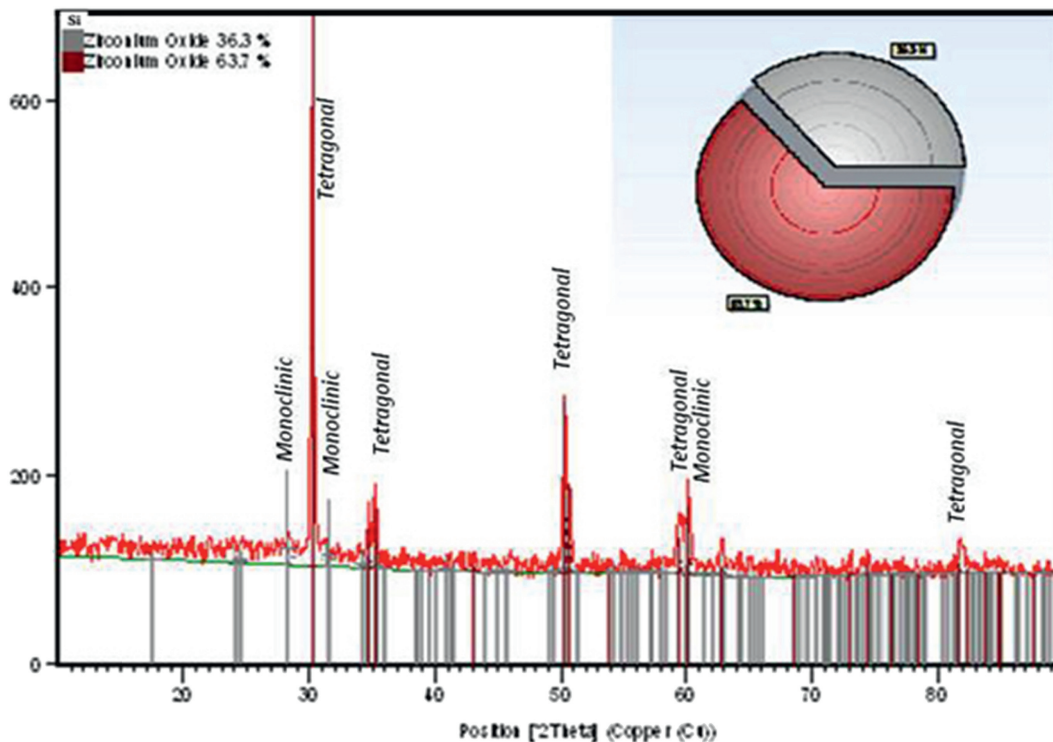


Fig. 14. XRD analysis of Y-TZP ceramic after silica plasma coating.

luting cement type [47,48]. Resin cementation seems to be the favorable choice for cementing zirconia restorations [46,47].

Multilink Automix resin cement was used in this study. It contains dimethacrylate, HEMA and silica fillers which provide the improved mechanical properties of the cement represented by high

flexural strength (70 MPa) according to the manufacturers. These improved mechanical properties could be responsible for high bond strength results of Multilink Automix [39]. There is some evidence that improved adhesive bonding to Y-TZP ceramics might be achieved using materials with a chemical affinity for metal oxides

Table 7

Mean, standard deviation (SD) values and results of one-way ANOVA and Tukey's tests for comparison between XRD results after different surface treatments.

Surface treatment	Control		Sand-blasted		Oxygen plasma treated		Silica plasma coated		P-value
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
Zirconium oxide tetragonal phase	100 ^a	0	29 ^d	1.5	81.2 ^b	2.6	63.7 ^c	1.5	<0.001*
Boehmite	0 ^b	0	23 ^a	1.1	0 ^b	0	0 ^b	0	<0.001*
Zirconium oxide Anorthic phase	0 ^b	0	48 ^a	2.1	0 ^b	0	0 ^b	0	<0.001*
Zirconium oxide monoclinic phase	0 ^c	0	0 ^c	0	18.8 ^b	1.7	36.3 ^a	2.1	<0.001*

*: Significant at $P \leq 0.05$, Different letters in the same row are statistically significantly different according to Tukey's test.

Table 8

Mean, standard deviation (SD) values and results of one-way ANOVA test for comparison between surface roughness (Ra) after different surface treatments.

Surface treatment	Mean	SD	P-value
Control	0.74 ^c	0.12	0.005*
Sandblasted	1.26 ^b	0.20	
Oxygen plasma treated	1.76 ^a	0.15	
Silica plasma coated	1.56 ^a	0.19	

* Significant at $P \leq 0.05$, Different letters are statistically significantly different according to Tukey's test.

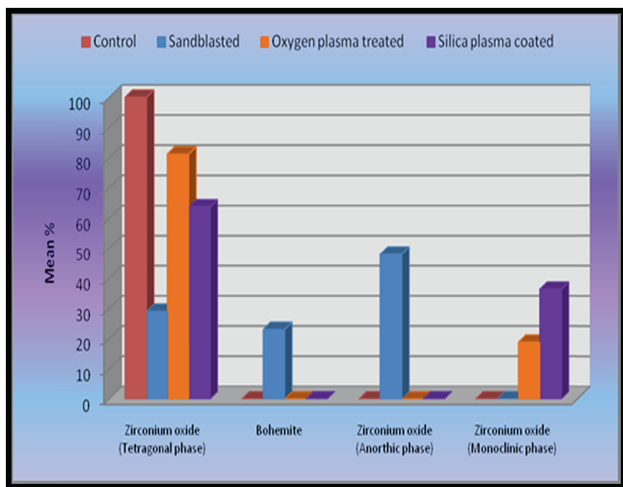


Fig. 15. Bar chart representing mean % of different compounds after different surface treatments.

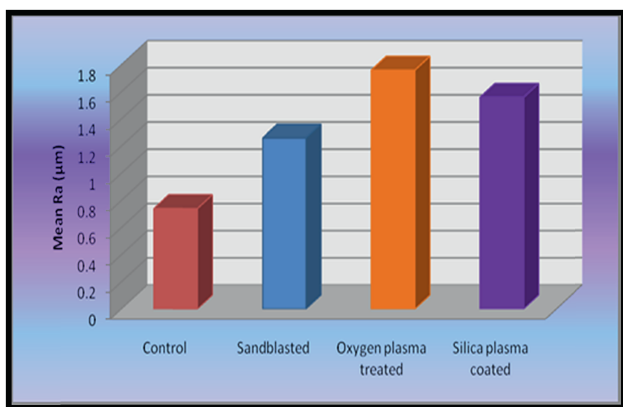


Fig. 16. Bar chart representing mean values for comparison between surface roughness (µm) after different surface treatments.

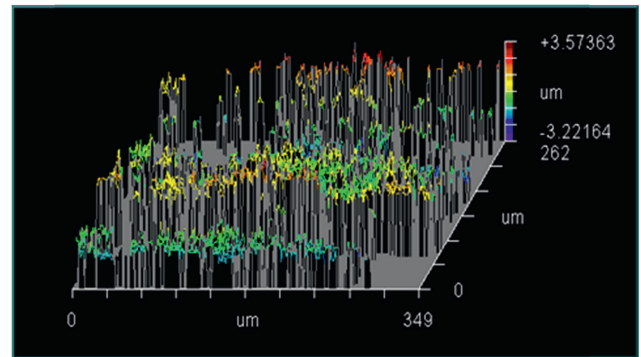


Fig. 17. 3D-optical (Ra) image of untreated Y-TZP ceramic.

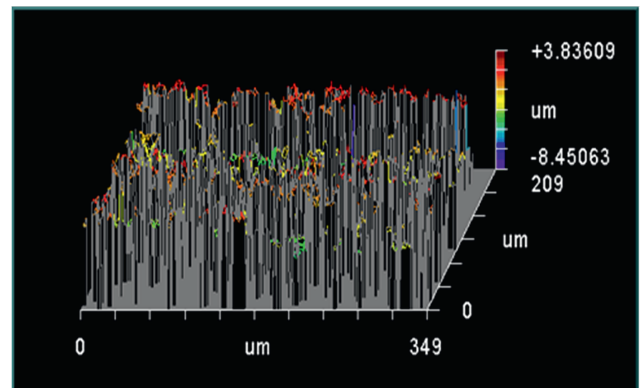


Fig. 18. 3D-optical (Ra) image of Y-TZP ceramic after sandblasting.

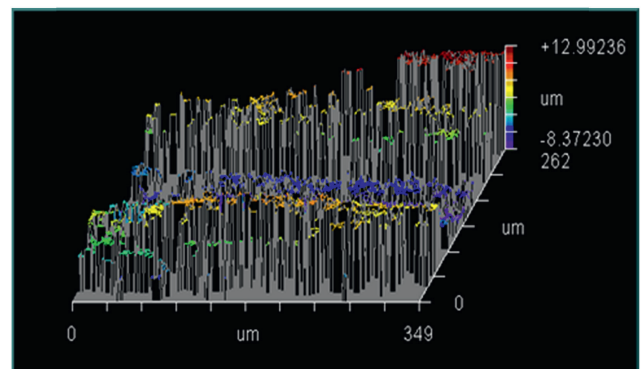


Fig. 19. 3D-optical (Ra) image of Y-TZP ceramic after oxygen plasma treatment.

[22,49–51]. Phosphate ester monomers, such as MDP (10-methacryloyloxy- decyl-dihydrogenphosphate), chemically react

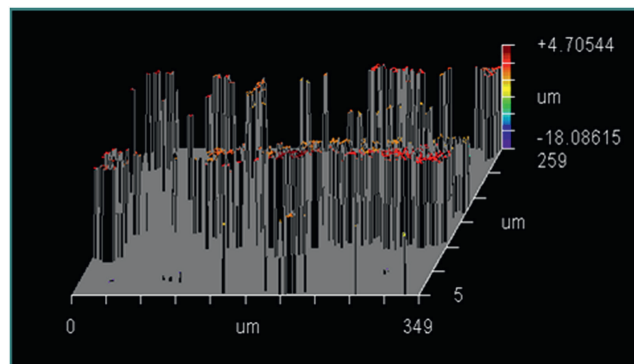


Fig. 20. 3D-optical (Ra) image of Y-TZP ceramic after silica plasma coating.

with zirconium dioxide, promoting a water-resistant bond to densely sintered zirconia ceramic [22]. So Metal/Zirconia Primer which contains MDP and other monomers, including VBATDT (6-[4-vinylbenzyl-n-propyl]amino-1,3,5-triazine-2,4-dithione), MEPS (thiophosphoric methacrylate) and MTU-6 (6-methacryloyloxyhexyl-2-thiouracil-5-carboxylate) [52,53] was also used.

According to Heikkinen [54], and Blatz et al., [55] silane coupling agent in the present study was mixed together with zirconia primer. Monobond Plus not only act as hybrid inorganic-organic bifunctional molecules that copolymerize with the organic matrix of the resin cement [56], but also improve the surface energy and wettability of zirconia to resin cements [57].

Thermocycling was performed to simulate thermal changes that occur in the mouth and act as a fastened aging process [37,42,51,58]. Samples were subjected to 3000 thermal cycles between 5 and 55°C. It is considered as an important factor that has been shown to decrease the bond strength in in-vitro studies [55,59].

Shear bond strength test is one of the most widely used for evaluation of adhesion in dentistry [43,46,49,59–62], because of being easy, fast and reflects the clinical situation. The stresses applied during the shear test were directed mainly at the zirconia/cement interface resulting in a relatively uniform distribution of inter-facial stresses because non-uniform distribution may cause mainly cohesive failure within the cement or the ceramic, which may lead to erroneous conclusions [23].

In the present study and in order to improve the bond strength between composite resin cement and zirconia ceramic surface, not only chemical retention was performed through using a mix of silane agent and metal/zirconia primer, but also different surface treatments of zirconia were performed to provide micromechanical retention [39,63, 64]. The mean values of bond strength of resin cement to Y-TZP zirconia ceramic presented in this study varied between 6.5 and 19.6 MPa depending on the surface treatment method applied. It was noticed that untreated, control (group (1)) samples showed the statistically significantly lowest mean shear bond strength, (6.5 MPa). These data were also supported by SEM evaluation (Fig. 7) which showed that the control group samples have slight roughness produced during milling procedures caused by diamond burs. This finding concurs with Blatz et al., [59] Al Hussaini and Wazzan [64] and Hummel and kern [65]. Because such slight roughness was not enough to produce a retentive surface in comparison with other surface treatments (groups (2), (3) & (4)), and 3D-optical profilometer control group samples recorded the lowest mean values of irregularities; 0.74 μm .

The shear bond strengths of sandblasted samples (group (2)) in the present study showed statistically significantly higher mean

values than those of control group, (13.7 MPa). Sandblasting was recommended as a preferred surface treatment method for densely sintered oxide ceramics by Nothdurft et al., [39] Cavalcanti et al., [63] and Zhu et al. [67]. Moreover, in this study deposition distance was controlled using a specially constructed device as recommended by Ozcan et al. [66].

Sandblasting was identified as a key-factor in establishing a durable bond between the luting agent and the ceramic, when combined with 10-Methacryloyloxyde-cyl dihydrogen phosphate (MDP monomer), either contained in the adhesive primer (as in the present study) or in the cement itself [59,68,69]. Oyagüe et al. [42], and Blatz et al. [55], assumed that Al_2O_3 abrasive particles removed any organic contaminants, produced an activated micro-roughened zirconia surface, increased the bonding area, modifying the surface energy and wettability [68], so they improve the bond strength by allowing for micromechanical interlocking of the resin cement [70].

SEM evaluation showed that, Al_2O_3 sandblasting produced an increased roughness and irregularities with grooves and sharp edges [71,72] (Fig. 8) these are considered to be important for the interlocking of the composite resin cement to Y-TZP ceramic. Moreover, 3D-optical profilometer analysis of the sandblasted group (Table 8, Fig. 18) revealed higher mean value of irregularities 1.26 μm , compared to control group. Both silica plasma coated and oxygen plasma treated groups showed the statistically significantly highest mean shear bond strength values. Although there was no statistically significant difference between them, silica-plasma coated group in this study recorded higher mean value of shear bond strength (19.6 MPa), followed by oxygen plasma-treated group (18.1 MPa).

Plasma deposition techniques could change the surface properties by attaching a film to the surface of the material [73], in a fast process that can be performed at low temperatures. Moreover the thickness and chemical composition of the film can be controlled [74,75] through the sputtered material from the target, the gas and the plasma source used [73,76].

In the present study silica deposition process using argon gas was performed, where Si-O bonds were available on the entire Y-TZP surface, promoting the chemical adhesion achieved by the silane coupling agent [42,77]. These results are consistent with those of Derand et al. [40], who showed that treating zirconia surface with plasma spraying increases shear bond strength to the resin cement, compared to untreated group. They are also consistent with those of Zhu et al. [67], who found that silica coating/salinization was most effective in improving the bond strengths of the resin cements to zirconia, compared with Al_2O_3 sandblasting group due to the chemical bond formed via the silica layer on ceramic surface, silane coupling agent, and resin cement.

In contrast, Kern and Wegner [22], and Blatz et al. [59], concluded that air-abrasion combined with MDP containing resin composite provides superior long-term shear bond strength than silica-coated zirconia bonded to Bis-GMA resin cement. SEM evaluation of Si-coated plasma samples in this study (Fig. 10) support the shear results, where a typical honeycomb pattern and clusters of silica particles covered loosely the surface, creating chemically reactive islets on the surface of the samples and could thus have chemically modified the surface of zirconia to enable a better reaction with the primers. Microcracks, some roughness and porosities, were also supported by higher mean of irregularities, 1.6 μm , as indicated by 3-D profilometer analysis (Table 8, Fig. 20).

These results are in agreement with those of Della Bona et al., [70] who showed that silica coating increase the surface roughness of zirconia. On the other hand Ozcan et al. [66], reported that surface roughness (Ra) of zirconia samples was the highest with 50 mm Al_2O_3 sandblasting than that of silica coated samples, also SEM images ($\times 500$) showed rougher surface of sandblasted group

compared to silica coated one.

Another application of plasma that was used in this study is the treatment of Y-TZP samples with oxygen gas, which allow physical etching that could improve the micro-roughness, increasing surface oxidation; allowing for modification of surface-bound chemical groups, and provide hydrophilic surfaces; improving wettability as well as the affinity toward the resin cement [26,78,79]. These results (Table 1) concur with those of Piascik et al. [80], who reported a more chemically reactive surface of Y-TZP samples when plasma treated by oxy-fluoride gas, thus allowing for covalent bonding between zirconia surface and resin cement, resulting in higher bond strength compared with control group. On the other hand plasma treated zirconia samples were found by Piascik et al. [74], to display the highest shear bond strengths and improved chemical bonding than control, polished, sandblasted and even Si-coated groups. SEM results (Fig. 9) showed roughness, irregularities, and both irregular and rounded pits and porosities, where the primer and adhesive resin can infiltrate and interlock, this was compatible with the highest mean of irregularities 1.8 μm showed by profilometer (Table 8, Fig. 19).

X-ray diffractometer which was used to identify crystalline phases in several studies [66,81–83] was used in this study to compare crystalline phases present in zirconia after different surface treatments as well as in control group. Although transformation of pure zirconia from the tetragonal to the monoclinic phase takes place during cooling after material sintering, in this study, the sintered control specimens did not have any monoclinic zirconia and only tetragonal phase predominated at 100% by volume (Table 3, Fig. 13). This was in accordance to Piconi and Maccauro [5], and Garvie et al. [84], who showed that Y-TZP zirconia, that is partially stabilized with 3 Mol% of yttrium oxide is stabilized in the more durable tetragonal phase even at room temperature and after sintering. In the present study zirconia blocks were milled in green, pre-sintered stage to avoid the negative effect of pre-cracks, flaws and subsequent phase transformation that was indicated in several studies [72,85,86] when grinding zirconia in fully sintered stage.

Since managing the sintering temperature and time is important, control samples were sintered according to the manufacturer's instructions from room temperature to 1500 °C using a rise time of 3 h and kept at 1500 °C for 2 h. Although Chevalier [87] demonstrated that the amount of the cubic phase in zirconia increases when the sintering temperature reaches 1500 °C for a long sintering time (5 h), but smaller zirconia grains about 0.4 μm size that was used in this study as described by manufacturer have a lower transformation rate. This was in agreement with Munoz-Saldana et al. [88], and Tsukuma et al. [89], who observed that the bigger the zirconia grain size; larger than 1 μm , the more prone the grains are for spontaneous t-m transformation. In this study, and in order to avoid t-m transformation in sandblasted group, manufacturer recommendations was carried out to apply low pressure about 29 PSI, for short time period only 10 s for sandblasting the Y-TZP zirconia samples. Although, x-ray diffraction (Table 7, Fig. 14) showed the absence of monoclinic phase in sandblasted zirconia samples, the appearance of new anorthic zirconia phase 29% by volume, in addition to tetragonal zirconia 48%, also supports the principle of zirconia phase transformation under stresses. Anorthic phase is also known as triclinic phase (with close similarity to monoclinic), is a crystal system with the lowest symmetry; where lattice vectors: a, b and c are non-equal, also α , β and γ angles are not equal. This was compatible with the results of Kosmac et al. [86], Hannink and Swain [90], and Swab [91], who demonstrated that the tetragonal zirconia is meta-stable and the surface t-m phase transformation can be initiated by surface grinding or sandblasting.

Cağlar and Yaniloglu [92] also found that sandblasting with

Al_2O_3 resulted in high surface roughness compared to Er Yag laser treated and control groups. Sandblasting had the potential to induce flaws in zirconia ceramic discs, with concomitant t-m transformation. Guazzato et al. [72], and Kosmac et al. [86], also indicated that sandblasting produced the most effective tetragonal to monoclinic phase change when compared to fine polishing, grinding with an abrasive wheel, or grinding using a diamond bur. In the present study, X-ray diffraction of sandblasted zirconia samples also showed the appearance of alumina particles (Boehmite) at 23% by volume. These results were consistent with Özcan and Vallittu [60], Tiller et al. [93], Tiller et al. [94], and Lorrente et al. [95], who suggested that when using particles of Al_2O_3 for sandblasting of zirconia, there are complex reactions on the substrate surface taking place, which consist of the separation and accumulation of certain elements at the substrate surface. Both silica coated and oxygen plasma treated groups in the present study (Tables 5 and 6, Figs. 15 and 16), showed t- m phase transformation. The effect of repeated shots of plasma ion beam that was charged at 12 KV and at 7 cm distance between the source and the zirconia surface, together with the particles bombardment of samples can explain the phase transformation due to creation of stresses and high temperatures.

This was compatible with many studies [86,90,91] which concluded that t-m phase transformation of zirconia can take place under the effect of stress or temperature changes. While oxygen-plasma treated group, shows predominant tetragonal phase 81.2%, and only 18.8% monoclinic phase in the form of zirconium dioxide; which supports the appearance of surface oxides, silica-coated group by plasma technology shows higher monoclinic phase at 36.3%. This is due to higher kinetic energy created by argon gas used in case of silica-coating as it has higher atomic mass (40), compared with less atomic mass of oxygen (16), in addition it was indicated by Giacobbe [96], that argon gas can cause more heat transfer at more rapid rate between the plasma jet and the object.

5. Conclusions

Within the limitations of this in vitro study the following conclusions were evident:-

1. Surface treatments of Y-TZP ceramic together with MDP primer (Metal/Zirconia primer) and silane-coupling agent (Monobond Plus) application improve the bond strength to resin cement.
2. Plasma-Silica coating and plasma-oxygen treatment, both are valuable methods that improve the bond strength of resin cement to Y-TZP ceramic.
3. Silica coating by plasma technology provides durable bond strength and can be a promising alternative pretreatment before silane application to enhance bonding with zirconia ceramic.
4. Plasma surface treatment for zirconia induce tetragonal-monoclinic phase transformation.

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