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EFFECT OF DIFFERENT SURFACE TREATMENT MODALITIES ON CRYSTAL STRUCTURE AND BIAXIAL FLEXURAL STRENGTH OF SUPER TRANSLUCENT ZIRCONIA

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ABSTRACT

Objectives: To study the outcome of different surface treatments on X ray diffraction analysis (XRD) and flexural strength of super translucent zirconia.

Materials and Methods: Forty identical discs were milled from presintered super translucent zirconia blank (VITA YZ ST, VITA Zahnfabrik, Germany). Specimens were classified into 4 groups according to the surface treatment used: Group C: no treatment (control group), Group S: discs were airborne particle, Group P: discs were treated with primer and Group SP: discs were airborne particle then, they were treated with primer. All specimens were subjected to XRD analysis and biaxial flexural strength test.

Results: The highest biaxial flexural strength recorded value was for the C group (462.11±17.33 MPa) while the lowest one was recorded for S group (310.33±7.78 MPa). Applying one way ANOVA and Tukey test revealed a statistically significant difference among all tested groups. The XRD analysis of specimens that were treated with different surface treatments revealed the evolution of monoclinic phase transformation in groups S and SP.

Conclusions: The airborne particle abrasion resulted in the evolution of tetragonal to monoclinic phase transformation on the zirconia surface which in turn negatively affected the flexural strength of super translucent zirconia.

KEYWORDS: Super translucent zirconia, Biaxial flexure strength, Airborne particle abrasion, Primer, XRD.

INTRODUCTION

In recent decades, the increased need for esthetic led to the replacement of metal-ceramic restorations with indirect metal-free ones. [1] Zirconia has gained an important role in these cases, because of its superior mechanical and biocompatible properties.[2] In the past, it was used only as frameworks for all-ceramic restorations. Lastly, with the development of translucent zirconia with its superior esthetic characteristics, allowed for the use of monolithic

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prosthesis with numerous advantages; elimination of chipping problems, favorable mechanical properties, manufacturing of thinner restorations resulting in a more conservative dental preparation and the possibility of manufacturing by CAD/CAM method. [3,4]

Strong bond strength between fixed restoration and luting cement is obligatory for long lasting restorations, manufacturers claimed that zirconia restorations can be effectively luted with either conventional or adhesive cements, [5] however, a few zirconia fixed dental prosthesis demonstrated decreased retention with their supporting abutments. [6] A strong, long-standing bond between adhesive resin and zirconia restoration is set up by micromechanical interlocking and chemical bond formation.[7] Achieving an efficient and stable bond with zirconia is a challenge, because zirconia is a polycrystalline material with restricted vitreous phase, neither etching with hydrofluoric acid nor silanization can accomplish strong bond between zirconia and resin. [8] So, different surface treatments are presented to form a strong long-lasting bond between zirconia and resin cements.

Air abrasion is utilized to obtain mechanical interlocking, clean the superficial layer, remove contaminations, increase surface irregularities, and alter the wettability and surface energy. [9]

Primers have a dominant role in adhesive procedures, particularly for zirconia-based ceramics.[10] Chemical surface pre-treatment of zirconia with MDP-containing primer resulted in a durable bond between resin and translucent zirconia.[11,12] The adhesive functional monomers are believed to make hydrogen bonds with the metal oxides at the interface of zirconia / resin, resulting in improved wettability.[13]

Since results are not usually notable, the combination of primers especially those containing MDP and airborne particle abrasion is liable to form good bond strength, particularly in long term. [14-17]

The zirconia strength could be affected by variable surface treatment modalities, like airborne particle abrasion, silica coating, etching with acids and mixing of any of these methods [18,19] Many studies revealed negative effects on zirconia strength as a result of using different surface treatments. [20-22]

Distinctive surface flaws were noticed after zirconia surface treatment with airborne particle abrasion, which is considered as areas of stress concentration resulting in probable crack initiation and propagation. [23,24] Otherwise, some studies showed an increase in zirconia strength with airborne particle abrasion because of the creation of compressive stresses resulted from the transformation of tetragonal to monoclinic phases on the zirconia surface. [5,25] Other studies considered this transformation to be responsible for deterioration of the zirconia mechanical properties. [26-29] So, the target of this study was to assess the effect of variable surface treatments on X ray diffraction analysis and flexural strength of super translucent zirconia. The null hypotheses to be tested were that application of different surface treatments on super translucent zirconia would affect its crystalline structure and its biaxial flexure strength.

MATERIALS AND METHODS

One acrylic resin disc (Pattern resin LS, GC America Inc. Alsip, IL 60803 USA) with a circumference of 12 mm and thickness of 1.2 mm was made using a special mold.[30] Scanning of the disc was done using an optical scanner (Ceramill map400, Amman Girrbach, Germany). Using the special software (Ceramill Mind design software), forty identical copies of the scanned disc were designed on the presintered super translucent zirconia blank (VITA YZ ST, VITA Zahnfabrik, Germany) and dry milled using 5-axis milling machine (Ceramill Coolstream, Amman Girrbach, Germany). Sintering, finishing, and glazing of the discs were done following the manufacturer's recommendations.

Specimens grouping and surface treatments

The discs were randomly divided into 4 groups (n=10) according to the surface treatment used; Group C: no treatment was used (control group), Group S: discs were airborne particle abraded with 50 µm Al_2O_3 at right angle to the surface at distance of 10 mm and 4 bars pressure for 20 seconds[5] (Renfert Gmbh, Germany), Group P: discs were treated with zirconia primer (Z-primer Plus, Bisco Inc, USA) by application of 2 uniform coats of primer which were dried with air spay for 5 seconds according to the manufacturer's recommendations. Group SP: discs were airborne particle abraded with 50 µm Al_2O_3 at right angle to the surface at distance of 10 mm and 4 bars pressure for 20 seconds then, they were treated with 2 uniform coats of zirconia primer which were dried with air spray for 5 seconds.

X-Ray Diffraction analysis (XRD)

Characterization of phase transformation of zirconia surface following the different surface treatments was performed by X-Ray Diffraction analysis (XRD). Three randomly selected specimens from each group were analyzed in a diractometer (X'Pert PRO, PANalytical co., Holland).

XRD patterns were collected using conventional 2θ–θ method and the grazing angle method (incident angle θ =1°and 2°) with Cu $K\alpha$ radiation at 40 kV voltage and 40 mA current. Diffract grams were obtained from 25°to 36° at a scan speed of 0.5°/min. Rietveld analysis of XRD patterns was performed with software (Bruker AXS, Karlsruhe, Germany) to quantify phase contents and lattice parameters.

The amount of monoclinic phase (Xm) was calculated using Gravie and Nicholson equation [31] as follows:

$$X\text{m} = \frac{\boldsymbol{Im}\,(111) + \boldsymbol{Im}\,(-111)}{\boldsymbol{Im}\,(111) + \boldsymbol{Im}\,(-111) + \boldsymbol{It}\,(111)}$$

Where ***Im (111)*** is the monoclinic peaks intensity at 2θ=28° and ***Im (-111)*** is the monoclinic peaks intensity at 2θ = 31° degrees, and ***It (101)*** is the tetragonal peaks intensity at 2θ =30°.

The volumetric fraction of monoclinic phase (Vm) was calculated using the following equation[32]:

$$V\text{m} = \frac{0.3111\,\boldsymbol{Xm}}{1 + 0.3111\,\boldsymbol{Xm}}$$

Biaxial flexural strength test:

All specimens were exposed to biaxial flexural strength test using piston-on-three balls technique (Figure 1), each specimen was positioned on the top of three steel balls (3.2 mm in diameter and 120° apart forming a tripod). Load was applied at right angle to the center of the upper surface of the specimen by a round tipped cylinder steel piston with a diameter of 1.6 mm at a crosshead speed of 0.5 mm/min. in a Universal Testing machine (Instron 3345, USA) using its special software (Bluehill Universal software, Instron, USA) till failure occurred. The flexural strength, in MPa, was calculated using the following equations according to the guidelines of ISO 6872, 1998 [33]:

$$S = -0.2387\, P\, (X-Y) / d^2$$
$$X = (1+ \nu)\, \ln (r_2 / r_3)^2 + [(1- \nu) /2]\, (r_2 / r_3)^2$$
$$Y = (1+\nu)\, [1+\ln (r_1/ r_3)^2] + (1- \nu)\, (r_1/ r_3)^2$$

Where ***S*** is the maximum tensile stress in MPa, ***P*** is the load at fracture (N), ***d*** is thickness of the specimens (1.2 mm), ***ν*** is the Poisson's ratio for zirconia (0.32), $_1$ is the supporting ball radius (3.2 mm), r_2 is the radius of the piston tip (1.6 mm), and r_3 is the specimen radius (12mm).

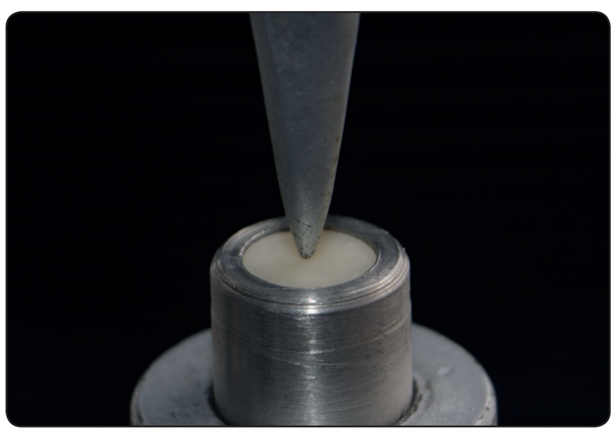

Fig. (1) Biaxial flexure strength test

The resultant data were collected, tabulated, and analyzed statistically.

RESULTS

The XRD analysis of the different surface treatments revealed the evolution of monoclinic phase transformation in groups S and SP (Figure 2) with volumetric fraction of monoclinic phase of 6 and 7% respectively, while no phase transformation was observed in the C and P groups.

The biaxial flexure strength values and their standard deviations for all tested groups are presented in table 1. The highest reported value was for the control group (C) (462.11±17.33 MPa) while the lowest one was reported for the airborne particle abraded group (S) (310.33±7.78 MPa).

Analysis of data was done in two steps; one way analysis of variance ANOVA was conducted for the comparison between all groups which revealed a statistically significant difference ($p < 0.05$) between all groups. Tukey test for multiple comparisons was also performed to differentiate between each group in relation to the other 3 groups and revealed statistically significant difference among groups.

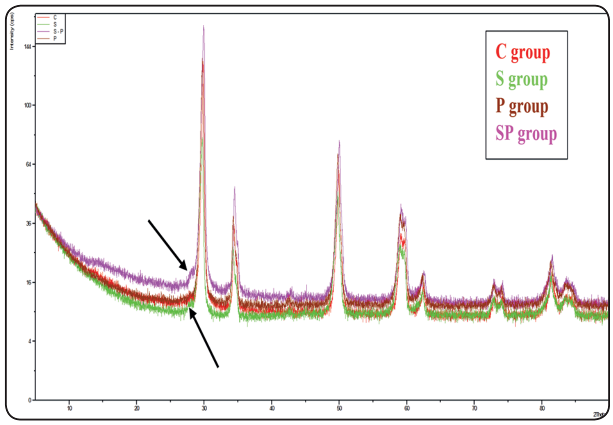

Fig. (2) XRD analysis of the different surface treatment methods. The arrows represent the monoclinic phase.

TABLE (1) Means and standard deviations in MPa for biaxial flexure strength of all groups (one way ANOVA and Tukey tests)

	C group	S group	P group	SP group	test of sig.
Mean±SD	462.11±17.33[a]	310.33±7.78[b]	443.34±11.73[c]	412.96±11.88[d]	F=286.43 P<0.001*

The same superscripts indicate insignificant different pairs of values.

DISCUSSION

The ceramic material used in this study was a super translucent zirconia where the manufacturer claims high strength combined with high esthetics. The surface treatments used were airborne particle abrasion and/or primer application as they were reported to perform better adhesion with the adhesive resin cements. [7,11]

XRD method was used to evaluate the surface structure changes and characterization in zirconia resulting from different surface treatment applications as it is considered a non-destructive reliable method. [34]

Biaxial flexural strength method was used as it is considered a simple and easy method to perform, their results are more precise and accurate when compared with uniaxial tests. [35]

Based on the results of the XRD analysis, the first hypothesis was accepted as the airborne particle abrasion resulted in phase transformation from tetragonal to monoclinic on the surface of the super translucent zirconia in groups S and SP, while the C and P groups showed no phase transformation. This tetragonal to monoclinic transformation may occur when an external mechanical stress is applied on the zirconia surface. [36] The results of this study were in accordance with those of Kosmac et al [23], Moon et al [5], Jain et al [24] although they used conventional and translucent zirconia and Inokoshia et al [37] who used high translucent zirconia.

Regarding the results of the biaxial flexure strength, the second hypothesis was also accepted as the different surface treatments affected the strength of super translucent zirconia. Based on the statistical analysis of the results, the control group showed the highest value of biaxial flexure strength while the airborne particle abraded group showed the lowest value, this may be attributed to the effect of air blasting which produce micro cracks on the zirconia surface and due to the transformation of tetragonal to monoclinic phase. These surface flaws affect the mechanical behavior of the zirconia negatively resulting in decreased flexure strength values. The results of this study were in accordance with those of Wang [38] although he used 50 and 120 µm aluminum oxide particles at 0.35 MPa pressure for 25 seconds at 20 mm distance. Also, Monaco [39] results agreed with this study, he used 30 µm silica-coated alumina particles and 50 and 120 µm aluminum oxide particles at 0.35 MPa pressure for 25 seconds at 15 mm distance. Yoshida et al [29] was also in agreement with this study, they used 50 µm Al_2O_3 at 0.1, 0.15, 0.2, and 0.3 MPa for 15 seconds.

However, there were disagreement with the results of Fonseca et al [40] as they used 30 and 110 µm silica-modified Al_2O_3, and 110 µm Al_2O_3 at 0.28 MPa for 20 seconds, Jain et al [24] who used 30 µm silica-coated alumina particles at 0.28 mm pressure and Yilmaz et al [41] who used 110 µm Al_2O_3 at 2 bars for 20 seconds and used four-point flexural strength test. All of them found that the airborne particle abrasion increased the flexural strength of zirconia which may be due to the difference in particle size, pressure applied, type of flexure strength test used, and type of zirconia used.

The SP group showed higher flexural strength than the S group although the surface characterization of both groups revealed tetragonal to monoclinic transformation also both groups were subjected to the same airborne particle abrasion procedure, this may be attributed to the application of the zirconia primer which may seal the initially formed micro cracks and chemically bond to the zirconia surface.

CONCLUSIONS

Within the restrictions of this *in-Vitro* study, it was deduced that:

1. The airborne particle abrasion of super translucent zirconia resulted in phase transformation from tetragonal to monoclinic.
2. Airborne particle abrasion has a negative effect the flexural strength of super translucent zirconia.
3. The application of zirconia primer on air abraded super translucent zirconia counter acts the negative effect of air abrasion on the biaxial flexural strength.

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