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Evaluating the Effects of Sandblasting on Micro-Shear Bond Strength and Flexural Strength of Zirconia

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ABSTRACT

Background and Objective: Various techniques have been introduced to improve bond strength of zirconia which may negatively affect flexural strength. This study aims to evaluate the effects of different sandblasting parameters on micro-shear bond strength (MSBS, Mpa) and biaxial flexural strength (BFS, Mpa) of Zirconia.

Methods: In this in vitro study, zirconia blocks were cut into 180 discs for MSBS and BFS tests (90 specimens in each group). For each test, they were divided into one control group and 8 experimental groups according to the type of sandblasting regimen (pressures of 4 or 6 bar, duration of 14 or 21 seconds, and alumina powder sizes of 50 or 110 µm, n=10). A universal testing machine was used to determine MSBS and BFS. X-ray diffraction (XRD) analysis was then performed.

Findings: According to the results, groups with 110 µm particles (4 bar- 14 s, 6 bar- 14 s, 4 bar- 21 s and 6 bar- 21 s with MSBS values of 34.43±5.99, 35.21±6.39, 27.17±3.95 and 28.66±3.92 Mpa, respectively) had significantly higher MSBS values compared to the control group ($p<0.001$, $p<0.001$, $p=0.034$, $p=0.005$, respectively). Groups with 110 µm- 21 s sandblasting regimen (with pressure of 4 and 6 bar with BFS values of 1031.69±90.00, 1062.56±91.29, 962.30±93.24, respectively) and those with 50 µm- 6 bar- 21 s sandblasting regimen had statistically significant lower BFS values compared to the control group ($p<0.001$). According to XRD analysis, groups with 110 µm powder size and 21 s sandblasting resulted in more monoclinic phase.

Conclusion: This study demonstrated that sandblasting with larger powder size and shorter duration could increase MSBS without any negative effects on BFS.

Keywords: *Flexural Strength, X-Ray Diffraction, Yttria-Stabilized Tetragonal Zirconia, Air Abrasion.*

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Introduction

In modern dentistry, there is an increasing demand for aesthetic and biocompatible restoration (1, 2). These materials must be aesthetic and also withstand the stresses created in restoration, following the occlusal function. Alumina and zirconia are new generations of biocompatible polycrystalline ceramics which have such aesthetic properties, and can also withstand masticatory stresses (3, 4).

Zirconia materials have polymorphic features and appear in three different forms based on temperature including monoclinic phase (m) that is stable at temperatures below 1170°C, tetragonal phase (t) which exists between 1170° and 2370°C, and cubic phase (c) that is stable beyond 2370°C (3). This ceramic has high fracture toughness due to the phenomenon of transformation toughening that is related to localized increase in unit-cell volume at crack tip resulting in compressive stresses, which inhibit crack propagation (3). Zirconia, with a partially stabilized tetragonal phase is the only other material that demonstrates the same stress-induced transformation toughening. Since tetragonal phase is only stable between 1170° and 2370°C, it should be stabilized to room temperature with oxides such as yttrium oxide (Y_2O_3, mostly 3% yttrium) or cerium oxide (CeO_2). This allows t→m transformation of zirconia under stresses during function (5, 6).

Despite their favorable strength, zirconia restorations are resistant to acid etching, hence their bonding is tougher than silica-based materials. This has been a concern to find out chemical or mechanical approaches to improve bond strength of zirconia to tooth structure (7). So far, several different surface treatments have been introduced to obtain higher bond strength of zirconia to resinous materials. These include surface abrasion or roughening (8, 9), tribochemical silica coating (10, 11), chlorosilane treatment (10, 12), selective infiltration-etching (SIE) technique (13, 14), and application of phosphate ester primers and phosphate modified resin cements (13, 15).

Although sandblasting with airborne alumina particles is one of the most commonly used techniques, there is a general consensus that roughening zirconia surface with 50-µm and 110-µm alumina powder improves bond strength due to micromechanical retention (7). Although it is beneficial for bonding, there is a premise that it can create structural defects reducing zirconia strength during function due to phase transformation (16). Therefore, the abraded yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) strength depends on the ability of the ceramic for phase transformation and the severity of the air-particle abrasion process, such as particle size, pressure, and duration of sandblasting.

According to controversial data achieved from previous studies, the present study was conducted to evaluate the effects of different parameters of sandblasting procedure on micro-shear bond strength (MSBS) and biaxial flexural strength (BFS) of zirconia-based restorations.

Methods

This in vitro study was approved by ethics committee of Mashhad University of Medical Sciences with the code IR.MUMS.SD.REC.1394.168. Cylindrical zirconia blocks (VITA ZIRCONIA YZ, Bad Säckingen, Germany) were cut into discs (1mm in thickness and 13mm in diameter, measured with electronic digital Vernier caliper [Louisware, ROSIMO Co., China]). Cutting procedure was accomplished by a computer numerical control (CNC) machine (NAJI60, Nemov Fanavaran Pars Mashhad, Iran).

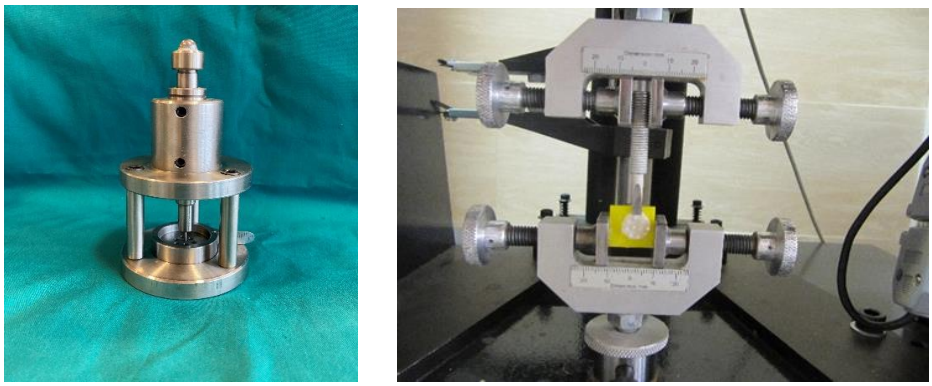
After sintering at 1400°C for 12 hours in a furnace (F2L-1720, Azar Kureh, Tehran, Iran), the specimens were mounted in epoxy resin (KER 828, KUMHO P&B CHEMICALS, Korea) and cleaned using an ultrasonic cleaner (JP-4820 Skymen Cleaning Equipment Shenzhen Co, China) in deionized water for 5min to eliminate any contamination. To determine flexural strength and MSBS, zirconia discs were divided into nine experimental groups according to the aforementioned parameters, and sandblasting was performed (Table 1). Sandblasting parameters was chosen according to previous studies (13, 16, 17). The sandblasting (Denta Part Dental Dual Function Sandblast, Tehran, Iran) procedure was undertaken from a distance of approximately 10mm and perpendicular to the surface of the disc.

To evaluate MSBS, zirconia discs (n=10) were decontaminated in deionized water using an ultrasonic cleaner. Tygon tubes (1 mm in diameter and 2 mm in height) were then placed on zirconia discs and were filled with self-adhesive cement (G-CEM LinkAce, Tokyo, Japan) and light cured for 20 seconds with a light curing unit (Bluephase Style, Ivoclar Vivadent, Liechtenstein). After that, the specimens were kept in deionized water for 24 hours and were loaded for MSBS testing by a universal testing machine (UTM, STM-20, SANTAM, Iran) at the crosshead speed of 1 mm/min (Figure 1-A).

A universal testing machine and a custom-made stainless-steel piston-on-three-ball apparatus (Figure 1-B) were employed to determine biaxial flexural strength. The apparatus has three hardened steel balls (with a diameter of 2.5-6.5 mm) for the support of test specimens. These balls are positioned on the 120˚ apart on a disk (with a diameter of 10-12 mm). The specimens were placed on these supports and the load was applied with a flat punch (with a diameter of 1.4±0.2 mm) at the center. The apparatus was then moved to the UTM and loaded at a crosshead speed of 1 mm/min until fracture occurred.

Table 1. Different parameters in sandblasting process

Group	Sandblasting particle size (µm)	Sandblasting Time (S)	Sandblasting pressure (bar)
1	50	14	4
2	50	14	6
3	50	21	4
4	50	21	6
5	110	14	4
6	110	14	6
7	110	21	4
8	110	21	6
9	No treatment	-	-



A **B**

Figure 1. A: MSBS testing unit, B: BFS testing unit

The maximum tensile stress (that is related to biaxial flexural strength) was calculated considering the recommended test standard (ASTM F394-78) equation: S= -0.2387P(X-Y)/d2
S, P, X, Y, and d were determined as follows:
S= maximum tensile stress (MPa), P=load at fracture (N), and d=specimen thickness (mm) at fracture origin.
X= (1+ν) in (B/C)2+((1-ν)/2)(B/C)2
Y= (1+ν)(1 + in(A/C)2)+ (1-ν)(A/C)2
ν= Poisson's ratio that was assumed 0.25 (it is assumed 0.25 if Poisson's ratio is unknown for that ceramic), A= radius of support circle (mm), B= radius of piston tip (mm), and C= radius of specimen (mm) (18).

One disc from each group was randomly selected and prepared for X-ray diffraction (XRD) analysis. The XRD (EXPLORER, GNR Analytical Instruments Group, Italy) pattern of the ceramic specimens was prepared to detect any crystalline structure changes due to sandblasting. Scanning was conducted in the angular range of $20<2\theta<80°$ at 0.01°/second scanning speed and an accumulation time of 2 seconds at each point using Copper (Cu) K-alpha (K-α) radiation. Phase identification and quantitative phase analysis through the Rietveld refinement were consequently performed by the X'Pert High Score (version 3) software. Data were analyzed using one-way analysis of variance (ANOVA), Tukey's post-hoc test, and Dunnett's test and $p<0.05$ was considered significant.

Results

Dunnett's test showed that groups 5, 6, 7, and 8 (sandblasted with 110 µm particles) with MSBS values of 34.43±5.99, 35.21±6.39, 27.17±3.95 and 28.66±3.92 Mpa respectively had statistically significant differences in MSBS compared to the control group ($p<0.001$, $p<0.001$, $p=0.034$, $p=0.005$, respectively) while there were no significant differences between other groups (Table 2). The results of one-way ANOVA revealed significant differences in BFS regarding different types of surface treatments ($p<0.001$). Groups 4, 7, and 8 (with flexural strength values of 1031.69±90.00, 1062.56±91.29, 962.30±93.24, respectively) showed significantly lower BFS compared to the control group ($p<0.001$, Table 3). However, groups 7 and 8 (sandblasted for 21 seconds) showed significantly higher bond strength, they also had significantly lower flexural strength. In groups sandblasted with 50µm particles, only group 4 (sandblasted with 6-bar pressure and for 21 seconds) had significantly lower flexural strength compared with the control group. According to the results obtained from XRD analysis (Figure 2), more monoclinic phases existed in all experimental groups compared to the control group. Groups sandblasted with 110 µm particles also had more monoclinic phases (in comparison to those with 50 µm particles and control groups).

Table 2. Comparison of Mean and Standard variation values of micro-shear bond strength between experimental groups and control group

	Group	Mean(MPa)±SD	p-value
1[a**]	4 bar - 14 s -50 µm	20.38±3.08	>0.999
2[a]	6 bar - 14 s - 50 µm	23.96±5.25	0.568
3[a]	4 bar - 21 s -50 µm	24.69±7.91	0.354
4[a]	6 bar - 21 s -50 µm	19.04±3.94	0.986
5	4 bar - 14 s -110 µm	34.43±5.99	<0.001
6	6 bar - 14 s -110 µm	35.21±6.39	<0.001
7	4 bar - 21 s -110 µm	27.17±3.95	0.034
8	6 bar -21 s -110 µm	28.66±3.92	0.005
9[a]	Control	20.59±3.84	

[**]**Distinct letters in the same column indicate significant differences between control and other groups in terms of micro-shear bond strength**

Table 3. Comparison of Mean and Standard variation values of flexural strength between experimental groups and control group

	Group	Mean(MPa)±SD	p-value
1[a**]	4 bar- 14 s- 50 µm	1175.96±60.37	0.460
2[a]	6 bar- 14 s- 50 µm	1135.81±93.29	0.057
3	4 bar- 21 s- 50 µm	1129.55±89.47	0.037
4	6 bar- 21 s- 50 µm	1031.69±90.00	<0.001
5[a]	4 bar- 14 s- 110 µm	1180.89±11.69	0.543
6[a]	6 bar- 14 s- 110 µm	1167.76±71.96	0.333
7	4 bar- 21 s- 110 µm	1062.56±91.29	<0.001
8	6 bar- 21 s- 110 µm	962.30±93.24	<0.001
9[a]	Control	1160.81±83.62	

[**]Distinct letters in the same column indicate significant differences between control and other groups in terms of flexural strength

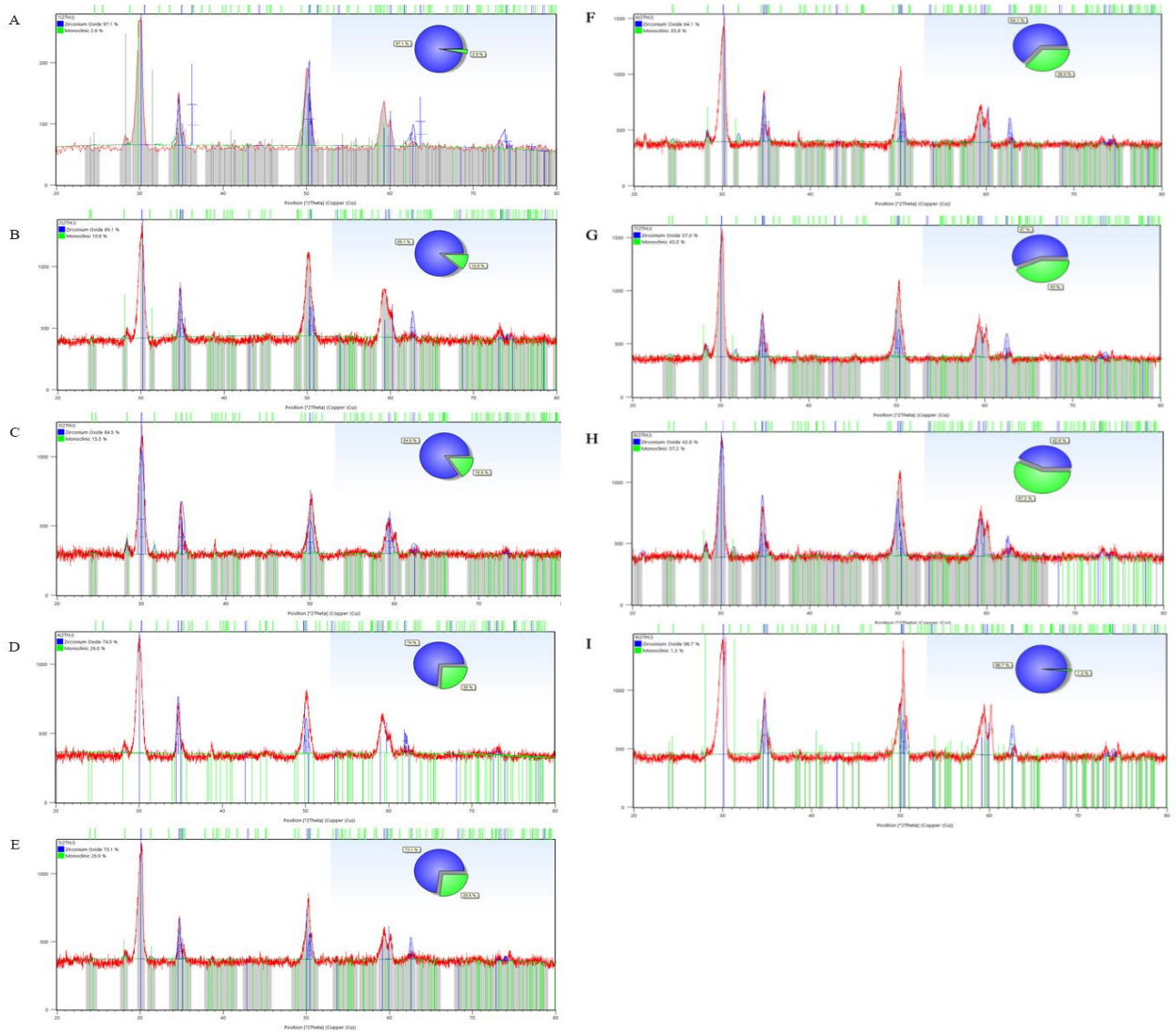


Figure 2. XRD (X-ray diffraction) analysis results for group 1-9 (A: 4 bar- 14 s- 50 µm, B: 6 bar- 14 s- 50 µm, C: 4 bar- 21 s- 50 µm, D: 6 bar- 21 s- 50 µm, E: 4 bar- 14 s- 110 µm, F: 6 bar- 14 s- 110 µm, G: 4 bar- 21 s- 110 µm, H: 6 bar- 21 s- 110 µm, I: control group- no treatment)

Discussion

This in-vitro study showed that MSBS values were higher in most of the experimental groups after sandblasting, and sandblasting with 110-µm particles (regardless of duration and pressure) resulted in higher MSBS values. According to the data of present study, it was concluded that the sandblasting powder size was the most important factor in determining MSBS. This study also revealed statistically significant differences between experimental groups in terms of biaxial flexural strength. The flexural strength was lower in groups 4, 7 and 8. Increased time of sandblasting treatment may produce micro-cracks and defects at the location of maximum stress that can impair zirconia and cause fracture. Regarding the particles size, the present study showed that biaxial flexural strength could decrease significantly after sandblasting with 110 µm particles.

As stated, surface grinding and roughening improves bond strength through micromechanical adhesion (19). It also prepares a wider contact area for bonding, and increases surface free energy (20). In this respect, Wolfart et al. established that grinding with aluminum oxide powder with particles of 50-110 µm in size could be effective in roughening and decontamination of zirconia surface (21). However, in the present study, only 110µm particles had a statistically significant effect on MSBS. Similarly, Aboushelib et al. found that surface roughening was a key factor in zirconia bonding (9). Also, Kim et al. recommended Al2O3 sandblasting with 110 µm sand for 3Y-TZP. They concluded that this combination results in better micro-interlocking with resin cement (22), which is in line with present study.

On the contrary, Borges et al. had rejected the correlation between zirconia surface treatment and bond strength and had reported no differences in shear bond strength when the surface was acid etched or sandblasted compared to the untreated control group. This study had also expressed that air abrasion could have minor effects on surface roughening and it could fail to produce reliable resin bond strength (23). This investigation was not consistent with the present study due to different preparation conditions. Furthermore, Smith et al. and Blatz et al. had supported the idea that mechanical adhesion was not enough for resin bonding to zirconia, and emphasized the necessity of chemical adhesion (13, 24). In the study by Su et al. pressure of more than 1 bar had been found to have no effect on bond strength of zirconia (18). Similarly, in the present study, pressures of 4 and 6 bar were evaluated and it was concluded that the size of the sandblasting particles was more important than pressure.

Similar to our study, Guess et al. had reported that particles greater than 50µm could make zirconia more prone to radial cracks during function through structural defects made by sandblasting (25).

The results of XRD analysis in this study revealed that all the experimental groups had more monoclinic phase than the control group. Sandblasting with 110 µm particles also generated higher monoclinic phase than groups with 50 µm particles and the control group. XRD analysis also showed that differences in 110 µm particles were more obvious especially for sandblasting duration of 21seconds (Group 8 (6 bar- 21 s- 110 µm) and Group 7 (4 bar- 21 s- 110 µm) that had significantly lower flexural strength and the most frequent monoclinic phase). As previously stated, these groups had lower flexural strength probably due to phase transformation after sandblasting procedure.

To summarize this study with regard to the data obtained, the size of sandblasting particles was the most significant parameter affecting MSBS and sandblasting duration had the most adverse effect on biaxial flexural strength and phase transformation.

Considering the limitations of the present study, it was concluded that sandblasting zirconia restorations with 110 µm particles improved bond strength, but the duration of sandblasting procedure was not allowed

to exceed 14 seconds, no matter what the pressure value was. Therefore, sandblasting with larger powder size and lesser duration could increase MSBS without any negative effects on BFS.

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