

# Effects of Various Chairside Surface Treatments on Zirconia-Resin Cement Bond Strength

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## Abstract

**Introduction:** Various surface treatments have been used to improve the adhesion of resin cement to zirconia restorations. The present study aimed to investigate the effects of different surface treatments on the bond strength of resin cement to zirconia (Y-TZP) in clinical practice. **Methods:** thirty square Y-TZP samples were classified into three groups of 10, including group SB (50  $\mu\text{m}$  sandblasted  $\text{Al}_2\text{O}_3$  particles), group B (diamond burs), and group C (control). One sample from each group was subjected to X-ray powder diffraction, scanning electron microscopy (SEM), and profilometer analysis. The shear bond strength (SBS) of zirconia-resin cement was measured using a universal testing machine at the crosshead speed of 1 mm/min until bonding failure. SBS values were analyzed using analysis of variance (ANOVA) and Tukey's HSD test ( $\alpha=0.05$ ). **Results:** According to the results of ANOVA, SBS was significantly affected by the treatment method. Tukey's HSD test showed significant differences between the groups ( $P<0.05$ ). Groups SB ( $9.99\pm 0.78$  MPa) and B ( $9.30\pm 0.67$  MPa) had significantly higher SBS values compared to group C ( $6.47\pm 1.33$  MPa) ( $P<0.05$ ), while they had no significant differences with each other in this regard ( $P>0.05$ ). In addition, SEM evaluations indicated morphological differences between the Y-TZP samples. According to the results of X-ray diffractometer, monoclinic phase transformation was observed in group SB only (28%). **Conclusion:** According to the results, grinding and sandblasting were both effective in chairside surface treatments for improving the bond strength of the resin cement to Y-TZP. However, it should be considered that sandblasting may cause phase transformation.

**Keywords:** Bond Strength, Chairside, Surface Treatment, Zirconia.

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## Introduction

Zirconia-based ceramics have become popular in dentistry since their metal-free structure meets the aesthetic demands of patients. These ceramics provide superior mechanical and physical properties, such as high flexural strength, low elastic modulus, high fracture toughness, and damage tolerance with a stress-induced transformation toughening mechanism compared to other ceramic systems (1-4). Zirconium oxide (ZrO) is a polymorphic material with three allotropes. The monoclinic phase (room temperature-1,170°C) could be transformed into the tetragonal phase (1,170-2,370°C) and the cubic phase (2,370°C-melting point) (5). The phase transformation of tetragonal ZrO to the monoclinic phase is induced by stressors such as grinding, impact or fracture. The transformation leads to volume expansion (3-5%) and crack propagation (4).

The cementation process is essential to the clinical success of zirconia restorations (6). Although conventional methods could be applied for the cementation of zirconia restorations, they do not ensure sufficient bond strength. Therefore, using resin cement is preferred since it provides better marginal seal, while enhancing the retention and fracture resistance of the restorations (6-8). Adhesion of resin cements to zirconia restorations has proven difficult since the chemical composition of zirconium lacks the glass phase (9). Therefore, various techniques have been proposed to enhance the adhesion between zirconia restorations and resin cement, including airborne-particle abrasion, laser irradiation, tribochemical silica coating, plasma spraying, heat-induced maturation, use of organofunctional silanes, and use of phosphate-modified monomers (MDPs) in resin cement. Since zirconia has a high crystalline content, neither hydrofluoric acid etching nor silanization could improve the bond strength for zirconia-based ceramics (1, 6, 7, 10, 11).

Air abrasion systems remove loose, contaminated layers and modify the surface topography of zirconia through increasing its roughness and wettability, thereby improving bond strength through mechanical interlocking. The system encompasses various particle sizes within the range of 30-250  $\mu\text{m}$  (10-13). There are speculations that some manufacturers do not recommend air particle abrasion prior to cementation since abrasion might create microfractures that reduce fracture strength and cause catastrophic failure (8, 14). However, several studies have denoted that the bond strength to zirconia ceramics improves after air abrasion (15-17).

Grinding with diamond burs may be an alternative technique in this regard, which has been described in the literature for the roughening of zirconia restorations with favorable effects on the bond strength of resin cement (16, 18). Grinding with a fine bur enhances the flexural strength and reliability of zirconia, while grinding with a

coarse bur decreases its flexural strength and reliability (3). Additionally, severe grinding leads to deep defects in the surface and stress concentrations of the material (18).

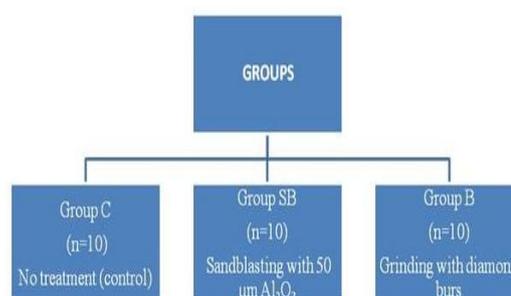
Previous studies have demonstrated that air abrasion and grinding with burs have no significant effects on the improvement of the bond strength of zirconia to resin cements (1, 17, 19, 20). Furthermore, grinding and sandblasting could both trigger the tetragonal phase to monoclinic phase transformation (21).

Several studies have examined the effects of various treatment methods on the bond strength of resin to zirconia restorations (22, 23), and other studies have been focused on the clinically applicable methods in this regard (24, 25). The present study aimed to investigate the effects of two chairside treatment methods, including airborne-particle abrasion and grinding with diamond burs, on the bond strength of zirconia-resin cement.

## Materials and Methods

In this study, 30 square-shaped (10×10×2 mm) zirconia samples (Y-TZP) (Zirkonzahn, Zirkonzahn USA Inc., USA) were prepared using a copy-milling machine (Yenamak, Ezcama, İstanbul, Turkey) and sintered in accordance with the instructions of the manufacturer. The samples were embedded in a PVC ring (height: 20 mm, diameter: 25 mm) using an autopolymerizing acrylic resin block.

The surfaces of Y-TZP samples were ground-finished using silicon carbide abrasive papers (240, 400, 600, 800 grits) and a grinding machine at 600 rpm (Minitech 233, Presi, Grenoble, France) under running water (cooling) for one minute. Following that, the surfaces were cleaned with acetone and dried in air stream. The samples were randomly classified into three groups of 10 (Fig.1).



**Figure 1.** A Schematic View of Study Groups

The samples in group SB were abraded using Al<sub>2</sub>O<sub>3</sub> particles (50 μm) with the airsonic mini-sandblaster (Hager Werken, Germany) at the two-bar pressure and distance of 10 millimeters between the nozzle and surface for 20 seconds. In this process, the airsonic mini-sandblaster was perpendicular to the Y-TZP surface. The samples in group B were prepared using high-speed green-labeled FG burs (Dia-Burs, Mani, Utsunomiya, Tochigi, Japan; grit size: 150 μm) with water cooling. The surface grinding procedures were performed using a high-speed turbine (BA International Ltd., Northampton, England) at 400 krpm. The burs were employed to grind the sample surfaces with minimal pressure in one direction, and they were replaced afterwards.

After the surface treatments, all the samples were ultrasonically cleaned in 96% isopropyl alcohol for 380 seconds and dried. An extra sample from each group was selected for scanning electron microscopy (SEM) (magnification: 100X, Zeiss Evo LS10, Carl Zeiss Microscopy GmbH, Jena, Germany) and 3D surface profilometer (Nanomap 500LS Stylus Profiler, Ankara, Turkey). Moreover, X-ray diffraction (XRD) analysis was performed to examine the samples in terms of the effects of the treatments on the phase composition and occurrence of phase transformations in the material.

The samples were evaluated by XRD (D5000, Siemens, Germany) using CuKα radiation after the surface treatments. Relative amounts of the transformed monoclinic zirconia on the treated surfaces were determined based on the integral intensities of the monoclinic M(111) and M(111̄), as well as the tetragonal T(111) peaks (Equation 1) (18):

$$(1) \quad X_M = \frac{I_{M(111)} + I_{M(11\bar{1})}}{I_{M(111)} + I_{M(11\bar{1})} + I_{T(111)}}$$

A cylindrical Teflon mold with a hole (inner diameter: 4 mm, height: 3 mm) was fabricated. The PVC ring was seated at the centre of the hole, and the resin cement (RelyX™U100 Self-Adhesive Resin Cement, 3M ESPE, U.S.A) was applied in the hole and polymerized using an LED curing light (Elipar, 3M ESPE) for 20 seconds at the light intensity of 800 mW/cm<sup>2</sup>. The Teflon mold was gently removed, and all the samples were preserved in a desiccator at room temperature for 24 hours prior to shear bond strength (SBS) testing.

A universal testing machine (Shimadzu AGS-X, Shimadzu Corporations, Tokyo, Japan) was used to test the SBS of zirconia to resin cement at the crosshead speed of 1 mm/min until bonding failure. After the SBS testing, one sample from each group was evaluated using

the SEM. In this process, the samples were examined in stereomicroscope (Olympus SZX10) with a 100X magnification in terms of the type of failure (adhesive, cohesive or mixed).

Based on the data obtained from the pilot study, sample size was estimated at 10 using a power analysis to provide the statistical significance (α=.05) with 80% test power. In addition, the ultimate stress value (MPa) of the Y-TZP-resin cement was calculated based on the following formula (Formula 1) (26):

$$(1) \quad \text{Stress} = \frac{\text{Failure Load (N)}}{\text{Surface Area (mm}^2\text{)}}$$

The SBS values were analyzed in SPSS version 11.5 (SPSS, Chicago, ILL). In addition, the Kolmogorov-Smirnov normality test was used to assess the data distribution of the groups, and the homogeneity of the variances was examined using Levene's test. Since the test results were indicative of the normal distribution of data in the groups and variance homogeneity, one-way analysis of variance (ANOVA) and Tukey's HSD test were employed for the comparison of the groups. ANOVA was also used to investigate the data in terms of significant differences, and Tukey's HSD test was applied to perform multiple comparisons. In all the statistical analyses, the significance level was determined to be P<0.001.

## Results

The mean SBS values of the three groups are presented in Table I. According to the results of one-way ANOVA, SBS was significantly affected by the treatment method (P<0.001). In addition, the results of Tukey's HSD test regarding the SBS values of zirconia bonded with resin cement revealed significant differences between the groups (P<0.05).

According to the findings, groups SB and B had significantly higher SBS values compared to group C (P<0.05), while they had no significant difference with each other (P>0.05). Mode of failure in the study groups is shown in Table II.

**Table I.** Mean Shear Bond Strength Values in Study Groups: Group SB (Sandblasting with 50 μm Al<sub>2</sub>O<sub>3</sub>), Group B (Grinding with Diamond Burs), Group C (Control)

Groups	N	Mean (MPa)	SD
<b>Group SB</b>	10	9.99 <sup>a</sup>	±0.78
<b>Group B</b>	10	9.30 <sup>a</sup>	±0.67
<b>Group C</b>	10	6.47 <sup>b</sup>	±1.33

\*Different superscripts letters indicate statistically significant differences in mean values (P<0.05)

**Table II.** Mode of Failure: Group SB (Sandblasting with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$ ), Group B (Grinding with Diamond Burs), Group C (Control)

Group	Adhesive	Cohesive	Mixed
SB	7	x	3
B	8	x	2
C	10	x	x

### SEM Evaluations

The SEM images of the zirconia surfaces after surface treatment are depicted in Figure 2. Morphological differences were observed between the Y-TZP samples after surface treatments. Comparison of the groups indicated that group B had parallel, deep scratches, while group SB exhibited randomly oriented pits.

### Profilometer Evaluations

The 3-D profilometer images showed the surface and depth irregularities of each treatment method (Fig. 3). In the profilometer evaluations, the distance between the top and base was measured in the three groups, as follows: group C surface with 8.56  $\mu\text{m}$  in depth, group SB surface with 21.56  $\mu\text{m}$  in depth, and group B surface with 88.06  $\mu\text{m}$  in depth.

### X-Ray Diffractometer

Comparison of the groups indicated the monoclinic phase transformation rate to be 28% in group SB, while the other groups showed no phase transformation.

## Discussion

The non-durable bond strength of resin cement to Y-TZP, which is an acid-resistant ceramic, is considered to be a major concern among clinicians (27). Therefore, various treatment methods have been developed to improve the bonding performance of zirconia restorations. In the light of these findings, the present study aimed to investigate the effects of two chairside surface treatments (i.e., sandblasting and grinding with diamond burs) on the bond strength of Y-TZP to resin cement. The current research was based on the challenges faced by clinicians regarding sandblasting as a surface treatment method.

Although sandblasting is often preferred as a surface treatment for dental ceramics, it has been reported to cause major complications, such as crack propagation. The alternative method (i.e., grinding with diamond burs) has not been extensively studied in the literature. Therefore, the present study aimed to assess whether the latter could prevent the unwanted results observed in sandblasting.

In the current research, there were three groups, one of which was control without any interventions by

clinicians. The other groups were experimental, in which sandblasting (group SB) and grinding with diamond burs (group B) were used to compare their effectiveness in terms of increasing the bond strength of Y-TZP to resin cement. According to the obtained results, the bond strength of Y-TZP to resin cement improved in both the experimental groups. Based on the findings, the null hypothesis which stated that grinding with diamond burs would not improve the bond strength of Y-TZP to resin cement was ruled out. The increased bond strength in the experimental groups may have resulted from the increased surface area, wettability, and micromechanical retention (28).

As can be seen in Fig. 3, surface roughness enhanced in groups B and SB, which is consistent with the SEM images (Figure 2). These findings are in line with the studies by Curtis et al. (3) and Kosmac et al. (18), who stated that grinding with coarse diamond burs caused a significant increase in surface roughness compared to fine grinding. Grinding with diamond burs may lead to positive and negative outcomes. With regard to the positive outcomes, grinding induces compressive stresses on the surface, thereby increasing the bond strength of Y-TZP to resin cement. As for the negative outcomes, grinding causes defects on the surface, which could exceed the depth of the compressive layers of the surface and lead to stress concentration (4, 18).

In the present study, profilometer was used to evaluate the results of grinding with diamond burs, which has not been applied in the previous studies in this regard. According to the profilometer data (Figure 3), grinding with diamond burs significantly increased the surface roughness.

In a study in this regard, Queblawi et al. (2) denoted that grinding with diamond burs resulted in higher flexural strength (1,727 MPa) compared to airborne-particle abrasion (798 MPa). The higher bond strength value of the grinding procedure was attributed to the grit size of the diamond burs (fine grit), which differed from those used in the present study (coarse grit). These findings were compatible with the results obtained by Kosmac et al. (29), which indicated that dental grinding and sandblasting had counteractive effects on the performance of zirconia as they measured flexural strength with thermal cycling. Thermal cycling is a factor that could affect the SBS value of resin cement and plays a key role in the simulation of oral conditions (30).

As stated earlier, sandblasting is the preferable surface treatment method for dental ceramics (31) since it improves surface roughness, surface energy, and microporosity. However, it may function as a crack initiator, causing the weakening of the material (32). In the current research, a close improvement was observed in the SBS values of group SB ( $9.99 \pm 0.78$  MPa) and group B ( $9.30 \pm 0.67$  MPa). In the case of surface

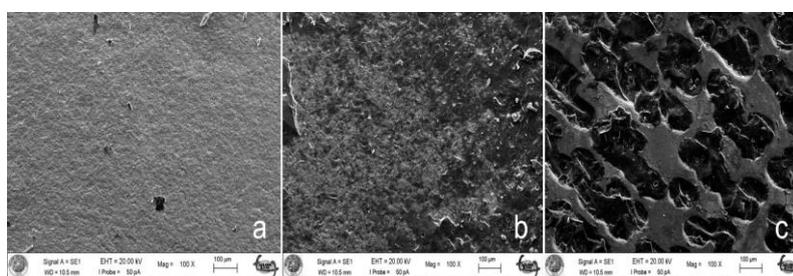
treatment with sandblasting, the previous studies in this regard have demonstrated that high bond strength was only observed on air-abraded surfaces with the following application of an MDP-containing composite resin (6, 15, 33).

In another research, Kern and Wegner reported favorable initial bond strength with BIS-GMA resin cement to zirconia treated with air-particle abrasion (19). In the present study, we applied RelyX U100 Self-adhesive resin cement, which had high bond strength and had not been previously used in a research. The material contains neither BIS-GMA nor MDP monomers.

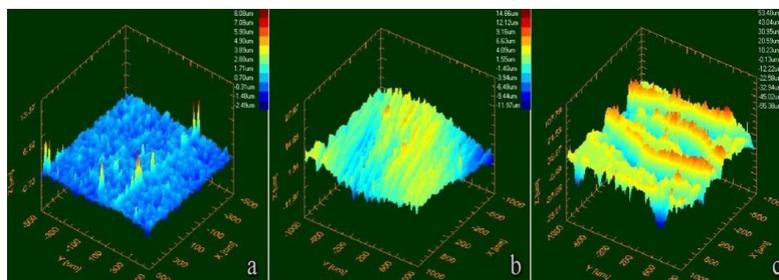
Our findings were inconsistent with the results obtained by Kosmac et al. (18), who investigated the effects of sandblasting and grinding. According to the

mentioned research, surface grinding using diamond burs with coarse grits decreased mean strength and increased local temperature, thereby leading to phase transformation. Contrary to the findings of the mentioned study, our findings indicated that grinding with diamond burs could not increase the monoclinic phase contents as shown by the XRD analysis (Fig. 4).

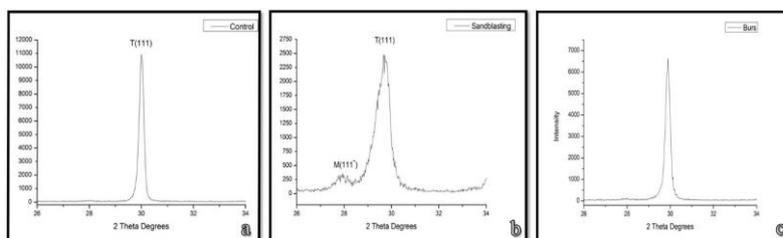
One of the limitations of this *in-vitro* study was that since it was not a funded research project, it yielded limited profilometer results. Had the project been funded, it would have provided the profilometer analysis for each subject in all the study groups for more accurate results. Another limitation was the absence of the thermal cycling effect and local temperature measurement, which was generated during the treatment process.



**Figure 2.** SEM Micrographs of Zirconia Surface: a) Group C, b) Group SB, c) Group B



**Figure 3.** Profilometer Images of Zirconia Surface: a) Group C, b) Group SB, c) Group B



**Figure 4.** XRD Analysis of Zirconia: a) Group C, b) Group SB, c) Group B

## Conclusion

Within the limitations of the present study, the following conclusions were drawn:

1. Compared to 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  sandblasting, grinding could effectively increase the bond strength of resin cement to the Y-TZP surface.
2. The SBS value of 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  sandblasting was similar to the SBS value of grinding although 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  sandblasting led to phase transformation.
3. Grinding and sandblasting were both effective in chairside treatments to enhance the bond strength of resin cement to Y-TZP.

It is recommended that further investigations in this regard assess the effects of thermal cycling, different grit sizes, and heat generation during the grinding procedure.

## Conflicts of interest

The author declare no potential conflicts of interest.

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