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Comparative evaluation of micro-tensile bond strength between zirconia core and all-ceramic layering with different surface treatments: an *in vitro* study

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ABSTRACT

Background: The quest for newer and stronger materials for replacing teeth has resulted in Zirconium oxide's introduction, which possesses excellent mechanical strength and toughness. However, uncertainty exists in the relationship between its bond strength and surface treatment method adopted and the mode of failure at the interface.

Aim: The study aimed to evaluate the comparison of micro-tensile bond strength between the zirconia core and all-ceramic layering with different surface treatments and analyse their failure mode by Scanning Electron Microscope (SEM).

Materials and methods: Zirconia cores (Ceramill ZI 71 XS) were fabricated by CAD-CAM into discs with 5mm diameter and 3.5mm height. Then the cores were divided into four groups. Among which, Group-I was the control group, and the remaining were surface treated. Group-II specimens were treated with sandblasting, followed by acid etching; Group-III and Group-IV were treated with zirliner and glass beads, respectively. After that, the veneering material (IPS Empress, E.max Ceram Dentin) of 2×2 mm was adhered to the zirconia core and then kept in the ceramic furnace. The specimens were mounted on a Universal Testing Machine, and tensile stress was applied. The obtained data were subjected to One-way ANOVA and Tukey-HSD tests for statistical analysis.

Results: The samples treated with sandblasting followed by acid etching showed more micro-tensile bond strength at core and veneer interface. Furthermore, the SEM study revealed a cohesive failure in Group-II, whereas, in Group-I and -III, there was an adhesive failure. Group-IV specimens exhibited a mixed failure. One -way ANOVA showed significant differences ($p=0.001$) within the groups. In posthoc analysis, Group-III showed significant differences with Groups -I, II, and IV.

Conclusion: Increased surface roughness of zirconia obtained by sandblasting with aluminium oxide particles, when coupled along with chemical etching with hydrofluoric acid, enhanced the micro-tensile bond strength between the Y-TZP zirconia core and veneering ceramic.

1. Introduction

Teeth are considered the most important component of the stomatognathic system and their loss leads to an imbalance in harmony in the masticatory apparatus [1]. Porcelain holds a special place in dentistry as it is considered most

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aesthetic restorative material with good translucency. Further, it is highly biocompatible among the materials used in dentistry [2]. Combining better manufacturing techniques such as computer-aided design/computer-aided manufacturing (CAD-CAM) with stronger materials has increased the possibility of using all-ceramic restorations for dental applications. The advancement in technology has improved the fracture toughness, wear resistance, machinability, hardness and flexural strength of ceramics [3-7]. Zirconium oxide was introduced as a core material for all-ceramic restorations due to its good chemical properties, high mechanical strength, toughness, and Young's modulus similar to that of stainless steel [8].

All-ceramic material failures reported, were either as delamination of veneering ceramic from the core ceramic or sometimes in the form of cracks on the core material itself. There are various reasons for these types of failures, such as, crack propagation of core material which reduces bonding effect, insufficient thermal expansion due to a sudden change in temperature during the conversion of the liquefied stage to the solidification stage and various transformations of crystalline stage [9].

Several studies suggested that a tensile bond strength test may be more appropriate for evaluating adhesive interfaces' bond strength because of more uniform interfacial stresses [2,3]. The micro-tensile bond strength test was developed to eliminate the non-uniform stress distribution at the adhesive interface, and it has been used to measure the bond strength [8]. Hence, this study was designed to evaluate the effect of various surface treatments on the micro tensile bond strength of layered ceramic on the zirconia core and compare them. Further, this study has also analysed the mode of failure using a Scanning Electron Microscope (SEM).

2. Materials and methods

Pre-sintered Y-TZP zirconium oxide blanks (Ceramill ZI 71 XS, Dental arch form, h=12mm Amann Dental Gmbh and Girrbach Dental Gmbh, AmannGirrbach, Austria) were used for core fabrication using CAD-CAM technology.

2.1 Fabrication of Aluminium metal dies for core

A standard metal disc having a 14 mm external diameter, 10mm of internal diameter and a thickness of 4mm with 3.5mm height was fabricated from aluminium blocks by lathe milling. The inner surface was smoothened using files and abrasive papers (Ecomet, Buehler LTD, Evanston, Ill, USA).

2.2 Fabrication of Aluminium Metal Die for Layering Ceramic

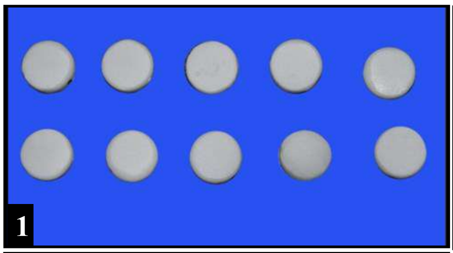
An aluminium disc measuring 6mm external diameter, 2mm internal diameter and thickness of 2mm with a height of 2mm was fabricated from aluminium blocks by lathe milling. The aluminium block was shaped and trimmed using the lathe cutter. The die measurements were verified using electronic Vernier Calipers (Mitutoyo Corp, Tokyo, Japan).

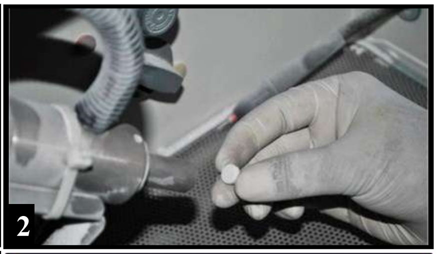
2.3 Fabrication of Core

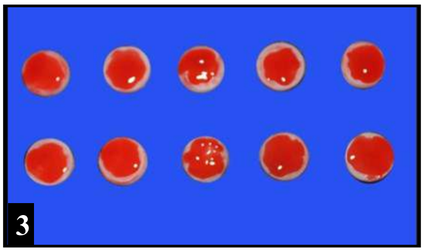
The standard metal disc was used for the preparation of 40 core samples. Pre-sintered Y-TZP Zirconium oxide blanks (Ceramill ZI 71 XS, Dental arch form, h=12mm Amann Dental Gmbh and Girrbach Dental Gmbh, AmannGirrbach, Austria) were used for core fabrication using CAD-CAM technology. A Ceramill map 300 scanning machine was used to scan the metal die, and later, the design was transferred to the Ceramill motion, and zirconia samples were prepared (Figure 1).

2.4 Surface treatment for Core

Forty samples were fabricated by the Ceramill motion milling machine and were steam washed adequately. The samples were then divided into four different groups (Table 1). Group-I with no surface treatment was used as the control group against which the other three surface treatments were compared. For Group-II samples, first sandblasting with 120µm of aluminium oxide (Al_2O_3) (Cobra, Renfert, Strahimittei, Germany) was done for 1 minute (Figure 2). The samples were then etched by 5% hydrofluoric acid (IPS ceramic Refill, Ivoclar Vivadent, Germany) for 1 minute (Figure 3). The surfaces of the Group-III samples were treated with Zirliner (IPS-E.max, Ivoclar Vivadent, Germany). It was mixed into a creamy consistency and then was applied to the zirconia core until an even greenish colour effect was achieved. After the application, the zirliner was properly dried and fired in the ceramic furnace (Programmat-P 100, Ivoclar Vivadent AG, Schaan, Liechtenstein) according to the manufacturer's instructions. Group IV samples were surface treated with glass beads of 50µm (Rolloblast, Renfert, Strahlmittel, Germany) for 1 minute (Figure 4).







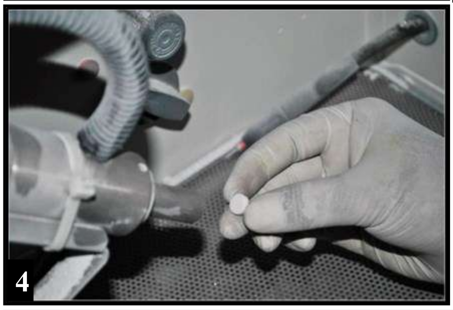


Figure 1. Prepared Zirconia core Discs.
Figure 2. Sandblasting the core ceramic surface with alumina.
Figure 3. Etching the ceramic Core with hydrofluoric acid.
Figure 4. Sandblasting the core surface with glass beads.
Figure 5. Veneering ceramic on the zirconia core before heating.
Figure 6. Prepared zirconia core and veneer after heating.

Figure 7. Evaluation of Micro-tensile bond strength at core ceramic and layered ceramic.

Table 1. Surface treatments used in various groups

Groups	N	Surface treatment
I	10	No surface treatment
II	10	Sand blasting+Hydrofluoric acid
III	10	Zirconia liner
IV	10	Glass beads

Table 2. Mean and standard deviation of Micro-tensile bond strength (MPa) of Ceramic specimens.

Groups	N	Mean ± SD#	Significance
Group –I	10	81.43±26.87	0.001*
Group –II	10	99.89±21.97	
Group –III	10	54.28±18.85	
Group –IV	10	76.68±30.49	

Standard Deviation; * Significant

Table 3. Comparison of micro-tensile bond strength of ceramic specimens (Post-hoc analysis)

Groups		Mean Difference	Significance
Group -I	Group -II	18.46	0.098
	Group -III	27.15	0.005*
	Group -IV	4.76	0.931
Group -II	Group -III	45.61	0.001*
	Group -IV	23.21	0.022*
Group -III	Group -IV	22.40	0.029*

* Significant

2.5 Application of Layer Ceramic to the Core Specimen

Aluminium dies fabricated for specified dimension was used for layering procedure. The recommended ratio of layering powder and liquid were taken and mixed on the ceramic mixing slab. The layering with the dentin body of D4 shade was kept in the ceramic furnace (Programmat-P 100, Ivoclar Vivadent AG, Schaan, Liechtenstein) (Figures 5 and 6).

2.6 Evaluation of Micro-tensile bond strength (MTBS)

The samples were attached with nickel-chromium stands of 3cm length to the layering ceramics to secure it on the testing machine. The specimen was mounted for testing in Universal Testing Machine (Instron) and tensile load was applied at a crosshead speed of 1 mm/ min (Figure 7).

2.7 Scanning Electron Microscope (SEM) analysis

The failure modes at the fracture site were analyzed using Scanning Electron Microscope (Quanta200 F). The specimens obtained following the MTBS testing were subjected to SEM under a specific magnification of 250X.

The data were subjected to One-way ANOVA and Tukey-HSD tests for statistical analysis using SPSS for Windows, Version 21.0., SPSS Inc.

3. Results

The Normality test results, Kolmogorov-Smirnov and Shapiro-Wilk tests showed that the sample did not follow the normal distribution. Therefore, to analyse the data, non-parametric tests were applied. To compare micro-tensile bond strength (MPa) between all the four groups, Kruskal Wallis was applied, and for pairwise comparison, Mann-Whitney U tests with Bonferroni corrections were used.

The mean micro-tensile bond strength of the groups is given in Table 2. The ceramic specimens treated with both sandblasting and hydrofluoric acid (Group-III) exhibited more micro-tensile bond strength among the groups (Table 2). The ceramic specimens treated with Zirconia liner demonstrated the least micro-tensile bond strength. One-way ANOVA showed significant differences (p=0.001) within the groups.

In Post-hoc analysis, Group-III showed significant differences with Groups I (p=0.005), -II (p=0.001) and -IV (p=0.029). The group-II specimens also exhibited significant differences (p=0.022) with group IV (Table 3).

SEM analysis showed adhesive and cohesive debonding in the layers of ceramic (Figures 8 a-d). The ceramic specimens in group-I and -III showed adhesive failure and the group -II specimens showed cohesive failure (Table 4). The specimens in group-IV exhibited both adhesive and cohesive failures.

4. Discussion

Material selection, performances, and clinical recommendations on layered all-ceramics are based on standard mechanical testing methodologies [9]. Information on the best combination of zirconia core and veneering ceramic could help the clinician predict possible fracture or debonding at the core-veneering ceramic interface [10]. In this study, Yttria-stabilized Tetragonal Zirconia Polycrystals (Y-TZP) were used for the core fabrication. A low fusing nano-fluorapatite glass-ceramic was used for the veneering of the Zirconia core. The presence of nano-fluorapatite crystal structure (100-200 nm) with a length of 1-2 µm enhanced the material’s optical property. The material could also be used as a single layering material for veneering [11,12]. With the application of layered ceramic on the Zirconia core, there have been many changes in the stress distribution pattern that makes its performance to be hardly predictable in a clinical scenario [13]. Thus, the present study aimed to determine the bond strength between the Zirconia Oxide core and the layering ceramic.

For improving bond strength, sandblasting is a popular means by increasing surface roughness and providing undercuts [14,15]. However, sandblasting also initiates phase transition, affecting the mechanical strength and, most probably, the material's bonding capacity [16]. On this score, the effect of sandblasting on the mechanical strength of Y-TZP & the bond quality to veneering ceramics is thus, an intensely studied subject and was adopted as one of the surface treatments in the present study. Having employed a low-fusing nano-fluorapatite glass-ceramic as a veneer material for CAD/CAM Y-TZP zirconia core, an etchant gel was used after sandblasting to study the effect on bond strength. The application of liner material to mask the white colour of Zirconia and to improve the

Table 4. Percentage of mode of failure in different groups .

Groups	Adhesive Failures (Between core and veneer)	Cohesive Failure (Within veneer)	Mixed Failure (Combination of both)
I	100%	0%	0%
II	0%	100%	0%
III	100%	0%	0%
IV	50%	40%	10%

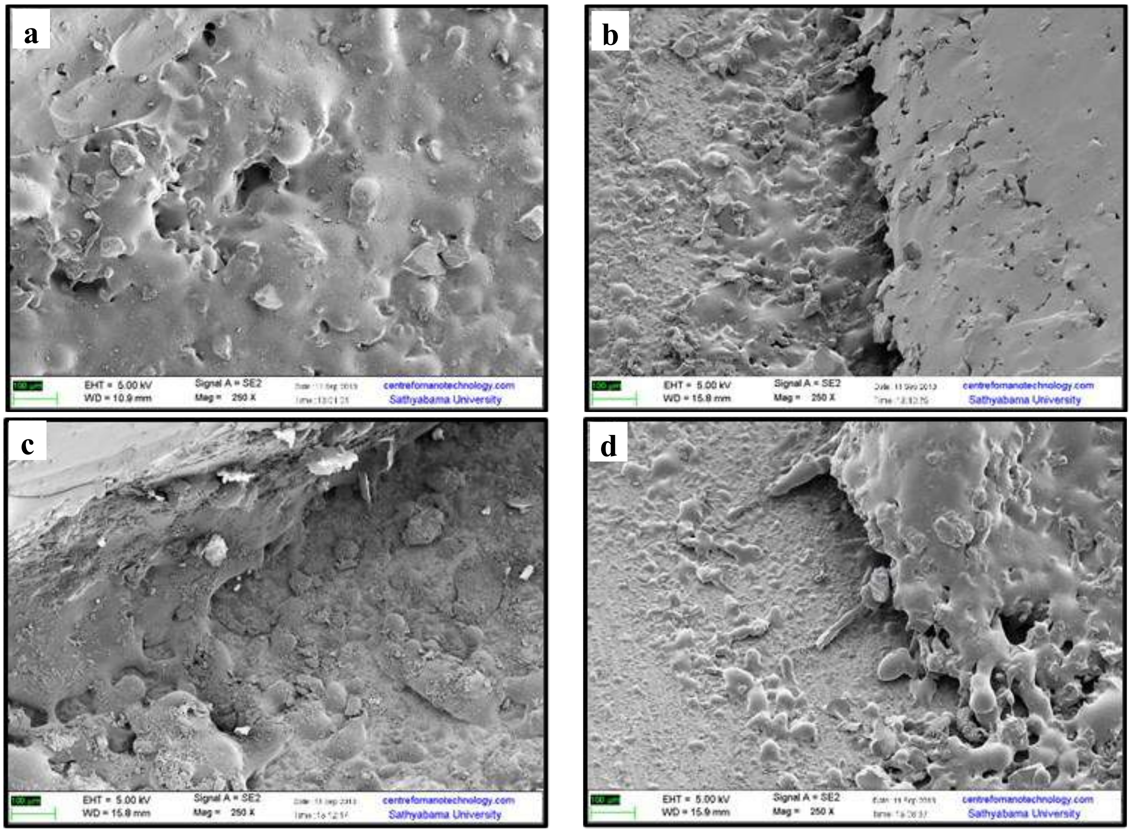


Figure 8. SEM analysis of bonding failure. Where a. Adhesive failure observed in group-I specimens; b. Cohesive failure observed in group-II specimens; c. Adhesive failure observed in group-III specimens; and d. Group-IV specimens exhibited both adhesive and cohesive failures.

bond strength between the core and the veneer layers has been studied before [17]. With IPS e.max Ceram as layering material, the corresponding liner was used to evaluate the effect of bond strength. The non-abrasive glass beads for smoothing and condensing of the ceramic surface have been used in the study with 50μm, to decrease the crack formation during function [18]. With the several perplexities among the different surface treatments of bilayered ceramics, a comparison was made to emanate the results.

This study showed that Group II had the highest mean bond strength compared to other groups (Table 2). The p-value was significant (0.010) as was found in the Kruskal-Wallis Test. Group-III demonstrated the least bond strength among all the four groups. When each group was compared with the control group (Group-I), specimens with sandblasting along with acid etching (Group-II) elicited the highest micro-tensile bond strength values (MTBS). Minimal variation of the standard deviation of all four groups with enhanced MTBS values could be attributed to the use of high-performance zirconia oxide in tetragonal metastable form as core and compatible low fusing fluorapatite glass-ceramic as veneer material [17]. In the control group, debonding occurred at the interface between the core and the veneer material as revealed by the SEM images. Hence, an adhesive mode of failure materialized in Group-I. This may be partly due to large differences in the flexural strengths between the two ceramics and more significantly, any mismatch in the elastic moduli [19].

The MTBS values when compared with Group II showed little less bond strength, confirming the results of the previous studies in which the various surface treatments showed an improvement in MTBS [20]. Group-II specimens where both mechanical and chemical mode of surface treatments were employed showed the highest MTBS, and they were in compliance with prior studies [21,22]. After the mechanical interlocking with airborne abrasives, hydrofluoric acid etchant application was done on the ceramic layer. Use of acid alone on zirconia is difficult due to its chemical inertness [23]. However, acid etching is a commonly used method for silica-based glass-ceramic surfaces and hence was coupled along with sandblasting [24].

The results of Group-III were in contrast to the study done by Fleming *et al.* (2004) [25] where an increase in strength was found at the smooth interfacial surface. But the outcome supported the research done by Aboushelib *et al.* (2010) [23]. In Group III, no mechanical porosities were created, and only Zirliner was applied, which could be attributed for the least MTBS.

The use of glass beads in Group-IV showed a nominal difference in MTBS compared to the control group. A mixed adhesive and cohesive debonding was detected in the layers of ceramic (Table 4).

Non-abrasive particles, unlike Al_2O_3, usually employed for surface treatment and divesting of all-ceramic materials, increased the bond strength but not to a very great extent. In general, the thicker the zirconia and veneering ceramics, the higher the residual stresses. The specimen ratio in this study was high, with 5mm thick zirconia disc and 2mm veneering porcelain than represented in typical dental restorations [20].

5. Conclusion

The increased surface roughness of zirconia obtained by sandblasting with aluminium oxide particles, coupled with chemical etching with hydrofluoric acid, enhanced the micro-tensile bond strength between the Y-TZP zirconia core and veneering ceramic.

Conflicts of interest: Authors declared no conflicts of interest.

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