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Evaluation of Micro Tensile Bond Strength between Surface Treated Zirconia and Ceramic: An Invitro Study

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Abstract:
Purpose: The purpose of the present study was to compare the micro tensile bond strength at the core – veneer interface of zirconia restorations after using Al_2O_3 airborne particle abrasion, chemical etching with 5:5 mixed solution of hydrofluoric acid and nitric acid and both sandblasting and chemical etching. MATERIALS AND METHODS:-A total of 60 specimens of sintered zirconia ceramic of size 10×5×5 mm were used for the experiment. Sixty samples were randomly divided into four groups according to the surface treatment method used. Group 1(control): CAD/CAM milled surface without any surface treatment. Group 2: Al_2O_3 airborne particle abrasion was performed using a sandblasting machine with 110µ particle size. Group 3: Chemical etching using 5:5 mixed solution of hydrofluoric acid and nitric acid for 2 hrs. Group 4: sandblasting with Al_2O_3 particles and chemical etching with 5:5 mixed solution of hydrofluoric acid and nitric acid. Sectioning of specimens was done until 1×1×14 mm^3 microbars were obtained. The microbar with metal bars was attached to the opposing arms universal testing machine. RESULTS:-The highest mean micro tensile bond strength value was measured in the sandblasting, and chemical etching group (46.4 MPa), and the lowest value was measured in the control group (30.84 MPa). CONCLUSIONS:-There is a significant increase in the mean micro tensile bond strength between experimental groups and the control group. There is also a considerable increase in value in group 4 from groups 2 and 3. But there is no significant difference between group 2 and group 3.
Key words: micro tensile bond strength, zirconia, ceramic, sandblasting, chemical etching.

Introduction:

In clinical situations that require highly demanding esthetic restorations, all-ceramic restorations have the potential to be a more effective selection when compared to metal-ceramic restorations. The alloy structure in metal restorations may create an opaque appearance, while ceramic materials seem to generally produce a more translucent look that replicates the appearance of the natural tooth[1].
The increasing demand for esthetics has increased the popularity of all-ceramic restorations. The inherent properties of ceramics, such as brittleness, have limited their application, especially in posterior teeth[2]. Advances in ceramic materials over the years have resulted in the introduction of zirconia (ZrO2) as a viable material for use in dental prosthetics[3]. However, zirconia is a material of choice for the substructure due to its superior mechanical properties, without the limitations related to the size or position of the restoration.
The excellent mechanical and chemical properties of zirconia (ZrO2) have led to its use both as an alternative to traditional dental porcelain alloys and for the fabrication of posterior fixed partial dental

prostheses (FDPs). Dental zirconia is often yttria-stabilized tetragonal zirconia polycrystals(Y-TZP) or yttria-partially stabilized zirconia (YSZ). Y-TZP has shown to have greater flexural strength (900 to 1200 MPa) and fracture toughness (9 to 10 MPa/m2)than other ceramic systems due to a transformation toughening mechanism[4,5].
Chipping of the veneering ceramic or delamination of the veneer from the core is the most frequently occurring technical complications in core-veneered zirconia restorations.
Chipping was reported in 25% of the posterior 3-unit FDPs after 31.2months of observation in 1 study[4]. In another study, after a 3-year follow-up, porcelain fractures were reported in 11.4% of the posterior FDPs[6]. In a third study, veneering porcelains fractured in 13% of the posterior FDPs(having a terminal abutment) after a 48-month follow-up, and in 12%of the posterior FDPs (cantilever design)after a 50-month follow-up[7]. The estimated 5-year complication rates ranged from 10% to 60% [8]. These results were attributed primarily to insufficient bond strength between zirconia frameworks and veneering porcelains.
The clinical success and reliability of these restorations are highly dependent on the bond strength at the interface between the veneering ceramic and core.
Bond strength is affected by many variables, including the surface treatment of the substructure, the development of defects at the core-veneer interface, the residual stresses due to the thermal mismatch between the core and veneering porcelains, the reactions at the interface between substrates and veneering porcelains, and the wetting properties of the surface[9].
Surface treatment of zirconia for higher bond strength can be achieved by removing or adding surface materials. Airborne-particle abrasion is a routine way to roughen and clean resin or porcelain bonding surfaces of zirconia [10, 11]. It is essential to consider that airborne particle abrasion results in a phase transition at the surface, changing the crystal structure from tetragonal to monoclinic [12]. These crystal structures exhibit different coefficients of thermal expansion (CTE)[13].
In general, the structure of veneering ceramic has been described as an amorphous and glassy matrix that consists of a random network of cross-linked silica in a tetrahedral arrangement, which is embedded in varying amounts of un-dissolved feldspar and leucite crystals. For ceramic surface treatment, the acid reacts with the glassy matrix that contains silica and formshexafluorosilicates. This glassy matrix is selectively removed, and the crystalline structure is exposed. As a result, the surface of the ceramic becomes rough, which is expected for micromechanical retention on the ceramic surface. This roughly etched surface also helps to provide more surface energy [14].
The purpose of the present study was to compare the micro tensile bond strength at the core – veneer interface of zirconia restorations after using Al_2O_3 airborne particle abrasion, chemical etching with 5:5 mixed solution of hydrofluoric acid and nitric acid and both sandblasting and chemical etching.

Materials and Methods:

A total of 60 specimens of sintered zirconia ceramic (3Y-TZP, AcuceraInc., New York, NY, USA) of size 10×5×5 mm were used for the experiment.

Surface treatment methods:

Sixty samples were randomly divided into four groups according to the surface treatment method used.
Group 1(control): CAD/CAM milled surface without any surface treatment.
Group 2: Al_2O_3 airborne particle abrasion was performed using a sandblasting machine with 110µ particle size for 15 seconds at a 10 mm distance from the surface and with a pressure of 3.5 bar.
Group 3: Chemical etching using 5:5 mixed solution of hydrofluoric acid and nitric acid for 2 hrs.
Group 4: sandblasting with Al_2O_3 particles and chemical etching with 5:5 mixed solution of hydrofluoric acid and nitric acid.

Application of porcelain:

All the samples were veneered with porcelain on one side to a height of 4 mm. One sample from each group is left without veneering to study the surface topography under the Scanning Electron Microscope. The

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specimens were then fired in a ceramic furnace according to the manufacturer's instructions.

A special metal mold was prepared for sectioning which is sealed at both the ends. A three component resin (AMPSET, Cold Mounting Systems, and Turkey) was mixed and poured into the mold until the base of the mold was covered. After the final setting time of 24 hours cyanoacrylate adhesive(Mitreapel, Beta Chemical Industry, Istanbul, Turkey) was used to secure the specimens to the resin base to prevent mobility while the rest of the mold was filled with the resin. The samples were inside the sectioning machine and were sectioned by a rotating diamond coated disc under cold water irrigation. Sectioning of specimens was done until $1 \times 1 \times 14$ mm^3 microbars were obtained. The microbars of the first row were excluded due to the possibility of the defects. The dimensions of the microbars were examined using a digital caliper. Fifteen such microbars having identical sizes were chosen for micro tensile bond strength testing. They were fixed to the metal bars with cyanoacrylate glue with the zirconia ceramic interface at the junction of the two metal bars. The microbar with metal bars was attached to the opposing arms universal testing machine. A tensile load was applied to the microbars at a crosshead speed of 1 mm/ minute, and the maximum load upon fracture was recorded (Table no. 1).

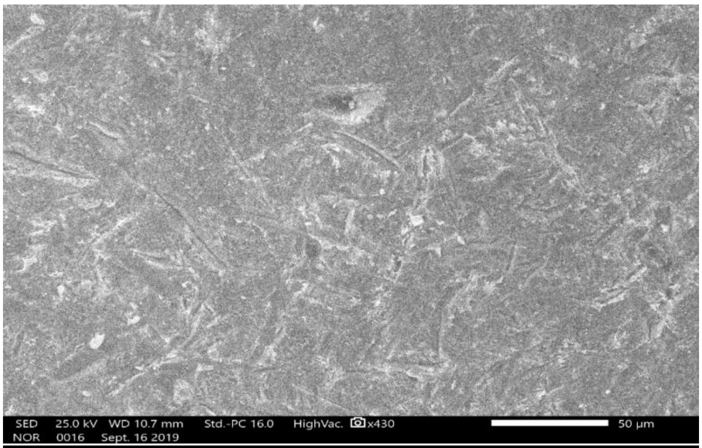

Figure no. 1: SEM analysis of surface of zirconium block with no surface treatment (Group 1)

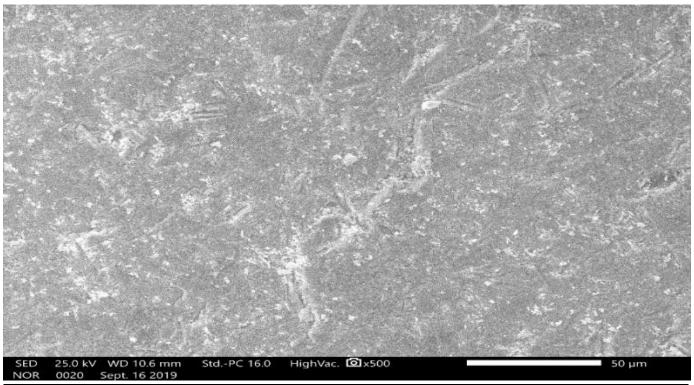

Figure no. 2: SEM analysis of surface of zirconium block surface treated with sand blasting (Group 2)

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Figure no. 3:SEM analysis of surface of zirconium block surface treated with chemical etching (Group 3)

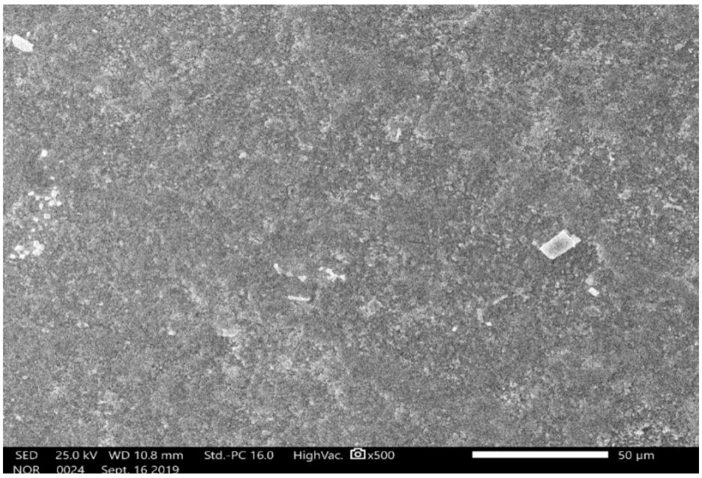

Figure no. 4: SEM analysis of surface of zirconium block surface treated with sand blasting and chemical etching (Group 4)

S. No.	Group I	Group II	Group III	Group IV
1.	30.80	36.56	33.45	45.23
2.	30.02	34.12	32.33	46.23
3.	29.70	38.56	37.78	44.12
4.	42.58	24.12	26.46	43.56
5.	41.06	32.18	31.22	48.23
6.	28.07	33.87	30.78	47.23
7.	24.96	42.58	38.41	44.89
8.	35.04	44.71	41.12	40.12
9.	32.01	38.12	33.46	39.12
10.	39.08	30.12	31.88	56.12
11.	24.07	29.32	28.52	50.67
12.	27.08	30.14	30.29	54.12
13.	20.36	38.24	37.46	46.12
14.	20.77	37.96	33.68	45.29
15.	37.15	34.78	32.89	45.01

Table no. 1: Micro tensile bond strength (MPa)

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Results

Statistical analysis:

One way ANOVA was used for the detection of possible statistically significant differences in the mean standard deviation of the micro tensile bond strength values of the four study groups. The highest and lowest mean values and standard deviation of micro tensile bond strength between four groups were given in Table 2 and Figure 5. The highest mean micro tensile bond strength value was measured in the combined sandblasting and chemical etching group (Group 4) (46.4 MPa), and the lowest value was measured in the control group (Group 1)(30.84 MPa)(Table no. 2). The micro tensile bond strength values in sandblasting and chemical etching groups were 35.004 MPa and 33.30 MPa, respectively (Figure no. 5). The results of one way ANOVA proved that there is significant increase in the mean microtensile bond strength value for group 4 compared to the remaining groups (Table no. 2).

Tuckeys multiple post hoc test indicates that there is no significant difference between the control group and sand blasting group as the p value is greater than 0.05(p = 0.1524). A comparision between control group and chemical etching group (p = 0.5887)and between sand blasting group and chemical etching groups(p = 0.8165) also shows no significant difference. On comparing combined sand blasting and chemical etching group(Group 4) with all the three remaining groups shows significant difference as p value is less than 0.05(Table no. 2). The SEM analysis of surface roughness shows highest roughness on group 4 samples (Figure no. 4) followed by group 2(Figure no. 2), group 3(Figure no. 3) and group 1(Figure no. 1) in their order of surface roughness.

Table no.2: Pair wise comparisons of four groups (Group 1, Group 2, Group 3 and Group 4) with Micro tensile bond strength byTukeys multiple posthoc procedures

Materials	Group 1	Group 2	Group 3	Group 4
Mean	30.85	35.03	33.32	46.40
SD	6.99	5.37	3.94	4.54
Group 1	-			
Group 2	p=0.1524	-		
Group 3	p=0.5887	p=0.8165	-	
Group 4	p=0.0002*	p=0.0002*	p=0.0002*	-

*p<0.05 indicates significant difference

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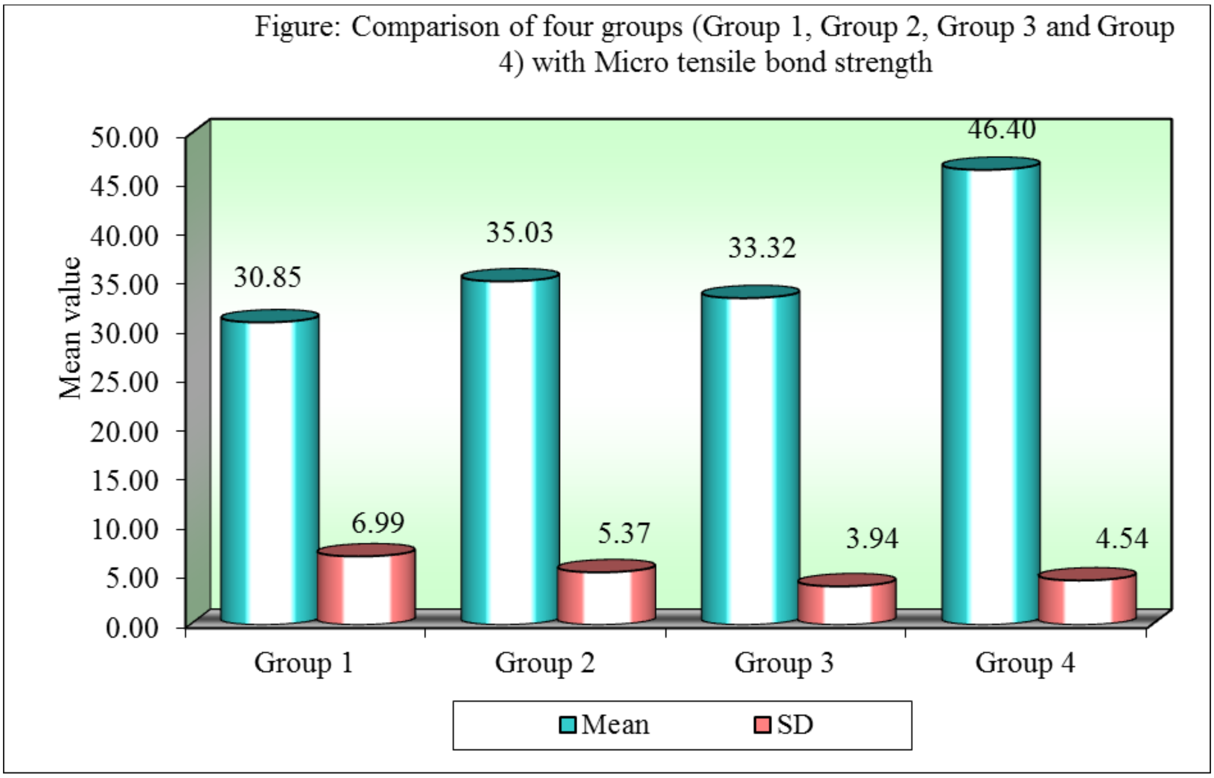

Graph no. 1: Comparison of four groups (Group 1, Group 2, Group 3 and Group 4) with Micro tensile bond strength.

Discussion

The purpose of the present study was to evaluate the efficacy of three different surface treatments of zirconia core and to compare the micro tensile bond strength at zirconia core – ceramic veneering interface among the treated surfaces. The studies on the bond strength of core-porcelain veneer have mostly used shear strength tests, 3-point/4-point flexural tests, or biaxial flexural tests, which are associated with the structural failure of the specimens [15]. The samples used in the shear test settings receive uneven stress during loading [16].

Application of micro tensile bond strength test on dental ceramics needs careful handling of the specimens to avoid the creation of structural defects [2].

Several variables such as the surface condition of the zirconia core, residual stress due to the difference in thermal expansion coefficient, interfacial flaws or defects, surface wettability and shrinkage of veneering material may affect the bond strength of zirconia – veneering porcelain [17].

Aboushelib et al. recommended that the difference in thermal expansion coefficient between zirconia core and veneer should be as small as possible [18].

The fracture behavior of the zirconia – veneer bilayer was evaluated through shear bond strength testing. Cohesive failure of the veneering material itself resulted from the inferior physical properties of the veneering porcelain and the low bond strength between the veneer and zirconia core[19].

Several approaches have been introduced to increase the surface roughness of ceramics to obtain satisfactory mechanical interlocking. Phark et al. [20] proposed a porous surface for zirconia ceramic, which significantly increased the longterm SBS of zirconia to composite resin. However, no studies testing this surface-modifying method for zirconia to porcelain bonding were identified.

Group 3 samples, as described by Yen et al., [21] hydrofluoric acid can react preferentially with the silica phase in a glassy matrix to form hexafluorosilicates. As a result, the surface of the ceramic becomes rough, which is required for micromechanical retention.

At low hydrofluoric acid concentrations, the crystalline phase could have greater durability [21].Therefore, using a lower concentration may cause less of an etching effect on the ceramic surface, thereby resulting in

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a lower surface roughness value [1]. Consequently a higher concentration of hydrofluoric acid is used in this study.
It might be assumed that airborne particle abrasion would enhance the bond strength of cores to veneering porcelains by increasing surface roughness and providing undercuts [12]. The samples of group 4, which were treated with both sandblasting with 110µ Al_2O_3 particles and chemical etched with 5:5 mixed solution of hydrofluoric acid and nitric acid showed the highest micro tensile bond strength which was supported by SEM analysis showing more roughness on their surface. Adding nitric acid increases the etching rate (Figure no. 4).

Conclusion:
There is a significant increase in the mean micro tensile bond strength between experimental groups and the control group. There is also a considerable increase in value in group 4 from groups 2 and 3. But there is no significant difference between group 2 and group 3.
The limitations of the present study include the following: the layered ceramic specimens did not represent the clinical shape of zirconia restorations, and the effects of aging conditions on the bond quality of zirconia to veneering porcelain were not evaluated. Therefore, further studies are needed.

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