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The effect of fusion sputtering surface treatment on microshear bond strength of zirconia and MDP-containing resin cement☆

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ABSTRACT

Purpose. The aim of this study is to evaluate the effect of fusion sputtering surface treatment on the microshear bond strength of zirconia and self-adhesive MDP-containing resin cement.

Materials. Thirty-six zirconia discs received one of the following treatments: fusion sputtering, airborne particle abrasion with 50-μm aluminum oxide particles, while as-sintered specimens served as a control. Four treated zirconia samples from each group were examined using 3D laser scanning microscope to assess the surface roughness and scanning electron microscope to study the surface topography. The specimens of each group were bonded to composite micro discs using MDP-containing self-adhesive resin cement (Panavia SA cement plus). The specimens were thermocycled for 5000 cycles between 5 and 55 °C. Microshear bond strength test was performed using universal testing machine until bonding failure. Failure modes and fracture surfaces was evaluated using scanning electron microscope.

Results. The fusion sputtering surface treatment significantly influenced zirconia-resin bond strength ($p < 0.001$). The highest mean microshear bond strength value was observed in fusion sputtering treatment (23.18 ± 4.38). The lowest value was observed in as-sintered zirconia surfaces (7.23 ± 6.26).

Significance. Fusion sputtering surface treatment enhanced the microshear bond strength of zirconia and resin cement.

1. Introduction

Indirect ceramic restorations made of alumina and zirconia have gained widespread popularity in the field of dentistry. Yttria-tetragonal zirconia polycrystal (Y-TZP) provides higher fracture toughness and strength compared to other dental ceramics [1]. The clinical success of resin bonding procedures for indirect ceramic restorations depends on the quality and durability of the bond between the ceramic and the resin cements [2]. Etching the ceramic surface with hydrofluoric acid (HF) and subsequent silanization of the silica-based

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ceramics is a well-established method to achieve durable bond of resin-based materials [3]. Unfortunately, neither acid etching nor applying silane-coupling agents has resulted in adequate resin bond to high-alumina [4–6] or zirconia ceramics [5–7]. The lack of a glassy matrix and the absence of SiO_2 make acid etching and silane application incapable of treating the zirconia surface [8,9].

Fusion sputtering described by Aboushelib 2012 is a new technique used to create a rough zirconia surface by spraying an air-water jet carrying microscopic zirconia particles onto unsintered zirconia; upon impact with the surface, the particles achieve good contact and adherence. After sintering, these particles become structurally fused with the underlying framework and create undercuts suitable for establishing mechanical retention with resin cements [10].

In order to simplify adhesive cementation procedures, various self-adhesive resin cements consist of phosphate monomers; including MDP have been developed [11,12].

Although several studies have evaluated the efficacies of surface treatment methods, there is still no consensus regarding the best surface treatment for achieving optimal bond strength between zirconia and resin cement [13].

Therefore, the aim of this study was to evaluate the effect of fusion sputtering method on the micro shear bond strength between Y-TZP ceramics and MDP-containing self-adhesive resin cement. The null hypnosis tested suggested that the fusion sputtering will not influence the micro shear bond strength of zirconia and resin cement.

2. Materials and methods

2.1. *Zirconia blocks preparation*

Thirty-six zirconia discs (11 mm × 13 mm) were prepared by manually cutting IPS e.max ZirCAD (Ivoclar Vivadent, Australia) using (MICRACUT 150, Milno, Italy). Before application of any treatment, the surfaces of the zirconia specimens to be treated were ground using 600-grit Al_2O_3 polishing paper under water to produce a smooth polished surface, and randomly divided into three groups. Each group contains 12 discs according to the surface treatment [14].

2.2. *Surface treatment of zirconia*

- Fusion Sputtering according to Aboushelib [10]: 5 g of unsintered zirconia powder were placed in a plastic capsule along with a 1-mm zirconia ball. The sealed capsule was placed in an electronic mixer for 45 min to allow fine grinding of the zirconia powder. Only 7–12 μm particles were selected by shaking the ground powder through fine stainless steel mashes. The process was repeated several times until 50 g of the required powder was obtained. Ten grams of the selected particles were added to a glass jar filled with 10 ml of 50% ethyl alcohol and the mixture was placed on an ultrasonic shaker to allow homogenous distribution of the particles. Immediately after mixing, the suspension was transferred to Air Brush (BADGER 155 ANTHEM U.S.A.) used to spray paint, and the air pressure was adjusted to 0.3 MPa. The adjusted ceramic spraying nozzle was kept at constant distance from the disk specimens (20 mm) using a plastic rod attached to the nozzle. A manual flow controller was used to maintain a constant spray. First, two short jets were released from the nozzle onto a black paper until a constant mixture was observed, then the surface of the zirconia discs were sprayed for 5 s. The sputtered zirconia discs were stored at 60 °C for 2 h to allow proper drying of the surface before sintering according to the manufacture's instruction (a sintering program at a maximum temperature of 1500 °C for 6 h) [10].
- After sintering, Airborne-particle abrasion; Using 50 μm aluminum trioxide particles, Al_2O_3 (S-U-Alustral, Schuler-Dental; Ulm, Germany) for 10 s at 10 mm distance and a pressure of 2 bar (Twin-Pen sandblaster).
- No treatment (as-sintered) served as control group.

2.3. *Surface structure and surface roughness assessment*

The treated surfaces of four extra zirconia samples were examined using 3D laser scanning microscope (Keyence VK-X100, Japan) to assess the surface roughness and a scanning electron microscope (JEOL JSM -5300 Scanning Microscope, Japan) to study the surface topography of each group.

2.4. *Bonding procedure*

All zirconia specimens were ultrasonically cleaned in 90% ethanol for 20 min to clean the surface.

Composite micro discs (Filtek Z250) were prepared by incrementally filling a silicon mold (2 mm × 2 mm), followed by light polymerization 20 s for each surface (Elipar™ free light 2, 3M-ESPE, St. Paul, MN, USA).

Six composite micro discs were cemented on each zirconia disc. The composite discs were cemented on the zirconia using MDP containing self-adhesive resin cement Panavia SA (Kuraray America, Inc., New York, NY) following the manufacturer's recommendations. Any residual cement was removed using a micro brush and the margins were light cured for 20 s per surface. The cement was cured under a 500 g constant load, for 5 min to assure complete setting of the cement. The complete zirconia sample holds six cemented composite micro discs (Fig. 1).

The specimens were stored in distilled water at 37 °C for 24 h, then thermocycled in water at temperatures between 5 and 55 °C for 5000 cycles [15].

2.5. *Micro shear bond strength test*

A copper mold to hold the zirconia sample and a shearing blade of 3 mm was fabricated. The micro shear test was applied on each composite micro disc cemented on the zirconia sample. The Micro shear bond strength was measured by applying an axial load on the bonded interface until failure by using a universal testing machine (AGS-X 5 KN Shimadzu Japan). Bond strength was calculated in MPa by dividing the load at failure by the adhesive surface area (mm^2).

Fig. 1 – Showing zirconia sample, which holds six composite micro discs cemented by PANAVIA SA resin cement.

2.6. Failure assessment using stereomicroscope and scanning electron microscope (SEM)

Following Micro shear testing, all fractured surface of the zirconia side were examined by stereomicroscope (OLYMPUS stereomicroscope sz11, Olympus Optical Co., Ltd., 2-43-2, Tokyo, Japan) and scanning electron microscopy (JEOL JSM-5300 Scanning Microscope, Japan) to identify the failure pattern and determine the micro morphological topography. The failure pattern was classified either as cohesive fracture, where the fracture line travelled through the adhesive resin or composite resin material, or adhesive fracture, where the fracture line travelled along the zirconia/resin interface leading to exposure of the zirconia surface.

3. Results

Laser scanning microscope revealed that Fusion sputtering increased surface roughness (Ra = 4.14 μm) of the treated zirconia surfaces (Fig. 2). Air born particle abrasion with 50-μm aluminum oxide also increased the surface roughness of the abraded zirconia surface (Ra = 0.68 μm) (Fig. 3). The as sintered zirconia revealed a smooth flat surface with average (Ra = 0.31 μm) (Fig. 4).

SEM images of the treated zirconia surfaces showed in Figs. 5–7. The fusion sputtering surface treatment resulted in the creation of high retentive beads made of zirconia dioxide particles attached to the treated surfaces (Fig. 5). Air born particle abrasion with 50-μm aluminum oxide resulted in creation of sharp scratches and grooves (Fig. 6). The as-sintered zirconia revealed a smooth flat surface (Fig. 7).

Regarding the microshear bond strength, the fusion sputtering surface treatment showed significant higher μ-SBS value than other surface treatments $p < 0.001$. Mean and stan-

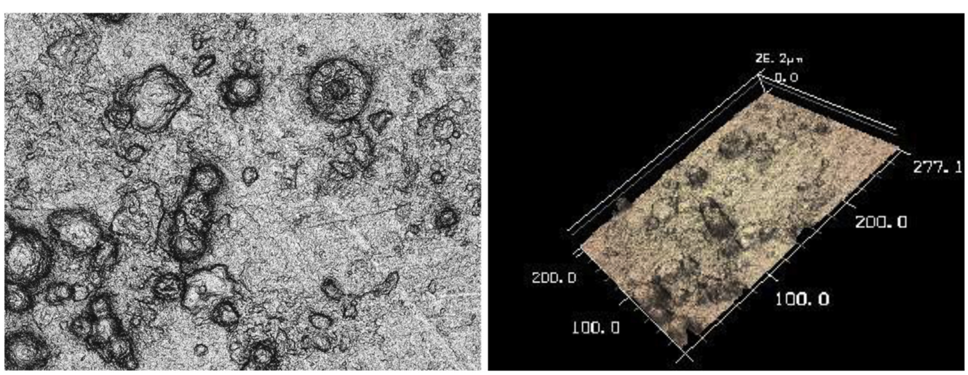

Fig. 2 – 3D laser microscope image of fusion sputtering treatment showing high retentive beads made of zirconia dioxide particles on the treated surfaces.

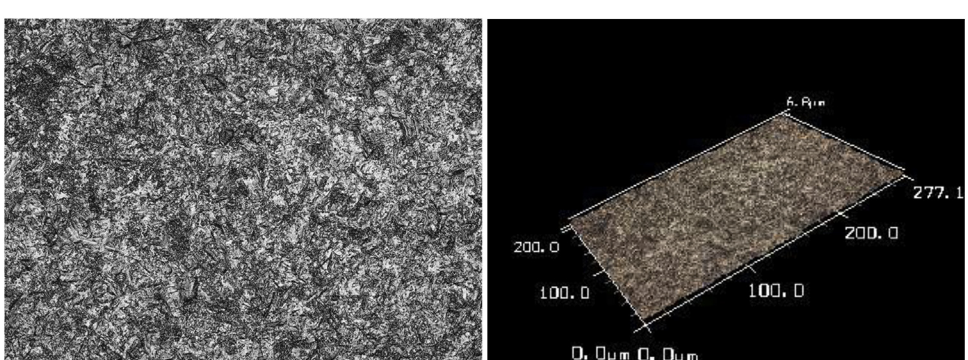

Fig. 3 – 3D laser microscope image of air abraded zirconia showing creation of sharp scratches and grooves.

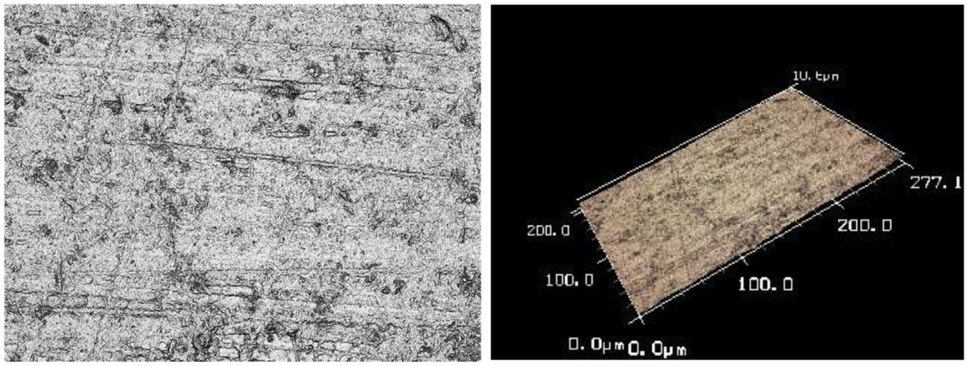

Fig. 4 – 3D laser microscope image of as-sintered zirconia showing smooth flat surface.

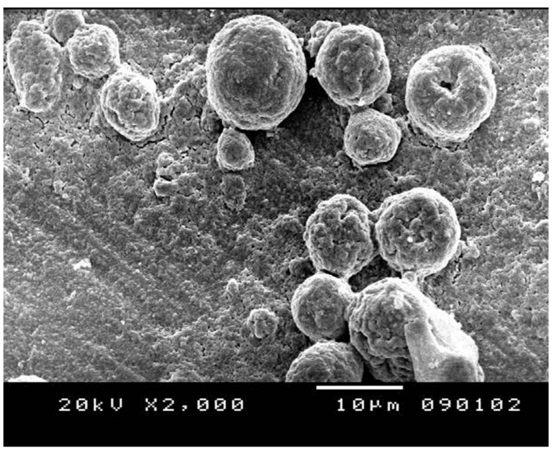

Fig. 5 – SEM image 2000×, demonstrating the fused beads created on the surface of fusion-sputtered specimens.

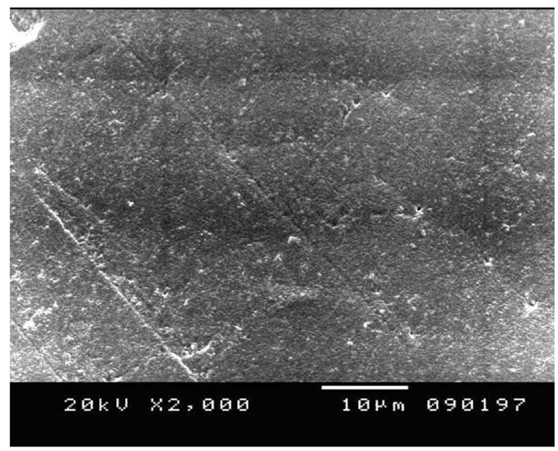

Fig. 7 – SEM image 2000×, demonstrating the as-sintered zirconia surface.

dard deviation (SD) of μ-SBS for different Surface treatments are summarized in (Table 1).

With stereomicroscope and SEM examination of the fractured surface of the zirconia (Table 2), 100% cohesive failure was found in the fusion sputtering group, 80% cohesive failure was found in air abrasion group, 100% adhesive failure was found in the as-sintered group.

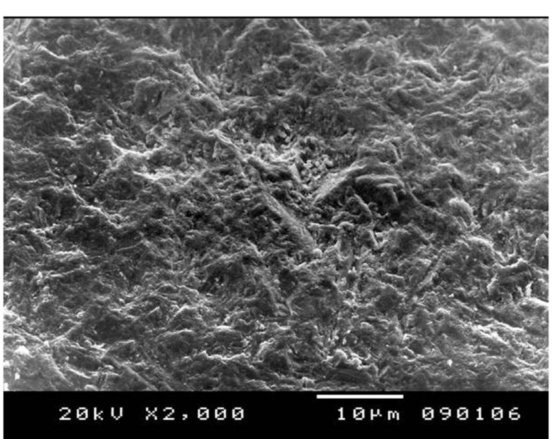

Fig. 6 – SEM image 2000×, demonstrating the air abraded zirconia surface.

Table 1 – Mean and standard deviation (SD) for Bond strength for different surface.

	Surface treatment						p-Value
	FS		AA		AS		
	Mean	SD	Mean	SD	Mean	SD	
Bond strength	23.18[a]	4.38	17.60[b]	5.59	7.23[c]	6.26	<0.001*

Means with the different letter indicates significant difference $p \leq 0.05$.
* Statistically significant at $p \leq 0.05$.

Table 2 – Failure type of different test groups.

Surface treatment	Pretest failures	Failure type
As-sintered (control)	6	100% Adhesive
Air abrasion	0	80% Cohesive
Fusion sputtering	0	100% Cohesive

4. Discussion

Numerous factors affect the bond durability of resin cement to zirconia ceramics, these include roughness of the ceramic surface, the ability of resin cements to wet the zirconia surface, composition of the resin cements, possible contamination during bonding procedures and cleaning methods of the restoration luting surface [16].

This in-vitro study was an attempt to evaluate the effect of fusion sputtering on the micro shear bond strength of zirconia and MDP containing self-adhesive resin cement (Panavia SA). A significant increase in the μ-SBS was found by surface treatment of zirconia with fusion sputtering or air particle abrasion. Thus the null hypnosis of this study was rejected.

In the present study as regard the results of surface roughness; fusion sputtering surface treatment produced the highest Ra value (4.14 μm) through the formation of high beads of zirconia oxides particles that became attached with the zirconia surface after sintering. This result is consistent with the study of Aboushelib [10], where Ra of the fusion sputtering was (3.7 μm).

A point that must be considered is that air-abrasion might compromise the mechanical strength of ceramic itself by initiating surface defects [17]. The critical defect size in zirconia ceramic varies between 15 and 40 μm [18]. The surface-produced damage resulted in a significant reduction in flexural strength of zirconia and in reduction in its fatigue resistance as well. The deteriorating effect becomes more evident when coarse aluminum oxide particles are used (100–120 μm) [19]. However, in a systematic review 2016, Aurelio et al. concluded that the flexural strength of Y-TZP is enhanced by airborne-particle abrasion, independent of particle size, blasting time, air pressure, and the presence of aging [20].

Regarding the present study results of μ-SBS test for different surface treatment, the results of μ-SBS test were consistent with the results of the surface roughness. The control group, in which the zirconia surface was polished and received no further treatment, presented the smallest value of bond strength. The highest bond strength value achieved in fusion sputtering surface treatment followed by air particle abrasion. This indicated that the surface treatment can influence the surface topography and increase the average surface roughness of the zirconia thus improve bond strength of luting agents to the ceramic substrate [21]. The created surface beads in the fusion sputtering samples become part of the framework and create 3-dimensional undercuts suitable for establishing micromechanical retention with the resin cement. Additionally, fusion sputtering increased the surface area of the bonding surface, which explains the high microshear values between resin cement and fusion-sputtered specimens.

The phosphate ester group of the adhesive monomers and zirconia oxides chemically creates direct bonds [22–24]. The use of Panavia SA that contains MDP has bifunctional ends consisting of long organic hydrophobic chain molecules. Hydrophilic phosphate ester groups at one end bond strongly to Y-TZP, and vinyl groups react with the monomers of the resin cement at the other end [25,26]. In agreement with other studies, this study showed that phosphate monomers are chemical agents that provide durable bond strength to zirconia [2,4,16,27,28].

The specimens modified by air abrasion had micro-shear bond strength value of 15.03 ± 5.09 MPa. Airborne particle abrasion promotes the formation of resin-ceramic micromechanical interlocking by increasing the bonding area, inducing an activated micro-roughened zirconia surface, and modifying wettability and surface energy of the ceramic surface [5,15,13,29–34]. This results consistent with many studies that concluded that particle abrasion combined with phosphate monomers improves resin bonding to zirconia [22,35,36].

The results of shear bond strength test of the present study are consistent with the failure modes observed. The groups with the highest microshear bond strength showed cohesive failure mode. Fusion sputtering showed cohesive failure, which may be due to the combined effect of the increased contact area with the Y-TZP ceramic surface and the improved chemical interaction with the MDP in the resin cement. Air particle abrasion showed mainly cohesive failure mode as the most common type of failure mode with 20% adhesive failure. As-sintered group showed lower bond strength mean values than other surface treatments and adhesive type failures.

5. Conclusion

Within the limitation of this study we concluded that fusion sputtering and air abrasion increased the surface roughness of zirconia and significantly influenced the bond strength between zirconia and self-adhesive resin cement.

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