

# The Impact of Plasma Treatment of Cercon® Zirconia Ceramics on Adhesion to Resin Composite Cements and Surface Properties



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Published online 29 August 2017



## Abstract

**Introduction:** In recent years, the use of ceramic base zirconia is considered in dentistry for all ceramic restorations because of its chemical stability, biocompatibility, and good compressive as well as flexural strength. However, due to its chemical stability, there is a challenge with dental bonding. Several studies have been done to improve zirconia bonding but they are not reliable. The purpose of this research is to study the effect of plasma treatment on bonding strength of zirconia.

**Methods:** In this in vitro study, 180 zirconia discs' (thickness was 0.85-0.9 mm) surfaces were processed with plasma of oxygen, argon, air and oxygen-argon combination with 90-10 and 80-20 ratio (n=30 for each group) after being polished by sandblast. Surface modifications were assessed by measuring the contact angle, surface roughness, and topographical evaluations. Cylindrical Panavia f2 resin-cement and Diafill were used for microshear strength bond measurements. The data analysis was performed by SPSS 20.0 software and one-way analysis of variance (ANOVA) and Tukey test as the post hoc.

**Results:** Plasma treatment in all groups significantly reduces contact angle compare with control ( $P=0.001$ ). Topographic evaluations revealed coarseness promotion occurred in all plasma treated groups which was significant when compared to control ( $P<0.05$ ), except argon plasma treated group that significantly decreased surface roughness ( $P<0.05$ ). In all treated groups, microshear bond strength increased, except oxygen treated plasma group which decreased this strength. Air and argon-oxygen combination (both groups) significantly increased microshear bond strength ( $P<0.05$ ).

**Conclusion:** According to this research, plasmatic processing with dielectric barrier method in atmospheric pressure can increase zirconia bonding strength.

**Keywords:** Plasma laser; Zirconia ceramics; Resin composite; Surface properties.

## Introduction

Nowadays, applications of ceramic based zirconia in restorative dentistry due to its esthetic and mechanical properties have been widespread.<sup>1,2</sup> Martensitic transformation of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics causes high flexural strength and toughness compared to other dental ceramic materials.<sup>3-6</sup> Despite these advantages, it is hard to obtain a proper bonding to other substrates because of its hydrophobic surface.<sup>7,8</sup>

Several studies have shown that unmodified surfaces display low bond strength and eventually leads to adhesive failure,<sup>9-11</sup> so there is a need to intensify surface properties of these ceramics to reach an effective bonding.<sup>12</sup> These surface modifications are based on roughening the surface and increasing its wettability.<sup>13-16</sup> Conventional adhesion

techniques used for silica-based ceramic (such as etching by hydrofluoric acid) cannot be used for zirconia ceramics, because it fails to achieve adequate surface roughness and will lead to poor bonding strength.<sup>10,17-19</sup>

The airborne  $Al_2O_3$  particle abrasion of zirconia ceramics surface has been found an effective method for achieving durable ceramic-resin bonding. This method increases the surface roughness and wettability, which leads to mechanical and chemical bonding between resin and ceramic surface.<sup>20-24</sup> However, it has been reported that particle abrasion by alumina can create mechanical damage on the surface of ceramic, which could affect bonding performance in long term.<sup>25-27</sup> To deal with this problem, it is recommended to use softer and rounder abrasive particles<sup>26</sup> or omitting these particles.<sup>28</sup>

To avoid these problems, researchers tried to find

alternative surface treatment for improving bonding performance of zirconia ceramics: selective infiltration etching,<sup>29</sup> chlorosilane combined with water vapor,<sup>7</sup> the fluorination vapor techniques,<sup>24</sup> ion bombardment,<sup>15,30</sup> and plasma-coating techniques.<sup>15,31,32</sup>

Recently, application of plasma in dentistry has thrived specially as an effective tool for surface enhancement.<sup>32</sup> It has been suggested that plasma modification of surfaces can improve zirconia-resin bonding performance by changing functional groups on the surface creating reactive sites on surfaces.<sup>31,33</sup> Valverde et al reported that application of non-thermal plasma for zirconia ceramic surface treatment significantly increases the micro tensile bond strength.<sup>31</sup>

The objectives of the present study were to (1) evaluate surface properties of Cercon® zirconia ceramics, (2) specify the impact of non-thermal plasma on zirconium bonding strength to resin composite cements.

## Methods

### Specimen Preparation

Nine blocks of Cercon® base colored 47 blocks (DeguDent, Hanau, Germany) were cut by low speed diamond saw (Secotom-50, Struers, Ballerup, Denmark) in 180 discs (thickness was 0.85-0.9 mm) and were polished in 3 sandblasting phases by 800 grit, 1500 grit, 3000 grit, respectively. According to the factory's data, these ceramics have 5% yttrium oxide, below 2% hafnium oxide, and below 1% aluminium oxide and silicon oxide. The discs surfaces were washed for 5 minutes with distilled water in ultrasonic machine and sintered after one-day drying; the thickness was 0.75 mm in this phase. Sample coarseness increased after sintering. The surfaces were polished with 1000 grit emery to make a flat sample surface. Prior to surface modification treatments, samples were washed after polishing with acetone, iso-propanol, and distilled water in 3 phases for 5 minutes in ultrasonic set.

The specimens were placed in reactor in order to surface modification via plasma. Plasma treatment was performed based on their experimental groups, with a low density cold active inert argon, oxygen, and air gas plasma beam for 20 seconds over a distance of 10 millimeters. There were 6 study groups based on treatments:

- 1) ceramics treated by oxygen plasma
- 2) ceramics treated by argon plasma
- 3) ceramics treated by plasma of air
- 4) ceramics treated by 20% oxygen and 80% argon combination
- 5) ceramics treated by 10% oxygen and 90% argon combination
- 6) untreated ceramics (control).

Tektronix DPO 3012 oscilloscope (Beaverton, Oregon, USA), Tektronix TCP 202 flow probe (Beaverton, Oregon, USA) and Tektronix p6015 (Beaverton, Oregon, USA) high voltage probes were used to measure the input voltage for producing plasma.

### Contact angle measurement

In order to measure contact angle in specimens (n=60), the sessile drop technique was performed by a micrometer set (EasyDrop, Kruss, Germany) using 2 different liquids with different polarities (distilled water and diiodomethane). Before this test, the 60 specimens were randomly allocated into 6 groups, with 5 of them treated by plasma (n=50) and 1 group untreated (n=10). After 5 seconds, a CCD camera made digitalized standard photos. We used ImageJ software (National Institutes of Health, Bethesda, Maryland, USA) and a plugin (DropSnake) for image processing, which was previously described by Stalder et al.<sup>34</sup>

### Topographical Evaluation and Surface Roughness Measurement

Phase imaging of the discs used was performed in the previous step (n=30) via an atomic force microscopy (easyscan 2, Nanosurf AG, Liestal, Switzerland) to assess the topography and coarseness of discs' surfaces. Each specimen's surface was scanned in 2 points at the center, 2 points at the perimeter, and 2 points between the center and perimeter. 256 × 256 resolution and 1 Hz scan rate were utilized to obtain topography on a 90 μm × 90 μm area. The accuracy of this setting is lower than 0.5 nm in Z axis and was 1 nm in XY axis. Prior to the scanning, all surfaces were blown through with cold air and cleaned by alcohol.

### Microshear Bond Strength Measurement

After surface processing, a cemented probe was applied to the sample to perform micro tensile and cutting bond strength tests in remaining disks (n=120). PANAVIA F 2.0 resin composite cement (Kuraray, Okayama, Japan) was filled in custom made tygon silicone tubes with an inner diameter 1.14 mm and 1 mm height on top of each disc. After light curing (Flashsoft, CMS dental, Denmark) for 40 seconds, the resin blocks were incubated at 37°C for 24 hours to achieve maximum chemical polymerization. Micro tensile tester (Bisco, Schaumburg, IL, USA) with tension velocity of 1 mm/min was used in this study. Micro-shear bond strengths were calculated by dividing peak load by the cross-sectional area of the composite cylinder. A stereomicroscope (Karl Zeiss, Germany) was used to evaluate bonding failure at ×10 magnification.

### Statistical Analysis

Data analysis was performed by SPSS 20.0 software (IBM Corp., Armonk, NY, USA). One-way analysis of variance (ANOVA) at a 0.05 confidence level was used to compare bonding strength data and Tukey test as the post hoc.

## Results

### Contact Angle

Table 1 shows descriptive statistics of treated groups and untreated group (control). Plasma treatment in all groups significantly reduces contact angle compare with control ( $P=0.001$ ). Also, the comparisons between groups showed

**Table 1.** Means of Contact Angle in Different Plasma Treated Groups and Without Treatment Group (Control)

Study Groups	Mean	Standard Deviation
Control	63.00	2.000
Oxygen	32.00	4.000
Air	27.00	1.000
Argon	33.00	3.000
10% Argon-90% Oxygen	34.00	1.000
20% Argon-80% Oxygen	23.00	2.000

**Table 2.** Means of Surface Coarseness in Different Plasma Treated Groups and Without Treatment Group (Control)

Study Groups	Mean	Standard Deviation
Control	8.00	0.000
Oxygen	23.03	1.000
Air	10.00	0.000
Argon	4.00	0.000
10% Argon-90% Oxygen	14.00	0.000
20% Argon-80% Oxygen	17.00	0.000

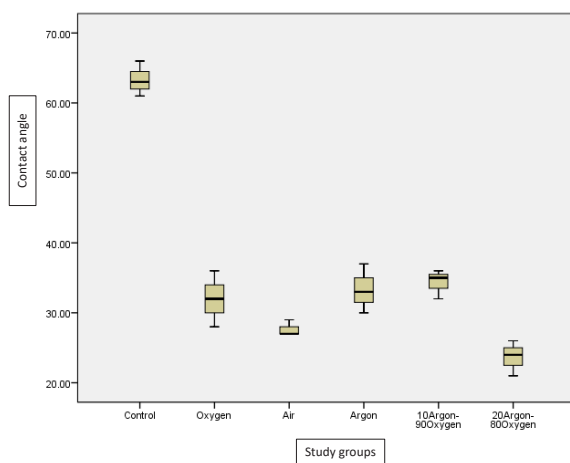
significant difference ( $P < 0.05$ ) except between air plasma and 10% argon-90% oxygen plasma ( $P = 0.094$ ). Figure 1 shows the boxplots of contact angles in study groups.

**Surface Roughness**

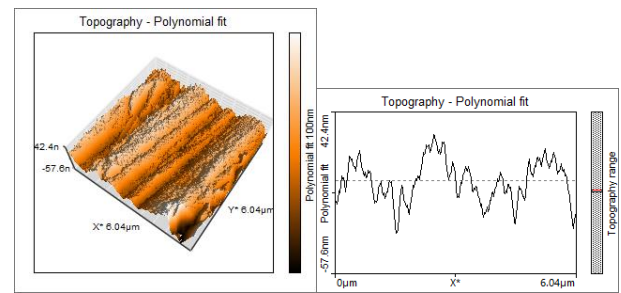
Descriptive statistics of study groups are presented in Table 2. Topographic evaluations of each group are showed in Figure 2 to 6. Coarseness promotion occurred in all plasma treated groups which was significant when compared to control ( $P < 0.05$ ) except for the argon plasma treated group that significantly decreased surface roughness ( $P < 0.05$ ). The boxplots of surface roughness are showed in Figure 7.

**Microshear Bond Strength**

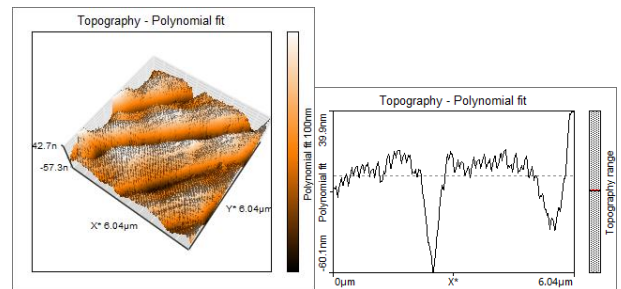
In all treated groups, microshear bond strength increased, except for the oxygen treated plasma group which



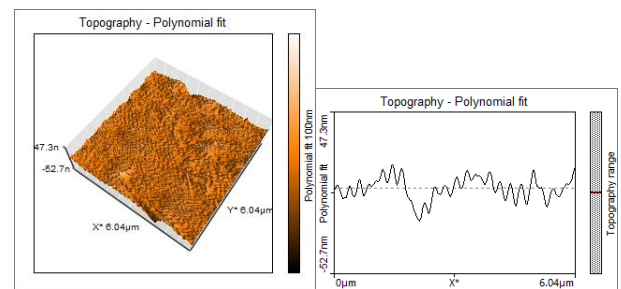
**Figure 1.** Boxplots for Contact Angles (Degree) of Different Plasma Treated Groups and Without Treatment Group (Control).



**Figure 2.** Topographical Evaluations of Specimens' Surface of Untreated Group (Control).



**Figure 3.** Topographical Evaluations of Specimens' Surface of Argon Plasma Treated Group.

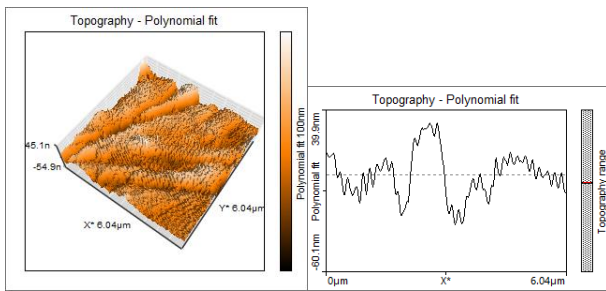


**Figure 4.** Topographical Evaluations of Specimens' Surface of Air Plasma Treated Group.

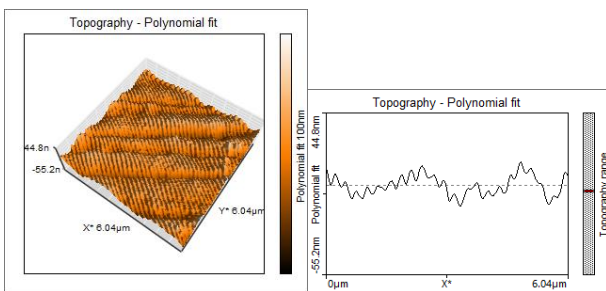
decreased this strength (Table 3). Air and argon-oxygen combination (both groups) significantly increased microshear bond strength ( $P < 0.05$ ). Figure 8 shows the boxplots of means of microshear bond strength in different plasma treated groups and without treatment group.

**Discussion**

Due to mechanical properties of ceramic based zirconia, obtaining appropriate bonding of Y-TZP in dental procedures is difficult.<sup>7,11,35</sup> Several treatments have been used in studies to improve bonding properties of these materials.<sup>7,15,20,22,24,29,30,36,37</sup> Recently, it has been suggested that the application of plasma can lead to enhanced zirconia-resin bonding by making changes on the surfaces of Y-TZP.<sup>31-33</sup> In the present study, we assessed the effects of various types of non-thermal plasmas on Y-TZP in terms of surface properties and bonding strength.



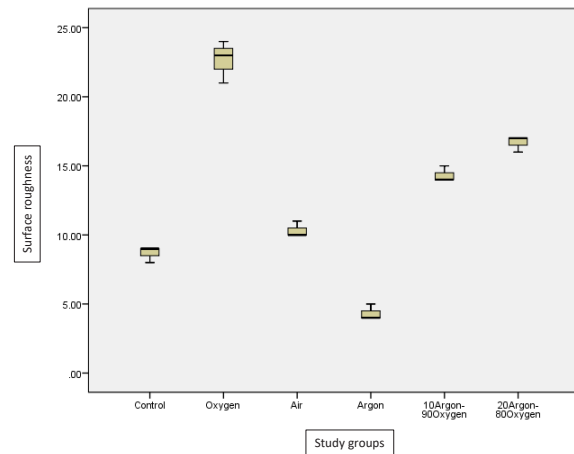
**Figure 5.** Topographical Evaluations of Specimens' Surface of Oxygen Plasma Treated Group.



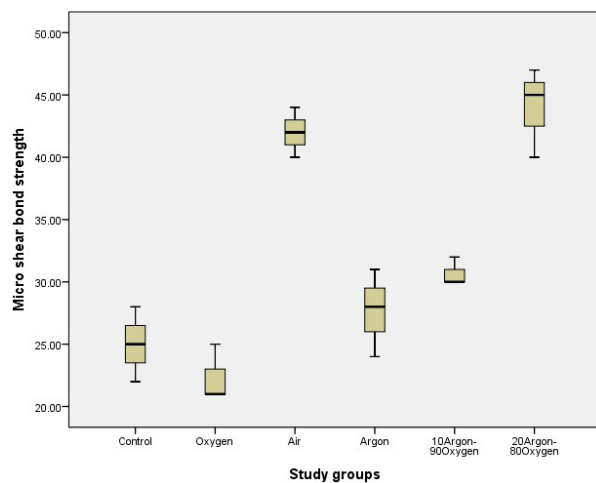
**Figure 6.** Topographical Evaluations of Specimens' Surface of Combination of Argon and Oxygen Plasma Treated Group.

Surface energy (SE) is a term that expresses the intermolecular forces on the surface of a material and it depends on the values of the polar and disperse components of surface.<sup>38</sup> Greater value of the SE leads to better wettability of the surface and better bonding properties of the material.<sup>39</sup> A common way for calculating the SE of a surface is contact angle measurements of liquids which amount has a reverse relationship with SE.<sup>38</sup> The findings of the present study indicated that all kinds of plasma treatments reduced the contact angle of zirconia. These findings are consistent with Noro et al<sup>40</sup> and Valverde et al<sup>31</sup> studies. In Valverde et al<sup>31</sup> study, it has been suggested that the reason for this phenomenon is that after plasma application, O elements in the surface will increase which leads to higher polarity. It also has been reported that gas of the plasma increases the formation of the active peroxide radicals and excessive functional groups (such as C-O and C-OH) on the treated surfaces of inert materials like Y-TZP, which cause higher SE.<sup>41,42</sup> Formation of oxygen functional groups on zirconia after using plasma was confirmed by XPS analysis in Noro et al<sup>40</sup> study. Due to higher polarity which could result in greater SE value and wettability, the adhesion properties and bonding strength of zirconia will be enhanced by the application of plasma.<sup>31,39,43</sup>

In the field of microshear bond strength, air and argon-oxygen groups increased microshear bond strength, but there were no significant differences between control group and other study groups. In Derand et al study,<sup>10</sup> it has been reported that shear strength between zirconia surface and resin intensified by the RF plasma treatment. Also,



**Figure 7.** Boxplots for Surface Roughness (nm) of Different Plasma Treated Groups and Without Treatment Group (Control).



**Figure 8.** Boxplots for Microshear Bond Strength (MPa) of Different Plasma Treated Groups and Without Treatment Group (Control)

**Table 3.** Means of Microshear Bond Strength in Different Plasma Treated Groups and Without Treatment Group (Control)

Study Groups	Mean	Standard Deviation
Control	25.05	3.00125
Oxygen	22.00	2.08986
Air	42.00	2.04158
Argon	27.00	3.00000
10% Argon-90% Oxygen	30.00	1.05276
20% Argon-80% Oxygen	44.00	3.00000

It et al<sup>44</sup> suggested that the application of atmospheric-pressure low temperature plasma enhance shear bonding strength of zirconia. As it has been mentioned before, application of plasma can enhance bonding strengths by increasing the SE value. Another mechanism for this improvement, is that gas plasma treatment removes organic contamination of zirconia by breaking C-H and C-C bonds.<sup>31,45</sup>

As previously mentioned, the airborne Al<sub>2</sub>O<sub>3</sub> particle abrasion of zirconia ceramics surface has been reported

as effective treatment in some literatures.<sup>20-24</sup> Ito et al<sup>44</sup> compared this treatment with gas plasma treatment. They reported that these treatments both increased the shear bond strength but there were no significant differences between them. They suggested that Al<sub>2</sub>O<sub>3</sub> particle abrasion affect crystal structure of zirconia and generate micro-cracks which leads to higher SE and better wettability and bonding properties.

One of advantages of this study was performing the process in a simpler environment. The reactor used in this study was cheaper and easily made. This reactor works in atmosphere pressure and therefore do not need vacuity equipment. This study used direct plasma processing in which ions together with electrons have great role in making reactions, while indirect processing has lower efficiency. Using air as a plasma environment producing gas was the next benefit that results in isolated chamber deletion from reactor and this can lead to easier processing in shorter time.

### Conclusion

Based on our findings, it could be concluded that; (1) plasma processing of Y-TZP with oxygen, argon, air and the combination of oxygen and argon decreases the contact angle and in other words increases the SE. (2) Plasma processing of Y-TZP with above gases decreases the coarseness of surface. (3) Plasma processing of Y-TZP increases the shear bond strength using air and the combination of oxygen and argon as plasma gases. Since we conducted bonding immediately after treatment and then assessed the bonding properties of Y-TZP surface, we suggest that further studies investigate long term effect of plasma treatment of Y-TZP because the active surface state changes over time.

### Ethical Considerations

The proposal of study was approved by the Ethics Committee, Deputy of Research, Shahid Beheshti University of Medical Sciences.

### Conflict of Interests

Authors declare that they have no competing interests.

### Acknowledgements

The authors would like to thank the Research Deputy of Shahid Beheshti University of Medical Sciences for financially supporting this study.

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