# Zirconia-Composite Bonding After Plasma of Argon Treatment

Luigi Canullo, DDS, PhD[a]/Costanza Micarelli, DDS[a]/Laura Bettazzoni, CDT[a]/
Brunilda Koçi, DDS, PhD[b]/Paolo Baldissara, MD, DDS[c]

***Purpose:*** To compare the shear bond strength (SBS) values of resin cement to zirconia treated with a new activating method. ***Materials and Methods:*** Forty-five zirconia specimens were divided into three groups: no treatment (group 1), plasma of argon cleaning for 375 seconds (group 2), and plasma of argon cleaning for 750 seconds (group 3). Composite cylinders were bonded with a self-adhesive cement. After 40 days of water storage, specimens were subjected to the SBS test. Data were analyzed with one-way analysis of variance and the Neuman-Keuls multiple comparison test. ***Results:*** Test groups obtained SBS values significantly higher (101% for group 2 and 81% for group 3) than controls. ***Conclusion:*** Plasma of argon appeared to improve bonding between zirconia and resin cement. *Int J Prosthodont 2014;27:267–269. doi: 10.11607/ijp.3686*

Different problems with zirconia restorations have been reported at the core-ceramic interface (veneering ceramics chipping or delamination) and at the core-cement interface (loss of retention).[1] For this last complication, different causes have been reported, including: (1) convergence angles of abutment preparation higher than 12 degrees are recommended for obtaining easier scanning and milling processes but decrease the mechanical retention,[2] and (2) the lack of adhesion between zirconia and commonly used cements (the absence of silica phase makes zirconia not etchable). In addition, the hydrophobic behavior of zirconia causes low wettability of zirconia surfaces by the adhesive cements commonly used.[3] To improve superficial adhesion of zirconia, different modalities and conditioning/cementing materials have been suggested.

The purpose of this study was to investigate the influence of plasma of argon treatment on the adhesion properties of yttria tetragonal zirconia polycrystal (Y-TZP zirconia) ceramic to composite cement. The shear bond strength (SBS) values of untreated and plasma-treated specimens were statistically compared.

## Materials and Methods

Forty-five tablets of densely sintered Y-TZP zirconia (Echo, Sweden-Martina) were air-abraded, using 110 µm aluminum oxide at 2 bar pressure, and distributed in three groups according to the surface treatment: steam cleaned at 8 bar (group 1), plasma of argon cleaning for 375 seconds (group 2), and plasma of argon cleaning for 750 seconds (group 3).

Plasma treatment was performed using a plasma reactor (Yocto, Diener Electronic) with argon at 75 W of power and 10 MPa of pressure (Fig 1).

A primer containing phosphate monomer (Z Primer Plus Bisco) was applied on the specimen. Then a composite cylinder was cemented with a dual cure, self-adhesive cement (BisCem, Bisco) (Fig 2).

After water storage (40 days at 37°), the SBS test was performed using an Instron machine (4465, Instron) with a 0.5-kN load at a descending speed of 0.5 mm/minute until debonding. Specimens were then observed under a stereomicroscope (Wild M10, Leica) to describe the failure pattern as adhesive, mixed adhesive, mixed cohesive, or cohesive (Fig 3).

Data were analyzed with one-way analysis of variance and the Neuman-Keuls multiple comparison test. The null hypothesis was that the SBS mean values of the plasma-treated groups were not statistically different from the control group ($\alpha$ = .05).

[a]*Independent Researcher, Rome, Italy.*
[b]*Researcher, Department of Prosthodontics, University of Tirana, Tirana, Albania.*
[c]*Professor, Department of Oral Sciences, Section of Prosthodontics, Alma Mater Studiorum, University of Bologna, Bologna, Italy.*

***Correspondence to:*** *Costanza Micarelli, via Gregorio VII 416 00165 Rome, Italy. Email: costanza@micarelli.eu*


**Fig 1** Yoko reactor working at 75 W and pressure of 10 MPa.

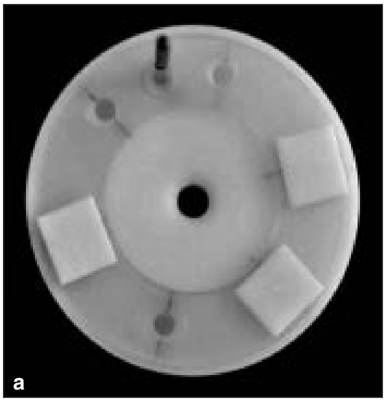

**Fig 2** Specimen preparation. **(a)** Tablets were placed on the lower part of a polyammide mold. **(b)** After primer application, the upper piece of the mold was positioned and secured using a central screw, then a layer of luting cement was placed in the cavity (3.3 mm in diameter) and light cured. After polymerization, the cavity was filled with composite and light cured again.

**Table 1** SBS Values (MPa) and Percentage of Improvement*

| Group | Mean | SD | SBS improvement (%) |
|---|---|---|---|
| 1 (control) | 8.54$^{B}$ | 4.1 | 0 |
| 2 (plasma treated for 375 s) | 17.25$^{A}$ | 3.1 | 101 |
| 3 (plasma treated for 750 s) | 15.46$^{A}$ | 4.8 | 81 |

SBS = shear bond strength.
*Groups with the same superscript letter are not statistically different. One-way analysis of variance and Neuman-Keuls multiple comparison test; f = 19.1; $P < .0001$.

**Table 2** Frequency Distribution of the Failure Patterns as Determined by Stereomicroscope Analysis*

| Group | Adhesive (%) | Mixed adhesive (%) | Mixed cohesive (%) | Cohesive (%) |
|---|---|---|---|---|
| 1 (control) | 33.3 | 20.0 | 40.0 | 6.7 |
| 2 (plasma treated for 375 s) | 0 | 60.0 | 33.3 | 6.7 |
| 3 (plasma treated for 750 s) | 0 | 73.3 | 26.7 | 0 |

*Note the absence of pure adhesive failure patterns in plasma-treated groups, which suggests that the increase of the SBS in plasma-treated specimens is due to a stronger zirconia-cement interface bonding.

## Results

Groups 2 and 3 showed statistically higher SBS values compared with group 1 (Table 1); thus, the null hypothesis was rejected. In groups 2 and 3, adhesive failures were absent, but were 33% in group 1 (Table 2). In group 2 (375 seconds), the percentage of mixed cohesive and cohesive failures was slightly higher than in group 3 (750 seconds).

## Discussion

The present study showed that the plasma cleaning allows for a statistically higher strength at the zirconia-cement interface compared to the untreated sample.

The different fracture modalities confirmed this assumption: among untreated specimens, a consistent amount of adhesive fractures (between zirconia and cement) was found (Fig 3). Controversially, among treated specimens, most detachments were adhesive/cohesive or pure cohesive (within the cement bulk, with part of the cement remaining attached to the zirconia) (Fig 4). The difference in SBS values between groups 2 and 3 was not statistically significant.

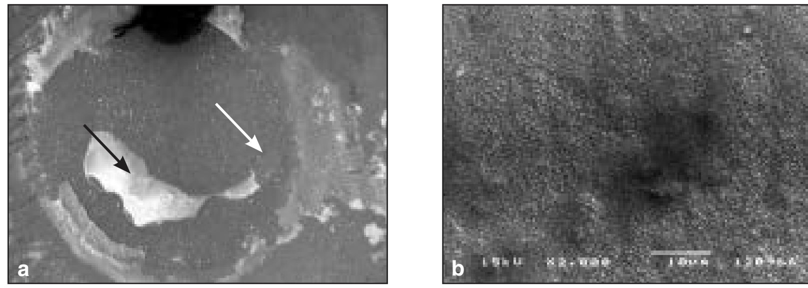

**Fig 3 (a)** Stereomicroscope and **(b)** SEM image of a control specimen that had a mixed adhesive failure pattern. The gray material *(white arrow)* partially covering the zirconia surface in the stereomicroscope image is the cohesively failed cement. The white material, which lies on the cement layer, was part of the bulk of the composite cylinder. The brilliant areas *(black arrow)* where zirconia is exposed correspond to the adhesively failed interface. The SEM image shows a zirconia exposed area where adhesive interface failed. Zirconia grains are clearly visible and some cement remnants are still on the ceramic surface.

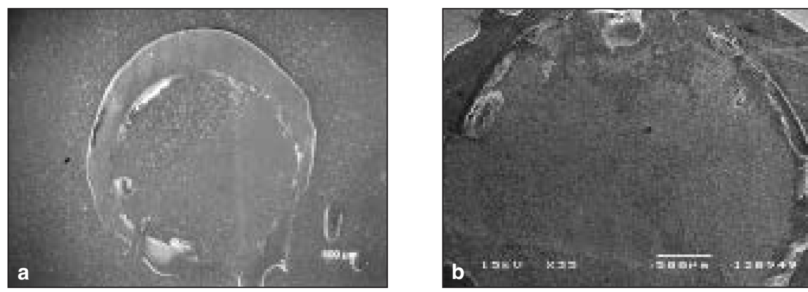

**Fig 4** A mixed-cohesive fracture pattern has been attributed to a group 2 specimen. The brilliant areas in **(a)** the stereomicroscopy image are adhesive failure sites. In the **(b)** SEM image, remnants of cement on the zirconia surface are clearly visible.

In this study, a dual auto adhesive cement was used with primer containing MDP (3-trimethoxysilylpropyl methacrylate), whose efficacy has been demonstrated. Since MPD directly interacts with zirconia, a deeply cleaned surface with increased wettability (as results from plasma cleaning treatment) might allow for superior primer behavior.[4]

However, to explain stronger adhesion, an additional advantage of plasma cleaning treatment could be advocated: unlike air-abrasion and tribochemical treatment, plasma cleaning does not produce pollutions or affect zirconia surface configuration. This might positively affect the bond strength and its long-term stability.[5]

SBS results achieved in the present study might be difficult to compare with other studies because of different aging protocols. In fact, in the present study a prolonged water storage was performed.

## Conclusions

Within the limitation of this laboratory study, the following conclusions can be drawn: *(1)* plasma of argon cleaning appeared to be effective in increasing adhesion between zirconia and composite cement and *(2)* the results obtained in this study may not be directly comparable to data present in the literature, due to differences in materials, techniques, and protocols. Further studies on plasma sources are needed.

## Acknowledgments

The authors reported no conflicts of interest related to this study.

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