



# Effect of atmospheric plasma versus conventional surface treatments on the adhesion capability between self-adhesive resin cement and titanium surface

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**PURPOSE.** The aim of this study was to evaluate the effects of atmospheric plasma (APL) versus conventional surface treatments on the adhesion of self-adhesive resin cement to Ti-6Al-4V alloy. **MATERIALS AND METHODS.** Sixty plates of machined titanium (Ti) discs were divided into five groups (n=12): 1) Untreated (CNT); 2) Sandblasted (SAB); 3) Tribochemically treated (ROC); 4) Tungsten CarbideBur (TCB); 5) APL treated (APL). SEM analysis and surface roughness (Ra) measurements were performed. Self-adhesive resin cement was bonded to the Ti surfaces and shear bond strength (SBS) tests, Ra and failure mode examinations were carried out. Data were analyzed by one-way analysis of variance and chi-squared test. **RESULTS.** The lowest SBS value was obtained with CNT and was significantly different from all other groups except for APL. The ROC showed the highest SBS and Ra values of all the groups. **CONCLUSION.** It was concluded that the effect of APL on SBS and Ra was not sufficient and it may not be a potential for promoting adhesion to titanium. [*J Adv Prosthodont 2015;7:249-56*]

**KEY WORDS:** Titanium; Surface treatment; Non-thermal atmospheric pressure plasma; Micromechanical retention; Self-adhesive resin cement; Shear bond strength

## INTRODUCTION

Titanium (Ti) and its alloys are widely used as dental materials in prosthodontics and implantology because of their low density, high corrosion resistance, superior biocompatibility and good mechanical properties.<sup>1</sup> Using titanium as a prosthetic superstructure requires a strong and durable

bonding of cement to the titanium to resist various challenges in the oral environment.<sup>2-4</sup>

Adhering a luting cement to a surface depends on both micromechanical interlocking and physicochemical bonding.<sup>5</sup> Many different surface modification techniques have been proposed for increasing the binding and are classified as: micromechanical retention, chemical bonding, and micromechanical retention and chemical bonding.<sup>6</sup>

To provide micromechanical retention, airborne-particle abrasion with Al<sub>2</sub>O<sub>3</sub> particles is typically used.<sup>3,4,7-9</sup> Additionally, surface roughening with a bur has been reported to be quite practical and safe method for modifying the material surface.<sup>10</sup> These methods change the wettability characteristics of dental materials for improved adhesion and bonding. These procedures also clean the surface and increase the surface area.<sup>3,4,7-10</sup>

Chemical coupling can be obtained with metal primers and silanes. Metal primers contain active monomers such as 10-methacryloyloxydecyl-dihydrogen phosphate (MDP), 4-methacryloxyethyl trimellitate anhydride (4-META) and

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others that react chemically with the oxides present on the metal surface.<sup>11</sup> Silanes, due to their bifunctional characteristics, establish a chemical bond between the resin matrix and the metal surface.<sup>12</sup>

One method known to achieve both micromechanical retention and chemical bonding is tribochemical silica coating, which uses airborne-particle abrasion with silica-modified Al<sub>2</sub>O<sub>3</sub> particles. This method consists of a coating of abrasive sand, a silane coupling agent, a light-cured bonding resin and an opaque resin. This surface treatment provides a combination of micromechanical and chemical adhesion.<sup>3,4,6,10</sup>

Plasma treatment has been frequently used to modify the surface of products in the industrial sector.<sup>13,14</sup> Plasma is an electrically neutral ionized gas, electrons, ultraviolet irradiation and free radicals under normal pressure conditions.<sup>14</sup> Previously, this technique has been successfully used to deactivate bacteria even in bio-films on titanium surfaces.<sup>15,16</sup> Plasma irradiation does not cause environmental pollution, as it does not require chemical additives.<sup>13</sup> Plasma temperature is depending on the input power and varying according to the distance of the plasma flame. Its temperature can range between 37°C and 62°C, which is why it was defined biocompatible.<sup>17</sup> It was reported that, with the proper application of plasma treatment, an oxide layer is formed and surface becomes hydrophilic nature, which may influence the surface adhesion properties.<sup>18</sup> As distinct from a high pressure plasma requires a complex reactor, atmospheric plasma may operate at atmospheric pressure. A plasma device that works at an atmospheric pressure can be easily used in the oral environment. Since the first report on low temperature atmospheric pressure plasma (APP) in 1968, the homogeneous dielectric barrier discharge in atmospheric air has been explored in many applications ranging from industry to biology and medicine.<sup>13-21</sup>

Although numerous studies have evaluated the bond strength of resin cements to Ti alloys, very few have compared the effects of different surface treatments including roughening with a bur, sandblasting (conventional surface treatment), tribochemical silica coating, and plasma treatment (atmospheric pressure), on bond strength. The adhesion mechanism of Ti and the adhesive luting cements remains unclear.<sup>22</sup> Various publications are mentioned that there have been many different surface treatment techniques which increase the bond strength.<sup>3,4-8,11,12,22</sup>

The purpose of this study was to evaluate the effects of different surface treatments on the bond strength of a resin cement to Ti Grade 5 alloy. The null hypothesis was that the use of different surface treatments would not influence the shear bond strength at the titanium/cement interface. This test was intended to demonstrate the effect of atmospheric plasma treatment versus conventional surface treatment methods.

## MATERIALS AND METHODS

Sixty plates of machined Ti Grade 5 (Ti-6Al-4V) alloy (Biotec srl, Povolaro di Dueville, Italy) discs (10 mm in diameter and

2.0 mm thick) were machined and then cleaned in an ultrasonic bath (Sonorex, Bandelin, Berlin, Germany) filled with distilled water for 5 minutes.

The specimens were randomly divided into five experimental groups (n=12) that were subjected to one of the following surface treatment techniques: 1) Untreated control (Group CNT); 2) Sandblasted (Group SAB); 3) Tribochemically surface treated (Group ROC); 4) Roughened with a tungsten carbide bur (Group TCB); and 5) Plasma treated (Group APL).

**For Group CNT**, the test surface was polished with silicone carbide abrasive paper from 600 through 1200 grit (English Abrasives: London, England) under water irrigation.

**For Group SAB**, the sample surface was cleaned to remove residues and dried before the surface was blasted with 110 µm high-purity aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) sand (Rocatec™ pre, 3M ESPE, Seefeld, Germany, Lot No: 372623) for 15 seconds at a pressure of 2.8 bar from a vertical angle, and the distance was maintained at 10 mm from the specimen surface via the coating unit (Rocatec™ Junior, 3M ESPE, Seefeld, Germany).

**For Group ROC**, the sample surface was blasted with Rocatec™ pre as described above. This was followed by tribochemical coating of the microblasted surface with 110 µm silica-modified aluminum oxide (Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>) sand (Rocatec™ plus, 3M ESPE, Seefeld/Germany, Lot: 371315) according to the manufacturer's instructions. The blast pressure was adjusted to 2.8 bar to ensure an adequately high level of energy for creating the tribo-plasma. Any residual blast-coating agent was removed with a stream of oil-free air. After the Rocatec™ plus surface treatment, the silane solution (ESPE™ Sil, 3M ESPE, Seefeld, Germany, Lot No: 391601) was applied to the bonding surface and the solution was allowed to dry for 5 minutes. During this time, care was taken to prevent any impurities from contacting the silanized surface.

**For Group TCB**, to improve mechanical retention, the bonding surface of the specimens was drilled with a cylindrical tungsten carbide bur (Jota AG Rotary Instruments, Rüthi, Switzerland, Lot No: 779929), rotating at 40,000 rpm with consistent force under water spray. In order to provide a standard application, drilling was performed in parallel to the sample surface and cylindrical cavities of approximately equal volume (4 mm wide, 2 mm deep) were obtained with the use of a milling device (Paraskop M, Model No. 26060, Bego, Bremen, Germany) and drill depth was controlled.

**For Group APL**, plasma surface treatment was performed with an atmospheric plasma torch (Plasmatrete GmbH, Bisamweg 10, Steinhagen, Germany) to modify the titanium surfaces for bonding. This device does not require any special gas and generates plasma at room temperature. It was operated at a frequency of 21 kHz with an applied voltage of 5 kV. The treatment speed was 5 m/min. with pressure of 2 bar, and the approximate jet power was 1 kVA. It was used for 5 seconds, with a distance of 5 mm between the nozzle and the test surface. It was attempted to always keep the discs within the plasma. Manufacturer information of the materials used was shown in detail at Table 1.

**Table 1.** Materials evaluated

Group	Material/Method	Composition	Manufacturer	Lot No.
-	Ti Grade 5	Ti-6Al-4V	Biotecsrl, Povolaro di Dueville, Italy	698
-	RelyXTM U 200 self-adhesive resin cement	phosphoricacid, silanated fillers methacrylate monomers.	3M Deutschland GmbH, Neuss, Germany	517597
CNT	Abrasive paper applied	From 600 to 1200 grit	English Abrasives, London, England	-
SAB	Sand blasted	110 $\mu\text{m}$ $\text{Al}_2\text{O}_3$	Rocatec™ pre, 3M ESPE, Seefeld, Germany	372623
ROC	Sand blasted & Tribochemical coating	110 $\mu\text{m}$ $\text{Al}_2\text{O}_3$ + $\text{SiO}_2$	Rocatec™ plus, 3M ESPE, Seefeld/Germany	371315
TCB	Roughened with a burr	Tungsten Carbide	Jota AG Rotary Instruments, Rütthi, Switzerland	779929
APL	Atmospheric plasma applied	Atmospheric pressure	Plasmatreat GmbH, Bisamweg 10, Steinhagen, Germany	208134

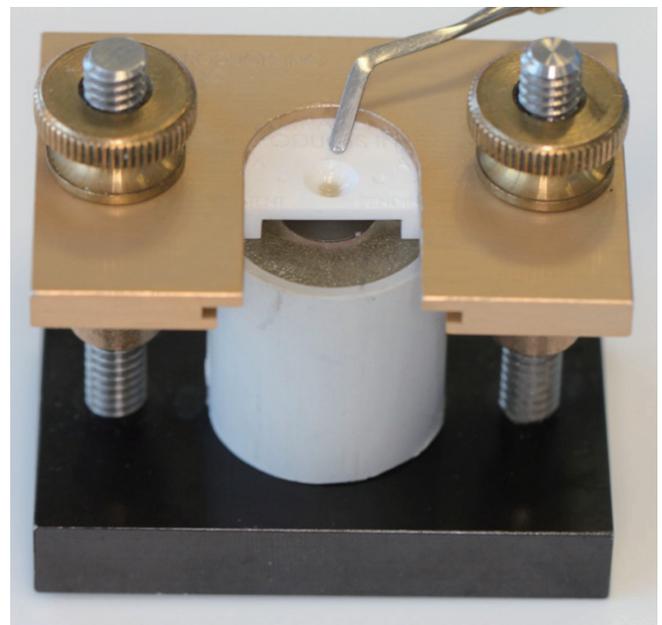
Following the surface modification procedures, all specimens were rinsed under distilled water and exposed to dry air spray; then, the samples were placed in a slot prepared in polymethyl methacrylate resin (Meliodent, Bayer Dental, Newbury, UK) that covered with polyvinyl chloride tube (20.0 mm in diameter and 27.0 mm high).

**SEM Analysis:** One fractured specimen from each group was examined under a scanning electron microscope (SEM, JSM-5600 Scanning Microscope; JEOL Ltd., Tokyo, Japan). Representative specimens were captured at  $\times 500$  magnification.

**Surface Roughness Measurements:** After each surface treatment, surface roughness measurements were carried out using a profilometer (Perthometer M2, Mahr GmbH, Göttingen, Germany) to measure the Ra (average roughness height) in micrometers ( $\mu\text{m}$ ). The roughness measurement surfaces of samples were not used for the shear bond test because the profilometer destroyed the surface. The mean roughness value was calculated from 3 single measurements.

**Resin Cement Bonding:** RelyX™ U 200 self-adhesive resin cement (3M Deutschland GmbH, Neuss-Germany, Lot: 517597) was condensed into an Ultradent Teflon mold (Ultradent Product, Inc., Utah, USA) and cured for 40 s as proposed by the manufacturer. The specimens were clenched in the Ultradent Bonding Jig (Ultradent Products Inc., South Jordan, UT, USA), and Teflon molds (2.3 mm in internal diameter, 3 mm in height) were used to build and connect the resin cement on the Ti plate surface (Fig. 1). After the cement had set via light curing (VOCO Celalux S/N, VOCO GmbH, Cuxhaven, Germany), the Teflon mold was disassembled.

**Shear Bond Strength tests:** SBS tests were conducted with a mechanical testing machine (Lloyd-LRX, Lloyd Instruments, Fareham, UK) with a 1 N load cell. A Sharp blade shearing sleeve, operating at a crosshead speed of 0.5 mm/min was used to apply a parallel shearing load as closely as possible to contact the cement/titanium interface. The failure strength was recorded in megapascal (MPa) unit.



**Fig. 1.** Making samples with the use of Teflon mold and bonding jig.

**Stereomicroscopy:** Each specimen was examined under a stereomicroscope (MZ 12; Leica Microsystems; Glattbrugg, Switzerland) at  $\times 20$  magnification, and images were observed and analyzed with a software. The failure mode was scored by the same evaluator as either adhesive (between titanium and resin cement), cohesive (within the resin cement), or combined (areas of adhesive and cohesive failure). For the last classification, the adhesive area was divided into quadrants, and the pre-dominant type of failure was observed in each specimen according to the method of Santos *et al.*<sup>23</sup>

**Statistical Analysis:** SBS and Ra values were analyzed by one-way analysis of variance (ANOVA). According to assumption of homogeneity of variance, post-hoc Tukey's and Games-Howell multiple comparison tests were used for SBS and Ra values, respectively. Statistical significance was set at the 0.05 probability level. In addition, the failure mode data were analyzed by the chi-squared test.

**RESULTS**

The results of one-way ANOVA (Table 2) indicated that the surface treatment methods affected the bonding and roughness values significantly. Table 3 and Fig. 2 show the mean SBS values (MPa), mean Ra values (µm) and standard deviations for each group, as well as the statistical analysis results.

**Table 2.** One-way analysis of variance (ANOVA) results for mean shear bond strength (SBS) and mean surface roughness (Ra) values

Source of variation	SBS (MPa)					Ra (µm)				
	SS	df	MS	F	Sig.*	SS	df	MS	F	Sig.*
Between groups	1652.7	4	413.1	32.7	.000	35.3	4	8.8	448.6	.000
Within groups	693.3	55	12.6			1.0	55	0.02		
Total	2346.0	59				36.3	59			

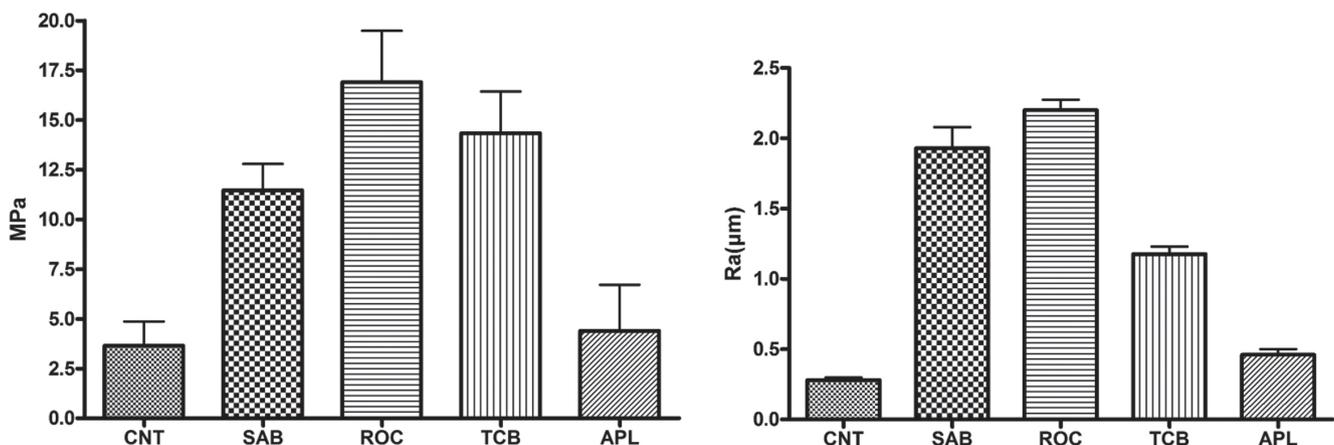
\*Sig. indicates significance.

**Table 3.** Mean ± standard deviation (SD) of the shear bond strength (SBS) and surface roughness (Ra) values

Group	N	SBS (MPa)			Ra (µm)		
		Min.	Max.	Mean (± SD)*	Min.	Max.	Mean (± SD)**
CNT	12	1.40	8.46	3.65 (± 2.11) <sup>a</sup>	0.24	0.35	0.28 (± 0.03) <sup>A</sup>
SAB	12	8.58	15.06	11.47 (± 2.30) <sup>b</sup>	1.46	2.49	1.93 (± 0.26) <sup>B</sup>
ROC	12	11.11	25.22	16.91 (± 4.48) <sup>c</sup>	2.01	2.39	2.20 (± 0.13) <sup>C</sup>
TCB	12	8.70	22.22	14.01 (± 4.11) <sup>b,c</sup>	1.11	1.33	1.19 (± 0.07) <sup>D</sup>
APL	12	1.05	13.31	4.40 (± 4.02) <sup>a</sup>	0.34	0.59	0.46 (± 0.07) <sup>E</sup>

\*Values having same lower case superscripted letters were not significantly different for post-hoc Tukey test (P>.05).

\*\*Values having different upper case superscripted letters were significantly different for post-hoc Games-Howell test test (P<.05).



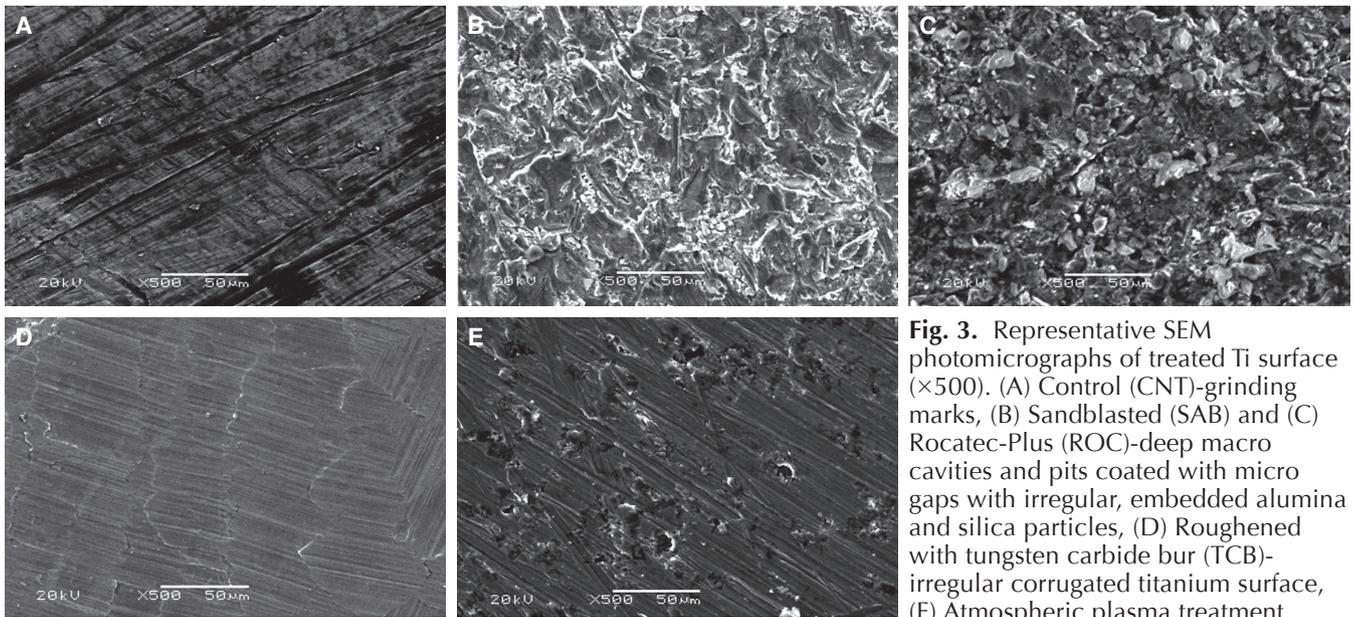
**Fig. 2.** Mean ± standard deviation of the shear bond strength (MPa) and surface roughness (µm) values.

**SBS:** The lowest shear bond strength values were obtained for the CNT group and significantly differ from all other groups except from the APL group. The ROC group showed the highest test results and is significantly different from the other groups ( $P<.05$ ) except from the TCB group ( $P>.05$ ). Comparisons of the test values that were obtained for the CNT and APL groups, the SAB and TCB groups, and the ROC group and the TCB groups did not show significant differences ( $P>.05$ ). When the groups abraded with  $110\ \mu\text{m}\ \text{Al}_2\text{O}_3$  particles were compared, it was observed that silica-modified  $110\ \mu\text{m}\ \text{Al}_2\text{O}_3$  (Rocatec™ plus) promoted significantly higher SBS than  $110\ \mu\text{m}\ \text{Al}_2\text{O}_3$  alone ( $P<0.05$ ).

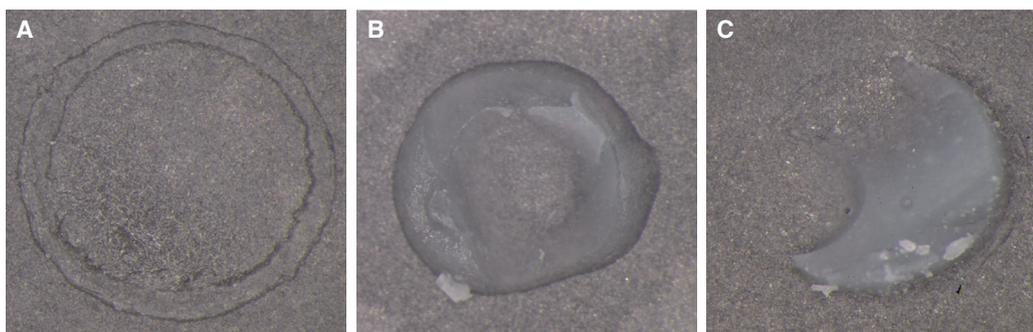
**Ra:** The ROC group showed the highest surface roughness value of all the groups (Table 3). There were significant differences between all groups ( $P<.05$ ) in terms of surface roughness. In support of this finding, SEM examination showed that groups with different Ra values had different surface topography from that of the control group.

**SEM:** The original CNT surface was in a roughened form because of the abrasive paper, which was no longer visible after treatment with SAB, ROC and TCB. SEM images revealed that more grooves were present in the SAB and ROC groups than in the TCB and APL groups (Fig. 3). For the SAB and ROC groups, there is no difference in terms of surface rheology, and they have deep macro cavities and pits coated with micro gaps, which possibly resulted from the high impact of the abrasive particles. The Ti surface appeared rough, with irregular, embedded alumina and silica particles. In the APL group, a superficially fissured coarse surface with carbon spots was observed, but the morphological characteristics of the Ti surface did not differ in any important way from that of the CNT group.

**Stereomicroscopy:** Adhesive, cohesive and mixed failure mode images are represented in Fig. 4. The failure mode was predominantly adhesive for all cements, as shown in Table 4. The relation between failure mode and treatment method was statistically significant ( $X^2=22.565$ ,  $df=8$ ,  $P<.05$ ).



**Fig. 3.** Representative SEM photomicrographs of treated Ti surface ( $\times 500$ ). (A) Control (CNT)-grinding marks, (B) Sandblasted (SAB) and (C) Rocatec-Plus (ROC)-deep macro cavities and pits coated with micro gaps with irregular, embedded alumina and silica particles, (D) Roughened with tungsten carbide bur (TCB)-irregular corrugated titanium surface, (E) Atmospheric plasma treatment (APL)-a superficially fissured coarse surface with carbon spots.



**Fig. 4.** Stereomicroscope images of failure mode between resin cement and Ti surface: (A) Adhesive failure mode, (B) Cohesive failure mode, (C) Mix failure mode.

**Table 4.** Distribution of failure modes per group and resin cement

Group	Failure mode			Total
	Adhesive	Cohesive	Mixed	
CNT	12	0	0	12
SAB	9	1	2	12
ROC	8	2	2	12
TCB	11	0	1	12
APL	8	0	4	12
Total	48	3	9	60

## DISCUSSION

The results of this study demonstrate that the APL group showed the lowest values in terms of both surface roughness and bond strength. In contrast, the ROC group presented significantly higher surface roughness and bond strength values than the other groups ( $P < .05$ ). Although the ROC-TCB and SAB-TCB groups had similar bond strengths internally, there were no significant differences between these pairs. Therefore, the null hypothesis was rejected.

The self-adhesive cement used in this study has a high degree of adhesion.<sup>8,12,24,25</sup>

It has been reported that a strong and durable bond at the metal/cement interface depends on a combination of micromechanical retention and chemical bonding. In cementation with non-adhesive resin cements, both bonding mechanisms (chemical and micromechanical) must be ensured.<sup>3,4</sup> However as shown in the present study, in the case of self-adhesive resin cements, an appropriate adhesion can be obtained by abrasion with airborne  $Al_2O_3$  particles<sup>8</sup> and roughening with a bur to increase surface area.<sup>10</sup>

In this study, two different chemical compositions were used for the airborne-particle abrasion:  $Al_2O_3$  particles (Rocatec™ pre) and silica-modified  $Al_2O_3$  particles (Rocatec™ plus). Sandblasting of a material surface is often used to obtain a micro-retentive topography and increased surface area; thus, the wettability of the material can be increased.<sup>7</sup>

Significantly higher bond strength was observed for samples having both micromechanical retention and chemical bonding with the silica coating system (Rocatec™ plus) than for samples treated by airborne-particle abrasion with  $110 \mu m$   $Al_2O_3$  particles alone ( $P < .05$ ). This result was in agreement with the findings of previous investigations.<sup>26-29</sup> Significant differences were observed between the SAB and ROC groups for both Ra and SBS values ( $P < .05$ ). A possible explanation for these statistical results is the superiority of combined micromechanical retention and chemical bonding that was provided with the  $SiO_2$  coating of the same-sized  $Al_2O_3$  particles.

There are many studies on the effects of atmospheric plasma surface treatment on the adhesion of certain types of dental material, primarily for polymers<sup>18-20,30,32-34</sup> and less so for zirconia<sup>24,31</sup> and titanium.<sup>14,22,31</sup> Some authors have reported that plasma surface modification procedures allow the formation of stronger bonds, improve wettability, and produce chemically active sites for the adhesion of other molecules.<sup>21,30,31</sup> To the best of our knowledge, there is limited information in the dentistry literature about the effects of plasma treatment of titanium surfaces on surface roughness and adhesion.

Valverde *et al.*<sup>24</sup> detected a significant increase in the micro-tensile bond strength to Y-TZP surfaces when non-thermal plasma was applied alone or in combination with ceramic primer and/or  $Al_2O_3$  sandblasting. In addition, Silva *et al.*<sup>31</sup> used surface characterization to investigate the effects of non-thermal plasma exposure on Ti and Y-TZP and reported that the plasma treatment of both material surfaces resulted in increases in surface energy in parallel with increases of the execution time.

However, in the present study, the APL group had the lowest value for bond strength after the control group. This may be attributed to effects caused by the time of treatment, the range of applied power, the waveforms of the applied voltage or the distance between the sample surface and the plasma applicator nozzle, as noted by many publications.<sup>20,21,31,33</sup>

Han *et al.*<sup>21</sup> and Cho *et al.*<sup>34</sup> demonstrated that the application of non-thermal APP provided much lower adhesive strength than that obtained with conventional hydrofluoric acid etching and silane application to enhance the adhesion of composite resin to dental ceramic, which is in line with our findings. However, these authors<sup>21,34</sup> also emphasized that this approach could contribute to the enhancement of bond strength with dental adhesives.

Roughening the Ti surface increases surface area and wettability; it also provides mechanical interlocking with resin cements and is considered to be a significant factor influencing bond strength.<sup>6,35,36</sup> In this study, airborne-particle abrasion of the ROC and SAB groups resulted in greater degrees of roughness on the Ti surface than did the other treatments (Table 3). An increase in the surface roughness leads to an increase in surface area, which, in turn, may improve the Ti-resin cement bonding. However, in the comparison between groups for bond strength and roughness values, this was not the case. For surface roughness measurements, the TCB group value was less than the SAB group value. However, the shear bond strength value of the TCB group was higher than that of the SAB group (Table 3), which reveals that roughening the surface of the titanium does not increase the bonding strength. Therewithal, it was mentioned that surface roughness causes intense stress locations at the metal-cement interface and forms sharp angled edges that could avoid completely wetting.<sup>37</sup> This result of the present study is in agreement with those of some previous studies.<sup>35-38</sup>

This experimental study revealed that an inadequate Ti/

resin-cement bond was produced in the untreated control group. The analysis of failure modes showed variations in the test groups. Mixed failures occurred more frequently in groups with higher bond strength values (Table 4). This finding is in accordance with those of previous studies.<sup>38-39</sup>

According to the requirements of ISO 10477,<sup>40</sup> the minimum acceptable SBS value at the interface of resin-based materials and substrate is 5 MPa. Matsumura *et al.*<sup>41</sup> reported that the resin-to-metal SBS value should be at least 10 MPa for clinically acceptable results. In the present study, SBS values above 10 MPa were observed in all groups except for the APL and CNT groups.

Plasma treatment did not bring about any structural change in the titanium surface, in contrast to treatments such as sandblasting and treatment with Rocatec™ plus, which can change the surface of the titanium morphologically.

Limitation of this study is the use of certain parameters for plasma application. Future studies are needed to evaluate in different execution time and applied power of plasma discharge which may bring different results. Also, only one category of dental cement and one type of titanium were tested; these findings may not apply to other cements or titanium alloy materials.

## CONCLUSION

In this study, the effect of atmospheric plasma treatment on bond strength was not sufficient to replace the current protocol involving micromechanical roughening for titanium surfaces.

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