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Airborne-particle abrasion parameters on the quality of titanium-ceramic bonds

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INTRODUCTION

Because of its good biocompatibility, titanium has been used in many fields of medicine, including dentistry.[1-9] Its excellent biological and mechanical properties seem to make it an ideal material for use in the human body. Dental implants made of titanium have come to be used in everyday dental practice. However, because of its unique properties, the way titanium joins with ceramic material is also unique.

The highly oxidative nature of titanium is regarded as the main cause of the poor strength of its bonds with ceramic materials. Therefore, methods are being sought to improve the bond strength, such as etching with acids, alkalis, or salts; etching with a laser; applying intermediate silicon layers by the sol-gel method; applying a bonding layer with a high gold content; using low-melting ceramics; fusing in a vacuum; and fusing in an argon atmosphere. In the majority of such studies, specimens are airborne-particle-abraded with Al_2O_3 before they are modified, or specimens after airborne-particle abrasion are used as a control group for the surfaces tested. Neither the method of airborne-particle

ABSTRACT

Statement of problem. Airborne-particle abrasion of titanium is a clinically acceptable method of surface preparation. It is crucial to know the effectiveness of bond strength between the metal substructure and the veneering ceramics after this kind of surface treatment.

Purpose. The purpose of this study was to determine how the particle size of the abrasive material and pressure affected treated surfaces and the strength of titanium-ceramic bonds.

Material and methods. Disks made of titanium (Tritan CpTi grade 1, Dentaurum, 99.5% Ti) were treated in an airborne-particle abrasion process with 50, 110, and 250 μm aluminum oxide (Al_2O_3) at pressures of 0.2, 0.4, and 0.6 MPa. To characterize the treated surfaces, the following values were measured: roughness, free surface energy, and the quantity of abrasive particles attached to the surface. Subsequently, the strength of the metal-ceramic bond was determined. Apart from the strength tests, fractures were observed to determine the character and fracture location in the course of the strength tests. The results of the experiment were analyzed with 2-way ANOVA and the Tukey HSD test (α=.05).

Results. Both the pressure and the particle size of Al_2O_3 used in the airborne-particle abrasion affected the strength of the titanium-ceramic bond (P<.05). A statistically significant difference was found between the group subjected to airborne-particle abrasion under a pressure of 0.4 MPa with 110-μm Al_2O_3 particles and the other experimental groups (P<.05).

Conclusion. This study demonstrates that the highest bond strength between a ceramic and titanium substructure can be achieved after airborne-particle abrasion at an angle close to 45 degrees with 110-μm Al_2O_3 particles under 0.4 MPa of pressure. (J Prosthet Dent 2015;■:■-■)

abrasion nor the process parameters are standardized; thus they vary considerably from one study to another.[1,10-13]

Despite numerous attempts to apply various methods of surface treatment to increase the strength of the bond between a ceramic material and the titanium base, the only effective and available method is surface development by airborne-particle abrasion and ensuring a mechanical bond between the 2 materials. Airborne-particle abrasion of a titanium specimen surface results in

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morphological changes. The geometric structure of the surface can be determined according to various parameters: the arithmetic mean deviation of the Ra profile (this is the most frequently used parameter of roughness and considered the most significant), the maximum height of the Rz profile, and Rt (total profile height). The Ra parameter for titanium is frequently determined for dental implants to increase the surface available for osseointegration. Airborne-particle abrasion is the main method of preparing the surface of implants before further modifications. Frequently, specimens that are airborne-particle abraded with Al_2O_3 are used as controls for modifications aimed at increasing the strength of a titanium-ceramic bond.[5,7,14-19] Although airborne-particle abrasion is a commonly used procedure, no cross-sectional studies have been done to determine the effect of the basic parameters of such treatment on the structure of a metal surface. Moreover, the parameters of roughness, wetting angles, and free surface energy and their effect on the quality of a titanium-ceramic bond have yet to be analyzed.

The absence of clear guidelines for the airborne-particle abrasion of titanium elements has encouraged the authors to investigate how the main parameters of the process affect the strength of titanium-ceramic bonds.

MATERIAL AND METHODS

One hundred and thirty-eight titanium disks (Tritan CpTi grade 1; Dentaurum) with a diameter of 21 mm and a thickness of 5 mm were used in the experiment. The minimum content of titanium was equal to 99.5% with traces of Fe, O, H, N, and C, according to the classification of the American Society for Testing and Materials. To make the surface uniform before airborne-particle abrasion, the disks were ground on a rotary grinder (Metasinex; Measinex Row) with SiC abrasive paper with a grit size of 220, 400, 600, and 800 under water cooling. The specimens were washed with water, dried with compressed air after each grinding, and divided into 12 groups. Three groups (10 specimens in each group) were designed for preliminary examination. The rest of the specimens were treated with an airborne-particle abrasion process (Mikroblast Duo; Prodento-Optimed) using Al_2O_3 and were then subjected to tests for strength,

roughness, and free surface energy and to fractographic examination.

First, an experiment was conducted to determine the effect on the surface parameters of the inclination angle of the specimen during the process. Thirty specimens, divided into 3 groups, were used. Two groups of titanium disks, with 10 specimens in each group, were airborne-particle-abraded (Renfert GmbH) with Al_2O_3 (Renfert GmbH) at glancing angles of 45 degrees and 90 degrees and at a distance of 10 mm from the end of the blasting device nozzle. Another 10 specimens were airborne-particle-abraded with the nozzle angle changing during the process. For this group, an approximate distance of 10 to 15 mm was used, which, combined with variation of the angle at which particles of Al_2O_3 hit the surface, simulated conditions similar to those in which dental technicians work. The experiment was conducted for an Al_2O_3 particle size of 110 μm.

Roughness measurements were then performed on the prepared specimens with a laser scanning microscope (LSM; Nikon) at ×500 magnification and a measurement section length of 275 μm. The following surface parameters were determined: arithmetic averages of the Ra profile deviation, maximum height of the Rz profile, maximum elevation of the Rp profile, depth of the lowest hollow of the Rv profile, average value of the height of the Rc profile elements, total height of the Rt profile, quadratic mean of Rq profile ordinates, coefficient of asymmetry of the Rsk profile, and coefficient of inclination of the Rku profile.

A preliminary analysis of the roughness parameters did not reveal any significant differences related to the glancing angle. A fixed angle of 45 degrees and a distance of 10 mm from the airborne-particle abrasion nozzle (Mikroblast Duo; Prodento-Optimed) were chosen for further experiments. The abrasion time of the specimens was established at 10 seconds.

One hundred and eight cylindrical specimens were used in the main experiment; they were divided into 9 groups with 12 specimens in each group. Specimens in groups were treated with an airborne-particle abrasion process (Mikroblast Duo; Prodento-Optimed) with Al_2O_3 with a particle size of 50, 110, and 250 μm and under pressures of 0.2, 0.4, and 0.6 MPa. After the process, the titanium disks were cleaned with steam under pressure, washed in deionized water in an ultrasonic washer for 8 minutes, and dried with compressed air. In the next step, 2 specimens from each group were examined microscopically and their roughness and surface energy determined. Ceramic material (Super Porcelain Ti-22; Noritake) was fused onto the other specimens in accordance with the guidelines of the manufacturer, except for those which applied to airborne-particle abrasion.
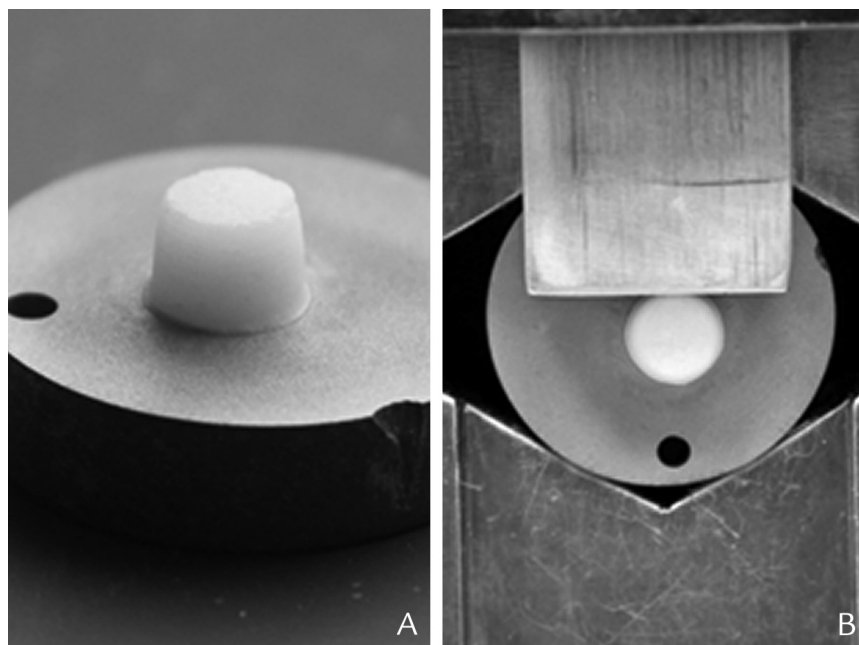
Figure 1. Test stand for measuring metal-ceramic bond strength. A, Specimen with ceramic material fused to it. B, Specimen position during shear strength test.

Ceramic material was formed on the surface under treatment into 10-mm high, 5.6 mm internal diameter cylinders to a height of 4 mm. The ceramic material was fused onto the central part of the area that was previously airborne-particle-abraded. The shape and height of a specimen was adjusted with a straight handpiece and a milling cutter. Layers of ceramic material were fused in the following sequence: BP bond, opaquer, dentin 1, dentin 2, enamel. After the ceramic material was fired, the diameter and height of the specimens were checked with a caliper (MIB DIN 862) with an accuracy of 0.02 mm.

Shear strength (Zwick/Roell Z005) was tested on 90 specimens to determine the strength of the bonds between the ceramic material and the titanium. The specimens were loaded at a crosshead speed of 2 mm/min until failure of the titanium-ceramic bond; the maximum force was recorded (Fig. 1), and together with the diagrams of the test course, was entered into a computer program integrated with the testing device. Subsequently, the bond strength was calculated from the formula: $Rt = F/S$, where Rt is the shear force [Pa], F the force acting on the specimen [N], and S the surface area of the specimen [m^2].

The results were statistically analyzed using 2-way ANOVA (α=.05). The null hypothesis was formulated as follows: Ho: $\mu1=\mu2=\mu3...\mu i$, assuming an absence of statistically significant changes among the analyzed specimens. The null hypothesis was rejected when $F > F_{critical}$. If the test showed that this condition was met in the analyzed case, then the null hypothesis was rejected. This indicated that statistically significant changes had occurred in the studied group. The Tukey HSD test was used to determine differences between a pair of specimens.

After the strength tests were performed, cross sections of the fractured specimens were analyzed with an electron scanning microscope (SEM S-3000N; Hitachi High-Technologies Corp) to determine the nature of the fractures formed in the shearing process and the locations where the specimens fractured (Fig. 2).

Roughness was determined with a laser scanning microscope (SEM S-3000N; Hitachi High-Technologies Corp) (the device and parameters of the measurement were the same as in the preliminary tests). The following surface parameters were determined: arithmetic averages of the Ra profile deviation, maximum height of the Rz profile, and total height of the Rt profile. The Pearson correlation method was used to determine the correlation between the strength of a titanium-ceramic bond and the roughness coefficient (Rz, Rt, Ra).

The free surface energy of the specimens (γ_s) was determined by measuring the wetting angle with a device (FM40 EasyDrop; Krüss GmbH).[20] Two test liquids were used: distilled water and diiodomethane. The liquids were selected so that one of them had a small dispersive component of surface energy (γ_L^d) and a large polar component of surface energy (γ_L^p), while these characteristics for the other were the opposite, that is, a large value of γ_L^d and a small value of γ_L^p. Liquids were added at 0.8 μL. The wetting angle was determined with the DSA15 software by measuring the geometry of a droplet
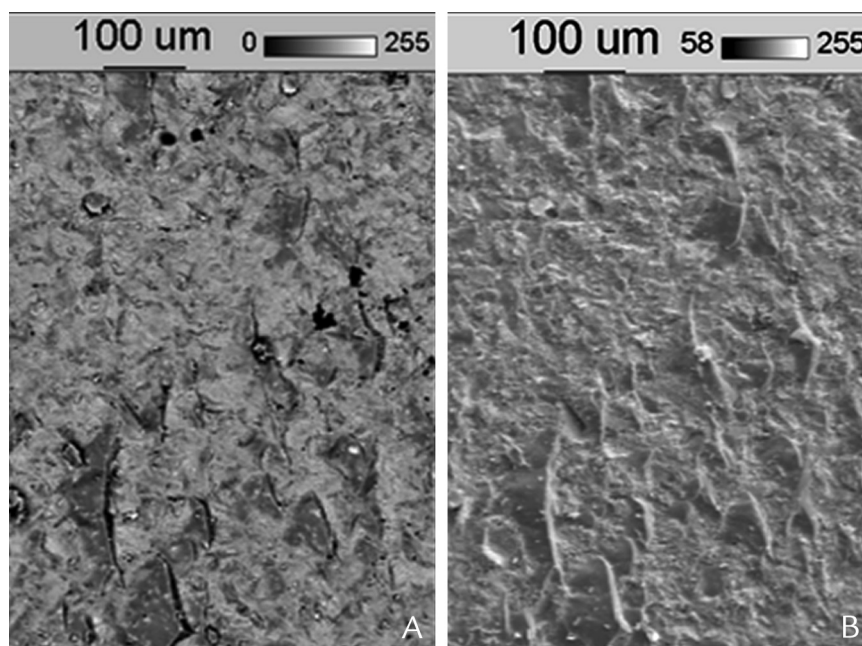
Figure 2. Microscopic image of fracture surface of specimen that was airborne-particle-abraded with 250-μm particles under pressure of 0.4 MPa. A, Image obtained with back-scattered electrons (BSE) at ×500 magnification. B, Image obtained with scattered electrons (SE) at ×500 magnification.

of the test liquid placed on the tested surface. The values of individual dispersive (γ_S^d) and polar (γ_S^p) components for the tested specimens were determined with the Owens-Wendt model. The components thus calculated were used to determine the free surface energy of the tested surfaces.

RESULTS

Statistical analysis of the results of the strength tests (Table 1) showed that the pressure applied in the airborne-particle abrasion process and the particle size of Al_2O_3 affected the strength of a titanium-ceramic bond. The Tukey test revealed statistically significant differences among the groups in which specimens were airborne-particle-abraded with Al_2O_3 with a particle size of 110 μm under a pressure of 0.4 MPa, 50 μm under a pressure of 0.2 MPa, 50 μm under a pressure of 0.4 MPa, 50 μm under a pressure of 0.6 MPa, and 250 μm under a pressure of 0.2 MPa. Statistically significant differences ($P<.05$) were also observed between the 50 μm/0.6 MPa and the 250 μm/0.4 MPa groups.

Not all results of the strength test met the ISO 9693 standard, in which the minimum strength of a metal-ceramic bond is specified as 25 MPa. Only some groups in our experiment meet those criteria; these were the specimens that were airborne-particle-abraded with 110-μm particles under a pressure of 0.2 and 0.4 MPa and the 250 μm/0.4 MPa group.

The nature of fractures formed after the strength tests was similar for all of the specimens. In most cases,

Table 1. Results of strength tests for titanium-ceramic bonds

Bond Strength (MPa)	50 μm (SD)	110 μm (SD)	250 μm (SD)
0.2	19.37 (4.08)	27.41 (3.8)	22.99 (4.54)
0.4	22.27 (3.87)	32.28 (5.67)	25.93 (3.68)
0.6	19.78 (3.66)	22.99 (2.83)	24.32 (5.36)

SD, standard deviation.

the fracture ran along the border between the ceramic material and the titanium. In all of the specimens, a much larger area of the surface was rich in titanium (Fig. 3). However, ceramic residue was found on the surface; it was visible on maps of the surface distribution of elements as silicon and tin, which are ingredients of ceramic materials (Fig. 4). Because tin appears on the surface in all cases, fractures appeared to occur in the area of the bond or opaquer because only those components contained tin (Fig. 4B). Aluminum, visible in the specimens, comes from particles of the abrasive material (Fig. 3B).

The results of the roughness measurement reveal an increase in Ra with an increase in the size of Al_2O_3 particles in each group at the same processing pressure; similar findings were observed with Rz and Rt (Table 2). However, when 110-μm particles were used, the variability of Ra and Rz was not significant between 0.4 MPa and 0.6 MPa. The values of all of the surface parameters increased noticeably in the group in which the specimens were airborne-particle-abraded at a pressure of 0.6 MPa with 250-μm particles, with the values being nearly twice as high as in the 0.2 MPa group. Determination of the coefficients of the
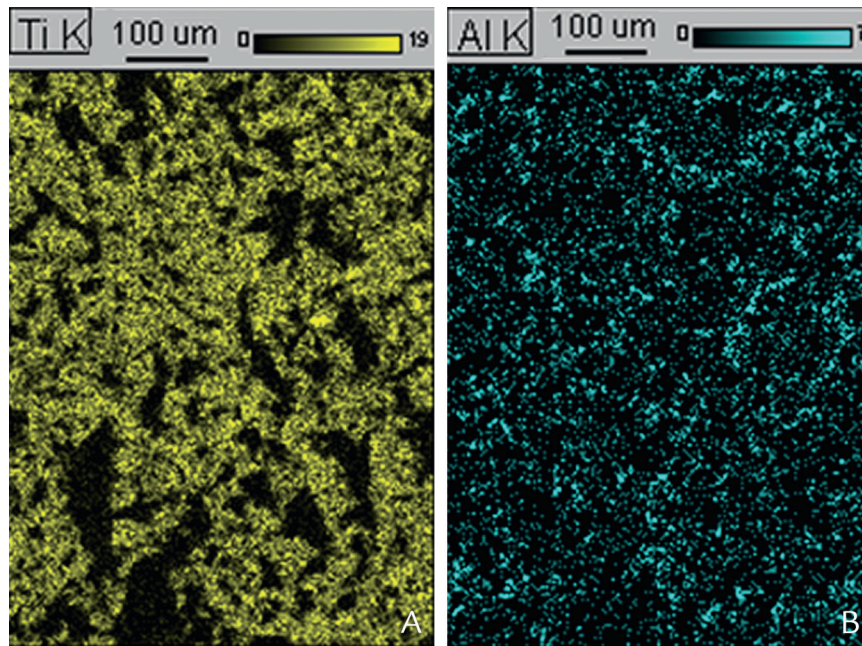
Figure 3. Distribution of elements on surface of specimen that was airborne-particle abraded with 250-μm particles under pressure of 0.4 MPa. A, Surface distribution of Ti. B, Surface distribution of Al.
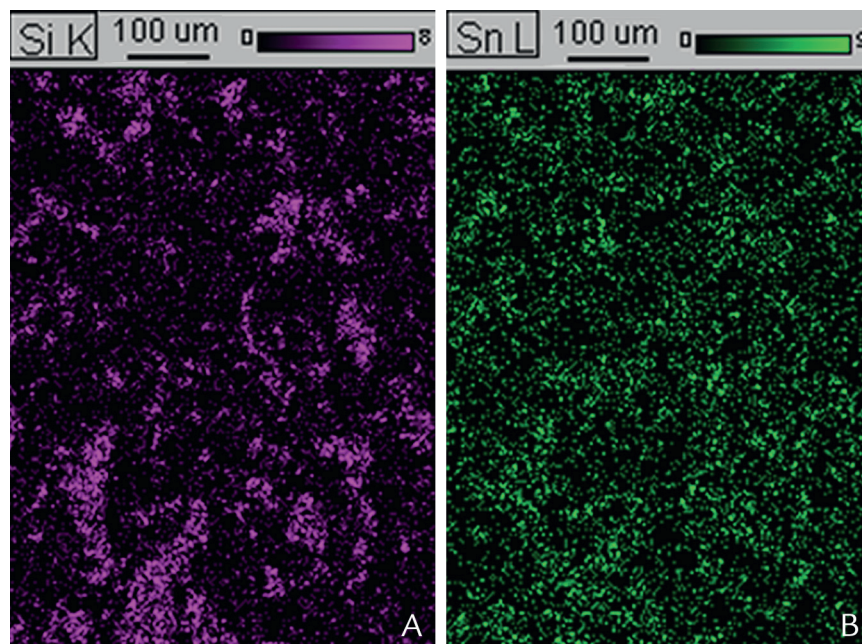

Figure 4. Distribution of elements on surface of specimen that was airborne-particle-abraded with 250-μm particles under pressure of 0.4 MPa. A, Surface distribution of Si. B, Surface distribution of Sn.

Pearson correlation leads to the conclusion that the strength of a titanium-ceramic bond is not correlated with Rz or Rt (correlation coefficients < .02). For Ra, the coefficient of the Pearson correlation equal to 0.24, indicates a weak positive correlation between the roughness coefficient Ra and the strength of a titanium-ceramic bond.

Wetting angles for water θ_w and diiodomethane θ_j, as well as free surface energy γ_s and its dispersive (γ_s^d) and polar (γ_s^p) components, are shown in Table 3. For free surface energy γ_s and its dispersive (γ_s^d) and polar (γ_s^p) components, their correlation with the bond strength is weak (<.1).

Table 2. Mean values of parameters of roughness for tested surfaces

Pressure, MPa	Ra, μm (SD)			Rz, μm (SD)			Rt, μm (SD)		
	50 μm	110 μm	250 μm	50 μm	110 μm	250 μm	50 μm	110 μm	250 μm
0.2	0.340 (0.010)	0.580 (0.058)	1.315 (0.148)	3.245 (0.095)	5.418 (0.371)	10.745 (1.164)	3.963 (0.131)	7.095 (0.595)	15.033 (1.659)
0.4	0.556 (0.017)	1.029 (0.026)	1.622 (0.203)	4.472 (0.120)	7.605 (0.252)	13.350 (1.829)	5.553 (0.164)	10.053 (0.260)	19.167 (2.803)
0.6	0.792 (0.044)	1.092 (0.034)	2.248 (0.220)	6.247 (0.382)	8.750 (0.598)	20.383 (2.735)	8.220 (0.461)	12.317 (0.662)	28.400 (4.757)

Ra, arithmetic averages of profile deviation; Rt, total height of profile; Rz, largest height of profile; SD, standard deviation.

Table 3. Wetting angles for water θ_w and diiodomethane θ_j and free surface energy γ_s and its dispersive (γ_s^d) and polar (γ_s^p) components related to variable parameters of surface treatment, that is, size of Al_2O_3 particles and pressure

Parameter	θ_w [deg]	θ_j [deg]	γ_s [mJ/m²]	γ_s^d [mJ/m²]	γ_s^p [mJ/m²]
50 μm, 0.2 MPa	116.0	84.3	15.71	15.65	0.06
50 μm, 0.4 MPa	115.6	90.6	12.47	12.09	0.38
50 μm, 0.6 MPa	108.7	76.4	19.68	19.36	0.32
110 μm, 0.2 MPa	62.9	52.0	42.03	25.06	16.98
110 μm, 0.4 MPa	62.3	42.9	44.98	29.76	15.22
110 μm, 0.6 MPa	63.2	56.9	40.60	22.22	18.38
250 μm, 0.2 MPa	69.0	55.9	37.62	23.76	13.86
250 μm, 0.4 MPa	82.9	53.9	33.20	28.21	4.98
250 μm, 0.6 MPa	105.0	57.3	31.38	31.37	0.01

θ_w, wetting angles for water; θ_j, wetting angles for diiodomethane; γ_s, free surface energy; γ_s^d, dispersive of free surface energy; γ_s^p, polar of free surface energy.

DISCUSSION

Determining the influence of parameters of airborne-particle abrasion was of interest in this study. Parameters of airborne-particle abrasion, particle size of the abrasive material, and pressure were found to influence the treated surfaces and the strength of titanium-ceramic bonds.

The results of strength tests show the importance of selecting the appropriate parameters of airborne-particle abrasion. The size of Al_2O_3 particles and the pressure applied in airborne-particle abrasion affected the strength of titanium-ceramic bonds. The lowest strength of all of the pressure groups was recorded for specimens that were airborne-particle-abraded with 50-μm particles. This can be attributed to the largest percentage of Al, the largest number of Al_2O_3 particles abraded into the surface, and the largest volume share of the particles on the surface of a titanium specimen, all of which have an adverse effect on the strength of a titanium-ceramic bond. According to Gilbert et al,[19] particles of Al_2O_3 can contaminate a titanium surface, which can weaken the anchoring of the ceramic material and decrease resistance to corrosion and biocompatibility. The lowest strength of the titanium-ceramic bonds in the group may be the result of the lowest values of the parameters of the geometric structure of the surface, which may be associated with the wetting angle and free surface energy (Table 3). The group in which specimens were airborne-particle abraded with 50-μm particles of Al_2O_3 deviates

from the others, where the process was conducted with 110- and 250-μm particles.

An analysis of the free surface energy values shows that the highest values (42.02 to 44.98 mJ/m²) were achieved for the surfaces that were airborne-particle-abraded with 110-μm particles. Satisfactory results ranging from 31.38 to 37.62 mJ/m² were also obtained for specimens treated with 250-μm particles, with the value decreasing with increasing working pressure. The lowest values (12.47 to 19.68 mJ/m²) were achieved for the surfaces airborne-particle-abraded with 50-μm particles. The values of free surface energy appear to correlate, to a considerable extent, with the results of strength tests. The greatest strength of a titanium-ceramic bond was observed in the group in which specimens were airborne-particle-abraded with 110-μm Al_2O_3 particles at a pressure of 0.4 MPa; the value of free surface energy was also the highest (44.98 mJ/m²), while the value of the wetting angle was the lowest both for water (62.3) and for diiodomethane (42.9), which has a beneficial effect on the wetting of a titanium surface by a ceramic material. A comparison of the other groups, that is those airborne-particle-abraded with 50- and 250-μm particles, reveals a clear association between the wetting angles and free surface energy on the one hand and the titanium-ceramic bond strength on the other.

No immediate relationship was found between the geometric parameters of the surface and the strength test results because the latter did not increase steadily for each group of grain size and treatment pressure. Kim and Cho[12] arrived at similar conclusions. The values of Ra increased after intermediate layers of SiO_2 and SiO_2-TiO_2 were applied; despite an increase in the joint strength for individual groups, they cannot be made conditional on an increase in Ra, because SiO_2 and SiO_2-TiO_2 also affect the chemical bonds between the ceramic material and titanium.[14]

An analysis of the nature of the fractures formed after the strength tests shows that they were similar in all of the specimens. In most areas, a fracture ran along the border between the titanium base and the ceramic material, which could indicate adhesive damage to the bond, and a higher content of titanium on the surface was observed on all of the tested specimens. However, ceramic residue was also found on the surface; elements were visible on the maps of the surface distribution,

which are ingredients of ceramic materials (Figs. 3, 4). This in turn indicates the mixed (cohesive-adhesive) nature of the damage. This is also confirmed by the fact that tin appeared on the surface in all cases, which may indicate that fractures in the ceramic material occur in the area of the bond or opaquer because those components contain tin.

The limitations of the study include the selection of the test specimen geometry used to measure joint strength, which is different from that in the ISO standard for testing the bond strength of dental metal-ceramic specimens. However, the specimen geometry was selected to suit the study methodology; in particular, it had to allow the specimens to be mounted in a strength testing machine.

One of the areas that should be explored in further research is the effect on the quality of a metal-ceramic bond of the particles of an abrasive material blasted into the alloy.

CONCLUSION

Within the limitations of this in vitro study, the following conclusions were drawn:

1. The strength of a titanium-ceramic bond depended both on the size of Al_2O_3 particles and on the pressure used for airborne-particle abrasion ($P<.05$).
2. The highest strength of a titanium-ceramic bond was achieved for an Al_2O_3 particle size of 110 μm and a pressure of 0.4 MPa ($P<.05$).
3. The highest strength of a bond between ceramic material and a titanium suprastructure can be achieved as a result of airborne-particle abrasion with Al_2O_3 particles with a size of 110 μm at a pressure of 0.4 MPa and at an angle close to 45 degrees.

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