

Effect of surface treatment on bond strength of low-fusing porcelain to commercially pure titanium

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Statement of problem. Due to the pronounced oxidative nature of titanium at high temperatures, an excessively thick layer of TiO₂ may form on the surface. This oxide layer could adversely affect titanium-porcelain bonding.

Purpose. The purpose of this study was to investigate the effect of bonding agent and surface treatment using airborne-particle abrasion and hydrochloric acid on the bond strength between a low-fusing porcelain and commercially pure cast titanium.

Material and methods. A casting unit was used to cast 60 specimens of commercially pure titanium (25.0 × 3.0 × 0.5 mm). The specimens were equally divided into 3 groups. The first group received no surface treatment and served as the control, the second group was subjected to airborne-particle abrasion, and the third group was treated with hydrochloric acid. The specimens in each group were further divided into 2 subgroups of 10 each. Ten specimens were treated with bonding agent (Noritake), and 10 specimens were not treated with bonding agent. Low-fusing porcelain (Noritake) was fired onto the surface of the specimens. A universal testing machine was used to perform the 3-point bending test. The titanium-ceramic interfaces were subjected to scanning electron microscopic analysis. The bond failure data (MPa) were analyzed with a 2-way analysis of variance and Tukey multiple range tests ($\alpha=.05$). Four specimens from each group were selected for scanning electron microscopic examination.

Results. The debonding test showed that surface treatment with airborne-particle abrasion followed by application of a bonding agent resulted in the strongest (35.60 ± 8.15 MPa) titanium-ceramic bond ($P<.001$), followed by airborne-particle abrasion alone (25.6 ± 5.4 MPa) and bonding agent alone (24.7 ± 6.3 MPa). Hydrochloric acid surface treatment provided no beneficial effect to the titanium-ceramic bond strength compared to untreated specimens ($P=.975$). The photomicrographs of the titanium surface after debonding demonstrated residual porcelain retained on the metal surface for all groups.

Conclusions. Surface treatment using either airborne-particle abrasion or bonding agent alone enhanced the bond strength of cast commercially pure titanium to low-fusing porcelain. The combination of airborne-particle abrasion and bonding agent provided the greatest improvement in titanium-ceramic bond strength. Titanium surface treatment with hydrochloric acid, with or without bonding agent, produced values that were not statistically different than the control. (J Prosthet Dent 2005;94:350-6.)

CLINICAL IMPLICATIONS

This study demonstrated that the combination of airborne-particle abrasion and bonding agent application significantly improves the bonding strength of the commercially pure titanium to the low-fusing porcelain tested.

The metal-ceramic restoration (MCR) is a widely accepted esthetic restoration. For a metal to be used in an MCR, it should be biocompatible so that it does not cause harmful toxicological or allergic reactions to the patient or the dental team members. In addition, it should have adequate physical properties, be easy to manipulate, and be relatively inexpensive.¹ The success of the MCR depends on the presence of a strong bond between porcelain and metal substructures.² Proper tech-

niques and careful selection of porcelain and metal combinations are essential for clinically successful MCRs. For proper bonding, the coefficient of thermal expansion of the ceramic and metal should be compatible.³

The significant increase in the price of gold in the 1970s led to a substantial rise in the price of gold dental alloys. This prompted development of less expensive alternative alloys. However, these substitutes, particularly base metal alloys, have been less than ideal, as their hardness makes clinical and laboratory manipulation very difficult.¹ Also, they are potential biological hazards.⁴⁻⁶ Biologic safety concerns, including adverse

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local or systemic reactions from exposure to certain elements in metal biomaterials, have been reported.^{7,8}

The use of titanium in dentistry has increased because of its superior biocompatibility, corrosion resistance, desirable physical and mechanical properties, and relatively low cost.⁹⁻¹² Titanium is used for endosseous implants and removable and fixed partial dentures.¹³⁻¹⁵ However, titanium reacts strongly with gaseous elements such as oxygen at high temperature, and yields an excessively thick layer of TiO₂.^{16,17} Such an oxide layer is considered a detriment to titanium-porcelain bonding, as it can easily break or spall. The metal oxide layer should be a monolayer in thickness to create an effective metal-ceramic bond.¹⁸ Therefore, it is essential that porcelain firing should occur below 800°C to prevent excessive oxide formation.^{16,17} Adachi et al¹⁷ reported that the commercially pure titanium specimens oxidized at 750°C showed a well-adhering oxide layer, 32 nm in thickness, whereas the oxide layer formed on specimens heated to 1000°C was approximately 1 μm thick, with significantly lower adherence.

Another possible source of conventional porcelain-titanium bond failure is the stress caused by the mismatch of the thermal expansion coefficient of titanium and ceramic that may affect the flexural bond strength of the titanium ceramic system. Dental ceramics with coefficient of thermal expansion values less than 8.5×10^{-6} °C are appropriate for titanium-ceramic restorations.¹⁹

Some manufacturers have introduced low-fusing porcelains designed for bonding to titanium with low-fusing temperatures (<850°C) and favorable thermal expansion coefficients.²⁰⁻²² Several studies have evaluated the quality of bonding low-fusing porcelain to titanium.^{17,20-26} These authors have shown that such bonding was acceptable,^{20-22,24,25} but variations exist between different titanium and ceramic systems.²² Some studies reported that the bond strength was comparable to that of the NiCr and Pd alloys and conventional ceramics systems,²⁴ while others reported inferior values.^{20-22,25,26}

Several secondary factors may enhance the titanium-ceramic bond strength. These include alteration of the titanium surface using airborne-particle abrasion,²⁷ acid etching,²⁷ and application of a bonding agent^{22,24,27} prior to ceramic application. Although airborne-particle abrasion of the titanium surface prior to ceramic application improves the adhesion of the ceramic to the titanium substrate,²⁷ this procedure remains technique sensitive. Derand and Hero²³ observed that the use of larger alumina particles with a diameter of 250 μm, compared with 50-μm particles, significantly improved the bond between titanium and ceramic. It is possible that the small particles may embed in the titanium surface. In fact, Gilbert et al²⁴ found that airborne-particle abrasion could contaminate the surface of titanium with alumina particles, which could weaken the

mechanical interlocking of the porcelain and titanium. Contamination of the titanium surface might also decrease its corrosion resistance and biocompatibility.²⁸

Reyes et al²⁷ evaluated several chemicals for treating the titanium surface prior to ceramic application. The authors observed that specimens treated with hydrochloric acid required more energy to break bonds compared with airborne-particle-abraded specimens and attributed this to the importance of a roughened surface. Moreover, the authors studied the effect of sulfuric acid and peroxide on titanium-ceramic bond strength and reported that although the use of peroxide resulted in the highest surface roughness, it yielded the lowest bond strength. The authors concluded that "increasing surface roughness does not necessarily result in an increase in bond strength." The use of gold-based bonding agents on gold alloys²⁹ or on titanium²³ did not enhance the metal-ceramic bond strength. As a result, some manufacturers provide a specially formulated bonding agent for use when bonding low-fusing porcelains to titanium, which has been shown to enhance titanium-ceramic bonding.^{22,24,27}

The aims of the present study were to investigate the following: (1) the bond compatibility of a low-fusing porcelain to a commercially pure titanium with and without porcelain bonding agent, (2) the effect of titanium surface treatment with airborne-particle abrasion or hydrochloric acid on the titanium-ceramic bond, and (3) the effect of using a combination of surface treatment and bonding agent on titanium-ceramic bond. The research hypothesis was that the bonding agent and the proposed surface treatment would enhance titanium-ceramic bond.

MATERIAL AND METHODS

Sixty specimens were cast with commercially pure titanium type I (Rematitan; Dentaaurum Inc, Pforzheim, Germany) using a casting unit (Rematitan autocast; Dentaaurum Inc). To obtain same-sized specimens, wax patterns were fabricated with an aluminum mold with dimensions of 28.0 × 3.2 × 0.8 mm. Eight patterns were sprued together and invested in 1 casting ring. The wax patterns were invested with silica- and phosphate-free, alumina and magnesia-based investment (Rematitan Plus; Dentaaurum Inc). The investment and casting procedures were completed according to the manufacturer's instructions. The closed interconnecting 2-chamber system of the casting machine allowed melting and casting to occur, first under a vacuum and then under argon gas with pressure of 0.8 bar, followed by automatic melting and casting.

The resultant metal specimens were sequentially wet-ground with 500-, 600-, and 1000-grit silicon carbide papers (Polo250/3; Jean Wirtz, Dusseldorf, West Germany) to ensure complete removal of the alpha (α) case layer, and to achieve the final dimensions of 25.0

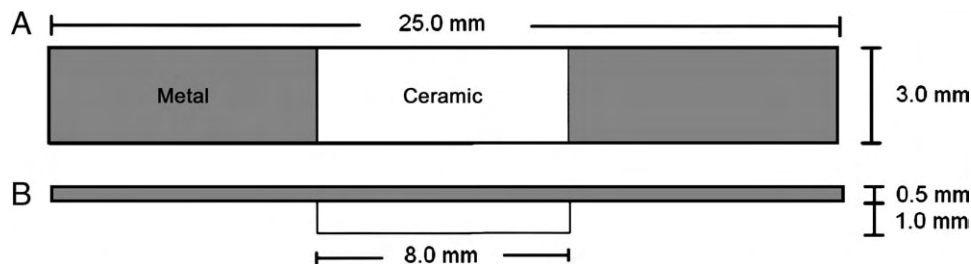


Fig. 1. Specimen design (ISO 9693).³⁰ A, Top view. B, Side view.

Table I. Titanium-ceramic bond strength (N=10)

	Bonding agent			No bonding agent		
	NST	APA	HCA	NST	APA	HCA
Mean	24.65 ^a	35.60	24.65 ^a	14.35 ^b	25.60	15.10 ^b
SD	6.34	8.15	4.61	4.01	5.41	2.97

Mean values with similar letters were not significantly different ($P > .05$). NST, No surface treatment (control group); APA, airborne-particle abrasion; HCA, hydrochloric acid.

Table II. Two-way ANOVA

	Sum of squares	df	Mean square	F	P
Bonding agent	1485.037	1	1485.037	48.919	<.001
Surface treatment	1589.175	2	794.588	26.175	<.001
Bonding × Treatment	1.425	2	0.713	0.023	.977
Error	1639.275	54	30.357		
Total	4714.912	59			

× 3.0 × 0.5 mm as required by the International Standard Organization (ISO) 3-point bending test specification 9693 (Fig. 1).³⁰ The thickness of the specimens was verified using a micrometer (Mitutoyo; Mitutoyo Corp, Tokyo, Japan) to the nearest 0.01 of a millimeter. The specimens were placed on a glass slab and visually observed to ensure planar configuration. The specimens were divided into 3 groups of 20 each to receive different treatment. The specimens in group NST received no surface treatment and served as the control, those in group APA were subjected to airborne-particle abrasion, and those in group HCA were treated with hydrochloric acid (HCl).

The airborne-particle abrasion was accomplished using 250-μm aluminum oxide particles at 2 to 3 bars air pressure (Sandstorm; Vaniman Inc, Fallbrook, Calif). The HCl treatment was performed by immersing the specimens in 10% by weight aqueous solution of HCl in a heat-resistant glass container and boiling for 30 minutes, taking care to avoid contact between specimens. Subsequent to the respective surface treatments, all specimens were subjected to ultrasonic cleaning (Vitasonic II; Vita Zahnfabrik, Bad Sackingen, Germany) using distilled water for 10 minutes and then rinsed in distilled water. Specimens were left to dry before the application of porcelain. Finishing procedures for all the specimens were performed by a single investigator.

Porcelain application

Low-fusing porcelain (Noritake Super Porcelain; Noritake Dental Supply Co Ltd, Nagoya, Japan) was

used in this investigation. The specimens in each group were further divided into 2 subgroups of 10 each. Ten specimens were not treated further, and a bonding agent, which was supplied with the porcelain, was applied to 10 specimens. Heat pretreatment of the specimens was performed immediately after cleaning procedures in a furnace (Multimat Mach 2; Dentsply Intl, York, Pa), according to the manufacturer’s instructions. Porcelain-bonding agent powder and liquid were mixed according to the manufacturer’s instructions. A thin layer was painted with a short bristle brush on the 8 × 3-mm porcelain-bearing area in the central portion of the respective specimens. The bonding agent was dried at the muffle entrance of the furnace and fired according to the manufacturer’s instructions.

Porcelain was added to the metal specimens to the dimensions of 8 mm in length, 3 mm in width, and 1 mm in thickness in the central portion of each metal strip, according to the ISO 9693 specifications (Fig. 1).³⁰ The entire process followed the manufacturer’s instructions and began with the application of 2 uniform coats of opaque porcelain, using a brush, on the porcelain-bearing area. The body porcelain was formed on the opaque layer using an aluminum matrix and fired at 500°C to 760°C with a heat rate of 40°/min under a vacuum of 72 cm/Hg. The firing shrinkage was compensated for by applying a second layer of body porcelain, yielding a final total thickness of 1 mm. Finally, the glaze layer was applied and fired at 500°C to 760°C with a heat rate of 50°/min with no vacuum. Porcelain application for all the specimens was accomplished by a single investigator.

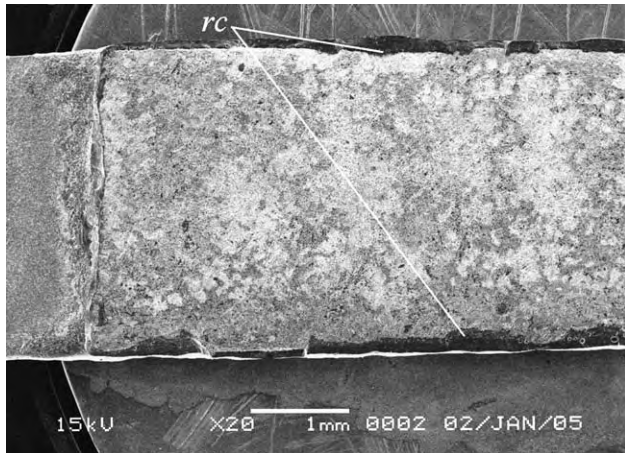


Fig. 2. Photomicrograph of failed surface of NST without bonding specimen. Dark area represents retained ceramic (*rc*); light area represents titanium (Original magnification $\times 20$).

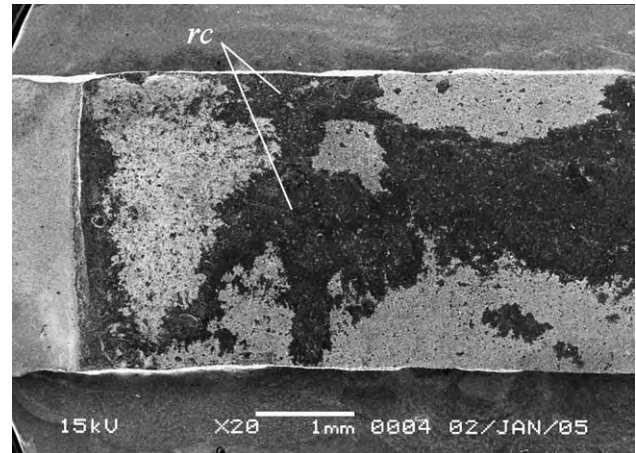


Fig. 3. Photomicrograph of failed surface of APA with bonding specimen. Dark area represents retained ceramic (*rc*); light area represents titanium (Original magnification $\times 20$).

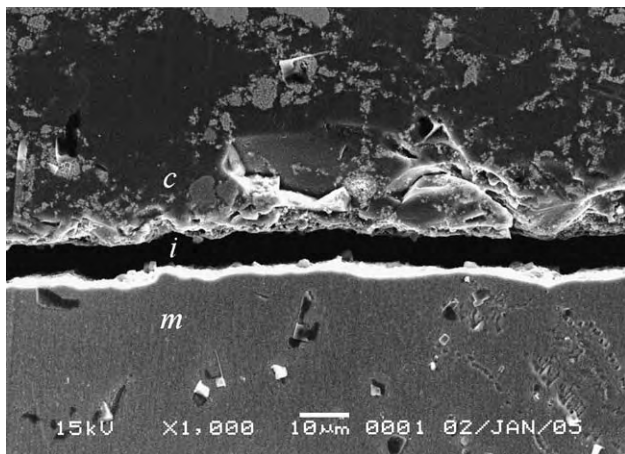


Fig. 4. Photomicrograph of cross-section of titanium-ceramic interface for NST without bonding specimen. *c*, Ceramic; *i*, metal-ceramic interface; *m*, metal (Original magnification $\times 1000$).

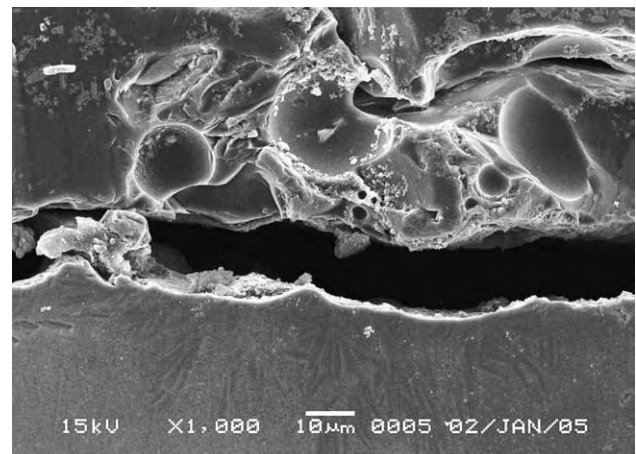


Fig. 5. Photomicrograph of cross-section of titanium-ceramic interface for HCA with bonding specimen (Original magnification $\times 1000$).

Testing procedures

The bond strength testing was performed with a 3-point bending test on a servo-hydraulic universal testing machine (Instron 8500R; Instron Corp, Canton Mass) using a load-cell capacity of 0.01 KN. The specimens were positioned on a specially fabricated metal support with the porcelain facing downward. The metal support was used to align and stabilize the specimens. A compressive load was applied at the midpoint of the metal strip with a rounded-tip loading rod at a crosshead speed of 0.5 mm/min until a sudden drop in load occurred in the load-deflection curve, indicating the bond failure. The failure load was recorded digitally with a personal computer using software provided by the manufacturer

of the testing machine. The bond strength (σ) was calculated by the following equation given in ISO 9693:³⁰

$$\sigma = k.F \text{ (N/mm}^2\text{)}$$

where *F* is the maximum force applied in Newtons before debonding (failure load), and *k* is a constant determined from a graph in ISO 9693 with units of mm^{-2} . The value of *k* depends on the thickness of the metal substrate and the elastic modulus of the metallic material, and for the commercially pure titanium tested it was determined to be 4.6 mm^{-2} .

Statistical software (SPSS for Windows, Release 11.0.0, 2001; SPSS Inc, Chicago, Ill) was used to generate descriptive statistics and perform inferential tests. A 2-way analysis of variance (ANOVA) and Tukey

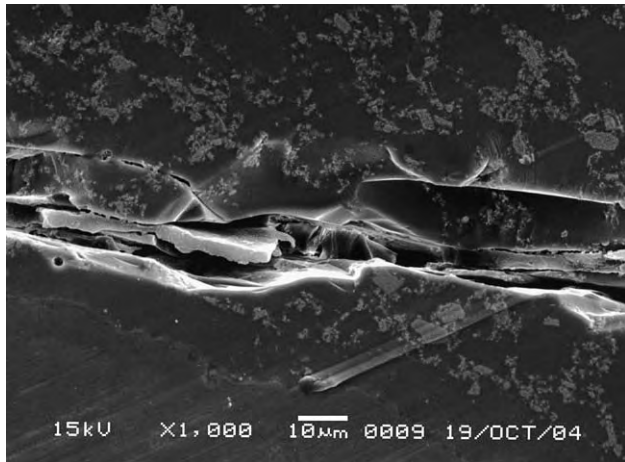


Fig. 6. Photomicrograph of cross-section of titanium-ceramic interface for APA with bonding specimen (Original magnification $\times 1000$).

multiple range tests were used to determine the statistical significance of the mean differences between groups ($\alpha=.05$).

SEM analysis

Two specimens from each group were selected for examination of the metal-ceramic interface under SEM (JSM-636OLV; JOEL Ltd, Tokyo, Japan). These specimens were embedded in clear autopolymerizing acrylic resin (Orthoresin; Dentsply). The specimens were sectioned in the midsection with a low-speed saw (Isomet 2000 precision saw; Buehler Ltd, Lake Bluff, Ill) along their width so that the cross-sectional area could be examined. The sectioned specimens were ultrasonically cleaned for 10 minutes and manually polished with an abrasive paper (Polo250/3; Jean Wirtz) with grits of 240, 320, 400, and 600. Final polishing of the specimens was accomplished with a rotary polisher (Automata; Jean-Wirtz) with aluminum-oxide polishing paste (Micropolish II; Buehler Ltd). Another 2 specimens from each group were selected for examination of the failed surfaces of the metal-ceramic interface. These specimens were ultrasonically cleaned in distilled water for 10 minutes prior to the making of the SEMs. Photomicrographs were made of a different region of each specimen to evaluate the metal-ceramic interface.

RESULTS

The mean load at bond failure and SD values are presented in Table I. The 2-way ANOVA showed significant differences ($P<.001$) among the titanium-ceramic bond strengths with the bonding agent and different surface treatments (Table II). The results of the Tukey multiple range tests showed significant differences in the load at bond failure between APA with bonding agent group and the rest of the groups ($P<.001$), indicating that

the combination of airborne-particle abrasion surface treatment and bonding agent provide the greatest effect on the titanium-ceramic bond as compared with all tested groups. Statistical analyses also showed that using a bonding agent or surface treatment with airborne-particle abrasion enhanced the titanium-ceramic bond strength. Specimens treated with HCl, with or without bonding agent, showed no significant difference compared with the control specimens ($P=.975$).

Scanning electron microscopy

The photomicrographs of the cast titanium surface after debonding demonstrated residual porcelain retained on the metal surface for all groups (Fig. 2 and Fig 3). This observation indicates a combination of cohesive and adhesive bond failures. However, more traces of porcelain were observed on specimens that were treated with the combination of airborne-particle abrasion and bonding agent, which may indicate that the failure was primarily adhesive for the rest of the groups.

Photomicrographs of cross-sections of tested specimens demonstrated that the fracture line was located more often between ceramic and metal for all the groups except for specimens that were treated by the airborne-particle abrasion and bonding agent combination, where the fracture line, in some areas, occurred primarily within the porcelain material (Figs. 4 to 6).

DISCUSSION

The study results suggested that part of the research hypothesis was accepted. The use of surface airborne-particle abrasion and bonding agent enhanced the titanium-ceramic bond. However, titanium surface treatment with HCl produced no effect on the titanium-ceramic bond. The minimum debonding/crack-initiation strength for porcelain/metal combinations set by ISO 9693 is 25 MPa.³⁰ The titanium-ceramic bond of the control group, which had no surface treatment or bonding agent, was the weakest among all the tested groups (14.35 MPa)—far below the ISO requirement. This study indicates that an unmodified titanium surface, that is, a surface not receiving airborne-particle abrasion or bonding agent, may lead to an unsatisfactory titanium-ceramic bond. This is in agreement with previous studies.^{24,27} Wang and Fung³¹ have indicated that the unmodified titanium surface produces a weak, porous, nonprotective and nonadherent oxide layer that is unsuitable for porcelain bonding. As seen in the photomicrograph (Fig. 2), the titanium specimen shows some areas with remnants of porcelain. While the porcelain remnants indicate some degree of bonding, it was not adequate to meet the ISO standard.

The group treated by airborne-particle abrasion produced significantly greater bond strength than the

control group ($P < .001$). This finding concurs with those of Reyes et al.²⁷ Airborne-particle abrasion likely improves the bond strength by removing loosely attached furrows, overlaps, and flakes of metal created by grinding procedures, provides mechanical interlocking, increases surface area, and increases wettability.^{27,32-34}

The present study demonstrated that HCl surface treatment did not have a significant effect on the titanium-ceramic bond as compared with the control group. The mean bond strength of the HCA group without bonding (15.1 MPa) was significantly below the lower limit value of 25 MPa in the ISO 9693 standard. The specimens appeared greenish in color after boiling in HCl for 30 minutes, indicating the formation of an oxide layer. This might explain the weak bond strength. It is possible that a shortened period of boiling may have resulted in a thinner oxide layer; however, further study is needed. Photomicrographs showed that HCl produced a greater degree of roughness in the surface of titanium than the control group (Figs. 4 and 5). Surface roughness should increase surface area and, thus, may improve the titanium-ceramic bond; however, for the HCA group without bonding this was not the case. It appears that roughening of the metal surface does not necessarily enhance the flexural bond strength.²⁷ Also, the surface roughness may increase the stress concentration at the metal-ceramic interface, and generate steep reentrant angles that may prevent complete wetting and result in voids at metal-ceramic interfaces.³⁵ Surface roughness measurement was not part of this study; however, it is possible that HCl may produce an overroughened titanium surface which may interfere with complete wetting and production of voids. This may be seen in the cross-section of an HCA specimen (Fig. 5).

Reyes et al.²⁷ observed that HCl-treated specimens require more energy to break the titanium-ceramic bonds than airborne-particle-abraded specimens, which is in conflict with the present study. The disagreement may be explained by differences in the methodology. Reyes et al.²⁷ used 50- μm abrasive particles, while 250- μm particles were used in the present study. Derand and Hero²³ observed that the use of 250- μm alumina particles, compared with 50- μm particles, significantly improved the bond between titanium and ceramic. Gilbert et al.²⁴ found that airborne-particle abrasion could contaminate the surface of titanium with alumina particles, which could weaken the mechanical interlocking of the porcelain and titanium.

The use of a bonding agent significantly enhanced the titanium-ceramic bond ($P < .001$). Several studies have reported similar results.^{22,24,27} It is believed that the bonding agent may enhance the titanium-ceramic bond strength by preventing the formation of a nonadherent oxide, which is otherwise formed when titanium is exposed to high temperatures.^{24,27} The fine particles

of titanium in the bonding agent act as oxygen scavengers, inhibiting progressive build-up of a nonadherent oxide layer with each firing cycle.²⁴ In addition, bonding agents contain a mixture of titanium and ceramic particles that may reduce the thermal expansion mismatch between metal and the ceramic material.²⁴ The authors believe that the titanium-ceramic bonding mechanisms are complicated, inadequately understood, and require further investigation.

Atsu and Berksun²² investigated 3 titanium-ceramic systems, 2 of which were provided with a bonding agent. The authors demonstrated that the bond strength of the ceramic system used without a bonding agent was greater than one of the systems that used a bonding agent. The authors reported that the thickness of the bonding agent that produced the weakest bond strength was 30 to 40 μm at the interface as observed in SEM. The excessive thickness, as well as the composition of bonding agent, may have affected the metal-ceramic bond. The authors also reported that the highest bond strength was recorded with the same bonding agent that was used in the present study.

Derand and Hero²³ reported that using bonding agent on titanium causes a significant decrease in the bond strength. This may be related to the fact that the authors used a bonding agent made to be used with gold alloys. Also, the bonding agent and the ceramic used in that study were from different manufacturers and might have been incompatible with each other.

Surface treatments using airborne-particle abrasion and bonding agent together produced significantly greater bond strength when compared with all the tested groups. This may be due to the enhancement of metal wettability when the bonding agent is applied over an airborne-particle abraded surface. The HCl surface treatment and bonding agent combination did not provide any improvement in the titanium-ceramic bond strength compared with the control group. This finding indicates that the roughness produced by airborne-particle abrasion may be more favorable to the bonding agent than the roughness produced by HCl.

It has been shown that the weak area of the titanium-ceramic bond was the excessive and nonadherent oxide layer at the interface, and this problem must be solved to obtain greater bond strengths.^{16,17} The present study results suggest that the use of surface airborne-particle abrasion along with an appropriate bonding agent will provide the highest bond strength of the porcelain to the titanium tested. A limitation of this study was that only 1 brand of low-fusing porcelain and 1 brand of titanium were tested; the findings related to these 2 products may not be extrapolated to similar materials. Also, if measuring the oxide layer thickness had been part of the study, helpful observations might have been obtained to better understand the behavior of the materials. In addition, the mode of failure may not directly

correspond to the clinical situation, although the 3-point bending test is widely accepted and used by several investigators to evaluate the metal-ceramic bond strength.^{20-22,24-27}

CONCLUSIONS

Within the limitations of this investigation and for the materials used in this study, the following conclusions were made:

1. Surface treatment using airborne-particle abrasion significantly enhanced the bond between the cast, commercially pure titanium to low-fusing porcelain.

2. The use of a bonding agent significantly improved the titanium-porcelain bond strength.

3. The combination of airborne-particle abrasion and bonding agent produced the most significant improvement in the titanium-ceramic bond.

4. Titanium surface treatment with hydrochloric acid, with or without bonding agent, produced no effect on the titanium-ceramic bond, as compared to the control specimens.

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