Original Article

Effects of Sandblasting and Silicoating on Bond Strength between Titanium and Porcelain

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alumina particles (110 µm) on titanium (Ti) surface to identify the most effective method of increasing the bond strength between porcelain and Ti. Materials and Methods: Thirty rectangular plates (15 mm \times 50 mm \times 1 mm) of commercially pure Ti (Cp Ti) Grade 5 (GC Dental Industrial Corporation, Tokyo, Japan) were divided into three groups for different surface modification procedures (n = 10). Ti bonder porcelain, opaque, and dentin layers were fired separately on Ti plates. All specimens were placed in a bending jig for four-point bending test. The load and crosshead displacement data were collected to calculate the strain energy release rate as a G value. **Results:** Lowest mean G values in J/m^2 were in the group sandblasted with 150 μ m Al₂O₃ particles (Group 2) (18.6 ± 5), followed by the group sandblasted with 50 μ m Al₂O₂ particles (Group 3) (20.8 ± 6.1) and the group sandblasted with 110 μ m silicoated Al₂O₂ particles (Group 1) (24.5 ± 4.1). The one-way ANOVA and Bonferroni post hoc tests indicated that there was a statistically significant difference between Group 1 and Group 2 (P < 0.05). There were also no statistically significant differences between Group 1 and Group 3 and Group 2 and Group 3 (P > 0.05). Conclusion: The size of alumina particles is not a factor that is directly effective in enhancing the bond strength of Ti-porcelain systems. The bond strength of Ti-porcelain systems can be extremely improved by the application of sandblasting with silica-coated alumina particles.

KEYWORDS: Bond strength, dental porcelain, surface treatment, titanium

Purpose: The aim of this study was to evaluate the effects of the different

sized alumina particles (50 and 150 µm) and tribochemical silica-modified

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INTRODUCTION

Metal-porcelain restorations continue to be the most widely used restorations owing to their adequate mechanical strength and pure esthetics.^[1] Metal substrates have an effect on the physical properties of metal-porcelain restoration. Nonprecious alloys have the disadvantages of poor biocompatibility, low corrosion resistance, poor bond strength, and easy discoloration of porcelain. On the other hand, usage of commercially pure titanium (Cp Ti) and its alloys as a nonprecious metal substrate in dentistry has increased in recent decades due to their excellent biocompatibility, high strength, high heat resistance, high corrosion resistance, and low cost.^[2,3] Ti is a nonprecious metal used in dental implants, removable and fixed partial dentures.^[4-6] Not

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only is Ti more expensive than common base metals such as nickel and chromium for the patient, but its usage is also complicated for dentists. The highly oxidative nature of the surface of Ti is an assumption as the cause of the weak bond strength between porcelain and Ti.^[7]

Intimate contact between metal and porcelain can be accomplished by increasing wettability of the metal surface, which can be made by increasing surface energy.^[8] Many physical and chemical surface treatments

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of Ti have been proposed such as sandblasting with alumina + steam cleaning or ultrasonic cleaning,^[9] coating with Au,^[10] silicon nitride,^[11] Cr and ceramic,^[12] acid treatments (HF, H_2SO_4 , and H_2O_2), or varied surface modification techniques (Silicoater, Silicoater MD, Rocatec, Siloc Systems). All these surface treatments have been used to achieve a better bonding between porcelain and Ti until today. Bonding agents are also considered to play a major role in Ti–porcelain bonding. The fine particles of Ti in bonding agents act as oxygen scavengers and protect the surface from excess oxidation.^[13]

Sandblasting with alumina particles is the most common method recommended for creating surface roughness and providing mechanical interlocking force for porcelain. Airborne-particle abrasion procedure of the Ti surface before ceramic application is technically sensitive.^[14] Airborne-particle abrasion could contaminate the surface of Ti with alumina particles, which could weaken the mechanical interlocking between the porcelain and Ti.[13] Caustic and acid baths such as nitric and hydrochloric acids or sodium hydroxide aqueous solutions have been successfully used for the cleaning of contaminated Ti surface after heat treatment. The size of alumina particles is another effective factor on the bond strength between porcelain and Ti. The use of a large particle size of alumina was advantageous in increasing the surface roughness and interlocking of Ti with porcelain.^[15,16] However, it is true that there is not adequate information concerning the influence of Ti surface modification procedures in improving the bond strength between Ti and porcelain.

The aim of this study was to evaluate the effects of the differently sized alumina particles (50 and 150 μ m) and tribochemical silica-modified alumina particles (110 μ m) on Ti surface so as to identify the most effective method for increasing the bond strength between porcelain and Ti.

MATERIALS AND METHODS

Thirty rectangular plates (15 mm \times 50 mm \times 1 mm) of Cp Ti Grade 5 (GC Dental Industrial Corporation, Tokyo, Japan) were machined using 5-axis milling machine (AVAMILL Chrome VHS-5000-5A, AvaDent computer-aided design/computer-aided manufacturing [CAD/CAM], Izmir, Turkey). The machined Ti plates were ground on a 600-grit silicon carbide paper (Atlas Zimpara, Seza Group, Istanbul, Turkey) under running water and then cleaned in ultrasonic bath (Bandelin, Sonorex, Germany) filled with distilled water for 5 min.

The thirty specimens were divided into three groups according to surface modification procedures as stated below (n = 10).

Group 1

The specimens were sandblasted with silicoated alumina particles with diameter of 110 μ m (Rocatec PreUniversal Bonding System, 3M ESPE, 3M Deutschland GmbH, Neuss, Germany) according to the manufacturer's instructions (13 s duration, 2.8 bar pressure, 10 mm distance). Then, the silane solution (ESPE Sil, 3M ESPE, 3M Deutschland GmbH, Neuss, Germany) was applied on the silicoated Ti surfaces and allowed to dry for 5 min.

Group 2

The specimens were abraded with alumina particles with diameter of 150 μ m using a dental airborne-particle abrasion unit (EasyBlast, Bego GmbH and Co. KG, Bremen, Germany) according to the manufacturer's instructions (20 s duration, 2 bar pressure, 15 mm distance). Then, all specimens were cleaned in an ultrasonic bath (Bandelin, Sonorex, Germany) and filled with distilled water for 5 min.

Group 3

The specimens were abraded with alumina particles with diameter of 50 μ m using a dental airborne-particle abrasion unit (EasyBlast, Bego GmbH and Co. KG, Bremen, Germany) according to the manufacturer's instructions (20 s duration, 2 bar pressure, 15 mm distance). Then, all specimens were cleaned in an ultrasonic bath (Bandelin, Sonorex, Germany) and filled with distilled water for 5 min.

After the surface treatment of all Ti specimens, a thin layer of bonder porcelain (GC Initial Ti Bonder, GC Dental Industrial Corporation, Tokyo, Japan) was applied, followed by opaque and dentin layers (GC Initial Ti, GC Dental Industrial Corporation, Tokyo, Japan). Then, all layers were fired separately according to the manufacturer's instructions in a dental vacuum porcelain furnace (Programat P500, Ivoclar Vivadent, Germany). All porcelain surfaces that would be in contact with the rollers of four-point bending jig were ground to achieve flat and smooth surfaces using 240-grit and 320-grit silicon carbide papers (Atlas Zimpara, Seza Group, Istanbul, Turkey), respectively.

As part of the specimen preparation for four-point bending test, the specimens were notched across their widths and entirely through the depth of the porcelain layer at the middle of the specimen using a water cooled low-speed diamond saw (Isomet, Buehler GmbH, Düsseldorf, Germany). Then, the specimens were placed in a bending jig and the applied load was controlled by a screw-knob on the control panel. The precrack started from the base of the notch and extended along the interface with a total length of 2 mm approximately [Figure 1]. Tamac, et al.: The bond strength between porcelain and titanium

The precracked specimens were then placed in a custom-designed four-point bending jig mounted in a universal testing machine (Autograph AG-50kNEe, Shimadzu, Japan) with the inner rollers 14 mm and outer rollers 26 mm. They were subjected to load at a crosshead speed of 0.05 mm/min until the crack reached the inner rollers. The load and crosshead displacement data were collected for calculation of the strain energy release rate as a *G* value. The following formula is used to calculate the strain energy release rate, *G* value for each specimen:^[17]

$$G = \frac{h(P^2 l^2 [1 - v_m^2])}{E_m b^2 h^3}$$

where, P, load

l, distance between inner and outer rollers

 v_{m} , Poisson's ratio of metal

 E_{m} , Elastic modulus of metal

b, width of specimen

- $h_{\rm m}$, thickness of metal
- h_{n} , thickness of porcelain
- h, thickness of specimen

The nondimensional parameter η is calculated as follows:

$$\eta = \left(\frac{3}{2}\right)\left(\frac{1}{\left(h_{m} / h\right)^{3}} - \frac{\lambda}{\left(\frac{h_{p} / h^{3} + \lambda[h_{m} / h]^{3}}{+3\lambda[h_{p}h_{m} / h^{2}]}\right)}\right)$$
$$\lambda = \frac{E^{m}\left(1 - v_{p}^{2}\right)}{E_{p}\left(1 - v_{m}^{2}\right)}$$

All data sets were subjected to a Shapiro–Wilk test to evaluate the normality of the distribution. Data were analyzed with one-way ANOVA and the Bonferroni *post hoc* test using SPSS 19.0 (SPSS Inc. Chicago, IL, USA) for Windows.

RESULTS

The mean values and standard deviations of adhesion or bond strength (Gc) of Ti–porcelain bonding systems together with 95% confidence interval are presented in Table 1. The group sandblasted with 110 μ m silicoated Al₂O₃ particles (Group 1) demonstrated the highest mean bond strength value (24.5 ± 4.1) (J/m²). The lowest mean bond strength value (18.6 ± 5) (J/m²) was seen in a group

Table 1: Mean and standard deviation of the strain energy release rate (J/m²) of all groups						
Groups	$G_{c}(J/m^{2})$	95% CI for mean				
	-	Lower bound	Upper bound			
Group 1	24.5±4.1	21.5375	27.5179			
Group 2	18.6±5	15.0064	22.1829			
Group 3	20.8±6.1	16.4822	25.2374			
Total	21.3±5.5	19.2472	23.4076			
CI=Confider	nce interval					

 Table 2: One-way ANOVA on strain energy release rate
Sum of squares df Mean square Р F 179.282 G_a value 2 89.641 3.358 0.048 between groups Within groups 27 720.729 26.694 900.011 29 Total

Table 3: Results of the Bonferroni post hoc analysis						
Comparison	rison Mean P 95% CI for m		for mean			
between	difference		Lower	Upper		
the surface			bound	bound		
modification						
procedures						
Group 1						
Group 2	5.9330099*	0.048	0.035374	11.830646		
Group 3	3.6678307	0.372	-2.229805	9.565466		
Group 2						
Group 1	-5.9330099*	0.048	-11.830646	-0.035374		
Group 3	-2.2651792	1.000	-8.162815	3.632457		
Group 3						
Group 1	-3.6678307	0.372	-9.565466	2.229805		
Group 2	2.2651792	1.000	-3.632457	8.162815		

*P = 0.05. CI=Confidence interval



Figure 1: Placement of the specimens in a custom-designed four-point bending jig. l = Distance between inner and outer rollers, b = Width of specimen, h metal = Thickness of metal, h porcelain = Thickness of porcelain, P = Force

sandblasted with 150 μ m Al₂O₃ particles (Group 2). The one-way ANOVA test indicated that there was a significant difference between groups (*P* = 0.048) [Table 2].

For the results of Bonferroni *post hoc* test, Group 1 showed significantly higher mean G_c values than Group 2 (P < 0.05). However, there were no statistically

significant differences between Group 1 and Group 3 and Group 2 and Group 3 (P > 0.05) [Table 3].

DISCUSSION

Although the use of Ti and its alloys has many advantages in the field of dentistry, the surface of Ti is really technically sensitive on the Ti porcelain interface. Growth of thick oxide layer on Ti alloys at high temperatures, adherence of self-formed oxide to Ti alloys, and bonding by fusion of self-formed oxide with porcelain were reported as failures in Ti-porcelain bonding. Ti reacts strongly with oxygen at high temperatures (1000°C) and forms a thick (1 µm) TiO, layer that interferes with Ti-porcelain bonding. Therefore, lower temperature (750°C) porcelain firing is required to form a thin (32 nm) TiO₂ layer and to prevent excessive oxide formation. On the other hand, the mismatch of coefficient of thermal expansion between porcelain and Ti alloys leads to residual stresses within the porcelain and flexural bond strength of the metal-ceramic systems, which may contribute to failure.

The fabrication of Ti-porcelain restorations has technically sensitive procedures such as fabrication of Ti coping, Ti surface treatment for increasing bond strength, and application and firing of porcelain onto Ti coping to complete the restoration.

Casting is the traditional production method for the fabrication of Ti copings. On the other hand, Ti has a high melting point (1.668°C) and strong affinity to oxygen, nitrogen, and carbon at high temperatures, which require casting circumstance to be in a vacuum or inert gas chamber. Because of several technical problems associated with the Ti casting method such as shrinkage and porosity formation, CAD/CAM machining and laser sintering of Ti were developed as alternative production methods of Ti copings.^[16]

Ti-porcelain bonding is still a subject that should be researched. Ti-porcelain bonding is influenced by the surface properties of the Ti substrate.^[18] The technique of sandblasting with alumina particles has been commonly employed for many purposes in dentistry, including removal of contaminants, increase of effective surface area, and improvement of wetting ability of porcelain.^[14,19] Previous studies reported that the use of a large particle size of alumina was advantageous in increasing the surface roughness and promoting mechanical interlocking of Ti with porcelain.^[20] However, when the Ti surface was sandblasted with different sized conventional alumina particles (50 µm, 150 µm) in the present study, no statistically significant differences were detected in bond strength of porcelain to Ti between these two groups. Wang et al. also found that there was no statistically significant difference among sandblasted

Ti surface with alumina particles with a diameter of 50 and 125 μ m.^[16] Another factor affecting the bond strength seemed to be amount and behavior of alumina particles embedded in Ti. The alumina particles are embedded into Ti by the process of sandblasting.^[15,21] If the particles are loosely embedded in Ti, the porcelain will be peeled off from Ti surface with alumina particles. On the contrary, if the particles are tightly embedded in Ti, the alumina particles will give an effect of interlocking and enhance the bonding.

Silica-modified alumina particles are the aluminum oxide particles coated with a thin layer of silica. The sandblasting with silica-modified alumina particles could form a tribochemical coating on the surface of the adherent surface.^[22,23] This process is known as "cold silicatization" as it prevents the thermal stressing within the metal and the distortion of metal. In the previous studies^[9,16] that examined the metal surface sandblasted with silica-modified alumina particles by X-ray microanalysis, the researchers pointed out the existence of the embedded particles or trace of silica on the concave wall after the particle had removed. The bond strength of porcelain to Ti is improved with the presence of silica. In this in vitro study, the results with the highest bond strength of Ti-porcelain systems were found in the group sandblasted with silica-coated alumina.

Measuring the bond strength of metal-ceramic systems has been traditionally carried out using shear tests. However, these tests ignore the nature of the stresses generated within the adherence zone, which can have a significant effect on the mode of failure. Three-point bending test is commonly used for this measurement. However, recently, four-point bending test started to be preferred over the three-point bending test because of its advantages. Four-point bending test has some advantages such as stability of crack extension along the interface, prevention of high stress concentration, prevention of overload occurring at the initiation of crack growth, and measurement without the influence of other variables in the metal substrate or veneering ceramics.^[17,24,25] The strain energy release rate can be used for comparison of bond strengths of various biomaterial systems in dentistry.^[26] Therefore, the four-point bending test was selected for adhesion testing in this study.

CONCLUSION

Within the limitations of this study, the following conclusions are made:

1. The bond strength of Ti–porcelain systems can be extremely improved by the application of sandblasting with silica-coated alumina particles 2. The size of alumina particles is not the directly effective factor in enhancing the bond strength of Ti-porcelain. Larger sized particle only increases the surface roughness of Ti.

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Conflicts of interest

There are no conflicts of interest.

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