EGYPTIAN
DENTAL JOURNAL
www.eda-egypt.org

Vol. 69, 1397:1407, April, 2023

Print ISSN 0070-9484 • Online ISSN 2090-2360

Fixed Prosthodontics and Dental Materials

Submit Date : 29-10-2022 • Accept Date : 05-02-2023 • Available online: 10-04-2023 • DOI : 10.21608/edj.2023.171597.2329

INFLUENCE OF SURFACE TREATMENT AND THERMOCYCLING ON PORCELAIN BONDING TO TWO FORMS OF TITANIUM

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ABSTRACT

Statement of problem. Porcelain fracture or chipping are considered a frequent clinical problem of titanium-ceramic restorations. Although titanium-ceramic bonding remains a challenge, data on the influence of surface treatment on titanium-ceramic bonding are lacking.

Purpose. The purpose of this in vitro study was to investigate the influence of surface treatment and thermocycling on porcelain bonding to two forms of titanium.

Material and methods. Forty titanium specimens (25x3x0.5 mm) were machined by using wire-cut electric discharge machining (WEDM) technology and assigned into two groups as per the material form (n=20): Cp Ti grade 2 and Ti-6Al-4V alloy. The specimens in each group were further assigned into two subgroups (n=10) as per the tested surface treatment: airborne-particle abrasion and hydrochloric acid etching treatment. In addition, Ni-Cr alloy specimens (n=10) were provided as a control. Ultra-low fusing porcelain was applied at the center of the treated titanium specimens. Half of the titanium specimens in each subgroup (n=5) were subjected to thermocycling (5000 cycles). Flexural bond strength measurements for all specimens were determined by using a universal testing machine at a crosshead speed of 0.5 mm/min. A representative specimen from each group was observed by using a scanning electron microscope at ×200 magnification. Data were analyzed statistically by using 2-way ANOVA and the Student-Newman-Keuls (SNK) post hoc tests (α=.05).

Results. No statistically significant difference in bond strength was found between both forms of titanium and the control group (*P*=.805). The mean and ±SD bond strength values (before and after thermocycling) ranged from 44 ±2 MPa to 48 ±4 MPa. In addition, the effect of surface treatment and thermocycling on porcelain bonding to both forms of titanium was not statistically significant (*P*=.481 and .864). The SNK post hoc tests showed no significant difference between the Cp Ti grade 2 and Ti-6Al-4V alloy specimens (*P*>.05).

Conclusions. The bond strength of both forms of titanium (Cp Ti grade 2 and Ti-6Al-4V alloy) to ultra-low fusing porcelain was comparable to that of the control group. Both airborne-particle

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abrasion and hydrochloric acid etching treatment resulted in adequate porcelain bonding to both forms of titanium; however, the difference in bond strengths between both forms of titanium was not statistically significant. Thermocycling did not affect porcelain bonding to both forms of titanium. Owing to their acceptable bond strength, both forms of titanium can be used in clinical practice.

Clinical Implications: Treating Cp Ti grade 2 and Ti-6Al-4V alloy by using airborne-particle abrasion with 110-µm Al_2O_3 and 10% hydrochloric acid for 10 minutes results in adequate porcelain bonding to both forms of titanium.

KEYWORDS: Cp Ti grade 2 , Ti-6Al-4V alloy, airborne-particle abrasion , hydrochloric acid etching , Flexural bond strength.

INTRODUCTION

Commercially pure titanium (Cp Ti) and Titanium-6Aluminum-4Vanadium alloy (Ti-6Al-4V) are considered promising alternatives to nickel-chromium, cobalt-chromium, and gold alloys because of their good corrosion resistance, low thermal conductivity, low density, outstanding mechanical properties, and excellent biocompatibility.[1-5] In prosthetic dentistry, both forms of titanium have been used to fabricate removable dental prostheses, frameworks for metal-ceramic fixed dental prostheses (FDPs), and implant-supported prostheses by using different fabrication techniques, including the conventional casting technology, computer-aided design and computer-aided manufacturing (CAD-CAM), electric discharge machining (EDM), and selective laser melting (SLM).[6-12]

However, the casting of titanium and its alloys when used in metal-ceramic FDPs remains a challenge because of their high melting temperature (1672 °C) and extreme chemical reactivity with elements in the investment such as oxygen at high temperatures.[2,4,13] This resulted in the development of a thick layer (α-case) on the titanium surface containing mainly titanium oxides.[14-16] The presence of this brittle, nonadherent layer can result in diminished mechanical properties, reduced ductility, increased surface roughness and porosities, and inferior titanium-ceramic bonding.[17,18]

To overcome casting problems, titanium restorations can be milled from prefabricated titanium blocks by using the CAD-CAM technology, thus eliminating the problems of porosities and contraction of titanium castings.[19-21] In addition, unconventional machining methods, including wire-cut electric discharge machining (WEDM) technology have provided an alternative method for the fabrication of titanium restorations.[22-24] Both CAD-CAM and WEDM technologies reduce the formation of the α-case layer.[2,8]

Adequate bonding between metal substructures and veneering ceramics is considered an important factor to ensure the clinical longevity of metal-ceramic FDPs.[25,26] Factors that influence titanium-ceramic bonding include the formation of an oxide layer at high temperatures, the tendency of the resulting oxides to adhere to the titanium surface or porcelain, and the development of stresses on the titanium-ceramic interface attributed to the incompatibility between coefficients of thermal expansion (CTEs) of titanium and porcelain.[27] For these reasons, dental manufacturers improved the bond strength by developing ultra-low fusing porcelains (<850 °C) with favorable CTEs.[28] However, bonding porcelain to different forms of titanium remains a challenge and has been questioned.[29]

Several surface treatments have been proposed to successfully achieve acceptable porcelain bonding to titanium, including acid etching, airborne-particle abrasion, thin chrome coating, silicon nitride coating, and application of a bonding agent.[26-38] The disparity in bond strength outcomes in these trials could be related to differences in testing methodologies, the material type, the geometry of the specimen, and surface treatment methods.[2] Furthermore, the bond strength may be compromised by the repeated stresses on the titanium-ceramic interface during thermocycling to mimic clinical conditions, leading to porcelain chipping and fracture.[28,39]

The purpose of this in vitro study was to determine the influence of surface treatment and thermocycling on porcelain bonding to two forms of titanium. The null hypotheses were that no difference in bond strength would be found between porcelain and both forms of titanium after surface treatment and thermocycling.

MATERIAL AND METHODS

The materials evaluated are specified in Table 1. Based on a study by Tróia et al,[33] a sample size of 45 specimens had an effect size (F=0.693) and a power of 95% with a significance level (α=.05) to test the null hypothesis that no difference in bond strength would be found between porcelain and both forms of titanium after surface treatment and thermocycling. To accomplish more consistent results, the number of specimens was raised to 50 (n=10) for each group. In 95% (the statistical power) of those experiments, *P* was <.05. A statistical software program (G*Power v3.1.9.2) was used to calculate the sample size. A schematic presentation of the specimen design and 3-point bending test apparatus is shown in Figure 1.

Forty titanium specimens were machined to the desired dimensions from 1.0 mm sheet stock and assigned into 2 groups as per the material form (n=20): Cp Ti grade 2 and Ti-6Al-4V alloy. These specimens (n=40) were machined in standardized thickness (25×3×0.5 mm), in compliance with the requirements of International Organization for Standardization (ISO) standard 9693-1:2012 specifications.[40]

TABLE (1) Materials evaluated

Metal/alloy	Manufacturer	Composition (wt.%)	Lot No.	Fabrication Technique
Cp Ti Grade 2	RMI Titanium Co	Ti (99.8), C (0.01), N (0.008), Fe (0.06)	451102	EDM
Ti-6Al-4V alloy	RMI Titanium Co	Ti (89), Al (6.11), V (4.05), C (0.02), N (0.02), Fe (0.19)	9150840	EDM
Ni-Cr alloy (4all)	Ivoclar Vivadent AG	Ni (61.4), Cr (25.7), M (11.0), Si (1.5), Mn (<1.0), Al (<1.0), C (<1.0)	K16250	Lost-wax

EDM, electric discharge machining.

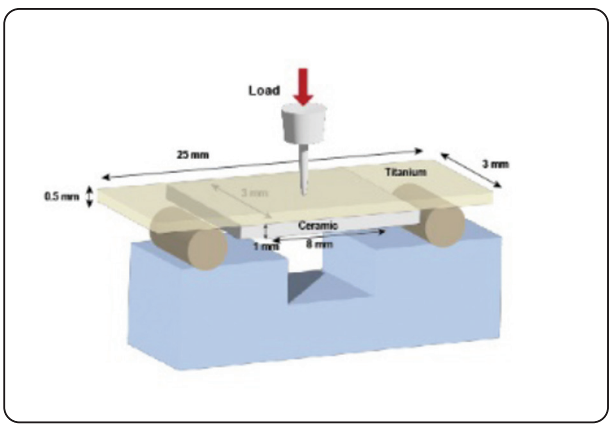

Fig. (1) Schematic representation of specimen design and 3-point bending test apparatus.

The WEDM machine (CX-20; Mitsubishi) consists of a worktable, wire electrode, dielectric supply system, servo control system, and power supply. A copper wire (Ø 0.25 mm) was used as a tool and distilled water was used as a dielectric fluid. The desired dimensions were obtained with electricity by using spark erosion under carefully controlled conditions. These sparks were generated between a negative electrode (metal workpiece) and a positive electrode (copper wire). Both electrodes were immersed in a dielectric fluid (distilled water) that flushes away the metal flakes produced by the sparks. The produced sparks resulted in high temperatures on the workpiece surface, resulting in the melting and vaporizing of a small area of the metal workpiece until the required shape was obtained. The machining parameters for this process were a 40-V power, 5-mm/min erosion speed, 5-seconds pulse on time, and 24-seconds pulse off time.

The resultant titanium specimens were sequentially wet-polished by using 200-grit up to 1000-grit silicon carbide abrasive paper (Buehler Ltd.) to assure α-case layer removal and to accomplish the desired dimensions, as recommended in ISO standard 9693-1:2012.[40] Digital calipers (Dial Caliper D; Aura Dental) with 0.1 mm accuracy were used to confirm width and length.

For the control group, 10 Ni-Cr alloy specimens were cast by using conventional casting technology. To ensure a uniform thickness of the specimens, autopolymerizing acrylic resin patterns (Duralay; Polidental) (n=10) were fabricated by using a brass mold with the dimensions of (25.1x3.1x0.7mm). Sprues with a 3 mm diameter were attached to the resin patterns and subsequently invested by using a phosphate-bonded investment (Ceramvest; Protechno) in a metal ring according to the manufacturer's instructions. After setting, the metal ring was heated to 850 °C for 25 minutes in a burnout furnace (Vulcan A-130; Dentsply Sirona) and then placed in casting equipment (SM-2 CEN MOTOR; Mestra) to cast Ni-Cr alloy specimens. The ring was subsequently devested by using 250-µm Al_2O_3 particles (Protechno) in an airborne-particle abrasion unit (ESB 2; Eurocem Srl) under 0.2-MPa pressure for 10 seconds at a 10 mm distance. All specimens were observed for planar configuration and checked for fit inside the brass mold for any refinements.

Each group of the produced titanium specimens was divided into two subgroups (n=10) as per surface treatment: airborne-particle abrasion (APA) and hydrochloric acid etching treatment (HCA). For APA, specimens (n=20) were airborne-particle abraded by using 110 μm Al_2O_3 particles (Protechno) for 10 seconds under 0.2-MPa pressure at a 10-mm distance. For HCA, specimens (n=20) were submerged in a 10 wt% aqueous solution of hydrochloric acid and boiled for 10 minutes in a heat-resistant glass vessel. For the control group, APA was accomplished as above. Subsequently, all specimens were steam cleaned (EGV 18; Eurocem Srl) for 15 seconds and then allowed to dry for 10 minutes before porcelain application.

For the titanium specimens (n=40), ultra-low fusing porcelain (Triceram; Dentaurum GmbH & Co KG) was applied at the center of each titanium specimen to the dimensions of (8x3x1 mm), in accordance with the requirements of ISO standard 9693-1:2012 specifications (Fig. 2).[40] A specially designed brass mold was constructed to provide 1 mm mold space, thus ensuring a standardized ceramic thickness. A thin uniform layer of paste bonder (Triceram; Dentaurum GmbH & Co KG) was applied onto the titanium specimen and fired in a calibrated oven (Vacumat 40T; VITA Zahnfabrik GmbH & Co KG) from 500 °C to 795°C and held for 60 seconds at 795 °C. After cooling, 2 opaque porcelain layers (Triceram; Dentaurum GmbH & Co KG) were applied onto the specimen covered by the bonder and fired with the same firing parameters as above. After opaque firing, the thickness of the opaque layer (0.3 mm) was verified by using the same calipers. Subsequently, 2 dentin porcelain layers were applied onto the opaque layer and fired from 500 °C to 755 °C and held for 60 seconds at 755 °C. For the control group, felspathic porcelain (VMK 95; VITA Zahnfabrik GmbH & Co KG) was applied by using the same brass mold following the same sequence of layering as above and fired according to the manufacturer's instructions.

After porcelain application, Half of the specimens in each subgroup (n=5) were subjected to thermocycling (5 ºC to 55 ºC, 5000 cycles) in a thermocycling machine with a 5 second transfer time between baths and 10 second dwell time in each bath.[31,41]

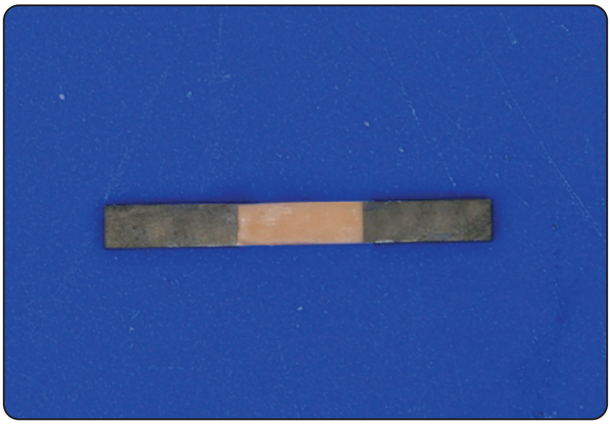

Fig. (2) Titanium-ceramic specimen with dimensions as recommended in ISO 9693-1:2012. ISO, International Organization for Standardization.

All specimens were submitted to the 3-point bending test in a universal testing machine (Model LRX-plus; Lloyd Instruments Ltd), in compliance with the requirements of ISO standard 9693-1:2012 specifications.[40] The specimens were positioned on 2 supports with a span distance of 20 mm and then loaded (5 KN) at the midline of the metal specimens by using a bi-beveled metallic chisel at a speed of 0.5 mm/min until failure occurred (Fig. 3). The failure resulting from loading was recorded by using a software program integrated with the testing machine (Nexygen MT; Lloyd Instruments). The bond failure load was recorded in newtons (N) and bending strength (in MPa) was calculated by using the following formula[42]: $\Sigma=3PI/2bd^2$, where Σ=flexural bond strength (MPa), P=maximum load to failure (N), I=span between the supports (mm), b=specimen width (mm), and d=specimen height (mm). Furthermore, the failure mode of a debonded specimen from each treated group (before and after thermocycling) was evaluated by using scanning electron microscopy (SEM) (JSM-6360LV; Jeol Ltd) at a 10 mm working distance with a 7-kV acceleration voltage at ×200 magnification. Failure modes were categorized as adhesive failure between the ceramic and titanium specimen; cohesive failure within the ceramic or titanium specimen; or mixed (both adhesive and cohesive) failure.[4,33]

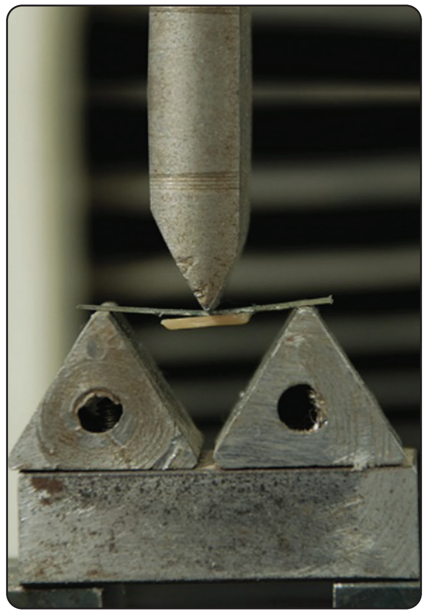

Fig. (3) Titanium-ceramic specimen submitted to 3-point bending test in universal testing machine, with porcelain side being under tension. Porcelain veneer peeling off indicated bond failure.

Numerical bond strength data were represented as mean and standard deviation values. One-way ANOVA was used to compare the mean values among all groups. Two-way ANOVA followed by the Student-Newman-Keuls (SNK) post hoc tests were used to determine the difference among the mean values of the groups and the effects of surface treatment and thermocycling on porcelain bonding to two forms of titanium (α=.05). Statistical analysis was performed by using a statistical software program (GraphPad Prism,v4.0; GraphPad, Inc).

RESULTS

The mean values and standard deviations of the bond strength measured in MPa for all groups (before and after thermocycling) are summarized in Table 2. No statistically significant difference in bond strength was found between both forms of titanium and the control group (P=.805) (Table 3). In addition, the effect of surface treatment and thermocycling on porcelain bonding to both forms of titanium was not statistically significant (*P*=.481 and .864) (Table 4). The SNK post hoc tests showed no significant difference between the Cp Ti grade 2 and Ti-6Al-4V alloy specimens (*P*>.05). The SEM images of titanium specimens after debonding showed remnants of porcelain on the metal surfaces for all groups (before and after thermocycling). This suggested that the bond failure between titanium and ultra-low fusing porcelain was primarily adhesive; however, a mixed (cohesive/adhesive) bond failure was evident. Representative SEM images of debonded specimens are displayed in Figures 4 and 5.

TABLE (2) Mean values ±SDs of metal-ceramic bond strength (MPa) for all treated specimens

Materials Used	Bond Strength (Mean Values ±SDs)			
	APA		HCA	
	Before TC	After TC	Before TC	After TC
Cp Ti Grade 2	45 ±3[a]	45 ±5[a]	47 ±2[a]	45 ±3[a]
Ti-6Al-4V alloy	46 ±4[a]	46 ±3[a]	48 ±4[a]	44 ±2[a]
Ni-Cr alloy (Control)	48 ±2[a]			

APA, airborne-particle abrasion; HCA, hydrochloric acid; TC, thermocycling; SD, standard deviation. Same superscript uppercase letters indicate no significant differences (P >.05).

TABLE (3) One-way ANOVA for titanium and control groups

Source	SS	df	MS	F calc.	*P*	F tab.
Between groups	56.863	8	7.107	0.556	.805	2.208
Within groups	459.989	36	12.777			
Total	516.852	44				

df, degrees of freedom; MS, mean square; SS, sum of squares; calc., calculated; tab., tabulated.

TABLE (4) Two-way ANOVA assessing effects of surface treatment and thermocycling on bond strength

Source	SS	df	MS	F calc.	*P*	F tab.
Surface treatment	49.446	1	12.361	0.891	.481	2.714
Thermo cycling	43.191	1	6.169	0.445	.864	2.359
Error	388.086	28	13.860			
Total	480.723	30				

df, degrees of freedom; MS, mean square; SS, sum of squares; calc., calculated; tab., tabulated.

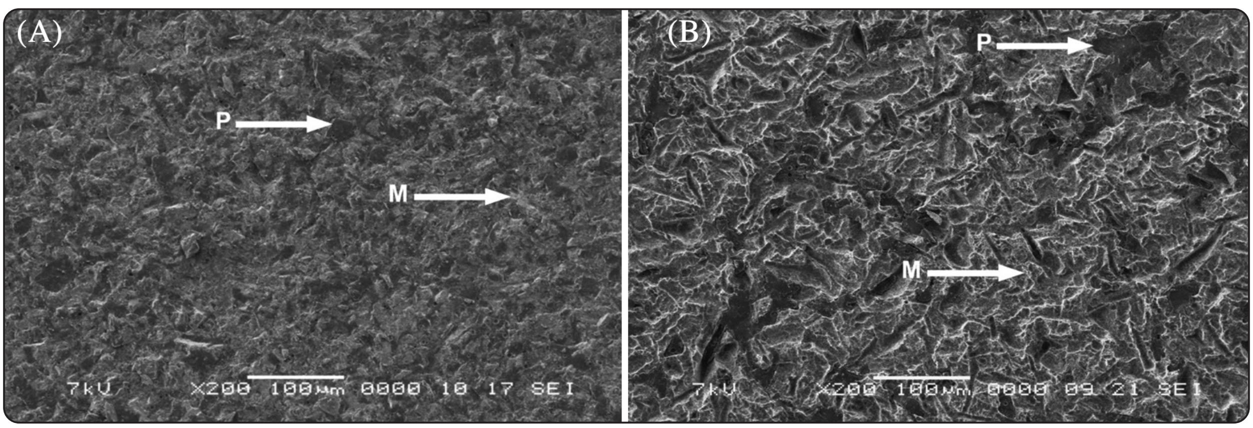

Fig. (4) A,B

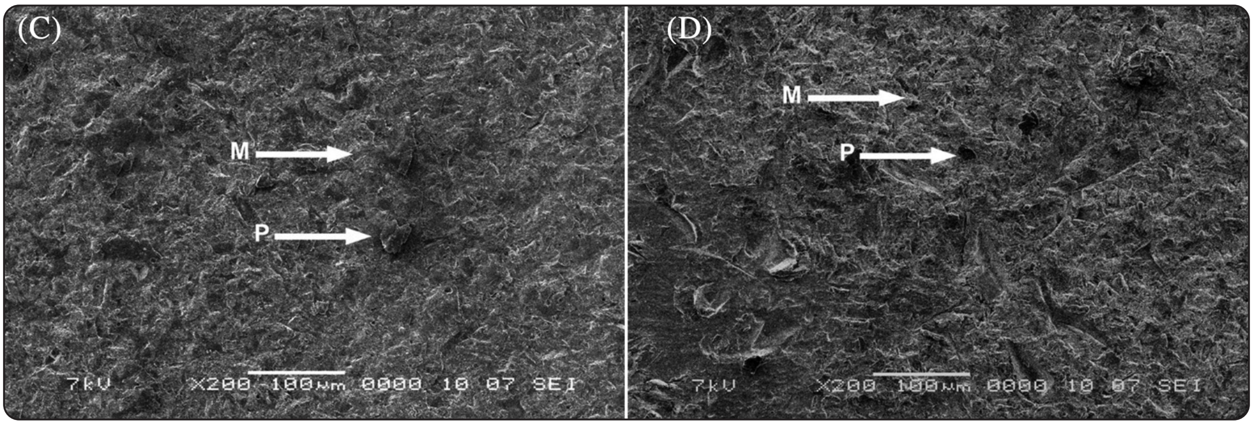

Fig. (4) Scanning electron microscope images of treated Cp Ti grade 2 surface before and after thermocycling. A, Air-borne particle abrasion. B, Hydrochloric acid treatment. C, Air-borne particle abrasion+thermocycling. D, Hydrochloric acid treatment+thermocycling. Cp Ti, Commercially pure titanium. M, Metal. P, Porcelain. Original magnification ×200.

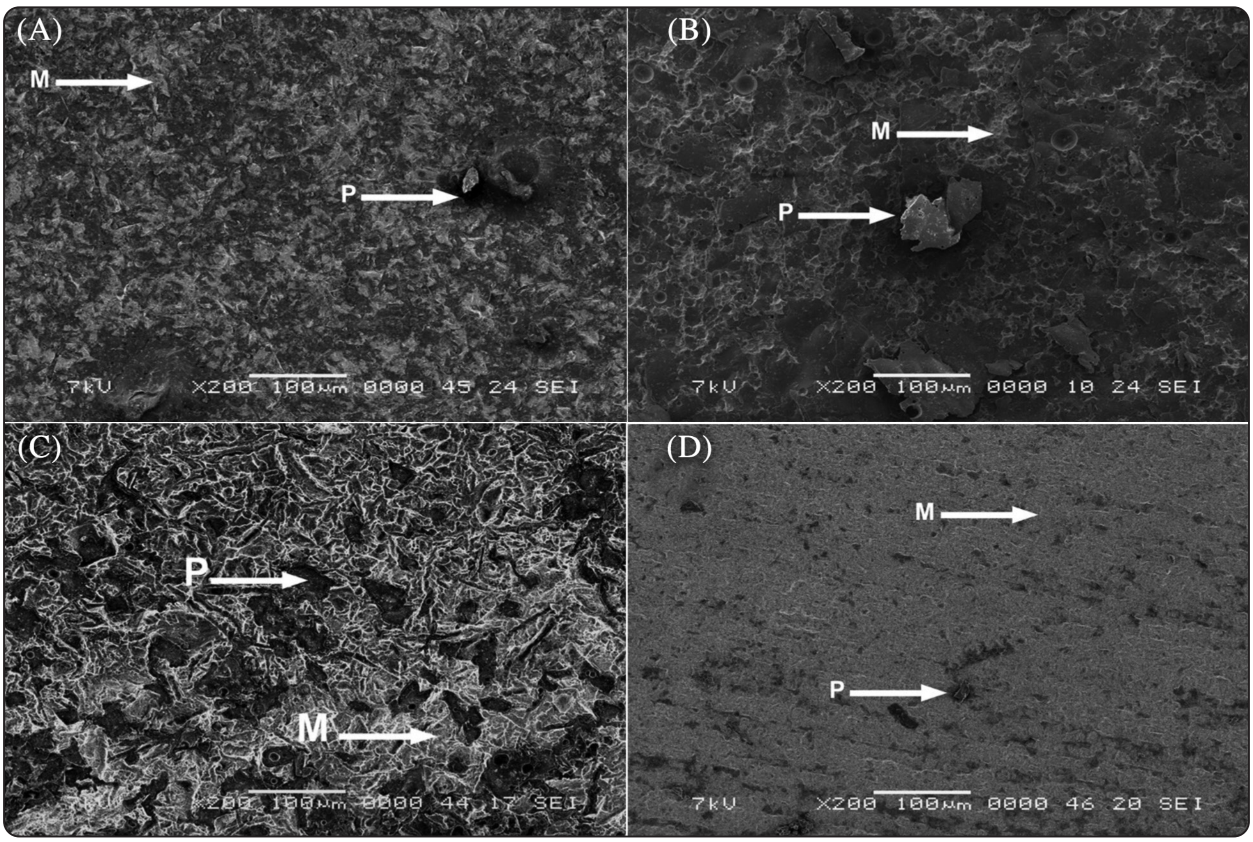

Fig. (5) Scanning electron microscope images of treated Ti-6Al-4V alloy surface before and after thermocycling. A, Air-borne particle abrasion. B, Hydrochloric acid treatment. C, Air-borne particle abrasion+thermocycling. D, Hydrochloric acid treatment+thermocycling. Ti-6Al-4V Titanium-6aluminum-4vanadium. M, Metal. P, Porcelain. Original magnification ×200.

DISCUSSION

This in vitro study examined the influence of surface treatment and thermocycling on porcelain bonding to two forms of titanium. The null hypotheses, stating that no difference in bond strength would be found between porcelain and both forms of titanium after surface treatment and thermocycling were not rejected.

In the present study, the mean bond strength values for both forms of titanium specimens after surface treatments (before and after thermocycling) ranged from 44 ±2 MPa to 48 ±4 MPa, thus exceeding the minimal value of 25 MPa recommended in the ISO 9693-1:2012 for metal-ceramic systems.[40] These findings demonstrated that airborne-particle abrasion (APA) and hydrochloric acid (HCA) surface treatments resulted in higher mean bond strength values than the recommended minimum values; however, the influence of both surface treatments on porcelain bonding to both forms of titanium was not significant (P>.05). Furthermore, no significant difference was observed among the titanium and control groups (P>.05).

The accepted bond strength between both forms of titanium specimens and ultra-low fusing porcelain might be because of the improved surface roughness of the titanium specimens resulting from APA and HCA surface treatments. This surface roughness provided mechanical interlocking between the metal and porcelain and increased the surface area for effective bonding.[35,36] Although roughening seems to be the main mechanism by which APA increases bond strength, it is probable that APA also works by reducing pronounced surface imperfections created by machining.[1,4] In contrast, hydrochloric acid treatment may produce over roughened titanium surface, resulting in voids at the metal-ceramic interface and preventing complete wetting. Moreover, placing the specimens for a long time in hydrochloric acid may produce an oxide layer which may affect titanium-ceramic bond strength.[4] Thus, boiling the specimens in hydrochloric acid for 10 minutes in the present study resulted in enhanced porcelain bonding to both forms of titanium. The findings obtained in this study were consistent with those of previous studies testing titanium-ceramic bond strength.[2,3,6,33] In contrast, the obtained results were against those of Reys et al,[35] who measured the bond strength of airborne-particle abraded and hydrochloric acid-etched specimens. They reported that more energy is required for the acid-treated specimens to break the titanium-ceramic bond. The conflict might be because of the disparities in particle–size used. Another explanation for the resulting bond strength is the use of a paste bonder that controls the surface oxidation of titanium during porcelain firing cycles.[1,2,4,34,35] In addition, the incorporation of ceramic and titanium particles in the bonding agents lessens the mismatch in CTEs between the ceramic material and metal,[19] possibly explaining the improved porcelain bonding to both forms of titanium.

Wire-cut electric discharge machining (WEDM) is a nontraditional machining and electrothermal process for metal removal by using a series of electric discharges (sparks) under controlled settings.[24] The main advantages of WEDM include the ability to machine hard metals or alloys, the production of desired shapes by using a single electrode, and the elimination of the α-case layer, thus enhancing titanium-ceramic bond strength.[24,43] Therefore, it was used in this study as an alternative to traditional fabrication techniques to produce surface roughness and improve bond strength.

Ultra-low fusing porcelain (Triceram) was used in this study for its low firing temperature (800 °C), thus preventing the formation α-case layer which could affect the titanium-ceramic bond strength.[25] In addition, Triceram has a low CTE, 8.6 to 9.2×10^{-6} °C compared with 9.6×10^{-6} °C for titanium, enabling a strong interfacial bonding.[2,4,26] Yilmaz and Dincer[6] reported that the acceptable difference in CTE between metal and porcelain at a given temperature is approximately 1.0 x 10^{-6} °C. For the control group, conventional porcelain was used because its outstanding properties were extensively studied in the dental literature.

In the present study, half of the specimens (n=20) were subjected to thermocycling of 5000 cycles to simulate 4 years of clinical practice.[28,31,39] The findings of this study showed that thermocycling had no significant effect on porcelain bonding to both forms of titanium, consistent with those of Tróia et al.[33] In contrast, the results conflicted with those of Parchańska-Kowalik et al,[32] who recorded that thermocycling reduced the strength of the titanium-ceramic bond, regardless of the surface treatment.

The results of this in vitro study suggested that both forms of titanium-ceramic systems can be used in clinical practice. However, information is lacking on the long-term performance of titanium-ceramic restorations. Limitations of this study included that only one fabrication technique (WEDM) and one type of ultra-low fusing porcelain (Triceram) were tested. Furthermore, the failure mode might not exactly be modeled to the clinical conditions. Clinical trials are necessary to confirm the results.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The bond strength of both forms of titanium (Cp Ti grade 2 and Ti-6Al-4V alloy) to ultra-low fusing porcelain was comparable to that of the control group.
2. Both airborne-particle abrasion and hydrochloric acid treatment resulted in porcelain bonding to both forms of titanium that exceeded the minimal bonding values of 25 MPa; however, the difference in bond strengths between both forms of titanium was not statistically significant.
3. Thermocycling did not affect porcelain bonding to both forms of titanium.
4. Owing to their acceptable bond strength, both forms of titanium can be used in clinical practice.

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