

BOND STRENGTH OF PORCELAIN TO TITANIUM AND TITANIUM ALLOY, A COMPARATIVE STUDY WITH CONVENTIONAL METAL-CERAMIC SYSTEMS

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ABSTRACT

Purpose: Titanium metal's affinity for gaseous element such as oxygen may affect the titanium-ceramic bonding. The purpose of this study was to evaluate the bonding potential between a low fusing porcelain and commercially pure titanium and titanium alloy, and to compare the bond strength with those of two conventional metal-ceramic systems.

Materials and Methods: Forty specimens were fabricated, 10 specimens for each group. Titanium casting unit was used to cast the commercially pure titanium and titanium alloy (Ti-6Al-4V) specimens and Noritake low fusing porcelain was applied to them. The gold-based and the Ni-Cr alloys were melted and cast with an automatic centrifugal casting machine and Vita VMK 95 conventional porcelain material was applied to them. A universal testing machine was used to perform the 3-point bending test. The metal-ceramic interfaces were subjected to scanning electron microscopic examination.

Results: The mean bond strength values obtained with the four different metals were 33.12 ± 6.16 MPa for the commercially pure titanium, 14.01 ± 5.63 MPa for the titanium alloy, 44.73 ± 5.63 MPa for the gold-based alloy and 40.76 ± 7.62 MPa for the Ni-Cr alloy. The gold-based alloy exhibited significantly greater bond strengths compared to the commercially pure titanium ($P < .001$). Furthermore, the commercially pure titanium group showed significantly greater bond strength than the titanium alloy group ($P < .0001$). There was no significant difference in the bond strength between the gold-based and the Ni-Cr groups ($P = .503$).

Conclusions: The bond strength of the conventional metal-ceramic combination was significantly greater than the bond strengths of the cast commercially pure titanium-Noritake ceramic combinations. Noritake porcelain showed significantly higher bonding strength to commercially pure titanium than to titanium alloy.

INTRODUCTION:

For any metal to be clinically successful as part of metal-ceramic restoration (MCR), it must be biocompatible so that it does not cause harmful toxicological or allergic effects to the patient or the

dental team members. Also, it should have adequate physical properties, be easy to manipulate, and relatively inexpensive.¹ Furthermore, the metal-ceramic chemical bond should be achievable. The optimum bond between the ceramic and the

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metal framework is essential for the clinically successful MCR. Several investigations have evaluated the quality of metal-ceramic bond for different porcelain-alloys combinations.²⁻⁵

The use of titanium in dentistry has noticeably increased because of its superior biocompatibility, corrosion resistance, desirable physical and mechanical properties, and low cost.⁶⁻⁹ The growing trend involves the use of titanium as an economical and biocompatible alternative for the existing alloys in dentistry. In prosthodontics, its use goes beyond the endosseous implants to removable and fixed partial dentures frameworks, either tooth supported or implant supported.¹⁰⁻¹² However, titanium's affinity for oxygen at high temperatures affects the titanium-ceramic bond since it may yield a thick nonadherent oxide layer. Therefore, porcelain firing should take place below 800°C in order to prevent excessive oxide formation.^{13,14} In addition, the mismatch of the coefficient of thermal expansion of titanium and porcelain may affect the flexural bond strength of titanium-ceramic system.¹⁵

Some manufacturers have introduced low-fusing porcelains designed for bonding to titanium. It has the potential to match the oxidation characteristics and low thermal expansion coefficient of titanium. Several studies have evaluated the quality of titanium-low fusing porcelain bonding.^{13,16-22} Pang et al¹⁹ compared bond strengths of three porcelain systems: cast titanium to Duceratin porcelain, machined-milled titanium to Procera porcelain and palladium-copper alloy to VMK 68 porcelain. The specimens were subjected to a three-point bending test. They found that bond strength of palladium-copper to VMK 68 porcelain was significantly greater than that of the two titanium-low fusing porcelain combinations. Moreover, it was reported that the bond strength of low-fusing porcelain bonded to cast commercially pure titanium or Ti-6Al-4V alloy was significantly lower than the conventional combination of porcelain-Pd-Ag alloy.²¹ Yilmaz and Dincer²² using three-point bending test, found that NiCr-VMK 68 porcelain bond was significantly stronger than the commercially

pure titanium-vita titan porcelain. Atsu and Berk-sun¹⁶ investigated the bonding strength of three commercial titanium low fusing porcelains (Vita Titankeramik, TiBond and Noritake Ti22), fired in vacuum and in argon atmosphere to cast and non-cast commercially pure titanium using a 3-point bending test. They used Ni-Cr alloy-conventional porcelain combination as control. They found that the Ni-Cr-conventional porcelain system fired in argon atmosphere had significantly higher bond strength than all the other systems. They also reported that the Ni-Cr-conventional porcelain and titanium-Noritake systems were the only systems that exceeded the lower limit of the bonding strength value in DIN 13927 (ISO 9693). They concluded that Titanium-Vita Titankeramik and Titanium-TiBond porcelain systems showed significantly lower bond strengths than the Ni-Cr-porcelain system.

Some of the manufacturers provide a special bonding agent to control the thickness of the oxide layer. It enhances the titanium-ceramic bonding.^{16,18,23,24} In addition, evidence suggested that the roughened titanium surface created by the sandblasting could also improve the titanium-ceramic bond.^{17,23,24} Derand and Hero¹⁷ reported that contamination of titanium surface was observed after casting and found it to be due to contact with the investment during solidification. They concluded that a thin surface layer (50-100 µm) of the casting should be removed before firing of the porcelain. They also found significant lower bond strength when the sandblasting alumina grains were reduced from 250 to 50 µm. They indicated that grinding the titanium surface with stones and subsequent blasting with 250µm sand removed a layer of approximately 100µm.

Several titanium alloys were suggested to be used in dentistry.^{25,26} Titanium can be alloyed with various elements primarily to improve the mechanical properties, such as strength, high temperature performance, creep resistance, weldability, and formability.^{8,27} Among the various titanium alloys, the titanium-aluminum-vanadium (Ti-6Al-4V) alloy was FDA-approved and one of the commonly

used titanium alloys in implant dentistry because of the improved physical and mechanical properties in comparison to commercially pure titanium.^{9,27}

The purpose of this study was to evaluate the bond strengths of a low fusing porcelain to commercially pure titanium and Ti-6Al-4V alloy after sandblasting and porcelain bonding agent application, and to compare them with those of two conventional metal-ceramic systems.

MATERIALS AND METHODS

The four metals tested in this investigation were commercially pure titanium (PTi), Ti-6Al-4V alloy (TiA), gold-based alloy (AuA) and nickel-chromium alloy (NiCr). The tested metals and their compositions are listed in table I. To obtain same-sized specimens, wax patterns were fabricated with an aluminum mold with dimensions of 28.0 x 3.2 x 0.8 mm. Forty specimens were fabricated, 10 of each metal. Titanium casting unit (Rematitan auto-cast, Dentaaurum Inc) was used to cast the PTi and TiA specimens. The wax patterns were invested with silica-free 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. For the AuA and NiCr groups, the wax patterns were invested with a phosphate-bonded investment material (FastFire 15, Whip Mix Corporation, Louisville, KY, USA) and cast according to manufacturer's recommendations. The alloys were melted and cast with an automatic centrifugal casting machine (Fornax 35EM, Bego, Bremen, Germany).

The resultant metal strips were sequentially wet ground with 500, 600, and 1000 grit silicon carbide papers (Polo250/3, JeanWirtz, Dusseldorf, West Germany) to ensure complete removal of the alpha case layer and to achieve the final dimensions of 25.0 x 3.0 x 0.5 mm as required by International Standard Organization (ISO) 3-point bending test specification 9693 (Fig. 1).²⁸ The thickness of the specimens was monitored using a micrometer (Mitutoyo, Mitutoyo Corporation, Tokyo, Ja-

pan) to the nearest 0.01 of a millimeter. The specimens were placed on a glass slab and visually observed to insure planar configuration. The specimens were sandblasted (Sandstorm, Vaniman Inc, Fallbrook, CA, USA) using 250 μ m aluminum oxide particles at 2-3 bars air pressure. After that, specimens were subject to ultrasonic cleaning (Vitasonic II; Vivadent, Irvine, Calif) using distilled water for 10 minutes, then rinsed in distilled water.

Low fusing porcelain (Noritake Super porcelain, Nagoya, Japan), supplied with a special bonding agent, was applied on the PTi and TiA specimens. For the AuA and NiCr specimens the conventional porcelain Vita VMK 95 (Vita, Bad Sackingen, Germany) was used. Heat pretreatment and porcelain application were completed according to the manufacturer's instructions. For the PTi and TiA specimens, a thin layer of the porcelain-bonding agent was painted with a short bristle brush on the porcelain bearing area in the central portion of the specimens. The bonding powder and liquid were mixed according to manufacturer instruction. The bonding agent was dried at the muffle entrance of the furnace for 5 minutes and fired according to the manufacturer's instructions.

The opaque porcelain was prepared and two uniform coats were applied with a brush on the designated area of the metal strips. The body porcelain was subsequently formed on the opaque layer using an aluminum matrix and fired. The firing shrinkage was compensated by applying a second application of the body porcelain yielding a final total thickness of 1 mm with a veneer area of 8 mm in length and 3 mm width, according to the ISO 9693 specifications (Fig. 1).²⁸ Finally, the glaze layer was applied and fired. The entire process of porcelain application followed the manufacturer's instructions and completed by one investigator.

The bond strength testing was performed with a 3-point bending flexure on a universal testing machine (Instron 8500R, High Wycombe, Bucks, UK). The specimens were positioned with the porcelain facing down on a specially fabricated metal

support that facilitates proper alignment and stability of the specimens. A compressive load was vertically applied at the midpoint of the metal strip with a rounded loading rod at a crosshead speed of 0.5 mm/min until a sudden drop in load was occurred in the load-deflection curve indicating bond failure. The failure load was recorded digitally with 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 9693:²⁸

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

where F is the maximum force applied in Newtons before de-bonding (failure load), and k is a constant determined from a graph in ISO 9693. The value of k depends on the thickness of the metal substrate and the elastic modulus of the metallic material.

One specimen from each group was randomly selected to examine the cross-section of the metal-ceramic interface under SEM (JSM-636OLV; JOEL Ltd, Tokyo, Japan). These specimens were embedded in clear autopolymerizing acrylic resin (Orthoresin; Dentsply, Weybridge, Surrey, England) and horizontally sectioned in the middle of the specimens with low speed saw (Isomet2000 precision saw, Buehler, Lake Bluff, Ill, USA). The sectioned specimens were ultrasonically cleaned for 10 minutes and manually polished with an abrasive paper (Polo250/3, JeanWirtz) through grits 240, 320, 400, to 600. Final polishing of the specimens was accomplished on a rotary polisher (Automata, Jeanwirtz, Charlottenster, West Germany) with aluminum oxide polishing paste (Micropolish II, Buehler). Another specimen from each group was selected to examine the failed surfaces of the metal-ceramic interface. These specimens were ultrasonically cleaned in distilled water for 10 minutes prior transferred to the SEM. Photomicrographs were taken from different region of each specimen to evaluate the metal-ceramic interface.

Statistical Package for Social Sciences (SPSS for Windows, Release 11.0.0, 2001, SPSS Inc, Chi-

cago, Ill) was utilized to generate descriptive statistics and inferential tests. A 1-way analysis of variance (ANOVA) and the Tukey's multiple range tests were used to determine the statistical significance of mean differences between groups. The significance level was set at 5%.

RESULTS

The mean load at bond failure and standard deviation values obtained with the four different metals are shown in table II. The table suggests that the bond strength of the TiA group (14.01 MPa) was the only tested group below the lower limit value set by the ISO 9693 standard for the 3-point bending test (25 MPa).

The 1-way ANOVA showed that the type of metal significantly influenced bond strengths ($P < .0001$) (Table III). Tukey's test confirmed that AuA group exhibited significantly greater bond strengths compared to the PTi and TiA groups ($P < .001$). Furthermore, the PTi group showed significantly greater bond strengths than the TiA group ($P < .0001$). No significant difference in the bond strength between AuA and NiCr groups was found ($P = .503$).

The photomicrographs of the metal surfaces after debonding showed residual porcelain retained on the metal surface for all groups (Figs. 2 to 4). This observation indicates a combination of cohesive and adhesive bond failures. However, more porcelain traces observed in specimens of AuA and NiCr indicating that the failure was mainly cohesive for these groups. TiA specimen surface showed good amount of voids of various sizes (Fig. 3).

Photomicrographs of cross section of tested specimens demonstrated that the fracture line was occurred more often within the porcelain material for AuA, NiCr and PTi groups. However, a fracture line between ceramic and metal was witnessed in some areas (Figs. 5 and 7). For TiA, the fracture line was located more often between ceramic and metal. Also, TiA specimen surface showed voids of various sizes in the bonding agent layer (Fig. 6).

Table I. Alloys studied

Alloy type	Product name	Manufacturer	Composition*	Code
Commercially pure titanium	Rematitan	Dentaurum Inc, Pforzheim, Germany	> 99.5% Ti, 0.003% N, 0.1% C, 0.015% H, 0.02% Fe, 0.18% O	PTi
Titanium alloy	Ti6Al4V	VSMPO, Verkhnyaya Salda, Russia	> 89% Ti, 6.5% Al, 4.2% V, 0.01 C, 0.17% Fe, <0.0005% Y, 0.178% O, 0.008% N, 0.0003% H	TiA
Gold-base	Heraloy G	Heraeus Kulzer, Hanau, Germany	51.5% Au, 37.9 % Pd, 8.5 % In, 2.0 % Ga, <0.1 % Ir, <0.1% Ru	AuA
Base metal	Remanium CS	Dentaurum Inc, Pforzheim, Germany	61.0% Ni, 26.0% Cr, 11.0% Mo, 1.5% Si, <1.0% Fe, Ce, Al, Co	NiCr

* provided by the manufacturers.

Table II. The metal-ceramic bond strength (n=10)

Alloy type	Mean (MPa)	Std. Deviation
PTi	33.12 a	6.16
TiA	14.01 b	5.63
AuA	44.73 c	5.63
NiCr	40.76 c	7.62

Mean values with similar superscript letters were not significantly difference.

Table III. One-way ANOVA

	Sum of Squares	df	Mean Square	F	P
Between Groups	5584.412	3	1861.471	46.745	<0.0001
Within Groups	1433.598	36	39.822		
Total	7018.010	39			

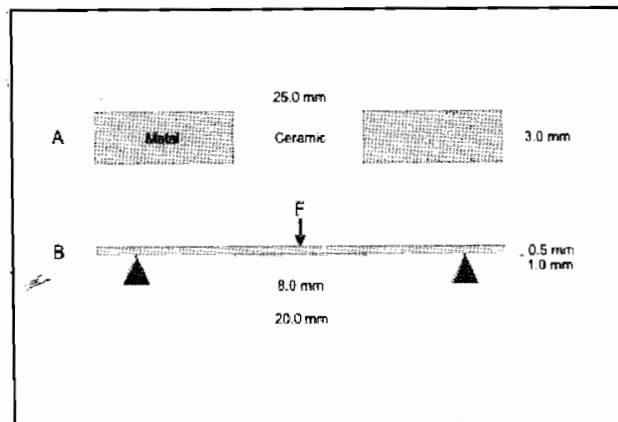


Fig. 1. Scheme of the specimen and 3-points flexure bond test (ISO 9693). A, top view; B, side view; F, force.

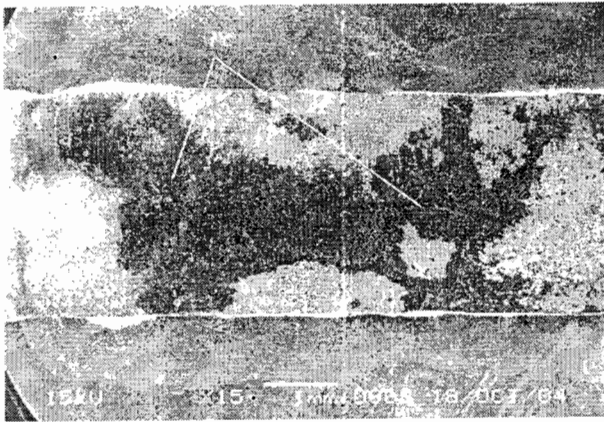


Fig. 2. Photomicrograph of the failed surface of PTi specimen. Dark area represents retained ceramic (rc), light area represents metal. (Original magnification x15.)

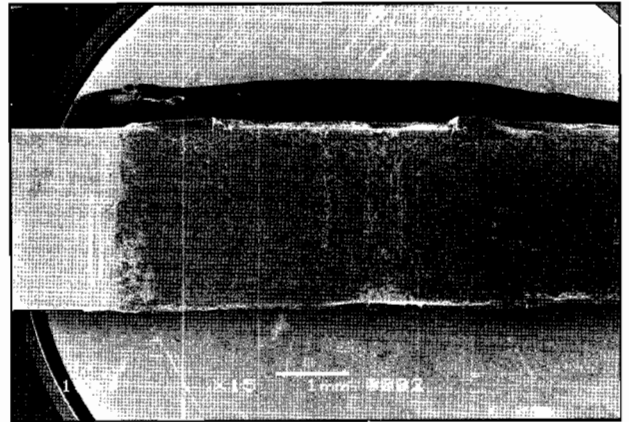


Fig. 3. Photomicrograph of the failed surface of TiA specimen. (Original magnification x15.)

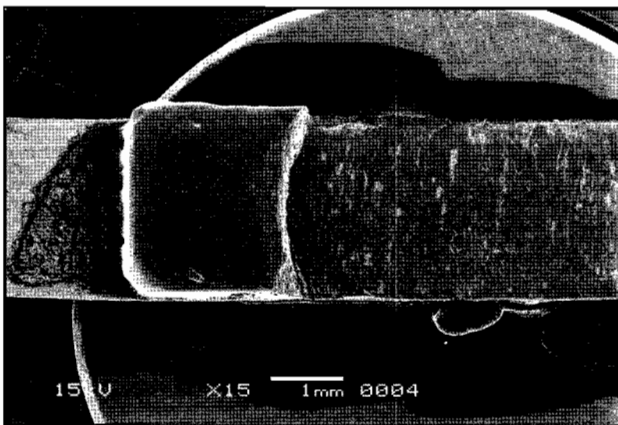


Fig. 4. Photomicrograph of the failed surface of AuA specimen. (Original magnification x15.)

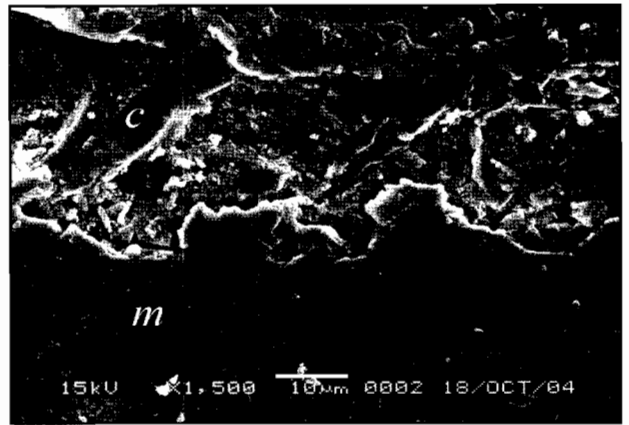


Fig. 5. Photomicrograph of cross section of metal-ceramic interface for PTi specimen. c, ceramic; m, metal. (Original magnification x1500.)

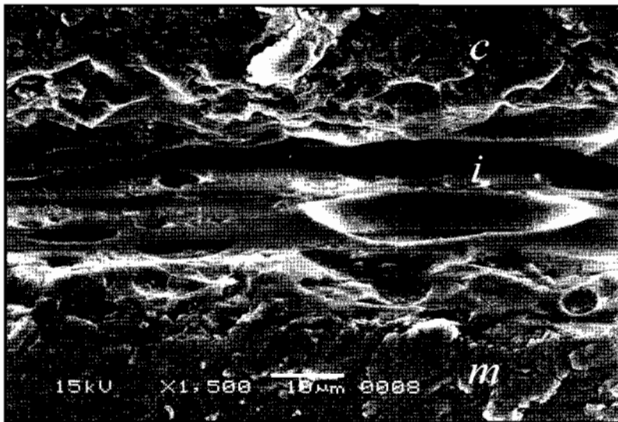


Fig. 6. Photomicrograph of cross section of metal-ceramic interface for TiA specimen. c, ceramic; i, metal-ceramic interface; m, metal. (Original magnification x1500.)

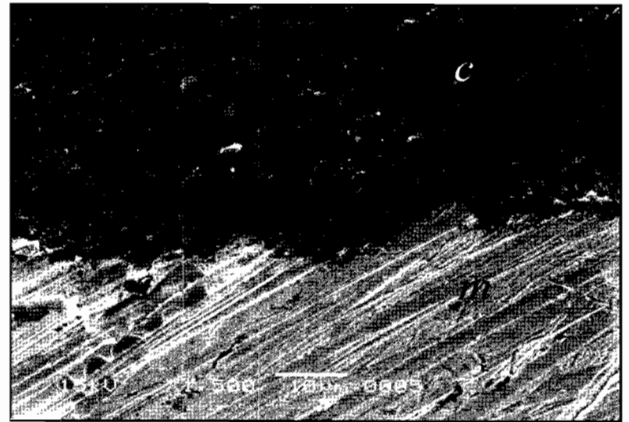


Fig. 7. Photomicrograph of cross section of metal-ceramic interface for AuA specimen. c, ceramic; m, metal (Original magnification x 1500.)

DISCUSSION

The study evaluated the bond strength of low fusing porcelain to type I cast commercially pure titanium and Ti-6Al-4V alloy, and compared them with those of conventional porcelain with gold-base and base metal alloys, using a 3-point bending test (ISO 9693). The results of the 3-point bending tests showed that the bond strength of conventional porcelain (Vita VMK 95) to AuA and NiCr alloys were significantly greater than those PTi- and TiA-low fusing porcelain (Noritake) systems. Inferior bond strength of titanium-low fusing porcelain in relation to Ni-Cr-conventional porcelain bond was reported by previous studies.^{16,20,22} In addition, Troia et al²¹ found that the bond strength of Pd-Ag alloy to conventional porcelain was significantly stronger than the low fusing porcelain bonded to cast commercially pure titanium or Ti-6Al-4V alloy. Also, Pang et al¹⁹ found that the bond strength of Pd-Co alloy to conventional porcelain was significantly greater than two titanium-porcelain combinations tested in their study. However, Gilbert et al¹⁸ concluded that when a titanium bonding agent is used, the porcelain fused to titanium bond strength is slightly but significantly larger than the high-palladium porcelain bond strength.

The titanium-porcelain bond strength ranged from 38% to 58% of the Ni-Cr-conventional porcelain bond as reported in 1996 by Probst et al²⁰ and from 33% to 60% as reported in 2000 by Atsu and Berksun.¹⁶ In the present study it was 81%. The improvement in the titanium-ceramic bond in this study maybe referred to the titanium surface preparation prior porcelain application, which was supported by previous studies' recommendation. The alpha-case layer was removed from the titanium's surface by grinding,¹⁷ then the surfaces were sandblasted^{17,23,24} and the designated bonding agent was applied.^{16,18,23,24}

However, this surface preparation was not enough to help in achieving PTi- and TiA-low fusing porcelain bond strength that was comparable to

that of conventional metal-ceramic systems. Controlling the titanium oxide layer by the use of low fusing ceramic and the designated bonding agent were not sufficient to reach the optimum titanium-ceramic bond. Adachi et al¹³ reported that titanium and Ti-6Al-4V showed good oxide adherence for the specimens oxidized at 750°C, but poor low fusing porcelain adherence. They suggested that the titanium or titanium alloy continue oxidizing during the firing process of the ceramic, which may lead the originally adherent oxide layer to become non-adherent. Titanium could obtain the necessary oxygen for oxidation from the reduction of the oxides in the ceramic via displacement reactions. However, this is not supported by the findings of Pang et al¹⁹ that reported the multiple firing schedules did not significantly affect the low fusing porcelain-grade II commercially pure titanium bond strength. On the other hand, it was believed that the bonding agent controls the formation of non adherent oxide during ceramic firing.^{18,24} The bonding agent contains a mixture of titanium and ceramic particles that may act as oxygen scavengers; therefore it inhibits progressive build-up of non adherent oxide layer with each firing cycle.¹⁸ If the brittleness of the titanium-oxide layer was taken into consideration, it is hard to understand how the bonding agents can be capable of improving the titanium-ceramic bond strength.

From the present study and other previous ones,^{16,19-22} it is seen that the commercially pure titanium-ceramic system was inferior in bond strength to the conventional metal-ceramic systems. However, the bond strength of PTi-Noritake system (33.12 MPa) exceeded the lower limit of 25 MPa specified in the ISO 9693 standard for the 3-point bending test.²⁸ Such findings have been also reported by previous studies.^{16,22,23}

The bond strength of PTi-Noritake system (33.12 MPa) was significantly higher than TiA-Noritake system (14.01 MPa) that was the only tested system below the ISO 9693 standard (25 MPa). Accordingly, Noritake porcelain was not

compatible with Ti-6Al-4V. It was clear that the surface preparation prior to porcelain application used in this study did not help enough to achieve the required TiA-Noritake porcelain bond. The amount of oxide coating produced on the commercially pure titanium surface was reported to be double than those formed on the titanium alloy (Ti-6Al-4V).^{13,25} Perhaps, TiA does not need bonding agent to control the formation of non adherent oxide during ceramic firing and to enhance the TiA-Noritake porcelain bond. The bonding agent can form an interlayer that cannot maintain its original properties during the firing and can be damaged due to the intermetallic reactions. A brittle interlayer may impair the TiA-Noritake porcelain mechanical compatibility. Photomicrographs of surface and cross section of TiA specimens showed voids of various sizes in the bonding agent layer (Figs. 3 and 6).

Mismatching of thermal expansion coefficients of Noritake ceramics and TiA should be considered as one of the other possible explanations for the low bonding strength.²⁹ Akagi et al²⁵ reported a higher coefficient of expansion with Ti-6Al-4V than with commercially pure titanium. This needs further investigation. Thermal expansion incompatibility might cause residual stresses, which develop during cooling from the glass transition temperature of ceramics to room temperature. In addition, cooling rate and number of firing cycles may have their own effects.

It seems that optimal metal-porcelain bond depends greatly on proper matching of metal-porcelain combinations.¹⁶ The authors believe that the titanium-ceramic bonding mechanisms are complicated and inadequately understood. Because of the usefulness of titanium for intraoral use, due to its superior biocompatibility and mechanical properties, further focus on the components and microstructure of the reaction zone is of great importance for optimizing the titanium-ceramic bonding.

CONCLUSIONS

Within the limitations of this study's design, the following conclusions were made:

1. The bond strength of the conventional metal-ceramic combination was significantly greater than the bond strengths of the cast commercially pure titanium- Ti-6Al-4V alloy-Noritake ceramic combinations.
2. Titanium-Noritake ceramic system showed significantly higher bond strengths than Ti-6Al-4V alloy-ceramic one.
3. Noritake porcelain is not recommended to be used with Ti-6Al-4V alloy.

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