Bond strength of porcelain to degassed cast titanium

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Purpose: This study examined the effect of degassing and sandblasting on the three-point flexure bond strength of low-fusing porcelain to cast titanium and compared the results with the strength of porcelain to a gold alloy.

Materials and Methods: Cast titanium plates (0.5 x 5.0 x 25.0 mm) were prepared and the surfaces were sandblasted with three different-sized alumina particles (50, 125, and 250 μm). After sandblasting, the cast plates were divided into two groups: one group to be degassed and the other that was not degassed. A low-fusing porcelain (1.0 x 5.0 x 8.0 mm) was then baked on the center of the cast titanium plate. Conventional gold alloy/porcelain specimens were also prepared as references. Degassing was performed according to heating schedules recommended by the manufacturer. Three-point bending tests were conducted using a universal testing machine at a crosshead speed of 1.5 mm/minute and a distance of 20 mm between the supporting rods. The mean value of the fracture force (N) and the deflections (mm) at fracture were measured.

Results: The degassed specimens withstood significantly ($p<0.05$) greater force to fracture and deflection compared to the non-degassed specimens for each corresponding alumina particle size. However, there were no statistical differences in force to fracture and in deflection among the specimens sandblasted with the 50, 125, and 250 μm alumina particles. There was no statistical difference in calculated bond strength between the degassed titanium and the gold alloy.

Conclusion: When a low-fusing porcelain system was used, the three-point flexure bond strength to degassed titanium was comparable to the strength of the porcelain to gold alloy.

(Int Chin J Dent 2002; 2: 67-74.)

Clinical Significance: When a low-fusing porcelain system was used, the reliable titanium-ceramic restorations could be obtained by degassing of titanium under vacuum condition.

Key words: bond strength, cast titanium, degassing, low fusion porcelain.

INTRODUCTION

Conventional porcelain-metal restorations have been extensively used in dentistry because of their esthetic appearance and good mechanical properties. However, mechanical failures often occur between

the metal and ceramic at the porcelain/metal interface. Clinically, such failures often begin as porcelain fractures. The success of porcelain-fused-to-metal alloy restorations acutely depends on the strong bonding between the porcelain and the metal substructure.[1,2]

Titanium has many advantages as a dental prosthetic material because of its good corrosion resistance and excellent biocompatibility.[3-5] Also, due to the passive oxide film that forms on its surface, the resin adheres to titanium as effectively as it adheres to other non-noble alloys.[6-8] However, titanium has a high melting temperature and increased chemical reactivity at high temperatures, making it difficult to cast and solder and to bond to porcelain.

Casting of titanium can be carried out in special casting machines under argon gas atmosphere that prevents the oxidation of titanium, which is generally believed to proceed by the inward lattice diffusion of oxygen with a new oxide forming at the oxide-metal interface. At 882°C, titanium undergoes a phase change from α-Ti to β-Ti. Low-fusing porcelains (under 800°C) must be used because, at temperatures above 800°C, an increasingly thick oxidation layer will be created with a rather weak bond to the underlying titanium.[9,10] The use of a low-fusing porcelain on cast titanium has increased in recent years.[9-17]

In metal/ceramic restorative techniques, the degassing process causes an oxide layer to form that acts as a bonding link between metal and porcelain. However, there is little information on the correlation between degassing and the bond strength of low-fusing porcelain to cast titanium in metal/ceramic restorations. Kimura et al.[9] investigated the effect of heat treatments on the tension-shear bond strength of low-fusing porcelain to wrought titanium. They reported that increasing the heat treatment temperature, especially over 900°C, increased the oxide layer, resulting in a decrease of bond strength to titanium; they concluded that the conventional degassing procedure was not suitable for porcelain-pure titanium restorations.

The purpose of this study was to examine the effects of degassing and sandblasting on the three-point flexure bond strength of low-fusing porcelain to cast titanium and to compare the results with the strength of porcelain to a gold alloy-ceramic system.

MATERIALS AND METHODS

The metals used in this study were commercially pure titanium (CP Ti ASTM Grade II, Selec Co., Osaka, Japan) and a high gold-containing alloy (Au: 77.3%; Pt: 9.8%; Pd: 8.9%; Ag: 1.2%; Cu: 0.3%; others: 2.5%, Degudent Universal, Degussa AG, Hanau, Germany) for metal-ceramics restorations. The porcelains employed were a low-fusing porcelain (Super Porcelain Titan, Noritake Co., Nagoya, Japan) for titanium and a conventional feldspathic porcelain (Vita Omega 900, Vita Zahnfabrik Co., Bad Säckingen, Germany) for gold alloy.

Preparation of cast metal plates

The CP Ti was cast in plastic plate patterns [0.5 (d_M) x 5.0 (W) x 25.0 (I_M) mm] (Fig. 1) invested in mold rings with a magnesia-based investment material (Selevest CB, Selec Co.). The invested molds were allowed to bench-set at room temperature for 60 minutes and were then placed in a burn-out furnace. The

burn-out schedule before casting followed the manufacturer's instructions. The CP Ti ingots were cast into the molds in a centrifugal casting machine (Ticast Super R, Selec Co.). After casting, the molds were bench-cooled to room temperature, and each cast plate was then retrieved from the investment. To investigate the effect of sandblasting, the cast titanium plates were sandblasted with 50 μm (Hi Aluminas, Shofu Inc., Kyoto, Japan), 125 μm (Powder WA 100, Panasonic Heraeus Dental, Osaka, Japan), and 250 μm (Powder W, Panasonic Heraeus Dental) alumina powders. Sandblasting was applied perpendicularly to the surface of the cast Ti surface from a distance of 20 mm at pressure of 0.4 MPa for 20 s. All the cast plates were examined using radiography with a dental unit (Coronis 90, Asahi Roentgen Inc., Ltd., Kyoto, Japan) to determine whether there was any noticeable internal porosity. The 70 kVp X-ray source (current: 10 mA, exposure: 0.8 s) was positioned perpendicular to the film at a distance of 50 cm from the specimen. If there was any porosity in the cast plate, that specimen was excluded. The suitable cast plates were cleaned ultrasonically in acetone for 10 minutes. A total of ten cast plates were prepared for each sandblasting condition. Half of the cast plates was subjected to the degassing process (+O) before the porcelains were fired. The other half of the plates for each condition was not degassed (-O). Cast gold alloy plates sandblasted with 50 μm alumina were also prepared as a reference.

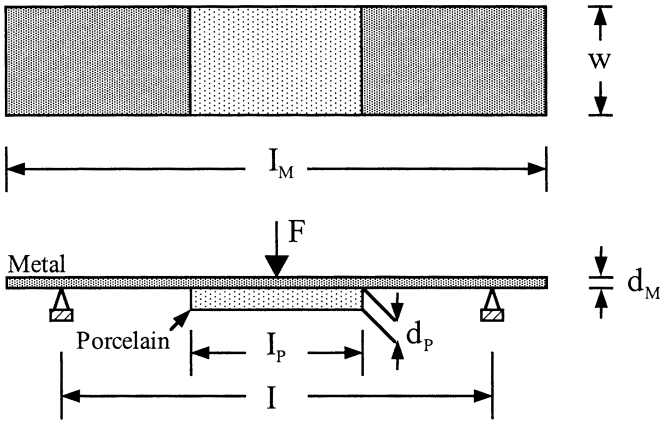

Fig. 1. Diagram of the three-point flexural test (Dimensions of the test configuration are: l_M = 25 mm, l_P = 8 mm, l = 20 mm, d_M =0.5 mm, d_P = 1 mm, w = 5 mm.).

Degassing of cast plates

The degassing schedules of each metal followed the manufacturer's instructions. For titanium degassing, the cast plates were heated from 500°C to 800°C at a heating rate of 50°C/minute and held in a furnace (Austromat 3001, Dekema, Honolulu, HI, USA) for 3 minutes under vacuum (9.8×10^4 Pa); the vacuum was then released into the air from 800°C to room temperature. The cast gold alloy plates were degassed from 550°C to 980°C at a heating rate of 50°C/minute under air atmosphere and held in the furnace for 1 minute, then cooled to room temperature.

Application of porcelain

Two layers (total thickness of approximately 0.2 mm) of opaque porcelain were applied to both metals on a 8.0 (I_P) x 5.0 (W) mm^2 area located at the central portion of each cast metal plate. A bonding opaque

porcelain was used for the first layer on the CP Ti. Two layers of body porcelain were then formed using a jig [8.0 (I_P) x 5.0 (W) x 1.0 (d_P) mm]. The second layer of body porcelain compensated for the firing shrinkage of the porcelain. After fusing the porcelains, the 8.0 (I_P) x 5.0 (W) surface of the body porcelain was then polished and adjusted to a final thickness of 1.0 (d_P) mm.

Three-point flexure testing

The metal/porcelain specimens were subjected to three-point bending testing using a universal testing machine (AGS-10kNG, Shimadzu, Kyoto, Japan) at a crosshead speed of 1.5 mm/minute and a distance of 20 mm between the supporting rods. The specimens were loaded at the center of the metal, and the force to fracture the specimens (Ff: N) was recorded. The deflections (Df: mm) at fracture were also measured. Five specimens (n = 5) were tested for each experimental condition, and the means and standard deviations were calculated. The stress for the three-point bending strength was calculated from the force to fracture the specimens (d_M = 0.5, sandblasted with 50 μm alumina) according to a numerical method using data generated in a previous study.[18] This method calculates the mean shear bond strength at the metal-porcelain interface as a function of the elastic modulus of the alloy, which was measured for each metal according to ISO standard 6871. Three-point bending strength is defined as the mean shear bond strength, J_b (MPa), at the moment of fracture:

$$J_b = F_{xy} \times F_f$$

where F_{xy} is the mean shear stress under unit load F = 1 N at the interface of the porcelain bonded to metal in the specimen geometry as described above. The value of F_{xy} was taken from a previous publication by Schwarz et al.,[18] and F_f is the force recorded at the failure of the porcelain veneer. Statistical analysis of the results of the three-point bending test was performed using an analysis of variance (ANOVA) with a Tukey-Kramer HSD test at a significance level of $\alpha = 0.05$.

RESULTS

Table 1. Fracture force (Ff: N) and deflection (Df: mm) at fracture of titanium specimens.

	SB (+O)			SB (-O)		
	50 μm	125 μm	250 μm	50 μm	125 μm	250 μm
Ff (N)	17.42 (0.60)	15.56 (1.80)	15.96 (1.00)	13.79 (1.10)	12.04 (1.20)	13.70 (0.70)
Df (mm)	0.17 (0.01)	0.15 (0.02)	0.15 (0.01)	0.14 (0.01)	0.11 (0.01)	0.13 (0.02)

One standard deviation in parentheses. +O: with degassing, -O: without degassing.

Table 1 presents the results of the force to fracture and the deflection at fracture of the specimens sandblasted with the different-sized alumina particles. These results are also depicted in Figs. 2 and 3, respectively.

The degassed specimens withstood greater force to fracture and deflection compared to the non-degassed specimens for each corresponding alumina particle size used for sandblasting. Statistical analysis indicated significantly ($p<0.05$) higher force was necessary to fracture the degassed specimens than the non-degassed ones. However, there were no statistical differences ($p>0.05$) in force to fracture and in deflection among the specimens sandblasted with the 50, 125, and 250 μm alumina particles.

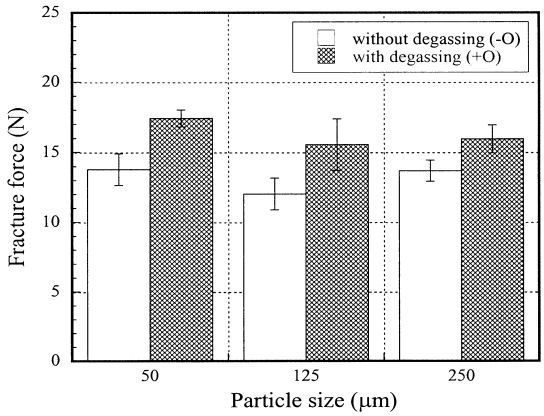

Fig. 2. Fracture force (N) at fracture of titanium specimens.

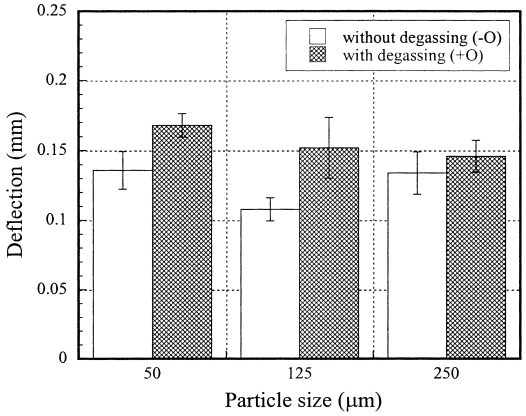

Fig. 3. Deflection (mm) at fracture of titanium specimens.

Table 2. Results of the modulus of elasticity (E_M), the mean shear stress (F_{xy}) under unit load F=1, fracture force (F_f) and calculated mean bond strength (J_b) of porcelain to Ti with degassing (+O) and without degassing (-O), and to degassed Au alloy.

	E_M (GPa)	F_{xy}	F_f (N)	J_b (MPa)
Ti (+O)	108	2.82	17.4 (0.6)	49.1 (1.7)
Ti (-O)	108	2.82	13.8 (1.1)	38.9 (3.1)
Au alloy	81.8	3.04	17.3 (0.7)	52.6 (2.1)

One standard deviation in parentheses.

The Ff and Df values of the gold alloy specimens sandblasted with 50 μm alumina were 17.30 (± 0.70) N and 0.23 (± 0.01) mm, respectively (numbers in parentheses indicate one standard deviation). The measured elastic moduli of the metals and calculated three-point flexure bond strengths are presented in Table 2. The calculated bond strength of porcelain to non-degassed titanium (38.9 MPa) was statistically ($p<0.05$) lower than that to the degassed titanium (49.1 MPa) and the gold alloy (52.6 MPa). There was no statistical difference in bond strength between the degassed titanium and the gold alloy.

DISCUSSION

The degassed titanium specimens in this study withstood significantly ($p<0.05$) greater fracture force and deflection compared to the specimens that were not degassed for each alumina particle size used for sandblasting. In their study of the effect of degassing on the bond strength of porcelain to wrought titanium, Kimura et al.[9] reported that increasing the degassing temperature, particularly over 900°C, decreased the tension-shear bond strength of low-fusing porcelain to titanium. In the present study, the force to fracture the titanium specimens degassed at 800°C was greater compared to the non-degassed specimens. Since the degassing procedures were conducted under vacuum in both studies, the differences in results may be due to the different types of low-fusing porcelain, different forms of titanium (wrought or cast), or the use of different surface treatments (polished or sandblasted). Cast titanium forms an oxidized layer from the reaction with the investment and the ambient elements on the cast surface. The manufacturer of the porcelain used in the present study recommends degassing and the use of bonding porcelain before applying the opaque porcelain. In general, oxidation (degassing) of a metal surface, such as a gold alloy surface, increases the bond strength between metal and porcelain; however, excessive oxidization on the metals, especially on titanium,[14] decreases the bond strength. However, it seems that the force to fracture sandblasted cast titanium degassed at 800°C under vacuum atmosphere increased using the low-fusing porcelain system employed in this study.

Another possible reason for the higher force to fracture the degassed specimens is the change that occurs in the mechanical properties (yield strength, elongation and modulus elasticity) of the cast titanium plates. Degassing causes the surface oxidation of the cast titanium plate and makes the cast titanium surface more brittle and hard, resulting in higher bending resistance compared with a non-degassed titanium plate. The high bending resistance increases the force to deflect at the point of porcelain fracture.

There were no statistical differences ($p>0.05$) in the effect of alumina particle size on the force to fracture and deflection for the specimens. These results indicate that roughening the bonding surface by sandblasting does not affect the force to fracture results of the three-point bending test. Roughening the metal surface might have a greater effect on the tension-shear or shear bond strength than on the three-point bending strength.[19]

The calculated three-point bend strength of porcelain to non-degassed titanium in this study was statistically ($p<0.05$) lower than that to the degassed titanium and the gold alloy. However, there was no statistical difference in bond strength between the degassed titanium and the gold alloy. Togaya et al.[17] investigated the properties of a metal-porcelain system and compared the bond strength (pull test) of porcelain to cast titanium that was not degassed. They reported that the pull-out bond strength of the cast titanium-porcelain system was comparable to the strength of a gold alloy/porcelain system and a Ni-Cr/porcelain system and concluded that the lower firing temperature was desirable for titanium-ceramic processing. Pröbster et al.[14] investigated the three-point bending strength of three low-fusing porcelain systems fired to cast titanium surfaces without the reaction layer, and compared it with results found for a Ni-Cr/porcelain system. They reported that the three porcelain systems had a significantly lower bond

strength compared to the Ni-Cr/porcelain system. The present study and previous studies have produced mixed results; it seems that the bond strength values have varied based on the types of procelains and metals used. Other possible factors affecting bond strength are test methods, the porcelain furnace used, and porcelain firing schedules, including degassing before porcelain fusing.

CONCLUSIONS

The following conclusions can be drawn from this study:

1. When a low-fusing porcelain system (Titan) was used, the three-point flexure bond strength to degassed titanium was comparable to the strength to gold alloy (Degudent Universal)-porcelain (Vita Omega 900) system.
2. No significant differences in force to fracture and in deflection among the specimens sandblasted with the 50, 125, and 250 μm alumina particles indicated that roughening the bonding surface by sandblasting does not affect the force to fracture in the three-point bending test.
3. Based on the results obtained in this study and in previous reports, the bond strength of a titanium-porcelain system depends on factors such as the porcelain used and the porcelain firing schedules (including degassing).

ACKNOWLEDGMENT

This study was supported in part by grant Y2001-Z from the Baylor College of Dentistry Faculty Intramural Grant Program. The project in Nagasaki University of the primary author Liu was supported in part by a grant from the Japan-China Medical Association (2000-2-5-19). Editorial assistance by Mrs. Jeanne Santa Cruz is also appreciated.

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Received on April 24, 2002. Revised on May 29, 2002. Accepted on May 29, 2002.