BOND COMPATIBILITY OF LOW-FUSING PORCELAIN TO RECAST TITANIUM

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ABSTRACT

The purpose of this study was to investigate the bond compatibility of low-fusing porcelain to recast commercially pure titanium. A 3-point bending test was used to evaluate bonding strength. Three groups of specimens were prepared according to the percentage of new and recast commercially pure titanium. Group A-100% as-received metal, Group B-1:1 ratio of new to once recast metal, and Group C-100% once recast metal. Titanium casting unit was used to cast 24 specimens, eight for each group, with dimensions of 25.0 x 3.0 x 0.5 mm. Low fusing porcelain (Noritake) was fired onto the surface of the titanium specimen. A universal testing machine was used to perform the 3-point bending test. There was significant difference in the load at bond failure of titanium-ceramic system between group A (33.35±5.16 MPa) and group B (24.35±5.14 MPa) [P=0.004]. Similar results were found between group A and group C (23.63±4.17 MPa) [P=0.002]. No significant difference was found between group B and group C (P=0.954). It can be concluded that the bond strength of the as-received commercially pure titanium-Noritake ceramic combinations was significantly greater than that of the recast commercially pure titanium-Noritake ceramic combinations. Previously cast commercially pure titanium should not be used again to fabricate metal-ceramic restoration.

Keywords: Bond strength, dental ceramics, commercially pure titanium, recasting.

INTRODUCTION

The significant increase in the price of gold in 1970s led to a substantial rise in the cost of the gold dental alloys; and prompted the development of less expensive alternative alloys. However, these substitutes, particularly base metal alloys, have been less than ideal from several aspects such as the increased hardness which makes the clinical and laboratory manipulation difficult,1 and the potential biological hazards.2,3 The use of titanium in dentistry has dramatically increased in the past few years because of its superior biocompatibility, corrosion resistance, desirable physical and mechanical properties, and low cost.4-6 Today, titanium is used for removable and fixed partial dentures, both tooth supported and implant supported.7,9 However, titanium reacts strongly with gaseous elements such as oxygen at high temperatures, and yields an excessively thick layer of TiO2.10,11 Such an oxide layer is considered detrimental to a good titanium-
porcelain bonding. Therefore, it is essential that porcelain firing should take place below 800°C in order to prevent excessive oxide formation.10-11 Adachi et al10 reported that the commercially pure titanium (CPTi) 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 2-3 times thicker with significantly lower adherence.

Some manufacturers have introduced low-fusing porcelains designed for bonding to titanium with low fusing temperatures (<850°C) and favorable thermal expansion coefficients.12-14 Several studies have shown that the quality of titanium-low fusing porcelain bonding was acceptable.12-17 Moreover, some of the manufacturers provide a special bonding agent to control the oxide layer thickness and to enhance the titanium-ceramic bonding.12,15,16 In addition, evidence suggests that the roughened surface created by sandblasting could also improve the titanium-ceramic bond.15,18

In many dental laboratories, previously used gold alloy may be combined with a portion of new alloy, as-received from the manufacturer. Textbook guidelines for recasting gold alloy vary from adding no new metal to adding up to 50% new metal to the buttons or sprues removed from the previously made castings.19-21 There have been several investigations of the effect of recasting on the physical and clinical properties of the type III gold,22-23 high-palladium,24-25 Co-Cr,26-27 and NiCr26-29 alloys.

A greater concern exists with re-melting of metal-ceramic alloys used for metal-ceramic restorations compared with those used for all-metal restorations because of the potential loss of trace base metals (e.g., Fe, In, Sn, and Zn) which play important role in metal-ceramic bonding.20 Rasmussen and Doukoudakis24 investigated the adhesion of dental porcelain to recast gold-palladium alloy; and found that a coping of 1:1 ratio of new to reused metal for a porcelain-metal restoration was an adequate safety margin. However, increased frequency and size of interfacial voids were observed in 85% or more recast metal. Papazoglou et al25 also reported that recasting without adding new alloy negatively affected some high-palladium alloys.

There appear to be no published studies on the effects of titanium recasting on the porcelain bonding to titanium. The purpose of this study was to investigate the bond compatibility of low fusing porcelain to recast CPTi.

**MATERIALS AND METHODS**

A 3-point bending test was performed to test the bonding strength of low fusing porcelain to recast CPTi in accordance with International Standard Organization (ISO) specification 9693 (Fig. 1).31 The CPTi (Tritan, Dentaurum Inc, Pforzheim, Germany) was >99.5% Ti, 0.03% N, 0.1% C, 0.015% H, 0.30% Fe, and 0.25% O as provided by the manufacturer.

Three casting protocols were selected in relation to the proportion of as-received and recast CPTi: Group A 100% as-received metal, Group B 50% wt new metal, 50% wt once recast metal, and Group C 100% once recast metal. Group C (recast) ingots were obtained by casting a new 31 gm ingot into a copper ring that acted as a mold. Two ingots of as-received and 2 ingots of once used were chosen to be sectioned into equal halves. Each of group B ingots was formed from one-half ingot of as-received and another half from once used.

Twenty four specimens were prepared, 8 for each group. In order to obtain precise dimensions of the metal strips, wax patterns were fabricated with the help of an aluminum mold with dimensions of 28.0 x 3.2 x 0.8 mm. The patterns were sprued and invested with silica-free and phosphate-free, alumina and magnesia-based investment (Rematitan Ultra, Dentaurum) and cast using a casting unit (Castmatic, Dentaurum). Manufacturer’s instructions were followed for the investment and casting procedures.

The resultant metal strips were sequentially wet ground with 500, 600, and 1000 grit silicon carbide paper 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 ISO 9693 (Fig. 1). The thickness of the specimens was controlled with a micrometer (Mitutoyo; Mitutoyo Corp, Tokyo, Japan) to the nearest 0.01 of a millimeter. The specimens were placed on a slab surface and visually observed to determine whether they were planar or curved. The bonding surface of the specimens was then sandblasted (Sandstorm, Vaniman Inc, Fallbrook, Calif, USA) using 125-250pm aluminium oxide and 2-3 bars air pressure and cleaned with distilled water in an ultrasonic bath for 10 minutes.
A thin layer of the porcelain bonding agent (Noritake, Nagoya, Japan) was painted in the central portion of the alloy sample (8 x 3 mm) with a short bristle brush. The specimens were heated in the furnace according to the manufacturer’s instructions. The low fusing porcelain (Noritake) was built up to dimensions 8 x 3 x 1 mm according to ISO 9693 (Fig. 1). Two uniform coats of opaque porcelain each approximately 0.2 mm thick were applied with a brush on an area 8 x 3 mm located in the central portion of each metal strip. The body porcelain was subsequently formed with the use of an aluminum thickness index and a vibrator. The firing shrinkage was compensated for with a second porcelain application and yielded a final thickness of 1 mm. Firing procedures for the porcelain were accomplished according to the manufacturer’s instructions.

The bond strength testing was performed with a 3-point bending test on a servo-hydraulic universal testing machine (Instron 8500R, High Wycombe, Bucks, UK). The titanium-ceramic specimens were positioned on a support (20 mm span distance) with the porcelain facing down. A compressive load was applied at the midline of the metal strip by a rounded loading rod using a crosshead speed of 0.5 mm/min. The load was applied until a sudden disruption of the load-deflection curve occurred 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 \( a \) was calculated with the following equation given in ISO 9693:31

\[
a = kF \text{ (N/mm}^2\text{)}
\]

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 with units of \( \text{mm}^{-2} \). The value of \( k \) depends on the thickness of the metal substrate and the elastic modulus of the metallic material and for the CPTi tested it was determined to be 4.75 mm\(^{-2}\).

Visual observations of the failed surfaces of the metal-ceramic interface were made to determine whether cohesive or adhesive failure of the titanium-ceramic bond occurred. The data were analyzed by statistical software (SPSS for Windows, Release 10.0). Descriptive statistic was used to summarize the data. The one-way ANOVA test was performed to determine the mean differences of bond strength among the groups and the Tukey’s multiple range test were used to find out the statistical significance of the mean differences between groups (\( a=.05 \)).

RESULTS

The mean titanium-ceramic bond strengths and standard deviations are presented in Table 1. The

![Fig 1. Diagram of specimen configuration and 3-points flexure bond test (all dimensions are in millimeters). A: Side view; B: Top view; F: Force.](image-url)
TABLE 1. TITANIUM-CERAMIC BOND STRENGTH (MPa)

<table>
<thead>
<tr>
<th>Groups</th>
<th>N</th>
<th>Mean</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>8</td>
<td>33.35</td>
<td>5.16</td>
</tr>
<tr>
<td>B</td>
<td>8</td>
<td>24.35a</td>
<td>5.14</td>
</tr>
<tr>
<td>C</td>
<td>8</td>
<td>23.63a</td>
<td>4.17</td>
</tr>
</tbody>
</table>

Mean values with similar superscript letters were not significantly different (p=.954).

The mean bond strength of 50% recast (24.35 MPa) and 100% recast (23.63 MPa) groups were below the lower limit value of 25.00 MPa in the ISO 9693 standard. For the as-received group all the specimens exceed the minimum debonding strength, whereas for the 50% recast group 3 specimens and for the 100% recast group only 2 specimens exceed the 25.00 MPa limit.

The one-way ANOVA test showed significant differences (P=0.001) among the titanium-ceramic bond strengths (Table 2). The results of Tukey’s multiple range test showed significant difference between the loads at bond failure of titanium-ceramic system between the as-received and the 50% recast groups (P=0.004). Similar result were found between the as-received and the 100% recast groups (P=0.002). However, no significant difference were found between the 50% recast and the 100% recast groups (P=0.954).

Visual analysis of the fracture sites surfaces displayed residual porcelain retained on the metal surface for as-received group. This observation indicates a combination of cohesive and adhesive bond failures. However, no visible porcelain on the titanium's surface was found for the 50% recast and the 100% recast groups, suggesting an adhesive bond failure. Also, visually the specimens' surfaces showed numerous interfacial porosity for the 50% recast and the 100% recast groups.

DISCUSSION

The results of the present study showed that the mean debonding/crack-initiation strength for the as-received CPTi (33.35 MPa) was significantly higher than the mean for 50% recast (24.35 MPa) and 100% recast (23.63 MPa) alloy. According to the ISO 9693 six specimens should be prepared, and the metal-ceramic system passes the test if four or more specimens exceed the minimum debonding/crack-initiation strength set at 25 MPa. For the as-received group all the 8 specimens exceeded the minimum debonding strength, whereas for 50% recast group, 3 out of 8 specimens and for 50% recast group, only 2 out of 8 specimens exceeded the 25.00 MPa limit.

This study indicates that recasting used CPTi may lead to an unsatisfactory titanium-ceramic bond. This may be explained by the potential oxidation of titanium during casting procedure which may affect its behavior after remelting. Taira et al reported that molten and heated CPTi reduced the oxide mold, and freed oxygen diffused from the surface into the interior of titanium castings, which increased the microhardness proportional to the amount of absorbed oxygen. The physical and mechanical properties of CPTi can be greatly influenced by the addition of small traces of other elements such as oxygen, iron, and nitrogen. In addition, oxygen is an alpha stabilizer by forming interstitial solid solutions of titanium.

The casting of used CPTi may lead to a thick layer of TiO₂ formation at the surface of the specimens. Such an oxide layer is considered detrimental to titanium-porcelain bonding as it can easily break. The visual observed adhesive bond failure for recast groups can be explained by the presence of the thick layer of TiO₂.

It was believed that the bonding agent controls the formation of non adherent oxide during ceramic fir-
The bonding agent contains a mixture of titanium and ceramic particles that may act as oxygen scavengers; therefore it inhibits progressive build-up of nonadherent oxide layer with each firing cycle. It was clear that the surface preparation that include sandblasting and bonding agent application prior to porcelain application did not help enough to achieve the required recast CPTi-Noritake porcelain bond. It has been shown that the weak area of the titanium-ceramic bond was the excessive and non-adherent oxide layer at the interface. Adachi ET al suggested that the titanium continue oxidizing during the firing process of the ceramic, which may lead the originally adherent oxide layer to become non-adherent. It was reported that the oxide formed on the titanium surface at ceramic firing temperatures is porous, nonadherent and unsuitable for ceramic bonding.

It was visually observed that specimens of recast CPTi groups showed numerous interfacial porosity. This has also been reported for 85% and more recast gold-palladium alloy. An interfacial void could seriously weaken a metal-ceramic bond as the ceramic failures may initiated at the voids which are considered to be the areas of stress concentration. Rasmussen and Doukoudakis found that a coping of 1:1 ratio of new to reused metal for a ceramic-metal restoration was an adequate safety margin for the gold-palladium alloy and the serious changes were found only for 85% and more recast metal. In the present study the CPTi acted differently when recast. This can be related to the pronounced oxidative nature of titanium at high temperatures, which may yields an excessively thick layer of TiO₂.

The results of the present study clearly indicate that the previously cast CPTi should not be used again to fabricate metal-ceramic restoration. However, one of the limitations of this study was that only one brand of low-fusing porcelain was tested; the findings of the tested product may not be extrapolated to similar materials. Also, if measuring the oxide layer thickness was part of the study, helpful observations might have been obtained to better understand the behavior of the materials. In addition, further investigations should include the compositional stability and the mechanical properties for recast CPTi.

**CONCLUSIONS**

Within the limitations of this study, the following conclusions were drawn:

1. The bond strength of the as-received CPTi-Noritake ceramic combination was significantly greater than that of the recast CPTi-Noritake ceramic combination.

2. Previously cast CPTi should not be used again to fabricate metal-ceramic restoration.

**REFERENCES**


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