Original Article

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Shear bond strength of dental ceramics to cast commercially pure titanium

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Abstract

Aim: The objective of this study was to evaluate the bond strength of four dental ceramics to commercially pure titanium. **Methods**: To measure the resistance of metal-ceramic bonding, ceramic rings (Noritake Ti22®, Triceran®, IPS®, Noritake EX-3®) were made around metal rods fused to commercially pure titanium. The area of metal-ceramic union was measured and, after mounting in type III plaster, the rings were subjected to a shearing force in a universal testing machine at a crosshead speed of 2 mm/s until failure occurred. The metal-ceramic shear bond resistance was calculated in MPa. **Results**: The shear bond strength means for the ceramics Triceram and Noritake Ti22 (42.50 MPa and 61 MPa, respectively) were higher than the minimum value required by the DIN 13927 standard (25 MPa). The ceramics IPS and Noritake EX3, although not specifically formulated for titanium, also had shear bond strength means above the ISO-recommended value (38.47 MPa and 29.04 MPa, respectively); however, there cracks in some specimens after burning and detachment of the ceramic from the metal**. Conclusions:** The ceramic Noritake Ti22 should be indicated for the commercially pure titanium casting due to its higher mean bond resistance compared to other ceramics utilized.

Keywords: shear bond strength, titanium ceramic system, low firing ceramic.

Introduction

The use of metal-ceramic restorations began in the late 1950's, allowing the development of prosthetic rehabilitation with better cosmetic results. However, the actual mechanism of adhesion of ceramic to metal is complex and not fully understood mainly due to differences in thermal expansion and formation of oxide layer on the surface of dental metal alloys. These factors make the chemical union complicated, acting as an adhesion blocker associated with adherence reduction[1-4].

Several metal alloys have been introduced to the fabrication of fixed partial dentures covered with ceramics. Recent developments in casting techniques have enabled the construction of dentures using titanium, as they present excellent biocompatibility, good corrosion resistance and acceptable physical properties, and this have increased the application of titanium and its alloys in dentistry[5-6]. However, the bonding of ceramic to titanium is still a problem in the current use of metal-ceramic restorations[6]. One of the characteristics of titanium is that in the presence of oxygen an oxide layer is formed and adheres to titanium surface. While this oxide layer confers corrosion resistance, it decreases considerably the

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bond strength at the metal ceramic interface. This layer is sometimes formed by oxides from the investment that react with titanium surface at high temperatures and interferes with the metal-ceramic[7]. Thus, many titanium surface treatments have been proposed to minimize the problem, such as blasting or metal surface treatment with acid, among others[2-8].

There are still some unfavorable characteristics of titanium, such as the presence of porosities, problems with soldering and bonding to the dental ceramics[9]. Some studies have shown that the adhesiveness of titanium to ceramics is comparable to the adhesion of ceramic to Ni-Cr alloys[10], so the ratio of adhesiveness of titanium to ceramic materials needs further study to answer the question of high reactivity of titanium with certain elements[11].

Considering all these aspects, the objective of this study was to test different metal-ceramic restorations, analyzing the bond strength of four dental ceramics to commercially pure titanium (CP Ti).

Material and methods

The four commercial ceramics used in study are listed in Table 1.

Table 1. Description of ceramic materials used in study.

Group	Ceramic	Manufacturer
A	Noritake Ti22®	Noritake Super Porcelain, Noritake, Nagoya, Japan
B	Triceran®	Triceram; Esprident GmbH, Ispringen, Germany
C	IPS®	Ivoclar-Vivadent, Schaan, Liechtenstein
D	Noritake EX-3®	Noritake SuperPorcelain EX -3; Noritake, Nagoya, Japan

Forty specimens of CP Ti (Tritan, grade 1; Dentaurum, Pforzheim, Germany) were prepared. For obtaining the titanium rods, brass rods (3.0 mm in diameter and 75.0 mm in length) were included in titanium coating (Rematitan Plus, Dentaurum Ispringen JP KG, Pforzheim, Germany). After setting of the coating, the rods were removed from the refractory mold and subjected to thermal cycling in the oven (EDG-7000 3P - EDG Equipment and Controls Ltd, Brazil), following the manufacturer's instruction.

Titanium casting was performed on the Rematitan machine (Dentaurum, Pforzheim, Germany) by means of voltaic arc and injection through positive pressure of argon gas in the upper portion of the cylinder, and negative through the vacuum in the lower cylinder. After casting, the CP Ti rods were divested and cleaned with carbide burs (702L; KG Sorensen Ind. Com. Ltda., Barueri, SP, Brazil) and airborne Al_2O_3 abrasion, which is a standard procedure recommended by composite manufacturers, was performed with particles of 110 µm in size for 20 s, under pressure of 30 to 40 psi and a distance of 3 to 5 cm[12-13].

After this treatment, the specimens were taken to the oven to 400° C to 750° C, where they remained for 10 min at 750° C without vacuum for pre-oxidation of the metal. During all these procedures the specimens were touched only by the opposite end where they would receive the ceramic ring, thus avoiding contamination of the site of ceramic application.

The area to receive the ceramic was drawn with the help of two silicone cursors (Optosil, Heraeus Kulzer South America Ltda, Brasil) and an acrylic spacer 2.5 mm thick (Plexiglass; Swedlow Inc., Gardengrove, CA, USA) to obtain a default in making the porcelain specimen, about 6.0 mm in diameter x 2.0 mm thick. In all specimens, ceramic application was performed by the same investigator. After the application of ceramics, the excess was removed with the aid of abrasive silicone polishers.

The specimens were numbered and measured with a digital caliper as follows: two perpendicular measurements from the rod diameter immediately above and below the ring, and four equally spaced measurements from the thickness of the ceramic ring. The mean of the measurements represents, respectively, the rod diameter and thickness of the ceramic ring. These mean values were used to calculate the area of the metal-ceramic bonding using the following equation (Eq. 1): $S = \pi.ø.e$, where = bonding area; ø = diameter of the rod; e = thickness of the ceramic ring[12].

To determine the resistance of the metal-ceramic bonding, the ceramic rings were embedded in stone cylinders (Vigodent SA, Ind. Com., Brazil), previously isolated with petroleum jelly and placed on a vibrator for plaster. The opposite end of the titanium rod was then attached to the liner and maintained centrally through the support resin with depression in the central of the PVC ring. After setting of the plaster and removing the PVC rings, the specimens were kept at room temperature for seven days so the plaster could dry, before the metal-ceramic bond resistance test.

The specimens were subjected to a shearing force in a universal testing machine (EMIC MEM 2000, EMIC Equipment and Systems Testing Ltd, Brazil) at displacement speed of 2.0 mm/min and load cell 200 kgf. After failure, the peak load recorded was used to calculate the rupture tension as an indicator of bond strength of the metal-ceramic union using the following equation (Eq.2): $T = F / S \times 9.8\ MPa$, where T = tension break; F = critical rupture load, S = area of the metal-ceramic union[13].

All data were analyzed by ANOVA and Tukey test to determine the adhesion of ceramics to CP Ti. Statistical analysis was performed with SPSS software for Windows, version 12.0 (SPSS Inc., Chicago, IL, USA). In each group, the interface between the ceramic and titanium were examined by scanning electron microscopy (SEM).

Results

The shear bond strength means of the titanium-ceramic bonding systems of each group are presented in Table 2. There was a statistically significant difference among the groups ($p < 0.05$) (Table 2 and 3). Group A presented the highest shear bond strength value (61.46 MPa) and groups B and C showed higher bond strength (42.50 and 38.47 MPa) than Group D (29.04 MPa).

SEM micrographs (Figure 1) of the surface area of the tested specimens showed unique characteristics of the reaction zone of each bonding system. The SEM micrographs of the

Table 2. Comparisons of the mean of metal-ceramic bond strength values (ANOVA).

	Sum of squares	*df*	Mean square	*F*	*p*-Value
Between groups	8342.3926	3	2780.7976	23.53	0.000*
Within groups	6617.2090	56	118.1644		
Total	14959.6016	59			

Table 3 - Metal-ceramic mean shear bond strength values (MPa) for the four experimental groups.

Groups	Mean (SD)
A	61.46 (± 13.50) a
B	42.50 (± 12.27) b
C	38.47 (± 9.00) bc
D	29.04 (± 7.67) c

Different letters indicate statistically significant difference at 5% (ANOVA and Tukey's test).

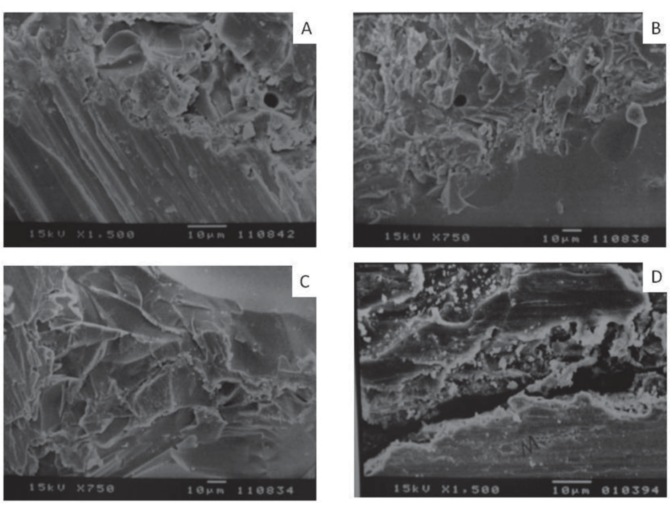

Fig. 1. SEM images of commercially pure titanium specimens with different ceramic materials used in study: Noritake Ti22® (A), Triceran® (B), IPS® (C) and Noritake EX-3® (D).

cross section of the tested titanium ceramic specimens demonstrated that the interface had porosities. Large gaps were present in the titanium-ceramic bonding interfaces of Group D, suggesting the poorest bond in this region.

The SEM micrographs of group B suggest that minimal differences existed at the metal ceramic interface compared to the other groups. It was observed that the bonding agent bonded to both the ceramic and titanium had no gap, but occupied a width of approximately 30 to 40 μm at the interface. This could be considered an excessive width for an optimal metal ceramic bond (Figure 1).

The titanium-ceramic specimens of Group A showed similar bonding characteristics as observed. The interface produced with the bonding ceramic in the group A was more definite and regular than for the other titanium ceramic groups. However, there was less interfacial porosity for the ceramic-titanium systems of groups B and C (Figure 1).

Discussion

The use of titanium for dental crown and bridge applications has increased the clinical importance of assessing their compatibility with ceramic systems. This study evaluated the bonding characteristics of four commercially available ceramic designed for use with titanium, by the shear bond strength test and SEM analysis. Shear bond strength refers to the force required for separating two parts, and it consists of two factors: mechanical bonding and chemical adhesion. Mechanical bonding is an anchoring effect that is related to the surface roughness of the alloy surface, and chemical bonding compatibility implies formation of a strong bond during porcelain firing. Therefore, both mechanical and chemical factors are essential to create stable bonding.

The results of those tests showed that the shear bond strength of titanium ceramic in group A was higher than those others titanium ceramic systems used in this study. The excellence of metal-ceramic adherence was exhibited by the presence of a dentin ceramic layer on specimen surface after the shear bond strength test. As a result, higher shear bond strength was detected on the ceramic titanium in all specimens because the titanium ceramic groups exceeded the lower limit of 25 MPa established in the DIN 13927 standard[14].

The ceramic-titanium systems of Group B showed higher results than the minimum established by the international standard (42.50 MPa), which may be due to low temperature burn, reducing the risk of overgrowth of metallic oxides which would result in a gain of adhesion, since one of the greatest problems of adhesion of ceramic to metal is to control the formation of the oxide layer[7].

The SEM analysis showed that the titanium-ceramic systems of groups C and D had the poorest metal-ceramic bonds (Figs. 1). The titanium ceramic-system of Group A showed the best bonding and interfacial characteristics (Fig. 1) because no definite oxide layer was observed at the interface between ceramic and titanium. However, it has been reported[15] that failures in the titanium-ceramic system occurred at the oxide metal interface, suggesting poor oxide metal adherence. Adachi et al.[16] reported complete delimitation of the ceramic from the titanium surfaces, after testing in a constant strain flexure apparatus, with the amount of the remaining ceramic being less than 1%, as occurred at the titanium ceramic interface in the other groups.

Moreover, lower shear bond strength between titanium and different low fusing ceramic, compared to other alloys and conventional ceramic, has been reported previously[5]. Pröbster et al.[17] found that the bond strengths of the titanium ceramic specimens ranged from 38-58% of the strength of the Ni-Cr ceramic control specimens. However, several studies[10,18-19] have reported bond strength values between traditional prosthetic alloys and ceramic to be twice or even three times higher than the corresponding values of cast titanium with low fusing porcelains. Only the results obtained in this study have proven to be much higher than the standard requires (42.50 MPa)[14].

It is likely that factors others than the efficient protection of the castings from contamination from phosphate-bonded investment materials may interfere with the metal ceramic bonding to titanium. One of them could be the high dissolution of oxygen within titanium and consequently its diffusion from the surface into the bulk material, as temperature increases. This diffusion may reduce the number of oxides, creating the conditions for a stronger metal ceramic bond, as previously reported[20]. It is claimed that at the firing temperature of the low fusing titanium ceramic (720º to 750º C) a dissociation of the superficial native titanium oxides takes place, followed by dissolution of elements within the titanium mass, accompanied by diffusion of the ceramic material elements, increasing the shear bond strength between titanium and ceramic in all groups.

Many researchers[1,10,20-22] have reported mean values of shear bond strength different to those of the present study. More specifically, shear bond strength mean values of 27.79 MPa and 32.2 MPa has been given for Noritake Ti22 ceramic, when a magnesia investment was used[1,21]. In other studies[10-22] shear bond strength mean values different from those of the present study have been documented, even though different ceramics were used.

When analyzing these results, it is important to examine the effects of these variables on both experimental groups separately because the ceramic properties and the fusion temperature are very different. However, from a clinical perspective, it was desirable to "rank" or arrange the effects of the interface variables on titanium ceramic adherence on the basis of the shear bond strength values regardless of the type of ceramic used.

It is also important to realize that even though this is an in vitro study, the clinical implications of the results may be important. The authors are aware of the differences that exist between the ceramic on which this experiment was conducted and the oral environment.

In conclusion, this study demonstrated that the type of ceramic affects the shear bond strength to commercially pure titanium. Noritake Ti22 should be indicated for commercially pure titanium casting due to its higher mean bond resistance compared to other ceramics used.

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