# Shear bond strength between composite resins to cast titanium and gold alloy

# Resistência ao cisalhamento entre resinas compostas e o titânio fundido e uma liga áurica.

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## ABSTRACT

*New indirect composites with improved qualities have been introduced as alternative to porcelain. There is little information regarding the bond strength of the new metal-resin bonding systems, mainly when they are applied onto titanium surfaces. This study evaluated the shear bond strength of two indirect composite resin (Artglass/Heraeus Kulzer and Targis/Ivoclar) to cast titanium (Ti) and gold alloy (Au). Twenty metallic structures (4mm in diameter, 4,0mm thick) of each alloy were cast shaped and abraded with 250*mm *aluminum oxide before the application of the bond system for each polymer. Artglass opaque, dentin and enamel composite were applied using teflon matrices onto titanium and gold alloy structures. The same procedure was achieved to Targis and they were polymerized according to the manufactures' recommendations. The samples were stored in distilled water for 24 hours at 37°C and thermocycled (5° and 55°C/3000 cycles). Shear bond strength tests were performed by using an Instron Universal testing machine at a crosshead speed of 5mm/min. Data were analyzed statistically with 2-way ANOVA and Tukey test (*a*=0,5). The results indicated that the gold alloy was statistically better than the cast titanium (18,44MPa and 9,81MPa respectively) and Targis ceromer (16,61 MPa) showed significant higher shear bond strength than Artglass polyglass (11,64MPa) in all tested alloys. The best result was achieved when the ceromer was applied onto gold alloy.*

## UNITERMS

*Titanium; shear; bond; resin*

## INTRODUCTION

In recent years titanium has become a material of great interest in prosthetic dentistry. Since the 1950's, titanium was referred as 'the wonder metal' because of its applications for the aerospace industry. Titanium is quite light in weight and its density (4,5g/cm$^3$) is considerably smaller than that of gold, Co-Cr, or 316 stainless steel (19,3; 8,5; or 7,9g/cm$^3$, respectively)[12]. Titanium has excellent biocompatibility, corrosion resistance, mechanical properties that are nearly similar to those of dental gold alloys [1,4,9,14]. It has a low price as a consequence of it is abundance on the Earth's crust, being some million times more plentiful than gold. Moreover one must remember that titanium is a poor conductor of heat[13] and with proper technique it can readily be machined or polished to very fine finishes. These advantages have increased considerably the interest in using titanium in dentistry during the last decade.

Recent improvements in casting technology have made it possible to accurately fabricate prostheses made from commercially pure titanium, thus expanding the use of this metal. Because of its high melting point (1670±50°C), its strong affinity with gases such as oxygen, hydrogen, nitrogen, carbon, and also its reactivity with investment materials[13], this procedure demands special machines and gas protection to avoid oxidation of the metal which can damage the bond strength of aesthetic materials on cast titanium.

Resin has been used as a veneering material since the early 1940s, but clinicians found many problems with the early generations of resins, such as fluid leakage at the metal-resin interface, discoloration, poor long-term aesthetics, and poor wear resistance. Improvements in the composition and polymerization of composite resins have resulted in the "second-generation" laboratory composite resins[17]. Depending on each manufacturer these materials can be entitled as ceromer (ceramic optimized polymers), glass polymer or indirect composite resin and they support that these biomaterials join the best of porcelain and composite. These advantages include greater elasticity, high fracture toughness, aesthetics, color stability, easiness of intra-oral repairs and adjustments, a high degree of mastication comfort, abrasion similar to natural tooth structure, and compatibility with most dental casting alloys[3]. The latest improvement in these materials was the creation of chemical bonding of resin to the metal surfaces with the pretreatment of the alloy surface and the application of coupling agents. The chemical adherence of the opaque layer on the metal substructure reduced the creation of marginal gaps, caused by the polymerization shrinkage of the resin and the appreciable differences in coefficient of thermal expansion of the two materials[11].

During the last decade, a great breakthrough was the development of new metal-resin bonding techniques that has resulted in a chemical bonding between resin and metal. For clinical success, the veneering material should be strongly bonded without interfacial leakage or delaminating.

The purpose of this study was to evaluate the shear bond strength of veneering composites to cast pure titanium and gold alloy.

## Material and Methods

Specimens for the shear bond strength test were prepared from commercially pure titanium (99,5% Grade I) and Type IV gold alloy (Degulor-M, Degussa, Germany). Cylindrical-shaped plastic patterns, 5mm long and 4mm in diameter (Fig.1) were milled and twenty of them were placed in a phosphate-bonded investment (Cristobalite, Kerr). After setting a one-hour period, they were inserted for burnout at 870ºC in an oven and cast in gold alloy with a centrifugal casting machine. The remaining patterns were placed in an investment (Rematitan Plus, Dentaurum, Pforzheim, Germany). The casting was processed under argon gas protection in a two-chamber casting machine (Rematitan, Dentaurum, Pforzheim, Germany) with temperatures around 1668ºC. All metallic structure were cleaned and sandblasted for 10s with 250 µm $Al_2O_3$ particles (at 3.0 bar) at a fixed distance of 20mm with a single reservoir of sand. Cleaning of the sandblasted specimens was performed according to the manufacturer's instruction before receive the bonding system. After surface preparation was completed, each group were divided into two other groups to receive the aesthetic materials (Table 1).

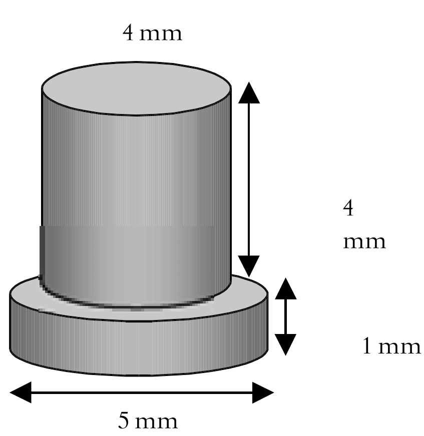


**FIGURE 1** - Metallic structure.

**Table 1 – Materials**

| Group | Metal | Polymer-glass |
|---|---|---|
| I | Gold alloy | Artglass |
| II | Gold alloy | Targis |
| III | Ti cp | Artglass |
| IV | Ti cp | Targis |

Before Artglass opaque handling, the Siloc system (Heraeus Kulzer, Wehrhein, Germany) was applied onto the circular (4.0mm) gold alloy surface (Group 1). Siloc-pre material was first applied uniformly with a disposable brush, and then allowed to dry for 2 minutes. The Siloc-pre was then activated in a proper chamber. These ten metallic cylinders were perfectly fitted in a Teflon matrix 4.0mm internal diameter and 4.0mm height (Fig.2). Two layers of Artglass opaque (Heraeus Kulzer, Wehrhein, Germany) were light polymerized with a xenostroboscopic unit (UniXS, Heraeus Kulzer) for 90s for each layer. Two layers of Artglass dentin were applied (2mm each layer), pre-polymerized and the whole metal/resin was carefully removed from the matrix. Then, the final polymerization was accomplished according to the manufacturer's instructions. For the second group (gold alloy/Targis), onto ten alloy surfaces, a thin layer of Targis Link (Ivoclar, Germany) was applied, with holding time of 5 minutes, followed by two opaque layers and two dentine layers which were light-polymerized by their proper machines (Targis Quick-10s and Targis Power- 11minutes, respectively). The same matrix was employed and removed as in the first group. For the two last groups (group 3 and 4) the same materials and sequences were repeated, except for using Ti cp metal instead of gold alloy. Hence, the third group was managed by Ti cp plus Artglass and the fourth group by Ti cp plus Targis. For all groups the specimens got the same measurements (Fig.3).

Specimens were tested in a mechanical testing machine (Model 4301, Instron Co, Canton, Mass.) with cell load of 500kg, with a crosshead speed of 0.5mm/min. A cylinder apparatus with a rectangular section was developed to execute the test and was composed by two independent parts: A-outer and B-inner part (Fig 4), which worked like a piston. Both of them had one whole with 4.0mm in diameter. When the wholes kept coincidence between themselves, the specimen was fitted through them, so that the resin part stayed inside the inner part and the metallic structure stayed inside the outer one (Fig 5)[6]. Force was applied onto the top of inner part until breakage occurred. The force, output from the machine was divided by the bonding surface area and the results reported in megapascal (MPa). The fractured specimens were evaluated in an optical microscope under magnification to determine the nature of the failure (cohesive, adhesive, or combination) and the interfaces, which were involved. Failure was described as adhesive if there was an absolute clean separation of the interfaces.

## Results

The fracture load was recorded for each of the fourthy specimens and the shear bond strength between titanium or gold alloy and resin materials was calculated. The obtained data were statistically treated through ANOVA and Tukey Kramer tests that revealed significant differences ($p<.05$) among the groups. The results are presented in Table 2. Considering only the metals, the gold alloy presented best shear bond strength for the combination with both Targis and Artglass. The binomy Au/Targis achieved the highest average (m=21.57MPa) in relation to the other combinations being significantly superior when groups were compared.

In relation to the composite resin, Targis presented the highest average (m=16.61MPa) when compared to Artglass (11.64MPa).

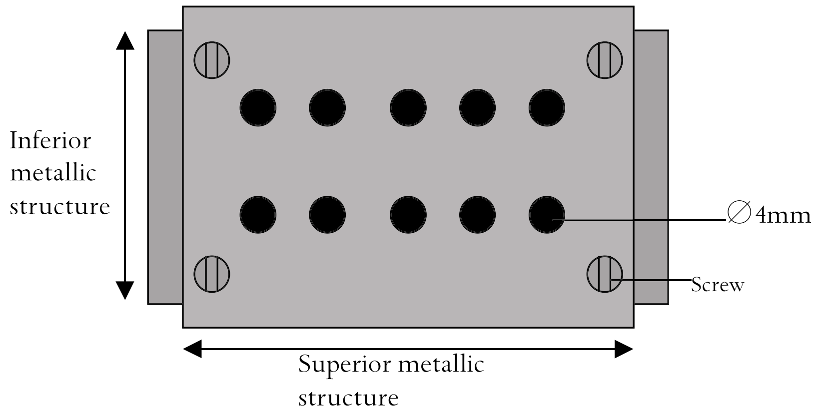


**FIGURE 2** - Teflon matrix.

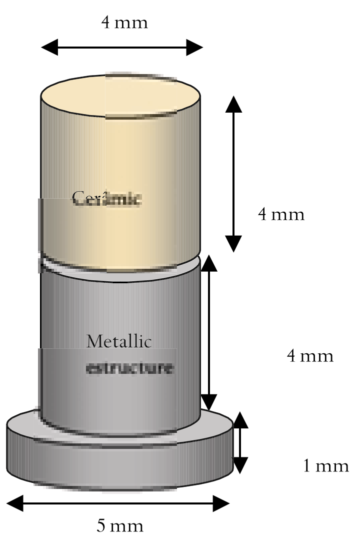


**FIGURE 3** - Specime.

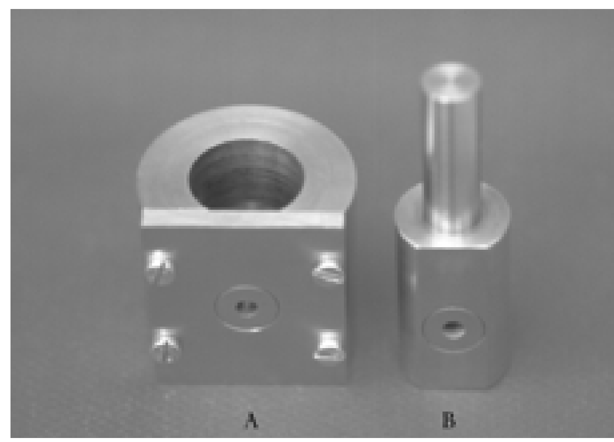


**FIGURE 4** - Mechanical device. **A** - Outer part; **B** - Inner part[6].

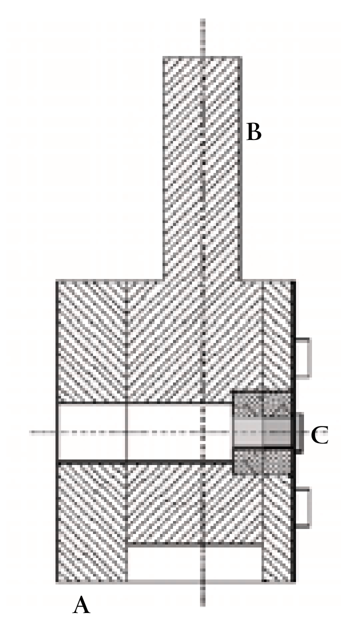


**FIGURE 5** - Specimen (**C**) fitted through mechanical device (**A** and **B**). Cross section view.

**Table 2 – Results of ANOVA for shear bond strengths (p=0,05)**

| Group | N | Mean | SD |
|---|---|---|---|
| 1- Artglass/Au | 10 | 15.30 a | 4.79 |
| 2- Artglass/Ti | 10 | 7.97 b | 2.78 |
| 3- Targis/Au | 10 | 21.57 c | 3.81 |
| 4- Targis/Ti | 10 | 11.65 d | 3.98 |

## DISCUSSION

Considering the technological advances, the inconveniences of acrylic resins used as aesthetic veneering of prosthetic restorations have been overcome with great efficiency by light activated materials[20]. The incorporation of glass microparticles in the resin mass increases its mechanical and aesthetic characteristics, assuring longer lasting to installed prosthesis[18]. It has also been part of this evolution, the establishment of the chemical link between the resin and the pretreated metallic surfaces followed by the application of bonding agent. Relying on this bonding manufactures have not suggested the production of mechanical retentions, favoring the final aesthetic result of the restoration as it permits larger space for the dentin/enamel layer, especially in limited areas[10].

Trying to establish and improve the adherence between titanium and resin or ceramic materials is a elaborated procedure. The excess of the oxide layer generated during the process of casting seems to damage the adherence of materials of aesthetic veneering once the elevated temperatures make titanium extremely reactive, generating a crust in its surface[5]. Other factors may also influence the adherence significantly. The composition of investments used during the casting process[16], the conditions offered by the equipment, the pressure in which molten titanium is introduced inside the investment[19] as well as the finishing of the surface after casting[8] should be considered when studying the adherence between titanium and aesthetic material.

In this research, it was possible to verify the shear bond strength of the interface between cast titanium and two second generation resins for laboratory. It was used the gold alloy as a control group and this achieved the best averages for both resins tested. The gold alloy also obtained the largest capacity for bonding to porcelain when compared to palladium, nickel-chromium and commercially pure titanium[15]. However, another study using a shear test based on torsional load showed that the bonding of titanium to porcelain was better than that of gold-porcelain system[2].

Some researchers believe that the success of bonding aesthetic materials on precious and nonprecious alloys is related to surface roughness, consequently promoting better mechanical interlocking between them. Despite the fact that it was not realized the scanning electron micrograph after metallic structures sandblasting, this procedure might suggest reasons for fracture resistance superiority of gold alloy and composite resin interface. The lower hardness surface of gold when compared with titanium can create more degree of surface roughness.

The observation with optical microscopy of the metallic surfaces of titanium and gold alloy didn't reveal any remain of the bonding agent, opaque or resin (Fig. 6). Therefore, the aspect suggested totally adhesive failure between the resin and the metal. On the resin surface of the same specimens there were not metallic remains, including the oxide layer, which was expected to be detached from the titanium metallic structure (Fig. 7).

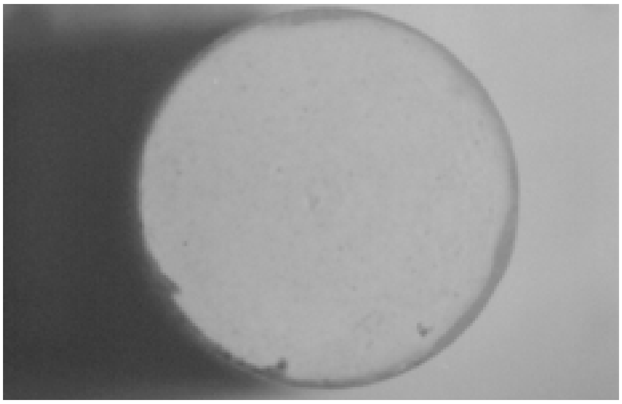

**FIGURE 6** - Polymer surface after mechanical test.

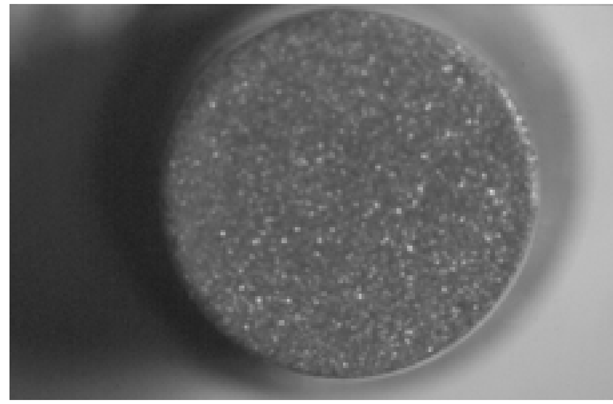

**FIGURE 7** - Metal surface after mechanical test.

## Conclusions

According to the results, the following conclusions were drawn:

1. The gold alloy established a better bond strength to polymers when compared to commercially pure titanium.
2. Targis/gold alloy interface achieved the highest mean of shear bond strength being the best combination realized.
3. For all tested combinations, the failures, in optical microscopy, were adhesive, not being found any remains of material in the metallic or resin surfaces.


## Resumo

Novos compósitos indiretos com propriedades melhoradas têm sido introduzidos no mercado como alternativa à porcelana. Existem poucas informações observadas na união adesiva entre estes sistemas de compósitos e o metal, principalmente quando são aplicados na superfície do titânio. Este estudo avaliou a resistência ao cisalhamento de dois polímeros (Artglass/Heraeus Kulzer e Targis/Ivoclar) com o titânio fundido (Ti) e uma liga áurica (Au). Vinte estruturas metálicas (4mm de diâmetro e 4mm de altura) de cada liga foram fundidas e jateadas com óxido de alumínio de 250_m antes da aplicação do sistema adesivo de cada polímero. O opaco e dentina do Artglass foram aplicados sobre a estrutura metálica usando uma matriz de teflon. O mesmo procedimento foi conduzido para o Targis e polimerizado de acordo com as recomendações dos fabricantes. As amostras foram armazenadas em água destilada por 24 horas a 37ºC e termocicladas (5º e 55ºC/3000 ciclos). A resistência ao cisalhamento foi realizada em uma máquina de teste universal (Instron) com velocidade de 5mm/min. Os dados foram analisados estatisticamente com os testes two-way e Tukey (_=0,5). Os resultados indicaram que a liga de ouro foi estatisticamente superior ao titânio (18,44MPa e 9,81MPa, respectivamente) e o cerômero Targis (16,61MPa) apresentando resistência maiores significantes em relação ao polímero de vidro Artglass (11,64MPa) em todas as ligas. O melhor resultado foi encontrado quando o cerômero foi aplicado sobre a liga de ouro.



## Unitermos

Titânio; cisalhamento; adesivo; resina

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