Effect of Laser Welding on the Titanium Composite Tensile Bond Strength

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The aim of this study was to analyze the shear bond strength between commercially pure titanium, with and without laser welding, after airbone-particle abrasion (Al_2O_3) and 2 indirect composites. Sixty-four specimens were cast and divided into 2 groups with and without laser welding. Each group was divided in 4 subgroups, related to Al_2O_3 grain size: A - 250 µm; B - 180 µm; C- 110 µm; and D - 50 µm. Composite rings were formed around the rods and light polymerized using UniXS unit. Specimens were invested and their shear bond strength at failure was measured with a universal testing machine at a crosshead speed of 2.0 mm/min. Statistical analysis was carried out with ANOVA and Tukey's test (α=0.05). The highest bond strength means were recorded in 250 µm group without laser welding. The lowest shear bond strength means were recorded in 50 µm group with laser welding. Statistically significant differences (p<0.05) were found between all groups. In conclusion, airborne particle abrasion yielded significantly lower bond strength as the Al_2O_3 particle size decreased. Shear bond strength decreased in the laser welded specimens.

Key Word: laser welding, titanium, composite resin, shear bond strength.

INTRODUCTION

The use of titanium and its alloys for cast restorations and partial denture frameworks has increased substantially over the last years, because of their excellent biocompatibility, corrosion resistance, high strength-to-weight ratio, and low cost (1,2). Also, this trend can be mainly attributed to the development of casting technology for titanium alloys, such as new casting machines, investment materials and the extensively reported advantages of titanium over other base metal alloys (1,2). However, the presence of an excessive oxide layer is a possible cause of decreased bonding durability (3). Nevertheless, the union of titanium to resin materials in metal-composite crowns and fixed partial dentures remains problematic because the success of these restorations relies on the establishment of a strong bond between composite and titanium substructures (4).

Metal-composite restorations have long been used as an alternative for dental restorations. Indirect light-cured composite resins have been extensively used in tooth restoration because they can provide acceptable aesthetics, wear resistance similar to tooth structure, and are easy to manipulate in the laboratory and to repair. However, durable bonding between composite resins and metal frameworks has been a challenge because the deficient bonding of indirect composites to the metallic alloys could promote the formation of marginal gaps and causing weak bond strength. Several adhesive systems have been introduced in an attempt to solve this problem (5). In addition, retention of indirect composites for fixed prosthesis and crowns to metal frameworks can be obtained by micromechanical (air-abrasion, electrolytic etching, porous metal coating), macromechanical (mesh, beads, rough surface with particles) and chemical (4-META composites, phosphate-based

composites), and adhesive layer application (tin plating, silanization) (5,6).

Some studies (6) have shown that silicoating systems improve the metal-composite bond strength with titanium and its alloys. However, the stability of titanium-composite bond is still questionable. While studies (4-7) have shown that shear bond strength results were considerably affected by thermal cycling or long-term water storage, other studies (7-9) have demonstrated that a number of the composite/adhesive systems evaluated under various metal surface conditioning methods exhibited considerably high and durable bond strength. Airborne-particle abrasion of dental alloys with alumina particles is commonly used to clean the pieces and to increase micro-retention and surface area. Although this is a common technique used in dental alloys for removal of metal oxide or debris and to improve bonding, it may be as problematic in its use. However, contamination (8) and distortion (9) of dental prostheses caused by airborne-particle abrasion might be main disadvantages increase the surface energy (8-9).

Despite the improvements in bonding of materials to cast metal frameworks, failures may still occur. To overcome these shortcomings, soldering of composite-metal crowns has become increasingly more frequent. This procedure reduces the laboratorial time (10) because it is performed directly on the cast model in areas very close to the esthetic material without causing damage, fracture or color change due to the use of techniques that do not affect the metal or alloy structure. Laser welding is one of the currently available options. This soldering technique has been extensively evaluated (11) and its reported advantages include high mechanical strength, minimal zone of heat influence and hence lesser deformation, which allows repairs and use with almost all dental alloys (12).

Such controversial and limited results concerning adhesive performance between indirect composite and titanium alloys has led to the lack of an informed design rationale for bonding composite materials to titanium and its alloys. The current study evaluated the bond strength of two indirect composite/adhesive systems to commercially pure titanium subjected to different particle sizes of airborne aluminum oxide (Al_2O_3) abrasion after laser welding. The hypotheses to be tested were that the bonding techniques involving airborne-particle abrasion and laser welding of the titanium surface produce higher bond strength values.

MATERIAL AND METHODS

This study was designed to evaluate the bonding characteristics of commercially pure titanium (CpTi - Tritan - grade 1, Dentaurum, Pfozheim, Germany) to two indirect composites: Artglass; Heraeus Kulzer, Wehrheim, Germany and Solidex; Shofu, Kyoto, Japan.

Sixty-four CpTi specimens were prepared. To obtain precise dimensions of the CpTi specimens, brass cylindrical rods (3.0 mm diameter x 60 mm length) were used as patterns and invested in a commercial phosphate-bonded investment for titanium (Rematitan Plus; Dentaurum J.P. Winkelstroeter KG, Pforzheim, Germany) in casting rings. After investment setting and removal of the brass rods, the casting rings were preheated in a furnace (EDG 7000 3P; EDG Equipamentos e Controles Ltda., São Carlos, SP, Brazil). Thereafter, the casting rings were heated, as *per* manufacturer's instructions.

Casting of the CpTi specimens was performed in a Discovery Plasma Ar-arc vacuum-pressure casting machine (EDG Equipamentos e Controles Ltda.), which produces electric arc melting in vacuum and argon-inert atmosphere, with vacuum-pressure injection of the alloy into the mold. The CpTi rods were divested and cleaned with carbide burs (702L; KG Sorensen Ind. Com. Ltda., Barueri, SP, Brazil) followed by 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 4 s under 2-bar air pressure.

The CpTi rods were assigned to 2 groups (n=16), according to the: Group 1 - with laser welding (Desktop Laser; Desktop, Dentaurum, Pforzheim Germany; 10 ms impulse duration and 1 Hz frequency), according to manufacturer's instruction; and Group 2 (control) - without laser welding. Each group was divided into 4 subgroups (n=4), according to the surface treatment (airborne-particle abrasion) prior to application of composites rings: A: 250 μm Al_2O_3 particles; B: 180 μm Al_2O_3 particles; C: 110 μm Al_2O_3 particles; D: 50 μm Al_2O_3 particles. To ensure that they were airborne-abraded in a single direction, the titanium rods were rotated by an engine at approximately 800 rpm during 10 s, maintaining 2 mm from the metal handpiece of a modified unit (Bijato; F&F, Araraquara, SP, Brazil).

The area that should receive the composites application was demarcated by 2 silicone slides (Optosil, Bayer, Leverkusen, Germany) and a 2.5 mm-thick polyester spacer (Plexiglass; General Electric, Mt. Vernon,

IN, USA) (14) in order to obtain a standard composites rings for each specimen. In all specimens, composite application was performed by a single investigator. The final dimensions of the composite rings around the rods were 6.0 mm diameter x 2.0 mm thickness.

For Artglass group, the Siloc Bond (Heraeus Kulzer) was uniformly applied. After 5 min for activation and drying to occur, a thin layer of opaque paste (Artglass Opaque; Heraeus Kulzer) was applied with a brush and polymerized for 90 s in the light-polymerizing unit (UniXS; Heraeus Kulzer), which has 2 Xenon strobe lamps (wavelength range: 320-520 nm; 1000 mW/cm^2; 20 Hz). After activation, the composite was stratified into 2 phases with the aid of a spatula, and then brushed with a fine layer of modeling liquid (Artglass liquid, Heraeus Kulzer GmbH) to help shape the composite externally. The specimens were placed in the light-polymerizing unit (UniXS) for 180 s in each phase. For the Solidex composite group (Shofu Dental Corp., San Marcos, CA, USA), a coat of Solidex Opaque liquid adhesive was applied and light cured for 1 min (UniXS; Heraeus Kulzer), and the specimens were polymerized for 3 min in the light-polymerizing unit (UniXS). The Solidex composite was then applied to shape the composite rings and polimerized for 3 min in the light-polymerizing unit (UniXS).

The specimens were individually tested to assess the shear bond strength at metal-composite interface. Two measurements, perpendicular to each other, were made of the rod diameter immediately above and below the ring as well as 4 equally distanced measurements of the composite rings thickness. The mean value of these measurements was assumed to represent the rod diameter and the composite ring thickness, respectively. The mean values were used to calculate the metal-composite bond area with the formula: $S = \pi.\varphi.e$, where: S = metal-composite bond area; φ = rod diameter, and e = composite ring thickness.

To determine the metal-composite bond shear strength, the composite rings were embedded in the center of a PVC cylinder with type IV gypsum (Vigodent S.A., Ind. e Com., Rio de Janeiro, RJ, Brazil) and a dental surveyor (Bio-Art Equipamentos Odontológicos Ltda., São Carlos, São Paulo, Brazil). After gypsum set, the PVC rings were removed from the specimens and pulled in a universal testing machine (EMIC MEM 2000; EMIC Equipamentos e Sistemas de Ensaio Ltda., São José dos Pinhais, PR, Brazil) until fracture. The test was done in a standardized manner at a constant crosshead speed of 2.0 mm/min (13).

The rupture peak load was used to calculate the shear bond strength, indicator of metal-composite shear bond strength, using the following equation (Eq. 2): T = (F/S) x 9.8 MPa (2), where: T = shear bond strength; F = critical rupture load; S = metal-composite bond area.

Data were subjected to ANOVA and Tukey's complementary test. All statistical analyses and calculations were undertaken using the SPSS 12.0 for Windows statistical software (SPSS Inc., Chicago, IL, USA) at 5% significance level. In each group, the airborne-particle abrasion CpTi rod surface was examined under scanning electron microscopy (SEM). Representative SEM micrographs were made of a different region of each specimen.

RESULTS

Table 1 summarizes the mean bond strength values and standard deviations of the 2 groups investigated. Tukey test showed that composites, laser welded, and airborne-particle abrasion protocol in CpTi rods exhibited significant influence on the mean shear bond strength values (p<0.05). There were interactions between composites and laser welded (p<0.05) and between composites and airborne-particle abrasion protocol (p<0.05). The mean shear bond strength values of the

Table 1. Shear bond strength means (±SD) of composites Artglass (A) and Solidex (S) to commercially pure titanium without laser (WL) and with laser welding (L).

Group		Aluminum oxide particle (μm)			
		250	180	110	50
Artglass	WL	20.82 a (±2.97)	20.04 a (±2.10)	17.23 ab (±2.56)	19.12 a (±5.08)
	L	19.30 a (±2.73)	13.91 b (±2.27)	14.62 b (±1.91)	11.60 b (±2.55)
Solidex	WL	19.55 a (±1.17)	19.11 a (±2.60)	17.96 ab (±1.81)	15.45 b (±1.86)
	L	18.00 a (±1.42)	17.18 ab (±2.80)	16.59 b (±2.21)	14.11 b (±2.07)

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Artglass group were greater than those of Solidex group.

The Tukey's test showed that the Artglass no-welded group had the highest mean shear bond strength value, with Artglass without laser welded (AWL) in the different group by airborne-particle abrasion protocol. Because of the interaction, ANOVA and the Tukey test were conducted to determine the effect of surface roughness procedure for each airborne-particle abrasion protocol. The results showed that the group remained similar for the Artglass and Solidex group and that there was no statistical difference within the 250 µm group. On the other hand, the specimens airborne-particle abraded with of 180, 110 and 50 µm particle size had statistically different bond strength (p<0.05) (Table 1).

For composites, the analysis of the bond strength values showed statistically significant difference by the airborne-particle abrasion protocol and laser welded (p<0.05). The Tukey's test showed that AWL group had the highest mean bond strength value, followed by Solidex without laser welding (SWL) in the same group by airborne-particle abrasion (Table 1).

Examination of surface morphologies with SEM showed that both modes of failure were present for all groups of specimens. The appearance of titanium on the surface was different in both conditions (airborne-particle abrasion and laser welding techniques). Figure 1 shows irregularity surfaces of no welded specimens and combined the different airborne-particles abrasion in the different particles sizes. Figure 2 shows the surface of welded specimens and the Figure 2D revealed a distinct surface on the specimen with no alteration after airborne-particle abrasion protocol.

DISCUSSION

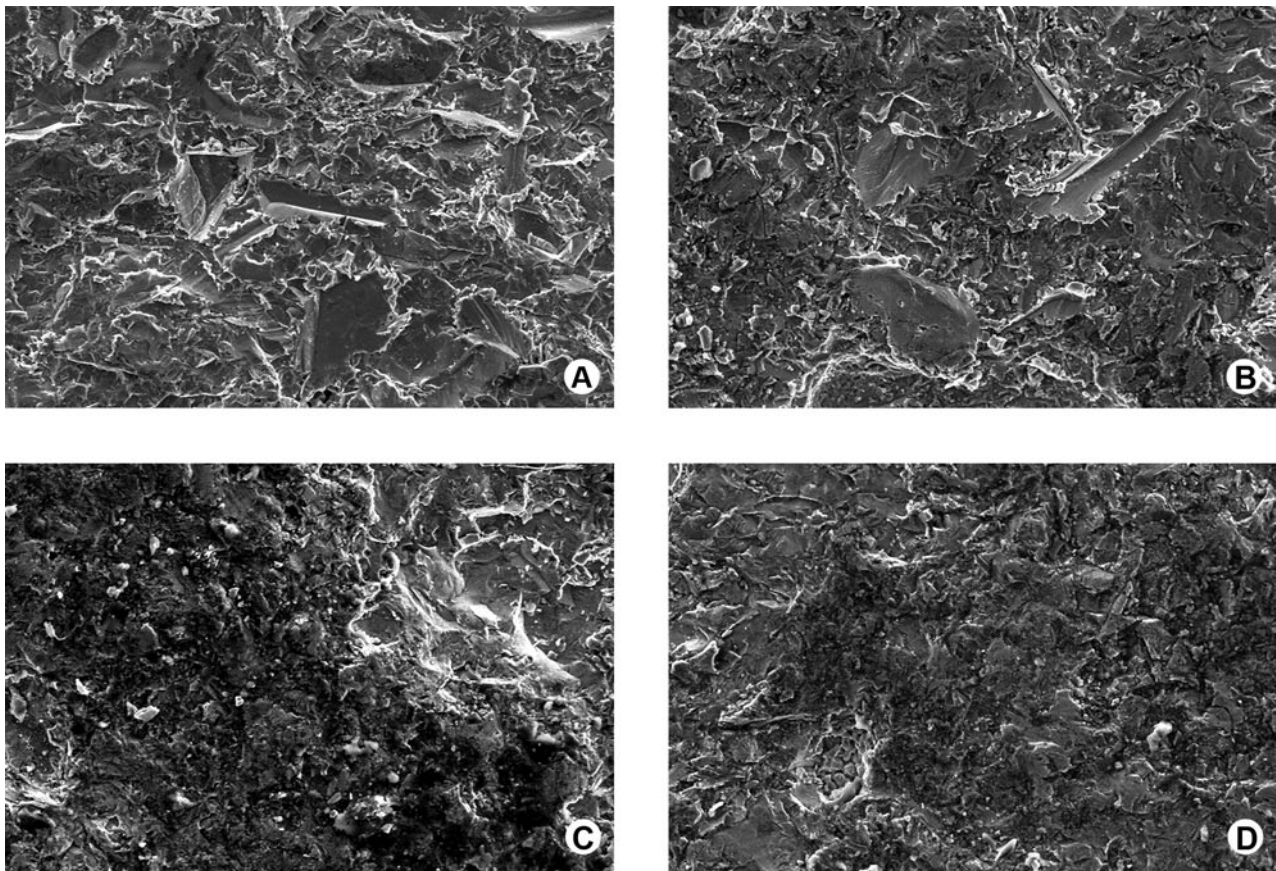Metal-composite restorations have not been well

Figure 1. SEM micrographs of non-welded commercially pure titanium rods after airborne particle abrasion with different aluminum oxide particle sizes: 250 µm (A), 180 µm (B), 110 µm (C) and 50 µm (D).

accepted generally due to what has been perceived as an insufficient ability to bond composite to metal castings. However, several systems have been developed over the last two decades in order to improve the bond strength of composites to metals, including titanium. These systems involve treatment of titanium surface to render it more reactive to bonding agents either by coating the metal surface with silicate (enabling bonding through a silane coupling agent or laser welded) or by airborne-particle abrasion with different Al_2O_3 particles (14).

Airborne-particle abrasion with Al_2O_3 not only results in surface micromechanical roughness, but left aluminum particles embedded in the surface. The role of this Al_2O_3 on bonding for the composite/titanium systems is not well known. In bonding systems containing chemical active products which are applied directly to the airborne-particle abrasion surface, bonding this embedded aluminum might also play an important role

in bonding mechanism, mainly in association with new techniques like laser welding, because airborne-particle abrasion with alumina created micromechanical roughening of the surface (15). Airborne-particle abrasion of titanium alloys also might have an effect on bond strength because they remove not only the impurities on the surface of titanium, but also the oxide films formed on the surface, influencing the formation of the new oxide film. The airborne-particle abrasion process may introduce physicochemical changes, which affect surface energy and wettability. The decreases in the surface energy of the airborne-particle abraded metal might be attributable to changes in surface composition by residues from the abrasive media (7,14).

Results from this study showed higher values of the 250 μm Al_2O_3 particles group in both composites compared with all of the experimental groups, which confirm the superiority of composites adherence to
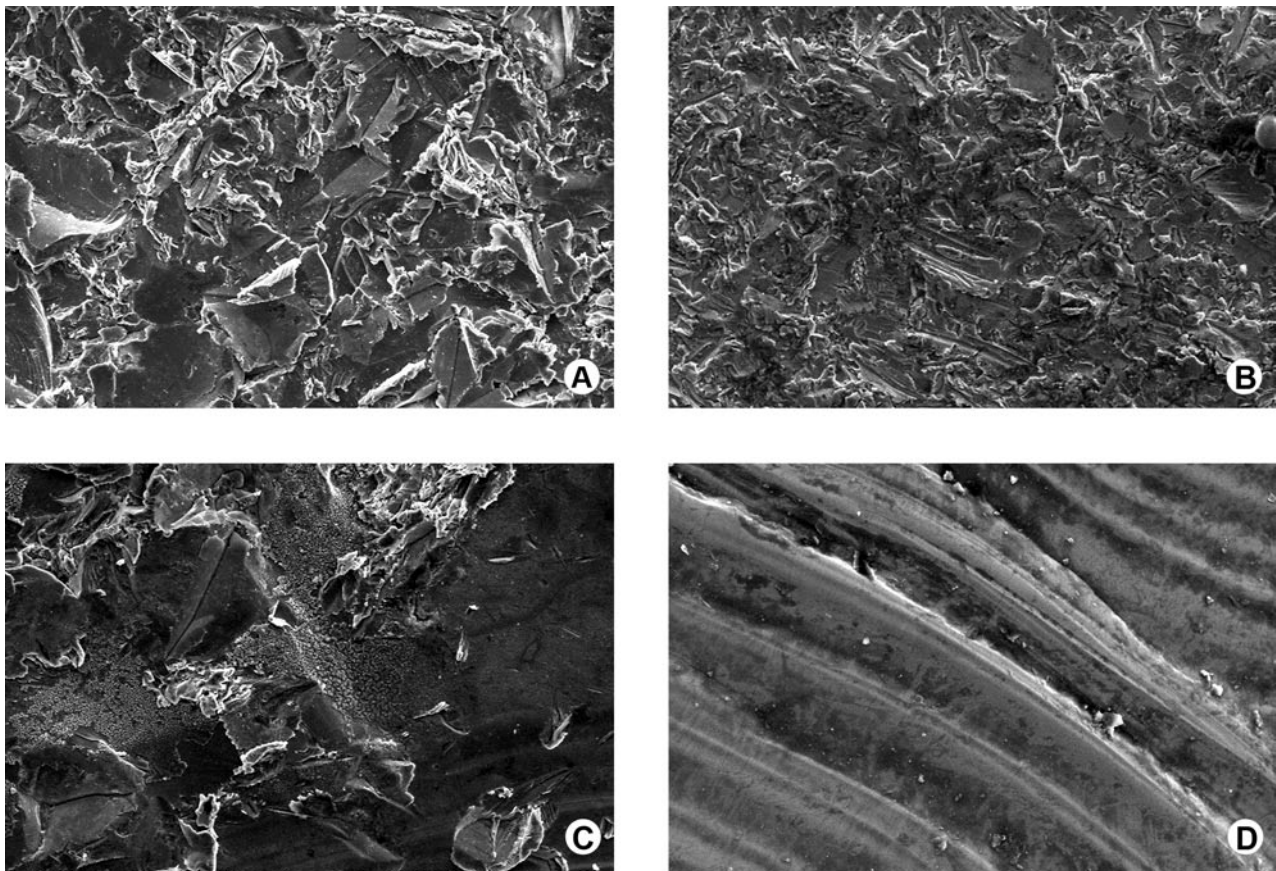

Figure 2. SEM micrographs of commercially pure titanium rods after laser welding and airborne particle abrasion with different aluminum oxide particle sizes: 250 μm (A), 180 μm (B), 110 μm (C) and 50 μm (D).

commercially pure titanium (CpTi) in more irregular surface. In addition, the mechanical union has relation with the surface roughness and usually the airborne-particle abrasion with Al_2O_3 increased the roughness that leads to the increase of adhesion area (16). The CpTi surface no laser welded presented more irregular surface that provided the union of the composite to the surface, especially with the 250 µm aluminum oxide (Al_2O_3), that has the potential to remove significant amounts of substances, like casting impurity and investment, and could affect the bond strength (17). Material loss that results from airborne-particle abrasion roughness surface is important to the clinical fit of restorations (16,18). However, unnecessary airborne-particle abrasion of the restorations should be avoided because it is likely to damage the margins of the restorations (17).

The lower values obtained with a welded group of the different composites can be explained by the surface. The metal area which received the laser weld showed the lower alterations in the surface after airborne-particle abrasion with the aluminum oxide particles. This is probably because the physical and mechanical properties can be different in the surface after laser welding. These results also indicate that airborne-particle abrasion of titanium with laser welded requires a different technique, as a particle size, from that normally used for conventional dental alloy. However, because the titanium reactivity, even when the oxide layer was removed from the surface, re-oxidation of the titanium proceeded immediately (19), and this can be a problem for titanium adhesion.

The Artglass and Solidex composites specimens, the no welded group specimens airborne-particle abraded with the different oxide particles showed higher bond strength compared with the laser welded group. These results indicate that the mechanical retention over no laser welded alteration is obtained when did not exist any modification in the CpTi surface.

There are some reports in the literature on the aluminum content of titanium after airborne-particle abrasion (1,14,17,19). However, in previous studies on various dental alloys (20), the high amount of aluminum was found after airborne-particle abrasion. Ultrasonic cleaning of the airborne-particle abraded samples resulted in a slight decrease in the aluminum. However, based on the variations within the groups the reductions found in the surface are statistically significant. In addition, SEM findings in this study suggest that the increase

in the particles size during the airborne-particle abrasion procedure could increase the shear bond resistance, due to the alteration of the surface irregularity.

As mechanical bonding would play an important role in maintenance of the bond structure in this situation, increased mechanical retention is essential for durability of the adhesion interface. It is speculated from the results shown in Figures 1 and 2 that the irregular surfaces formed by airborne-particle abrasion are effective for increasing mechanical retention and establishing reliable bonding.

It is also important to realize that even though this is an *in vitro* study, because the clinical implication of the results may be important. Future clinical studies are recommended to verify the findings of this study. To better understand titanium-composite bonding, further investigations with x-ray photoelectron spectroscopy to determine the oxidation status of titanium, and transmission electron microscopy to closely analyze structure (critical defects) and composition at the titanium-composite interfaces are needed.

Within the limitations of this study, the treatments based on airborne-particle abrasion (aluminum oxide) of the titanium surface produced the highest mean repair bond strength values, dependent of the laser welded and composite (Solidex and Artglass), did not confirm the hypothesis of the study. Airborne-particle abrasion plays a critical role in composites bonding to commercially pure titanium by creating roughened surface and this is combined between titanium and composite did increase the bond strength significantly when decrease the aluminum particle.

RESUMO

O objetivo deste estudo foi analisar a força de união entre o titânio comercialmente puro com solda e sem solda modificado por partículas de óxido de alumínio (Al_2O_3) e duas resinas indiretas. Um total de 64 espécimes foram fundidas e divididas em dois grupos sem solda e com solda a laser. Cada grupo foi novamente divididos em 4 subgrupos, de acordo com o tamanho de partículas de óxido de alumínio utilizado: A - Al_2O_3 (250 µm); B - Al_2O_3 (180 µm); C - Al_2O_3 (110 µm); D - Al_2O_3 (50 µm). Anéis de resina foram polimerizados ao redor das hastes de titânio no equipamento UniXS. Os espécimes foram embutidos em gesso e a força de união foi mensurada com auxílio da máquina de ensaios universais. ANOVA e teste de Tukey (p<0,05) foram utilizados para análise estatística. Os maiores valores de força de união foram registrados no grupo de 250 µm sem solda a laser. Os menores valores foram registrados para o grupo de 50 µm com solda a laser. Alterações estatisticamente significantes foram

observadas entre todos os grupos (p<0,05). As forças de união diminuíram significantemente com a diminuição dos tamanhos das partículas de óxido de alumínio. A força de união diminuiu nas amostras que receberam a solda a laser.

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