

# Effect of Air Abrasion and Different Silicoating Techniques on the Bond Strength of Particulate Filler Composite to Titanium

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## Research Article

Volume 2 Issue 3

Received Date: March 10, 2017

Published Date: August 03, 2017

DOI: 10.23880/oajds-16000139

## Abstract

**Background/purpose:** One of the problems associated with titanium metal-composite restorations is insufficient ability to bond resin to titanium metal castings. The purpose of this study was to evaluate the effect of two different silicoating techniques and air abrasion with different particles on the bond strength of a particulate filler composite to titanium alloy.

**Materials and Methods:** 40 titanium bar specimens divided in 5 groups were air abraded with 50 µm aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) particles (Group C); air abraded with Al<sub>2</sub>O<sub>3</sub> and then flamed with the Silano-Pen device (Bredent, Germany) (Group CSP); air abraded with silica-coated aluminum oxide particles (Group S); air abraded with synthetic diamond abrasives (Group D); and air abraded with diamond abrasives and flamed with the Silano-Pen device (Group DSP). The composite resin build-up was fabricated according to the manufacturer's recommendations. Shear bond strength of resin composite to the titanium surface were evaluated after 24 h storage in water at 37 °C.

**Results:** Data were analyzed by one-way analysis of variance (ANOVA), and the mean values were compared by post-hoc Tukey compromise test (p=0.05). The SEM photomicrographs were developed at a magnification of ×500 for visual inspection. To assess the mode of failure (cohesive, adhesive or mixed failure) the specimen surfaces were examined. There was no significant difference in bond strength values of group C and the other surface treated groups. (p>0.05). Application of Silano-Pen increased shear bond strength in treated with only synthetic diamond abrasives group. Group CSP showed higher bond strength than group S and D. Group DSP showed higher bond strength than group D, too.

**Conclusion:** Although Silano-Pen application after air abrasion with synthetic diamond abrasives effected the bond strength of titanium to composite, there was not any difference from only air abraded surface treatment.

**Keywords:** Silicoating; Titanium; Particulate filler composite

## Introduction

The development of new processing methods- such as lost-wax casting, computer-aided machining and electric discharge machining- has allowed the use of pure titanium and its alloys for dental implants, surgical splints, stents and fasteners, partial denture frameworks, fixed partial dentures and root posts [1-9]. Although various metal alloy can be used to fabricate fixed partial dentures, titanium presents several advantages as a prosthetic material, such as excellent biocompatibility, good mechanical properties, high corrosion resistance, low density and low cost compared to noble metals [2-11]. However, problems with porcelain bonding have been reported when titanium is used in metal-ceramic restorations as thick and non-adherent layers of titanium oxide are formed. The presence of an excessive oxide layer is a possible cause of decreased bonding durability. For this reason, particulate filler composite resin materials used on titanium castings as an alternative to porcelain fused to metal restorations for esthetic anterior/posterior restorations [9-12]. 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 [3,8,9]. However, one of the problems associated with titanium metal-composite restorations is insufficient ability to bond resin to titanium metal castings. The deficient bonding of indirect composites to the metallic alloys could promote the formation of marginal gaps and causing weak bond strength [2,3,6,8,9]. Like other metal alloys, use of titanium alloy for fixed partial denture requires a strong and stable bond between metal and esthetic veneering material (porcelain or composite resin) or metal and resin cement [11,13].

Several systems including chemical and mechanical surface treatment methods have been developed in order to improve the bond strength of resin materials to titanium. Macro-(mesh, beads, rough surface with particles) or micro-mechanical treatments (air-abrasion with alumina particles, electrolytic etching, porous metal coating, and acid etching), chemical treatments (4-META composites, phosphate-based composites) and adhesive layer application (tin plating, titanate coupler, silane coupling, carboxylic monomers, a phosphate monomer) were the only mechanisms for bonding composite materials to metallic substrates [2,3,6-9,14].

Other 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), conditioning the metal surface with organic acids in solvents, embedded in methacrylate-based liquids and polydimethylsiloxane coating that bond to the titanium oxides and to the resin composites [7-10,15]. Airborne-particle abrasion with aluminum oxide particles is commonly used for to clean the surfaces by removal of metal oxide or debris, to clinical fit of restoration and to increase micro-mechanical roughening and physicochemical changes which affect surface energy and wettability of the surface retention [6,9].

In dentistry, one alternative air abrasion material is synthetic diamond particles. It was previously applied onto the glass-infiltrated aluminous oxide ceramic surface to increase the surface roughness and surface area [16,17] then it was used on zirconia core, Ni-Cr and Co-Cr surface [18,19]. Several studies have shown that silicoating and functional monomer systems improve the composite-to-metal bond strength with noble and non-noble dental alloys and with titanium and its alloys [2,8,11,19]. The aim of this study was to evaluate the effect of two different silica-coating techniques and air abrasion with different particles on the bond strength of a particulate filler composite to commercially pure titanium alloy. The research hypothesis tested was that the shear bond strength would increase after application of alternative surface treatment methods.

## Material and Methods

The materials used in this study were presented in (Table 1). Titanium bars of commercially pure titanium (Grade 2, Bağsan Tic. A.Ş., Turkey) were sectioned with a lathe (Hawk TC-150, Cincinnati Machine Ltd. Co., UK) and 40 square-shaped specimens (10 × 10 mm, 2 mm in thickness) were obtained. All specimens were embedded in the centers of autopolymerizing acrylic resin blocks (Meliodent; Heraeus Kulzer, Armonk, NY). Then the surface of each specimen was ground with a series of silicon carbide papers (280-, 400-, 600-, 800- and 1000-grit) (3M ESPE, St. Paul, USA) for 10 sec on a 300 rpm grinding machine (Buehler Metaserv, Buehler, Germany) under running water in order to provide flat and uniform surface and ultrasonically cleaned for 3 min deionized water and air-dried. All specimens were divided 5 groups, each containing 8 specimens. The specimens in the groups were air abraded with 50 µm aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) particles (Group C); air abraded with 50 µm aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) particles and then flamed with the Silano-Pen device (Bredent GmbH, Senden, Germany) (Group

CSP) for 5 s/cm<sup>2</sup>; air abraded with 30 µm silica-coated aluminum oxide particles (SiO<sub>x</sub>) and application of a thin silane layer (Espe-Sil, 3M ESPE), which was left to dry for 5 minutes (Group S); air abraded with 30-50 µm synthetic diamond abrasives (Group D); and air abraded with 30-50 µm synthetic diamond abrasives and then flamed with the Silano-Pen device (Group DSP) for 5s/cm<sup>2</sup>. After specimens were flamed with Silano-Pen device,

specimens were cooling down to room temperature and then the Silano-Pen bonding agent was brushed on and air-dried for 3 min. Air abrasion procedure was performed using an intraoral air abrasion device (Microetcher, Danville Engineering Inc, San Ramon, Calif, USA) at an air pressure of 2.5 bars for 10 sec at a distance of approx. 10 mm.

Material	Composition	Manufacturer
Signum Metal Bond I+II	Inorganic pigments, SiO <sub>2</sub> , methyl methacrylate, acetone	Heraeus Kulzer, Hanau, Germany
Signum Composite	Bis-GMA and TEGDMA - SiO <sub>2</sub> ,	Heraeus Kulzer, Hanau, Germany
	Ba-Al-Si (1,0 µm)	
Silano-Pen	alcoholic solution of 3-methacryloyloxypropyltrimethoxy silane (MPS), Ethanol	Bredent, Senden, Germany
CoJet	30 µm silica-coated Al <sub>2</sub> O <sub>3</sub> particles	3M ESPE, Seefeld, Germany
Korox® 50	99.6% 110 µm Al <sub>2</sub> O <sub>3</sub>	BEGO, Bremen, Germany
Micron+MDA M3050	30-50 µm synthetic diamond particles	Elementsix, Shannon, Ireland

Table 1: Materials used for this study.

After surface treatments a thin layer metal bond (Signum Metal Bond I) was applied onto the all specimens surface and wait 15 seconds for it evaporate then a thin layer metal bond (Signum Metal Bond II) was applied onto the all specimens surfaces and then polymerized 90 seconds with a high performance light curing unit that has 300 W power input (Heraflash, Heraeus Kulzer, Hanau, Germany). A teflon mold (2 mm thick) with a circular hole (6 mm in diameter) was positioned on the center of the specimens' surfaces. Composite resin (Signum Composite, DA2) build-up was completed by applying small increments according to the manufacturer's recommendation and polymerized 90 seconds in a light-curing unit (Heraflash). The specimens were stored for 24 h in distilled water at 37 °C. Shear bond strength of an indirect composite on the titanium surface after different surface treatment were evaluated after 24 h storage in water at 37°C. Shear bond strength was then determined using a universal testing machine (Lloyd LRX; Lloyd Instruments, Fareham, Hampshire, UK) at a cross-head speed of 1 mm/minute where shearing load was applied parallel to the bonded interface. The means and standard deviations of 40 specimens were calculated for each group. The Kolmogorov-Smirnov test showed that the data had a normal distribution ( $P > 0.05$ ). A homogeneity of variance test was done using Levene's test (F ratio: 1.288,  $P > 0.05$ ). Data were analyzed by one-way analysis of variance (ANOVA), and the mean values were compared by post-hoc Tukey compromise test ( $p=0.05$ )

following one-way ANOVA. Titanium specimens submitted to five surface treatments including a control were selected for microphotographic evaluation. All specimens were examined under a field-emission scanning electron microscope (JSM-6335F; JEOL, Tokyo, Japan) at 10 kV. The SEM photomicrographs were developed at a magnification of ×500 for visual inspection. To assess the mode of failure (cohesive, adhesive or mixed failure) the specimen surfaces were examined under stereomicroscope (Leica EZ4 D, Germany).

## Results

The means and standard deviations (SD) of the shear bond strength values are presented in (Table 2). Result of one-way ANOVA showed that surface treatments effected shear bond strength between groups ( $p<0.001$ ) (Table 2). There was no significant difference in bond strength values of group C and the other surface treated groups. ( $p>0.05$ ). Application of Silano-Pen increased shear bond strength in treated with only synthetic diamond abrasives group. Group CSP showed higher bond strength than group S and D. Group DSP showed higher bond strength than group D, too. Stereomicroscopic examination of the fractured surfaces showed that mixed fractures were most common types of failure for all groups. Pure composite resin cohesive failures were not observed. The specimens in Group C, S and CSP exhibited 100% mixed

failures. Group D showed 25% and Group DSP showed 12.5% adhesive failure between alloy and composite resin. SEM photomicrographs of titanium alloy surfaces after different surface treatments are presented in (Figure 1). The topographic patterns differed between the specimens. The SEM photographs showed the control group had an irregular surface (a). The application of the sandblasting procedure with silano-pen did not change the surface topography of titanium alloy surface from the control group (b). The application of air abrasion with 30  $\mu\text{m}$  silica-coated aluminum oxide particles ( $\text{SiO}_x$ ) and application of a thin silane layer filled the surface irregularities but not changed the surface texture (c). The application of air abrasion with 30-50  $\mu\text{m}$  synthetic diamond abrasives filled the deep surface irregularities (d). The application of air abrasion with 30-50  $\mu\text{m}$  synthetic diamond abrasives and then flamed with the Silano-Pen device created a more irregular and rough surface than the synthetic diamond abrasive application alone (e).

Groups	Surface treatment	Mean $\pm$ SD
<b>C (Control)</b>	Air abrasion with 50 $\mu\text{m}$ $\text{Al}_2\text{O}_3$	14.63 $\pm$ 2.27 abc
<b>CSP</b>	Air abrasion with 50 $\mu\text{m}$ $\text{Al}_2\text{O}_3$ +Silano-Pen	16.82 $\pm$ 1.40 c
<b>S</b>	Air abrasion with 30 $\mu\text{m}$ $\text{SiO}_x$	13.83 $\pm$ 1.24 ab
<b>D</b>	Air abrasion with 30-50 $\mu\text{m}$ synthetic diamond abrasives	12.06 $\pm$ 1.77 a
<b>DSP</b>	Air abrasion with 30-50 $\mu\text{m}$ synthetic diamond abrasives+ Silano-Pen	16.45 $\pm$ 2.22 bc

Table 2: Mean (MPa) and Standard Deviation (SD) values of shear bond strength.

## Discussion

The bond strength between metal surface and esthetic restorative material (composite or porcelain) was effected by multiple factors such as thermal condition, chewing stimulus, features and characterization of esthetic material and metal [10]. This study set out to evaluate the influence of various surface conditioning methods on improving bond strength. Bonding with a composite resin and the oxide film of a metal is hydrophilic bond and this hydrophilic interface attracts water. The bond strength between alloy and composite depends on the dipole-dipole interactions, but the bond between metal and composite will be weakened through hydrolysis, and then might fail. Silicoating systems and functional monomer are used to reduce the hydrophilic character of the surface [10]. Silicoating systems cover the

metal surface with thin layer of silica by the way tribochemical or pyrolytical methods. In the present study a pyrolytical method Silano-Pen was used (Table 3).

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	122.11	4	30.53	9.12	0.001
Within Groups	117.14	35	3.35		
Total	239.26	39			

Table 3: One-way ANOVA results.

Air abrasion affects the bond strength of resins to titanium alloys, because airborne particle abrasion removes not only the impurities on the surface of titanium, but also the oxide films formed on the surface contaminated with aluminum oxides influencing the formation of the new oxide film, moreover sandblasting allows the remaining aluminum particles to chemically bond to resins [6,9,11]. Galo, et al. [9] stated that increasing of grid size of  $\text{Al}_2\text{O}_3$  (50 to 250  $\mu\text{m}$ ) particles increased shear bond strength of indirect composite resin to titanium alloy [9]. It was evaluated silicoating and a functional monomer application on the bond strength of grade 1 titanium surface showed that they were not statistically affect the shear bond strength to titanium alloy and thermal cycle procedure reduced the bond strength in all groups [10]. Similarly, in the present study silicoating did not statistically affect the shear bond strength to titanium alloy. The Mohs hardness of titanium, silicon dioxide, aluminum oxide and synthetic diamond are 6, 6.5, 9 and 10, respectively [20]. Hardness of abrasive material did not affect the bond strength [18]. The lowest shear bond strength value was obtained with group D. Sandblasting with synthetic diamond abrasive might fill the deep surface irregularities on titanium alloy surface. With the application of silicoating, lower bond strength values were obtained when compared air abraded and Silano-Pen applied surfaces. Silano-Pen application after air abrasion procedures increased shear bond strength but no significant difference was found between group C, CSP and DSP ( $p>0.05$ ). In a study which was evaluated the effect of three different surface treatment and luting agents on shear bond strength to titanium alloy showed that alkaline-heat treatment showed the highest bond strength values when compared other surface treatment [7]. In a previous study Yanagida, et al. [2] showed that higher shear bond strength values were obtained with primer and Siloc System [2]. Siloc System is based on the principle of burning of silica on a

metallic alloy. Similar to Siloc System Silano-Pen system uses heat treatment on metal surface. In this study higher shear bond strength values were obtained with group CSP and DSP than group S and group D respectively. Silane coupling with heat treatment may be effective on titanium surface to increase shear bond strength [20]. When compared traditional surface treatment methods, with this system more favorable results were obtained in adhesive prostheses, metal-composite fixed partial denture and crowns, telescopic crowns and removable partial dentures [12]. But when compared the Siloc system, silano-pen application is useful and easy handling in clinical and laboratory condition. Air abrasion with only synthetic diamond abrasive applied group showed lower bond strength values than groups with Silano-Pen was used significantly. This can be attributed to the effectiveness of heat treatment on the bond strength of titanium alloy and filler particulate composite. Also air abrasion with synthetic diamond abrasive applied groups showed only adhesive failures and other groups were mixed failures. This can be attributed to the adhesive debonding of synthetic diamond abrasives between titanium surface and composite resin.

Within the limitations on this study, the following conclusions were drawn. The effects of cyclic mechanical and thermal loading on the bond strength were not investigated in the present study. This limitation should be addressed in further studies, which should evaluate the effects of these parameters. Also alternative nano-surface treatment methods can be evaluated.

## Conclusion

Although Silano-Pen application after air abrasion with synthetic diamond abrasives effected the bond strength of titanium to composite, there was not any difference from only air abraded surface treatment. For easy handling in clinical and laboratory conditions, Silano-pen application might improve bond strength of titanium alloy and filler particulate composite.

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