

Comparison of Adhesive and Mechanical Effects on the Bond Strength of Indirect Composite to Titanium

İndirekt Kompozit ve Titanyum Arasındaki Bağlantı Dayanımı Üzerine Adeziv ve Mekanik Etkilerin Karşılaştırılması

ABSTRACT

The objective of this study was to compare the bonding properties of new dental adhesive systems to the surface of titanium for the express purpose of evaluating these systems for composite veneering of titanium restorations. Fifty-four titanium specimens (10 x 10 x 2 mm) were divided into six groups according to the surface conditioning method to be applied. One of the groups served as control and experimental groups consisted of air abrasion with aluminum oxide particles; application of dental adhesive and metal primer. Surface treatments increased the shear bond strength of indirect composite resin to titanium alloy surface ($P < 0.05$). The highest bond strength values were obtained with application of air abrasion and dental adhesive (26.84 ± 2.25) and significant differences were found between other groups ($P < 0.05$). The current study revealed that air abrasion followed by applying dental adhesive with specific functional monomers improves the bond strength of indirect composite to titanium.

Key words: Titanium, Indirect composite, Surface treatment, Shear bond strength

ÖZ

Bu çalışmanın amacı titanyum restorasyonların hızlıca veneerlenmesi için kompozitlerin kullanılabilmesi açısından yeni dental adeziv sistemlerin titanyum yüzeyine bağlanma özelliklerinin karşılaştırılmasıdır. Elli dört titanyum örnek (10 x 10 x 2 mm) yüzey hazırlama metodlarına göre altı gruba ayrıldı. Bir grup kontrol grubu olarak kullanılırken, deney grupları alüminyum oksit ile kumlama ardından, adeziv ve metal primerinin uygulanmasıyla hazırlanmıştır. Yüzey uygulamaları titanyum ve indirekt kompozit yüzeyleri arasındaki makaslama dayanımını artırmıştır ($p < 0.05$). En yüksek bağlanma dayanımı kumlama ve dental adezivin uygulamasında (26.84 ± 2.25) bulunmakla birlikte, diğer gruplar arasında da belirgin farklılıklar bulunmuştur. ($p < 0.05$). Bu çalışma kumlama sonrasında spesifik fonksiyonel monomer içeren dental adeziv uygulamasının indirekt kompozit ve titanyum arasındaki bağlanma dayanımını artırdığını göstermiştir.

Anahtar sözcükler: Titanyum, İndirekt kompozit, Yüzey uygulaması, Makaslama kuvveti

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INTRODUCTION

Ti-based alloys have received widespread attention due to their favorable mechanical properties including high specific strength, good corrosion stability, and biocompatibility after implantation (1,2). This trend can be mainly attributed to the development of casting technology for titanium alloys, such as new casting machines and investment materials and the extensively reported advantages of titanium over other base metal alloys(3-5). 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 at the high temperatures used for the porcelain fused to metal (PFM) technique(6-8). Low-fusing porcelains have been developed to minimize the difference in thermal expansion between titanium and porcelain, as well as to minimize high-temperature oxidation(6,9).

Resin composite veneering on titanium castings has been considered as an alternative for esthetic restorations. They are used in veneered crowns (partial or complete veneers), pontics for fixed partial dentures, removable partial dentures and implant prostheses(10,11). Recently introduced laboratory-processed resin composite systems attempt to resolve some of the problems inherent with dental ceramics. These new generation indirect resins have a higher density of inorganic ceramic filler than those of traditional direct and indirect composites(12). The ceramic optimized polymer (ceromer) is composed of a large number of ceramic particles. Various modes of polymerization (light polymerization, heat and pressure polymerization, argon polymerization, and vacuum polymerization) were used to improve mechanical properties such as compressive strength, bending strength, elastic modulus, abrasion resistance, and to decrease polymerization shrinkage(13,14). They use a postcuring process that results in superior flexural strength to feldspathic porcelain, minimal polymerization shrinkage, and wear rates comparable to tooth enamel(15). Also, favorable esthetics, repairability, and fast simple laboratory procedures are the advantages of these veneering materials(16).

The strong bonding between titanium and resin plays an important role in the longevity of the prosthesis. Bonding between metallic substructure and composite resin is usually obtained by macro-mechanical retentions such as undercuts, beads, loops, wires, posts and meshes. However, this process results in bulkier framework, and occurs with about 20 μm of gap on the

resin-metal interface and leads to discoloration and detachment of resin. To overcome these defects, air-abrasion, electrolytic etching technique and chemical etching technique are used to obtain a micro-roughness on the metals. However, microleakage cannot be solved completely because all the techniques are based on mechanical bonding. Using a metal primer, silicoating, heat treatment, and adhesive layer application (tin plating, silanization) are chemical bonding methods and among those, a metal primer and silicoating are clinically preferred(17). The availability of resin based adhesive primers for base metals capable of chemically bonding to casting dental alloys has simplified the procedures for surface preparation of base metal alloys(18).

Despite the improvements and the studies in bonding of composites to titanium frameworks, surface treatment effect has not been clarified. The objective of this study was to compare the bonding properties of dental adhesive systems to the surface of titanium for the express purpose of evaluating these systems for composite veneering of titanium restorations. The hypotheses to be tested were that all the surface treatments produce higher bond strength values and combined micromechanical and chemical surface treatment was the highest bond strength value. One-way analysis of variance (ANOVA) (SPSS 12.0; SPSS Inc., Chicago, Ill., USA) was planned to determine the significant differences among surface treatments and their interactions.

METHODOLOGY

The materials used in this study were presented in Table I. Titanium bars of commercially pure titanium (Grade 3, ASTM B 348, Gebze, Turkey) were sectioned and square-shaped specimens (10 \times 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 silicone 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 a flat and uniform surface and ultrasonically cleaned for 3 min with deionized water and air-dried.

Subsequently, the specimens were divided into six groups, according to the surface conditioning method to be applied (N= 54, n=9 per group). One of the groups served as control and no surface treatment was applied (Group 1). Experimental groups: air abrasion with 250

Table I: Materials used for this study

Material	Composition	Manufacturer
All-bond Universal	Ethanol, Bis-GMA.	Bisco, Inc., Schaumburg, USA
Z-prime Plus	Biphenyl dimethacrylate, MDP, Ethanol	Bisco, Inc., Schaumburg, USA
Signum Metal Bond I+II	Inorganic pigments, SiO ₂ , methylmethacrylate, acetone	Heraeus Kulzer, Hanau, Germany
Signum Composite	Bis-GMA and TEGDMA - SiO ₂ , Ba-Al-Si (1,0 µm) - 70 wt%	Heraeus Kulzer, Hanau, Germany
Titanium Bar	Grade 3 pure titanium	ASTM B 348, Gebze, Turkey

Bis-GMA: bisphenol A diglycidyl ether dimethacrylate, MDP: 10-methacryloxydecyl dhydrogen phosphate, TEGDMA: triethylene glycol dimethacrylate.

µm aluminum oxide (Al₂O₃) particles using an intraoral device (Microetcher; Danville Eng., San Ramon, California, USA) (Group 2); application of light-cured dental adhesive (All-bond Universal, Bisco, USA) and polymerized with a light-cured unit for 10 sec (Hilux LED 550; Benlioglu Dental Inc., Ankara, Turkey) (Group 3); application of a thin layer of metal primer and then dried with an air syringe for 3-5 seconds (Z-prime Plus, Bisco, USA). (Group 4); air abrasion with 250 µm aluminum oxide (Al₂O₃) particles and application of light-cured dental adhesive for 10 sec with light-cured unit (Group 5); air abrasion with 250 µm aluminum oxide (Al₂O₃) particles and application of metal primer (Group 6).

In Group 1 and 2 a thin layer metal bond (Signum Metal Bond I&II) was applied onto the all specimens surface then polymerized 90 seconds with a curing unit (Heraflash, Heraeus Kulzer, Hanau, Germany). 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 bars for 10 sec at a distance of approx. 20 mm.

After surface treatments a polytetrafluoroethylene mold (Isoflan, Diemoz, France) with a circular hole in the center (2 mm thick×3 mm in diameter) was positioned on the center of the specimens' surfaces. A thin layer of opaque paste (Signum Opaque F; Heraeus Kulzer GmbH, Hanau, Germany) was applied with a brush and polymerized for 90 s in the light-polymerizing unit (Heraflash, Heraeus Kulzer GmbH, Hanau, Germany). The composite resin (Signum Composite, DA2, Hanau, Germany) build-up was completed by applying small increments according to the manufacturer's recommendation and polymerized in the light-polymerizing unit (Heraflash) for 3 minutes. Shear bond strength of indirect composite to 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 test machine (Lloyd LRX; Lloyd Instruments PIC., Fareham, Hampshire, England) at a cross-head speed of 0.5 mm/minute where shearing load was applied parallel to the bonded interface. Force was applied to the composite resin-metal interface. The shear bond strength values were calculated in megapascal (MPa) by dividing the failure load (N) to the area of the composite resin ($N/\pi r^2$).

Data were statistically analyzed. The Kolmogorov-Smirnov test showed that the data was of a normal distribution ($P<0.05$). A homogeneity of variance test was done using Levene's test ($F: 4.926, P<0.001$). The means and standard deviations (SD) of shear bond strengths were calculated. One-way analysis of variance (ANOVA) (SPSS 12.0; SPSS Inc., Chicago, Ill., USA) was used to determine the significant differences among surface treatments and their interactions followed by a multiple comparisons' test performed using a post hoc Tukey test ($\alpha=0.05$).

To evaluate the effects of surface treatment on the surface morphology of titanium alloy, six additional titanium specimens were treated with the same experimental protocols as described previously. Then 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 ×1000 for visual inspection.

RESULTS

The results of one-way ANOVA revealed a significant difference among the groups ($P<.001$) (Table II). Surface treatments increased the shear bond strength of indirect composite resin to the titanium surface. The mean shear bond strength values and the differences among groups are shown in Table III. The highest shear bond strength

values were obtained in Group 4(26.84 MPa ±2.25) and significant differences were found among other groups ($P<.05$). Lower bond strength values were obtained in Group 1 (12,96 MPa±0.52). There were no significant differences among air abraded, bonded and air abraded, and primer applied groups ($P>.05$). Significant difference was found among primer and air abraded, and then primer applied groups ($P<.05$).

Stereomicroscopic examination of the fractured surfaces showed that failure modes of all specimens were combined adhesive and cohesive failures except for the control group and Z-prime group (100% mixed failures). Adhesive failure was observed in the control (67%) and Z-prime group (50%). (Table IV)

SEM photomicrographs of titanium alloy surfaces after different surface treatment are presented in Figure 1. The topographic patterns differed between the specimens. The SEM photographs showed the untreated titanium surface had an irregular surface (A). The application of the sandblasting procedure modified the surface topography of titanium alloy surface by increasing the irregularities on the surface (B). The application of light cure adhesive resin filled the surface irregularities (C). The application of light cure adhesive resin cement after sandblasting filled the deep surface irregularities (D). The metal primer application did not affect the titanium alloy surface. Smooth surfaces were obtained compared the other surface treatment except control surface (E). The application of metal primer after sandblasting created a

Table II: One-way ANOVA results

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1013.993	5	202.799	79.247	.000
Within Groups	122.835	48	2.559		
Total	1136.828	53			

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

Groups	Surface	Mean± SD
1	Untreated (control)	12.96±0.52 a
2	Sandblasting	17.34±0.94 bc
3	All-Bond	17.01±1.79 bc
4	Sandblasting +All-Bond	26.84±2.25d
5	Z-Prime	15.24±0.70 b
6	Sandblasting +Z-Prime	18.27±2.33 c

* Values having same letters were not significantly different for Tukey test ($P<.05$)

Table IV: Mode of failure of the groups

Groups	Surface	Mode of failure with percentage
1	Untreated (control)	Adhesive 67 %
2	Sandblasting	Combined
3	All-Bond	Combined
4	Sandblasting +All-Bond	Combined
5	Z-Prime	Adhesive 50 %
6	Sandblasting +Z-Prime	Combined

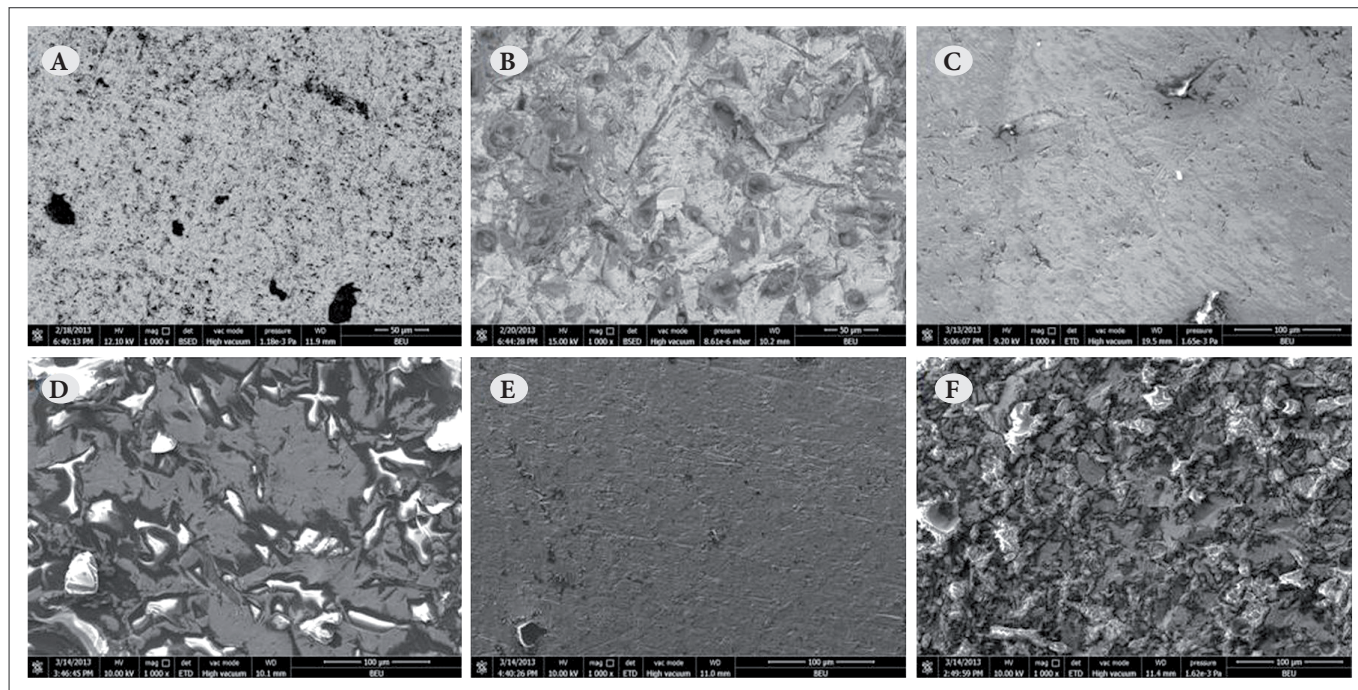


Figure 1A-F: The SEM photographs showed A) The untreated titanium surface B) The application of the sandblasting procedure C) The application of light cure adhesive resin D) The application of light cure adhesive resin cement after sandblasting filled E)The metal primer application F) The application of metal primer after sandblasting.

more irregular and rough surface than the metal primer application alone and the control surface (F).

DISCUSSION

The results of this study support acceptance of the research hypotheses that the shear bond strength of indirect composite resin to titanium alloy would be higher after application of surface treatments. Surface treatments increased the shear bond strength of indirect composite resin to titanium alloy surface ($P < 0.05$). The lowest shear bond strength values were obtained with the specimens that had no surface treatments.

The wettability of an alloy surface is an important factor for bonding between polymeric materials and metal; better wetting with metal surface conditioner and resin materials affects the interlocking between the resin and the metal surface. Both chemical and mechanical bonding techniques have been proposed to avoid a detachment of the ceromer materials from the alloys(17). According to the results of the present study for both chemical and mechanical surface conditioning methods, mean shear bond strengths to the titanium material tested were significantly different. The different groups contained specimens in which bonding was achieved either by micromechanical retention only, by chemical retention

only or by both bonding mechanisms (micromechanical retention and chemical) in the present study.

Several systems have been developed during the last two decades in order to improve the bond strength of composites to metals, including titanium. These systems involve treatment of the 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(18). The pretreatment by airborne particle abrasion of the metal surface produces micromechanical roughness that allows the resin to be mechanically joined and increases the area that adheres chemically with the resin. Furthermore, this treatment improves the wettability of the metal surface(19). Tanaka et al.(20), reported that the bond strength between composite resins and alloys was clearly improved by applying only sandblasting. El-Sherif et al.(21), reported that retainer surfaces prepared by air abrading with 250 μm aluminum oxide were superior in retention to those made by the electrochemical etching techniques.

In this study, only treatment of sandblasting with 250 μm increased shear bond strength of composite resin to titanium surface. On the other hand, it was reported

that the mechanical techniques yielded lower shear bond strength values than the chemical and mechanochemical bonding techniques(22). Airborne-particle abrasion creates surface roughness by cleaning the surface of metal oxides and increases the mechanical or chemical bond strength between metal substrates and composite material(23). In the present study, the airborne-particle abrasion significantly increased the bond strengths between the indirect composite and titanium framework.

Although air borne particle abrasion was applied, it was not enough to have a stronger shear bond strength without chemical retention. Independent variables that will affect the results of air abrasion include: particle size, air abrasion pressure, particle shape, incidence angle of the particles and wet versus dry particles(24). In a study, it was examined that the influence of eight different air pressures (0.00 to 0.40 MPa) and alumina grain size in air-abrasion (50, 90, 125 and 250 μm) on bonding between an indirect composite resin and titanium alloy. Among the air-pressure conditions employed for abrading with 50 μm alumina, the greatest bond strengths were recorded with air-pressures of 0.30, 0.35 and 0.40 MPa. and it was claimed that larged particle sized alumina may cause a reduction in the weight of alumina remaining on the titanium surface, while increasing surface roughness(25). In SEM observations in the present study, air abrasion with 250 μm alumina also caused a rougher surface. Besides air abrasion with 250 μm alumina did not affected more shear bond strength of resin to titanium alloy.

Chemical bonding minimized gap formation at the composite-metal interface(17). The chemical attachment of an opaque layer to the metal surface limited microleakage at the resin-metal interface that occurred because of polymerization shrinkage and mismatch of the coefficient of thermal expansion between the composite and the metal. However, chemical bonding is technique sensitive and a possible contamination of the interfacial adhesive layer may substantially decrease the metal-composite bond strength (26).

Metal primers are easy to apply, and saddled with other advantages of a good price performance and no need for proprietary apparatuses. They are usually supplied as single-liquid primers composed of a polymerizable monomer in a suitable solvent. These products are often called primers despite the fact that they are also coupling agents. Typically, these systems are considered as simplified chairside applications(24). In the present study, Z Prime Plus was used as a metal primer, where by it contained 10-methacryloxydecyl dihydrogen phosphate

(MDP) monomer. The surface treatment consisted of sandblasting and using a primer together is acceptable for achieving clinically high and stable bond strength to titanium alloy, surpassing the minimum level of 20 MPa for resisting masticatory forces(27). Although significant differences were found between metal primer and both sandblasting and metal primer groups, 18.27 MPa mean shear bond strength value was obtained in the present sandblasting and primer experimental group. Before the bonding procedure, the priming systems require air-abrasion with alumina to mechanically clean the surfaces and to increase the surface bonding area(28).

Several studies have shown that functional monomer systems improve the composite-to-metal bond strength with titanium and its alloys(3-5). Some studies suggested that primers containing MDP or thiophosphate monomer (MEPS) were effective for chemical bonding between resin and Ti(29-31). The choice of a chemical bonding system for prosthodontic application may depend on factors such as expense, availability, time requirement, and shelf life of the perishable components(32). The functional monomer contained in priming agents can be classified into two categories; an acidic derivative carboxylic or phosphate monomer effective for base metals(23). The phosphoric groups such as MDP have an affinity to the oxide layer generated on base metals, including chromium, nickel, aluminum, titanium, and zirconium oxide(24). Therefore, the application of priming agents containing specific phosphoric ester groups significantly enhances the bond strength of the composite material to the titanium frameworks(23). There also was a research on chemically increasing the Ti affinity using the primer, or increasing Ti bonding using silane(33,34). A light cured single-component dental adhesive that combines etching, priming and bonding in one bottle; All-Bond Universal was used in the study. The content of Bis-GMA was its difference from the other primer and bonding agents. The highest bond strength value 26.84 MPa that is significantly different from the other groups was found in the application of this adhesive after sandblasting. Signum composite which included Bis-GMA monomer was attributed to this result.

From the results shown in Figure 1 the irregular surfaces formed by airborne-particle abrasion with the chemical bonding techniques increased the adhesion between resin and titanium alloy. Adhesive failure was observed in the control and Z-prime group. This finding supports the possibility of strong adhesion by airborne-particle abrasion. Moreover, the control group showed significantly lower bond strength than the treated five

groups. Therefore, airborne-particle abrasion followed by applying a light-cured dental adhesive bonding to the titanium surface would improve the bond strength of the indirect composite to titanium assessed in the present study.

As the present study, in vitro evaluation is the first step of testing any technique or material to examine the properties and potential it possesses. It is the prospective randomized controlled trials that present the ultimate test (34). 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.

CONCLUSION

Within the limitations of the present study, it can be concluded that airborne-particle abrasion followed by applying dental adhesive with specific functional monomers improves the bond strength of indirect resin composite to titanium.

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