Effect of Different Surface Treatments of Titanium Surfaces on the Shear Bond Strength between Titanium and Zirconia Surfaces

Sukita Seekaewsiu[1], Patcharanun Chaiamornsup[2], Pornpot Jiangkongkho[2] and Yosnarong Sirimethawong[2]*

1. Department of Dentistry, Angthong Hospital, Angthong, Thailand.
2. Department of Prosthodontics, Faculty of Dentistry, Naresuan University, Phitsanulok, Thailand.

Abstract

The study evaluated the effect of different titanium surface treatments on the shear bond strength (SBS) between titanium and zirconia bonded with resin cement.

Fifty titanium disks were embedded in acrylic blocks. Titanium specimens without surface treatment were used as controls. Surface treatment methods such as etching with 37% phosphoric acid for 15 min, etching with 9% hydrofluoric acid for 15 s, treating with 50% hydrogen peroxide for 15 min, and sandblasting for 20 s with 50 μm alumina particles were assessed. Fifty zirconia disks (surface treated through sandblasting and applied with ceramic primer) were bonded with resin cement onto the titanium treated surfaces (5 groups; n = 10/group). All specimens were stored in water at 37 °C for 24 h. After 5000 cycles of thermal cycling, SBS test was performed. One-way ANOVA and Tukey HSD tests were used for statistical analysis.

The titanium samples that were sandblasted or treated with 50% hydrogen peroxide exhibited significantly higher shear bond strength compared to the rest of the specimens. Treating a titanium surface with sandblasting or 50% hydrogen peroxide increases its SBS with zirconia. Surface treatment with 50% hydrogen peroxide is simple, low cost, and does not require specialized equipment.

Experimental article (J Int Dent Med Res 2022; 15(3): 959-965)

Keywords: Surface Treatment, Titanium, Zirconia, Shear Bond Strength.

Received date: 23 April 2022 **Accept date:** 17 May 2022

Introduction

Implant abutments are components that connect to implant fixtures to support and/or retain a prosthesis. Titanium has been extensively used as an implant abutment material because of its excellent biocompatibility and high mechanical strength.[1] However, using titanium abutment for patients with a thin gingival biotype and high smile line may lead to compromises the esthetics due to an unnatural greyish appearance of the surrounding soft tissue.[2, 3] Recently, zirconia implant abutments have attracted attention because of their superior esthetic outcomes[4], reduced bacterial accumulation[5] and good mechanical stability.[6, 7]

***Corresponding author:**
Yosnarong Sirimethawong,
99 Faculty of Dentistry, Naresuan University, Tapo, Muang, Phitsanulok, 65000, Thailand.
E-mail: yosnarongs@nu.ac.th

Zirconia implant abutments can be of two types.[8] The first is the one-piece zirconia abutment, in which the entire component is made of zirconia. The one-piece zirconia abutments have high failure rates because of fractures at the implant-abutment connection[9] and abutment screw loosening.[10] The second is the two-piece zirconia abutment, which consists of a standardized titanium base and a zirconia coping abutment. The two components are bonded by a resin-based luting agent. The metal interface between metal base of two-piece zirconia abutment and metal fixture reduces fracture at connection of the zirconia abutment and wear phenomena at abutment-fixture interface.[11] Therefore, two-piece zirconia abutments can be advantageous for mechanical stability and wear resistance.[12]

However, because two-piece zirconia abutments are two different materials, interfacial debonding between the titanium base and zirconia coping abutment has been frequently observed. Particularly, when using a short titanium base in the limited interocclusal space of posterior teeth, the high risk of adhesive failure in the union between the titanium base and zirconia coping abutment has been observed.[13] Surface treatment of the materials before adhesive bonding is crucial for increasing the bonded joint strength and durability.[14] Although zirconia is an inert material that is difficult to treat the surface, several studies have found that pre-treatment zirconia surface by air-abrasion with Al_20_3 particle combined with the use of primer containing MDP can achieve superior long-term durability and increase tensile and shear bond strength between zirconia and resin cement.[15, 16] In contrast, a protocol for titanium surface treatment has not yet been established. Smielak et al.[17] reported that titanium without surface treatment bonded to zirconia using resin cement resulted in low shear bond strength between titanium and zirconia and adhesive fracture occurring between the titanium disks and the resin cement. The titanium surface seemed to be the weakest point of bonding construction. From this point, the authors focused on modifying titanium surfaces to improve the bond strength with zirconia. Recent studies have reported that surface treatment methods such as sandblasting[8, 18, 19], etching with acid[20-22], and treating with H_2O_2 solution[23-25] can enhance the bond strength of titanium with another material.

Nevertheless, a protocol for titanium surface treatment to improve bond strength with zirconia have not been elucidated. It is important to optimize the bond strength and assure for the longevity of prosthodontic restoration. Therefore, the purpose of this in vitro study was to evaluate the effect of different titanium surface treatments on the shear bond strength between titanium and zirconia by resin cement. The null hypothesis was that the different surface treatment methods would not influence the shear bond strength values at the titanium-zirconia interfaces.

Materials and methods

Specimen preparation

Titanium rods (CpTi Grade IV, Kobe steel Co., Kobe, Japan) 10 mm in diameter and 250 mm-thick were cut to 5 mm-thick disks using a linear precision saw (Isomet 5000, Buehler, Lake Bluff, Illinois, USA). Fifty titanium disks were embedded into polyvinyl chloride tubes with an auto-polymerized acrylic resin (Fast Curing Custom Tray Acrylic Resin; Instant Tray Mix, Lang Dental Manufacturing Company, Wheeling, Illinois, USA). The pre-bonding surfaces of titanium disks were polished with 240-, 400-, 800-, 1000-, and 1200-grit silicon carbide grinding papers using a grinder polisher (Phoenix Beta, Buehler, Lake Bluff, Illinois, USA) under water cooling. Then, the disks were ultrasonically cleaned in deionized water for 8 min.

Polished titanium disks were divided into five groups based on the surface treatment method used (n=10): no surface treatment (Control), etching with 37% phosphoric acid (H_3PO_4) (ActivEtch Gel, Dentalife Australia Pty Ltd) for 15 min, etching with 9% hydrofluoric acid (HF) (Ultradent Porcelain Etch, Ultradent Products Inc.) for 15 s, treating with 50% hydrogen peroxide (H_2O_2) (Hydrogen peroxide 50%, World Chemical Center) for 15 min, and sandblasting with 50 µm alumina particles for 20 s at a pressure of 0.25 MPa and a distance of 10 mm following Mehl's protocol.[26]

After titanium surface treatment, all the specimens were ultrasonically cleaned in deionized water for 8 min and dried with compressed air. The surface roughness and topography of the specimens were examined using an atomic force microscope (AFM) (Flex-axiom, Nano-surf, Liestal, Switzerland) and a scanning electron microscope (SEM) (Carl Zeiss Leo 1455VP, Jena, Germany). The mean value of roughness was analyzed at three different areas from each quadrant on the titanium surface using an AFM that represent morphological nanoscale features. The resolution of images at lateral size scanning for 50×50 μm^2. The roughness was calculated by using the Nanosurf analysis tool (C3000 control software version 3.10.0, Nano-surf, Liestal, Switzerland). All titanium specimens were selected with a surface roughness value of 165-175 nm. to control the initial surface roughness before surface treatment.

Zirconia blocks (Zirlux 16$^+$, Henry Schein Inc., Melville, New York, USA) were milled and sintered to a diameter and a thickness of 5 mm by a dental technician. The pre-bonding surfaces of the 50 zirconia disks were polished and cleaned using the same procedure as that used for the titanium disks. The bonding surfaces of all zirconia disks were surface treated by sandblasting with 50 µm alumina particles for 20 s at a pressure of 0.25 MPa and a distance of 10 mm. Then, the zirconia disks were ultrasonically cleaned in deionized water for 8 min. Ceramic primer (Clearfil Ceramic Primer Plus, Kuraray Noritake Dental Inc., Kurashiki, Okayama, Japan) was applied to the disks, and the disks were blow dried before cementation.

Bonding procedure

Titanium disks were bonded to zirconia disks with dual cure resin cement (Panavia V5, Kuraray Noritake Dental Inc.) and seated with a constant force of 1 kg applied by a specimen preparation jig (S4660A, Instron, Norwood, Massachusetts, USA). The excess cement was removed with a microbrush, and the specimen borders were light polymerized for 20 s with a dental polymerizing light (S.P.E.C. 3, Coltene Whaledent Inc., Cuyahoga Falls, Ohio, USA; 1600 mW/cm^2) placed at a distance of 10 mm to initiate polymerization and left for 5 min for definitive polymerization. All specimens were stored in water at 37 °C for 24 h. Subsequently, all specimens were aged by thermocycling for 5000 cycles at 5° C and 55 °C with a dwell time of 15 s.[27]

Shear bond strength test

The specimens were tested for shear bond strength (Figure. 1A) using a universal test machine (Model 8872, Instron, Norwood, Massachusetts, USA) with a 10 kN load cell. The shear load was applied at the resin cement-titanium interface (Figure. 1B).

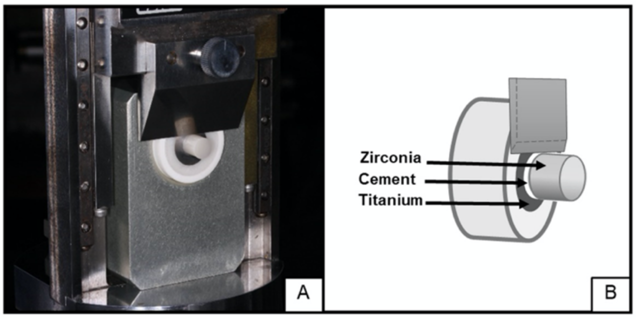

Figure 1. (A) Shear bond strength testing apparatus; (B) Schematic illustration of shear load.

A chisel shearing rod running at a crosshead speed of 1 mm/min were used to load on the specimens until fracture. Maximum forces were recorded and the shear bond strength was calculated from the following formula: Rt = F/S, where Rt is the shear bond strength (MPa), *F* is the maximum force acting on the specimen (N), and *S* is the surface area of the specimen (mm^2). Separated disks were viewed under a stereomicroscope (Olympus SZX16, Spacemed, Tokyo, Japan) at ×20 magnification to determine the mode of failure. Representative specimens were analyzed by SEM to determine the nature of the fractures formed during the shearing procedure.

Statistical analysis

The mean values and standard deviations of surface roughness and shear bond strength were calculated for each group. The data were analyzed using a statistical analysis software (SPSS version.22, IBM, Armonk, NY, USA). Statistical significance was analyzed using a one-way ANOVA, and the differences between the groups were determined using Tukey HSD post hoc test (α=0.05).

Results

The means ± standard deviations (SD) bond strength values range from 3.64 ± 2.11 MPa (control) to 36.01 ± 2.80 MPa (sandblast), as shown in Figure 2.

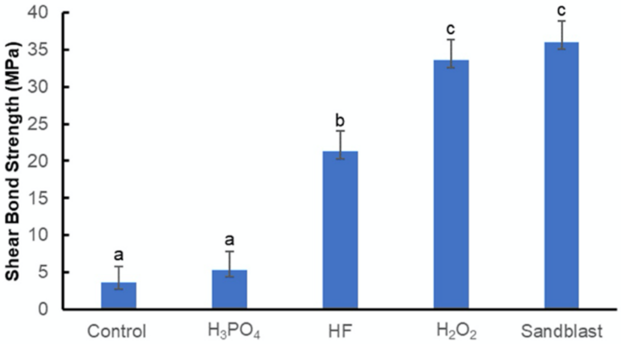

Figure 2. Mean and standard deviation values of shear bond strength. Different superscripts indicate significant differences among groups ($p<0.05$).

Statistical analysis revealed a significant influence of titanium surface treatment on the shear bond strength ($p<0.05$). The Tukey HSD test showed that the titanium surfaces treated with sandblasting (36.01 ± 2.80 MPa) and with H_2O_2 (33.57 ± 2.74 MPa) exhibited significantly higher shear bond strengths compared to other groups ($p< 0.05$), while the group that underwent surface treatment with HF (21.27 ± 2.76 MPa) exhibited a significantly higher shear bond strength than the control group (3.64 ± 2.11 MPa) ($p<0.05$). In contrast, the group surface treated with H_3PO_4 (5.29 ± 2.50 MPa) showed no significant difference compared to the control group (3.64 ± 2.11 MPa) ($p>0.05$).

The means ± SD values of the titanium surface roughness before and after surface treatment are shown in Figure 3. The titanium surface roughness values of the control, H_3PO_4-treated, and H_2O_2-treated groups were significantly lower than those of the HF and sandblast groups ($p<0.05$). The group that was sandblasted showed the highest surface roughness value among all groups ($p<0.05$).

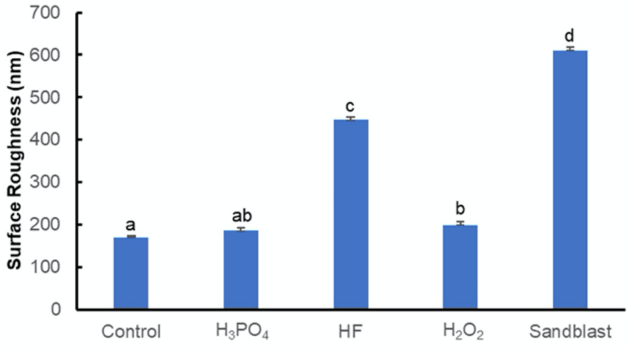

Figure 3. Mean and standard deviation values of surface roughness. Different superscripts indicate significant differences among groups ($p<0.05$).

The AFM and SEM images of the control group revealed a macroscopically and microscopically smooth surface, with evident minor parallel grooves, which were attributed to the polishing process (Figures 4A and 4B). The H_3PO_4- and H_2O_2-treated groups showed mean values of roughness similar to those of the control group. The H_3PO_4- and H_2O_2-treated disks exhibited slight alterations in the surface topography. As a result, the parallel grooves on the titanium surface became less prominent (Figures 4C, 4D, 4G, and 4H). For the groups that underwent surface treatment with sandblasting and with HF, the mean roughness values were significantly higher than those of the other groups, and a significant difference between the aforementioned two groups was observed ($p<0.05$). The HF-treated disks showed several peaks on a flat surface and protruding particles, which appeared as small and large irregular shapes with round edges (Figures 4E and 4F). The sandblasted disks revealed numerous irregular cavities, cracks, and sharp edges, which differed from the other groups (Figures 4I and 4J).

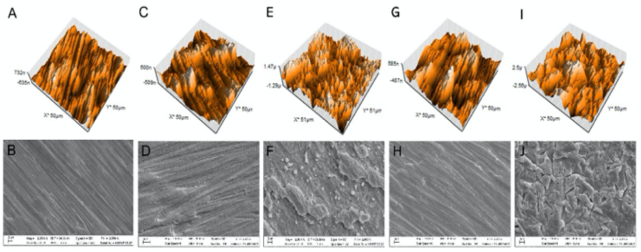

Figure 4. AFM (scanning area: 50×50 μm^2) (upper row) and SEM images (magnification ×2000) (lower row) for control group (A and B), H_3PO_4-treated group (C and D), HF-treated group (E and F), H_2O_2-treated group (G and H), and sandblast-treated group (I and J).

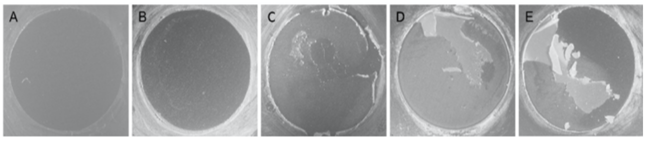

Figure 5. Stereomicroscope images (magnification ×20) of fractured titanium surfaces of the control group (A), H_3PO_4-treated group (B), HF-treated group (C), H_2O_2-treated group (D), and sandblast-treated group (E).

The distribution of fracture patterns is summarized in Table 1. The fracture pattern analysis showed that the control and H_3PO_4-treated groups demonstrated only the adhesive fracture pattern between the titanium disks and resin cement (Figure 5). In these two groups, no resin cement remnants were found on the titanium surfaces; however, all the resin cement layers were found on the zirconia surface. The groups with surfaces treated with HF, H_2O_2, and sandblasting have mixed fracture patterns, and more residual cement tended to remain on the zirconia surfaces than on the titanium surfaces.

Group	Adhesive fracture between titanium surface and resin cement	Mixed fracture pattern
Control	10	-
H_3PO_4	10	-
HF	6	4
H_2O_2	3	7
Sandblast	3	7

Table 1. Fracture pattern distribution.

Discussion

The use of a two-piece zirconia abutment as a prosthetic superstructure for dental implants requires an effective bond between the zirconia coping and titanium base. The bonding between these two materials is a key factor for the long-term success of the abutment. The results of the present study reject the null hypothesis, which considers that different surface treatments of titanium surfaces do not influence the shear bond strength between the titanium and zirconia surfaces. The surface treatment methods significantly affected the shear bond strength between the titanium and zirconia surfaces ($p<0.05$) as revealed by the analysis results.

In this study, the zirconia surface treatment method included sandblasting as per Mehl's protocol[26] and the application of a primer containing an adhesive phosphate monomer (MDP) as per manufacturer instructions. Several studies have shown that when an MDP primer is added to sandblasted zirconia surfaces[8], the resin cement retention increases. Furthermore, superior long-term tensile and shear bond strengths between zirconia and resin cement can be achieved.[28]

This study on the effect of surface treatment on the shear bond strength of the titanium and zirconia interface observed adhesive failure in the titanium-resin cement interface of the control group. A similar failure was observed by Maltzahn et al.[19], who reported that titanium without any mechanical surface treatment resulted in a low bonding strength between the titanium and ceramic materials. Therefore, the present study focused on titanium surface modification.

The surface treatment of titanium is beneficial for increasing the bond strength at the titanium-resin cement interface.[8, 20, 23] Sandblasting is a favorable method for titanium surface modification to improve its bonding with other materials.[25] Other methods such as etching with acid or treating with solution have been reported to improve the bond strength between titanium and resin cement.[20, 24] A previous study reported that etching titanium abutments with 9% HF for 15 s enhances the retention of restorative materials.[21] A pilot study showed that titanium surface treatment with 37% H_3PO_4 for 15 min resulted in higher bond strength values than surface treatment with 37% H_3PO_4 for 5 min, which was the duration considered in the previous study for titanium pre-treatment.[21] Another pilot study also demonstrated that the surface treatment of titanium with 50% H_2O_2 for 15 min had higher bond strength values with resin cement when compared to treatments with 30% H_2O_2 for 5 and 10 min.[24] Therefore, titanium surface treatment in this study was performed with sandblasting, etching with 9% HF 15 s, etching with 37% H_3PO_4 15 min, and treating with 50% H_2O_2 15 min to evaluate the effect of micromechanical surface treatments. The results of this study showed that the shear bond strength of the titanium surfaces increased by sandblasting, H_2O_2, and HF surface treatment methods. The increased shear bond strength of the sandblast- and HF-treated groups correlated with the increase in surface roughness, while the increased shear bond strength of the H_2O_2-treated group was not correlated with the surface roughness.

The sandblast- (36.01 ± 2.80 MPa) and HF-treated (21.27 ± 2.76 MPa) groups presented high shear bond strengths, resulting from increased surface roughness, which enhanced the bonding surface area. The cement could penetrate and form a micromechanical interlock with the titanium surface. This is consistent with the results of Gehrke et al.[8], who found that the sandblast method increases the surface roughness and improves the retention between the components of two-part abutments. However, excessive pressure or prolonged sandblasting resulted in a drastic change in the titanium surface.[18] Moreover, during the HF etching process, the etchant was agitated to enhance the contact area of the chemical reaction.[20] This observation is in agreement with a previous study, which reported that the HF etching effect resulted in micromechanical retention on the titanium surface and improved the bond strength between titanium and restorative materials.[21]

The titanium surface treated by immersing in H_2O_2 (33.57 ± 2.74 MPa) had a high shear bond strength and was not significantly different from the sandblast-treated group; however, the H_2O_2-treated group had a lower surface roughness than the sandblast- and HF-treated groups. The results of this study showed that the increased shear bond strength for H_2O_2-treated titanium surface is not related to the surface roughness or to micromechanical bonding. The high shear bond strength of the H_2O_2-treated group may be due to the increased oxide layer thickness, which was because of increased titanium oxidation; this was supported by the coloration changes in H_2O_2 treated disks. In agreement with a previous report, when titanium was immersed in H_2O_2, the titanium surface was oxidized to improve the resin bond strengths.[23] The oxidation mechanism was presumed to follow the Fenton reaction, forming hydroxyl radicals.[23] Therefore, surface treatment with H_2O_2 solution may have altered the natural composition of the titanium surfaces and produced active hydroxyl groups on the surface oxide film, thereby resulting in the increased bond strength of the resin cement to titanium through a chemical adhesion mechanism. However, Yoshida et al.[23] reported that prolonged H_2O_2 treatment of titanium decreased the bond strength due to the excessive thickness of the surface oxide film formed. Therefore, immersion of titanium in H_2O_2 solution is a promising surface treatment method; however, further studies are required to better understand the mechanism by which H_2O_2 enhances the bond strength.

The H_3PO_4-treated group had low shear bond strength (5.29 ± 2.50 MPa) and low surface roughness, which are not significantly different from that of the control group ($p>0.05$). Tsuchimoto et al.[22] reported that H_3PO_4 is strongly adsorbed on the titanium surface after the surface treatment. As a result, H_3PO_4 potentially inhibits the subsequent adsorption of the phosphoric groups of MDP on titanium surfaces. Therefore, the results of this study indicate that the titanium surface should not be pretreated with H_3PO_4, which would otherwise significantly decrease the bond strength of the resin cement to titanium restoration.

The analysis of fracture patterns and identification of the failure mode play a significant role in determining weak points resulting in bond failure. This study showed that the failure modes in the HF-, H_2O_2-, and sandblast-treated groups were mainly mixed fractures with residual cement on both the titanium and zirconia surfaces, thereby indicating adequate bonding on each surface. In a similar report, Maltzahn et al.[19] reported the occurrence of mixed fractures between titanium and zirconia, which were surface treated by the sandblasting method. However, the control group (without treatment) and H_3PO_4-treated group showed the lowest shear bond strength values with adhesive fractures, thereby indicating the presence of cement remnants on the zirconia surface. The fracture pattern analysis indicated that the bond between the titanium surface and resin cement could be the weak point. This is likely because the titanium surface is not modified or H_3PO_4 is not suitable for titanium surface treatment. In addition, the MDP primer was used only on the zirconia surface. This made the resin cement adhere to zirconia better than to titanium. A previous study reported that more cement remnants were found on the zirconia abutment when the MDP primer was applied to zirconia.[19]

The results of this study reveal that surface treatment with 50% H_2O_2 for 15 min yielded a high shear bond strength, which was close to that obtained using the sandblasting method. The clinical implications are that treatment with 50% H_2O_2 for 15 min does not require specialized equipment, is more controllable, and is easier than sandblasting. This technique may be a useful surface treatment alternative for clinicians to improve the bond strength between the titanium base and zirconia coping of two-piece zirconia abutments.

The limitation of this study is that the geometry used to measure the shear bond strength was different from that of clinically used abutments. The specimen geometry was selected to be appropriate for the study methodology, which required the specimens to be mounted on the universal test machine. Further studies should be conducted with clinical abutments to represent the realistic geometry of dental abutments. Moreover, the chemical interactions resulting from titanium surface treatment, which could increase the bond strength between titanium and zirconia surfaces, should be further investigated. In this in vitro study, the clinical conditions in the oral cavity, such as dynamic fatigue loading, pH fluctuation, and long-term aging, could not be simulated. Clinical application protocols should be confirmed in a clinical study.

Conclusions

Based on the results and limitations of this in vitro study, it can be concluded that the titanium surface treatment methods before bonding have a significant influence on the shear bond strength between the titanium and zirconia surfaces. The titanium surfaces treated with sandblasting or 50% H_2O_2 had the highest shear bond strength compared to other groups. Although sandblasting is an effective method for titanium surface treatment, immersion of titanium in H_2O_2 can be an alternative surface treatment method due to its simplicity and high shear bond strength value.

Declaration of Interest

The authors report no conflict of interest.

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