©Operative Dentistry, 2020, **45-4**, 426-434

Effect of Various Surface Treatments on Ti-Base Coping Retention

K Kemarly • SC Arnason • A Parke • W Lien • KS Vandewalle

Clinical Relevance

Mechanical surface roughening of the titanium-abutment base is necessary to increase the pull-off bond strength of the lithium disilicate abutment material. Additional chemical surface treatment may further increase the bond strength, but the effects are product specific.

SUMMARY

Objective: The titanium-cement interface of a Ti-Base implant crown must be able to resist intraoral pull-off forces. The purpose of this study was to evaluate the effect of mechanical and chemical surface treatments of a titanium-abutment base (Ti-Base, Dentsply/Sirona) on the pull-off bond strength of a lithium disilicate abutment coping.

Methods and Materials: Ti-Bases were divided into nine groups of 10 copings each that varied in both mechanical surface treatment (none; Al_2O_3 air abrasion; CoJet silicoating, 3M ESPE) and chemical treatments (none; Monobond Plus, Ivoclar Vivadent; Alloy Primer, Kuraray). Lithium disilicate abutment copings (IPS e.max CAD, Ivoclar Vivadent) were designed and milled. After crystallization, the copings were cemented onto the Ti-Bases with a resin cement (MultiLink Hybrid-Abutment Cement, Ivoclar Vivadent) according to the manufacturer's recommendations. The copings were torqued to a mounted implant, and the access channel was sealed with composite. After 24-hour storage and 2000 thermal-cycles in distilled water, the copings were subjected to a removal force parallel to the long axis of the interface until fracture. Data were analyzed with multiple one-way analyses of variance and Tukey post hoc tests (α=0.05).

Results: Significant differences were found between groups based on type of surface treatment ($p<0.05$).

Conclusions: Chemical surface treatment with Monobond Plus and mechanical surface treatment with CoJet silicoating or Al_2O_3 air abrasion resulted in the greatest pull-off bond strength. Alloy Primer did not provide a statistically significant increased pull-off bond strength when the surfaces were mechanically treated with Al_2O_3 air abrasion or CoJet silicoating. The lack of any mechanical surface treatment resulted in the lowest pull-off bond

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https://doi.org/10.2341/19-155-LR

strength regardless of the type of chemical surface treatment.

INTRODUCTION

The advent of computer-aided design/computer-aided manufacturing (CAD/CAM) dentistry has yielded several advantages over traditional dental workflows. Narrowing the focus on implant-supported prostheses, the Ti-Base and ScanPost system from Dentsply/Sirona (Charlotte, NC, USA) have made possible the fabrication of a ceramic implant abutment and prosthesis without the need for a traditional impression and cast.[1] Joda and Bragger[2] showed that this workflow decreased the cost to patients by 30% and halved the laboratory workload.

CAD/CAM technology is compatible with multiple restorative materials. For the posterior region, high occlusal forces have required materials with high fracture strengths, and zirconia has been traditionally shown as an effective material for abutment and restoration of implants in this critical region.[3-5] Zirconia also features high biocompatibility as well as more acceptable esthetics compared with metallic restorations.[3]

Recently, Ivoclar Vivadent has introduced a lithium disilicate CAD/CAM block marketed for use with the Ti-Base system. Lithium disilicate does not share a similar strength with zirconia; however, Elsayed and others[4] concluded that lithium disilicate abutments and hybrid-abutment crowns displayed sufficient strength during dynamic loading, lasting more than 1.2 million fatigue load cycles with "higher forces than physiological occlusal forces."[4] More esthetic restorations are possible with lithium disilicate in comparison with zirconia because of the higher degree of translucency. Also, lithium disilicate can be crystallized in the oven with a significantly shorter heating cycle, making it a very attractive restorative choice in terms of workflow.[6] Lithium disilicate also has the advantage of a more predictable adhesive bond than zirconia. The silica nature of the lithium disilicate allows for surface etching with hydrofluoric acid prior to silanization. The polycrystalline nature of zirconia may require other methods of surface treatment that may rely more on mechanical rather than chemical retention.[7]

Studies have investigated the effect of different mechanical and chemical surface treatments on titanium but not specifically the Ti-Base implant abutment base. The mechanical surface treatments commonly include air abrasion with Al_2O_3 and tribochemical silica coating. Air abrasion increases retention by roughening the titanium surface. Ebert and others[8] showed that air abrasion significantly increased bonding between zirconia copings and titanium compared with the control. Von Maltzahn and others[9] also investigated the mechanical surface treatment of titanium but included tribochemical coating. Tribochemical coating serves to embed silica particles into the surface of a material via high-speed impact from an air abrasion unit. In that study, it was found that the tribochemical surface treatment was less retentive than air abrasion with Al_2O_3.[9]

In addition to mechanical surface treatments, chemical surface treatments also exist for modifying titanium. According to the manufacturer, Monobond Plus (Ivoclar Vivadent, Amherst, NY, USA) and Alloy Primer (Kuraray, Houston, TX, USA) increase the bond strength to metals, with the use of Alloy Primer specifically mentioned for titanium. Both use functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) to promote chemical bonding between the metal and the cement.[10] Specifically, Monobond Plus contains ethanol, trimethylpropyl methacrylate (silane), methacrylated phosphoric acid ester (10-MDP), and disulfide acrylate. Alloy Primer contains methacrylated phosphoric acid ester (10-MDP) as well as 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol (VBATDT) in acetone. Little research has been done studying the bond strength of Monobond Plus to titanium, but Veljee and others[10] showed that the addition of Alloy Primer increased the retention of a resin cement to pure titanium to a statistically significant level. They postulated that the Alloy Primer promotes wettability, thus increasing the adhesive bonding. Yanagida and others[11] also found that using only Alloy Primer combined with a resin cement showed significantly higher bond strength to pure titanium compared with air abrasion or tribochemical modification alone.

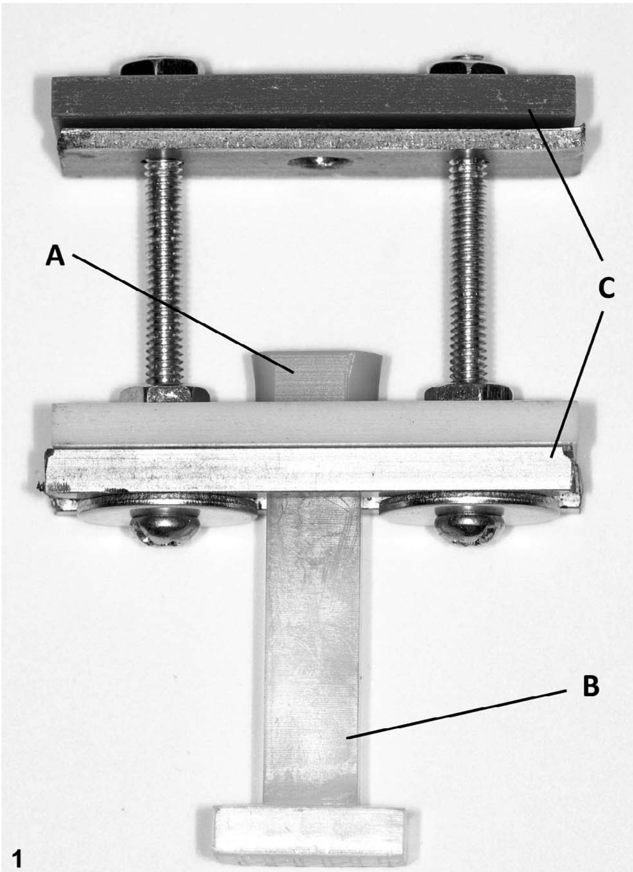
While research has shown that there is potential for differences in surface treatment in relation to the bond strength of titanium to resin cement, no research has evaluated the pull-off bond strength between the lithium disilicate abutment material and the titanium implant abutment base.[12] It is important to note that the Ti-Base is a medical grade 5 titanium aluminum alloy, which might behave differently than the pure titanium used in aforementioned studies. It is important to ensure that the restorations placed on the Ti-Bases are retentive and able to serve the patient in the long term. The purpose of this study was to evaluate several surface treatments in differing combinations and their effect on the pull-off bond strength of lithium disilicate to the Ti-Base implant abutment base. The null hypothesis tested was that there would be no difference in pull-off bond strengths of the lithium disilicate copings from the Ti-Base regardless of surface treatment modality.

METHODS AND MATERIALS

A custom coping was designed in SolidWorks CAD three-dimensional (3D) software (Dassault Systemes, Vélizy-Villacoublay, France). In addition, a custom cradle was designed to adapt an existing vice grip of the universal testing machine (model 5543, Instron, Norwood, MA, USA) to fit intimately with the coping to allow for even distribution of pull-off forces without possible fracturing due to compression of the lithium disilicate from the vice clamps. The cradles were 3D printed (Objet 260 Dental Selection, Stratasys Ltd, Eden Prarie, MN, USA). The coping was milled in lithium disilicate (IPS e.max CAD abutment, LT, shade A2, Ivoclar Vivadent) on a five-axis milling unit (CORiTEC 450i, imes-icore GmbH, Eiterfeld, Germany) and placed on an implant lab analog (Certain 4.1 mm, Biomet 3i, Palm Beach Gardens, FL, USA) and 3D scanned into the InLab software (v16.0, Dentsply/Sirona). Ninety copings were milled from the IPS e.max CAD abutment using a milling unit (MCXL, Dentsply/Sirona). The lithium disilicate copings were crystalized in a ceramic oven (Programat P500, Ivoclar Vivadent) following the manufacturer's instructions. To properly hold the implants, a custom base was designed in Solid Works with a channel. Holding towers were 3D printed (SLA Viper si2, 3D Systems, Rock Hill, SC, USA), and an implant (Certain 4.1 mm, Biomet 3i) was threaded into each. Each "implant tower" was analyzed to ensure that the implant was placed parallel to the long access of the tower to ensure pull-off forces would also be parallel.

In preparation for cementation, the titanium bases (Ti-Base, BC 4.1L, Dentsply/Sirona) were temporarily held in an implant lab analog (Certain 4.1 mm, Biomet 3i). Ninety Ti-Bases were divided into three groups of 30 each. Thirty of the Ti-Bases received no surface treatment. Thirty were air abraded (Basic Quattro IS, Renfert, Chicago, IL, USA) using 50 μm Al_2O_3 at 2.0 bar and then steam cleaned (i700B, Reliable, Toronto, Ontario, Canada). The remaining 30 Ti-Bases were treated with tribochemical silica coating (CoJet Sand, 3M ESPE, St Paul, MN, USA) at 2.0 bar for 15 seconds until the metal turned a uniformly dark color per the manufacturer's recommendation and steam cleaned. In each of the three groups of 30 Ti-Bases, 10 were primed with Monobond Plus primer applied to the Ti-Base bonding surface, allowed to react for 60 seconds, and gently blown dry with a three-way syringe. Ten were treated with Alloy Primer with a cotton pellet and left to dry per manufacturer recommendations. And the remaining 10 received no chemical treatment.

The intaglio surface of the custom lithium disilicate coping was etched for 20 seconds with hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) and rinsed thoroughly with water from a three-way syringe. Monobond Plus primer was applied to the etched surfaces and allowed to react for 60 seconds and gently blown dry with a three-way syringe. The specimens were cemented to the Ti-Base using an autopolymerizing resin cement (MultiLink Hybrid Abutment Cement, Ivoclar Vivadent) according to the manufacturer's recommendations. Glycerin gel was applied to the cementation interface for seven minutes and then rinsed off with a three-way syringe. During setting of the cement, the specimens were set in a custom-made jig that allowed for placement of a 100-g weight onto the specimen to ensure standardized pressure during setting of the cement. After removal of the glycerin gel, the cement interface was polished to mimic actual clinical procedures. Next, the Ti-Base specimens were torqued to the implant in the experimental apparatus at 20 N/cm. Clearfil SE Bond (Kuraray) was applied to the screw channel and light cured (Bluephase G2, Ivoclar Vivadent). Irradiance was recorded with a power meter (Powermax, Coherent Inc, Santa Clara, CA, USA) and considered acceptable since it was greater than 1000 mW/cm^2. Filtek Z250 (3M ESPE) was placed incrementally and light cured. The cameo surface of the composite was polished with Enhance and Pogo polishing tips (Dentsply). The assembled specimens (Figure 1) were then placed in distilled water and stored in an incubator (model 20 GC, Quincy Labs, Chicago, IL, USA) for 24 hours at 37°C. Then, the specimens were thermal-cycled in distilled water for 2000 cycles at 5°C and 55°C with a dwell time of 30 seconds at each temperature (Sabri Dental Enterprise, Downers Grove, IL, USA). Each specimen was then loaded under tension in a universal testing machine (Instron) with a pair of customized vice jig assemblies holding the lithium disilicate restoration on one side and the 3D-printed resin tower in the other (see Figure 2). The universal testing machine subjected the lithium disilicate copings to a removal force parallel to the long axis of



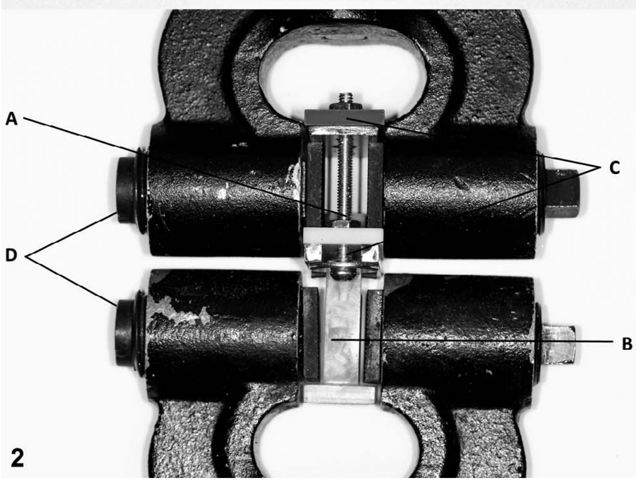


Figure 1. *Cemented specimen (A) loaded onto an implant threaded into a 3D-printed tower (B). The specimen is cradled by a custom adapter (C) designed to fit into the universal testing machine.*

Figure 2. *Cemented specimen (A) loaded onto an implant and threaded into a 3D-printed tower (B). The specimen is cradled by a custom adapter (C) fitted into the grips of the universal testing machine (D).*

the interface at a speed of 1 mm/min until the copings fractured or separated from the Ti-Bases. The maximum force between components was recorded in newtons.

Once the pull-off tests were complete, the fractured surfaces of all specimens were analyzed using a stereomicroscope at 10× magnification (SMZ-1B, Nikon, Melville, NY, USA). The fractured surfaces were evaluated and classified into the following failure modes: cement remaining on Ti-Base only, cement remaining on lithium disilicate only, cement remaining on both Ti-Base and lithium disilicate, fractured lithium disilicate with no cement remaining on Ti-Base, fractured lithium disilicate with some cement remaining on Ti-Base, and fractured lithium disilicate with a portion of lithium disilicate still bonded to the Ti-Base. The surface roughness of nine Ti-Bases was analyzed after mechanical modification (three per group). Surface roughness (Ra) was measured using a noncontact profilometer (3D Laser-Scanning Confocal Profilometer, Keyence, Itasca, IL, USA) and then analyzed using its proprietary software. The morphology of the Ti-Base surfaces was investigated by field-emission scanning electron microscopy (Sigma VP, Carl Zeiss, Oberkochen, Germany). The elemental composition of the Ti-Base surfaces was characterized by energy-dispersive spectroscopy (X-Max, Oxford Instruments, Abingdon, UK).

A mean removal force (N) and standard deviation were determined for each of the nine groups. Data were analyzed using a two-way analysis of variance (ANOVA) to evaluate the effect of mechanical (three levels) or chemical treatments (three levels) of the Ti-Base surface on the pull-off strength of the lithium disilicate specimens ($\alpha=0.05$).

RESULTS

The results of the two-way ANOVA found significant differences between groups based on mechanical surface treatments ($p<0.001$) and chemical surface treatment, but also there were significant interactions ($p<0.001$). The data were further evaluated by multiple one-way ANOVAs per mechanical or chemical surface treatment (see Table 1).

Chemical surface treatment with Monobond Plus and mechanical surface treatment with Al_2O_3 air abrasion (896.0±173.1 N) or CoJet silicoating (1011.5±120.2 N) resulted in the greatest pull-off bond strengths, but they were not significantly different from each other. Both groups were significantly greater than Al_2O_3 air abrasion (650.3±54.7

Table 1: *Pull-off Bond Strength of Lithium Disilicate Abutment Copings After Various Mechanical and Chemical Surface Treatments of the Titanium-abutment Base* [a]

Mechanical Surface Treatment	Pull-off Bond Strength (SD), Newtons		
	Chemical Surface Treatment		
	Monobond Plus	Alloy Primer	None
Al_2O_3 air abrasion	896.0 (173.1) Aa	759.5 (127.1) ABa	650.3 (54.7) Ba
CoJet silicoating	1011.5 (120.2) Aa	491.1 (102.3) Bb	501.8 (49.0) Bb
None	340.9 (95.5) Ab	332.4 (85.4) Ac	393.1 (65.3) Ac

[a] *Groups with the same uppercase letter per row or lowercase letter per column are not significantly different* ($p > 0.05$).

N) or CoJet silicoating (501.8±49.0 N) without any chemical treatment.

Chemical surface treatment with Alloy Primer and mechanical surface treatment with Al_2O_3 air abrasion (759.5±127.1 N) did not provide a significant increase in pull-off bond strength compared with no primer (650.3±54.7 N). Similarly, treatment with Alloy Primer and CoJet silicoating (491.1±102.3 N) did not provide a significant increase in pull-off bond strength compared with no primer (501.8±49.0 N). The lack of any mechanical surface treatment resulted in the lowest pull-off bond strength regardless of the type of chemical treatment. No mechanical treatment and Monobond Plus (340.9±95.5 N) was not significantly different from Alloy Primer (332.4±85.4 N) or no primer (393.1±65.3 N).

The most frequently observed failure mode for each group was as follows: Monobond Plus/Al_2O_3 abrasion, 100% had fractured lithium disilicate with a fragment of the lithium disilicate firmly bonded to the Ti-Base (see Figure 3); Monobond Plus/CoJet silicoating, 100% had fractured lithium disilicate with a fragment of the lithium disilicate firmly bonded to the Ti-Base; Monobond Plus/no mechanical, cement on both the lithium disilicate and the Ti-Base; Alloy Primer/Al_2O_3 abrasion, fractured lithium disilicate with a fragment of the lithium disilicate firmly bonded to the Ti-Base; Alloy Primer/CoJet silicoating, fractured lithium disilicate with a fragment of the lithium disilicate firmly bonded to the Ti-Base. Alloy Primer/no mechanical, fractured lithium disilicate with some cement left on the Ti-Base; no chemical/Al_2O_3 abrasion, cement on both the lithium disilicate and the Ti-Base; no chemical/CoJet silicoating, cement on both the lithium disilicate and the Ti-Base; no chemical/no mechanical, fractured lithium disilicate with no cement left on the Ti-Base. See Figure 4.

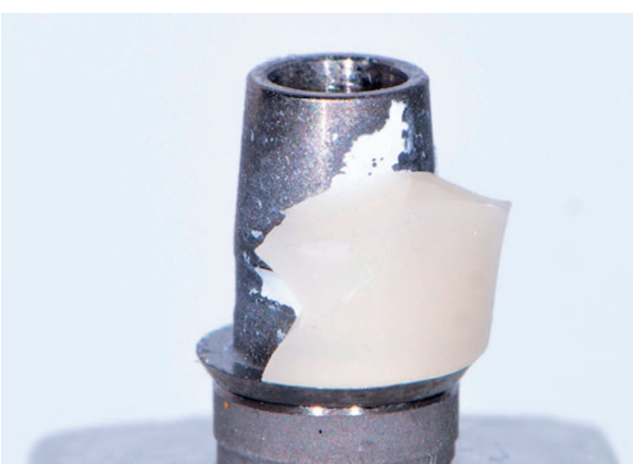


Figure 3. *Fragment of lithium disilicate bonded to the cervical area of the Ti-Base with cement remaining.*

In comparing surface roughness, Al_2O_3 abrasion gave an overall rougher surface (0.925±0.124 μm) compared with CoJet silicoating (0.555±0.000 μm) and control (0.297±0.040 μm). Evaluation of surface composition showed that the CoJet silicoating samples did in fact contain a higher surface composition of Si by weight (5.73%) compared with the Al_2O_3 abrasion (0.25%) or control (0.23%). Both the Al_2O_3 and CoJet silicoated Ti-Bases had less available Ti than the untreated Ti-Bases (35.53% and 27.29% compared with 70.9%). Scanning electron micrograph photos of the treated Ti-Base surfaces can be seen in Figures 5-7.

DISCUSSION

The purpose of this study was to evaluate several surface treatments in differing combinations and their effect on the pull-off bond strength of lithium disilicate to the Ti-Base implant abutment base. Within the limitations of this laboratory study, the null hypothesis was rejected because the results of the study found statistically significant differences in pull-off bond strengths of the lithium disilicate copings from the Ti-Base dependent on surface treatment modalities. Based on the results, it would appear that the most important factor in bonding to the Ti-Base is the use of some form of mechanical treatment. Without any mechanical surface treat-

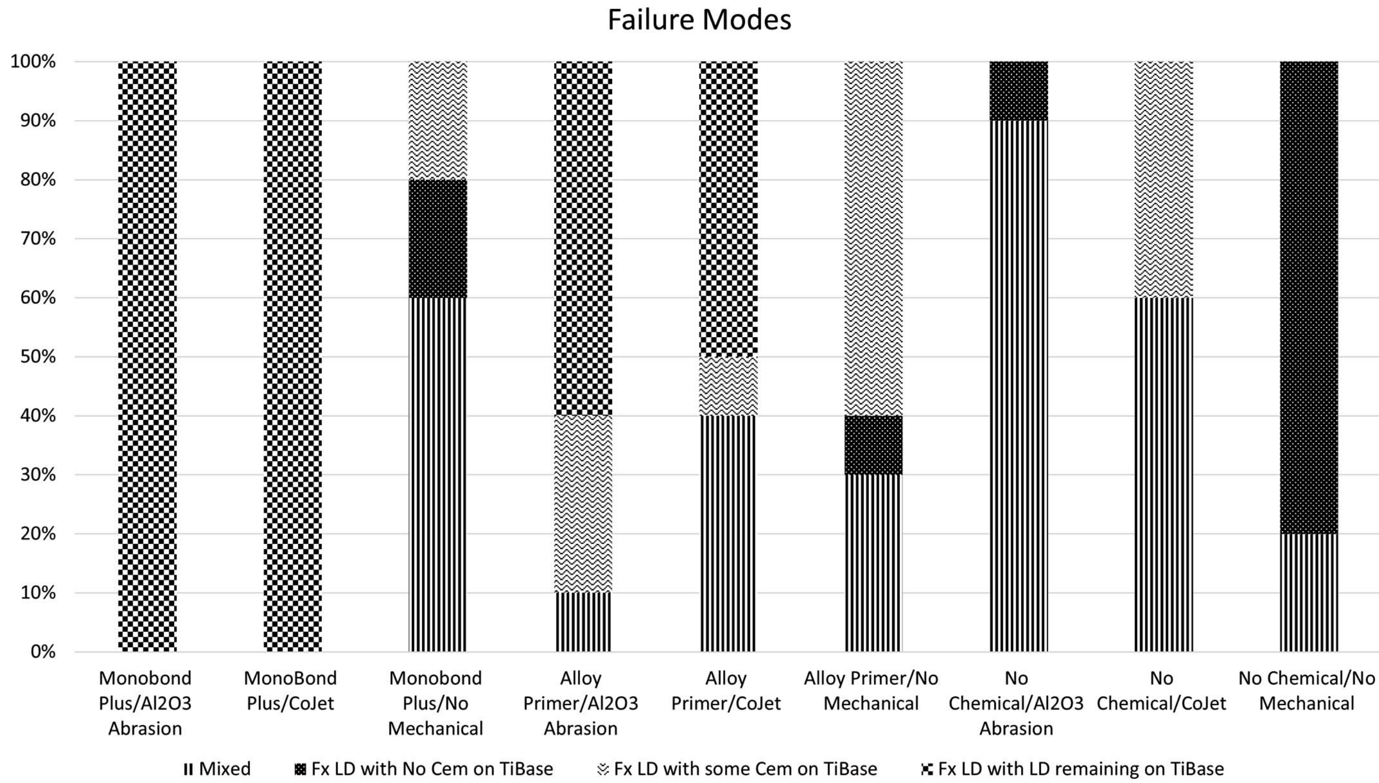
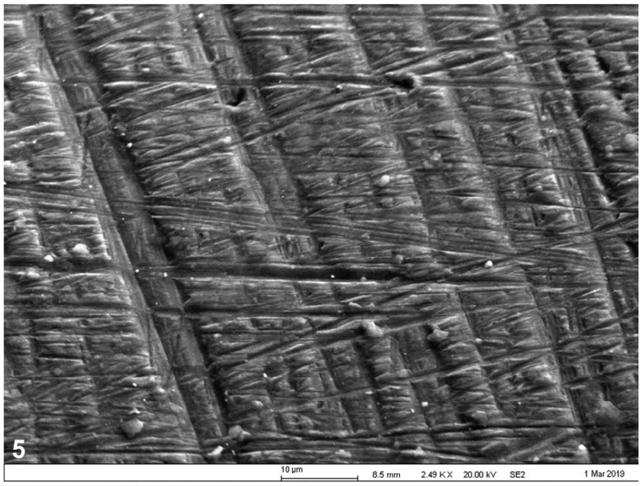


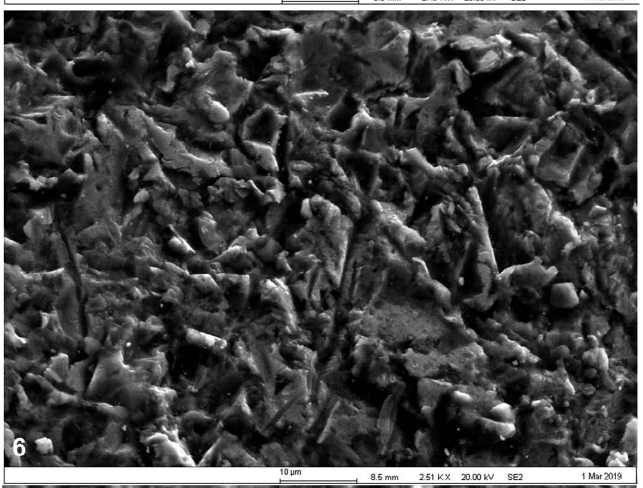
Figure 4. *Graphical representation of the various failure modes.*

ment, there was no statistically significant effect by any of the three chemical treatments. This is likely because of the increase in the bonding surface area due to increased surface roughness. The pull-off strengths correlated with the measured roughness of the Ti-Bases, with Al_2O_3 producing the roughest surface and the highest overall force with no chemical surface treatment. Papadopoulos and others[13] showed that the use of a large particle size increased the surface roughness and promoted increased mechanical retention when firing porcelain onto titanium. The effect of air abrasion on grade 5 alloy was shown to increase the shear bond strength to lithium disilicate.[14] However, a recent study by Linkevicius and others[15] showed that air abrasion of Ti-Bases with Al_2O_3 had a negative effect on retention of the zirconia coping. That study, however, used a different brand of titanium base (BioHorizons IPH Inc, Birmingham, AL, USA) that contains built-in retentive grooves. The air abrasion was shown to dull the retentive grooves, which could account for the discrepancy.[15]

The use of CoJet was overall less retentive than Al_2O_3 without chemical surface treatment. CoJet uses 30-μm particles, per the manufacturer. As described in this study, the smaller particle size yielded a smaller surface roughness according to the profilometer scan and is consistent with Fonseca and others,[16] who also showed particle size had a significant effect on bonding. Per the manufacturer's instructions, CoJet requires silane to be effective for bonding. When the silane containing Monobond Plus was added, the pull-off strength nearly doubled compared with the use of Alloy Primer, which does not contain silane. In this study's methodology, CoJet was applied at 2 bar. Per the manufacturer, this is the minimum accepted pressure that creates enough energy to embed the silica particles into the substrate. Were the maximum of 3 bar used, the bonding might have been significantly increased, but the authors felt it important to maintain consistency with the Al_2O_3 groups.

Monobond Plus was highly effective when combined with mechanical roughening of the Ti-Base with Al_2O_3 or CoJet silicoating. The effectiveness was likely due to a combination effect of each of its three functional components: trimethylpropyl methacrylate (silane), methacrylated phosphoric acid ester (10-MDP), and disulfide acrylate. As mentioned, silane in addition to CoJet allows for effective bonding. Air abrasion in addition to MDP and sulfur-containing compounds have also been shown to be





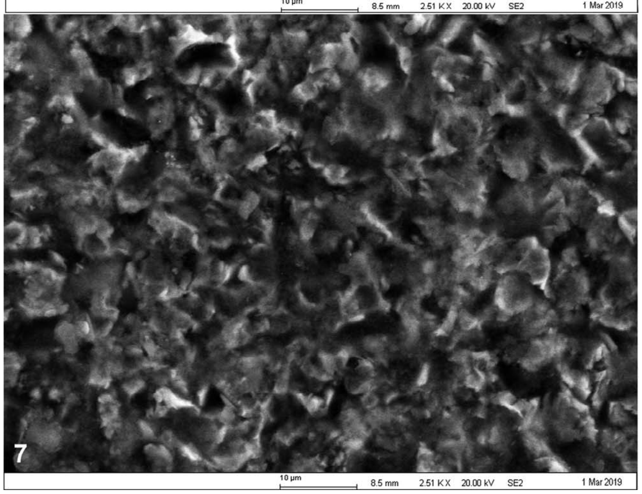


Figure 5. *Scanning electron micrograph photo of unmodified Ti-Base.*

Figure 6. *Scanning electron micrograph photo of Al_2O_3-treated Ti-Base.*

Figure 7. *Scanning electron micrograph photo of CoJet-treated Ti-Base.*

effective in bonding to titanium.[17-19] This study used Multilink Hybrid Abutment Cement, which is manufactured by Ivoclar Vivadent and is intended to be used with MonoBond Plus.

Unlike previous studies, the application of Alloy Primer did not appear to improve bonding between the Ti-Base and resin cement despite sharing similar components with Monobond Plus. One possible explanation is the potential differences between formulations. Ethanol is used as a solvent for Monobond Plus, while Alloy Primer uses acetone. The greater volatility of acetone may decrease the substantivity of the Alloy Primer. In their evaluation of the effect of organic solvents, Amaral and others[17] found that the type of solvent (ethanol or acetone) had no effect on the degree of conversion or resin-dentin bond stability; however, their study evaluated 4-methacryloyloxyethyl trimellitate anhydride adhesive (4-Meta Sun Medical Co, Kyoto, Japan) and not a primer, as investigated in this study.[17] In addition, thermal-cycling might have contributed to a decrease in the effects of Alloy Primer. Hiraba and others[18] looked at the effect of primers, including Alloy Primer and Monobond Plus, on the bond between tri-n-butylborane–initiated resin and a gold alloy. Part of the study design compared bond strengths before and after thermal-cycling. Their data showed that after thermal-cycling, the mean bond strength exhibited a significantly greater decrease in the groups using Alloy Primer compared with Monobond Plus.[18]

Groups with the greatest pull-off bond strengths combined mechanical modification and chemical surface treatments and shared the most common mode of failure: a fragment of lithium disilicate remaining firmly bonded to the Ti-Base with some cement on both the dislodged lithium disilicate coping and on the Ti-Base (see Figure 3). This failure mode indicates that while there was partial adhesive failure between the Ti-Base and coping interface, the bond between the remaining fragment and the Ti-Base was stronger than the tensile strength of that area of lithium disilicate coping. In all but two specimens, the remaining fragment was on the most cervical aspect of the Ti-Base and encased the tab used by the Ti-Base system for orientation of the crown on the abutment. Because of the taper of the Ti-Base, the cervical area has the largest diameter and thus the largest surface area for bonding. It is possible that the coronal portion of the Ti-Base with less surface area might have debonded first, creating greater tension between the coronal and apical segments. The failure mode of the lithium disilicate copings in the remaining groups was more heterogeneous. As expected, the group with no modification of the Ti-Base featured the most cases of no cement remaining on the Ti-Base. The remaining groups all had some cement on both surfaces, indicating partial adhesive failure of both the Ti-Base cement interface and lithium disilicate cement interface.

The authors caution that this study used a single static test. While informative, static testing gives limited information on the effects of repeated forces on cement interfaces. In this study, the lowest tensile pull-off bond strength of 327.8 N was greater than the maximum jaw-opening strength of 142.86 N recorded in previous research.[20] In addition, more research is necessary using other types of surface primers and cements.

CONCLUSIONS

Based on the limitations of this study, when bonding lithium disilicate copings to Ti-Bases, mechanical roughening with either Al_2O_3 air abrasion or CoJet silicoating is recommended. Once mechanically modified, Monobond Plus appears to be the superior chemical primer of the materials tested for treating the Ti-Base when using MulitLink Hybrid Abutment Cement. Further studies are needed to compare additional combinations of materials for maximizing Ti-Base retention.

Disclaimer

The views expressed in this article are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, Uniformed Services University of the Health Sciences, or the United States government.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 4 September 2019)

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