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Effect of Various Surface Treatments on Ti-Base Coping Retention

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Abstract

The titanium-cement interface of a Ti-Base implant crown must be able to resist intraoral pull-off forces. **Objective:** 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 hybrid-ceramic abutment coping. **Methods:** Ti-Bases were divided into 9 groups of 10 abutments 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). Hybrid-ceramic abutment copings (VITA Enamic, VITA) were designed and milled. After treatment of the screw channel with hydrofluoric acid and a silane-containing primer, the copings were cemented onto the Ti-Bases with a resin cement according to the manufacturer's recommendations. Each coping was 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 Ti-Base until fracture (Instron). Data were analyzed with 2-way and 1-way ANOVAs/Tukey post hoc tests (alpha=0.05). **Results:** Significant differences were found between groups based on type of surface treatment ($p<0.05$). See table. The results of the 2-way ANOVA found that with mechanical treatment, the greatest bond strengths were produced overall by Al_2O_3 air abrasion > CoJet silicoating > no treatment ($p<0.001$). With chemical treatment, the greatest bond strengths were produced overall with Monobond Plus > Alloy Primer = no treatment ($p<0.001$). **Conclusions:** The greatest pull-off bond strength of a hybrid-ceramic abutment coping to a titanium-abutment base was produced by mechanical treatment with Al_2O_3 air abrasion and chemical treatment

with Monobond Plus. The lowest bond strength was produced with no mechanical and no chemical surface treatment.

Introduction

The advancement in computer-aided design/computer-aided manufacturing (CAD/CAM) dentistry has brought about a new era in modern dentistry. The advent of CAD/CAM dentistry has yielded several advantages over traditional dental workflows. Focusing 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 showed that this workflow decreased the cost to patients by 30% and halved the laboratory workload.[2]

CAD/CAM technology is compatible with multiple restorative materials. For the posterior region, high occlusal forces have required materials with high fracture strengths. Polycrystalline (zirconia) and more recently, lithium-disilicate glassceramics, have been used for abutment and restoration of implants in this critical region.[3-5] However, high mechanical loads can lead to potential bone loss around the implant. The use of materials with elastic properties for the fabrication of dental implant superstructures may be a promising technique to reduce functional occlusal forces on implants.[6]

Recently, VITA has introduced a hybrid-ceramic CAD/CAM block (VITA Enamic, Bad Säckingen, Germany) marketed for use with the Ti-Base system. VITA Enamic is composed of a predominately feldspar-ceramic network enriched with aluminum oxide (86% wt or 75% vol) and a polymethyl-methacrylate (14% wt or 25% vol) that permeates the feldspar-ceramic matrix to yield a restorative material that is both strong and elastic as claimed by the manufacturer.[7] VITA Enamic exhibits a number of potential advantages over other CAD/CAM materials. Unlike zirconia or lithium disilicate, it does not require firing after being milled but only requires polishing, significantly reducing the processing time.[7] Moreover, when repair of proximal contact is warranted, composite resin materials can easily be added onto VITA Enamic, greatly reducing the treatment time. Albero et al.[8] and Wendler et al.[9] showed that VITA Enamic exhibits a modulus of elasticity similar to dentin and mechanical properties between those of glass ceramic and resin-based composites. On the other hand, VITA Enamic showed inferior flexural strength compared to zirconia or lithium disilicate.[10] Pouranfer et al. concluded extrinsic staining of VITA Enamic may be necessary to achieve comparable esthetics to ceramic materials. However, the extrinsic staining and polish of VITA Enamic was more susceptible to color change after simulated tooth brush abrasion compared to lithium disilicate (IPS e.max CAD; Ivoclar Vivadent, Schaan, Liechtenstein)[11]

Very little research has been published examining VITA Enamic as an implant abutment material. According to a study by Weyhrauch et al., when bonded to a titanium abutment using different types of cements, VITA Enamic exhibited a similar fracture strength to a lithium-disilicate material (IPS e.max CAD).[12] A recent study by Rohr et al. found that when bonding VITA Enamic to a one-piece zirconia implant and abutment, cements containing a methacrylated phosphoric acid ester (10-MDP) provided a high chemical bond.[13] No research has been conducted evaluating the pull-off bond strength of VITA Enamic to the titanium-based Ti-Base implant abutment system.

According to Abi-Rached et al., although titanium has attractive physical properties, there is a need for improving the bond at the titanium/cement interface for improved longevity of the restoration.[14] Mechanical and chemical surface treatments are often employed to improve titanium bonding. The mechanical surface treatments such as air abrasion with aluminum oxide (Al_2O_3) and tribochemical silica coating are commonly used. Air abrasion roughens the titanium surface, thereby increasing the bonding surface area and retention. Ebert et al. demonstrated that air abrasion with Al_2O_3 significantly increased the bond strength between zirconia copings and titanium compared to the control.[15] Von Maltzahn et al. also investigated the effect of tribochemical coating of titanium, where silica particles are embedded into the titanium surface via high speed impact. The author concluded that the tribochemical surface treatment resulted in less retentive compared to air abrasion with Al_2O_3.[16]

Chemical surface treatment is often used in conjunction with mechanical treatment to further enhance the bonding to titanium. According to the manufacturers, Monobond Plus (Ivoclar Vivadent,) and Alloy Primer (Kuraray, Tokyo, Japan) increase the bond strength to metals. Whereas there is little research to date evaluating the effect of Monobond Plus to titanium bonding, the manufacturer specifically mentions the use of Alloy Primer on titanium. 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. Veljee et al. showed that the addition of Alloy Primer increased the retention of a resin cement to pure titanium at a statistically significant level.[17] They postulated that the Alloy Primer promotes wettability, thus increasing the adhesive bonding. Yanagida et al. noted that Alloy Primer further enhanced the bonding strength to resin cement when used in conjunction with air abrasion or tribochemical surface treatment. [18] A recent study by Kemarly et al found that when bonding a lithium-disilicate abutment coping (IPS e.max CAD) to a Ti-Base, 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. However, Alloy Primer did not provide a statistically significant increased pull-off bond strength when the Ti-Base surfaces were mechanically treated with Al_2O_3 air abrasion or CoJet silicoating.[19]

The purpose of this study was to evaluate several surface treatments in differing combinations and their effect on the pull-off bond strength of VITA Enamic coping cemented to a Ti-Base implant abutment base. The null hypothesis tested was that there will be no difference in pull-off bond strengths of the VITA Enamic copings from the Ti-Base regardless of surface treatment modality.

MATERIALS AND METHODS

A custom coping was designed in SolidWorks CAD 3-D software (Dassault Systemes, Velizy-Villacoublay, France). In addition, a custom cradle was designed to adapt to an existing vice grip of the universal testing machine (Model 5543, Instron, Norwood, MA) to fit intimately with the coping to allow for an even distribution of pull-off forces to prevent possible fracture due to direct compression of the VITA Enamic from the vice clamps. The cradles were 3D printed (Objet 260 Dental Selection, Stratasys Ltd., Eden Prairie, MN). The copings were milled in VITA Enamic on a 5-axis milling unit (CORiTEC 450i, imes-icore GmbH, Eiterfeld, Germany), placed on an implant lab analog (Certain 4.1mm, Biomet 3i, Palm Beach Gardens, FL), and 3D scanned into the inLab software (v18.1, Dentsply, Sirona). Ninety copings were milled from the VITA Enamic abutments using a milling unit (MCXL, Dentsply/Sirona). A custom implant holding base was designed in SolidWorks with a channel. The bases were 3D printed (SLA Viper si2, 3D systems, Rock Hill, SC) and into each, an implant (Certain 4.1mm, Biomet 3i) was hand threaded. Each implant tower was inspected to ensure that the implant was placed parallel to the long axis so that the pull-off strength would be parallel and evenly distributed.

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

The intaglio surface of the custom VITA Enamic coping were degreasedqw with alcohol, etched for 60 seconds with 5% hydrofluoric acid (VITA ADIVA CERA-ETCH, Zahnfabrik), rinsed thoroughly using water with a 3-way syringe, then dried for 20 seconds. Monobond Plus (Ivoclar Vivadent) was applied to the etched surfaces and allowed to react for 60 seconds and gently air dried using a 3-way syringe. The specimens were cemented to the Ti-Bases using a resin cement, Multilink Hybrid-Abutment Cement (Ivoclar Vivadent) according to the manufacturer's recommendations. During the setting of the cement, the specimens were loaded onto a custom jig with a 100g weight on the coping, to ensure uniform, standardized pressure. Glycerin gel was applied to the cement margin for 7 minutes, then rinsed off with a 3-way syringe. After removal of the glycerin gel, the cement interface was polished to simulate a clinical procedure. Next, the Ti-Base specimens were torqued to each implant tower 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) and considered acceptable if greater than 1000 mW/cm^2. Teflon tape was placed in the access chamber, followed by Filtek Z250 (3M/ESPE), which was placed incrementally, light cured, and polished with Enhance and Pogo polishing points (Dentsply). The assembled specimens were then placed in distilled water and stored in an incubator (Model 20 GC, Quincy Labs, Chicago, IL) for 24 hours at 37 ^oC. The specimens were then thermal cycled in distilled water for 2000 cycles at 5^{o}C and 55^{o}C with a dwell time of 30 seconds at each temperature (Sabri Dental Enterprise, Downers Grove, IL). Each specimen was then loaded under tension in a universal testing machine (Model 5543, Instron) with a pair of customized vice jig assemblies holding the VITA

Enamic restoration on one side and the 3D printed resin tower with an embedded implant on the other. The universal testing machine subjected the VITA Enamic copings to a removal force parallel to the long axis of the interface at a speed of 1mm/min until the copings fractured or separated from the Ti-Bases. The maximum force between components was recorded in Newtons.

Upon completion of the pull-off test, the fractured surfaces of all specimens were analyzed using a stereomicroscope at 10x magnification (SMZ-1B, Nikon, Melville, NY). The fractured surfaces were evaluated and classified into the following failure modes: cement remaining on Ti-Base only, cement remaining on VITA Enamic only, cement remaining on both Ti-Base and VITA Enamic, fractured VITA Enamic with no cement remaining on Ti-Base, fractured VITA Enamic with some cement remaining on Ti-Base, and fractured VITA Enamic with portion of VITA Enamic still bonded to the Ti-Base. The surface roughness of 9 Ti-Bases were analyzed after mechanical modification (3 per group). Surface roughness (Ra) was measured using a non-contact profilometer (3D Laser-Scanning Confocal Profilometer, Keyence, Itasca, IL) and then analyzed using its proprietary software. The morphology of the Ti-Base surfaces was investigated by a field-emission scanning electron microscopy (Sigma VP, Carl Zeiss, Oberkochen, Germany). The elemental composition of the Ti-Base surfaces was characterized by an energy dispersive spectroscopy (X-Max, Oxford Instruments, Abingdon, United Kingdom).

A mean pull-off force (N) at fracture and the standard deviation was determined for each of the 9 groups. Data were analyzed using a two-way analysis of variance (ANOVA) to evaluate the effect of mechanical (3 levels) or chemical treatments (3 levels) of the Ti-Base surface on the pull-off strength of the VITA Enamic specimens (alpha = 0.05).

The sample size of 10 specimens per group provided 80% power to detect a moderate effect size (0.29, or approximately 0.58 standard deviation) difference among means for the main factor of mechanical or chemical treatment, and a moderate effect size (0.345, or approximately 0.69 standard deviation) difference among means for the interaction term when testing with a two factor ANOVA at the alpha level of 0.05 (SPSS, Chicago, IL).

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 ($P<0.001$) and there was no significant interaction ($P=0.55$). The data were further evaluated by multiple one-way ANOVA's per mechanical or chemical surface treatment. See Table 1.

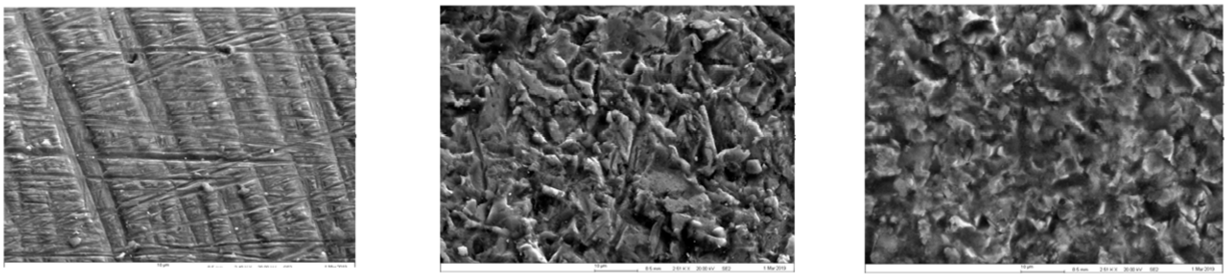
Among the groups treated with mechanical surface treatment with Al_2O_3 air abrasion, Chemical surface treatment with Monobond Plus (774.6 ± 221.3 N) resulted in the greatest pull-off bond strength and it was significantly different from the group that received no chemical surface treatment (500.2 ± 176.6 N), but not significantly different from the group treated with Alloy Primer (669.4 ± 203.1 N). Among the groups treated with Monobond Plus chemical surface treatment, no significant difference in pull-off bond strength was found with the group treated with Al_2O_3 air abrasion (774.6 ± 221.3 N), CoJet silicoating (648.5 ± 221.3 N) or the group that did not receive mechanical surface treatment (560.1 ± 186.3 N). The lowest pull-off bond strength was found with the group withoutchemical or mechanical surface treatment (110.4 ± 23.7 N), which was significantly different from the group treated with Monobond Plus/no mechanical surface treatment (560.1 ± 186.3 N) but not significantly different than Alloy Primer chemical treatment/ no mechanical surface treatment (172.5 ± 91.5 N). See Table 1.

Table 1: Pull-off bond strengths of the Vita Enamic copings from the Ti-Bases after chemical and mechanical surface treatments.

Mechanical Surface Treatment	**Pull-Off Bond Strength** Newtons (st dev)		
	Chemical Surface Treatment		
	Monobond Plus	**Alloy Primer**	**None**
Al_2O_3 Air Abrasion	774.6 (221.3) Aa	669.4 (203.1) ABa	500.2 (174.6) Ba
CoJet Silicoating	648.5 (201.3) Aa	288.4 (99.0) Bb	257.9 (86.5) Bb

When evaluating failure modes, the group without any mechanical or chemical surface treatment showed cement remaining on VITA Enamic only. All other groups exhibited fractured VITA Enamic with portion of VITA Enamic still bonded to the Ti-Base.

SEM photos of the Ti-Base surface treated with Al_2O_3 air abrasion and CoJet silicoating can be seen in Figure 1 (a-c) below. Al_2O_3 provided the roughest surface (0.925 ± 0.124 μm) compared to CoJet (0.555 ± 0.000 μm) or no mechanical treatment (0.297 ± 0.040 μm) groups. Higher composition of Silicon by weight was noted in the samples treated with CoJet (5.73%) compared to Al_2O_3 air abrasion (0.25%) or no mechanical treatment (0.23%). Less availability of Titanium was seen with both Al_2O_3 air abrasion (35.53%) or CoJet (27.29%) compared to no mechanical treatment (70.9%).



a. b. c.

Figure 1 (a-c) SEM photo of unmodified Ti-Base (a); Al2O3 treated Ti-Base (b) and CoJet Treated Ti-Base (c).

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 cemented Vita Enamic to the Ti-Base implant abutment base. Within the limitations of this *in vitro* study, the null hypothesis was rejected because the results of the study found statistically significant differences in pull-off bond strengths of the Vita Enamic copings from the Ti-Base dependent upon surface treatment modalities. Based upon the results, it appears that the surface treatment with Monobond Plus was the single most important factor in bonding to the Ti-Base. Combining mechanical surface treatment with Monobond Plus did not further improve the pull-off strength at a statistically significant level. However, the highest pull-off strength was seen in the group with combined application of Al_2O_3 air abrasion with Monobond Plus. This is likely due to the increase of 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 et al showed that use of a large particle size increased surface roughness and promoted increased mechanical retention when firing porcelain onto titanium.[20] However, a recent study by Linkevicius et al that showed air abrasion of Ti-Bases with Al_2O_3 had a negative effect on retention of a zirconia coping. That study, however, utilized a different brand of titanium base (BioHorizons IPH, Inc, Birmingham, AL) that contains built-in retentive grooves. The air abrasion was shown to dull the retentive grooves which could account for the discrepancy of these results.[21]

The use of CoJet was overall less retentive than Al_2O_3 regardless of the chemical surface treatment modality used. CoJet was only effective in increasing the pull-off strength at the statistically significant level when used in combination with Monobond Plus. 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 more than doubled compared to the group where CoJet was used with Alloy Primer, which does not contain silane. In this study's methodology, CoJet was applied with 30 μm particles at 2 bar. Fonseca et al that also showed particle size had a significant effect on bonding.[22] Per the manufacturer, this is the minimum accepted pressure that creates enough energy to embed the silica particles into the substrate. Utilization of CoJet with different particle size or at higher pressure could have yielded further increase in bonding.

Monobond Plus was highly effective regardless of the mechanical surface treatment modality used. 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 shown to be effective in bonding to titanium.[23-25] Additionally, 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[12, 19] using other abutment materials, this study found that the application of Alloy Primer was only effective when combined with air abrasion with Al_2O_3. When used with CoJet or no mechanical surface treatment, 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. Monobond Plus utilizes ethanol while Alloy Primer contains acetone, which is more volatile, and may decrease the substantivity of the Alloy Primer. In their evaluation of the effect of organic solvents, Amaral et al found that the type of solvent (ethanol or acetone) had no effect on degree of conversion or resin-dentin bond stability, however their study evaluated 4-Methacryloyloxyethy trimellitate anhydride adhesive (4-Meta Sun Medical Co, Kyoto, Japan) and not a primer as investigated in this study.[23] Additionally, thermal cycling might have contributed to a decrease in the effects of Alloy Primer. Hiraba et al 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. One aspect of the study design compared bond strengths before and after thermal cycling. Their data showed that after thermal cycling, the mean bond strength dropped significantly more with the groups using Alloy Primer compared to Monobond Plus.[24]

All groups but the group without mechanical or chemical treatment shared the same mode of failure - a fragment of Vita Enamic remaining firmly bonded to the Ti-Base with some cement on both the dislodged Vita Enamic coping and on the Ti-Base. 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 Vita Enamic coping. In most 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. Due to 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 group without mechanical or chemical treatment showed no cement remnant on the Ti-Base, signifying inadequate bonding between the resin cement and the Ti-Base.

In a similar study by the same research group, the use of a lithium-disilicate abutment produced a higher increase in pull-off strength with the use of CoJet silicoating and Monobond Plus (1011.5 ± 120.2 N) over Al_2O_3 air abrasion with Monobond Plus (896.0 ± 173.1 N) when compared to the baseline of no mechanical treatment with Monobond Plus (340.9 ± 95.5 N). In contrast, with the Vita Enamic abutment, Al_2O_3 air abrasion with Monobond Plus (774.6 ± 221.3 N) exhibited a greater increase in pull-off strength over CoJet silicoating with Monobond Plus (669.4 ± 203.1 N), compared to the baseline of no mechanical treatment with Monobond Plus (560.1 ± 186.3 N). However, in both studies, no significant difference was observed with the use of different mechanical treatment with the use of Monobond Plus, while a significant difference was seen over the respective group with no mechanical treatment.[19]

With the use of a lithium-disilicate abutment, mechanical treatment with Al_2O_3 air abrasion with Alloy Primer (795.9 ± 127.1 N) did not provide significantly different pull-off strength when compared to Al_2O_3 air abrasion without chemical treatment (650.3 ± 54.7 N). CoJet with Alloy Primer (491.1 ± 102.3 N) did not provide a significantly different pull-off strength when compared to CoJet without chemical treatment (501.8 ± 49.0 N). No mechanical treatment with Alloy Primer (332.4 ± 85.4 N) did not provide significantly different pull-off strength when compared to no mechanical treatment withoutchemical treatment (393.1 ± 65.3 N). These results signify ineffectiveness of Alloy Primer when bonding a lithium-disilicate abutment onto a Ti-Base. In contrast, with Vita Enamic abutments, Alloy Primer showed significant increase in pull-off strength when used in conjunction with Al_2O_3 air abrasion (669.4 ± 203.1 N), compared to when Alloy Primer was used with CoJet (288.4 ± 99.0 N), or without mechanical treatment (172.5 ± 91.5N). This suggest effectiveness of Alloy Primer when used in conjunction with Al_2O_3 air abrasion when bonding a Vita Enamic abutment onto a Ti-Base.

The authors caution that this study utilized a single static test. While informative, static testing gives limited information on the effects of repeated forces on cement interfaces. In all but the group without any mechanical or chemical surface treatment, the average tensile pull-off bond strength was greater than the maximum jaw-opening strength of 142.86 N recorded in previous research.[26] Additionally, more research is necessary using other types of surface primers and cements.

CONCLUSIONS

Based on the limitations of this study, when bonding Vita Enamic copings to Ti-Bases, mechanical roughening with either Al_2O_3 air abrasion or CoJet silicoating, used in conjunction with Monobond Plus is recommended. Regardless of mechanical modification, Monobond Plus appears to be the superior chemical primer of the materials tested for treating a Ti-Base when using MulitLink Hybrid Abutment Cement. CoJet silicoating was only effective when used with Monobond Plus, whereas Alloy Primer was only effective when used in combination with Al_2O_3 air abrasion. Further studies are needed to compare additional combinations of materials for maximizing Ti-Base retention.

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