Effect of surface treatment and luting agent type on shear bond strength of titanium to ceramic materials

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PURPOSE. This study aimed to compare the effect of different surface treatments and luting agent types on the shear bond strength of two ceramics to commercially pure titanium (Cp Ti). MATERIALS AND METHODS. A total of 160 Cp Ti specimens were divided into 4 subgroups (n = 40) according to surface treatments received (control, 50 μm airborne-particle abrasion, 110 μm airborne-particle abrasion, and tribochemical coating). The cementation surfaces of titanium and all-ceramic specimens were treated with a universal primer. Two cubic all-ceramic discs (lithium disilicate ceramic (LDC) and zirconia-reinforced lithium silicate ceramic (ZLC)) were cemented to titanium using two types of resin-based luting agents: self-cure and dual-cure (n = 10). After cementation, all specimens were subjected to 5000 cycles of thermal aging. A shear bond strength (SBS) test was conducted, and the failure mode was determined using a scanning electron microscope. Data were analyzed using three-way ANOVA, and the Tukey-HSD test was used for post hoc comparisons (P < .05). RESULTS. Significant differences were found among the groups based on surface treatment, resin-based luting agent, and ceramic type (P < .05). Among the surface treatments, 50 μm air-abrasion showed the highest SBS, while the control group showed the lowest. SBS was higher for dual-cure resin-based luting agent than self-cure luting agent. ZLC showed better SBS values than LDC. CONCLUSION. The cementation of ZLC with dual-cure resin-based luting agent showed better bonding effectiveness to commercially pure titanium treated with 50 μm airborne-particle abrasion. [J Adv Prosthodont 2022;14:78-87]

KEYWORDS
Bond strength; Hybrid abutment; Lithium silicate; Resin-based luting agent; Surface treatment

INTRODUCTION
Implant-supported restorations have many proven properties, such as long-lasting use and esthetics, making them a viable treatment alternative.
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The composition and manufacturers of the materials used in the study are shown in Table 1. A total of 160 disc-shaped Cp Ti (10 mm in diameter and 3 mm in height) (Premium 5030 CNC milling machine; Etterfeld, Germany) were fabricated using the milling method and embedded in chemically polymerized acrylic resin (Meliodent; Heraeus Kulzer, South Bend, IN, USA). Discs were cleaned ultrasonically for 5 minutes and divided into four groups to receive surface treatments (n = 40). The first group of samples received no surface treatment and served as a control group. The second group received airborne-particle abrasion with 50 µm Al₂O₃ and the third group with 110 µm Al₂O₃ at 2 bar pressure for 10 s at an angle of 45 degrees. The fourth group received tribochemical coating with 30-μm silicatized Al₂O₃ particles (CoJet™ System; 3M ESPE, Seefeld, Germany) at 2 bar pressure for 15 seconds, as recommended by the manufacturer. After surface treatment, the samples were cleaned in an ultrasonic cleaner for 180 s with 10% alcohol. Each treated group was further divided into two subgroups.
(n = 20) to receive all-ceramic specimens: (1) lithium disilicate ceramic (IPS e.max CAD; Ivoclar Vivadent AG, Schaan, Liechtenstein), (2) zirconia-reinforced lithium silicate ceramic (Celtra Duo; Dentsply Sirona, Konstanz, Germany). Ceramic specimens were milled from blocks (5 mm diameter, 3 mm height) and fired in a ceramic oven (Programat P310; Ivoclar Vivadent, Schaan, Liechtenstein), following the parameters provided by the manufacturers (e.max CAD: 403°C start, 90°C/min heating rate, 820°C final #1, 10:00 holding time #1, 840°C final #2, 7:00 holding time #2; Celtra Duo: 500°C start, 55°C/min heating rate, 820°C final, 1:30 holding time). Zirconia-reinforced lithium silicate restorations were etched using 4.8% hydrofluoric acid etching gel (IPS Ceramic Etching Gel; Ivoclar Vivadent, Schaan, Liechtenstein) for 30 s, lithium disilicate restorations were etched with the same etching gel for 20 seconds, and both were then rinsed with water spray. Ceramic specimens were divided into two groups, according to resin-based luting agent type: Multilink Hybrid Abutment (Ivoclar Vivadent, Schaan, Liechtenstein) and Panavia V5 (Kuraray Noritake Dental, Tokyo, Japan). Monobond Plus was applied to titanium and ceramic specimens, allowed to react for 60 s, and air-dried for the Multilink Hybrid Abutment resin-based luting agent group. For the Panavia V5 group, Clearfil Ceramic Primer Plus was applied to titanium and ceramic specimens and dried. Resin-based luting agents were applied to ceramic surfaces and cemented to the Ti surface under 15 N pressure with a Gillmore device. Specimens of the Panavia V5 resin-based luting agent group were light-cured for 5 seconds using a light-emitting diode (LED) light (VALO™ Cordless; Ultradent, South Jordan, UT, USA). Specimens of the Multilink Hybrid resin-based luting agent group were autopolymerized for 7 minutes. Excess luting material on the margins was removed using a scalpel. All cementation procedures were performed by the same operator (G.S.O).

All specimens were stored in a humidifier for 24 h at 37°C and aged by thermal cycling (5000 cycles, 5°C - 55°C) with a dwell time of 20 s in distilled water. The bonded specimens were placed in a universal testing machine and loaded with a crosshead speed of 1 mm/min. The maximum shear load was recorded immediately before debonding. The following formula was used to calculate the SBS data: fracture load/bonding surface area = N/mm² = MPa. Failure types were examined using a reflected-light microscope (Olympus SZ40; Olympus Optical Co., Tokyo, Japan) at 20× magnification (Fig. 1). Failure types were determined according to the amount of luting material remaining on the titanium surface, from score 0 to 3, as indicated in a previous study where score 0 represents adhesive failure and score 3 is complete cohesive failure.16

We selected one specimen randomly from each sur-

| Table 1. Types and compositions of the materials used in the study |
|---------------------------------------------------------------|
| **Product** | **Composition** | **Type** | **Manufacturer** |
|----------------|-----------------|----------|-----------------|
| IPS e.max CAD | SiO₂ 57.0-80.0%, Li₂O 11.0-19.0%, K₂O 0.0-13.0%, P₂O₅ 0.0-11.0%, ZrO₂ 0.0-8.0%, ZnO 0.0-8.0%, Colorants 0.0-18.0% | Lithium disilicate ceramic | Ivoclar Vivadent, Schaan, Liechtenstein |
| Celtra Duo | SiO₂ 59.3%, Al₂O₃ 3%, Li₂O 14.5%, K₂O 1.2%, Na₂O 0.2%, P₂O₅ 4.9%, B₂O₃ 2%, MgO 0.01%, ZrO₂ 9.3%, SrO 0.0003%, CeO₂ 0.83%, V₂O₅ 0.61%, Tb₂O₃ 3.3%, Er₂O₃ 0.73%, HFO 0.21% | Zirconia reinforced lithium silicate ceramic | Dentsply Sirona, Hanau, Germany |
| Multilink Hybrid Abutment | Bisphenol A diglycidyl methacrylate ethoxylated (bis-EMA), urethane dimethacrylate (UDMA), 2-hydroxyethyl methacrylate, ytterbium trifluoride, dibenzoyl peroxide | Self-cure resin-based luting agent | Ivoclar Vivadent, Schaan, Liechtenstein |
| Panavia V5 | Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, barium glass filler, fluorooaluminosilicate glass, silica filler, initiators, stabilizers, pigments | Dual-cure resin-based luting agent | Kuraray Noritake Dental, Tokyo, Japan |
face treatment group for SEM imaging. Selected specimens were cleaned ultrasonically in distilled water for 5 minutes, dried, and coated with a thin Au-Pd layer (200 - 300 nm). SEM (Zeiss Supra 40 VP; Carl Zeiss AG, Germany) was used for surface examination and results were presented as photographs.

Statistical analysis was performed using standard statistical software (SPSS V23; IBM Armonk, NY, USA). The Kolmogorov-Smirnov test ($P > .05$ for all tests) and Levene test ($P = .437$) confirmed that all the data were normally distributed and homogeneous; therefore, parametric tests were used. The effect of surface treatment, resin-based luting agent, and ceramic on shear bond strength was examined by a 3-way analysis of variance. The Bonferroni test was used to examine the main effects, while multiple comparisons were examined with the Tukey HSD test for interactions ($P \leq .05$).

**RESULTS**

The mean and standard deviation values of the SBS test from surface treatment, ceramic, and resin-based luting agent are shown in Table 2. The total values at the end of each column show the main effect of the surface treatments. The total values at the end of the Total line show the main effect of the ceramics. The main effect of the resin-based luting agent is seen when the total values at the end of the row and column are matched. Other total values show interactions. A 3-way ANOVA was performed to determine the main effects of surface treatment, resin-based

![Fig. 1.](https://jap.or.kr)

**Table 2.** The mean and standard deviation values of SBS test from surface treatment, ceramic and resin-based luting agent

| Resin-based luting agent | Ceramic       | Control       | 50 µm airborne-particle abrasion | 100 µm airborne-particle abrasion | Tribochemical silica coating | Total     |
|--------------------------|---------------|---------------|---------------------------------|----------------------------------|-------------------------------|-----------|
| **Multilink Hybrid**     | e.max CAD     | 9.13 ± 0.91   | 15.89 ± 1.03                    | 15.56 ± 1.46                     | 12.89 ± 1.11                  | 13.37 ± 2.95C  |
|                          | Celtra Duo    | 9.62 ± 0.94   | 20.67 ± 1.19                    | 18.17 ± 3.34                     | 12.68 ± 1.19                  | 15.29 ± 4.79B  |
|                          | Total         | 9.38 ± 0.94D  | 18.28 ± 2.68AB                  | 16.86 ± 2.84BC                   | 12.79 ± 1.12D                 | 14.33 ± 4.07  |
| **Panavia V5**           | e.max CAD     | 12.6 ± 1.62   | 17.58 ± 1.37                    | 18.16 ± 1.3                      | 15.43 ± 2.11                  | 15.94 ± 2.71AB |
|                          | Celtra Duo    | 12.71 ± 1.98  | 20.74 ± 2.29                    | 17.38 ± 1.18                     | 15.77 ± 2.43                  | 16.65 ± 3.52A  |
|                          | Total         | 12.66 ± 1.76E | 19.16 ± 2.45A                   | 17.77 ± 1.28AB                   | 15.6 ± 2.22C                  | 16.3 ± 3.14   |
| **Total**                | e.max CAD     | 10.87 ± 2.19F | 16.73 ± 1.46Y                   | 16.86 ± 1.9Y                     | 14.16 ± 2.12                  | 14.65 ± 3.1   |
|                          | Celtra Duo    | 11.17 ± 2.19F | 20.71 ± 1.78F                   | 17.77 ± 2.47F                    | 14.23 ± 2.44F                 | 15.97 ± 4.23  |
|                          | Total         | 11.02 ± 2.17E | 18.72 ± 2.58b                   | 17.31 ± 2.22C                    | 14.19 ± 2.25d                 | 15.31 ± 3.76  |

$\text{a-d: There is no difference among the surface treatments with the same letter, A-E: There is no difference between surface treatment and resin based luting agent interactions with the same letter, X-T: There is no difference between surface treatment and ceramic interactions with the same letter.}$
luting agent type, and ceramic (Table 3). There was a statistical difference among all surface treatments, with the highest result obtained in the 50 µm airborne-particle abrasion group. Resin-based luting agent type was also statistically significant \((P < .001)\). The mean value was 14.33 MPa in the Multilink Hybrid Abutment resin-based luting agent group and 16.3 MPa in the Panavia V5 group. The ceramic type was also significant \((P < .001)\); the mean value was 14.65 MPa in the e.max CAD group and 15.97 MPa in the Celtra Duo group. Surface treatment and resin-based luting agent interaction were statistically significant \((P = .002)\). The highest results were obtained in the 50 µm airborne-particle abrasion Panavia V5 cement interaction \((19.16 \pm 2.45)\), and the lowest results were obtained in the control group Multilink Hybrid Abutment resin-based luting agent interaction \((9.38 \pm 0.94)\). Surface treatment and ceramic interactions were also statistically significant \((P < .001)\). The highest results were obtained in 50 µm airborne-particle abrasion with Celtra Duo interaction \((20.71 \pm 1.78)\) and the lowest results in Celtra Duo \((11.17 \pm 2.19)\) and e.max CAD \((10.87 \pm 2.19)\) ceramics of the control group. Resin-based luting agent and ceramic interactions were also statistically significant \((P = .027)\). The highest results were obtained in the Panavia V5 resin-based luting agent with Celtra Duo interaction \((16.65 \pm 3.52)\), while the Multilink Hybrid Abutment resin-based luting agent with e.max CAD interaction showed the lowest results \((13.37 \pm 2.95)\). No differences were found between Panavia V5 resin-based luting agent with the e.max CAD group and Multilink Hybrid Abutment resin-based luting agent with Celtra Duo interactions. The observed failure types by surface treatment, resin-based luting agent, and ceramic type are shown in Figure 2. Adhesive failure type (combination of scores 0 and 1) was more common than cohesive failure (combination of scores 2 and 3).

The SEM images of the surface treatment groups are shown in Figure 3. The control group showed the smoothest surface and followed by silica coating. Airborne-particle abrasion groups showed more irregularities and pores than control and silica-coated groups.

**DISCUSSION**

This study aimed to test the combined effects of routinely used surface treatments, various resin-based luting agent types, and ceramic types on bonding to titanium. The null hypothesis was rejected as surface treatments, ceramics, and resin-based luting agent type significantly affected shear bond strengths.

Previous studies investigating titanium bonding to ceramics reported that mechanical treatments applied to titanium surfaces increase the titanium-ceramic connection. Therefore, \(\text{Al}_2\text{O}_3\) air abrasion and tribochemical coating were chosen for the rough-
Examination of the partial eta squared results showed that surface treatment mainly affects bonding. Each of the surface roughening methods increased the bond strength of ceramics to titanium surfaces. The results of the present study are consistent with previous studies using shear or tensile tests that reported the positive effect of airborne particle abrasion application on bond strength to titanium before silane or adhesive primer application. Airborne-particle abrasion with 110 µm Al₂O₃ created more surface irregularities than airborne-particle abrasion with 50 µm Al₂O₃ in this study. However, this group showed lower bond strength than 50 µm Al₂O₃, indicating the importance of surface treatment as they can be applied in dental clinics and small laboratories. The group that did not receive any surface treatment served as a control.

Fig. 2. Failure types of specimens.
a: Control + e.max CAD + Multilink Hybrid, b: Control + e.max CAD + Panavia V5, c: Control + Celtra Duo + Multilink Hybrid, d: Control + Celtra Duo + Panavia V5, e: 50 µm airborne-particle abrasion + e.max CAD + Multilink Hybrid, f: 50 µm airborne-particle abrasion + e.max CAD + Panavia V5, g: 50 µm airborne-particle abrasion + Celtra Duo + Multilink Hybrid, h: 50 µm airborne-particle abrasion + Celtra Duo + Panavia V5, i: 110 µm airborne-particle abrasion + e.max CAD + Multilink Hybrid, j: 110 µm airborne-particle abrasion + e.max CAD + Panavia V5, k: 110 µm airborne-particle abrasion + Celtra Duo + Multilink Hybrid, l: 110 µm airborne-particle abrasion + Celtra Duo + Panavia V5, m: Cojet + e.max CAD + Multilink Hybrid, n: Cojet + e.max CAD + Panavia V5, o: Cojet + Celtra Duo + Multilink Hybrid, p: Cojet + Celtra Duo + Panavia V5.

Fig. 3. SEM images of surface treatments as control (A), 50 µm airborne-particle abrasion (B), 110 µm airborne-particle abrasion (C) and silica coating (D). 1000× magnification.
For the titanium surfaces treated with airborne-particle abrasion, two factors that need consideration are roughness and surface composition. Fonseca et al.\textsuperscript{23} found that Cp Ti abraded with 50 µm Al\textsubscript{2}O\textsubscript{3} and treated with adhesive showed higher bond strength than 120 µm Al\textsubscript{2}O\textsubscript{3} abraded groups. They discussed this result with the effect of surface composition changes after airborne-particle abrasion. For the present study, higher results in 50 µm Al\textsubscript{2}O\textsubscript{3} groups could be due to changes in surface composition. Silica coating has also been reported for improving bonding to titanium due to a chemical reaction between the silica layer and silane.\textsuperscript{22,25} However, similar to previous studies,\textsuperscript{17,19} the present study found no significant increase in bond strength after tribochemical silica coating. In order to ensure standardization in the present study, the Cojet system was used at the same pressure as airborne-particle abrasion. As described in previous studies, the manufacturers of the Cojet system claim that 2 bar is the minimum accepted pressure for creating sufficient energy to embed the silica particles into the substrate. It can be concluded that higher pressure can generate more energy and better bonding results. On the other hand, chemical silica-silane bonding could have decreased by thermal aging applied in this study. However, SEM images showed that the silica coating did not produce as much surface roughness as airborne-particle abrasion.

Surface treatment and ceramic selection are more critical than surface treatment and resin-based luting agent selection in titanium-ceramic bonding according to partial eta squared results. In the present study, surface preparation and cementation procedures were applied according to the recommendations of manufacturers. For this reason, hydrofluoric acid etching gel at the same concentrations (4.8%) was applied at different times. Fabian Fonzar et al.\textsuperscript{25} compared the shear bond strength of lithium disilicate and zirconia-reinforced lithium silicate ceramics using different concentrations of hydrofluoric acid and different etching times. They found higher bonding results with the zirconia-reinforced lithium silicate group in the 4.9% hydrofluoric acid concentration and indicated that etching time did not significantly affect adhesion. In the present study, Monobond Plus was applied to the Multilink Hybrid resin-based luting agent group and Clearfil resin-based luting agent group; these primers contain 10-methacryloyloxydecyl dihydrogen phosphate (MDP) for chemical bonding between the resin-based luting agent and the metal. Monobond Plus and Clearfil Ceramic Primer Plus contain silane (trimethylpropyl methacrylate and 3-Methacryloxypropyl trimethoxysilane, respectively) and ethanol as solvent. Several studies in the literature have evaluated the comparative effect of Monobond Plus and Clearfil Ceramic Primer Plus to Alloy Primer on bonding.\textsuperscript{17,26-29} Kemarly et al.\textsuperscript{17} used Monobond Plus and Alloy Primer with different surface treatments on titanium abutment base and investigated the pull-off bond strength of a lithium disilicate abutment coping. They found better results with Monobond Plus and reported that the silane content of Monobond Plus could produce better bonding. On the other hand, in contrast with the present study, Freifrau von Maltzahn et al.\textsuperscript{28} found better results with Monobond Plus than Clearfil Ceramic Primer Plus and Alloy Primer. However, applying Clearfil Ceramic Primer Plus only to ceramic surfaces and alloy primer to titanium surfaces may cause this difference in results. A previous study by the same researchers showed better results in specimens with Ceramic Primer Plus applied to titanium and ceramic surfaces than other groups.\textsuperscript{27} Various studies evaluating the bond strength of resin-based luting agents to titanium are available in the literature.\textsuperscript{16,20,29-33} However, different results emerged even in the studies using similar luting agent types. These differences could be due to the primers used, specimen differences in study design, or compositional differences in luting agents, including different ratios of monomers and chemical/light catalysts. According to ISO 9693:2019, the bond strength between metal-ceramic systems for dental restorations should be above 25 MPa, and in the present study, none of the groups could reach this value.\textsuperscript{34} It is noteworthy that this value is for veneering porcelains on metal substrates only. However, according to ISO 10477, the minimum acceptable SBS value for resin-based materials to different substrates is 5 MPa,\textsuperscript{35} and clinically, the resin-metal interface should be at least 10 MPa for satisfactory results.\textsuperscript{36} In the present study, all bonding values were...
above 10 MPa in surface-treated groups, even close to this value in the control group. Thus, it can be considered that both luting agents provided adequate clinical bonding. Dhesi et al.\textsuperscript{16} investigated the shear bond strength of lithium disilicate (e.max CAD) and four other ceramics to titanium with different luting agent types, including self-etch dual-cure resin and self-cure (Multilink Hybrid) resin-based luting agent in their study. Similar to the present study, they reported higher bond strengths with dual-cure resin-based luting agent than self-cure resin-based luting agent for the lithium disilicate group. On the other hand, Pitta et al. published a case report describing a bonding failure in a full contour zirconia crown cemented with Multilink Hybrid resin-based luting agent on a titanium base. In the cementation of the subsequently reconstructed crowns, the titanium bases were air abraded and bonded with glass ionomer luting agent, and no loss of cementation was observed in the 1-year clinical follow-up. However, in this study, it was not stated whether any surface treatment was applied to the first restorations and titanium bases. It is therefore unclear whether the debonding is solely dependent on the bonding agent.\textsuperscript{37} Failure modes after the shear bond test revealed a predominantly mixed cement distribution between Ti-base and ceramic surfaces for the airborne-particle abraded specimens. In contrast, all of the specimens showed adhesive failure in both luting agent groups of the control group. A systematic review and meta-analysis study reported that after the recommended surface treatment method (hydrofluoric acid etching and silanization) application, no significant difference was found in the bond strengths of lithium disilicate and zirconia-reinforced lithium silicate to resin-based luting agents.\textsuperscript{38} These observations support the benefit of airborne-particle abrasion to improve the adhesion to titanium surfaces and may explain the adhesive failure between the titanium and the cement in the untreated control group and the successful bonding between the resin-based luting agent and the ceramic.

In the present study, flat specimen surfaces were used for the bonding test. However, crown taper, crown height, and Ti base could affect retention. Due to this limitation, further studies should evaluate different ceramic and resin-based luting agent types with more retentive forms.

**CONCLUSION**

The shear bond test showed that the highest bond strength occurred with 50 µm Al\textsubscript{2}O\textsubscript{3} abrasion. Among the ceramic materials studied herein, the zirconia-reinforced lithium silicate ceramic group showed higher bond strength than the lithium disilicate ceramic group. Dual-cure resin-based luting agent provided higher bond strength between titanium surface and ceramic than self-cure resin-based luting agent.

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