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Composite Cement Components Stabilize the Bond between a Lithium-Disilicate Glass-Ceramic and the Titanium Abutment

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Purpose: To evaluate the effect of composite cement components and thermocycling on the bond strength of monolithic lithium-disilicate (LS2) glass-ceramic implant-supported restorations bonded to titanium (Ti) abutments.

Materials and Methods: Eighty LS2 blocks were treated with five types of composite cement and primer, then divided accordingly into groups: M (Multilink hybrid abutment), G (G-CEM LinkAce), GP (G-CEM LinkAce with G-Multi PRIMER), P (Panavia F2.0), and U (RelyX U200). Half of the 16 specimens from each group were subjected to thermocycling (groups T-M, T-G, T-GP, T-P, and T-U). The tensile bond strength (TBS) of all specimens was measured using a pull-off test. The cross section of the LS2 block from which the Ti abutment was removed was examined for mode of failure. Two-way ANOVA and Tukey's HSD test (significance level = 0.05) were used to determine the effect of composite cement composition and thermocycling on TBS.

Results: There was no difference in TBS between the five groups before thermocycling ($p = 0.16$). However, groups M ($p < 0.001$) and G ($p = 0.014$) showed significantly lower TBS than the corresponding thermocycled groups. Groups T-GP, T-P, and T-U did not show significant changes in TBS after thermocycling ($p > 0.05$). All failures occurred at the interface between the composite cement and Ti abutment and not between the cement and the LS2 block.

Conclusion: Thermocycling can reduce the bond strength between the composite cements and Ti abutment. The composite cements containing 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) or methacrylate phosphate ester monomers stabilize bonding.

Keywords: bond strength, cements, composite, lithium-disilicate glass-ceramic, thermocycling, titanium abutment.

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Monolithic lithium-disilicate (LS2) glass-ceramic crowns can be highly translucent and withstand mechanical stress.[3,18] When fabricated as a single structure, these partially crystallized glass-ceramics have a survival rate of 90% or more when observed over a period of 3-6 years.[4,24] In addition to possessing a fracture strength similar to metal-ceramic (porcelain-fused-to-metal) crowns, higher fracture resistance was reported compared to zirconia-veneering prostheses.[20,29] Moreover, for LS2 implant prostheses, sufficient prosthesis thickness can be secured to the natural tooth abutment, so that high fracture resistance can be expected.[32] Monolithic LS2 implant-supported restorations are prepared by bonding Ti inserts and bases with composite cement.[11,12,19] This process not only simplifies the fabrication, but also provides high fracture resistance due to the monolithic restoration. Previous studies have reported how to achieve stable adhesion between composite cements and LS2.[1,13,22,23,26,30,35] Several methods to improve adhesion between composite cement and metallic materials, including titanium, have also been studied. To increase the surface area, either a mechanical surface treatment method such as airborne-particle abrasion (APA), or a chemical surface treatment method (acid corrosion, methylene chloride solution, or application of a primer including a functional monomer).[6,8,12,21,34,36]

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Table 1 Description of materials used

Materials	Main composition	Manufacturer
Amber Mill	Lithium-disilicate glass-ceramic (SiO_2, Li_2O, K_2O, MgO, Al_2O_3, P_2O_3)	HASS
Sirona TiBase	Ti-6Al-4V	Dentsply Sirona
Multilink hybrid abutment	Bisphenol A diglycidyl methacrylate ethoxylated (bis-EMA), urethane dimethacrylate (UDMA), 2-hydroxyelthyl methacrylate, ytterbium trifluoride, dibenzoyl peroxide	Ivoclar Vivadent
G-CEM LinkAce	4-methacryloyloxyethyl trimellitic anhydride (4-META), UDMA, fluoro-alumino-silicate glass, pigment, dimethacrylate (DMA), distilled water, phosphoric ester monomer, initiator, and camphorquinone	GC
G-Multi PRIMER	10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), 10-methacryloyloxydecyldihydrogenthiophosphate (10-MDTP), 4-META	GC
Panavia F2.0	10-MDP, hydrophobic and hydrophilic DMA, silanized silica filler, sodium fluoride, camphorquinone, initiators	Kuraray Noritake
RelyX U200	Methacrylated phosphoric acid esters, TEG-DMA, alkaline basic fillers, initiator components, stabilizers and pigments	3M Oral Care

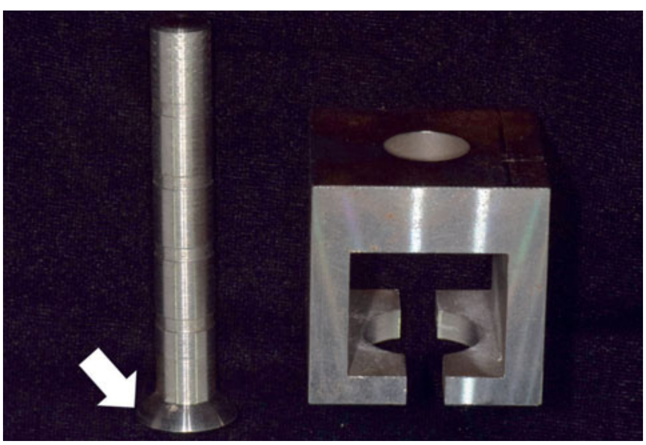

Fig 1 Customized metal jig. Arrow indicates the hemispherical bearing structure designed to uniformly subject all specimens to gravity along the y-axis.

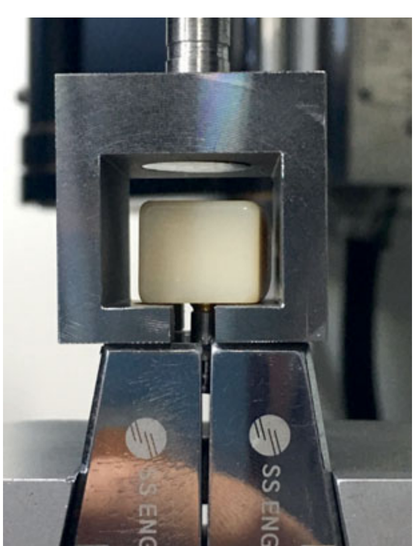

Fig 2 Arrangement of pull-off test.

Composite cement is composed of dimethacrylate-based monomers (eg, bisphenol A glycerolate dimethacrylate [bis-GMA], triethylene glycol dimethacrylate [TEG-DMA], and urethane dimethacrylate [UDMA]), fillers, initiators, and inhibitors. Depending on the components and their ratios in the composite cement, properties such as polymerization degree, viscosity, water absorption rate, and solubility differ.[2,7,14-16,33] When polymerized composite cement is exposed to the oral environment, absorption of water and dissolution of may occur, thus altering the properties of the composite cement due to hydrolysis of the components.[10,25] Although several studies have compared the bond strength of composite cement and its properties, a study on the bond strength between the LS2 and Ti abutment after thermocycling to reproduce the intra oral environment is needed.[5,9,17,27,28,31,37]

The purpose of the present in vitro study was to investigate the effect of composite cement components and thermocycling on the bond strength between LS2 and Ti abutment by measuring the tensile bond strength (TBS). The null hypothesis is that the type of composite cement used and thermocycling do not affect the TBS of LS2 and Ti abutments.

MATERIALS AND METHODS

Specimen Preparation

The inside of 80 LS2 blocks (Amber Mill, HASS; Gangneung, Korea) were mechanically milled and designed according to the shape of the implant Ti abutment (TiBase, Dentsply Sirona; Konstanz, Germany) were sintered. Eighty Ti abutments (TiBase, Dentsply Sirona) and laboratory analogs (3D-Analog, GeoMedi; Uiwang, Korea) were screwed together. For pretreatment, all LS2 (Amber Mill, HASS) blocks were etched and dried after being placed in 4% hydrofluoric acid (HF) etchant (4% Porcelain Etchant, Bisco; Schaumburg, IL, USA) for 30 s, and then dried for 1 min

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Table 2 Results of two-way ANOVA for tensile bond strength of lithium-disilicate glass-ceramic block to titanium abutment

Source	Type III Sum of Squares	Df	Mean Square	F	Significance
Cement	31.141	4	7.785	17.463	< 0.001
Thermocycling	3.635	1	3.635	8.153	0.006
Cement * thermocycling	16.939	4	4.235	9.499	< 0.001
Error	31.207	70	0.446		
Total	6516.466	80			
Corrected total	82.921	79			

Table 3 Mean (± SD) tensile bond strength (MPa) of composite cement according to presence or absence of thermocycling

Group	Cement	Thermocycling	Tensile bond strength (MPa)
M	Multilink Hybrid Abutment	–	8.92 ± 0.66^A
T-M	Multilink Hybrid Abutment	Thermocycling	7.16 ± 0.52^B
G	G-CEM LinkAce	–	9.03 ± 0.23^A
T-G	G-CEM LinkAce	Thermocycling	7.79 ± 0.82^B
GP,	G-CEM LinkAce with G-Multi RRIMER	–	9.13 ± 0.59^A
T-GP	G-CEM LinkAce with G-Multi RRIMER	Thermocycling	9.57 ± 0.21^A
P	Panavia F2.0	–	9.37 ± 0.49^A
T-P	Panavia F2.0	Thermocycling	9.91 ± 1.07^A
U	RelyX U200	–	9.46 ± 0.30^A
T-U	RelyX U200	Thermocycling	9.33 ± 1.08^A

Different superscript letters indicate statistically significant differences ($p < 0.05$).

after treatment with a silane coupling agent (Monobond N, Ivoclar Vivadent; Schaan, Liechtenstein) according to the manufacturer's instructions.

Table 1 shows the classification of experimental groups according to the composite cements and primers used: M (Multilink hybrid abutment, Ivoclar Vivadent); G (G-CEM LinkAce, GC; Tokyo, Japan); GP (G-CEM LinkAce with G-Multi PRIMER, GC); P (Panavia F2.0, Kuraray Noritake; Osaka, Japan); U (RelyX U200, 3M Oral Care; St Paul, MN, USA).

A total of 80 Ti abutments were cemented into the holes of the LS2 blocks using five kinds of composite cements. Sixteen specimens were prepared for each group, half of which were subjected to 6000 thermal cycles to reproduce the oral environment. The samples were immersed for 30 s each in 5°C and 55°C water baths and the transfer time was set to 2 s. Thermocycled groups are referred to as T-M, T-G, T-GP, T-P, and T-U.

Tensile Bond Strength (TBS)

A customized metal jig was fitted to hold LS2 blocks for bonding force measurements (Fig 1). The hemispherical bearing structure is designed so that at the interface between the upper and lower structures, all specimens are uniformly subjected to gravity along the y-axis (Fig 1, arrow). The specimen bonded to the metal jig was placed in a universal testing machine (Model 5982, Instron) and a pull-off test was performed in the longitudinal direction at a loading speed of 0.5 mm/min (Fig 2). The TBS was calculated in MPa by dividing this value by the area of the Ti abutment for each specimen (Table 3, Fig 3).

Failure Mode Analysis

Failures were analyzed by observing the surface of the separated Ti abutment and the inner surface of the LS2 block. Afterwards, one separated block was randomly selected from each group, and the cross section was observed using scanning electron microscopy (SEM, QUANTA FEG 250, FEI; Hillsboro, OR, USA; Fig 4).

Statistical Analysis

Statistical power analysis using G*Power 3.1 (Brunsbüttel, Germany) showed that the appropriate number of specimens required to ascertain the TBS was 8 per subgroup. To determine the effect of composite cement composition and thermocycling on TBS, two-way ANOVA was performed at a 95% confidence interval, followed by Tukey's post-hoc test. Data were analyzed using computer software (SPSS statistics v 23.0; Chicago, IL, USA).

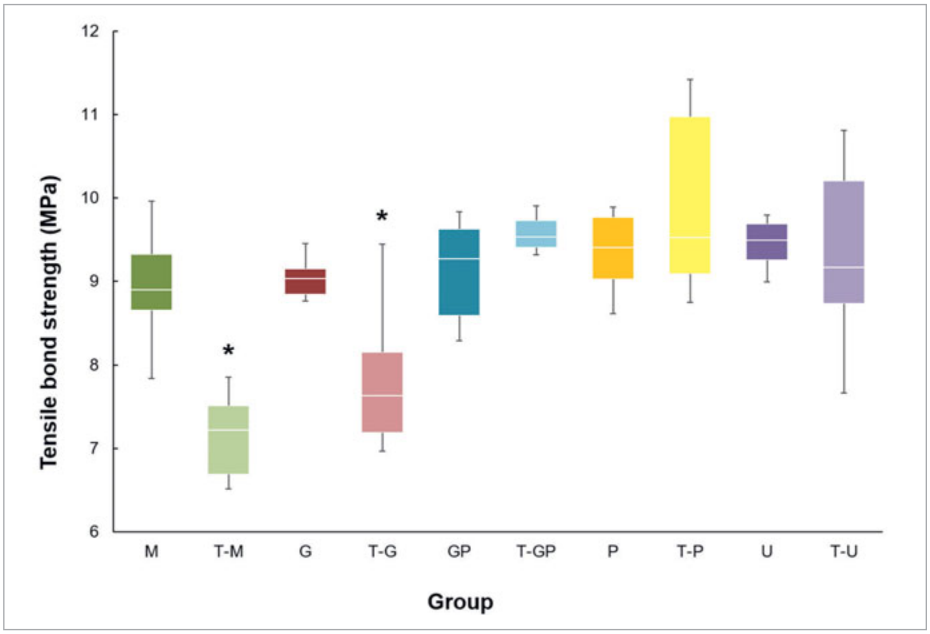

Fig 3 Box plot for comparing the mean tensile bond strength (TBS) by composite cement type and thermocycling. M: Multilink hybrid abutment, Ivoclar Vivadent; G: G-CEM LinkAce, GC; GP: G-CEM LinkAce with G-Multi PRIMER, GC; P: Panavia F2.0, Kuraray Noritake; U: RelyX U200, 3M Oral Care; T: thermocycling. *Statistically significantly lower TBS than the other groups.

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Fig 4 Cross section of lithium-disilicate (LS2) glass-ceramic block after pull-off test.

RESULTS

As a result of two-way ANOVA, significant differences were observed depending on cement type and thermocycling (with vs without; Table 2). TBS measurements from each experimental group are summarized in Table 3 and Fig 3. When comparing the bond strength of different composite cement compositions, no significant differences in TBS were observed between the groups without thermocycling ($p = 0.16$). However, after thermocycling, the T-M and T-G groups showed significantly lower TBS than did the T-GP, T-P, and T-U groups ($p < 0.05$). Specifically, the multilink hybrid abutment (groups M and T-M; $p < 0.001$) and G-CEM (groups G and T-G; $p = 0.014$) composite cements displayed significantly lower TBS after thermocycling. G-CEM LinkAce with G-Multi PRIMER (groups GP and T-GP groups), Panavia F2.0 (groups P and TP), and RelyX U200 (groups U and TU) composite cements did not exhibit significant changes in TBS after thermocycling ($p > 0.05$).

Adhesive failure between the Ti abutment and composite cement was observed in all specimens. Residual composite cement was observed on the bonded surface of the LS2 block, but not on the separated Ti abutment surface (Fig 4). The cross-sectional SEM image of the bonded interface obtained from a LS2 block cut from a Ti abutment also showed this failure pattern (Fig 5).

DISCUSSION

There was no significant difference in TBS between the experimental groups without thermocycling. However, groups T-GP, T-P, and T-U exhibited significantly higher bond strength than did groups T-M and T-G. For this reason, the different composite cement components and how they are influenced by thermocycling should be considered. Unlike multilink hybrid abutments that do not contain functional monomers in the composite cements, G-Multi PRIMER and Panavia F2.0 contain methacryloyloxydecyl dihydrogen phosphate (MDP), and RelyX U200 contains methacrylate phosphoric ester monomer. These functional monomers containing phosphate bonds play a major role in maintaining bond strength. Koizumi et al[13] reported that when primers containing 10-MDP were applied to titanium surfaces, the bond strength between titanium and the composite cement sig-

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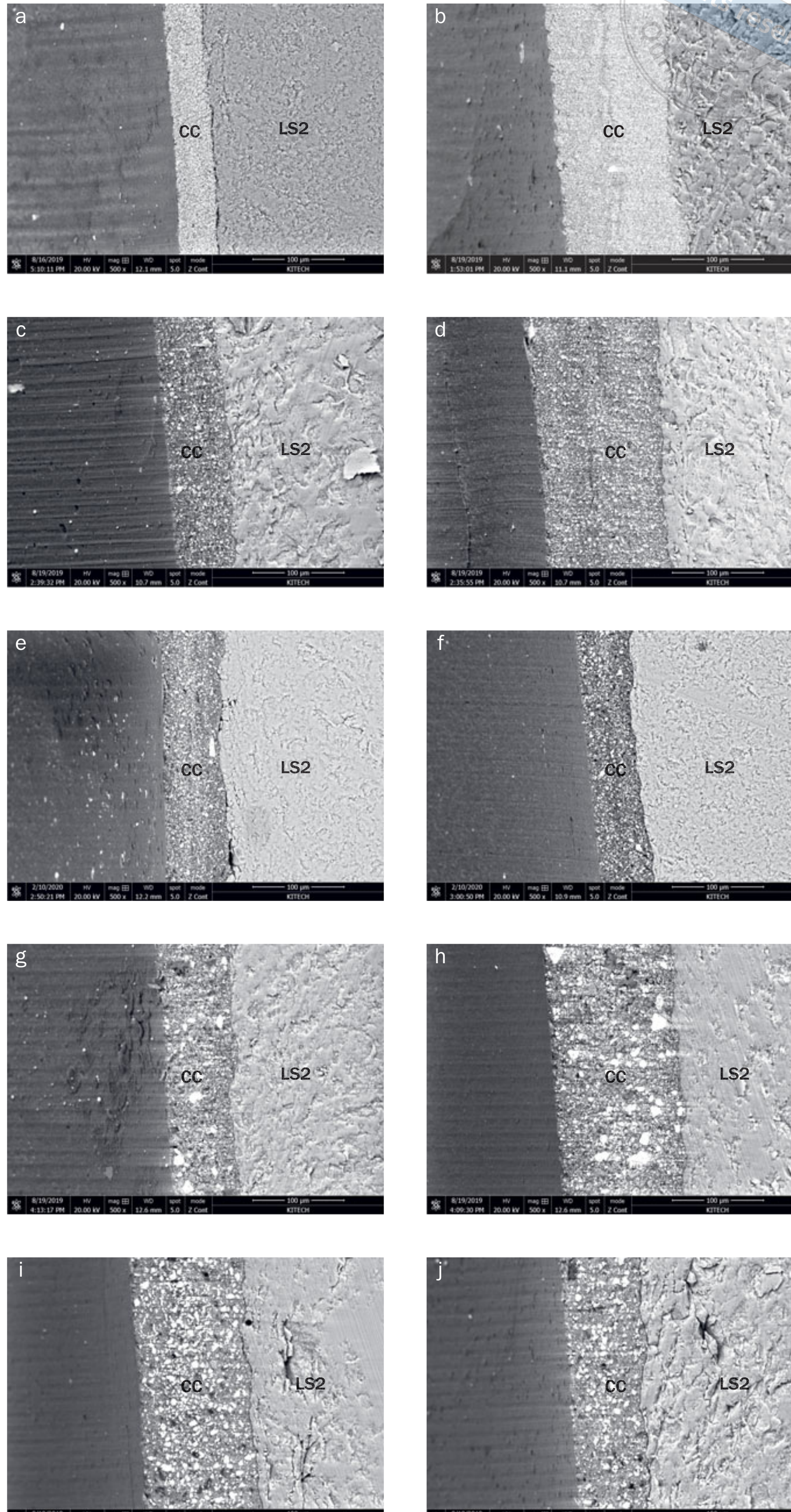
Fig 5 Scanning electron microscopy (SEM) images of the cut surface of LS2 glass ceramic block after failure (500X). a. ML group; b. T-ML group; c: G group; d: T-G group; e. GP group; f. T-GP group; g. P group; h. T-P group; i. U group; j. T-U group. CC: composite cement; LS2: lithium-disilicate.



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nificantly improved. The 10-MDP component in G-Multi PRIMER improves adhesion to precious metals.[36] The use of G-Multi PRIMER is not mandatory, but the T-GP group resulted in higher bond strengths than did the T-G group. Tsuchimoto et al[33] reported significantly higher bond strength between the composite cement and titanium when using primers containing MDP rather than primers containing 4-methacryloyloxyethyl trimellitic anhydride (4-META). G-CEM LinkAce contains functional monomers, 4-META, and phosphoric ester monomer.

Phosphate-based components in the composite cement and the primer may have a positive effect on the tensile bond strength with the titanium abutment. The methacrylate monomers responsible for the main polymerization reactions of composite cement are bis-EMA, UDMA, and TEG-DMA. The multilink hybrid abutment consists of ethoxylated bisphenol A dimethacrylate (bis-EMA) and urethane dimethacrylate (UDMA), G-CEM LinkAce consists of UDMA, and RelyX U200 consists of triethyleneglycol dimethacrylate (TEG-DMA). By comparison, TEG-DMA has a relatively low molecular weight and no intermolecular interactions (eg, hydrogen bonding), resulting in low viscosity and high polymerization rates due to the remaining fluid during polymerization. Gajewski et al[7] reported that the polymerization rate of TEG-DMA monomers is significantly higher than that of bis-GMA, UDMA, and bis-EMA monomers, indicating that the amount of unpolymerized monomers remaining is lower.

In studies comparing various composite cements, G-CEM cements possessed higher water sorption and residual monomer release than RelyX Unicem and Panavia F2.0. However, considering the results of groups G and GP, the detrimental effect of thermocycling on the bond strength can be reduced if the titanium surface is treated with a primer containing 10-MDP. Self-adhesive composite cements are characteristically initially hydrophilic due to their low pH, which gradually become hydrophobic due to pH neutralization as polymerization proceeds, although pH neutralization ability depends on the composite cement components. Roedel et al[25] reported the highest level of pH neutralization in RelyX Unicem 2, and Zorzin et al[37] reported that RelyX Unicem 2 showed higher levels of pH neutralization than G-CEM.

In this study, 6000 thermocycles were performed to simulate approximately 6 months of oral conditions. Because prostheses are exposed to a variety of harsh mechanical and chemical environments, it is therefore clinically important to select a composite cement that is more resistant to external influences for the long-term success of the prostheses. The SEM image of the LS2 block (Fig 5) indicates that adhesion between the composite cement and LS2 is stable, although adhesive faiure occurred between the composite cement and the Ti abutment (Fig 4). Various titanium surface treatment methods have been studied to increase the bond strength between these titanium and composite cements.[6,8,13,21,30,33] As mentioned earlier, high bond strengths have been reported with the use of phosphate-containing primers.[13] APA on the titanium surface provided stronger bond strength with composite cement than use of methylene chloride, 10% hydrogen peroxide, and 9% HF.[6] Guilherme et al[8] also reported high bond strength when 9.5% HF was applied. In addition, Özcan et al[21] reported the effect of spraying silica powder on a titanium surface to deposit silica particles.

In this study, no mechanical surface treatment was performed on the Ti abutment surface in order to focus on the bond strength between the primer and the composite cement components on the titanium surface. APA or primer treatment on the Ti abutment surface is recommended.[6,8] This enables improved bonding between Ti abutment and composite cement. Several studies have confirmed the effectiveness of APA.[21,30,34] Therefore, we expected that APA would counteract or mask the effects of thermocycling and the inherent effects of composite cement components. We wanted to quantify the bond strength with the Ti-Base as a function of the composition of the composite cement when this conditioning effect was intentionally blocked. In addition, thermocycling was applied to simulate the most unfavorable conditions. In particular, using a customized pull-off test device with the addition of a hemispherical cylinder, we strove to minimize errors that could cause the bond strength to be overestimated. As a result, a careful execution of the pull-off test in the direction of the Ti-base axis without APA treatment yielded low bond strength results. In addition, the effects of thermocycling become more evident.

Therefore, further studies are needed to improve the bond strength in combination with mechanical surface treatments. Another limitation of this experiment involves the tolerance of cement spaces formed from specimens for SEM images. Although errors in the measured value may occur depending on the direction the specimen was cut, the observed cement space was between 50 and 100 μm. In the future, precise milling could be employed for further improvements to reduce measurement errors. In spite of these shortcomings, significant differences in bond strength were observed related to the composite cement composition.

CONCLUSION

Overall, the TBS between the composite cement and the Ti abutment was affected by the composite cement composition, primer, and thermocycling, and the effect of thermocycling was different depending on the composition of the composite cement. TBS increased when the Ti abutment surface was treated with 10-MDP or phosphate-containing primer. Bonding between LS2 and the composite cement is stable, but the bond strength between the Ti abutment and the composite cement is relatively poor, which is the main cause of bond failure.

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Clinical relevance: The bond between a composite cement and the titanium abutment can be weakened by thermocycling. Composite cements or primers containing 10-MDP or phosphate monomers can prevent bond weakening due to thermocycling.