Silane primers rather than heat treatment contribute to adhesive bonding between tri-*n*-butylborane resin and a machinable leucite-reinforced ceramic

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The purpose of the present study was to evaluate the effects of silane primers with and without heat treatment on bonding between a resin and a leucite-reinforced ceramic (GN-Ceram). Six dental primers (GC Ceramic Primer, GP; Clearfil Ceramic Primer, CP; RelyX Ceramic Primer, RP; Tokuso Ceramic Primer, TP; Shofu Porcelain Primer, SP; and Porcelain Liner M, PM) and five experimental primers (MDS, MTS, MDES, MTES, and ATS) were evaluated. GN-Ceram specimen was primed, heated at 100°C for 60 min (Heat), and then bonded to a resin composite using a self-curing resin. Shear bond testing revealed that GP, GP/Heat, CP, CP/Heat, RP, RP/Heat, TP, TP/Heat, SP, SP/Heat, PM, PM/Heat, MDS/Heat, MTS, MTS/Heat, and ATS/Heat exhibited superior bond strengths. No-primer, No-primer/Heat, MDS, MDES, MTES, and ATS exhibited low bond strengths, while there were no significant differences in bond strength among SP, MDS, MDES/Heat, and MTES/Heat. It seemed that heat treatment improved the bonding performance for MDS, MTES, and ATS only.

Keywords: Adhesive bonding, Ceramics, Surface modification

INTRODUCTION

To meet the growing aesthetic demands of discerning patients, ceramic has become the predominant material of choice for the restoration of anterior and posterior teeth. The computer-aided design and computer-aided manufacturing (CAD/CAM) revolution has further boosted the use of ceramics for crown restorations and implant-supported prostheses¹. Dental CAD/CAM technology circumvents several shortcomings associated with the use of dental ceramics. Amongst which, the problem of potential marginal inaccuracy caused by the sintering shrinkage of conventional feldspathic ceramics can be avoided through the use of CAD/CAM systems. Since CAD/CAM restorations (such as crowns or copings) are machined from homogeneous and factory-standardized ceramic blocks, they retain the original physical properties of the starting material.

Several popular CAD/CAM systems, such as Aadvia (GC Corp., Tokyo, Japan), GN-I (GC Corp.), Decsy (Media Inc., Tokyo, Japan), and Cerec 3 (Sirona Dental Systems GmbH, Bensheim, Germany), use silica-based ceramic blocks. GN-Ceram (GC Corp.), a leucite-reinforced silica-based ceramic block for Aadvia and GN-I CAD/CAM systems, is designed for the fabrication of entire restorations—including the occlusal surfaces. GN-Ceram block consists of SiO₂, Al₂O₃, K₂O, Na₂O, and CaO as the major components. It has a glass matrix reinforced with a leucite crystalline phase (K₂O·Al₂O₃·4SiO₂)².

Adhesive bonding between ceramic restorations and abutment teeth is often enhanced using silane primers³. Silane monomers used to improve ceramic bonding included tris(2-methoxyethoxy)vinylsilane (which

was used to treat silica powder)⁴ and 3-methacryloxypropyltrimethoxysilane (MTS) which can be activated by inorganic acids, organic acids, or water⁵⁻⁸. It was reported that a commercially available silane primer containing MTS improved the bond strength between a silica-based ceramic and certain resin-based luting agents^{9,10}. Silane monomers that have been evaluated for ceramic bonding included 1-methacryloxymethyltrimethoxysilane, 3-acryloxypropyltrimethoxysilane, 3-methacryloxypropyltrichlorosilane, 3-(4-methacryloxyphenyl)propyltrichlorosilane, and a combination of 3-methacryloxypropylsilyltriisocyanate and 3-mercaptopropyltrimethoxysilane¹¹⁻¹³.

It was further reported that heat treatment of silane-treated ceramics could maximize their bond strength¹⁴⁻¹⁶. Therefore, in the fabrication of resin composites (which are generally composed of silanated particulate glass/ceramic fillers) and industrial glass products, post-silanization heat treatment is routinely performed on silanated ceramics.

The properties and effects of silane monomers are defined by their chemical structures. Although a certain degree of variation exists among silane monomers, there is a general paucity of information regarding the effect of heat treatment on the generic chemical structure of silane monomers and the eventual effect on the bonding capability of ceramics. Specifically, there begs the ultimate question if silane primers require heat activation to achieve optimal bonding.

The purpose of the present study was to investigate the efficacy of heat treatment in improving the bonding performance of silane primers. A machinable glass ceramic reinforced with leucite crystallites was treated with one of 11 silane monomer-containing primers (six commercially available and five experimental primers). With or without post-silanization heat treatment,

primer-treated ceramics were bonded using a tri-*n*-butylborane-initiated self-curing luting agent and their bond strengths evaluated.

MATERIALS AND METHODS

Materials

The substrate materials and luting agent used in the present study are listed in Table 1. A leucite-reinforced ceramic (GN-Ceram, GC Corp.) (Fig. 1) and a resin composite (Clearfil DC Core Automix, Kuraray Medical Inc., Tokyo, Japan) were used as the substrate materials. The luting agent used was a self-curing unfilled resin (MMA-TBB resin) containing methyl methacrylate (MMA) monomer and tri-*n*-butylborane (TBB) initiator, but no functional monomer.

The six commercially available dental silane primers (GC Ceramic Primer, GP; Clearfil Ceramic Primer, CP; RelyX Ceramic Primer, RP; Tokuso Ceramic Primer, TP; Shofu Porcelain Primer, SP; and Porcelain Liner M, PM) and five experimental silane primers (MDS, MTS, MDES, MTES, and ATS) used in the present study are listed in Table 2. The structural formulae of the silane monomers used in the experimental primers are shown in Fig. 2. All primers were single-bottle primers, except for GP, TP, and PM which were two-bottle primers.

Specimen preparation

1. Ceramic substrate specimens

A total of 144 rectangular disk specimens (8.0 mm × 7.0 mm × 3.0 mm thickness) were cut from GN-Ceram block using a high-speed air turbine (J. Morita Corp., Osaka, Japan) and a diamond point (GC Corp.). Surfaces of the GN-Ceram specimens were ground using 600-grit silicon carbide abrasive papers and then rinsed with water. A 40wt% phosphoric acid solution was applied to each specimen surfaces for 5 s, followed by a water rinse for 15 s. Specimens were randomly divided into 12 groups, 11 primer groups and one No-primer group, with 12

specimens in each group.

Onto the polished and rinsed surface of each ceramic substrate was applied 1 µL of each primer using a micropipette (Eppendorf AG, Hamburg, Germany). In the No-primer group, 12 control specimens with no primer application were prepared. Within each group, half of the specimens were baked at 100°C for 60 min in a furnace (KL100, Kuraray Medical Inc.), thereby yielding six heat-treated ceramic specimens for each of these groups: GP/Heat, CP/Heat, RP/Heat, TP/Heat, SP/Heat, PM/Heat, MDS/Heat, MTS/Heat, MDES/Heat, MTES/Heat, ATS/Heat, and No-primer/Heat. The remaining specimens in each group were not heat-treated.

2. Resin composite substrate specimens

A total of 144 round disk specimens (7.0 mm diameter, 3.0 mm thickness) were fabricated by polymerizing Clearfil DC Core Automix resin in a silicon mold using a

Fig. 1 Leucite-reinforced ceramic block (GN-Ceram) used in the present study.

Table 1 Substrate materials and luting agent used in the present study

Material	Composition	Manufacturer	Lot No.
Substrate material			
GN-Ceram	Silicon dioxide, aluminum oxide, potassium oxide	GC Corp., Tokyo, Japan	0611141
Clearfil DC Core Automix	Bis-GMA ^a , TEGDMA ^b , filler (barium glass, silicate glass), photoinitiator, chemical initiator	Kuraray Medical Inc., Tokyo, Japan	0083AA
Luting agent			
MMA-TBB resin	MMA ^c , tri- <i>n</i> -butylborane, PMMA ^d	Wako Pure Chemical Ind. Ltd., Osaka, Japan Sun Medical Co. Ltd., Moriyama, Japan Sun Medical Co. Ltd.	TWQ5264 RR22 RR11

^a Bis-GMA: bisphenol-A-diglycidylether dimethacrylate

^b TEGDMA: triethyleneglycol dimethacrylate

^c MMA: methyl methacrylate

^d PMMA: polymethyl methacrylate

Table 2 Primers used in the present study

Primer (Abbreviation)	Composition	Manufacturer	Lot No.
GC Ceramic Primer (GP)	Primer A: vinyl silane, ethanol Primer B: methacrylate, ethanol	GC Corp., Tokyo, Japan	0805192
Clearfil Ceramic Primer (CP)	MTS ^a , MDP ^b , ethanol	Kuraray Medical Inc., Tokyo, Japan	0007BA
RelyX Ceramic Primer (RP)	Silane monomer, ethanol, water	3M ESPE, Seefeld, Germany	2E+07
Tokuso Ceramic Primer (TP)	Primer A: silane monomer, ethanol Primer B: phosphate monomer, ethanol	Tokuyama Dental Corp., Tokyo, Japan	012038
Shofu Porcelain Primer (SP)	Silane monomer, dehydrated ethanol, others	Shofu Inc., Kyoto, Japan	020803
Porcelain Liner M (PM)	Liquid A: 5% 4-META ^c , MMA ^d Liquid B: 4% MTS ^a , MMA ^d	Sun Medical Co. Ltd., Moriyama, Japan	SM2
MDS primer	2% MDS ^e MMA ^d	Shin-Etsu Chemical Co., Tokyo, Japan Wako Pure Chemical Ind. Ltd., Osaka, Japan	904346 TWQ5264
MTS primer	2% MTS ^a MMA ^d	Shin-Etsu Chemical Co. Wako Pure Chemical Ind. Ltd.	907402 TWQ5264
MDES primer	2% MDES ^f MMA ^d	Shin-Etsu Chemical Co. Wako Pure Chemical Ind. Ltd.	902074 TWQ5264
MTES primer	2% MTES ^g MMA ^d	Shin-Etsu Chemical Co. Wako Pure Chemical Ind. Ltd.	907027 TWQ5264
ATS primer	2% ATS ^h MMA ^d	Shin-Etsu Chemical Co. Wako Pure Chemical Ind. Ltd.	909301 TWQ5264

^a MTS: 3-methacryloxypropyltrimethoxysilane

^b MDP: 10-methacryloxydecyl dihydrogen phosphate

^c 4-META: 4-methacryloxyethyl trimellitate anhydride

^d MMA: methyl methacrylate

^e MDS: 3-methacryloxypropylmethyldimethoxysilane

^f MDES: 3-methacryloxypropylmethyldiethoxysilane

^g MTES: 3-methacryloxypropyltriethoxysilane

^h ATS: 3-acryloxypropyltrimethoxysilane

light curing apparatus (α -Light II, J. Morita Corp.) for 180 s.

3. Bonded specimens

A 50- μ m-thick piece of polyester tape with a circular hole of 4 mm diameter was affixed to each rectangular ceramic disk to define the bonding area (Fig. 3). The ceramic specimen was bonded to the resin composite disk with MMA-TBB resin using the brush-dip technique. Bonded specimens for a total of 24 groups (No-primer, No-primer/Heat, GP, GP/Heat, CP, CP/Heat, RP, RP/Heat, TP, TP/Heat, SP, SP/Heat, PM, PM/Heat, MDS, MDS/Heat, MTS, MTS/Heat, MDES, MDES/Heat, MTES, MTES/Heat, ATS, and ATS/Heat) were prepared. All bonded specimens were stored at room temperature for 60 min. Then, they were immersed in water at 37°C for 24 h before being subjected to shear bond strength test.

Shear bond strength test

Each bonded specimen was embedded in a plastic mold with an acrylic resin (Unifast III, GC Corp.). The whole assembly was then seated in an ISO/TR 11405 shear testing jig (Wago Industrial Ltd., Nagasaki, Japan). Shear bond strength was determined using a universal testing device (AGS-10kNG, Shimadzu Corp., Kyoto, Japan) at a crosshead speed of 0.5 mm/min.

Mean bond strength and standard deviation of six specimens were calculated for each group. Statistical analysis was performed using two-way factorial analysis of variance (ANOVA), in which heat treatment and primer served as independent factors. The mean values were analyzed using Tukey's test ($\alpha=0.05$, $n=6$) following one-way ANOVA.

Failure mode analysis

Debonded specimen surfaces were observed using an

optical microscope (SMZ-10, Nikon Corp., Tokyo, Japan) at 20× magnification to determine failure mode. Failure modes were categorized as follows: Adhesive failure at ceramic-luting agent interface (Ad); cohesive failure within luting agent (Co); fracture of the ceramic (Frac); and combinations thereof (Ad/Co, Ad/Frac, and Ad/Co/Frac).

Fig. 2 Structural formulae of the experimental silane monomers used in the present study.

RESULTS

Shear bond strength

ANOVA revealed that shear bond strength was significantly influenced by heat treatment and primer application, and that their interaction was also significant (Table 3). The means and standard deviations of shear bond strength are summarized in Table 4, ranging between 13.9 and 53.0 MPa. GP, GP/Heat, CP, CP/Heat, RP, RP/Heat, TP, TP/Heat, SP, SP/Heat, PM, PM/Heat, MDS/Heat, MTS, MTS/Heat, and ATS/Heat exhibited superior bond strengths with no significant differences in the range of 39.4–53.0 MPa. Although SP, MDS, MDES/Heat, and MTES/Heat showed no significant differences, No-primer, No-primer/Heat, MDS, MDES, MTES, and ATS exhibited low bond strengths with no significant differences in the range of 13.9–26.6 MPa.

Among the five experimental silane primers, MTS yielded the highest bond strength—with or without heat treatment. Compared to No-primer/Heat, all of the other heat treatment groups exhibited significantly higher bond strengths. No significant differences were detected between the primers with and without heat treatment, except for MDS, MTES, and ATS. MDS/Heat, MTES/Heat, and ATS/Heat specimens showed significantly higher bond strengths than those of MDS, MTES, and ATS, respectively.

Failure modes

Table 5 summarizes the failure modes of the specimens in this study. 14 groups (No-primer, No-primer/Heat, RP, TP/Heat, SP/Heat, PM/Heat, MDS, MDS/Heat, MDES, MDES/Heat, MTES, MTES/Heat, ATS, and ATS/Heat) exhibited complete adhesive failure at the ceramic-luting agent interface (Ad). Complete or partial fracture within the ceramics (Frac and Ad/Frac) was observed in six specimens of five groups (GP, CP, SP, MTS, and RP/Heat). A combination of adhesive failure and cohesive failure within the luting agent (Ad/Co) was observed in 21 specimens of seven groups (TP, PM, MTS, GP/Heat, CP/Heat, RP/Heat, and MTS/Heat). No complete cohesive failures (Co) were observed.

Fig. 3 Schematic illustration of bonding procedure and shear bond strength test.

Table 3 Analysis of variance results

Source of variation	d.f.	Sum of squares	Mean square	F-value	P-value
Heat treatment	1	1,029.5	1,029.5	23.8	0.0001
Primer	11	16,862.8	1,533.0	35.5	0.0001
Heat treatment/Primer	11	3,129.1	284.5	6.6	0.0001
Residual	120	5,185.7	43.2		

Table 4 Shear bond strengths of MMA-TBB resin to GN-Ceram

No heat treatment		Heat treatment	
Group name	Mean (SD) ^a (MPa)	Group name	Mean (SD) ^a (MPa)
No-primer	15.2 (3.9) ^{AB}	No-primer/Heat	13.9 (6.0) ^A
GP	44.4 (12.3) ^{FG}	GP/Heat	44.3 (2.1) ^{FG}
CP	47.1 (3.0) ^{FG}	CP/Heat	53.0 (4.8) ^G
RP	52.6 (8.3) ^G	RP/Heat	42.6 (5.4) ^{EFG}
TP	44.1 (3.7) ^{FG}	TP/Heat	48.2 (5.7) ^{FG}
SP	39.4 (10.0) ^{DEFG}	SP/Heat	44.8 (10.2) ^{FG}
PM	44.9 (2.5) ^{FG}	PM/Heat	42.7 (3.7) ^{FG}
MDS	26.6 (5.5) ^{ABCD}	MDS/Heat	44.0 (4.5) ^{FG}
MTS	48.5 (6.7) ^{FG}	MTS/Heat	44.8 (4.1) ^{FG}
MDES	19.1 (8.2) ^{AB}	MDES/Heat	28.5 (11.8) ^{BCDE}
MTES	14.4 (7.4) ^A	MTES/Heat	37.2 (3.9) ^{CDEF}
ATS	24.4 (6.1) ^{ABC}	ATS/Heat	40.9 (2.6) ^{EFG}

^a Identical capital letters indicate values are not significantly different statistically ($p>0.05$).

Table 5 Failure modes of specimens

No heat treatment		Heat treatment	
Group name	Mode ^a (Number of specimens)	Group name	Mode ^a (Number of specimens)
No-primer	Ad(6)	No-primer/Heat	Ad(6)
GP	Ad(5), Ad/Frac(1)	GP/Heat	Ad(5), Ad/Co(1)
CP	Ad(5), Frac(1)	CP/Heat	Ad(1), Ad/Co(5)
RP	Ad(6)	RP/Heat	Ad(4), Ad/Co(1), Ad/Frac(1)
TP	Ad(1), Ad/Co(5)	TP/Heat	Ad(6)
SP	Ad(4), Ad/Frac(2)	SP/Heat	Ad(6)
PM	Ad(2), Ad/Co(4)	PM/Heat	Ad(6)
MDS	Ad(6)	MDS/Heat	Ad(6)
MTS	Ad(1), Ad/Co(4), Frac(1)	MTS/Heat	Ad(5), Ad/Co(1)
MDES	Ad(6)	MDES/Heat	Ad(6)
MTES	Ad(6)	MTES/Heat	Ad(6)
ATS	Ad(6)	ATS/Heat	Ad(6)

^a Ad: Adhesive failure at ceramic-luting agent interface; Co: Cohesive failure within luting agent; Frac: Fracture within ceramic.

DISCUSSION

The present study showed that the use of an appropriate silane primer alone, without additional heat treatment, was adequate and possible to achieve a strong bonding between a leucite-reinforced ceramic (GN-Ceram) and a

self-curing resin. A silane monomer has a degradable functional group and a polymerizable C=C double bond that are able to bind silicon dioxide (SiO₂) and methacrylates. GN-Ceram consisted of 59–60wt% SiO₂ as well as several other oxides (Al₂O₃, K₂O, Na₂O, and CaO)⁽²⁾. When the functional group of a silane monomer

is hydrolyzed with water molecules, the hydrolyzed silane monomer reacts with hydroxyl groups of the ceramic surface, forming covalent bonds or hydrogen bonds^{5,8)} (Fig. 4).

For the six commercial silane primers used in this study, no information was provided by their manufacturers on the types of silane monomers contained in the primers, except for PM and CP which indicated that the silane monomer used was 3-methacryloxypropyltrimethoxysilane. Therefore, to evaluate not only the effect of dental silane primers on bond strength, but also the role of the chemical structure of silane monomers, five experimental primers containing already-known silane monomers (MDS, MTS, MDES, MTES, and ATS) were used in this study.

Comparison of the silane primers MDS, MTS, MDES, and MTES suggested that the chemical structure of silane monomers affected the adhesive force between silane monomers and ceramic substrates. There were no significant differences in bond strength between MDS and MDES. However, MTS, which contained methoxy groups, exhibited remarkably higher bond strength than MTES, which contained ethoxy groups. Compared to the ethoxy group, it was probable that the methoxy group was more easily hydrolyzed and more readily converted into the silanol (Si-OH) group. The number of methoxy groups in the chemical structure also influenced bond strength. Therefore, MTS with three methoxy groups exhibited higher bond strength than MDS, which contained only two methoxy groups.

Results obtained in the present study supported a previous report that ATS improved the bonding between a glass plate and a resin composite¹²⁾. Although the chemical structures of ATS and MTS were similar, ATS and MTS had an acryl group and a methacryl group

respectively. The observed differences in bond strength between MTS, ATS, and ATS/Heat suggested that the chemical interaction between MTS and GN-Ceram occurred more readily than between ATS and GN-Ceram. In addition, the copolymerization of MTS with MMA might have proceeded more readily than that between ATS and MMA.

Heat treatment caused excess water to evaporate, thereby promoting the condensation of silane monomers on a ceramic surface. This then accelerated the chemical interaction between the silane monomers and the ceramic surface^{15,17)}, thereby causing MDS/Heat, MTES/Heat, and ATS/Heat to have improved bond strengths in the present study. According to previous reports^{14,15,17)} and based on our preliminary findings, a heat treatment of 100°C for 60 min in a furnace was employed in this study.

Silane monomers could also be activated by catalytic components in primers^{6-8,18)}. For primers CP, RP, and TP, their catalytic components should be the phosphate monomer and water, thereby accounting for their superior bond strengths. Similarly, primers GP and SP could have contained some catalytic components to activate the silane monomers, although no definitive information was published. Both PM and MTS primers contained 3-methacryloxypropyltrimethoxysilane as a silane monomer, but only PM primer contained 4-META as a catalytic component. There were no significant differences in bond strength ($p>0.05$) between 4-META-containing PM primer and non 4-META-containing MTS primer. This showed the negligible effect of 4-META on initial bond strength, and this result agreed with a pre-thermocycling porcelain bonding result in another study¹⁹⁾.

GP, CP, RP, TP, and SP had ethanol as their solvent,

Fig. 4 Hypothetical chemical interaction between 3-methacryloxypropyltrimethoxysilane (MTS) and ceramic surface.

while PM was an MMA-based primer. Nonetheless, these primers exhibited superior bond strengths with no significant differences, suggesting that both of these solvents were equally acceptable.

From a clinical viewpoint, results of the present study showed that a dental silane primer (GP, CP, RP, TP, SP, or PM) was effective in improving the adhesive bonding of MMA-TBB resin to GN-Ceram. Nonetheless, failure mode analysis revealed that adhesive force at the resin-ceramic interface was lower than the cohesive strength of resin: nearly all specimens exhibited either complete or partial adhesive failure. For this reason, it would be necessary to include an additional surface roughening treatment, such as etching and sandblasting, to further improve resin-ceramic bond strength^{20,21}.

CONCLUSIONS

Within the limitations of the present study, the following conclusions were drawn:

1. Heat treatment improved the bonding performances of MDS, MTES, and ATS primers only.
2. Chemical components of a silane primer, rather than heat treatment, contributed to optimal bonding between a tri-*n*-butylborane-initiated self-curing resin and leucite-reinforced ceramic, GN-Ceram.

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