Effect of silane and phosphate primers on the adhesive performance of a tri-*n*-butylborane initiated luting agent bonded to zirconia

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The purpose of this study was to evaluate the effects of primers on the bond strength and durability of an acrylic resin luting agent bonded to zirconia. Disk specimens were fabricated from zirconia partially stabilized with yttrium oxide. The disks were primed with one of the following materials: Alloy Primer (AP), Ceramic Primer (CP), Liquid A of the Porcelain Liner M (PLM-A), Liquid B of Porcelain Liner M (PLM-B), Porcelain Liner M (PLM-A+PLM-B), Monobond Plus (MP), and mixture of AP and PLM-B. The specimens were bonded with a tri-*n*-butylborane (TBB)-initiated luting agent. The shear bond strengths were determined both before and after thermocycling. The results were statistically analyzed with a non-parametric procedure. The highest post-thermocycling bond strength was generated from the groups primed with MP, CP, and AP. It can be concluded that the application of three phosphate primers is recommended for bonding the zirconia with the TBB-initiated luting agent.

Keywords: Bonding, Phosphate, Silane, Tri-*n*-butylborane, Zirconia

INTRODUCTION

Over the last decade, several all-ceramic systems have become established on the market, from metal-free restoration alternatives to wide spread metal-ceramic composite structures. Silica-based ceramics (leucite-reinforced feldspathic porcelain and lithium disilicate glass-ceramic) and oxide ceramics (glass-infiltrated aluminum-oxide ceramic, densely sintered aluminum-oxide, and glass-infiltrated zirconium-oxide ceramic) have been widely applied for conventional ceramic restorations¹.

High-strength ceramic zirconia is the latest development in ceramic framework materials². Zirconia has better mechanical properties than all of the previously introduced ceramics³. Furthermore, much clinical research into zirconia-based all-ceramic restorations has confirmed that zirconia ceramics exhibit high stability as a frame work material for full coverage crowns⁴⁻⁷, fixed partial dentures (FPDs)^{5,8-11}, and implant abutments¹²⁻¹⁴.

It has often been reported that full-coverage zirconia ceramic restoration and FPDs might not require adhesive cementation and that they can be cemented with conventional cements, including zinc phosphate^{4,6,8,11} or glass ionomer cement^{5,10}. However, the advantages of resin luting agents, *e.g.*, marginal seal, good retention, and improvement of fracture resistance have made them increasingly frequently used, even for high-strength

ceramics^{4,6,9}. Moreover, a sufficient resin bond has the aforementioned advantages and can become necessary in some clinical situations, such as for compromised retention and short abutment teeth.

Derand *et al.* suggested that present surface treatments (silica coating, air borne particle abrasion, and hydrofluoric acid treatment) have no positive effects on the resin bonds to zirconia ceramics¹⁵. However, several studies have proved that the application of luting cements and primers containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) results in a significantly greater bond strength to zirconia than with agents not containing MDP^{1,16-19}.

The current silane coupling agents (one-liquid or two-liquid type primers) contain some acidic monomers, including 4-methacryloyloxyethyl trimellitate anhydride (4-META) or MDP activated γ -MPTS²⁰. Therefore, acidic monomers have improved the wettability of the ceramic surface and have also enhanced bond strength^{21,22}. However, there have been frequent discussions about how silane coupling agents can affect the resin bond to conventional silica-based dental ceramics, resulting in unsatisfactory resin bonds to oxide-based dental ceramics^{23,24}. Similarly, the silanization of zirconia can improve the wetting ability of the surface and thereby result in small but increased bond strength values²⁵.

The aims of this study were to evaluate the effects of the chemical ingredients in primers on the bond strength and durability of a tri-*n*-butylborane (TBB)-initiated acrylic resin joined to a zirconia restorative

Color figures can be viewed in the online issue, which is available at J-STAGE.

Received Dec 10, 2013; Accepted Jan 7, 2014

doi:10.4012/dmj.2013-346 JOI JST.JSTAGE/dmj/2013-346

material and to investigate the effects of the addition of a combination of a silane coupling agent and a phosphate monomer to zirconia on adhesion. The following hypotheses were tested: (1) phosphate monomer MDP would enhance the bond strength to zirconia; (2) in determining the strength of bonding to zirconia, silane coupling treatment would be uninfuential; and (3) in terms of the bonding to zirconia, silane coupling treatment would inhibit the effects of phosphate monomer MDP.

MATERIALS AND METHODS

Materials

Yttrium oxide partially stabilized zirconia (YPS zirconia, 94.4% ZrO_2 and 5.4% Y_2O_3 , Katana, Kuraray Noritake Dental, Tokyo, Japan), designed for fixed prosthesis frameworks, was used as the adherend material (Table 1). Five priming agents, including Alloy Primer, Clearfil Ceramic Primer (Kuraray Noritake Dental), Porcelain Liner M (Liquid A and B, Sun Medical, Moriyama, Japan), and Monobond Plus (Ivoclar Vivadent, Shaaan, Liechtenstein) were assessed as bonding promoters. Porcelain Liner M is a two-component ceramic primer, whereas the other primers are single-liquid and contain at least one adhesive functional monomer, *i.e.*, MDP, VTD, γ -MPTS, and 4-META, respectively (Table 1). A methyl methacrylate (MMA)-based self-polymerizing resin initiated with partially oxidized tri-*n*-butylborane (TBB) was used as the luting agent. The TBB initiator and the powder component of this material were the

same as those of Super-Bond C&B resin (Sun Medical). A 99.8% MMA (Wako Pure Chemical, Osaka, Japan), which did not contain any functional monomers, was chosen as the monomer liquid of the MMA-TBB resin used in this study because the purpose of the current study was to evaluate the functional monomers in the priming agents.

Specimen preparation and shear bond strength

Figure 1 shows the procedure for the preparation of the specimens and shear bond testing.

A total of 154 disk specimens (11.4 mm in diameter, 3 mm in thickness) were prepared from zirconia and were divided into seven sets of 22 disks (groups 1 through 7, Table 2). All the disks were wet-ground with 1500-grit silicon carbide (Wet or Dry Tri-M-ite, 3M, St. Paul, MN, USA) abrasive paper, ultrasonically cleaned in distilled water, and then dried in a vacuum desiccator. A piece of double-coated tape with a circular hole 5 mm in diameter was placed on the disk surface of the 11.4 mm-diameter-zirconia disk to define the bonding area.

The specimen disks were primed with one of the following systems: Alloy Primer (AP), Ceramic Primer (CP), Liquid A of the Porcelain Liner M (PLM-A), Liquid B of Porcelain Liner M (PLM-B), Liquids A and B of Porcelain Liner M (PLM-A+PLM-B), Monobond Plus (MP), and a mixture of AP and PLM-B (AP+PLM-B) (Table 2).

A stainless steel ring (SUS303) with an inner diameter of 6 mm, and a 2-mm-high and 1-mm-thick wall was positioned around the 5-mm-diameter circular

Table 1 Materials used in this study

Material	Product name (Abbr.)	Manufacturer	Lot number	Composition (%)
Adherend material	Noritake Katana Zirconia Frame	Kuraray Noritake Dental Inc., Tokyo, Japan	200218	94.4 ZrO_2 , 5.4 Y_2O_3
Primer	Monobond Plus (MP)	Ivoclar Vivadent AG, Schaan, Liechtenstein	M44233	MDP, γ -MPTS, Sulfide methacrylate, Ethanol
	Clearfil Ceramic Primer (CP)	Kuraray Noritake Dental Inc.	00012D	MDP, γ -MPTS, Ethanol
	Alloy Primer (AP)	Kuraray Noritake Dental Inc.	00366A	MDP, VTD, Acetone
	Porcelain Liner M Liquid A (PLM-A)	Sun Medical Co., Ltd., Moriyama, Japan	VK1	MMA, 4-META
	Porcelain Liner M Liquid B (PLM-B)	Sun Medical Co., Ltd.	VK1	MMA, γ -MPTS
Luting agent	Super-Bond C&B Catalyst	Sun Medical Co., Ltd.	VK33F	TBB
	Super-Bond C&B Polymer	Sun Medical Co., Ltd	TV2	PMMA, TiO_2
	MMA	Wako Pure Chemical Ind., Ltd., Osaka, Japan	EPQ4043	99.8 MMA

MDP, 10-methacryloyloxydecyl dihydrogen phosphate; γ -MPTS, 3-(trimethoxysilyl)propyl methacrylate; VTD, 6-(4-vinylbenzyl-*n*-propyl)amino-1,3,5-triazine-2,4-dithione, -ditiol tautomer; MMA, methyl methacrylate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; TBB, tri-*n*-butylborane; PMMA, polymethyl methacrylate.

hole. The steel ring was filled with the MMA-TBB self-polymerizing resin, using a brush-dip technique.

After 30 min of bonding, the prepared specimens were immersed in water at 37°C for 24 h. This state was defined as 0 thermocycles, and half of the specimens (seven sets of 11 pairs) were tested at this stage. The remaining half of the specimens (seven sets of 11 pairs) were subsequently thermocycled in water between 5°C and 55°C for 10,000 cycles, with a 60-s dwell time per bath (Thermal Shock Tester TTS-

1 LM, Thomas Kagaku, Tokyo, Japan). The specimens were fixed in a steel mold and were seated in a bond test jig. The shear bond strength was determined with a mechanical testing device (Type 5567, Instron, Canton, MA, USA) at a crosshead speed of 0.5 mm/min. The average shear bond strengths and standard deviations of eleven replications were calculated for each group.

Debonded surface analysis

After the shear bond testing, the debonded surface was observed through an optical microscope (8×, SZX9, Olympus, Tokyo, Japan). The failure modes were classified into the following two categories: A, adhesive failure at the luting agent/adherent interface; and CA, combination of adhesive and cohesive failures.

Cohesive fracture ratios were calculated using surface analysis software (LM eye, Lasertec, Tokyo,

Fig. 1 Procedure for specimen preparation and shear bond testing.

Table 2 Adhesive primers assessed

Group	Primer (monomers)
Group 1	Monobond Plus (MDP, γ -MPTS)
Group 2	Clearfil Ceramic Primer (MDP, γ -MPTS)
Group 3	Alloy Primer (MDP, VTD)
Group 4	Alloy Primer+Porcelain Liner M Liquid B (MDP, VTD, γ -MPTS)
Group 5	Porcelain Liner M Liquid A+Liquid B (4-META, γ -MPTS)
Group 6	Porcelain Liner M Liquid A (4-META)
Group 7	Porcelain Liner M Liquid B (γ -MPTS)

Fig. 2 Representative debonded specimens. (a) Debonded surface of the zirconia specimen. This specimen was primed with the Clearfil Ceramic Primer material. The circular area represents the actual adhesive area. (b) The debonded surface was binarized by software and was cohesive in the resin area (CR) and adhesive area (Ad).

Japan) (Fig. 2). Cohesive fracture ratio is calculated as follows:

$$\begin{aligned} &\text{Cohesive fracture ratio (\%)} \\ &= \text{Cohesive fracture area (mm}^2\text{)} / \text{Adhesive area} \\ &\quad (19.63 \text{ mm}^2) \times 100 \end{aligned}$$

where cohesive fracture area is the differences in the primers. The average cohesive fracture ratios and standard deviations of eleven replications were calculated for each group.

Statistical analysis

A descriptive statistical analysis (mean, standard deviation, median, and interquartile range) of the shear bond strength and the cohesive failure ratio was conducted. The Kolmogorov-Smirnov test (IBM SPSS Statistics ver. 19, IBM, Somers, NY, USA) was primarily used. When the result of the Kolmogorov-Smirnov test did not show normal distribution in several groups, the Kruskal-Wallis test (IBM SPSS Statistics) was performed for both the pre- and post-thermocycling groups to evaluate the differences among primer variations. Based on the Kruskal-Wallis test, Steel-Dwass multiple comparisons (Kyplo5.0, KyensLab, Tokyo, Japan) were further performed to compare the differences among the seven priming groups for each of the pre- and post-thermocycling conditions. The difference between the pre- and post-thermocycling bond strength values for identical surface conditions was analyzed with the Mann-Whitney *U* test (IBM SPSS Statistics). The significance level was set at $\alpha=0.05$.

RESULTS

Shear bond strength

On the Kolmogorov-Smirnov test, the results of the shear bond strength test did not demonstrate a normal distribution for the two groups (post-thermocycling

groups 6 and 7). The Kruskal-Wallis test and Steel-Dwass multiple comparisons were therefore applied to the bond strength results. The Kruskal-Wallis test showed that the χ^2 value was 63.967 for the pre-thermocycling group and 63.439 for the post-thermocycling group. The *p*-value was less than 0.01 for both the pre- and post-thermocycling bond strengths. The pre- and post-thermocycling results were therefore analyzed separately using Steel-Dwass multiple comparisons.

The results for the shear bond strength are summarized in Table 3. The 24-h median bond strengths without thermocycling varied from a minimum of 17.1 MPa to a maximum of 29.7 MPa, and they were categorized into four groups (categories a–d). Among the pre-thermocycling groups, group 1 (Monobond Plus), group 2 (Clearfil Ceramic Primer), and group 4 (Alloy Primer+Porcelain Liner M Liquid B) showed the greatest bond strength (category a), whereas Group 7 (Porcelain Liner M Liquid B) showed the least bond strength (category d). The post-thermocycling median bond strengths varied from 0 MPa to 7.4 MPa and were again categorized into four groups (categories e–h). Among the post-thermocycling groups, groups 1, 2, and 3 showed the greatest bond strength (category e), followed by group 4. Groups 6 and 7 showed the least bond strength (category h).

The Mann-Whitney *U* test demonstrated significant differences between the pre- and post-thermocycling bond strengths for all the groups. The post-/pre-thermocycling bond strength ratios varied from 0.5% to 25.7%.

Failure mode analysis

On the Kolmogorov-Smirnov test, the results of the cohesive fracture ratio did not show a normal distribution for the six groups (pre-thermocycling

Table 3 Shear bond strength mean, standard deviation and median in MPa

Group	Primer	0 thermocycle			10,000 thermocycles				<i>p</i> -value
		Mean (SD)	Median	IQR	Mean (SD)	Median	IQR	Post-/Pre-bs ratio (%)	
Group 1	MP	28.6 (0.9)	28.8 ^a	1.5	7.7 (2.9)	7.4 ^e	5.1	25.7	<i>p</i> <0.01*
Group 2	CP	29.6 (0.9)	29.7 ^{a, b}	2.0	7.6 (1.7)	7.4 ^{e, f}	3.2	24.9	<i>p</i> <0.01*
Group 3	AP	26.8 (2.1)	27.9 ^b	2.4	5.5 (2.3)	6.6 ^{e, f}	4.4	23.7	<i>p</i> <0.01*
Group 4	AP+PLM-B	28.4 (1.1)	28.5 ^{a, b}	1.5	3.9 (1.6)	4.1 ^f	2.8	14.4	<i>p</i> <0.01*
Group 5	PLM-A+PLM-B	22.8 (2.4)	22.5 ^c	4.6	0.3 (0.2)	0.3 ^g	0.3	1.3	<i>p</i> <0.01*
Group 6	PLM-A	20.7 (2.0)	20.4 ^c	3.3	0.1 (0.1)	0 ^h	0.2	0.5	<i>p</i> <0.01*
Group 7	PLM-B	15.5 (3.8)	17.1 ^d	8.4	0.2 (0.1)	0.2 ^{g, h}	0	1.2	<i>p</i> <0.01*

n=11; SD, Standard deviation; Post-/Pre-bs ratio, Post-/Pre-thermocycling bond strength ratio (%); IQR, interquartile range; Identical letters indicate that the values are not significantly (*p*>0.05). * Significant difference between the pre- and post-thermocycling bond strengths (*p*<0.05) according to the Mann-Whitney *U*-test.

Table 4 Failure modes after shear bond testing

Group	Primer / Mode	0 thermocycle					10,000 thermocycles				
		A	CA	Cohesive fracture ratio			A	CA	Cohesive fracture ratio		
				Mean (SD)	Median	IQR			Mean (SD)	Median	IQR
Group 1	MP	0	11	78.4% (4.0)	80.0 ^j	7.0	0	11	50.4% (6.4)	48.0 ^m	10.0
Group 2	CP	0	11	91.1% (3.4)	92.8 ⁱ	3.7	0	11	52.3% (5.7)	53.0 ^m	4.3
Group 3	AP	0	11	71.4% (3.1)	70.0 ^k	5.7	1	10	37.1% (14.0)	40.0 ⁿ	15.4
Group 4	AP+PLM-B	0	11	76.6% (3.0)	77.0 ^j	1.8	1	10	23.8% (11.9)	24.0 ⁿ	10.7
Group 5	PLM-A+PLM-B	4	7	22.3% (26.4)	18.9 ^l	50.2	11	0	0% (0)	0 ^o	0
Group 6	PLM-A	5	6	18.3% (24.4)	13.6 ^l	23.7	11	0	0% (0)	0 ^o	0
Group 7	PLM-B	5	6	10.3% (21.8)	0.4 ^l	6.3	11	0	0% (0)	0 ^o	0

$n=11$; A, Adhesive failure at the resin-zirconia interface; CA, combination of cohesive and adhesive failures; SD, Standard deviation; IQR, interquartile range; Identical letters indicate that the values are not significantly ($p>0.05$). Optical microscope: 8× magnification and an analyzing software.

groups 2, 4, and 7, post-thermocycling groups 5–7). The Kruskal-Wallis test and Steel-Dwass multiple comparisons were further performed. The Kruskal-Wallis test showed that the χ^2 value was 61.464 for the pre-thermocycling group and 67.494 for the post-thermocycling group. The p -value was less than 0.01 for both the pre- and post-thermocycling bond strengths. The pre- and post-thermocycling results were therefore analyzed separately using Steel-Dwass multiple comparisons.

The results of the failure mode analysis and the cohesive fracture ratios are summarized in Table 4. The 24-h median cohesive fracture ratios varied from a minimum of 0.4% (group 7) to a maximum of 92.8% (group 2), and they were categorized into four groups (categories i–l). Among the pre-thermocycling groups, group 2 (Clearfil Ceramic Primer) showed the greatest cohesive fracture ratio (category i), whereas groups 5–7 showed the least cohesive fracture ratio (category l). The post-thermocycling median cohesive fracture ratios varied from a minimum of 0% (groups 5–7) to a maximum of 53.0% (group 2) and were again categorized into three groups (categories m–o). Among the post-thermocycling groups, groups 1 and 2 showed the greatest cohesive fracture ratio (category m), whereas groups 5–7 showed the least cohesive fracture ratio (category o). The cohesive fracture ratio generally decreased after the application of thermal stress.

DISCUSSION

This study evaluated the bonding characteristics of various surface treatment methods for YPS zirconia materials, using seven surface conditioning methods and a TBB-initiated luting agent. The TBB-initiated resin did not contain any adhesive monomer. This

composition enabled evaluation of the effects of functional monomers on the primers. The purpose of this study was to investigate the effects of the application of a combination of a silane coupling agent and a phosphoric acid ester monomer to zirconia on adhesion.

The choice of a chemical bonding system for prosthodontic application can depend on factors such as expense, availability time requirements, and the shelf life of the perishable components. The primers used in this study—Alloy Primer, Clearfil Ceramic Primer, and Monobond Plus—contain MDP, whereas Porcelain Liner M consists of two components: liquid A contained 4-META, and the liquid B component contained γ -MPTS. Regarding the monomer, γ -MPTS, it is not included in the Alloy Primer. Therefore, the interaction between MDP and the silane coupling agent would be indicated by mixing the alloy primer and liquid B.

In the present study, significant differences were observed between primers containing MDP and those not containing MDP; in addition, the silane coupling agent (PLM-B) showed the least bond strength before and after thermocycling. Thus, the first and second hypotheses were accepted. The third hypothesis was substantially rejected, as there were no significant differences between the MDP/silane coupling agent mixture and the silane coupling agent.

This study used shear bond strength determination in combination with thermocycling. Shear testing is a superior evaluation method for the shear bond strength of various resin cements. In this study, the steel ring was filled with the MMA-TBB self-polymerizing resin using a brush-dip technique. Compared with another method of bonding a pair of disk specimens, the ring filling method clearly resulted in low bond strength. Matsumura *et al.* suggested that the difference in the coefficient of thermal expansion between resin and

metal affects the shear bond strength²⁶⁾. Therefore, the greater the volume of filled TBB-resin is, the less bond strength would be detected.

Past studies have suggested that silane coupling agents improve the shear bond strength between silica-based ceramics and resin luting agents^{15,16,19,22,25)}. Zirconium oxide ceramic is different from conventional silica-based ceramic, and it has no silica phase or resistance to various types of surface conditioning^{1,16-19,24)}. Previous studies have reported that the application of an MDP-containing bonding/silane agent mixture increased the shear bond strength between zirconium oxide ceramics and resin luting agents^{1,16-19)}. In contrast, Derand *et al.* reported that a silane coupling agent inhibited bonding to zirconium oxide²⁵⁾. In this study, comparing group 3 and group 4, the author predicted that AP+PLM-B would show bond strength similar to that of MP and CP. In contrast, group 4 showed less bond strength than group 3, although there were no significant differences between group 3 and 4 after 10,000 thermal cycles. Two possibilities were considered: (1) the effect of MDP was inhibited by γ -MPTS; and (2) the concentration of MDP was diluted by the increase in the quantity of solvent (Ethanol, MMA, Aceton).

This study evaluated the percentage of cohesive failure area in the prescribed bonded area using surface analysis software (LM eye). As shown in Table 4, primers containing MDP (MP, CP, AP, AP+PLM-B) demonstrated a combination of cohesive and adhesive failures, whereas the others exhibited adhesive failures after thermocycling. Significant differences were shown between primers containing MDP and those not containing MDP. The authors suspect that the adhesive failure of the primers not containing MDP after application of thermocycling is a result of the penetration of water into the adhesive interface. It is also speculated that γ -MPTS and 4-META monomers are not effective for bonding zirconium oxide.

Within the limitations of this study, it can be concluded that the application of a silane coupling agent did not improve the shear bond strength. In contrast, it is possible that MDP was inhibited by the addition of γ -MPTS. Therefore, primers that contain MDP are recommended for the bonding of Katana zirconia material with a tri-*n*-butylborane (TBB)-initiated acrylic resin.

ACKNOWLEDGMENTS

This work was supported in part by a Grant-in-Aid for Scientific Research (C) 24592934 (2013) from the Japan Society for the Promotion of Science (JSPS), a grant from the Dental Research Center Nihon University School of Dentistry (2013), and a grant from the Promotion and Mutual Aid Corporation for Private Schools of Japan (2013).

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