Effect of two phosphate priming agents on bonding to alumina of two luting composites

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Purpose: The purpose of this study was to evaluate the effect of four ceramic bonding systems on adhesive bonding to high-purity alumina.
Materials and Methods: Different sized alumina disks were bonded with one of the following bonding systems; 1) Clapearl Bonding Agent and Clapearl DC, 2) Clapearl Bonding Agent and Clearfil Esthetic Cement, 3) Clearfil Ceramic Primer and Clapearl DC, and 4) Clearfil Ceramic Primer and Clearfil Esthetic Cement. Bond strengths were determined both before and after application of thermocycling.
Results: Average post-thermocycling bond strengths varied from 45.4 MPa to 55.2 MPa, and were categorized into two groups. Two groups, primed with the Clapearl Bonding Agent, recorded the maximal post-thermocycling bond strength. Bond strength of the two groups luted with the Clearfil Esthetic Cement increased statistically after application of thermocycling ($p \leq 0.01$).
Conclusion: Among the four bonding systems assessed, priming with the phosphate-based Clapearl Bonding Agent followed by luting with the Clapearl DC composite appeared to be the most consistent and durable system for bonding high-purity alumina. **(Asian Pac J Dent 2011; 11: 9-13.)**
Key Words: alumina, bonding, composite, MDP, primer

Introduction

The application of high-strength ceramic materials for anterior and posterior tooth-colored restorations has increased substantially. This trend is probably due to improvements in the layering technique between ceramic material and traditional tooth-colored porcelain. Aluminum oxide (alumina) has been used as a component of dental porcelain as the reinforcing medium.[1] High-purity alumina was introduced as a coping material in the Procera ceramic restorative system.[2] It is desirable that alumina or alumina-based coping material and dentin can be bonded durably.

A number of papers demonstrated the usefulness of adhesive systems for bonding alumina[3-17] and alumina-based ceramic materials.[18-20] An adhesive resin based on a carboxylic monomer enhanced bond strength to alumina.[3] Silane monomers and/or surface preparations with silicon compounds were introduced for bonding alumina ceramic restoratives.[3-6,8-11,13,14] The application of acidic monomers was also effective for bonding alumina.[3,6-17]

Although varying acidic adhesive systems for bonding tooth structure and ceramic restorations are being introduced, only limited information is available concerning bonding behavior of high-purity alumina, especially as related to chemical ingredients or functional monomers in the bonding and luting agents.[3,15-17] The purpose of the current study was to evaluate the effect of priming and luting agents on bond strength and durability of four bonding systems joined to high-purity alumina.

Materials and Methods

High-purity alumina (99.7%) sintered at 1,600˚C for 5 days (Furuuchi Chemical Corp., Tokyo, Japan) was used as the adherend material. Two priming agents; 1) Clapearl Bonding Agent (Kuraray Medical Inc., Tokyo, Japan) and 2) Clearfil Ceramic Primer (Kuraray Medical Inc.), were assessed. Both materials contained 10-methacryloyloxydecyl dihydrogen phosphate (MDP) as an adhesive functional monomer. Two dual-curable luting composites; 1) Clapearl DC (Kuraray Medical Inc.) and 2) Clearfil Esthetic Cement (Kuraray Medical Inc.), were employed, both of which did not contain any adhesive promoting monomer. Information about the materials is summarized in Table 1.

Table 1. Materials assessed

Material	Composition; (Lot)
Adherend material	
Sintered alumina	99.7 Al_2O_3, 0.08 SiO_2, 0.05 MgO (%)
Priming agent	
Clearfil Photo Bond	Catalyst: MDP, Bis-GMA, HEMA, dibenzoyl peroxide, CQ; (0409AA)
	Universal: initiators, accelerators, ethanol; (0507BA)
Clearfil Porcelain Bond Activator	MPTS; (0214AA)
Clearfil Ceramic Primer	MDP, MPTS, ethanol; (0010AA)
Luting composite	
Clapearl DC	A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, barium glass filler, colloidal silica, accelerators; (0058AB)
	B: Bis-GMA, TEGDMA, hydrophobic aromatic and hydrophilic aliphatic dimethacrylates, silanated silica, barium glass, colloidal silica, CQ, initiators, accelerators, pigments; (0046AA)
Clearfil Esthetic Cement	A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, silanated barium glass, colloidal silica, accelerators; (003AAA)
	B: Bis-GMA, TEGDMA, hydrophobic aromatic and aliphatic dimethacrylates, silanated silica, silanated barium glass, colloidal silica, CQ, initiators, accelerators, pigments; (003AAA)

Bis-GMA, bisphenol A diglycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; CQ, *dl*-camphorquinone; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MPTS, 3-(trimethoxysilyl)propyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; A, A paste; B, B paste. The Clapearl Bonding Agent consists of the following three-liquid; Clearfil Photo Bond (two-liquid) and Clearfil Porcelain Bond Activator (single-liquid)

A total of 88 pairs of disk specimens (10 and 8 mm in diameter by 3 mm thick) were wet-ground with a series of silicon-carbide (SiC) abrasive paper (400, 800, and 1500 grit) and ultrasonically cleaned with methanol. After cleaning, a piece of double-coated tape with a circular hole 5 mm in diameter and 50 μm in thickness was positioned on the surface of the 10 mm-diameter disk to define the bond area.

The 88 specimens of alumina disk pairs were divided into four sets (four adhesive systems; Table 2) of 22 specimen pairs. Specimen disks were primed with one of the two priming agents and air-dried. The 8- and 10-mm-disks were bonded with one of the two dual-polymerizing luting composites. After bonding, a 5.0 N load was applied to the specimens. Each specimen was then light exposed for 40 s with a halogen light polymerization unit (Optilux 501, Kerr Corp., Orange, CA, USA) from three directions. Accumulated exposure time period was 120 s.

After 30 minutes of bonding, the specimens were stored in 37°C water for 24 hours. This state was defined as pre-thermocycling. One half of the specimens (four sets of 11 pairs) were tested at this stage. The remaining one half of the specimens (four sets of 11 pairs) were subsequently thermocycled in water between 5°C and 55°C for 100,000 cycles with a 60-s dwell time per bath (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co. Ltd., Tokyo, Japan). The specimens were fixed in a steel mold and seated in an ISO TR 11405 bond test jig. Shear bond strengths were determined with a mechanical testing device (Type 5567, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm per minute. The average shear bond strength and standard deviation of 11 replications were calculated for each group.

The results were primarily analyzed by Levene test for evaluation of homoscedasticity (SPSS 16.0, SPSS Inc., Chicago, IL, USA). When the results of the Levene test showed homoscedasticity for all categories, one-way analysis of variance (ANOVA) and Tukey HSD multiple comparisons were performed with the value of statistical significance set at $\alpha=0.05$ level. Pre- and post-thermocycling bond strength within an identical bonding system was compared with the Mann-Whitney U-test.

Results

Levene tests run on the pre- and post-thermocycling groups showed p-values; 0.393 for pre-thermocycling and 0.439 for post-thermocycling bond strengths. Results of the pre- and post-thermocycling bond strengths were therefore analyzed by one-way ANOVA. The ANOVA results showed that p-values were less than 0.05 for both pre- and post-thermocycling bond strengths. The pre- and post-thermocycling results were analyzed subsequently with the post-hoc Tukey HSD test.

Table 2. Shear testing results (MPa)

Adhesive system Priming agent / Luting composite	Pre-thermocycling Bond strength mean Category (SD)	Post-thermocycling Bond strength mean Category (SD)	Post-/Pre- bond strength ratio (%)
Clearfil Ceramic Primer / Clearfil Esthetic Cement	39.7 a (2.8)	45.4 e (4.0)	114.4 p=0.01*
Clearfil Ceramic Primer / Clapearl DC	49.3 c (2.7)	48.7 e (3.3)	98.8 p=0.847*
Clapearl Bonding Agent / Clapearl DC	55.0 d (3.9)	54.5 f (4.9)	99.1 p=0.949*
Clapearl Bonding Agent / Clearfil Esthetic Cement	45.6 b (2.4)	55.2 f (3.0)	121.1 p<0.01*

Mean, n=11; SD, Standard deviation; Post-/Pre- bond strength ratio, Post-/Pre- thermocycling bond strength ratio (%); Category, Identical letters indicate that they are not statistically different at p=0.05 (Tukey HSD test). *Mann-Whitney U-test.

Results of shear bond testing are summarized in Table 2. Pre-thermocycling average bond strengths varied from a minimum of 39.7 MPa to a maximum 55.0 MPa, and were statistically different from each other (categories a-d). Post-thermocycling average bond strengths varied from 45.4 MPa to 55.2 MPa, and were categorized into two groups (categories e and f). Two groups, primed with the Clapearl Bonding Agent, recorded the maximal post-thermocycling bond strength (category f). Bond strength of the two groups luted with the Clearfil Esthetic Cement increased significantly after application of thermocycling ($p\leq0.01$), whereas that of the two groups luted with the Clapearl DC material remain unchanged ($p>0.05$). In addition, the Clapearl Bonding Agent generated greater post-thermocycling bond strength than the Clearfil Ceramic Primer regardless of the type of luting agent.

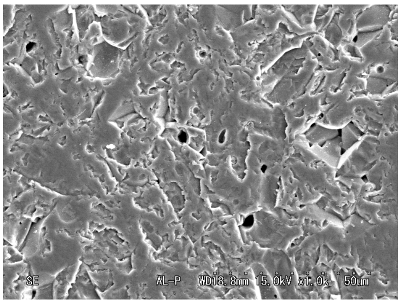
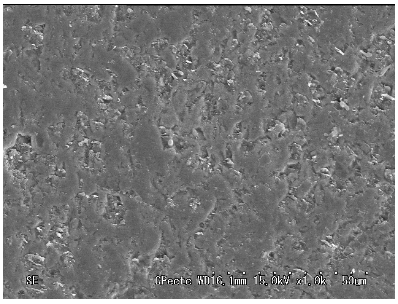
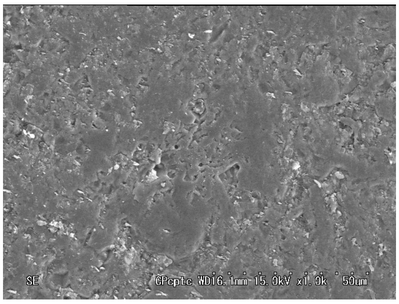
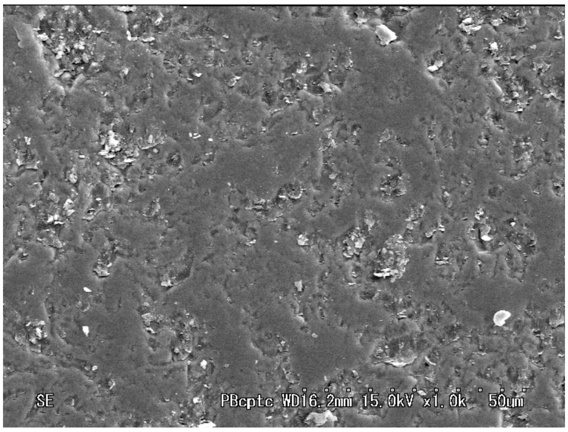




Fig. 1. Scanning electron micrograph of ground alumina (left).
Fig. 2. Debonded surface of alumina primed with Clearfil Ceramic Primer and bonded with Clearfil Esthetic Cement (Center).
Fig. 3. Debonded surface of alumina primed with Clearfil Ceramic Primer and bonded with Clapearl DC (right).

Figure 1 shows the ground alumina surface before bonding. Ground crystalline of alumina can be seen. Figures 2 through 5 depict debonded surface after thermocycling of alumina. Remnants of composite material were detected for all specimens. The micrographs indicate that composite materials were cohesively fractured substantially, and that bonding of the four bonding system is excellent even after thermocycling.



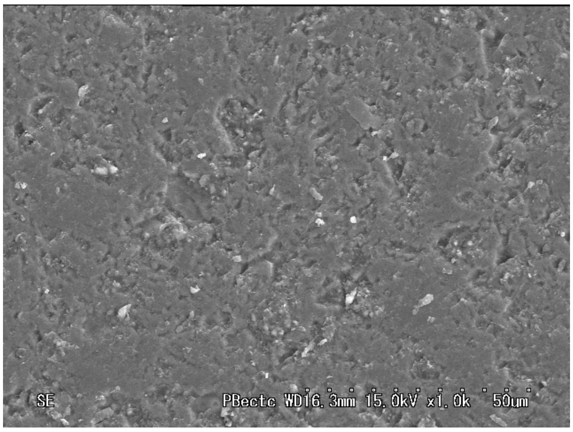


Fig. 4. Debonded surface of alumina primed with Clapearl Bonding Agent and bonded with Clapearl DC (left).
Fig. 5. Debonded surface of alumina primed with Clapearl Bonding Agent and bonded with Clearfil Esthetic Cement (right).

Discussion

This project aimed to evaluate the effect of four ceramic bonding systems on adhesive bonding to high-purity alumina. Clearfil Ceramic Primer was selected as a representative single-liquid pre-hydrolyzed silane primer without initiator. Clapearl Bonding Agent was employed as a three-liquid silane bonding agent with dual-initiation system. Although previous papers revealed that a representative silane monomer, MPTS, is not a critical compound for bonding alumina,[3,15-17] the authors used the Clearfil Porcelain Bond Activator (single-liquid unhydrolyzed silane) together with the Clearfil Photo Bond (two-liquid). Application of this three-liquid bonding agent, identified as the Clapearl Bonding Agent, made it possible to compare difference in bonding characteristics between a pre-hydrolyzed silane agent and an *in-situ* hydrolyzed silane bonding agent. Also comparison between the Clearfil Ceramic Primer and Clapearl Bonding Agent reveals difference in bonding characteristics between an MDP primer without initiator and an MDP bonding agent initiated with dual functional initiation system.

As shown in the shear testing results, it was apparent that the Clapearl Bonding Agent was superior to the Clearfil Ceramic Primer for both luting agents, and for both thermocycling conditions. The results suggest that the presence of an initiator is indispensable for durable bonding between MDP-based adhesives and metal oxide substrates. This finding is in agreement with the results of previous research concerning adhesive bonding of sintered porcelain,[21] although the bonding system employed was a little different.

Shear testing results in the current study exhibited an increase in bond strength after thermocycling of the Clearfil Esthetic Cement. The result was confirmed for both of the priming agents. Considering the fact that adherend material, priming agents, and light-exposure condition were identical, the authors speculate that the increase in bond strength after thermocycling is derived from lower double bond conversion in the matrix of the Clearfil Esthetic Cement at the pre-thermocycling stage. Although the manufacturer may have attempted to release a luting composite with improved color stability, the Clapearl DC is not a particularly problematic material in terms of color stability. Clinicians, therefore, should be aware that the newest version of the bonding system is not always the best system. Within the limitation of the current experiment, it can be concluded that traditional Clapearl Bonding Agent combined with the Clapearl DC luting composite is a suitable system for bonding high-purity alumina.

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