

Effects of Alumina-blasting and Adhesive Primers on Bonding between Resin Luting Agent and Zirconia Ceramics

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This study evaluated the effect of alumina-blasting and three commercial adhesive primers on the shear bond strength of a dual-cured resin luting agent to zirconia ceramics. Two different-sized zirconia ceramic specimens were treated with or without alumina-blasting and then treated with one of three adhesive primers. Subsequently, specimens were cemented together with Linkmax HV (GC). Half of the specimens were stored in water at 37°C for 24 hours and the other half thermocycled 10,000 times before shear bond strength testing. For groups treated with either alumina-blasting or primer, shear bond strength significantly decreased after thermal cycling. For groups treated with both alumina-blasting and one of the three primers, there were no significant differences in shear bond strength before and after thermal cycling ($p < 0.05$). It was thus concluded that the application of each of the three adhesive primers following alumina-blasting was effective for strong bonding of resin luting agent to zirconia ceramics.

Key words : Zirconia ceramic, Resin luting agent, Bond strength

INTRODUCTION

Patient demand for esthetic and metal-free restorations has increased recently, and this has resulted in frequent clinical use of all-ceramic restorations^{1,2}. Among these all-ceramic systems, computer-aided design and computer-aided manufacturing (CAD/CAM) has been commonly used as an alternative to conventional porcelain-fused-to-metal systems. In addition to development of CAD/CAM systems, the advent of new ceramics with high flexural strengths and strong fracture resistance has contributed to the proliferation of all-ceramic restorations.

Zirconia is a ceramic of high flexural strength exceeding 1000 MPa³, which is about six times stronger than feldspathic porcelains, and it is widely used as an orthopedic material (such as the head of hip prostheses)⁴. Polycrystalline zirconia is partially stabilized with Y₂O₃ and utilized in the tetragonal crystalline phase (Y-TZP)⁵. Y-TZP avoids exaggerated fracturing by undergoing transformation from tetragonal to monoclinic phase with a wide increase (about 4%) in volume⁶. To date, Y-TZP has been clinically used for all-ceramic post and core systems^{7,8} and copings for complete coverage of all-ceramic crowns and fixed partial dentures^{9–11}.

Various adhesive monomers have been developed for chemical adhesion with the aim of achieving a durable bond between resin luting agents and base and noble metals. The adhesive functional monomers bond strongly to pure base metals and alloys because they have affinity for metal oxides that exist on the surface of metals such as chromium, tin, titanium,

and copper^{12,13}. The zirconium surface is easily covered with a passive oxide film (ZrO₂), similar to the titanium surface (TiO₂)¹⁴. As such, the hypothesis that adhesive monomers may react with the Y-TZP ceramic surface as a metal oxide on base metal or alloy has been proposed, to that end where greater bonding between resin luting agent and Y-TZP ceramic may be obtained. Moreover, to enhance the bond strength of resin luting agents to the ceramic surface, a number of techniques have been reported that mechanically facilitate resin-to-feldspathic ceramic bonding^{15–20}. In particular, air particle abrasion with alumina prior to cementation is found to provide higher bond strengths for high-alumina ceramics²¹.

Limited information is available concerning the effect of adhesive monomers and/or alumina-blasting on bonding between zirconia ceramics and resin luting agents. Therefore, the purpose of this study was to evaluate and measure the shear bond strength of a dual-cured resin luting agent to Y-TZP ceramics treated with both alumina-blasting and one of three commercial adhesive primers.

MATERIALS AND METHODS

Zirconia ceramic

Industrially manufactured yttrium-oxide-partially-stabilized zirconia ceramic discs (Ohtsuka Ceramics Inc., Shimotsuma, Japan; 97.0 mol% ZrO₂ stabilized by 3.0 mol% Y₂O₃, Vickers hardness at 1,339.5±20.2) with diameters of 13.0 mm and 6.0 mm and a thickness of 2.0 mm were used for this study. The

ceramic surface was polished to $Ra=0.02\ \mu\text{m}$ by the manufacturer, and cleaned ultrasonically in distilled water for five minutes. This surface served as the control (none).

Alumina sandblasting

Alumina sandblasting was performed using mean particle size at 50, 75, 100, or $150\ \mu\text{m}$ of grain-sized aluminum oxide (Saint-Gobain Ceramic Materials Corp., Osaka, Japan.) for 20 seconds at 3.8-bar pressure (Combilabor, CL-FSG94, Heraeus Kulzer, GmbH & Co. KG, Hanau, Germany). Distance of the tip from the ceramic surface was about 10 mm.

Specimen surfaces before and after alumina-blasting were examined with a scanning electron microscope (SEM, TM-1000, Hitachi High-Technologies Corp., Hitachinaka, Japan) operating at an acceleration voltage of 15 kV.

Primers and resin luting agent

Three commercial primers (Alloy Primer, Super Bond Monomer Liquid, and Metal Primer II) containing adhesive monomers effective for base metals and a dual-cured resin luting agent (Linkmax HV) were used in this study. A trial primer containing silane coupling agent was also prepared. Their details are given in Table 1.

Preparation of specimens

Zirconia ceramic discs sandblasted with one of four mean particle sizes at 50-, 75-, 100-, and $150\ \mu\text{m}$ alumina were washed with steam cleaner for one minute and air-dried. The specimens were stabilized on a measuring table, and surface roughness was

measured using a profilometer (Surfcom 480A-12, Tokyo Seimitsu Co. Ltd., Tokyo, Japan) with a $2\ \mu\text{m}$ -radius stylus tip. Cut-off length and transverse length were set at 0.8 mm and 4.0 mm respectively. Specimens sandblasted with each of the four alumina particle sizes were scanned five times, and the average Ra values determined.

A piece of polyethylene tape with a circular hole of 4.0 mm diameter was positioned on the surface of 13.0-mm-diameter zirconia ceramic specimen to define the bonding area. For the control, one side of the zirconia ceramic specimen surface was unprimed; otherwise, one side was primed with one of four primers for 10 seconds, air-dried for five seconds, and then the mixed resin paste was placed within the circle on the 13.0-mm-diameter ceramic surface. Subsequently, the 6.0-mm-diameter ceramic specimen was placed on the resin paste. A sample holder secured the bonded specimens in a rigid position during bonding and controlled the cement film thickness to approximately $50\ \mu\text{m}$. Excess cement was removed before complete hardening of the resin luting agent.

Dual-cured resin luting agent was irradiated from four directions for 40 seconds, for a total exposure time of 160 seconds, using a visible light curing source (Quick Light, J. Morita Corp., Osaka, Japan) at a light intensity $>400\ \text{mW}/\text{cm}^2$. The specimens were allowed to stand for one hour at room temperature and then assigned randomly to two groups of seven specimens each. One group was stored in distilled water at 37°C for 24 hours, while the other group was likewise stored and then subjected to thermal cycling. For groups without or with alumina-blasting at one of four mean particle sizes, specimens

Table 1 Materials used in this study

Material	Abbreviation	Composition	Manufacturer
Alloy Primer	AP	10-methacryloxydecyl dihydrogen phosphate (MDP), 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithione (VTD), acetone	Kuraray Medical Inc., Kurashiki, Japan
Super Bond Monomer Liquid	SBML	4-methacryloxyethyl trimellitic anhydride (4-META), methyl methacrylate (MMA)	Sun Medical Co. Ltd., Moriyama, Japan
Metal Primer II	MP	thiophosphoric acid methacrylate (MEPS) ³⁴ , methyl methacrylate (MMA)	GC Corp., Tokyo, Japan
Silane coupling agent	SC	2.0 wt% γ -methacryloxypropyltrimethoxy silane (γ -MPTS) in ethanol	Tokyo Kasei Industry Co. Ltd., Tokyo, Japan
Linkmax HV	LM	A paste: fluoroaluminosilicate glass, urethane dimethacrylate, silica, camphoroquinone, amine B paste: fluoroaluminosilicate glass, urethane dimethacrylate, silica, benzoyl peroxide	GC Corp., Tokyo, Japan

were thermocycled 5,000 times. For groups treated with 50- μm alumina-blasting and/or one of three primers, specimens were thermocycled 10,000 times. Thermal cycling was performed between water baths (Rika-Kogyo, Hachioji, Japan) held at 4°C and 60°C with a dwelling time of one minute in each bath. Thermal cycling was performed to evaluate bond durability.

Shear test procedure

Shear bond strengths of specimens were measured in a universal testing machine (AGS-10kNG, Shimadzu Corp., Kyoto, Japan) at a crosshead speed of 0.5 mm/min. The means of each group were analyzed by two-way ANOVA with shear bond strength as the dependent variable and surface treatment and storage condition of specimens as independent factors. Tukey's compromise test with $p < 0.05$ was used to establish significance.

RESULTS

Table 2 shows the surface roughness (Ra, μm) values after sandblasting with one of four mean particle sizes of alumina, as well as the shear bond strengths of the resin luting agent to zirconia ceramics. Groups sandblasted with alumina showed significantly higher Ra values than those without sand-

blasting ($p < 0.05$). Increasing alumina particle size resulted in increase of surface roughness, whereby the group sandblasted with 150- μm alumina yielded the highest Ra value. Further, SEM analysis showed differences on the zirconia ceramic surface before and after alumina-blasting with different mean particle sizes. Fig. 1A showed a smooth surface, while Figs. 1B and 1C showed minor and major shallow irregularities respectively. There were no significant differences in shear bond strength among the four groups sandblasted with alumina and the control group before thermal cycling. After 5,000 thermocycles, shear bond strength significantly decreased regardless of mean particle size of sandblasting.

Table 3 shows the shear bond strengths of resin luting agent to zirconia ceramics treated with nine combination groups of alumina-blasting and primers at 0 and 10,000 thermocycles. Two-way ANOVA showed significant differences ($p < 0.0001$) between groups dependent on surface treatment, thermal cycling, and interaction between the two factors. Before thermal cycling, groups treated with a primer showed significantly higher shear bond strength than the control group ($p < 0.05$). However, after 10,000 thermocycles, the control and SC groups showed zero bond strength. When zirconia ceramics were treated with both alumina-blasting (mean particle size at 50 μm) and one of three primers, the shear bond

Table 2 Surface roughness (Ra) after sandblasting with 50-, 75-, 100-, and 150- μm aluminum oxide and shear bond strengths of resin luting agent to zirconia ceramics at 0 and 5,000 thermocycles

Particle size of alumina	Ra (μm), (): SD	Mean shear bond strength (MPa), (): SD	
		0 thermocycles	5,000 thermocycles
Control	0.021 (0.004) ^a	20.1 (3.6) ^{a,A}	0 (0) ^{a,B}
50 μm	0.378 (0.022) ^b	20.1 (2.8) ^{a,A}	4.3 (0.6) ^{c,B}
75 μm	0.532 (0.018) ^c	21.5 (4.2) ^{a,A}	3.7 (0.4) ^{b,B}
100 μm	0.594 (0.009) ^d	23.0 (2.0) ^{a,A}	0 (0) ^{a,B}
150 μm	1.092 (0.074) ^e	19.6 (1.5) ^{a,A}	0 (0) ^{a,B}

Means with same letters are not significantly different by Tukey's compromise test ($p > 0.05$).

Comparisons between four particle sizes of alumina at Ra and each thermocycling condition are represented by lower case letters.

Comparisons between two thermocycling conditions at each particle size of alumina are represented by upper case letters.

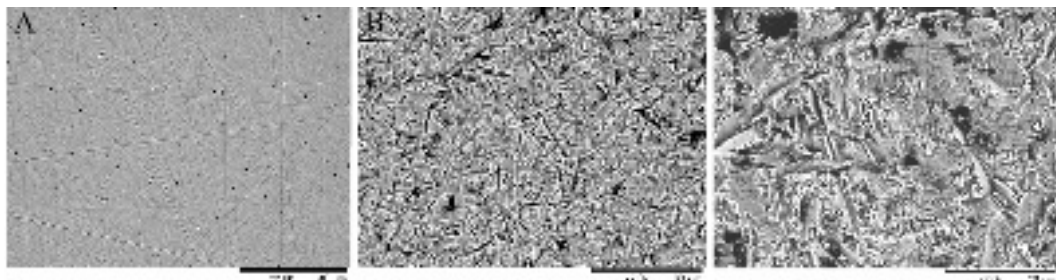


Fig. 1 Scanning electron micrographs ($2,000 \times$ original magnification) of zirconia ceramic specimen surfaces, where A: control non-air-abraded; B: air-abraded with 50- μm alumina; C: air-abraded with 150- μm alumina.

Table 3 Shear bond strengths of resin luting agent to zirconia ceramics at 0 and 10,000 thermocycles

Primer	Alumina-blasting (50 μm)	Mean shear bond strength (MPa), (): SD	
		0 thermocycles	10,000 thermocycles
None	—	20.1 (3.6) ^{a,A}	0 (0) ^{a,B}
SC	—	31.2 (3.2) ^{b,A}	0 (0) ^{a,B}
AP	—	38.4 (4.5) ^{b,c,A}	31.8 (3.3) ^{b,B}
SBML	—	37.6 (3.5) ^{b,c,A}	30.6 (1.4) ^{b,B}
MP	—	37.3 (5.7) ^{b,c,A}	25.5 (4.3) ^{b,B}
None	+	20.1 (2.8) ^{a,A}	0 (0) ^{a,B}
SC	+	39.4 (6.7) ^{b,c,d,A}	0 (0) ^{a,B}
AP	+	45.6 (8.8) ^{c,d,A}	49.0 (6.1) ^{d,A}
SBML	+	41.3 (6.3) ^{b,c,d,A}	42.3 (2.9) ^{c,A}
MP	+	50.2 (9.2) ^{d,A}	53.4 (6.7) ^{d,A}

Means with same letters are not significantly different by Tukey's compromise test ($p > 0.05$).

Comparisons between combinations groups of primer and alumina-blasting at each thermocycling condition are represented by lower case letters.

Comparisons between two thermocycling conditions of each combination group are represented by upper case letters.

strengths after 10,000 thermocycles were significantly higher than those treated with a primer only and without alumina-blasting. There were significant differences in shear bond strength before and after thermal cycling, except for combination groups of both alumina-blasting and one of three commercial primers ($p < 0.05$).

Surface observation of the debonded specimens revealed that all the control and alumina-blasted-only specimens failed at the zirconia ceramic-resin luting agent interface whether thermocycled or not. On the other hand, specimens treated with one of three primers only and without alumina-blasting showed complex adhesive failure and cohesive failure within the resin luting agent at 0 thermocycles, and then adhesive failure at 10,000 thermocycles. As for specimens treated with both alumina-blasting and one of three primers, complex adhesive failure and cohesive failure were observed for most debonded specimens regardless of thermal cycling.

DISCUSSION

It is well known that etching with hydrofluoric acid and application of silane coupling agents help to enhance the bonding between silica-based ceramics and resin luting agents. This is because silanization increases the wettability of the ceramic substrate; at the same time, silane coupling agents allow bonding between the silica contained in the ceramic and the organic matrix of the resin luting agent through siloxane bridges^{22–25}. However, neither etching with hydrofluoric acid nor application of silane coupling agents is sufficient to improve the bond strength between zirconia ceramics and resin luting agents because of two properties of zirconia ceramics: lack of silica and high resistance to acids²⁶. To date, only a few studies concerning the bonding methods of

zirconia ceramics to resin luting agents have been published^{27–29}. The aim of the present study, therefore, was to investigate methods to improve the bonding between resin luting agents and zirconia ceramics by means of alumina-blasting to promote micromechanical retention, and by the use of adhesive monomers to act like silane coupling agents and to promote chemical bonding.

SEM observations revealed that after air abrasion with different mean particle sizes of alumina, the surface roughness of zirconia ceramics increased in accordance with the increase of Ra value. However, in this study, no significant differences were observed in the shear bond strength between alumina-blasted groups and those not subjected to alumina-blasting before thermal cycling, although Ra increased significantly with increase of alumina particle size. At this juncture, it should be highlighted that different studies had yielded different results concerning the effect of alumina particle size by sandblasting on the bond strength of luting agents or composites, because the luted material and surface treatment method after sandblasting were different in each study^{30–33}. After 5,000 thermocycles in the present study, shear bond strengths decreased significantly ($p < 0.05$) regardless of alumina particle size in sandblasting. This result was in accordance with other studies showing that alumina-blasting was ineffective for durable bonding between zirconia ceramics and resin luting agents^{27,28}. Moreover, the findings of this study suggested that while alumina-blasting of zirconia ceramics produced a roughness corresponding to the mean particle size of alumina, the superficial irregularities were limited with minimal-sized undercuts only. Nonetheless, despite its absence of contributory effect on bond strength, the mean alumina particle size of 50 μm was selected because it is a typical size used clinically to clean the

inner surface of restorations.

Adhesive functional monomers bond strongly to the oxides of pure base metals and alloys¹³⁾. When zirconia ceramic was primed with one of three commercial primers or the trial primer containing silane coupling agent, the shear bond strength of the resin luting agent was significantly improved compared with the control group before thermal cycling ($p < 0.05$). The hydrophilic nature of MDP, 4-META, and MEPS³⁴⁾ in the commercial primers played an important role in the bonding of resin luting agent to zirconia. Previous studies have shown that 4-META and MDP acted as coupling agents, similar to silane coupling agents^{35,36)}. Adhesive monomers may bond chemically to the zirconia surface, due to reaction between the hydroxyl groups on zirconia ceramic surface and the carboxyl groups in 4-MET (hydrolyzed 4-META) or hydrogen groups in MDP or MEPS, similar to the surface reaction between silane coupling agents and silica-based ceramics.

After thermal cycling, shear bond strength treated with one of three primers and without sand-blasting decreased significantly ($p < 0.05$). In particular, the group treated with silane coupling agent showed zero bond strength after thermal cycling. This was because the silane coupling agent might have improved the wettability of the zirconia surface and thereby increased the initial bond strength, but this improvement was reduced after 10,000 thermocycles. In light of this result, a silane coupling agent is not recommended for zirconia ceramics with no silica content. It should also be mentioned that chemical surface treatment with primer only would not produce a long-term durable bond between zirconia ceramics and resin luting agents.

On the other hand, combined use of alumina-blasting and silane coupling agent showed improved bond strength between zirconia-alumina composite ceramics and resin luting cements containing adhesive monomers³⁷⁾. It would seem, therefore, that adhesive monomers, not silane coupling agents, were effective for durable bonding of resin cements to zirconia-alumina ceramics. Indeed, in a recent study by Atsu *et al.*³⁸⁾, it was found that when zirconia ceramics were surface-treated using a combination of tribochemical silica coating and the application of an MDP-containing bonding/silane coupling agent mixture, increased shear bond strength between zirconia ceramic and resin luting cement was achieved. In other words, MDP monomer in resin bonding might be essential to activating the reaction of the resin luting agent on the zirconia surface.

When zirconia ceramics were treated with both alumina-blasting at 50- μ m particle size and one of three primers, there were no significant differences in shear bond strength before and after thermal cycling. Alumina-blasting increased the zirconia surface area, and increased surface area allowed to a greater

extent the significant surface reaction between the hydrophilic adhesive monomers and zirconia ceramics. As a result, these groups maintained their shear bond strengths even after 10,000 thermocycles due to the greater anchorage and adhesion effects rendered by both alumina-blasting and adhesive monomers, *versus* the application of primer only without alumina-blasting.

After 10,000 thermocycles, the groups treated with alumina-blasting and either AP or MP showed significantly greater shear bond strength than the group treated with SBML ($p < 0.05$). The 4-MET monomer has carboxyl groups, while the MDP and MEPS monomers have hydrogen groups. The Linkmax HV (LM) material does not contain any adhesive monomer, which is in contrast to other commercially available resin luting agents. Therefore, difference in chemical structure for each adhesive monomer may have an effect on the long-term durability of the bonding between zirconia ceramics and resin luting agents. In addition, the hydrolysis reaction between hydroxyl groups on the zirconia ceramic surface and hydrogen phosphate groups may not occur easily after thermal cycling. Hence, the findings of this study suggested that surface treatment with a commercial adhesive primer following sand-blasting with a mean particle size of 50- μ m alumina was effective for strong and durable bonding between resin luting agents and zirconia ceramics.

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