

Research Article



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Comparison of bond strengths of ceramic brackets bonded to zirconia surfaces using different zirconia primers and a universal adhesive

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ABSTRACT

Objectives: The aim of this study is to compare the shear bond strengths of ceramic brackets bonded to zirconia surfaces using different zirconia primers and universal adhesive.**Materials and Methods:** Fifty zirconia blocks (15 × 15 × 10 mm, Zpex, Tosoh Corporation) were polished with 1,000 grit sand paper and air-abraded with 50 μm Al₂O₃ for 10 seconds (40 psi). They were divided into 5 groups: control (CO), Metal/Zirconia primer (MZ, Ivoclar Vivadent), Z-PRIME Plus (ZP, Bisco), Zirconia Liner (ZL, Sun Medical), and Scotchbond Universal adhesive (SU, 3M ESPE). Transbond XT Primer (used for CO, MZ, ZP, and ZL) and Transbond XT Paste was used for bracket bonding (Gemini clear ceramic brackets, 3M Unitek). After 24 hours at 37°C storage, specimens underwent 2,000 thermocycles, and then, shear bond strengths were measured (1 mm/min). An adhesive remnant index (ARI) score was calculated. The data were analyzed using one-way analysis of variance and the Bonferroni test ($p = 0.05$).**Results:** Surface treatment with primers resulted in increased shear bond strength. The SU group showed the highest shear bond strength followed by the ZP, ZL, MZ, and CO groups, in that order. The median ARI scores were as follows: CO = 0, MZ = 0, ZP = 0, ZL = 0, and SU = 3 ($p < 0.05$).**Conclusions:** Within this experiment, zirconia primer can increase the shear bond strength of bracket bonding. The highest shear bond strength is observed in SU group, even when no primer is used.**Keywords:** Ceramic bracket; Multi-mode adhesive; Orthodontic bracket; Universal adhesive; Zirconia; Zirconia primer

INTRODUCTION

Recently, the increased demand for esthetically-pleasing orthodontic treatments has led to the use of various new materials to produce more esthetic crowns or orthodontic brackets. Orthodontists frequently encounter patients who have had their teeth restored with metal, feldspathic porcelain, reinforced ceramics, and zirconia instead of natural tooth material. Because of the growing demand to maintain esthetics, the use of ceramic or resin brackets has become more popular than the use of metal brackets for orthodontic treatment.

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Originally, the zirconia core was covered with feldspathic porcelain powder as a veneer to maintain esthetics; however, owing to frequent fracture of the veneer, the use of monolithic zirconia crowns has increased [1,2]. In the past, they are used only for the posterior teeth because of the unaesthetic opacity problem. However, as the esthetics of monolithic zirconia crowns improve, they are being used not only for the posterior teeth, but also for anterior teeth [3,4].

However, zirconia is resistant to hydrofluoric acid etching, making it difficult to obtain proper surface roughness using this traditional technique. Thus, various studies have reported techniques to improve the bond strength between zirconia and resin cement by mechanical, chemical, or combined methods. In mechanical surface treatments, roughening using Al_2O_3 - or silica-coated particles leads to increased bond strength [5-7]. Several zirconia primers are available for chemical treatment [4,8-10]. Usually, these primers contain 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) as a key component. The phosphate group of the 10-MDP can react with the zirconium oxide, and this increases the bond strength. However, the separate use of a zirconia primer for increasing bonding requires time and increases the cost.

Universal or multi-mode adhesives for direct and indirect restoration with etch-and-rinse or self-etch modes were introduced recently. They usually contain 10-MDP and thus make bonding to zirconia possible without the use of zirconia primers. However, only a few studies on the bond strength between orthodontic brackets and zirconia crowns when these adhesives are used have been published [11-13].

There are various materials used to make orthodontic brackets, such as metal, ceramic, resin, and zirconia. Many patients now prefer transparent or white-colored brackets to metal brackets for esthetic reasons, but the bond strength between the bracket and the crown in these devices should be adequate for clinical treatment. A few studies on the shear bond strength between porcelain and metal or ceramic brackets have been published [14]. However, to our knowledge, there have been no such studies on zirconia and ceramic orthodontic brackets. The base of the ceramic bracket is different from that of the metal bracket; thus, the effect of surface treatments using zirconia primers or a universal adhesive to replace the zirconia primer for orthodontic purposes should be evaluated.

The aim of this study was to compare the shear bond strengths of orthodontic ceramic brackets bonded to zirconia surfaces using 3 different zirconia primers and a universal adhesive. The null hypothesis was that there was no difference in the bonding strength of orthodontic ceramic brackets bonded to zirconia surfaces using different primers and universal adhesive.

MATERIALS AND METHODS

Fifty yttria-stabilized zirconium oxide core (Zpex, Tosoh Corporation, Tokyo, Japan) specimens were produced ($15 \times 15 \times 10$ mm) using a copy milling machine and sintered. They were embedded in auto-polymerizing acrylic resin (Ortho-Jet, Lang Dental Manufacturing Co., Inc., Wheeling, IL, USA). Zirconia surfaces were polished with 1,000 grit silicon carbide paper, ultrasonically cleaned, and air-dried. Specimens were sandblasted with $50 \mu\text{m}$ Al_2O_3 particles for 10 seconds at 40 psi. The specimens were distributed into 5 groups ($n = 10$ in each group).

The control group (CO) was not treated with zirconia primer. Transbond XT Primer (3M Unitek, Monrovia, CA, USA) was applied on both surfaces and brackets were bonded using Transbond XT Paste (3M Unitek). Each specimen was light-cured for 15 seconds at 1,100 mW/cm² (Mr. Light LED curing light, Dent Zar, Tarzana, CA, USA).

Three different zirconia primers were applied on the zirconia surface. Metal/Zirconia primer (MZ; Ivoclar Vivadent, Schaan, Liechtenstein), Z-PRIME Plus (ZP; Bisco, Schaumburg, IL, USA), and Zirconia Liner (ZL; Sun Medical, Shiga, Japan) were applied to the specimens of the MZ, ZP, and ZL groups, respectively according to the manufacturer's instructions. Then, Transbond XT Primer was applied on both surfaces and brackets were bonded using Transbond XT.

For the SU group, Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, USA) was applied on the zirconia surface according to the manufacturer's instructions, and ceramic brackets were then bonded on the specimens in a manner similar to that used in the CO group. To minimize the difference, all procedures were done by one operator. The compositions of the 3 zirconia primers and universal adhesive are shown in **Table 1**.

The fifty specimens were stored in a distilled water at 37°C for 24 hours and subjected to 2,000 one-minute-long thermocycles at 5°C and 55°C. The bracket bonded zirconia specimens were mounted on the testing machine zig which bonded surface was perpendicular to the base and the shear bond strength was measured by a universal testing machine (EZ test, Shimadzu Co., Kyoto, Japan), at a crosshead speed of 1 mm/min until bonding failure occurred. The fractured surface was assessed using a stereomicroscope (X30, OPMI Pico, Carl Zeiss Meditec AG, Jena, Germany); the magnified surfaces were classified according to the adhesive remnant index (ARI) score (**Table 2**) [15]. To obtain a representative image, scanning electron microscope (SEM) images ($\times 100$) were obtained (**Figure 1**). For comparison of shear bond strength, we performed the one-way analysis of variance and *post hoc* multiple comparisons using the Bonferroni method at $\alpha = 0.05$. For comparison of the ARI index, we used Fisher's exact test ($p < 0.05$).

Table 1. Composition of primers and universal adhesive

Trade name (abbreviation)	Composition	Manufacturer
Metal/Zirconia Primer (MZ)	Tertiary butyl alcohol, methyl isobutyl ketone, phosphonic acid acrylate, benzoylperoxide	Ivoclar Vivadent, Schaan, Liechtenstein
Z-PRIME Plus (ZP)	Carboxylic acid monomer (BPDM), HEMA, ethanol, organophosphate monomer (10-MDP)	Bisco, Schaumburg, IL, USA
Zirconia Liner (ZL)	MMA, 10-MDP, 4-methoxyphenol (HQME)	Sun Medical, Shiga, Japan
Scotchbond Universal adhesive (SU)	Organophosphate monomer (10-MDP), dimethacrylate resins (BisGMA, etc.), HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane	3M ESPE, St. Paul, MN, USA

BPDM, biphenyl dimethacrylate; HEMA, hydroxyethyl methacrylate; BPDM, biphenyl dimethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MMA, methyl methacrylate; HQME, hydroquinone monomethyl ether.

Table 2. Adhesive remnant index (ARI) scores

ARI score	Criteria
0	No adhesive left on the tooth.
1	Less than half of the adhesive left on the tooth.
2	More than half of the adhesive left on the tooth.
3	All adhesive left on the tooth, with distinct impression of the bracket mesh.

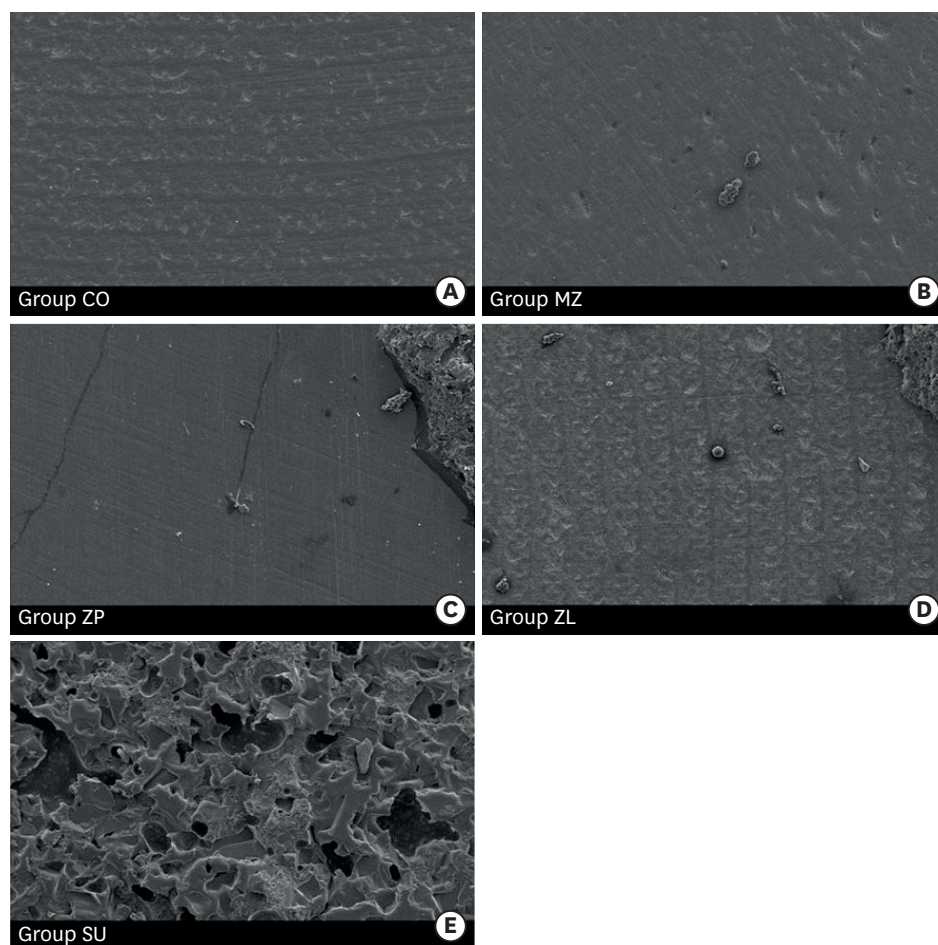


Figure 1. Scanning electron micrographs ($\times 100$) of zirconia surface. (A, B) adhesive failure pattern; (C, D) mixed failure pattern; (E) cohesive failure pattern within the cement. The remnants of the cement were observed on the zirconia surface.
CO, control; MZ, Metal/Zirconia primer (Ivoclar Vivadent, Schaan, Liechtenstein); ZP, Z-PRIME Plus (Bisco, Schaumburg, IL, USA); ZL, Zirconia Liner (Sun Medical, Shiga, Japan); SU, Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, USA).

RESULTS

The results of shear bond strength analysis are described in **Table 3**. Surface treatments with primers resulted in shear bond strength higher than that obtained without primer use in the CO group. The SU group showed the highest shear bond strength, followed by the ZP, ZL, MZ, and CO groups.

Table 3. Shear bond strengths (SBSS) of ceramic brackets bonded to zirconia surface (MPa, $n = 10$)

Group	SBS	Minimum	Maximum
CO	1.07 ± 0.81^d	0.22	2.60
MZ	5.16 ± 0.83^c	4.08	6.51
ZP	10.47 ± 2.69^b	5.91	13.37
ZL	9.55 ± 1.75^b	7.09	12.32
SU	13.85 ± 1.48^a	11.20	16.47

Same superscript indicates that the bond strength was not significantly different between the materials. CO, control; MZ, Metal/Zirconia primer (Ivoclar Vivadent, Schaan, Liechtenstein); ZP, Z-PRIME Plus (Bisco, Schaumburg, IL, USA); ZL, Zirconia Liner (Sun Medical, Shiga, Japan); SU, Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, USA).

Table 4. Adhesive remnant index (ARI) scores calculated depending on the remnants of adhesive on the zirconia surface after debonding

Group	ARI score					Mean \pm SD
	0	1	2	3	Median (Q1–Q3)	
CO	9	1	-	-	0 (0–0)	0.10 \pm 0.30
MZ	10	-	-	-	0 (0–0)	0.00 \pm 0.00
ZP	6	4	-	-	0 (0–1)	0.40 \pm 0.49
ZL	7	3	-	-	0 (0–1)	0.30 \pm 0.46
SU	-	-	4	6	3 (2–3)*	2.60 \pm 0.49

Q1, first quartile; Q3, third quartile; SD, standard deviation; CO, control; MZ, Metal/Zirconia primer (Ivoclar Vivadent, Schaan, Liechtenstein); ZP, Z-PRIME Plus (Bisco, Schaumburg, IL, USA); ZL, Zirconia Liner (Sun Medical, Shiga, Japan); SU, Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, USA).

*Statistically significant difference (Fisher's exact test; $p < 0.05$).

During the analysis of the debonded surfaces, most of the CO and MZ specimens showed adhesive failure patterns. However, some specimens of the ZP and ZL groups showed mixed failure (**Table 4**). The SU group showed mixed or cohesive failure patterns within the bonding resin and ceramic bracket. The SEM micrographs of the representative failure pattern are illustrated in **Figure 1**. Both the CO and MZ groups showed adhesive failure when observed using a stereomicroscope; the difference was small when SEM images were observed. In the CO group, no remnants were observed on the zirconia surface, and for the MZ group, small particles remained on the zirconia surface. Some of the specimens of the ZP and ZL groups showed mixed failure patterns and adhesive remnants were observed. For the SU group, large quantities of cement and adhesives remained on the zirconia surface.

DISCUSSION

The ideal bond strength required for orthodontic brackets is not the maximum bond strength. Instead, the bond strength should be adequate enough for enduring orthodontic treatment while being sufficiently weak to permit easy bracket removal. This is to avoid any concerns about inflicting damage to the restorations while debonding brackets that the clinician may have. In this study, 4 different surface treatments (MZ, ZP, ZL, and SU) were applied on zirconia surfaces to increase the bond strength between ceramic brackets and zirconia. Previous studies have shown that the bond strength between the orthodontic bracket and the restoration on tooth surface is acceptable if it exceeds 6–8 MPa [16,17]. The CO group showed the lowest shear bond strength, implying that only sandblasting is not an appropriate surface pretreatment method for bracket bonding on zirconia surfaces. The ZP, ZL, and SU groups showed significantly higher shear bond strengths than the CO group, but the shear bond strength of the MZ group was lower than 6 MPa, which might not be clinically acceptable for bracket bonding. These results are coincident with the ARI score. This might be due to the absence of 10-MDP.

It has been reported that combined treatment using silica-coating, silane, and MDP is reliable, and MDP-containing monomers improve the adhesion between resin cement and zirconia [4,18]. Many previous studies have reported that MDP-containing primers not only increase the bond strength by improving chemical bonding with zirconium oxide, but also maintain stable bonding after thermocycling [19–21]. Consistent with previous research, the ZP, ZL, and SU groups, which were treated with primer containing MDP, showed clinically acceptable shear bond strengths. ZP contains organophosphate and carboxylic acid monomers; phosphate monomers can co-polymerize with monomers of resin, and carboxylic monomer helps in substrate bonding [22,23]. It was reported that ZP application was associated with lower bond strength than the application of other MDP-containing

primers, because carboxylic acid monomers may have weakened the bonding with the methacrylate group of resin cement [24]. However, according to the results of this study, there was no significant difference between the bond strength in the ZP and ZL groups.

It has been reported that universal adhesives are stable alternatives to conventional bonding techniques [25]. In this experiment, the SU group had significantly higher shear bond strength than the other groups. This adhesive had not been originally developed as a zirconia adhesive, but as observed in this study, it improved bond strength better than other primers that are specialized for zirconia. A possible explanation is that the 10-MPD in the Scotchbond could increase the bond strength, and the Vitrebond copolymer, which is a unique component of the Scotchbond, may also help in bonding to the zirconia surface. Following these results, SU could replace the zirconia primer in the separate step implemented before application of the bonding agent, and it may simplify the bonding step.

In the study of bonding failure patterns, the SU groups showed the most amount of cement remnants on the zirconia surface, and this is consistent with the results of the shear bond strength analysis. Most of the CO and MZ samples showed adhesive failure patterns, reflecting low shear bond strengths.

CONCLUSIONS

Using the zirconia primers can increase the bond strength between the ceramic bracket and zirconia restoration. The SU group showed the highest shear bond strength, even without the use of the zirconia primer, suggesting that the Scotchbond adhesive can be used to simplify the bonding step. The shear bond strengths of the ZP, ZL, and SU groups are clinically acceptable.

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