Original Article

pISSN 2234-7518 • eISSN 2005-372X
http://dx.doi.org/10.4041/kjod.2015.45.4.164

THE KOREAN JOURNAL of ORTHODONTICS
KJO

Comparison of shear bond strength of orthodontic brackets using various zirconia primers

Ji-Yeon Lee[a]
Jin-Seok Kim[a]
Chung-Ju Hwang[b]

[a]Department of Orthodontics, National Health Insurance Corporation Ilsan Hospital, Goyang, Korea
[b]Department of Orthodontics, The Institute of Craniofacial Deformity, College of Dentistry, Yonsei University, Seoul, Korea

Objective: The aim of this study was to compare the shear bond strength (SBS) of orthodontic brackets bonded to zirconia surfaces using three different zirconia primers and one silane primer, and subjected to thermocycling. **Methods:** We designed 10 experimental groups following the surface treatment and thermocycling. The surface was treated with one of the following method: no-primer (NP), Porcelain Conditioner (PC), Z-PRIME Plus (ZP), Monobond Plus (MP) and Zirconia Liner Premium (ZL) (n=20). Then each group was subdivided to non-thermocycled and thermocycled groups (NPT, PC, ZPT, MPT, ZLT) (n=10). Orthodontic brackets were bonded to the specimens using Transbond™ XT Paste and light cured for 15 s at 1,100 mW/cm^2. The SBS was measured at a 1 mm/min crosshead speed. The failure mode was assessed by examination with a stereomicroscope and the amount of bonding resin remaining on the zirconia surface was scored using the modified adhesive remnant index (ARI). **Results:** The SBS of all experimental groups decreased after thermocycling. Before thermocycling, the SBS was ZL, ZP ≥ MP ≥ PC > NP but after thermocycling, the SBS was ZLT ≥ MPT ≥ ZPT > PCT = NPT ($p > 0.05$). For the ARI score, both of the groups lacking primer (NP and NPT) displayed adhesive failure modes, but the groups with zirconia primers (ZP, ZPT, MP, MPT, ZL, and ZLT) were associated with mixed failure modes. **Conclusions:** Surface treatment with a zirconia primer increases the SBS relative to no-primer or silane primer application between orthodontic brackets and zirconia prostheses.
[Korean J Orthod 2015;45(4):164-170]

Key words: Orthodontic brackets, Shear bond strength, Zirconia, Zirconia primer

Received November 21, 2014; Revised January 7, 2015; Accepted February 12, 2015.

Corresponding author: Chung-Ju Hwang.
Professor, Department of Orthodontics, The Institute of Craniofacial Deformity, College of Dentistry, Yonsei University, 50-1 Yonsei-ro, Seodaemun-gu, Seoul 120-752, Korea
Tel +82-1-2228-3106 e-mail hwang@yuhs.ac

The authors report no commercial, proprietary, or financial interest in the products or companies described in this article.

INTRODUCTION

Increased interest in an esthetic facial appearance has led to an increase in the number of adult orthodontic patients. Many patients underwent previous dental restorations to treat wear, attrition, missing teeth, and malformed teeth.[1] Distinct surface treatments are required to increase the bond strength between orthodontic brackets and the restorations because the bond between bracket and previous restorations is weaker than the bond to natural teeth.[2]

Esthetic demands and the development of ceramic systems have increased the use of all-ceramic crowns. Zirconia is a widely used core for all-ceramic crowns due to its high strength and superior esthetic appearance. To construct an esthetic prosthesis, a veneer is layered with ceramic powder onto the zirconia core. However, fracturing of the veneer is frequently reported in the posterior teeth, which apply strong masticatory forces,[3,4] prompting increased use of monolithic zirconia crowns without veneers.[5,6] In orthodontic patients with monolithic zirconia crowns, the orthodontic bracket should be bonded directly onto the zirconia surface. Currently, an additional zirconia surface treatment is required to obtain proper bond strength.[7]

In prosthodontics, various mechanical and chemical surface treatments have been studied to improve the bond strength between zirconia and resin cement. One such technique sandblasting was reported to increases mechanical retention,[8,9] and as a chemical method, many recently developed zirconia primers have been introduced.[10-13] However, few studies have examined the bond strength between orthodontic brackets and zirconia prostheses, and the effects of surface treatment with zirconia primers on bonding. Thus, the aim of this study was to compare the shear bond strength (SBS) of orthodontic brackets using three different zirconia primers and one silane primer, and subjected to thermocycling.

MATERIALS AND METHODS

A total 100 blocks of yttrium oxide-stabilized zirconia (NaturaZ α-series; DMAX International, Seoul, Korea) were embedded in acrylic resin (Ortho-Jet^{TM}; Lang Dental Manufacturing Co., Inc., Wheeling, IL, USA) polished with 1000-grit silicon carbide paper, and ultrasonically cleaned. All specimens underwent airborne-particle abrasion with 50-μm Al_2O_3 particles at 40 psi for 10 s at a 10-mm distance perpendicular to the surface. The specimens were randomly divided into five primer groups (n = 20 per group), and each primer was divided into two subgroups (n = 10 each) to examine by thermocycling protocols (Table 1). The composition of the primers used in this study is presented in Table 2.

Before bonding the orthodontic metal brackets (Gemini series^{TM}; 3M Unitek Corporation, Monrovia, CA, USA), all specimens were steam-cleaned and air-dried. In the control no-primer (NP) group, Transbond^{TM} XT Primer (3M Unitek Corporation) was applied to the zirconia surface and bracket base. The remaining specimen groups were treated with either Porcelain Conditioner (PC), Z-PRIME Plus (ZP), Monobond Plus (MP), or Zirconia Liner Premium (ZL) primer according to each

Table 1. Characteristics of experimental groups

Test group		**Surface treatment**
Non-thermocycled	**Thermocycled**	
NP	NPT	Sandblasting
PC	PCT	Sandblasting + PC
ZP	ZPT	Sandblasting + ZP
MP	MPT	Sandblasting + MP
ZL	ZLT	Sandblasting + ZL

NP, No-primer; NPT, no-primer with thermocycling; PC, Porcelain Conditioner; PCT, Porcelain Conditioner with thermocycling; ZP, Z-PRIME Plus; ZPT, Z-PRIME Plus with thermocycling; MP, Monobond Plus; MPT, Monobond Plus with thermocycling; ZL, Zirconia Liner Premium; ZLT, Zirconia Liner Premium with thermocycling.

Table 2. Composition of primers applied to zirconia specimens

Trade name	**Functional monomer**	**Manufacturer**
Porcelain Conditioner	Silane	Reliance Orthodontic Products, Inc., Itasca, IL, USA
Z-PRIME Plus	Organophosphate and carboxylic acid, biphenyl dimethacrylate and hydroxyethyl methacrylate	Bisco, Inc., Schaumburg, IL, USA
Monobond Plus	10-Methacryloyloxydecyl dihydrogen phosphate, silane methacrylate, ethanol, sulfide methacrylate	Ivoclar Vivadent AG, Schaan, Liechtenstein
Zirconia Liner Premium	Methyl methacrylate, phosphate ester monomer, 4-methoxyphenol	Sun Medical Co., Ltd., Shiga, Japan

manufacturer's instructions. All orthodontic brackets were bonded to the specimens using Transbond™ XT Paste and light polymerized for 15 s at 1,100 mW/cm^2 (Mr. Light LED curing light; Dent Zar, Tarzana, CA, USA). After bonding the orthodontic brackets, all specimens were stored at 37°C in distilled water for 24 hours. The five primer groups were then split into a total 10 subgroups, with one subgroup of specimens from each primer treatment subjected to thermocycling (KD-TCS30; Kwang-duk F.A., Gwangju, Korea) in 5°C and 55°C water for 2,000 cycles, yielding groups NPT, PCT, ZPT, MPT, and ZLT. The remaining five subgroups (NP, PC, ZP, MP, and ZL) did not undergo thermocycling.

Table 3. Modified adhesive remnant index (ARI) score

ARI score	Criteria
1	All composite remained on tooth.
2	More than 90% of the composite remained on tooth.
3	More than 10% but less than 90% of the composite remained on tooth.
4	Less than 10% of the composite remained on tooth.
5	No composite remained on tooth.

Table 4. Post-hoc test of pair differences comparing mean bond strengths between groups

Group	Difference of average bond strength (MPa)	Lower limit (MPa)	Upper limit (MPa)	Adjusted *p*-value
PC-NP	11.82	9.37	14.29	< 0.01
ZP-NP	15.16	12.70	17.62	< 0.01
MP-NP	13.72	11.26	16.18	< 0.01
ZL-NP	14.93	12.47	17.39	< 0.01
ZP-PC	3.33	0.87	5.79	< 0.01
MP-PC	1.89	–0.57	4.35	0.2
ZL-PC	3.10	0.64	5.56	< 0.01
MP-ZP	–1.44	–3.90	1.02	0.47
ZL-ZP	–0.23	–2.69	2.23	1.00
ZL-MP	1.21	–1.25	3.67	0.63
PCT-NPT	0.45	–2.12	3.01	0.99
ZPT-NPT	7.66	5.10	10.22	< 0.01
MPT-NPT	9.71	7.15	12.27	< 0.01
ZLT-NPT	10.35	7.78	12.91	< 0.01
ZPT-PCT	7.21	4.65	9.77	< 0.01
MPT-PCT	9.26	6.70	11.82	< 0.01
ZLT-PCT	9.90	7.34	12.46	< 0.01
MPT-ZPT	2.05	–0.51	4.61	0.17
ZLT-ZPT	2.69	0.13	5.25	< 0.05
ZLT-MPT	0.64	–1.92	3.20	0.95

NP, No-primer; NPT, no-primer with thermocycling; PC, Porcelain Conditioner; PCT, Porcelain Conditioner with thermocycling; ZP, Z-PRIME Plus; ZPT, Z-PRIME Plus with thermocycling; MP, Monobond Plus; MPT, Monobond Plus with thermocycling; ZL, Zirconia Liner Premium; ZLT, Zirconia Liner Premium with thermocycling.

The SBS was measured in all 10 groups using a universal testing machine (Model 3366; Instron® Co., Norwood, MA, USA) at a 1 mm/min crosshead speed. The failure mode was assessed by examination with a stereomicroscope (SZ61; Olympus, Tokyo, Japan), and the amount of bonding resin remaining on the zirconia surface was scored using the modified adhesive remnant index (ARI)[14] (Table 3). One representative specimen from each group was flecked with gold using an ion coater (IB-3; Eiko Co., Tokyo, Japan) and examined via scanning electron microscopy (S-800; Hitachi, Tokyo, Japan) to further evaluate the failure mode.

Statistical analysis

Statistical analysis was performed with IBM SPSS Statistics version 20.0 (IBM Co., Armonk, NY, USA). Two-way analysis of variance (ANOVA) was applied to detect significant differences in the SBS between the groups. The significance for all statistical tests was set at $p < 0.05$, and Tukey's honest significant difference post-hoc test was used to detect pairwise differences between the groups (Table 4).

Table 5. Comparison of shear bond strengths between primer and thermocycling groups

Group	Shear bond strength (MPa)
NP	11.59 ± 2.39 (7.61–16.40)
PC	23.42 ± 1.73 (20.90–26.58)
ZP	26.74 ± 0.94 (25.22–27.83)
MP	25.31 ± 1.62 (22.27–27.18)
ZL	26.52 ± 2.55 (22.91–31.45)
NPT	5.68 ± 0.85 (3.81–6.67)
PCT	6.12 ± 0.44 (5.30–6.75)
ZPT	13.33 ± 2.06 (9.90–17.29)
MPT	15.38 ± 2.95 (10.47–19.06)
ZLT	16.02 ± 2.54 (12.82–20.70)

Values are presented as mean ± standard deviation (range).
NP, No-primer; NPT, no-primer with thermocycling; PC, Porcelain Conditioner; PCT, Porcelain Conditioner with thermocycling; ZP, Z-PRIME Plus; ZPT, Z-PRIME Plus with thermocycling; MP, Monobond Plus; MPT, Monobond Plus with thermocycling; ZL, Zirconia Liner Premium; ZLT, Zirconia Liner Premium with thermocycling.

RESULTS

The SBS values for all groups are presented in Table 5. The variables and the interactions between the variables exerted significant effects on the SBS ($p < 0.05$; Table 6). In all five surface treatment groups, the SBS decreased after thermocycling (Figure 1). Among the groups that did not undergo thermocycling, group NP exhibited a significantly lower SBS than the other groups (PC, ZP, MP, and ZL; $p < 0.05$). Group PC, which was treated with a silane primer, showed significantly lower SBS than groups ZP and ZL ($p < 0.05$). However, groups PC and MP did not significantly differ between each other ($p > 0.05$), and no significant differences were detected among the groups treated with zirconia primers ($p > 0.05$; Figure 1).

In the thermocycling groups, there was no significant difference between groups NPT and PCT ($p > 0.05$). Groups ZPT, MPT, and ZLT, which used zirconia primers, were associated with a significantly higher SBS than the groups NPT and PCT ($p < 0.05$; Figure 1). There were no significant differences between groups ZPT and MPT or between groups MPT and ZLT ($p > 0.05$); however, groups ZPT and ZLT differed significantly between each other ($p < 0.05$; Figure 1).

For the ARI score, both of the groups lacking primer

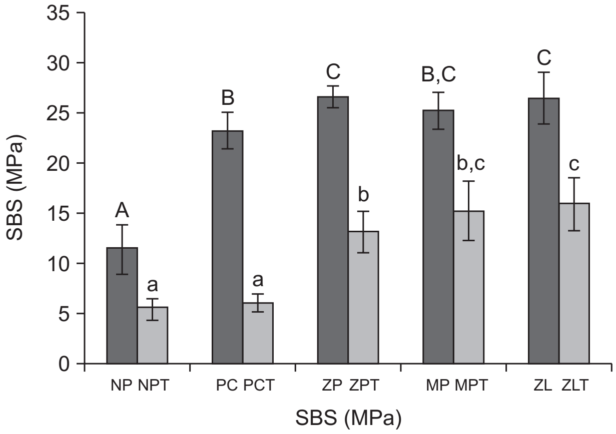

Figure 1. Comparison of shear bond strength (SBS) between non-thermocycled and thermocycled groups. Identical letters (A, B, C, a, b, c) indicate the lack of a significant difference between mean ($p > 0.05$). NP, No-primer; NPT, no-primer with thermocycling; PC, Porcelain Conditioner; PCT, Porcelain Conditioner with thermocycling; ZP, Z-PRIME Plus; ZPT, Z-PRIME Plus with thermocycling; MP, Monobond Plus; MPT, Monobond Plus with thermocycling; ZL, Zirconia Liner Premium; ZLT, Zirconia Liner Premium with thermocycling.

Table 6. Two-way ANOVA for experimental groups

Source of variation	df	Sum of squares	Mean square	F-value	*p*-value
Thermocycling	1	3,253.11	3,253.11	833.09	< 0.05
Primer	4	2,272.88	568.22	145.52	< 0.05
Thermocycling × Primer	4	359.60	89.90	23.02	< 0.05

ANOVA, Analysis of variance; df, degree of freedom.

Table 7. Frequency distribution of failure modes

Modified ARI scores	Thermocycled group					Non-thermocycled group				
	NP	PC	ZP	MP	ZL	NPT	PCT	ZPT	MPT	ZLT
1	-	-	-	-	-	-	-	-	-	-
2	-	-	7	5	6	-	-	6	7	8
3	-	4	3	5	4	-	5	4	3	2
4	-	3	-	-	-	-	3	-	-	-
5	10	3	-	-	-	10	2	-	-	-
Median	5	4	2	2.5	2	5	3.5	2	2	2
Mean	5	3.9	2.3	2.5	2.4	5	3.7	2.4	2.3	2.2
SD	0	0.83	0.46	0.5	0.49	0	0.78	0.48	0.45	0.4

ARI, Adhesive remnant index score; NP, no-primer; NPT, no-primer with thermocycling; PC, Porcelain Conditioner; PCT, Porcelain Conditioner with thermocycling; ZP, Z-PRIME Plus; ZPT, Z-PRIME Plus with thermocycling; MP, Monobond Plus; MPT, Monobond Plus with thermocycling; ZL, Zirconia Liner Premium; ZLT, Zirconia Liner Premium with thermocycling; SD, standard deviation.

http://dx.doi.org/10.4041/kjod.2015.45.4.164

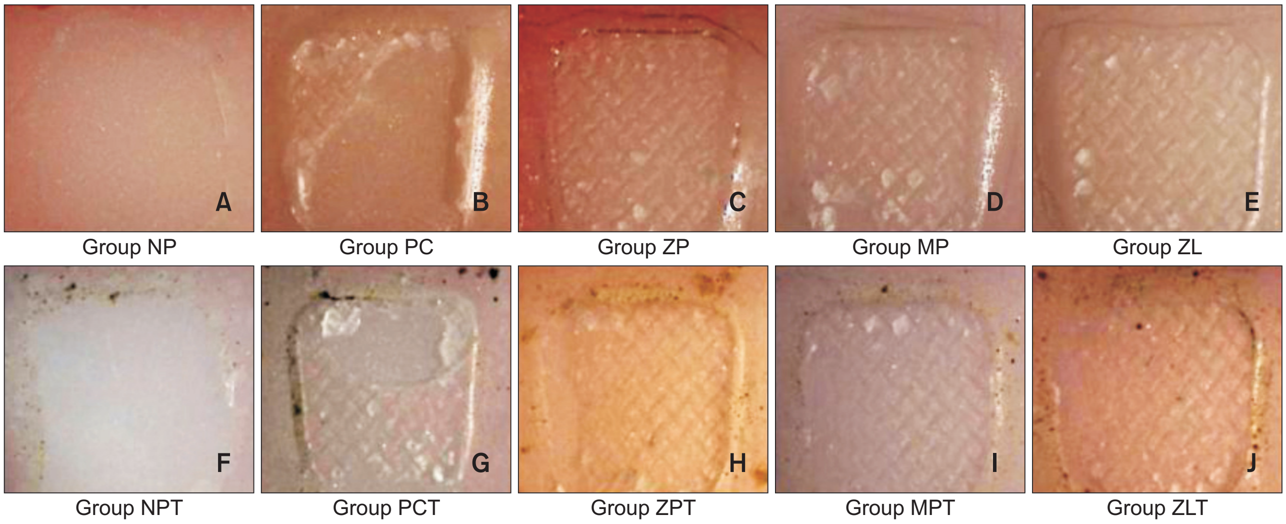

Figure 2. Representative stereoscopic micrographs (16×) of the adhesive failure mode (A, F) and the mixed failure mode (B–E, G–J). NP, No-primer; NPT, no-primer with thermocycling; PC, Porcelain Conditioner; PCT, Porcelain Conditioner with thermocycling; ZP, Z-PRIME Plus; ZPT, Z-PRIME Plus with thermocycling; MP, Monobond Plus; MPT, Monobond Plus with thermocycling; ZL, Zirconia Liner Premium; ZLT, Zirconia Liner Premium with thermocycling.

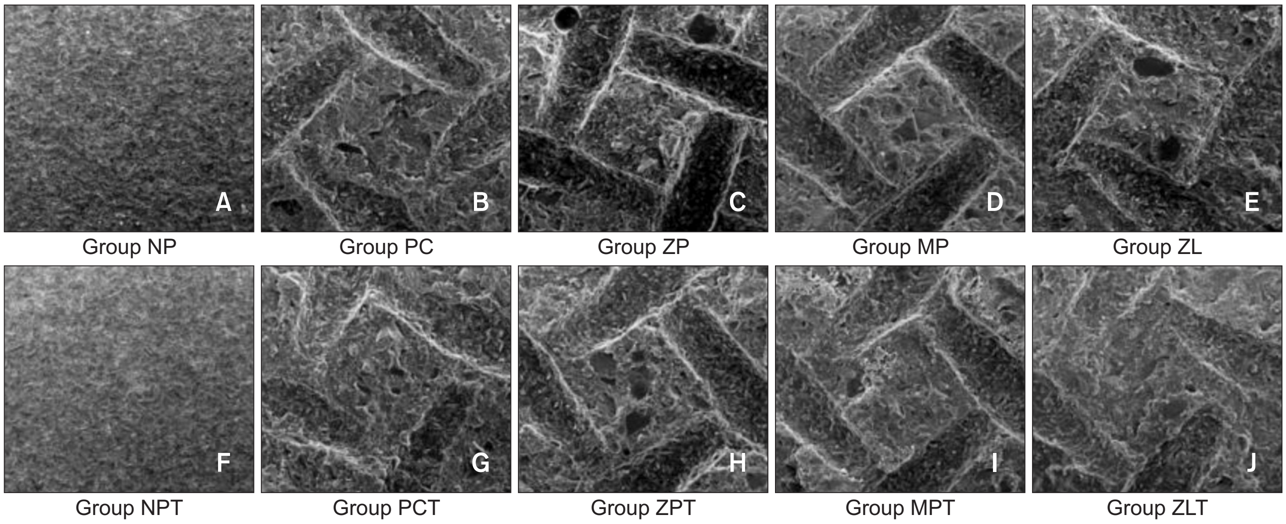

Figure 3. Representative scanning electron micrographs (200×) of the adhesive failure mode (A, F) and the mixed failure mode (B–E, G–J). NP, No-primer; NPT, no-primer with thermocycling; PC, Porcelain Conditioner; PCT, Porcelain Conditioner with thermocycling; ZP, Z-PRIME Plus; ZPT, Z-PRIME Plus with thermocycling; MP, Monobond Plus; MPT, Monobond Plus with thermocycling; ZL, Zirconia Liner Premium; ZLT, Zirconia Liner Premium with thermocycling.

(NP and NPT) displayed adhesive failure modes, but the groups with zirconia primers (ZP, ZPT, MP, MPT, ZL, and ZLT) were associated with mixed failure modes (Table 7). Micrographs of representative failure modes are shown in Figures 2 and 3.

DISCUSSION

In this study, the effect of zirconia primers on the SBS of orthodontic metal brackets was determined. Kern and Wegner[15] reported that sandblasting treatment increased the roughness of the zirconia surface, but compared to other alloys, it also decreased the number of undercuts and failed to maintain bond stability after water invasion. Similarly, the SBS of group NPT was 49% of the bond strength for group NP.

The control groups that lacked primer showed the lowest SBS both with and without thermocycling (Table

5). Reynolds[16] indicated that the minimum SBS able to resist normal orthodontic forces is 5.9−7.9 MPa, and McCarthy and Hondrum[17] reported a minimum value of 7 MPa. Based on these prior studies, groups NPT (5.68 MPa) and PCT (6.12 MPa) lack proper SBS. Group PC, which used a silane primer, exhibited significantly higher SBS than group NP ($p < 0.05$). The SBS of group PCT (which underwent thermocycling) was only 26.1% of the bond strength of group PC, and no significant difference was detected between PCT and NPT ($p > 0.05$).

Z-PRIME Plus contains biphenyl dimethacrylate and hydroxyethyl methacrylate. Monobond Plus largely comprises silane methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate (MDP), and sulfide methacrylate. Zirconia Liner Premium is similar to Monobond Plus, but does not contain sulfide methacrylate. Z-PRIME Plus is a mixture of phosphate and carboxylic acid monomers. Phosphate monomer can co-polymerize with resin monomers because its functional group bonds with the metal oxide of the substrate via its phosphoric acid group. Another monomer, carboxylic acid, helps form the bond to the substrate.[12,18] However, Lorenzoni et al.[19] reported that Z-PRIME Plus displayed lower bond strength than MDP-containing primers because the carboxylic acid monomer of Z-PRIME Plus weakened the bonding with the methacrylate group of the resin cement. Many studies have examined the efficacy of MDP monomer in chemical bonding with zirconium oxide on a zirconia surface[15,20,21] and found that MDP-containing primers maintain bonding stability both before and after thermocycling of a zirconia surface treated with sandblasting.[15,17,22,23] In the current study, MDP-containing primers also showed less strength reduction than observed in the other groups (39.2% MDP in groups MP and MPT; 39.6% MDP in groups ZL and ZLT).

Koizumi et al.[24] reported that the sulfide methacrylate monomer in Monobond Plus affects bond strength, but in our study, there was no significant difference in SBS between the MDP-containing primers (Monobond Plus and Zirconia Liner Premium) ($p > 0.05$).

For the ARI score, control groups NP and NPT exhibited an adhesive failure mode, reflecting the low SBS in these groups.[25] The groups using zirconia primers displayed mixed failure modes in which most of the bonding resin remained on the zirconia surface (Figure 2 and Table 7).[26]

CONCLUSION

Following the results of this study, regardless of thermocycling application, the groups without primer (NP and NPT) showed lower SBS and adhesive failure but the groups using zirconia primers (ZP, ZPT, MP, MPT, ZL, and ZLT) showed higher SBS and mixed failure. Thus, surface treatment with zirconia primer contributes to an increased in SBS compared with no-primer and silane primer application for orthodontic bracket bonding to zirconia prostheses.

REFERENCES

1. Kokich VG, Spear FM. Guidelines for managing the orthodontic-restorative patient. Semin Orthod 1997;3:3-20.
2. Wood DP, Jordan RE, Way DC, Galil KA. Bonding to porcelain and gold. Am J Orthod 1986;89:194-205.
3. Ortorp A, Kihl ML, Carlsson GE. A 3-year retrospective and clinical follow-up study of zirconia single crowns performed in a private practice. J Dent 2009;37:731-6.
4. Raigrodski AJ, Chiche GJ, Potiket N, Hochstedler JL, Mohamed SE, Billiot S, et al. The efficacy of posterior three-unit zirconium-oxide-based ceramic fixed partial dental prostheses: a prospective clinical pilot study. J Prosthet Dent 2006;96:237-44.
5. Guess PC, Zavanelli RA, Silva NR, Bonfante EA, Coelho PG, Thompson VP. Monolithic CAD/CAM lithium disilicate versus veneered Y-TZP crowns: comparison of failure modes and reliability after fatigue. Int J Prosthodont 2010;23:434-42.
6. Zhang Y, Chai H, Lee JJ, Lawn BR. Chipping resistance of graded zirconia ceramics for dental crowns. J Dent Res 2012;91:311-5.
7. Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. J Prosthodont Res 2013;57:236-61.
8. Wolf DM, Powers JM, O'Keefe KL. Bond strength of composite to etched and sandblasted porcelain. Am J Dent 1993;6:155-8.
9. Kato H, Matsumura H, Atsuta M. Effect of etching and sandblasting on bond strength to sintered porcelain of unfilled resin. J Oral Rehabil 2000;27:103-10.
10. Kitayama S, Nikaido T, Takahashi R, Zhu L, Ikeda M, Foxton RM, et al. Effect of primer treatment on bonding of resin cements to zirconia ceramic. Dent Mater 2010;26:426-32.
11. Kern M, Barloi A, Yang B. Surface conditioning influences zirconia ceramic bonding. J Dent Res 2009;88:817-22.
12. Magne P, Paranhos MP, Burnett LH Jr. New zirconia primer improves bond strength of resin-based cements. Dent Mater 2010;26:345-52.
13. Atsu SS, Kilicarslan MA, Kucukesmen HC, Aka PS. Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. J Prosthet Dent 2006;95:430-6.

http://dx.doi.org/10.4041/kjod.2015.45.4.164

14. Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984;85:333-40.
15. Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. Dent Mater 1998;14:64-71.
16. Reynolds I. A review of direct orthodontic bonding. Br J Orthod 1975;2:171-8.
17. McCarthy MF, Hondrum SO. Mechanical and bond strength properties of light-cured and chemically cured glass ionomer cements. Am J Orthod Dentofacial Orthop 1994;105:135-41.
18. Chen L, Suh BI, Brown D, Chen X. Bonding of primed zirconia ceramics: evidence of chemical bonding and improved bond strengths. Am J Dent 2012;25:103-8.
19. Lorenzoni FC, Leme VP, Santos LA, de Oliveira PC, Martins LM, Bonfante G. Evaluation of chemical treatment on zirconia surface with two primer agents and an alkaline solution on bond strength. Oper Dent 2012;37:625-33.
20. Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. Quintessence Int 2007;38:745-53.
21. Blatz MB, Sadan A, Martin J, Lang B. In vitro evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling. J Prosthet Dent 2004;91:356-62.
22. Lüthy H, Loeffel O, Hammerle CH. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. Dent Mater 2006;22:195-200.
23. Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. Dent Mater 2007;23:45-50.
24. Koizumi H, Nakayama D, Komine F, Blatz MB, Matsumura H. Bonding of resin-based luting cements to zirconia with and without the use of ceramic priming agents. J Adhes Dent 2012;14:385-92.
25. Lee SM, Hwang CJ. A comparative study of frictional force in self-ligating brackets according to the bracket-archwire angulation, bracket material, and wire type. Korean J Orthod 2015;45:13-9.
26. Ryu MJ, Gang SN, Lim SH. Effect of silica coating on bond strength between a gold alloy and metal bracket bonded with chemically cured resin. Korean J Orthod 2014;44:105-12.