Effect of primers on bonding of metal bracket to zirconia

MILIM LEE

The Graduate School

Yonsei University

Department of Dental Science

Effect of primers on bonding of metal bracket to zirconia

Directed by Professor CHUNG-JU HWANG

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MILIM LEE

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This certifies that the dissertation of

MI LIM LEE is approved.

Thesis supervisor: Chung-Ju Hwang

Jung-Yul Cha

Kyoung-Nam Kim

The Graduate School

Yonsei University

June 2014

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저자 씀

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ABSTRACT

Effect of primers on bonding of metal bracket to zirconia

MILIM LEE

Department of Dental Science

The Graduate School, Yonsei University

(Directed by Professor CHUNG-JU HWANG)

The use of zirconia in conservative aesthetic dentistry has become more common. With this trend, it's been extensively investigated to get acceptable bond strength with zirconia surface. But these studies are all focusing on resin luting cement with "naked" zirconia. Therefore, the aims of this study were to compare shear bond strength of resin cement to zirconia surface with using different primers and determine the effective surface treatment method for metal bracket bonding, zirconia primer or conventional porcelain primer.

Zirconia blocks(Zirkonzhan) were milled and embedded in cylinder-shaped acrylic resin required for using testing machine. Specimens were randomly assigned to four groups (n=10): NZ, Non-glazed surface / Sand blasting + Zirconia primer; GZ, Glazed surface / Sand blasting + Etching + Zirconia primer; GP, Glazed surface / Sand blasting + Etching + Porcelain primer and GZP, Glazed surface / Sand blasting + Etching + Zirconia primer + Porcelain primer. Mandibular incisor stainless steel metal bracket was bonded to target surface with resin cement and all specimens underwent thermal cycling for 2,000 cycles (5 ℃ to 55 ℃) with a dwelling time of 30 seconds.

Shear bond strengths of specimens were measured using a universal testing machine at a cross-head speed of 1 mm/min. Scanning electron microscopy, 3D optical profiler and stereoscopic microscopy were used for imaging the zirconia surface before and after surface roughening and debonding. The data were analyzed by one-way ANOVA and Fisher's exact test (p<.05).

The group GZ, using zirconia primer on glazed surface showed significantly lower shear bond strength than other groups. As for group NZ, GP and GZP, there was no statistically significant difference among them. While specimens in group NZ and GP showed adhesive failure between the resin cement/bracket base or complex adhesive and cohesive failure, all specimens in group GZ failed at the zirconia/resin cement interface.

Based on the results of this study, it does not seem proper to use a zirconia primer to bond metal bracket on the surface of full contour zirconia crown with resin cement. In such situations, the use of porcelain primer is more appropriate.

Key words: metal bracket bonding, zirconia, glazing, primer, shear bond strength

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MILIM LEE D.D.S.

Department of Dental Science

The Graduate School, Yonsei University

(Directed by Prof. CHUNG-JU HWANG, D. D. S., M. S. D., Ph. D.)

Ⅰ. INTRODUCTION

Zirconium oxide(ZrO_2), zirconia is characterized by superior mechanical properties such as high flexural strength, relatively low elastic modulus and a high fracture toughness compared to other ceramic materials (Manicone et al., 2007; Piconi and Maccauro, 1999). Yttria(Y_2O_3) is added to zirconia for improving mechanical properties, and yttria tetragonal zirconia polycrystal(Y-TZP) material is the type used in dentistry.

The use of zirconia in conservative aesthetic dentistry become more common due to increasing esthetic demands from patients and considerable developments of CAD/CAM (computer-aided design, computer–aided manufacturing) technologies including the performance of scanners, CAD software and net-worked machining centers (Cortellini et al., 2006). And such a trend has widen its area of application to the field of anterior and posterior fixed prosthesis, endodontic post, implant abutment and orthodontic bracket (Bertolini et al., 2014; Keith et al., 1994). As for fixed prosthesis, zirconia was just a substitution of metal for metal-free restorations in early days, and porcelain veneering was usually done because of the aesthetic limitations of zirconia. But, since the porcelain layer of zirconia-core crown is too thin to bear posterior heavy occlusal force, chipping or fracturing of veneering porcelain reported frequently (Agustin-Panadero et al., 2014; Ortorp et al., 2009). Recently, however, with improvement of opacity of Y-TZP and technique for precise machining and coloring zirconia block (Miyazaki et al., 2013), the clinical application of the full contour zirconia crown has been attempted actively (Guess et al., 2010).

With increasing of clinical application of zirconia and increasing demand for adult orthodontic treatment, an orthodontist has more chance to meet a zirconia surface on which one should bond orthodontic appliances. And even a crown with porcelain veneering on labial side often doesn't have veneering on lingual side, an orthodontist should know how to prepare the bonding surface made of zirconia for bonding of lingual bracket and fixed retainer.

Zirconia is separated material with feldspathic porcelain, so it's been extensively investigated to get acceptable bond strength with zirconia surface. As a result of these efforts, several methods have been suggested.

As there is no silica in its composition and it lacks a vitreous phase, conventional hydrofluoric acid etching and silanization procedures are incapable of modifying the zirconia surface (Foxton et al., 2011; Kitayama et al., 2010; Ozcan et al., 2011; Takeuchi et al., 2010). Sandblasting with Al_2O_3 particles is conventional roughening technique which is essential for mechanical interlocking of resin cement (Ozcan and Vallittu, 2003), but it can affect minimally on the zirconia surface (Borges et al., 2003). Sandblasting with silica-coated aluminum oxide particle followed by silane application has been attempted (tribochemistry) (Ruttermann et al., 2008). However, it has been questioned whether these transferring and embedding of silica from particles actually happen since the intrinsic hardness of the target material (Ernst et al., 2005). Recently, as another method, hot chemical solution etching has been attempted. This method utilizes the metallic nature of pure zirconium, but it is not applicable to the orthodontic clinic because the optimal temperature for processing is 100 ℃, for a period of 30 minutes (Xie et al., 2013).

To date, various adhesive primers for zirconia were developed. This chemistry enhance the adhesion between hydrophobic resin cements and indirect oxide-based substrates. The anhydride group present in 4-META monomer (Kumbuloglu et al., 2006), and for another type, the phosphate ester group of MDP monomer (Kumbuloglu et al., 2006; Wolfart et al., 2007) chemically bonds to zirconia ceramics. But these primers have been invented with focusing on “naked” zirconia surface – bonding between zirconia surface without glazing and resin cement.

Because there are many commercialized zirconia primer, an orthodontist possibly applies the primer on prosthesis made of zirconia for bracket bonding. However, we have to check a mechanism of zirconia primer and think about what proper surface preparation method is to get acceptable shear bond strength of resin cement with zirconia which had been done with glazing process for highly aesthetic, glossy surface. Therefore, the aims of this study were to compare shear bond strength of resin cement to zirconia surface with using different primers and determine the effective surface treatment method for orthodontic bracket bonding, zirconia primer or conventional porcelain primer.

Ⅱ. MATERIALS AND METHODS

1. Preparation of zirconia blocks and acrylic resin cylinders

Industrially manufactured Y-TZP ceramic blocks (Zirkonzhan, Prettau Blocks, Italy) were used. After design with Rhinoceros software, the zirconia blocks got CAD/CAM milled with size 8.0 x 8.0 mm^2 and a thickness of 5.0 mm (n=40), colored with Zirkonzhan color kit(dipping for 3 minutes according to manufacturers' instructions) and sintered in furnace for zirconia (firing at 1600 ℃ for 11 hours, Zirkonofen 600; Zirkonzhan)

As for glazed zirconia group, glaze liquid (INsitu Glaze Liquid, GC initial™, USA) was applied on sintered surface with brush and re-fired at 800 ℃ for 90 seconds in porcelain furnace. It was intended to reproduce the surface state delivered in dental clinic. All steps were done by one dental technician according to manufacturers' instructions for consistent surface production.

Total of 40 zirconia blocks were embedded in acrylic resin (Ortho-Jet™, Lang Dental Manufacturing Co., Inc., USA) with diameter 30.0 mm and a height of 15.0 mm for testing machine.

2. Surface roughening; Alumina sandblasting with/without HF etching

The specimens were randomly assigned to four groups of ten specimens each and treated according to the conditions as described in Table 1. Because hydrofluoric etching cannot change the surface structure of zirconia (Borges et al., 2003), etching process was only done with glazed specimens.

Table 1. Classification of study groups

Test group	Surface treatment
NZ	Non-glazed surface / Sand blasting + Zirconia primer
GZ	Glazed surface / Sand blasting + Etching + Zirconia primer
GP	Glazed surface / Sand blasting + Etching + Porcelain primer
GZP	Glazed surface / Sand blasting + Etching + Zirconia primer + Porcelain primer

Alumina sandblasting was performed with mean particle size at 50 ㎛ for 5 seconds at 40 psi pressure (PROPHYflex 3-2018, KaVo Dental GmbH). Distance of the tip from the surface was about 5 mm.

For specimens of GZ, GP and GZP group, sandblasted surface was etched with 9 % hydrofluoric acid (Reliance Porc-Etch™, Reliance Orthodontic Produces, Inc., USA) for 4 minutes.

3. Bracket bonding with primers and resin cement

After the surface roughening procedure, wash and dry surface thoroughly and apply one thin coat of zirconia primer (Zirconia Liner Premium, Sun medical, Japan) or porcelain primer (Reliance Porcelain Conditioner, Reliance Orthodontic Produces, Inc., USA).

Mandibular incisor stainless steel metal bracket (Tomy Inc., Japan) was bonded to target surface with resin cement (Transbond™ XT, 3M Unitek, USA). After removal of excessive resin around bracket, resin was polymerized with plasma arc lamp curing light (Flipo, LOKKI s.a., France) at a light intensity 1100 ㎽/㎠ for 10 seconds.

All materials were used according to manufacturers' instructions.

4. Shear bond strength test

All specimens underwent thermal cycling (KD-TCS30, Kwang-duk F.A., Korea) for 2,000 cycles (5 ℃ to 55 ℃), with a dwelling time of 30 seconds (Ando et al., 2001). After the procedure, all specimens were stored in distilled water at 37 ℃.

Shear bond strengths of specimens were measured by a universal testing machine (Model 3366, Instron® Co., USA) at a cross-head speed of 1 mm/min. And they were divided by area of bracket base(9.81 mm^2) and converted to units of MPa(Figure 1).

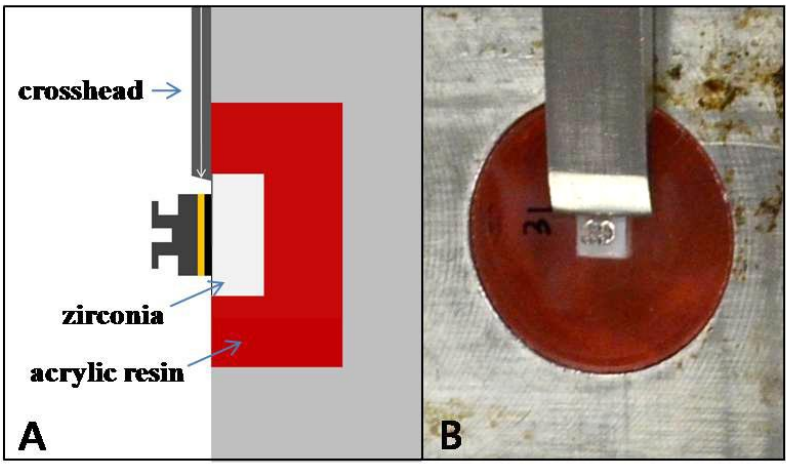

Figure 1. A, Schematic illustration for testing shear bond strength; B, Equipment for measuring shear bond strength.

5. Surface imaging

Scanning electron microscopy (S-3000N, Hitachi, Tokyo, Japan) was used for imaging the zirconia surface before and after surface roughening procedure with magnifications of x1,000 at an operating voltage of 15 kV.

Roughened surfaces were analyzed with a three-dimensional optical surface profiler (NewView 6300, Zygo Corp., Middlefield, CT,USA) based on non-contact scanning white light interferometry to evaluate three-dimensional surface.

Failure mode analysis was performed with a stereoscopic microscope (SZ61, Olympus, Tokyo, Japan) at x16 magnification and classified into three categories; adhesive failure between zirconia/resin cement, adhesive failure between resin cement/bracket base, complex adhesive and cohesive failure.

6. Statistical analysis

All preparation and testing procedures respectively were done by same operator to eliminate inter-operator variability. The data were submitted to one-way ANOVA and multiple comparison with Turkey's HSD(Honestly Significant Difference) test (SPSS version 21.0, SPSS Inc., Chicago, USA) to find differences of shear bond strength between groups depending on the type of primers used. Fisher's exact test was used for evaluation of relativeness between the primer and failure mode. The level of statistical significance was set at $\alpha = .05$.

Ⅲ. RESULTS

1. The effect of primers on shear bond strength of resin cement

Mean shear bond strength values(MPa) and standard deviations for each group are presented in Table 2 and Figure 2. One-way ANOVA revealed that the group GZ, using zirconia primer on glazed surface showed significantly lower shear bond strength values than other groups. As for group NZ, GP and GZP, there was no statistically significant difference among them(Table 3, 4).

Table 2. Comparison of the values of shear bond strength (unit: MPa)

Group	Mean	SD	Minimum	Maximum
NZ	13.53	2.60	9.98	18.04
GZ	3.79	1.23	2.07	5.51
GP	16.07	4.17	10.87	22.16
GZP	14.48	2.59	11.32	18.83

MPa, megapascal; SD, standard deviation

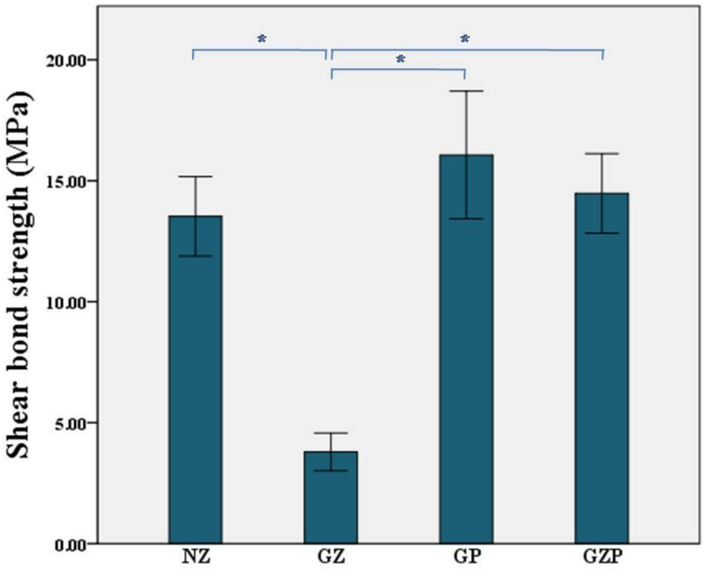

Figure 2. Comparison of the values of shear bond strength.

*. Statistically significant ($p < .05$)

Table 3. One-way ANOVA for experimental groups

	Sum of squares	df	Mean square	F-value	Sig.
Between group	923.42	3	307.81	37.97	0.000
Within groups	291.82	36	8.11		

df, degree of freedom; Sig., significance

Table 4. Tukey's HSD test

Group (I)	Group (J)	Mean difference (I-J)	Std. error	Sig.
NZ	GZ	9.74	1.27	*
NZ	GP	-2.54	1.27	NS
NZ	GZP	-0.95	1.27	NS
GZ	GP	-12.27	1.27	*
GZ	GZP	-10.68	1.27	*
GP	GZP	1.59	1.27	NS

Std., standard; Sig., significance; NS, non significant

*. Statistically significant ($p < .05$)

2. Surface imaging of the specimens

SEM images of zirconia surface before and after roughening procedure are presented in Figure 3. Figure 3A is a milled surface of zirconia(group NZ) which shows a slightly rough surface result of the cutting operation. Figure 3C is a glazed surface(group GZ, GP and GZP) which shows plane surface with a few pits on it.

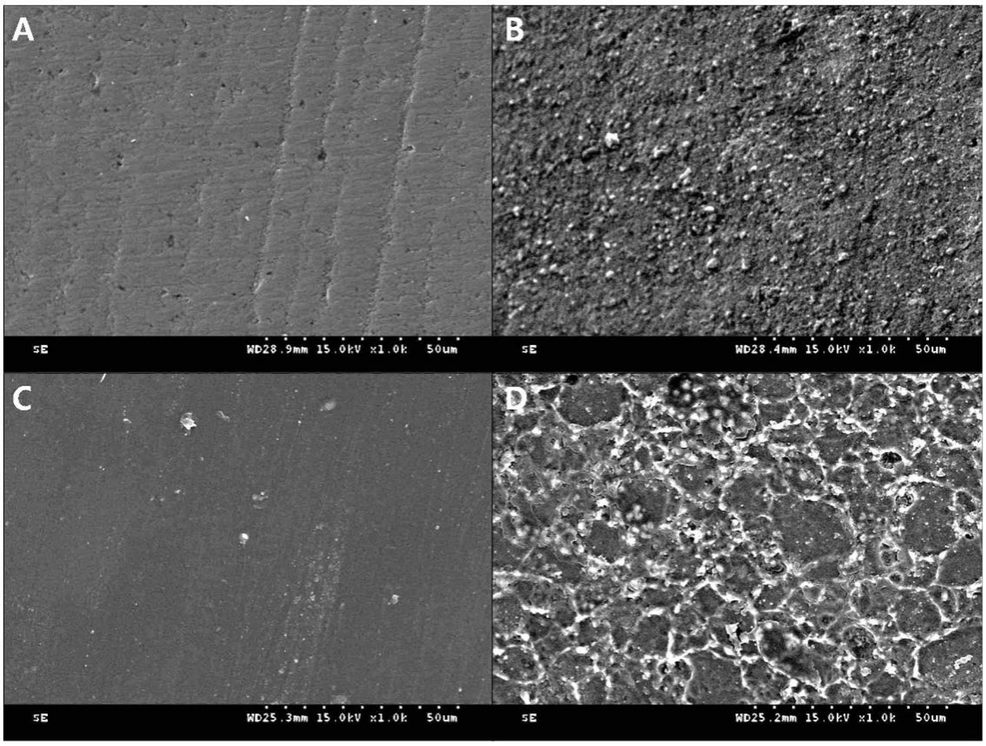

Figure 3. SEM images of zirconia surface (magnification x1,000). A, Zirconia without glazing, B, Sandblasting of A; C, Zirconia with glazing; D, Sandblasting and acid etching of C.

Sandblasting (and acid etching for glazed specimens) resulted in increase of surface roughness(A→B, C→D). Roughened surface of glazed zirconia(Figure 3D) has totally different condition from roughened surface of milled zirconia(Figure 3B), rather than simply the difference in the area of rough part.

3D optical profiler images corresponding to the SEM images are presented in Figure 4. Zirconia surface were minimally affected by sandblasting comparison with glazed surface.

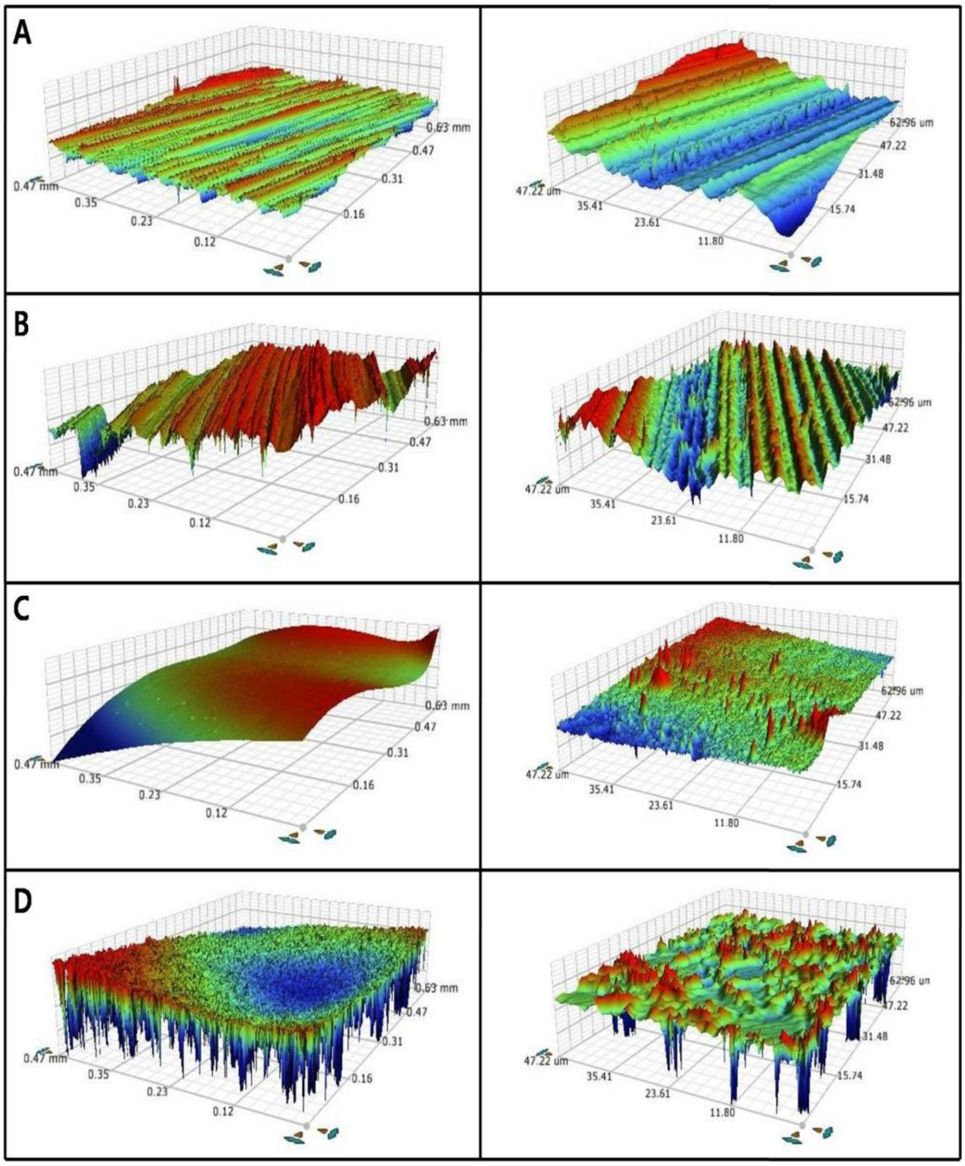

Figure 4. 3D optical profiler images of zirconia surface (magnification: left, x10; right, x100). A, Zirconia without glazing; B, Sandblasting of A; C, Zirconia with glazing; D, Sandblasting and acid etching of C.

3. Failure mode analysis

Failure mode analysis was performed with a stereoscopic microscope. There are pictures representing each failure mode(Figure 5), and the results are shown in Table 5.

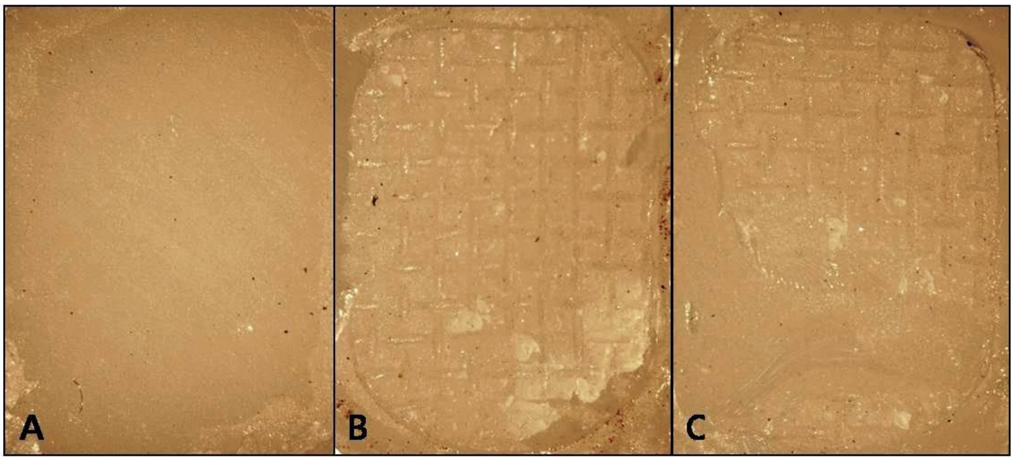

Figure 5. Stereoscopic micrographs of the debonded surface (magnification x16). A, Adhesive failure between zirconia/resin cement; B, Adhesive failure between resin cement/ bracket base; C, Complex adhesive and cohesive failure.

Table 5. Failure mode after shear bond strength testing

	NZ	GZ	GP	GZP
Adhesive btw zirconia/resin		10		
Adhesive btw resin/bracket	7		6	
Complex adhesive and cohesive	3		4	10

All specimens in group GZ showed adhesive failure between the zirconia/resin cement. In group NZ and ZP, the bonding failed at the resin cement/bracket base interface or showed complex adhesive and cohesive failure. As for specimens in group GZP treated with zirconia primer followed by porcelain primer, all showed complex adhesive and cohesive failure(Table 5). Fisher's exact test with $p < .05$ was used to establish significance among groups. Only group GZ was statistically distinguished from the other groups.

Ⅳ. DISCUSSION

In this study, shear bond strengths were measured to confirm which one is better for orthodontic bracket bonding to full contour zirconia crown, recently commercialized zirconia primer or conventional porcelain primer.

According to the results, group GZ, zirconia primer with glazed surface shows significantly lower shear bond strength, while previous studies on surface roughening procedure followed by using zirconia primer resulted in improvement of shear bond strength (Qeblawi et al., 2010; Tsuo et al., 2006). It means that glazing layer of full contour zirconia crown encountered in clinic is not removed by the conventional roughening method, and one cannot expect the zirconia primer's action when target surface had been glazed.

Zirconia Liner Premium, a zirconia primer used for this study, is mainly(97 %) composed of methyl methacrylate(MMA), and for the rest, there are 10-methacryloyloxydecyl dihydrogen phosphate(MDP) and 4-methoxyphenol(HQME), 3 %, 0.01 % respectively. The MDP monomer is effective to bond formation between resin cement and zirconia, by working in a way that the phosphate ester group of the MDP bonds directly to metal oxides such as chromium, nickel, aluminum, tin, titanium, and zirconium oxides (Yoshida et al., 2006).

Porcelain primer, silane coupling agent, is effective for creating a strong bond between resin cement and silica-based ceramics by increasing the wettability of the ceramic surface for the resin cement (Debnath et al., 2003).

As presented in this study, because the glazing layer which can be affected by silane coupling agent still remains after conventional roughening method, it doesn't seem proper to use a zirconia primer on glazed surface, instead, porcelain primer can provide acceptable shear bond strength between them. In other words, zirconia primer works only with non-glazed zirconia surface. And porcelain primer is required when bonding orthodontic brackets on glazed zirconia surface.

About failure mode, while specimens in group NZ and GP showed adhesive failure between the resin cement/bracket base or complex adhesive and cohesive failure, specimens in group GZ all showed adhesive failure between the zirconia/resin cement. This is consistent with the results of shear bond strength test.

Because aesthetics of lingual side of prosthesis is less important, glazing layer might be thin or rarely done, and the zirconia surface can be exposed during conventional roughening procedure. In such a situation, we have to decide which surface we should choose over another surface, or have to know whether the two different primers for each surface can be used at the same time without lowering their adhesion ability.

Specimens in group GZP were roughened with sandblasting and acid etching, and applied zirconia primer followed by porcelain primer. In the results, the frequency of complex adhesive and cohesive failure increased when using the zirconia primer and porcelain primer together compared to group NZ and GP dominant of adhesive failure between resin cement/bracket base. However, since there is no fully direct relationship between shear bond strength value with failure mode, even several specimens of group GZP showed greater bond strength values than others using single primer, and the measured values of group GZP in this study were far over 6-9 MPa, which is known as a minimum shear bond strength that can bear the force applied during orthodontic tooth movement (Lopez, 1980; McCarthy and Hondrum, 1994), it is a promising suggestion that using a zirconia primer and porcelain primer together is an alternative for getting acceptable bond strength between the two surface when it seems like part of glazed layer removed after roughening procedure.

Recently, there are several studies about crack development of zirconia surface after sandblasting (Guazzato et al., 2005; Karakoca and Yilmaz, 2009; Kosmac et al., 2000), and Tsuo et al. reported that even though the surface becomes rough as the alumina particle size increases, the shear bond strength does not increase proportionally (Tsuo et al., 2006). For this reason, it might be recommendable to use alumina particle size of 50㎛ which is typical size used clinically to clean the inner surface of restorations (Wang et al., 2008).

In this study, we measured shear bond strength under different conditions and concluded that primer for silica-based ceramics is effective method for pre-treating glazed zirconia surface to get clinically acceptable bond strength. Further studies with various types of bracket and adhesive materials would be more helpful for bonding attachments to zirconia restorations.

Ⅴ. CONCLUSIONS

The purpose of this study were to compare shear bond strength of resin cement to zirconia surface with different primers and determine the effective surface treatment method for orthodontic bracket bonding, zirconia primer or conventional porcelain primer.

The results were as follows.

1. Shear bond strengths were significantly low under condition of zirconia primer with glazed zirconia surface(group GZ) compared with other groups ($p<.05$).
2. There were no statistically significant difference between the shear bond strengths of zirconia primer with non-glazed zirconia surface(group NZ) and porcelain primer with glazed surface(group GP) ($p>.05$).
3. Using of both zirconia primer and porcelain primer together(group GZP) did not lower the shear bond strengths when compared with that of each primer to its surface alone(group NZ, GP) ($p>.05$).

4. All specimens of glazed zirconia with zirconia primer resulted in adhesive failure between the zirconia/resin cement. Zirconia primer with non-glazed surface and porcelain primer with glazed surface showed adhesive failure between the resin cement/bracket base or complex adhesive and cohesive failure. All specimens of two primers together(group GZP) resulted in complex adhesive and cohesive failure.

Based on the results described above, it does not seem proper to use a zirconia primer to bond metal bracket on the surface of full contour zirconia crown with resin cement. In such situations, the use of porcelain primer is more appropriate.

REFERENCE

Agustin-Panadero R, Roman-Rodriguez JL, Ferreiroa A, Sola-Ruiz MF, Fons-Font A: Zirconia in fixed prosthesis. A literature review. *J Clin Exp Dent* 6(1): e66-e73, 2014.

Ando F, Komori A, Kojima I: Tensile bond strength of light-cured resin-reinforced glass ionomer cement with delayed light exposure. *Odontology* 89(1): 45-48, 2001.

Bertolini MD, Kempen J, Lourenco EJ, Telles DD: The use of CAD/CAM technology to fabricate a custom ceramic implant abutment: A clinical report. *J Prosthet Dent*, 2014.

Borges GA, Sophr AM, de Goes MF, Sobrinho LC, Chan DC: Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent* 89(5): 479-488, 2003.

Cortellini D, Valenti M, Canale A: The metal-free approach to restorative treatment planning. *Eur J Esthet Dent* 1(3): 230-247, 2006.

Debnath S, Wunder SL, McCool JI, Baran GR: Silane treatment effects on glass/resin interfacial shear strengths. *Dent Mater* 19(5): 441-448, 2003.

Ernst CP, Cohnen U, Stender E, Willershausen B: In vitro retentive strength of zirconium oxide ceramic crowns using different luting agents. *J Prosthet Dent* 93(6): 551-558, 2005.

Foxton RM, Cavalcanti AN, Nakajima M, Pilecki P, Sherriff M, Melo L, et al.: Durability of resin cement bond to aluminium oxide and zirconia ceramics after air abrasion and laser treatment. *J Prosthodont* 20(2): 84-92, 2011.

Guazzato M, Quach L, Albakry M, Swain MV: Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic. *J Dent* 33(1): 9-18, 2005.

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* 23(5): 434-442, 2010.

Karakoca S, Yilmaz H: Influence of surface treatments on surface roughness, phase transformation, and biaxial flexural strength of Y-TZP ceramics. *J Biomed Mater Res B Appl Biomater* 91(2): 930-937, 2009.

Keith O, Kusy RP, Whitley JQ: Zirconia brackets: an evaluation of morphology and coefficients of friction. *Am J Orthod Dentofacial Orthop* 106(6): 605-614, 1994.

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* 26(5): 426-432, 2010.

Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L: Strength and reliability of surface treated Y-TZP dental ceramics. *J Biomed Mater Res* 53(4): 304-313, 2000.

Kumbuloglu O, Lassila LV, User A, Vallittu PK: Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods. *Oper Dent* 31(2): 248-255, 2006.

Lopez JI: Retentive shear strengths of various bonding attachment bases. *Am J Orthod* 77(6): 669-678, 1980.

Manicone PF, Rossi Iommetti P, Raffaelli L: An overview of zirconia ceramics: basic properties and clinical applications. *J Dent* 35(11): 819-826, 2007.

McCarthy MF, Hondrum SO: Mechanical and bond strength properties of light-cured and chemically cured glass ionomer cements. *Am J Orthod Dentofacial Orthop* 105(2): 135-141, 1994.

Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T: Current status of zirconia restoration. *J Prosthodont Res* 57(4): 236-261, 2013.

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* 37(9): 731-736, 2009.

Ozcan M, Cura C, Valandro LF: Early bond strength of two resin cements to Y-TZP ceramic using MPS or MPS/4-META silanes. *Odontology* 99(1): 62-67, 2011.

Ozcan M, Vallittu PK: Effect of surface conditioning methods on the bond strength of luting cement to ceramics. *Dent Mater* 19(8): 725-731, 2003.

Piconi C, Maccauro G: Zirconia as a ceramic biomaterial. *Biomaterials* 20: 1-25, 1999.

Qeblawi DM, Munoz CA, Brewer JD, Monaco EA, Jr.: The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement. *J Prosthet Dent* 103(4): 210-220, 2010.

Ruttermann S, Fries L, Raab WH, Janda R: The effect of different bonding techniques on ceramic/ resin shear bond strength. *J Adhes Dent* 10(3): 197-203, 2008.

Takeuchi K, Fujishima A, Manabe A, Kuriyama S, Hotta Y, Tamaki Y, et al.: Combination treatment of tribochemical treatment and phosphoric acid ester monomer of zirconia ceramics enhances the bonding durability of resin-based luting cements. *Dent Mater J* 29(3): 316-323, 2010.

Tsuo Y, Yoshida K, Atsuta M: Effects of alumina-blasting and adhesive primers on bonding between resin luting agent and zirconia ceramics. *Dent Mater J* 25(4): 669-674, 2006.

Wang H, Aboushelib MN, Feilzer AJ: Strength influencing variables on CAD/CAM zirconia frameworks. *Dent Mater* 24(5): 633-638, 2008.

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* 23(1): 45-50, 2007.

Xie H, Chen C, Dai W, Chen G, Zhang F: In vitro short-term bonding performance of zirconia treated with hot acid etching and primer conditioning etching and primer conditioning. *Dent Mater J* 32(6): 928-938, 2013.

Yoshida K, Tsuo Y, Atsuta M: Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. *J Biomed Mater Res B Appl Biomater* 77(1): 28-33, 2006.

국 문 요 약

지르코니아 표면에 교정용 메탈 브라켓 부착시 프라이머의 효과

이 미 림

연세대학교 대학원 치의학과

(지도교수 : **황 충 주**)

심미치과영역에서 지르코니아의 사용이 증가하면서 지르코니아 표면과 레진 시멘트 간에 임상적으로 허용 가능한 결합력을 얻기 위한 연구가 많이 이루어졌다. 하지만 이러한 연구들은 '절삭 가공 및 소결된' 지르코니아 표면과 레진 시멘트의 접착에 초점을 두었다.

본 연구에서는 글래이징 처리가 된 지르코니아 표면에 지르코니아 프라이머 처리를 한 군과 포세린 프라이머 처리를 한 군을 비교하여, 어느 것을 사용하는

것이 지르코니아와 레진 시멘트 간 적절한 전단결합강도를 얻기 위해 더 효과적인지 알아보고자 하였다.

지르코니아 블럭(Zirkonzhan)을 아크릴릭 레진에 고정하여 전단결합강도 실험을 위한 시편을 제작하였다. 시편을 임의로 네 군으로 각각 10개씩 배정하였다. 해당 군의 조건에 따라 표면 처리 후, 레진 시멘트를 이용하여 금속 하악 절치 브라켓을 부착하고 열순환 처리를 시행하고 만능재료시험기로 브라켓의 전단결합강도를 측정하였다. 지르코니아 시편에서 브라켓이 탈락하는 순간의 최대 힘(N)을 측정하고 이를 브라켓 베이스 면적으로 나누어 전단결합강도를 MPa 단위로 환산하였다. 샌드블라스팅 및 산부식 전과 후의 표면을 주사전자현미경과 3차원 광학식 형상측정기로 관찰하였고, 전단결합강도 측정 후 브라켓 접착면의 파절 양상을 실체 현미경으로 평가하였다.

글래이징 처리가 된 지르코니아 표면에 지르코니아 프라이머를 사용한 GZ 군의 전단결합강도가 나머지 세 군에 비해 통계적으로 유의하게 낮은 값을 보였다($p<.05$). NZ, GP, GZP 군 간에는 통계적으로 유의한 차이가 없었다($p>.05$). 장치 제거 시 파절 양상의 평가에서, NZ, GP 군의 시편은 레진 시멘트/브라켓 베이스 계면에서의 접착성 실패 또는 접착성과 응집성 실패 양상이 함께

관찰되는 혼합성 실패를 보인 반면, GZ 군의 시편은 모두 지르코니아/레진 시멘트 계면에서의 접착성 실패를 보였다.

본 연구의 결과, 임상에서 풀지르코니아 크라운(full contour zirconia crown)에 교정장치를 부착할 때에는 지르코니아 프라이머를 사용하는 것 보다 포세린 프라이머를 사용하는 것이 적절한 방법일 것이다.

핵심 되는 말: 교정용 메탈 브라켓 부착, 지르코니아, 글래이징, 프라이머, 전단결합강도