Comparison of shear bond strength of orthodontic tube in glazed zirconia prostheses according to the surface treatment methods

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아버지, 어머니 사랑합니다. 가장 가까운 곳에서 저를 응원해주고 도와주는, 평생을 함께할 사랑하는 남편에게도 감사의 마음을 전하며, 언제나 저에게 긍정적인 마음을 갖게 해주는 사랑하는 수미언니와 동생에게도 감사하는 마음을 전합니다. 마지막으로, 논문을 작성하는데 많은 조언와 응원을 아끼지 않은 담추금 선생님에게도 고마움을 전합니다.

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ABSTRACT

Comparison of shear bond strength of orthodontic tube in glazed zirconia prostheses according to the surface treatment methods

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Current studies on zirconia bond strength mostly focus on “naked zirconia”, the inside of the zirconia crown. In the orthodontic field, tube bonding is required on the surface-“glazed” crown. In this study, shear bond strengths were measured and compared by bonding tubes not on “naked zirconia” but on “glazed zirconia” using various methods.

A control group was established when bonding the tube on the glazed porcelain surface using the usual methods. After glazing one side of zirconia block and dividing the experimental groups into four random groups, the tubes were bonded by using different processing methods on each of the surfaces and using different primers.

Control group : 50-μm Al_2O_3 + HF + Porcelain primer

Experimental group 1 : 50-μm Al_2O_3 + HF + Porcelain primer

Experimental group 2 : 50-μm Al_2O_3 + Zirconia primer

Experimental group 3 : 30-μm silica-coated alumina particles + Porcelain primer

Experimental group 4 : 110-μm silica-coated alumina particles + Porcelain primer

Shear bond strength in each of the groups was measured by using a universal test machine. Surface characteristics were observed on the glazed surface by using SEM and a 3D optical profiler prior to applying primer, and specimens were classified according to the failure patterns. The results were as follows :

1. Shear bond strength of experimental group 2 was 10.59 MPa, which was significantly lower than all other groups ($p < .05$).
2. Two groups, the control group and experimental group 1 were clearly observed in the SEM images to have similar rough surfaces and cracks. However, experimental groups 2, 3 and 4 were observed as having particles attached. Experimental group 4 was observed as having bigger sized particles and more irregular rough surfaces compared to experimental groups 2 and 3. In addition, according to the results of analysis using a 3D optical profiler, the average surface roughness Sa value in experimental group 3 was significantly lower than the values for experimental groups 1, 2, 4 ($p < .05$). The Sa value in the control group was significantly lower than the values for experimental groups 2, 4 ($p < .05$). However, no significant difference was recorded among the other groups ($p > .05$).
3. As for the failure type of debonding surface, only adhesive failure was manifest between zirconia block surface and resin cement interface in experimental group 2. In the rest of the groups, adhesive failure between resin cement and tube base interface was mostly manifest. Therefore, this reveals a significant difference between the experimental group 2 and the rest of the groups ($p < .001$).

According to the results of this study, using porcelain primer after sandblasting with silica coating particles is clinically simpler, safer and more effective method in relation to glazed zirconia crown because it has a similar high shear bond strength compared to other methods without the need for hydrofluoric acid.

Key words : zirconia, glazing, shear bond strength, orthodontic tube, primer, surface treatment, bonding failure

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I. Introduction

As dental materials have recently undergone rapid development, many studies have been conducted focusing on aesthetic materials with excellent mechanical properties. Aesthetically outstanding materials have been utilized in various fields including inlays or onlays, orthodontic brackets, implant abutments, crowns, and posts using CAD/CAM (Guess et al., 2011; Meyenberg et al., 1995; Nakamura et al., 2010). PFM (porcelain-fused-to-metal) that is currently being widely used in the field of dental prosthesis has several advantages including appropriate strength, long life-span, convenient

manufacturing, and affordable pricing (Miyazaki et al., 2013; Pjetursson et al., 2007; Sailer et al., 2007). However, as aesthetic materials become more favored, PFM has recently been replaced by all ceramic crown, and it is starting to be utilized in more diverse fields (Al-Amleh et al., 2010; Miyazaki et al., 2013).

All ceramic comprised of zirconia core and porcelain veneering not only has the various advantages of zirconia core including high flexural strength (Christel et al., 1989; Guess et al., 2011), low Young's modulus compared to alumina core (Christel et al., 1989), high wear resistance (Studart et al., 2007), fracture toughness (Christel et al., 1989; Guess et al., 2011), non-cytotoxic (Lohmann et al., 2002), has excellent biocompatibility (Studart et al., 2007; Warashina et al., 2003), and color stability (Al-Amleh et al., 2010), etc. and also retains the aesthetics of porcelain. Therefore, it is much favored as an aesthetic material because it has a similar level of natural tooth translucence and a more appropriate margin color than existing metals (Jung et al., 2007).

However, full zirconia crown has started to receive greater attention than zirconia core and porcelain veneering crown (Miyazaki et al., 2013) as the latter two often undergo porcelain chipping and delamination (Al-Amleh et al., 2010; Manicone et al., 2007; Miyazaki et al., 2013) because a weak point is produced when these two materials come into contact with each other in the crown (Aboushelib et al., 2007). This defect results from a difference in the thermal expansion coefficients between core and ceramic, the firing shrinkage of porcelain, flaws on the veneering and poor wetting by the veneering on the core. Studies have been continuously conducted (Zhang et al., 2015; Zhang, 2014) to reduce opaqueness (Al-Amleh et al., 2010; McLaren and Giordano, 2005; White et al., 2005) and the lack of color diversity that had been pointed out as disadvantage compared to the porcelain from the beginning. Full zirconia crown has been widely used in the molar in the replacement of metal or gold fixed prosthesis due to its mechanical strength unlike all other ceramics (Raigrodski, 2004).

As zirconia has become more and more used, the adhesive properties of full zirconia and the natural tooth became an area of interest in studies. Zirconia is not processed with hydrofluoric acid and silanization procedures cannot be carried out because of its glass-free property (Inokoshi et al., 2014a; Kern and Wegner, 1998; Thompson et al., 2011).

Therefore, there have been many studies undertaken on hydrophobic phosphate monomers (Koizumi et al., 2012; Lehmann and Kern, 2009; Maeda et al., 2014) such as 4-META monomer (Komine et al., 2009), 10-metacryloyloxydecyl dihydrogen phosphate (MDP) in order to improve the adhesive properties of zirconia. In addition, there have been many studies conducted to improve surface roughness (Kern et al., 2009) or to utilize tribochemically coating the surface with silica-coated alumina particles (Chen et al., 2014; Thompson et al., 2011). However, these methods did not achieve sufficient bond strength in comparison to cement used for gold or porcelain (Al-Amleh et al., 2010) and also made the surface rough and reduced the mechanical strength (Thompson et al., 2011; Zhang et al., 2004).

In the orthodontics field, there has been increasing interest in zirconia due to the usage of zirconia brackets. In addition, there has an increase in the number of cases where a bracket is attached to the zirconia surface as the use of zirconia crown is expanded. If a patient has a zirconia core and porcelain veneering crown in the anterior teeth, it is required to attach a lingual bracket or fixed maintenance device on the zirconia core that is exposed to lingual side. In addition, if a patient has a full zirconia crown in the posterior teeth, a bracket or tube is attached to the buccal surface of zirconia. In particular, there has been no case where the buccal surface of posterior teeth has been grinded with an occlusal adjustment. Therefore, a bracket or tube is attached where the grazed area is left as it is. In the posterior teeth, a tube is most likely used.

However, previous studies investigating the bond strength of zirconia have evaluated the bond strength with a natural tooth using cement in the inner side of zirconia crown and hence focused on “naked zirconia” (Al-Amleh et al., 2010; Chen et al., 2014; Koizumi et al., 2012; Lehmann and Kern, 2009; Maeda et al., 2014; Manicone et al., 2007; Thompson et al., 2011). Therefore, studies focusing on the bond strength of zirconia with a “glazed surface” that is applied in the field of orthodontics have started to be undertaken (Bavbek et al., 2014; Cura et al., 2012); however, these studies are still in the beginning phase.

This study is intended to measure and compare shear bond strength by bonding tubes on the surface-glazed zirconia using various methods. The objective of this study is 1) to

discover the surface conditioning method that produces the highest shear bond strength when bonding a single tube on the glazed zirconia surface and compare that with the shear bond strength between glazed porcelain surface and tube, 2) to observe the characteristics of the surface after applying the surface conditioning method using a scanning electron microscope and 3-dementional optical profiler, and 3) to observe failure types of bonding on the glazed zirconia surface.

II. Materials and methods

1. Specimen preparation

Y-TZP blocks (Prettau-Zirkon, Zirkonzahn; Gais, Italy) were used as specimens for this experiment. The 50 blocks were manufactured with CAD/CAM using Rhinoceros software. The blocks were divided into five groups including one control group and four experimental groups, and each group was composed of ten specimens. These blocks were sintered in a zirconia furnace according to the instructions of the manufacturer (firing at 1600 ℃ for 11 hours, Zirkonofen 600; Zirkonzhan). As for the final size of the blocks, the length, width, and height were produced in the following dimensions: 10 mm x 10 mm x 3 mm respectively. For the control group, to veneer the porcelain on the zirconia core, a groove with length, width, and height of 8 mm x 8 mm x 1 mm, respectively, was made on the Y-TZP block (n=10) manufactured with length, width, and height of 10 mm x 10 mm x 3 mm, respectively, and porcelain powder (Ceramic Dentic C4, Zirkonzahn; Gais, Italy) was poured in the groove and they were sintered in the porcelain furnace for 90 seconds at a temperature of 800 ℃ in a vacuum according to the instructions of the manufacturer. All the specimens manufactured in this way were sintered in the porcelain furnace for 90 seconds at a temperature of 800 ℃ after applying a glazing solution (GC Initial® IQ Lustre Pastes NF, GC Corporation; Tokyo, Japan) on one side exposed with porcelain in the control group and also one side of the zirconia in the experimental group by a well-trained technician using a brush.

The 50 glazed blocks were placed on the floor in the middle of a circular cylinder manufactured with a diameter of 30 mm and height of 15 mm in accordance with the size of the universal testing machine so that glazed surface of porcelain and zirconia was exposed, and were fixed by pouring acryl resin (Ortho-jet™ Acrylic Resin, Lang Dental Mfg. Co.; wheeling, IL, USA).

2. Surface conditioning method

Different conditioning methods were applied to the glazed porcelain in the control group and the glazed zirconia in the four experimental groups. For the control group, 50-μm alumina (Dentaurum; Ispringen, Germany), a general surface conditioning method of porcelain, was used. A nozzle was placed 10 mm away from the block surface and sprayed for 15 seconds with a pressure of 40 psi. Afterwards, 9 % of hydrofluoric acid (Reliance Porc-Etch™, Reliance Orthodontic Products; Itasca, IL, USA) was applied for four minutes followed by being cleansed with water and completely dried.

For the experimental group 1, 50-μm alumina was used in the same way as the control group and sprayed in the same method. Afterwards, 9 % of hydrofluoric acid was applied for four minutes followed by being cleansed with water and completely dried.

For the experimental group 2, 50-μm alumina was used and sprayed in the same method followed by applying air to remove residues.

For the experimental group 3, 30-μm silica-coated alumina particles (Cojet Sand, 3M ESPE; St Paul, MN, USA) were used and sprayed in the same method followed by applying air to remove residues.

For the experimental group 4, 110-μm diameter silica-coated alumina particles (Rocatec plus, 3M ESPE) and specialized nozzles (Rocatec junior, 3M ESPE) were used and sprayed in the same method followed by applying air to remove residues.

3. Tube bonding procedure with primer and resin cement

For the control group and experimental groups 1, 3, and 4, porcelain primer (Reliance Porcelain Conditioner, Reliance Orthodontic Products) was applied thinly and the surface was dried for 60 seconds. For the experimental group 2, zirconia primer (Z-Prime™ plus, Bisco; Schaumburg, IL, USA) was applied one or two times followed by air drying for three to five seconds.

Adhesive (Transbond^{TM} XT Light Cure Adhesive Primer, 3M Unitek; Monrovia, CA, USA) was applied on both the control group and experimental groups followed by applying an appropriate amount of resin cement (Transbond^{TM} XT Light Cure Adhesive Paste, 3M Unitek) on the base of a standard single tube (Tomy incorporated; Tokyo, Japan) and slightly pushing it after placing the tube in the middle of the block. After removing excessive resin coming out of the tube with an explorer, a plasma arch lamp curing light (Flipo, LOKKI s.a., France) was set at 1100 mW/cm^2 and the resin was polymerized for ten seconds. Afterwards, it was preserved in a dried condition for 24 hours at room temperature.

The materials and methods used in the experiment are summarized in the Table 1 and 2.

Table 1. Description of main materials used

Product name	**Manufacturer**	**Chemical composition**	**LOT number**
Prettau-Zirkon	Zirkonzahn; Gais, Italy	ZrO_2 (Specifications), Y_2O_3 (4-6 %), Al_2O_3 (<1 %), SiO_2 (max 0.02 %), Fe_2O_3 (max 0.01 %), Na_2O (max 0.04 %)	ZB2105B
Ceramic Dentine C4	Zirkonzahn; Gais, Italy	SiO_2, Al_2O_3, P_2O_5, K_2O, Na_2O, CaO, F, TiO_2 and pigments	KB10564B
GC Initial® IQ Lustre Pastes NF (Lustre Paste Neutral)	GC Corporation; Tokyo, Japan	SiO_2 (45 %), Al_2O_3 (7 %), K_2O (4 %), Na_2O (6 %), Propylene-glycol (25.5 %)	201402181
Ortho-jet™ Acrylic Resin	Lang Dental Mfg. Co.; Wheeling, IL, USA	Liquid Methyl Methacrylate (>95 %), N, N-Dimetyl-p-Toluidine (<2 %) Powder Polymer (<90 %), Diethyl phthalate (<20 %)	1334-14CS
Blasting Medium, white	Dentaurum; Ispringen, Germany	50-μm Al_2O_3 particles	411808
Porc-Etch™	Reliance Orthodontic Products; Itasca, IL, USA	9 % hydrofluoric acid	146949
Cojet Sand™	3M ESPE; St Paul, MN, USA	30-μm silica-coated alumina particles	531388

Rocatec plus™	3M ESPE; St Paul, MN, USA	110-µm diameter silica-coated alumina particles	499179
Z-Prime™ plus	Bisco; Schaumburg, IL, USA	Biphenyl dimethacrylate (BPDM), Hydroxyethyl methacrylate (HEMA),10-metacryloyloxydecyl dihydrogen phosphate (MDP), ethanol	1400007581
Porcelain Conditioner	Reliance Orthodontic Products; Itasca, IL, USA	Acetone, ACS Grade (30-50 %) 3-(trimethoxysilyl) propyl-2-methyl-2-propenoic acid (1-5 %)	145505
Transbond™ XT Light Cure Adhesive Primer	3M Unitek; Monrovia, CA, USA	Bisphenol-adiglycidyl ether dimethacrylate (bis-GMA), triethylene glycol dimethacrylate, 4-(dimethylamino) benzeneethanol, dl-camphorquinone, hydroquinone	N492996
Transbond™ XT Light Cure Adhesive Paste	3M Unitek; Monrovia, CA, USA	Silane-treated quarts, silane-treated silica, bisphenol-a diglycidyl ether dimethacrylate (bis-GMA), bisphenol A BIS (2-hydroxyethyl ether) dimethacrylate, diphenyliodonium hexafluorophosphate	N499911
Single tube (standard, 022 slot)	Tomy incorporated; Tokyo, Japan	Stainless steel	B5Y4

Table 2. Surface conditioning and tube bonding sequences in this study

Groups	**Substrate**	**Step 1**	**Step 2**	**Step 3**
Control group	Porcelain veneering + glaze	50-μm Al_2O_3	HF	Porcelain primer
Experimental group 1	Zirconia + glaze	50-μm Al_2O_3	HF	Porcelain primer
Experimental group 2	Zirconia + glaze	50-μm Al_2O_3		Zirconia primer
Experimental group 3	Zirconia + glaze	Cojet sand™		Porcelain primer
Experimental group 4	Zirconia + glaze	Rocatec plus™		Porcelain primer

4. Shear bond strength test and failure type analysis

The tube base and bar installed in the universal testing machine (Model 3366, Instron® Co., USA) were placed on the same surface setting cross-head speed at 1 mm/min. The resistance power became greater as the tube was pushed more, and the greatest level of resistance power was measured at the moment immediately before the tube falls. This was divided by the area of the tube base, 14.00 mm^2, and that result was the shear bond strength (MPa) (Figure 1).

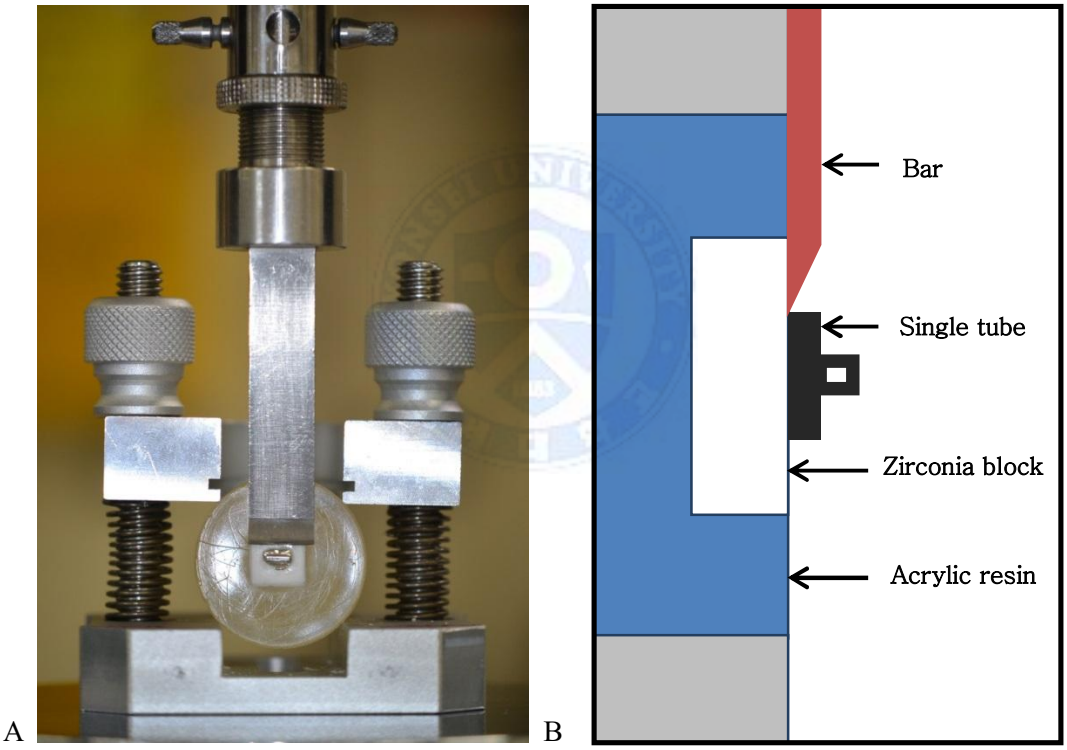

Figure 1. Shear bond strength testing. A, Measurement of shear bond strength using universal testing machine; B, Schematic illustration for testing shear bond strength.

5. Surface imaging

For the analysis of the surface of each group, surface imaging was conducted by using a scanning electron microscope (SEM) with a magnification of 1000X (S-3000N, Hitachi, Tokyo, Japan) before surface conditioning. In addition, the roughness of the surface was observed and the surface roughness Sa values were measured at three points of one block selected randomly from each group by using a 3-dimentional optical profiler (NewView 6300, Zygo Corp., Middlefield, CT, USA) with a magnification of 10X.

After the shear bond strengths were measured, failure types were classified by observing the debonding surface of each block by using a stereoscopic microscope with a magnification of 30X. The failure types were classified into three types as follows.

a) Adhesive failure 1: Adhesive failure between block surface and resin cement wherein all of the resin cements are left on the tube base

b) Adhesive failure 2: Adhesive failure between tube and resin cement wherein all of the resin cements are left on the block surface

c) Complex adhesive and cohesive failure: Mixed adhesive failure a and b or cohesive failure

6. Statistical analysis

After calculating the average and standard deviation of the shear bond strength of groups that each comprised ten specimens, the significance between the groups was analyzed in One-Way ANOVA. At a 0.05 significance level, Tukey's HSD (Honestly Significant Difference) method was used for post-hoc examination.

Correlation between each group and failure type was evaluated through Fisher's exact test at a 0.001 significance level.

After calculating the average and standard deviation of Sa values of groups that each comprised three specimens, the significance between the groups was analyzed in Kruskal-

Wallis test. At a 0.05 significance level, the least significant difference test using ranks was used for post-hoc examination.

III. Results

1. Effect of surface conditioning and primer on shear bond strength

As for the summary of average and standard deviation of shear bond strength in each group, results are shown in Table 3 and Figure 2. Shear bond strength was shown to be significantly lower in the experimental group 2 compared to all other groups ($p < .05$), and there was no significant difference among the other groups ($p > .05$).

Table 3. Shear bond strength results (MPa) for the groups

Group	N	Mean	S.D.
Control group	10	14.45[a]	1.77
Experimental group 1	10	15.88[a]	2.37
Experimental group 2	10	10.59[b]	1.97
Experimental group 3	10	13.71[a]	1.57
Experimental group 4	10	14.46[a]	3.12

SD, standard deviation
The same letters in the same column indicate no significant difference ($p > .05$).

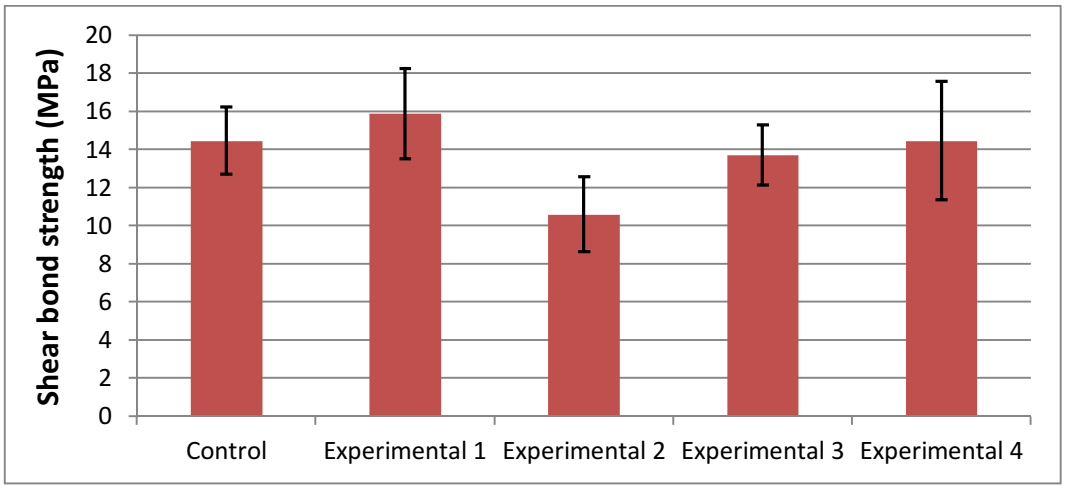

Figure 2. Mean shear bond strength results (MPa) and standard deviations for five different surface conditioning and tube bonding procedures.

2. Surface analysis

In order to identify the surface characteristics of the five groups, SEM was recorded on the surface after surface conditioning, and the relevant image is shown in Figure 3. Sandblasting and hydrofluoric acid were applied on the control group and experimental group 1 respectively, and in these two groups similar rough surfaces and cracks were observed. On the other hand, in the case of experimental group 2 that was only sandblasted, and in the case of experimental groups 3 and 4 where Cojet sand™ and Rocatec plus™, which are silica coating particles, were used, these groups had particles attached on the surface. Experimental group 4 was observed as having bigger sized particles and an irregularly rough surface compared to the experimental groups 2 and 3.

As for the second method for identifying surface characteristics among the five groups, the surface image was recorded using a 3D optical profiler with a magnification of 10X to evaluate surface roughness (Figure 4). The results of randomly measuring and comparing the surface roughness Sa values at three points by selecting one block randomly from each group are recorded in Table 4. The average surface roughness Sa value in the experimental group 3 was significantly lower than the values for experimental groups 1, 2, 4 ($p < .05$). The Sa value in the control group was significantly lower than the values recorded for experimental groups 2, 4 ($p < .05$). However, no significant difference was recorded among the other groups ($p > .05$).

Table 4. Sa values for groups

Group	N	Mean	S.D.
Control group	3	2.55^{ab}	0.10
Experimental group 1	3	2.78^{ac}	0.07
Experimental group 2	3	2.90^{c}	0.13
Experimental group 3	3	2.18^{b}	0.11
Experimental group 4	3	2.90^{c}	0.24

SD, standard deviation
The same letters in the same column indicate no significant difference ($p > .05$).

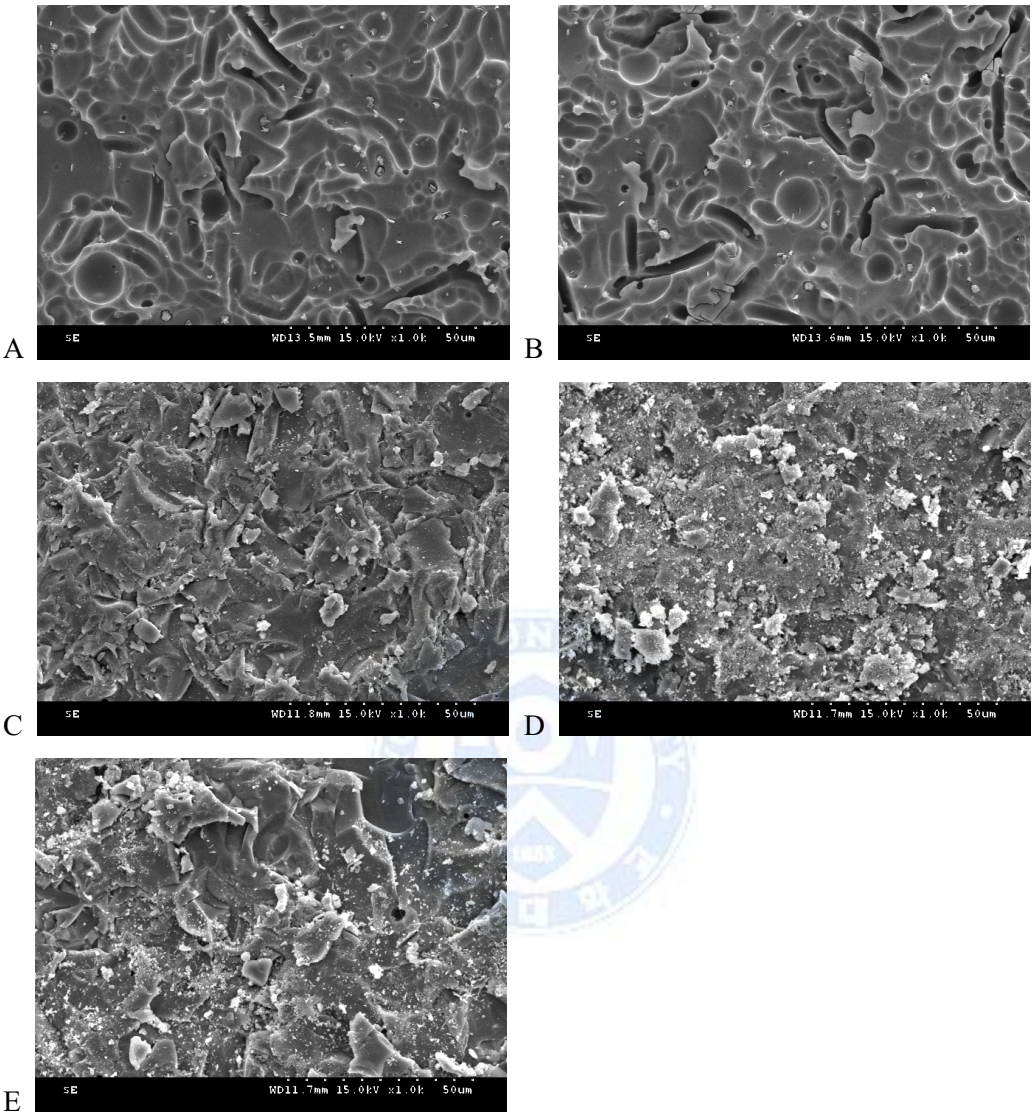

Figure 3. Scanning electron microscope (SEM) images of the conditioned glazed layer on blocks (1000X). (A) Control group; (B) Experimental group 1; (C) Experimental group 2; (D) Experimental group 3; (E) Experimental group 4

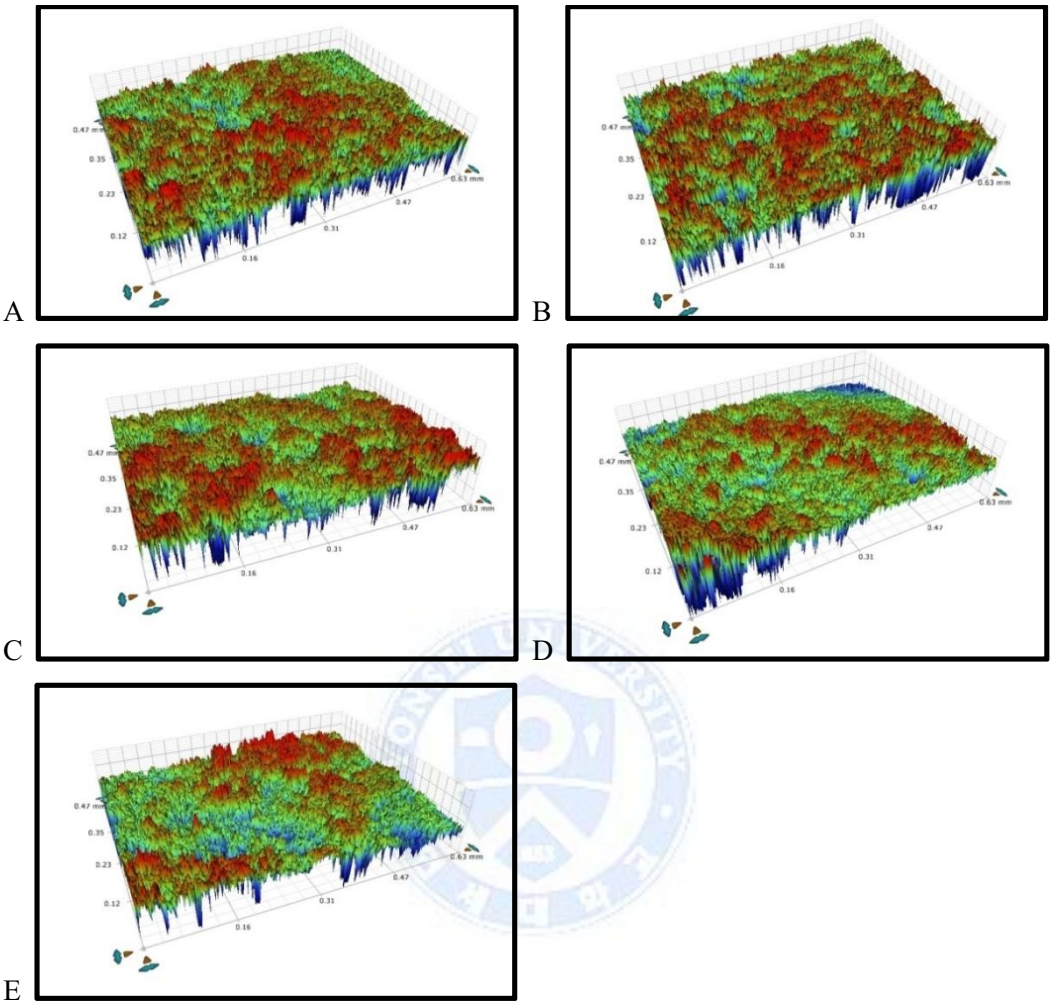

Figure 4. 3D optical profiler images of the conditioned glazed layer on blocks (10X). (A) Control group; (B) Experimental group 1; (C) Experimental group 2; (D) Experimental group 3; (E) Experimental group 4

3. Failure type analysis

Adhesive failure patterns classified by the stereoscopic microscope are shown in Figure 5, and the results are shown in Table 5. In the experimental group 2, adhesive failure was shown between the zirconia block surface and resin cement in all the ten specimens. Failure between the resin cement and tube base was shown to be the most common in all groups except for the experimental group 2. There was a significant difference in the level of $p < 0.001$ in the Fisher's exact test conducted on five groups. However, there was no significant difference in the level of $p = 0.531$ in the Fisher's exact test in four groups except for the experimental group 2. In other words, it was confirmed that only the experimental group 2 had a significantly different failure compared to other groups. ($p < 0.001$)

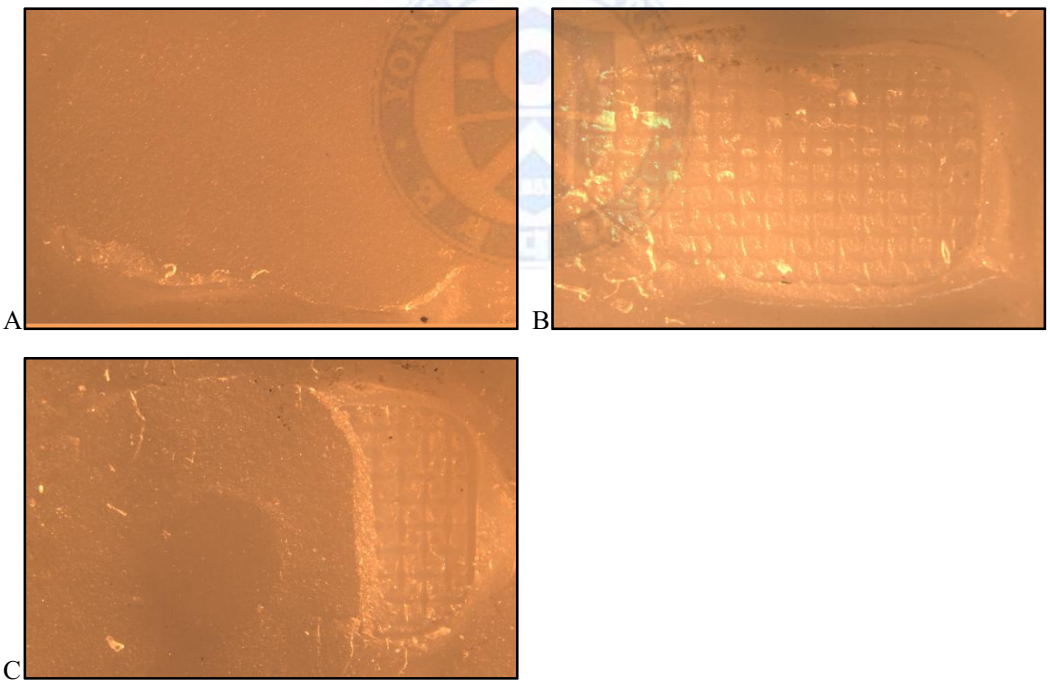

Figure 5. Stereoscopic micrographs of the debonded surface of blocks (16X). (A) Adhesive failure between block surface and resin; (B) Adhesive failure between resin and tube base; (C) Complex adhesive and cohesive failure

Table 5. Distribution of failure types for groups

	Control Group (N=10)	Experimental group 1 (N=10)	Experimental group 2 (N=10)	Experimental group 3 (N=10)	Experimental group 4 (N=10)
Adhesive failure between block surface and resin (A)	0	1	10	0	0
Adhesive failure between resin and tube base (B)	9	6	0	9	8
Complex adhesive (A+B) and cohesive failure	1	3	0	1	2

IV. Discussion

The objective of this study was to measure and compare shear bond strength by bonding tubes on surface-glazed zirconia using various methods. The most effective resin bonding method applied when attaching the tube on the surface-glazed zirconia was shown in the experimental group 1. However, there was no significant difference with the control group, and experimental groups 3 and 4. The experimental group 2 had a significantly lower shear bond strength compared to all other groups. In other words, it was confirmed that the experimental groups 1, 3, and 4 had a similar level of shear bond strength when compared to the method for bonding tube on surface-glazed porcelain in the control group. Results obtained in this study ranged from 10.59 to 15.88 MPa and hence represent a level of shear bond strength that exceeds the appropriate shear bond strength that ranges from 5.9 to 7.8 MPa which is required when providing orthodontic treatment as suggested by Reynolds (Reynolds, 1975). However, the results were similar or lower when compared to the bond strength between the natural teeth and brackets: 10.1-19.0 MPa (Scougall Vilchis et al., 2009), 10.8-16.3 MPa (Toodehzaeim et al., 2012) and 15.0-20.6 MPa (Tang et al., 2000).

According to other studies on the shear bond strength of tubes, shear bond strength between natural molar teeth and tubes turned out to be 3.0-7.0 MPa (Millett et al., 2001), 3.7-4.4 MPa (Johnston and McSherry, 1999), 1.7-3.9 MPa (Purmal and Sukumaran, 2010). In addition, the shear bond strength between porcelain and tubes was 3.5-3.6 MPa (Purmal et al., 2013). Therefore, the results of this study were several times higher than other studies using tubes.

The surface characteristics of each group according to different surface conditioning were observed in the SEM images. In addition, according to the results of measuring and comparing the surface roughness Sa values at three points by selecting one block randomly from each group using a 3D optical profiler with a magnification of 10X, the average surface roughness Sa value in experimental group 3 was significantly lower than the values for experimental groups 1, 2, 4. The Sa value in the control group was

significantly lower than the values for experimental groups 2, 4. However, there was no significant difference among the other groups. Even though the Sa value in the control group and experimental group 3 was lower than the other groups, shear bond strength was shown to be lower in experimental group 2. This means that shear bond strength depends more on primer characteristics; chemical cohesion instead of the surface roughness; mechanical coherence. However, the control group and experimental group 1 had the same surface conditioning method but produced significantly different results among the other groups. This indicates the possibility that the treated surface was not regular. Additionally, because of the small sample sizes, it would be desirable to compare more measured values so as to determine whether there is a significant difference in the actual clinical environment.

In experimental group 2 where 50-μm Al_2O_3 particles and zirconia primer were used, there was a significantly lower shear bond strength compared to all other groups. As shown in Table 1, the zirconia primer component used in the experiment includes Biphenyl dimethacrylate (BPDM), Hydroxyethyl methacrylate (HEMA), 10-metacryloyloxydecyl dihydrogen phosphate (MDP), and ethanol, etc. Organic phosphorous monomer such as MDP monomer most likely has an organofuctional portion where copolymerization is available with a monomer of resin cement such as the methacrylate group (Maeda et al., 2014; Matinlinna et al., 2006). These phosphate monomers also have phosphoric acid groups combined with zirconia (Magne et al., 2010). MDP components were designed to be combined with metal oxides. They are known to improve resin bond strength not only with metal oxides but also with zirconia and alumina metal oxide ceramics without applying silica coating or silane (Chen et al., 2014; Magne et al., 2010; Papia et al., 2014). Using primer or resin cement containing the MDP monomer after the sandblasting and/or silica coating treatment is currently known to be the most advantageous in bonding the zirconia (Cavalcanti et al., 2009; Inokoshi et al., 2014b; Kern and Wegner, 1998; Papia et al., 2014; Tanis et al., 2015; Wolfart et al., 2007). Nonetheless, the reason why the lowest level of shear bond strength was shown in experimental group 2 where Z-prime plus containing MDP was used was that the zirconia used in this experiment was “glazed

zirconia", while that which was used for zirconia using MDP in previous studies was only "naked zirconia."

Glazing in the ceramic is not only advantageous in terms of aesthetics but also in terms of improving flexural strength and decreasing surface flaws (Giordano et al., 1995). According to Douglas et al. (Douglas et al., 1981), glazing thickness in the zirconia was from 7.7 to 21.5 μm so that the maximum value was 31 μm when considering standard deviation. According to Cenk Cura et al. (Cura et al., 2012), glazing thickness was from 6.9 to 8.9 μm and so was not interruptive in crown fitting. However, even if multiple specimens were processed by one well-trained technician, it is still difficult to realize consistent thickness. Even though zirconia block was used in this experiment, glazing components were still left on the surface instead of zirconia after sandblasting and hydrofluoric acid processing. Major components of GC Initial™ IQ Lustre Pastes NF used in glazing are porcelain powder, so the surface that reacts with primers is not zirconia but contains porcelain components. This is the reason why a lower level of shear bond strength was shown in the experimental group 2 even after using zirconia primer containing MDP compared to other groups that utilized porcelain primer. In other words, a silanization procedure is required to obtain a higher level of bond strength through chemical combination on the surface on which glazing remains.

For silanization in porcelain, using porcelain primer after sandblasting and hydrofluoric acid processing is commonly used. However, since acid processing and the silanization effect is insufficient on the zirconia surface (Inokoshi et al., 2014a; Kern and Wegner, 1998; Thompson et al., 2011), a method employing tribochemical silica coating has been newly introduced. This method makes it possible to proceed with silanization by coating silica on the zirconia surface. It has been revealed in many papers that this method has an effect on zirconia surface (Bavbek et al., 2014; Chen et al., 2014; Thompson et al., 2011).

In this study, whether tribochemical silica coating with residual porcelain components on surface-glazed zirconia was effective or not was analyzed when compared with existing silanization of surface-glazed porcelain. Materials used in the experiment were Cojet sand™ that is used in clinical practice in dental clinics and Rocatec plus™ that is

used by technicians. The results of the experiments revealed that there was no significant difference among the control group, the group using Cojet sand™, and the group using Rocatec plus™. In addition, all three groups demonstrated a significantly higher level of shear bond strength than the experimental group 2 that used zirconia primer. In another study (Cheung et al., 2014), the group that used a silane coupling agent after processing the zirconia surface with Rocatec plus™ revealed an insignificantly similar shear bond strength compared to the group that used hydrofluoric acid and a silane coupling agent after glazing the zirconia surface in manner similar with this study. If at least a part of the zirconia that was exposed as a glazed layer was removed due to sandblasting in this experiment, tribochemical silica coating would have been more efficiently applied. In addition, since hydrofluoric acid was not necessary when using this product, it is a simpler and safer method in dental clinic practice than using sandblasting, hydrofluoric acid, or porcelain primer.

Studies seeking to obtain a high mechanical bond strength for zirconia are currently continuing. For silica-based ceramics, using hydrofluoric acid is the most widely employed method (Blatz et al., 2003). This makes the surface rough and clean, improving wettability and increasing surface area, so mechanical interlocking is feasible (Thompson et al., 2011). However, for nonsilica-based ceramics such as zirconia, it is difficult to make the surface rough to increase mechanical bond strength with hydrofluoric acid (Blatz et al., 2003; Cura et al., 2012; Kern and Wegner, 1998). Unlike hydrofluoric acid, sandblasting is effective in increasing mechanical bond strength in zirconia (Cavalcanti et al., 2009; Kern et al., 2009; Kosmac et al., 1999). In addition, it is known that flexural strength is improved as air abrasion creates a compressive layer on the surface since it causes tetragonal-to-monoclinic phase transformation (Cavalcanti et al., 2009; Curtis et al., 2006; Kosmac et al., 1999; Papanagiotou et al., 2006). Since surface flaws created by air abrasion do not exceed the thickness of compressive layers, it offsets the reduction in strength caused by flaws and finally improves flexural strength by air abrasion (Kosmac et al., 1999; Papanagiotou et al., 2006). Unlike air abrasion, the diamond bur of coarse grit (150μm) lowers strength and reliability (Kosmac et al., 1999).

According to adhesive failure types, there was an adhesive failure between the zirconia block and the resin only in the experimental group 2 and adhesive failure between the resin and the tube base was shown in most other groups. Since the bond strength between the tube base and the resin cement was the same, for experimental group 2, in which a failure was revealed between the zirconia block surface and resin, turned out to have a lower level of shear bond strength. In addition, there was no group that cohesive failure due to strong shear bond strength between block surface and resin cement was mostly shown. In other words, bond strength between specimens of each group and resin cement did not exceed the cohesive strength of the resin cement.

Thermocycling is an aging procedure that realizes the oral environment and hence is useful for providing stress on adhesive areas (Komine et al., 2009; Palmer et al., 1992). There are no specific criteria regarding temperature, time, and frequency, but there is a study that shows that 10000 cycles indicate a year (Gale and Darvell, 1999). However, there was no difference between when thermocycling was conducted 2000 times in 30 second intervals between 5 ℃ and 55 ℃ and when it was not conducted at all in the pilot test that was performed prior to this research. Therefore, thermocycling was omitted. According to a study by Cheung et al. (Cheung et al., 2014), there was no significant difference in terms of the shear bond strength before and after thermocycling in the glazed group. According to a study by Komine et al. (Komine et al., 2009), four types of primers out of a total of five primers revealed no change in shear bond strength before and after thermocycling. According to a study by Cure et al. (Cura et al., 2012), there was no change in shear bond strength at all before and after thermocycling. Also, in a study by Wegner and Kern (Wegner and Kern, 2000), there was no difference before and after thermocycling in the case of resin composite containing MDP.

In spite of the aforementioned results, since shear bond strength was shown to slowly decrease in water storage during 24 months when a stationary orthodontic device was used on average in orthodontic treatment (Oesterle and Shellhart, 2008), a study is needed that investigates shear bond strength when aging over 24 months is considered. Furthermore, there is a need to consider variables including photopolymerization time

and cross head speed in addition to aging (Finnema et al., 2010), and additional studies are needed to consider conditions in the mouth such as masticatory force.

V. Conclusions

Results of the shear bond strength experiment after proceeding with different surface conditioning techniques and using different types of primer in order to identify effective tube bonding methods on the surface-glazed zirconia are as follows.

1. Shear bond strength of the experimental group 2 was 10.59 MPa, which was significantly lower than all other groups ($p < .05$).
2. Two groups, the control group and experimental group 1 were clearly observed in the SEM images to have similar rough surfaces and cracks. However, experimental groups 2, 3 and 4 were observed as having particles attached. Experimental group 4 was observed as having bigger sized particles and more irregular rough surfaces compared to experimental groups 2 and 3. In addition, according to the results of analysis using a 3D optical profiler, the average surface roughness Sa value in experimental group 3 was significantly lower than the values for experimental groups 1, 2, 4 ($p < .05$). The Sa value in the control group was significantly lower than the values for experimental groups 2, 4 ($p < .05$). However, no significant difference was recorded among the other groups ($p > .05$).
3. As for the failure type of debonding surface, only adhesive failure was manifest between zirconia block surface and resin cement interface in experimental group 2. In the rest of the groups, adhesive failure between resin cement and tube base interface was mostly manifest. Therefore, this reveals a significant difference between the experimental group 2 and the rest of the groups ($p <.001$).

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국문요약

글레이징 처리된 지르코니아 보철물에서 표면 처리 방법에 따른 교정용 튜브의 전단 결합 강도 비교

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(지도 교수 : 유 형 석)

현재 지르코니아 잡착에 관한 연구는 주로 지르코니아 크라운의 내면 즉 순수 지르코니아 표면의 접착에 초점이 맞추어지고 있다. 교정영역에서는 글레이징된 크라운의 표면에서의 튜브 접착이 필요하므로, 이번 연구에서는 “naked zirconia”가 아닌, “glazed zirconia”에 다양한 방법으로 튜브를 접착하여 전단 결합 강도를 측정 비교 하였다.

글레이징된 포세린 표면에 일반적인 방법으로 튜브를 접착한 것을 대조군으로 설정하였다. 실험군은 지르코니아 블럭의 한쪽 표면을 글레이징하고, 무작위로 4개의 그룹으로 나눈 뒤, 각각 다른 표면 처리 방법, 다른 프라이머를 이용하여 튜브를 접착하였다.

대조군: 50-μm 알루미나 + 불산 + 포세린 프라이머
실험군 1 : 50-μm 알루미나 + 불산 + 포세린 프라이머
실험군 2 : 50-μm 알루미나 + 지르코니아 프라이머
실험군 3 : 30-μm 실리카 코팅 알루미나 + 포세린 프라이머
실험군 4 : 110-μm 실리카 코팅 알루미나 + 포세린 프라이머

만능 시험기을 이용하여 각 군의 전단 결합 강도를 측정하였다. 표면 처리 후 프라이머 도포 전 표면을 SEM, 3D optical profiler를 이용하여 표면 특성을 관찰하였으며 각 군의 접착 실패 양상을 분류하였다. 이번 연구의 결과는 다음과 같다.

1. 실험군 2 의 전단 결합 강도는 10.59 MPa 로, 다른 모든 그룹에 비해 유의성 있게 낮았다 ($p < .05$).
2. SEM 이미지에서 대조군, 실험군 1 의 두 그룹은 거칠어진 표면과 크랙이 유사한 양상으로 뚜렷하게 관찰되는 반면 실험군 2, 3, 4 의 표면은 파티클이 부착되어 있는 양상을 관찰 할 수 있었다. 실험군 2, 3 에 비해 실험군 4 는 파티클 사이즈가 더 크고 불규칙하게 거칠어진 표면이 관찰되었다. 또한 3D optical profiler 를 이용하여 분석한 결과에서는 실험군 3 의 평균 표면 거칠기 Sa 값이 실험군 1, 2, 4 에 비해 유의성 있게 낮게 나타났으며 대조군의 Sa 값은 실험군 2, 4 에 비해 유의성 있게 낮게 나타났다 ($p < .05$). 그 외 다른 그룹간의 유의차는 없었다($p > .05$).
3. 튜브가 탈락한 표면의 접착 실패 양상을 살펴보면, 실험군 2 에서는 지르코니아 블록 표면과 레진 시멘트 계면에서의 접착 파괴만이 나타났다. 나머지 군에서는 레진 시멘트와 튜브 베이스 계면에서의 접착 파괴가 주로 나타나 실험군 2 와 나머지 그룹간에는 유의성 있는 차이를 보였다 ($p < .001$).

이번 연구 결과에 따르면, 글레이징된 지르코니아 표면에 튜브를 접착 할 때, 실리카 코팅 파티클로 샌드블라스팅 후에 포세린 프라이머를 사용하는 방법이 불산의 사용 없이도 기존의 방법들과 유사하게 높은 전단 결합 강도를 나타낼 수 있어 임상적으로 안전하며 간단하면서도 효과적인 방법이다.

핵심 되는 말: 지르코니아, 글레이징, 전단 결합 강도, 교정용 튜브, 프라이머, 표면 처리, 접착 실패