



Influence of nano-structured alumina coating on shear bond strength between Y-TZP ceramic and various dual-cured resin cements

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PURPOSE. The purpose of this study was to evaluate the effect of nano-structured alumina surface coating on shear bond strength between Y-TZP ceramic and various dual-cured resin cements. **MATERIALS AND METHODS.** A total of 90 disk-shaped zirconia specimens (HASS CO., Gangneung, Korea) were divided into three groups by surface treatment method: (1) airborne particle abrasion, (2) tribochemicalsilica coating, and (3) nano-structured alumina coating. Each group was categorized into three subgroups of ten specimens and bonded with three different types of dual-cured resin cements. After thermocycling, shear bond strength was measured and failure modes were observed through FE-SEM. Two-way ANOVA and the Tukey's HSD test were performed to determine the effects of surface treatment method and type of cement on bond strength ($P<.05$). To confirm the correlation of surface treatment and failure mode, the Chi-square test was used. **RESULTS.** Groups treated with the nano-structured alumina coating showed significantly higher shear bond strength compared to other groups treated with airborne particle abrasion or tribochemical silica coating. Clearfil SA Luting showed a significantly higher shear bond strength compared to RelyX ARC and RelyX Unicem. The cohesive failure mode was observed to be dominant in the groups treated with nano-structured alumina coating, while the adhesive failure mode was prevalent in the groups treated with either airborne particle abrasion or tribochemical silica coating. **CONCLUSION.** Nano-structured alumina coating is an effective zirconia surface treatment method for enhancing the bond strength between Y-TZP ceramic and various dual-cured resin cements. [*J Adv Prosthodont 2017;9:130-7*]

KEYWORDS: Airborne particles abrasion; Shear bond strength; Zirconia; Alumina coating; Resin cement

INTRODUCTION

Zirconia has higher flexural strength than the existing ceramic and is widely used in fixed partial dentures.^{1,2} It is

recommended that prepared tooth with compromised taper be bonded with resin cement to improve the chances of crown retention.³ However, as zirconia does not contain silica and has resistance against acid etching, its bond strength with resin cements can be reduced.^{4,5} As a stable bond strength is critical in long-term prognosis of prosthesis, various mechanical and chemical zirconia surface treatment methods have been introduced to improve bond strength with resin cement. Airborne particle can be applied to enhance the mechanical retention of resin cement by increasing zirconia surface roughness. However, as zirconia has a high surface hardness, only minimal undercuts were observed indicating a failure to significantly increase the surface roughness.⁶ Also, airborne particle abrasion can form microcracks on the ceramic surface, which reduce the fracture resistance of zirconia.^{7,8} Tribochemical silica coating is widely used in order to obtain effective mechanical retention and chemical bonding.⁹ However, while the initial bond

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strength of resin cement to zirconia was high, it decreased after long-term storage or thermocycling in several surface treatments.¹⁰

Various resin cements contain functional monomers to improve chemical bonding with ceramics, such as methacrylated phosphoric ester, 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer, etc.¹¹⁻¹³ Özcan *et al.*¹⁴ reported that bond strength between zirconia and resin cement containing MDP monomer was high in dry conditions but decreased after thermocycling.

In this study, nano-structured alumina coating on zirconia surfaces was used to improve the bond strength with resin cement. This method can increase the bond surface area and improve the micro-mechanical retention through mechanical interlocking of the resin cement. The purpose of this study was to evaluate whether nano-structured alumina coating on zirconia surfaces can enhance the bond strength between zirconia and various dual-cured resin cements, compared to the existing surface treatment methods.

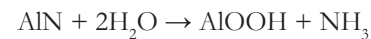
MATERIALS AND METHODS

Tetragonal zirconia polycrystal stabilized with 3 mol% yttria (Y-TZP) blocks (HASS Co., Gangneung, Korea) were sintered at 1,550°C for two hours. A total of 90 disk-shaped specimens were fabricated with a diameter of 15 mm and a thickness of 3 mm. The specimens were annealed in the air at a temperature of 1,200°C to release the surface stress occurring during tetragonal-to-monoclinic phase transformation. The specimens were divided into three groups of 30 specimens according to the surface treatment methods as follows:

- (1) Airborne particle abrasion: The surfaces of specimens were abraded with 50 µm aluminum oxide particles from a distance of 10 mm in a vertical direction at a pressure of 3 bar for 10 seconds.
- (2) Tribochemical silica coating: The surfaces of specimens were abraded with 110 µm silica-modified aluminum oxide particles (Rocatec plus, 3M ESPE, Seefeld, Germany) from a distance of 10 mm in a vertical direction at a pressure of 4 bar for 10 seconds. The silane coupling agent was applied and dried according to the manufacturer's instructions. ESPE Sil (3M EPSE AG, Seefeld, Germany) for

RelyX ARC and RelyX Unicem, and Clearfil ceramic primer (Kuraray, Okayama, Japan) for Clearfil SA Luting were used.

- (3) Nano-structured alumina coating: Ultrasonic cleaning of specimens was performed for 2 minutes in alcohol, acetone, and distilled water, respectively. 7.5 g of aluminum nitrate (AlN) powder (Grade C, 1.2 µm median particle size, surface area of 6 m²/g, Oxygen contents of 2.5 wt%O₂; H. C. Strack, Berlin, Germany) was dispersed in 250 mL of deionized water at 75°C to make a soluble suspension diluted with 3 wt% AlN powder. The specimens were immersed in the AlN suspension for 15 minutes. When dispersed AlN powder is exposed to hot water, a nano-structured boehmite coating starts to form on the surface as following reaction.



The coated specimens were dried at 110°C for 2 hours and treated with heat at 900°C for 1 hour using an electric resistance furnace. The heating rate was 10°C/min. Boehmite that was formed by heat is thermally decomposed, which will form transitional alumina and subsequently undergo continuous polymorphic transformation.¹⁵

Each group was divided into three subgroups of ten specimens, each of which was bonded with three different dual-cured resin cements (Table 1). The resin cements and components are shown in Table 2.

Table 1. Specimens preparation and their group

Group	Surface treatment	Resin cement
SA	Airborne particle abrasion	RelyX ARC
SU		RelyX Unicem
SC		Clearfil SA Luting
RA	Tribochemical silica coating	RelyX ARC
RU		RelyX Unicem
RC		Clearfil SA Luting
CA	Nano-structured alumina coating	RelyX ARC
CU		RelyX Unicem
CC		Clearfil SA Luting

Table 2. Dual-cured resin cements used in this study

Material	Components	Manufacturer
RelyX ARC	Bis-GMA ^a , TEGDMA ^b , Zirconia/silica filler, Amine, Photo initiator, Benzoyl peroxide	3M EPSE, Seefeld, Germany
RelyX Unicem	Methacrylated phosphoric ester, Dimethacrylate, Inorganic fillers, Fumed silica, Chemical and photo initiators	3M EPSE, Seefeld, Germany
Clearfil SA Luting	Bis-GMA, TEGDMA, MDP ^c , Dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, di-Camphorquinone, Benzoyl peroxide, Initiator, Accelerators	Kuraray, Okayama, Japan

^aBis-GMA: Bisphenol-A-glycidylmethacrylate; ^bTEGDMA: Triethyleneglycl dimethacrylate; ^cMDP: 10-Methacryloxydecyl dihydrogenic phosphate

Teflon tube with 3 mm inner diameters and 3 mm heights were filled with a composite resin (Estelite Σ Quick, Tokuyama, Tokyo, Japan) and light-cured for 20 seconds from two opposite sides. After the zirconia specimens were fixed in an adhesion mold, composite resin cylinders were bonded with three types of resin cements according to the manufacturer's instructions. Equal quantities of the base and catalyst of resin cement were mixed for 10 seconds. In the RelyX ARC, a thin layer of Adper Single Bond 2 (3M EPSE AG, Seefeld, Germany) was applied on the zirconia specimens and air-dried for 5 seconds and photo-polymerized for 10 seconds before bonding. Immediately after bonding of the resin cylinders to the zirconia specimens under a static load of 10 N, photo-polymerization was performed in four directions, each for 40 seconds, for a total of 160 seconds. The bonded specimens (Fig. 1) were kept at room temperature for 10 minutes. After storing the specimens in distilled water at a temperature of 37°C for 24

hours, 6,000 cycles of thermocycling were performed by immersion in 2 water tanks with temperatures of 5°C and 55°C water bath, each for 30 seconds. The shear bond strengths (SBSs) of the bonded specimens were measured using a universal testing machine (Model 4201, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min. The failure mode of the specimens were observed with Field-Emission Scanning Electron Microscopy (FE-SEM) after the SBS test. The failure modes were classified as (a) adhesive failure between the zirconia and resin cement; (b) cohesive failure in the resin cement; and (c) mixed failure when a combination of cohesive and adhesive failures occurred.

Two-way ANOVA and Tukey HSD post-hoc tests were performed to assess interaction of the surface treatments and type of resin cements, as well as their effects on the shear bond strength. In addition, the Chi-square test was performed to determine a possible correlation between surface treatment methods and failure modes. All analyses were performed using SPSS Statistics program (SPSS version 18. 0, SPSS Inc., Chicago, IL, USA) and the α level was set at .05.

RESULTS

The results of the shear bond strength test are shown in Fig. 2. According to the two-way ANOVA statistical analysis, significant differences were observed in shear bond strength depending on the surface treatment method and type of cement (Table 3). The interaction between the two variables indicated a statistically significant level at $P < .05$. The nano-structured aluminum oxide coating groups showed significantly higher shear bond strengths than the other groups. Also, Clearfil SA Luting cement showed sig-

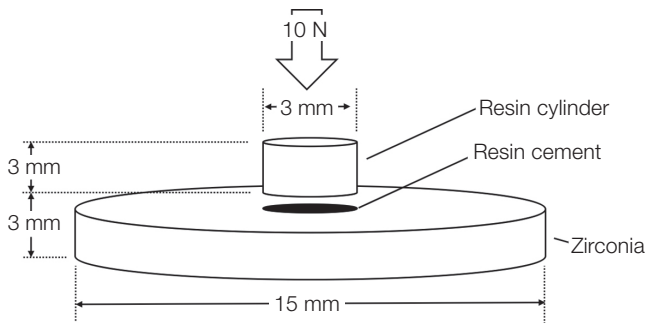


Fig. 1. Diagram of bonded specimen.

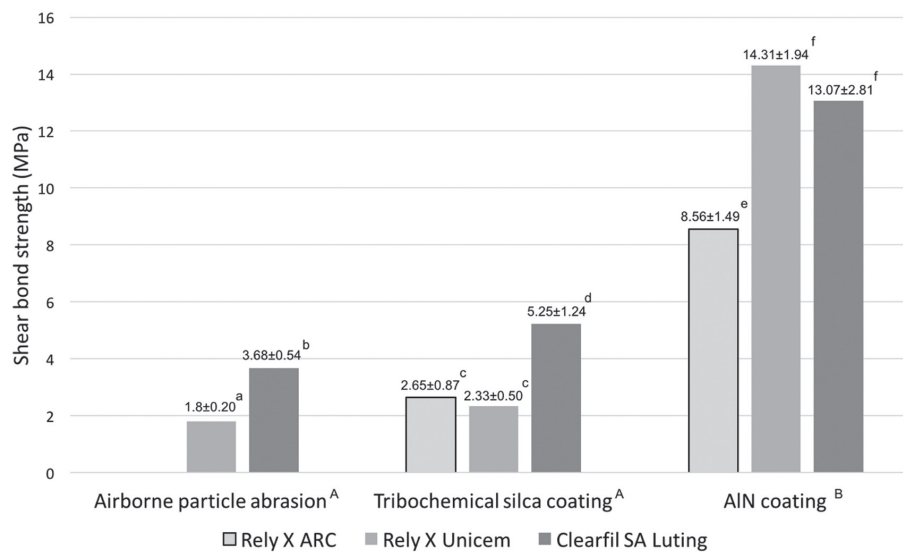


Fig. 2. Mean shear bond strength values for each experimental group ($P < .05$). Different superscript letters indicate statistically significant differences.

Table 3. Two-way ANOVA results for effect of different variables on shear bond strength

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Surface treatment	1582.594	2	791.297	377.988	<.001
Resin cement	151.628	2	75.814	36.215	<.001
Surface treatment * Resin cement	99.668	3	33.223	15.870	<.001
Error	150.728	72	2.093		
Total	1873.055	79			

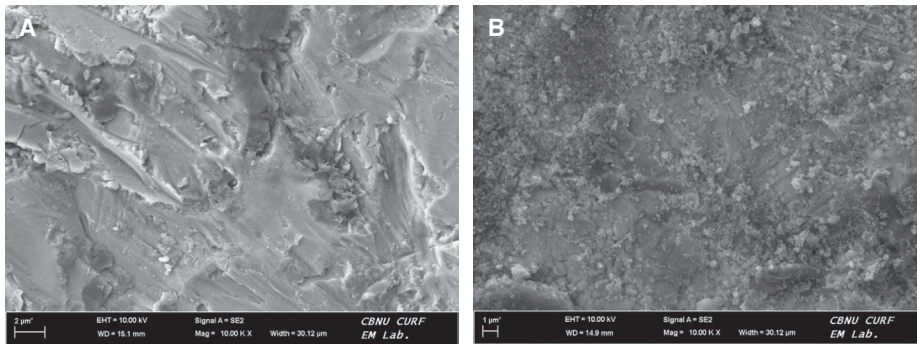


Fig. 3. (A) FE-SEM image showing the airborne particle abraded Y-TZP ceramic surface, (B) FE-SEM image showing the surface of Y-TZP ceramic after tribochemical silica coating.

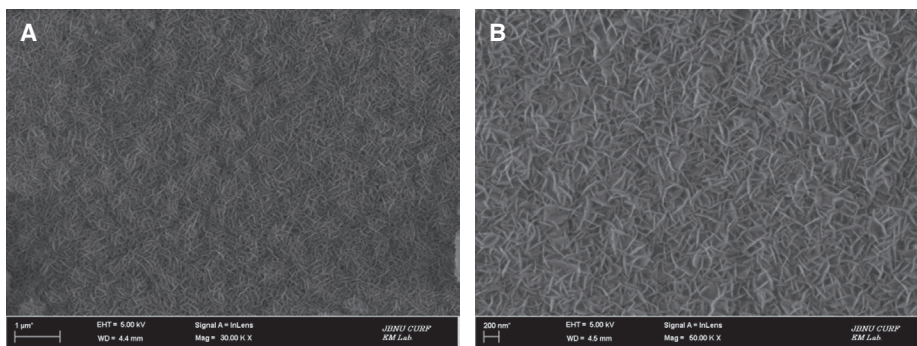


Fig. 4. (A) FE-SEM image showing the nano-structured alumina coating, (B) higher magnification FE-SEM image showing the nano-structured alumina coating.

nificantly higher bond strength than the other resin cements. Among the airborne particle abrasion groups, the shear bond strengths of RelyX ARC could not be measured due to spontaneous debonding, while Clearfil SA Luting demonstrated significantly highest shear bond strengths. Among the tribochemical silica coating groups, Clearfil SA Luting had significantly higher shear bond strengths than the other two types of resin cements. Among the nano-structured alumina coating groups, both RelyX Unicem and Clearfil SA Luting demonstrated significantly higher shear bond strengths than RelyX ARC. The groups of the nano-structured alumina coating showed significantly higher shear bond strengths than the groups of the other, regardless of type of cement.

Micro-undulated structures were observed on the surface of the specimens treated with airborne particle abrasion (Fig. 3A). Silica particles are distributed on the undulated surface, which was treated with tribochemical silica coating (Fig. 3B). Nano-structured alumina layer is evenly coated

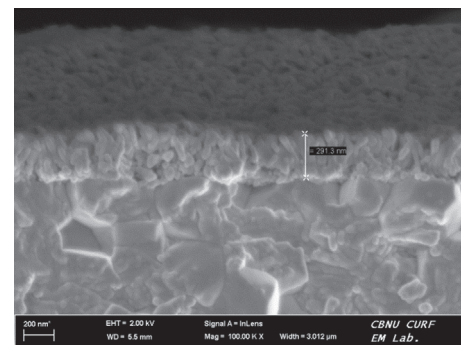


Fig. 5. FE-SEM image showing a section of nano-structured alumina coated Y-TZP ceramic.

on the surface of Y-TZP, which can increase the adhesion surface area and create micro-mechanical interlocking with the resin cement (Fig. 4). The even thickness of alumina coating layer was 290 nm (Fig. 5).

After measuring the shear bond strengths, the surface of all specimens were observed with FE-SEM (Fig. 6). The failure modes of each group are shown in Table 4. In the CA, CU, and CC groups, the aluminum oxide coating was covered with the resin cement and the cohesive failure was dominant (Fig. 7).

According to the Chi-square test result, there was a statistically significant difference in failure mode depending on surface treatment methods ($P < .05$). Among the airborne particle abrasion groups and tribochemical silica coating groups, the adhesive failure mode was dominant. In contrast, among the nano-structured alumina coating groups, cohesive failure was prevalent.

Table 4. Failure mode of experimental groups

Groups	Adhesive failure	Mixed failure	Cohesive failure
SA	10	0	0
SU	10	0	0
SC	5	5	0
RA	8	2	0
RU	8	2	0
RC	3	5	2
CA	0	2	8
CU	0	2	8
CC	0	0	10

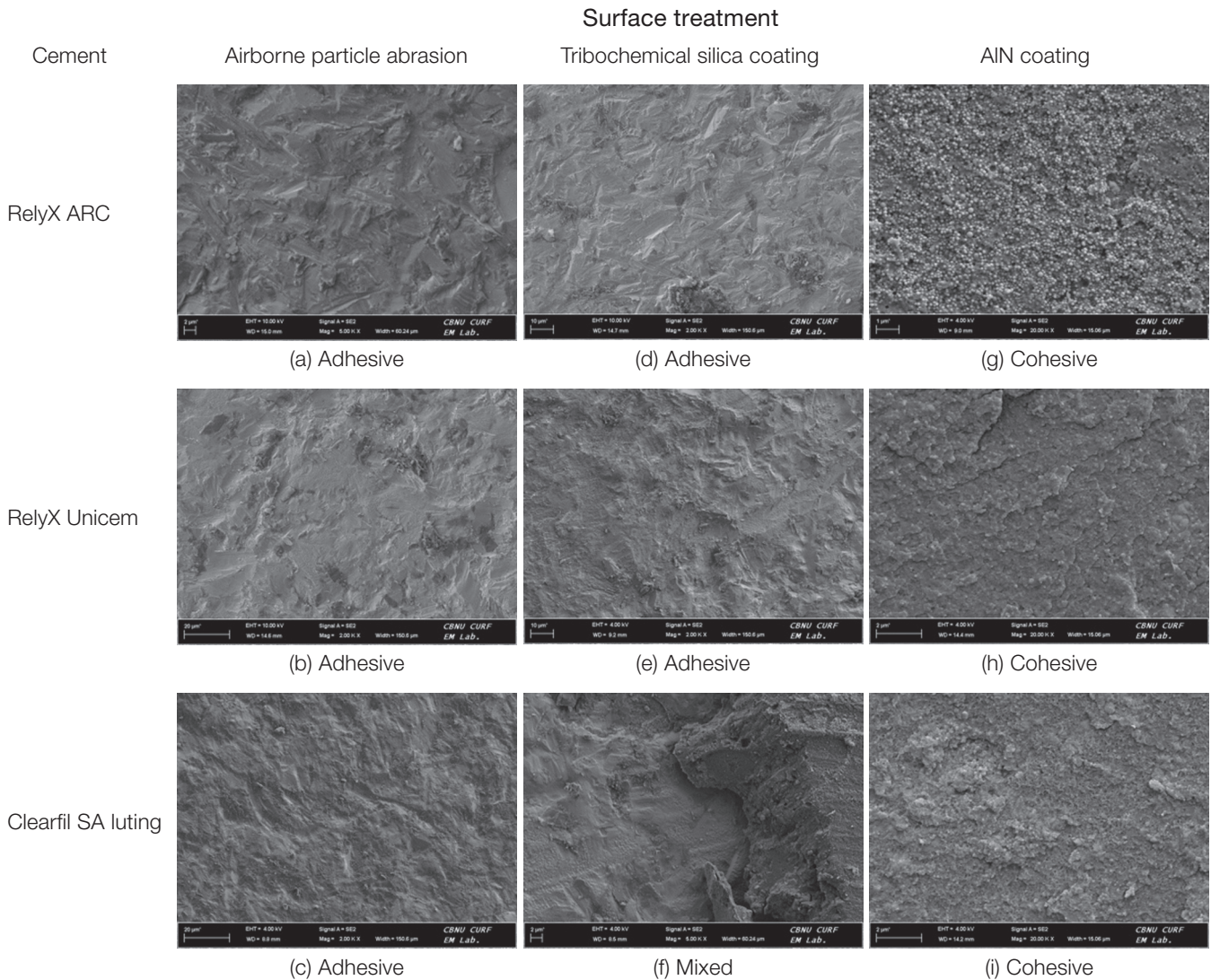


Fig. 6. FE-SEM images and dominant failure modes of specimens after measuring shear bond strength. Failure mode is described below the figures.

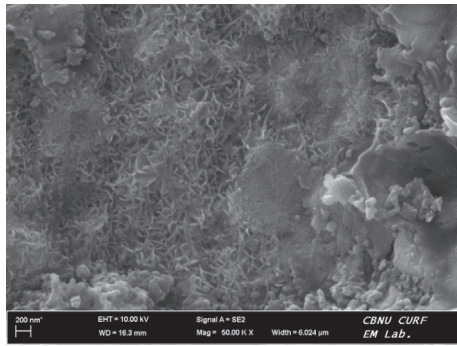


Fig. 7. Alumina coating covered by resin cement is shown more clearly in $\times 50,000$ magnified image.

DISCUSSION

Various mechanical or chemical methods have been used to enhance the bond strength between zirconia and resin cement. Several studies have reported that the bond strength could be reduced after thermocycling.^{16,17}

Although airborne particle abrasion can improve the roughness of surface, only a minimal undercut can be formed and abrasion does not lead to sufficient surface roughness because of high surface hardness. Hence, small increase of bond strength is obtained.⁶ Kern and Wegner¹⁸ also reported that the bond strength with conventional Bis-GMA resin cement after airborne particle abrasion did not remain stable for a long time. In this study, all specimens that were bonded using resin cement without functional monomer after airborne particle abrasion spontaneously debonded after thermocycling. Also, the groups that were bonded with other resin cements after airborne particle abrasion showed significantly lower bond strength than the other surface treatment groups. These results supported that as airborne particle abrasion created only a modest amount of undercut on the surface, it failed to achieve enough micromechanical bonding with the resin cement.

Many studies demonstrated that a tribochemical silica coating could achieve a higher bond strength than airborne particle abrasion for chemical bonding of a silica surface layer.¹⁹⁻²¹ The bond strength between the silica coated surface and resin cement was initially high but was reduced after long-term storage or thermocycling.¹⁰ In this study, the group bonded with RelyX ARC after airborne particle abrasion debonded spontaneously, while the bond strength of group coated tribochemical silica remained still after thermocycling. However, tribochemical silica coating groups showed significantly lower bond strength than the nano-structured alumina coating groups. This result supports that the siloxane bond created on the surface can increase the bond strength with resin cement in some degree, but it fails to achieve an effective improvement. A substantial amount of silica, which remains on zirconia surfaces after silica coat-

ing, is necessary for durable bonding. However, Matinlinna *et al.*²² reported that the amount of silica remaining after silica coating was not sufficient to obtain a silanization effect due to the high surface hardness of zirconia. It remains unclear whether the effect of tribochemical silica coating can endure on a long-term basis even in a clinical practice.

Nano-structured alumina coating groups showed remarkably higher bond strength with all types of resin cements. The surface treated with nano-structured alumina coating showed nano-scale undulated structures in FE-SEM images. Such structures can improve bond strength with resin cement by increasing the surface area and creating resin cement tags. Jevnikar *et al.*¹⁵ observed the infiltration of resin matrices into inter-lamellar spaces of alumina coating. The formation of such hybrid layers can help maintain durable bonding.

Recently, several resin cements containing functional monomers to increase bond strength were developed. The conventional Bis-GMA resin cements tend to show early fracture patterns due to low bond strength to zirconia.²³ RelyX Unicem resin cement, which consists of multifunctional phosphoric acid methacrylate and alkaline fillers, tends to show a relatively higher bond strength.^{21,24} The phosphate ester group of the MDP in Clearfil SA Luting cement is known to bond directly with oxidized metal surfaces or ceramics, including zirconia, and form a stable bond against hydrolysis. Hydrophilic phosphate groups of MDP monomers demineralize the tooth surface and bond with calcium ions or amino groups. Also, the phosphate groups directly react with hydroxyl groups on the zirconia surface. A number of studies have demonstrated that resin cements containing MDP monomers had higher bond strength compared to those containing different monomers.^{25,26} In this study, among the experimental groups with airborne particle abrasion or tribochemical silica coating, Clearfil SA Luting showed significantly higher shear bond strength than the other resin cements. However, among the experimental groups with nano-structured alumina coating, Clearfil SA Luting had significantly higher shear bond strength than RelyX ARC but did not show a statistically significant difference compared to RelyX Unicem. In the experimental groups treated with airborne particle abrasion or tribochemical silica coating, the shear bond strength could vary depending on the chemical bonding of resin cement monomers because the zirconia surface directly contacted the resin cement. However, in the experimental groups treated with nano-structured alumina coating, the importance of chemical bonding was less critical because the zirconia surface was not exposed to the resin cement by alumina coating and micromechanical bond strength was substantially increased.

When comparing failure modes depending on surface treatment methods, adhesive failure was common in the airborne particle abrasion groups and the tribochemical silica coating groups. In contrast, cohesive failure was prevalent in the nano-structured alumina coating groups. These results support that the nano-structured alumina coating has a

higher shear bond strength than other surface treatment.

Some studies suggested that if stress is occurred on a Y-TZP ceramic by airborne particle abrasion, the phase transformation, which creates a local compressive stress area, subsequently leads to an increase in fracture toughness.^{8,27} In contrast, other studies reported that if stress concentrates on a surface flaw, it can reduce the fracture strength.²⁸⁻³¹ However, the advantage of nano-structured alumina coating is that it can improve the bond strength with a resin cement without reducing the flexural strength of Y-TZP ceramics because it does not result in any surface flaws.

In clinical practice, the alumina coating layer thickness can affect the marginal discrepancy. In this study, the thickness of the alumina coating was approximately 290 nm, and the possibility of increasing the marginal gap was negligible. However, as this study applied nano-structured alumina coating to the disc-shaped zirconia specimens in the insufficient thermocycling cycle, further studies must be carried out to determine whether it is possible to form an even and uniform coating on the internal surface of a prosthesis for use in clinical practice.

CONCLUSION

Within the limitation of this study, nano-structured alumina coating of Y-TZP ceramics provides remarkably higher bond strength between zirconia and various dual-cured resin cements, compared to the other surface treatment methods. If a zirconia surface is treated with either airborne particle abrasion or tribochemical silica coating, application of resin cement containing MDP monomers is recommended. In addition, as a zirconia surface treated with nano-structured alumina coating can improve the bond strength between zirconia and resin cement, various resin cements can be used.

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