나노구조 알루미나 코팅 처리가 지르코니아 도재와 레진 시멘트 사이 전단 결합강도에 미치는 영향

김동운¹ · 이정진¹ · 김경아² · 서재민^{1*}

'전북대학교 치의학전문대학원 치과보철학교실 및 구강생체과학연구소, '을지대학교 의과대학 치과학교실

Influence of nano-structured alumina coating treatment on shear bond strength between zirconia ceramic and resin cement

Dong-Woon Kim¹, Jung-Jin Lee¹, Kyoung-A Kim², Jae-Min Seo^{1*}

¹Department of Prosthodontics, School of Dentistry and Institute of Oral Bio-Science, Chonbuk National University, Jeonju, Republic of Korea ²Department of Dentistry, School of Medicine, Eulji University, Daejeon, Republic of Korea

Purpose: The aim of this study was to investigate whether the application of nano-structured alumina coating to the surface of Y-TZP could enhance the bond strength with resin cement. **Materials and methods:** A total of 80 zirconia plates were prepared and divided into four groups. : 1) airborne particle abrasion treatment (A) : 2) Rocatec treatment after airborne particle abrasion (R) : 3) nano-structured alumina coating treatment after polishing (PC) and 4) nano-structured alumina coating after airborne particle abrasion (AC). Alumina coating was formed by the hydrolysis of aluminium nitride (AIN) powder and heat treatment at 900°C. Coating patterns were observed with FE-SEM. Resin block was bonded to treated zirconia ceramics using resin cement. The shear bond strengths were measured before and after thermocycling. **Results:** The FE-SEM images show a dense and uniform nano-structured alumina coating structure, which enhances shear bond strength by increasing micro mechanical interlocking to resin cement. PC and AC groups showed higher shear bond strengths than A and R groups before and after thermocycling. A and R groups displayed significant drops in shear bond strength after thermocycling. However, PC and AC groups did not show any meaningful decreases in shear bond strength after thermocycling. **Conclusion:** Treatment of Y-TZP ceramics with nano-structured alumina coating could significantly increase their shear bond strength. (*J Korean Acad Prosthodont 2016;54:354-63*)

Keywords: Zirconia; Shear bond strength; Alumina coating; Thermocycling; Resin cement

Introduction

Recently, as more and more patients show keen interests in the aesthetic aspects of dental restorations, dental ceramics are increasingly used not only for anterior restoration but also for posterior restoration, which requires strong strength.¹ Therefore, zirconia ceramics, which have excellent mechanical properties with high strength, have been increasingly applied in dental ceramics.^{2,3}

Zirconia restorations are fabricated by sintering yttria partially stabilized tetragonal zirconia ceramics (Y-TZP) using CAD-CAM tech-

* This work was supported by research funds from the Chonbuk National University in 2011.

This is an Open Access article distributed under the terms of the Creative Commons Attribution Non-Commercial License (http://creativecommons.org/licenses/by-nc/3.0) which permits unrestricted non-commercial use, distribution, and reproduction in any medium, provided the original work is properly cited.

^{*}Corresponding Author: Jae-Min Seo

Department of Prosthodontics, School of Dentistry and Institute of Oral Bio-Science, Chonbuk National University, 567 Baekjedaero, Dukjin-Gu, Jeonju 54896, Repulic of Korea +82 (0)63 250 2696: e-mail, jmseo@jbnu.ac.kr Article history: Received March 5, 2016 / Last Revision May 3, 2016 / Accepted July 5, 2016

nology.⁴⁵ However, the Y-TZP ceramic has its own drawbacks in that it has a low bonding strength with resin cement compared to other ceramics. The bonding strength relies on chemical bonding and mechanical interlocking due to micro-retentions created on the roughened surface.⁶ Hydrofluoric (HF) acid etching and sandblasting treatments are used to achieve micro-mechanical retentions by roughening ceramic surfaces.⁷ Existing silica-containing dental ceramic restorations can achieve a clinically stable bond strength through chemical bonding of the ceramics with resin cement by silane treatments.^{7,8} In contrast, as the Y-TZP ceramic does not contain silica and resists acid etching due to a high crystalline structure, it has limitations when using resin cements.^{9,10} Therefore, various surface treatments have been studied to improve the bond strength of Y-TZP ceramics with resin cement.^{11,12}

Airborne particle abrasion is mainly used to create mechanical retention by increasing surface roughness.^{13,14} The tribochemical silica coating method is a technique used to abrade the ceramic surface with silica-modified alumina particles, which enables the embedding of silica particles on the ceramic surface.^{15,16} The silica layer, which is created on ceramic surfaces during silica coating treatment, serves as a basis for the reaction of silane and precipitates chemical bonding.¹⁷ Therefore, the tribochemical silica coating method can achieve both airborne particle abrasion-induced mechanical retention and chemical bonding at the same time.¹⁸⁻²⁰

Many studies have been carried out on resin cements with special functional monomers that can be chemically bonded to Y-TZP ceramics. In recent years, many studies have been conducted on the application of adhesive primers including 10-methacryloyloxydecyl dihydrogenphosphate monomer (MDP) or phosphoric acid acrylate, which can increase the bond strength of Y-TZP ceramics to resin cement.^{12,21,22}

However, many studies maintain that the application of thermocycling after airborne particle abrasion or tribochemical silica coating can substantially reduce the bond strength.^{9,23} Akgungor *et al.*²⁴ reported that the application of MDP monomer did not improve the bond strength further.

Therefore, the aim of this study is to suggest a new non-invasive process to increase zirconia-resin bonding. This is the application of nano-structured alumina coating on the surface of Y-TZP, which can increase the bonded surface area and achieve micro-mechanical interlocking by creating nano-retentions on the surface.⁴

The objective of this study is to analyze the bonding potential of Y-TZP ceramic with a nano-structured alumina coating to resin cement and compare the bond strength to those of ceramic surfaces with various surface treatments.

Materials and methods

1. Specimens preparation

A total of 80 commercially available CAD-CAM zirconia blocks (Fulluster, 30 nm, HASS, Gangneung, Korea) made up of partially stabilized zirconia containing 3 mol% of yttria were used. After these blocks were sintered at 1550 °C in the air for 2 hours, they were abraded with a diamond wheel and then polished with a 1 μ m diamond paste (SPL-15, Okamoto Co., Yokohama, Japan) into disc-shaped specimens (15 nm in diameter and 3 mm thick). The specimens underwent annealing at a temperature of 1200 °C in the air for one hour after polishing to remove the mechanically affected layer caused by the tetragonal-to-monoclinic phase transformation due to high stress during abrasion. The specimens were divided into four groups of 20 depending on the surface treatment methods (Table 1).

Group A: Airborne particle abrasion

Group R: Tribochemical coating

- Group PC: Polishing + nano-structured alumina coating
- Group AC: Airborne particle abrasion + nano-structured alumina coating

All specimens were ultrasonically cleaned for 2 minutes in acetone, ethanol and deionized water, respectively.⁴

2. Surface treatment

All specimens were polished with silicone carbide paper (Abrasive Paper, Daesung, Seoul, Korea) of 100 grit to 1000 grit.

1) A&R group: To treat the surfaces of 40 specimens with airborne particle abrasion, 50 μ m aluminum oxide particles were sprayed at 3 bar pressure from a location 10 mm above the surface for 10 seconds. Among them, 110 μ m silica-modified aluminum oxide particles (Rocatec plus, 3M ESPE, Seefeld, Germany) were sprayed on 20 specimens at 4 bar pressure from a location 10 mm above the surface for 10 seconds. After that, the specimens were coated with silane

Group	Surface treatment		
А	50 μ m Al ₂ O ₃ airborne particle abrasion (3 bar, 10 s, 10 mm)		
(n = 20)			
R	50 μ m Al ₂ O ₃ airborne particle abrasion (3 bar, 10 s, 10 mm)		
(n = 20)	110 µm SiOx, Rocatec plus (4 bar, 10 s, 10 mm)		
	After applying silane coupling agent, 2 min. dry		
PC	Polishing : ascending stepwise approach		
(n = 20)	nano-structured alumina coating		
AC	50 μ m Al ₂ O ₃ airborne particle abrasion (3 bar, 10 s, 10 mm)		
(n = 20)	nano-structured alumina coating		

coupling agent (ESPE Sil, 3M ESPE, Seefeld, Germany) according to the instructions of the manufacturer and were dried.

2) PC&AC group: 20 specimens underwent additional polishing with silicone carbide paper of 1000 grit to 3000 grit for micro polishing. For airborne particle abrasion, the remaining 20 specimens were sprayed with 50 μ m aluminum oxide particles at 3 bar pressure from a location 10 mm above the surface for 10 seconds. After that, a total of 40 specimens (20 specimens treated with polishing and the other 20 specimens treated with airborne particle abrasion) were coated with nano-structured alumina.

The AlN powder used for nano-structured alumina coating was AIN Grade C (H.C. Starck, Berlin, Germany) with a median particle size of 1.2 μ m, a surface area of 6 m²/g, and an oxygen content of 2.5 wt%O₂. A diluted aqueous suspension containing 3 wt% of AlN powder was prepared by dispersing 7.5 g of AlN powder in 250 mL of deionized water preheated to 75 °C. Y-TZP specimens were immersed in the suspension dispersed with the AlN powder at a temperature of 75 °C for 15 minutes. Once exposed to water, dispersed AlN powder begins the following hydrolysis, resulting in the formation of a nano-structured boehmite (AlOOH) that coats the surface of immersed specimens.²⁵

 $AlN + 2H_2O \longrightarrow AlOOH + NH_3$

The coated specimens were subsequently air-dried in an oven at 110 °C for 2 hours, and were thermally treated by heating in an electric resistance furnace at 900 °C for one hour. The heating rate was controlled at 10 °C/min. Subsequent heat treatment causes the precipitated boehmite to thermally decompose, forming a transitional alumina that later undergoes a series of polymorphic phase transformations due to temperature changes.²⁶

$$\gamma \text{AlOOH} \xrightarrow{300^2 - 500^2} \gamma \text{Al}_2\text{O}_3 \xrightarrow{700^2 - 900^2} \delta \text{Al}_2\text{O}_3 \xrightarrow{900^2 - 1000^2} \theta \text{Al}_2\text{O}_3 \xrightarrow{1000^2 - 1100^2} \alpha \text{Al}_2\text{O}_3$$

3. Coating characterization

To monitor the surface morphology of each group, two specimens from each group were analyzed using field-emission scanning electron microscopy (FE-SEM) (Supra 40VP, Carl Zeiss, Jena, Germany) before the shear bond strength test. Also, the fractured cross sections of 5 specimens from each group were filmed after the shear bond strength test to monitor failure modes.

4. Shear-bond strength test

Composite resin cylinders with 3 mm in diameter and 3 mm in height were fabricated by filling Teflon tubes with a composite resin

356

Filtec Z250 (3M ESPE, Seefeld, Germany) in three increments. The test tube was filled with composite resin up to 1mm high in each increment, which was light polymerized for 20 seconds, so that each tube received a total of 60 seconds of light. After the light polymerization, the composite cylinders were bonded to the Y-TZP ceramic surfaces subjected to each of the different surface treatments with a resin luting cement Rely X Unicem (3M ESPE, Seefeld, Germany). The static load of 10 N was applied to the top surface of the resin cylinder. Excess cement was removed using cotton pellets, and the composite cylinder interface. After that, the bonded specimens were left for 10 minutes at room temperature.

Each surface-treated Y-TZP ceramic group was divided into two subgroups of 10 each. One subgroup was stored in deionized water at 37°C for 24 hours and the shear bond strength was measured. In contrast, the other subgroup was thermally cycled (TC) between 5°C and 55°C for 6,000 cycles after 24 hours of storage in deionised water at 37°C and the shear bond strength was tested.

The shear-bond strength was measured with a Universal Testing Machine (Model 4201, Instron Co., Norwood, MA, USA) at a crosshead speed of 0.75 mm/min. The bond strength (S) was calculated using the following formula:

S = L/A

L: load at failure (N), S: shear bond strength (MPa), A: adhesive area (mm²)

5. Statistical analysis

The statistical analysis was performed using SPSS version 18.0 program (IBM Co., Chicago, IL, USA). In order to compare the shear bond strength between groups, the data were analyzed using analysis of variance (ANOVA). The Tukey HSD was performed for the post-hoc test (α =.05). The paired t-test was carried out to assess the influence of thermocycling on the shear bond strength change of each group (α =.05).

Results

1. Coating characterization

FE-SEM images (\times 5,000) of the Y-TZP ceramic specimens subjected to each of the different surface treatments are shown in Figure 1. Line-shaped micro grooves are observed on a polished surface of the Y-TZP ceramic substrate (Fig. 1A). The rough structures are visible on the surface of the specimens treated with airborne particle abrasion (Fig. 1B). The silica particles are observed on the sur-



Fig. 1. (A) FE-SEM image (\times 5,000) demonstrating polished Y-TZP surface, (B) FE-SEM image (\times 5,000) demonstrating airborne particle abraded Y-TZP surface, (C) FE-SEM image (\times 5,000) demonstrating tribochemical coating treated Y-TZP surface.



Fig. 2. (A) FE-SEM image (\times 50,000) demonstrating the large retentive surface architecture created after completion of nano-structured alumina coating on polished Y-TZP surface, (B) FE-SEM image (\times 50,000) demonstrating the large retentive surface architecture created after completion of nano-structured alumina coating on air abraded Y-TZP surface.

face of the specimens treated with Rocatec (Fig. 1C). The FE-SEM images (\times 50,000) after the completion of nano-structured alumina coating are shown in Figure 2.

The nano-structured alumina coating show good surface coverage on the Y-TZP surface, which consists of nano-structured lamellae. The polished specimen with nano-structured alumina coating (Fig. 2A) displays the same morphology and surface coverage as the airborne particle abraded specimen with nano-structured alumina coating (Fig. 2B). However, the polished specimen (Fig. 2A) has an overall flat surface, while the airborne particle abraded specimen (Fig. 2B) displays an irregular surface topography along the basal rough structure.

2. Shear-bond strength

Two-way ANOVA was performed to identify a possible interaction between thermocycling and surface treatment on the bond strength of resin cement (Table 2). The results showed no correlation between the two variables.

The mean and standard deviation values of the shear bond strength of the four different surface treatment groups were analyzed using one-way ANOVA (Table 3).

Table 2. Results of two-way ANOVA						
Source of variation	SS	df	MS	F	P-value	
Thermocycling	516.38	1	516.38	33.09	<.01	
Surface treatment	2509.28	3	836.43	53.09	<.01	
Thermocycling * Surface treatment	63.53	3	21.18	1.36	.2629	
Model	3089.19	7	441.31	28.28	<.01	
Error	1123.75	72	15.61			
Total	4212.94	79				

Group	24 h in water (1 day)	24 h in water (1 day) + 6,000 TC		
	(Mean \pm SD)	(Mean \pm SD)		
А	8.41 ± 2.93 aa	$4.23\pm2.60^{ ext{cB}}$		
R	$12.96\pm3.81^{\mathrm{aA}}$	$5.12\pm1.47^{ ext{cB}}$		
PC	$17.86\pm5.87^{ ext{b}}$	$14.85\pm2.81^{ m d}$		
AC	$22.99\pm6.11^{ ext{b}}$	$17.70\pm3.62^{\scriptscriptstyle d}$		

Table 3. Shear-bond strength in MPa of composite resin cement (RelyX Unicem) to Y-TZP ceramic after different types of surface-conditioning

Same superscript small letters indicate no significant difference in the column, same superscript capital letters indicate no significant difference in the row. TC, Thermocycling.

Among the experimental groups stored in distilled water at room temperature for 24 hours after bonding, the AC group showed the highest shear bond strength at 22.99 MPa, followed by PC, R and A groups. A group showed the lowest bond strength at 8.41 MPa.

Among the experimental groups thermocycled for 6,000 cycles following 24 hours of storage in distilled water at room temperature after bonding, the AC group again showed the highest bond strength at 17.70 MPa, followed by PC, R and A groups.

Figure 3 shows the results of the Tukey HSD post-hoc test which was conducted to compare the shear bond strength between experimental groups following 24 hour storage in distilled water at room temperature after bonding.

PC and AC groups, which were treated with nano-alumina coating, displayed relatively higher shear bond strengths than A and R groups, which were not subjected to nano-alumina coating. PC groups did not show a statistically meaningful difference in shear bond strength compared to AC (P>.05), but possessed significantly higher shear bond strength than A and R groups (P<.05). However, there was no significant difference in shear bond strength between A and R groups (P>.05).

Figure 4 displays the results of the Tukey HSD post-hoc test which was carried out to compare the shear bond strength between the experimental groups following 24 hour storage in distilled water at room temperature and thermocycling for 6,000 cycles.

Similar to the results of the shear bond strength test before thermocycling, PC and AC groups, which were treated with nano-alumina coating, showed significantly higher shear bond strengths than A and R groups which were not subjected to nano-alumina coating (P<.05). However, there were no meaningful differences not only between PC and AC groups with coating treatment but also between A and R groups without coating (P>.05).

The influence of thermocycling on the shear bond strength of each group was analyzed and compared using the paired t-test, as shown in Figure 5. A and R groups, to which nano-alumina coating treatment was not applied, displayed significant drops in shear



Fig. 3. Shear-bond strength before thermocycling. The bars represent the SDs. * indicates significant difference (P < 05).



Fig. 4. Shear-bond strength after thermocycling. The bars represent the SDs. * indicates significant difference (P<.05).



Fig. 5. Influence of thermocycling on changes in shear bond strength of each group. * indicates significant difference (P<.05).

bond strength after thermocycling (P<.05). In contrast, PC and AC groups, which underwent the nano-alumina coating treatment, did not exhibit a statistically meaningful decrease in shear bond strength after thermocycling (P>.05).

3. Failure mode

After conducting the shear bond strength test, failure modes were observed using FE-SEM (Fig. 6, Fig. 7, Fig. 8).

Figure 6 and Figure 7 show the 2,000 times magnified images of failure modes in A and R groups respectively. In A group, rough structures are predominantly visible on the Y-TZP surface. In R group, it seems that the resin cement is mixed with rough structures. Therefore, A group predominantly represents an adhesive failure mode, while R group displays a mixed failure mode.

Figure 8 displays the 5,000 times and 40,000 times magnified images of a failure mode of PC group. At low magnification, most of the coating remains on the surface of the specimen (Fig. 8A). Magnifying



Fig. 6. FE-SEM image (×2,000) demonstrating adhesive failure mode of A group.

the red-colored box by 40,000 times shows the remaining coating on the surface of the specimen with resin cement bonded to the top (Fig. 8B).

Figure 9 shows the 2,000 times and 20,000 times magnified images of a failure mode in AC group. The low magnification image cannot confirm whether the coating is peeled off or not (Fig. 9A). However, if it is magnified by 20,000 times, it seems that a thin layer of resin cement is bonded to the top of the entire coated area (Fig. 9B). In the PC and AC groups, which underwent nano-alumina coating treatment, most of the nano-alumina coating remains even after the resin blocks are taken apart. These groups predominantly show a cohesive failure mode, in which the resin cement is retained within or on the remaining nano-alumina coating.



Fig. 7. FE-SEM image (×2,000) demonstrating mixed failure mode of R group.



Fig. 8. (A) FE-SEM image (×5,000) demonstrating cohesive failure mode of PC group, (B) FE-SEM image (×40,000) demonstrating high magnification of box in Fig. 8A.



Fig. 9. (A) FE-SEM image (\times 2,000) demonstrating cohesive failure mode of AC group, (B) FE-SEM image (\times 20,000) demonstrating high magnification of box in Fig. 9A.

Discussion

To achieve a strong and durable bond strength between resin cement and ceramic materials, it is important to cleanse, roughen and activate the adhesive surface area before cementation.²⁷

FE-SEM images (Fig. 2) show a dense and uniform nano-structured alumina coating structure on Y-TZP ceramics, which can enhance micro mechanical interlocking with resin cements. Consequently, the application of nano-structural alumina coating on the Y-TZP surface can enhance shear bond strength. The surfaces of PC and AC groups, which were treated with the coating, showed similar nanostructures though the overall surface of AC group appeared to be irregular. This is ascribed to the fact that the original surfaces of specimens are formed in different ways (polishing or airborne particle abrasion) before nano-alumina coating. Also, despite slight morphological difference between the PC and AC coating groups, there was no significant disparity in the bond strength between the groups. This indicates that the application of nano-alumina coating is a critical factor in expanding the surface area regardless of original surface treatments. Therefore, if nano-alumina coating is applied, no additional invasive surface treatments (ex. sandblasting) are needed to enhance the bond strength.

Airborne particle abrasion is commonly used as a mechanical method for creating micro-retentions on Y-TZP ceramic surfaces and its use under the right condition is often referred to as an important factor in boosting shear bond strength.^{9,28-30} Many research literatures have reported that such airborne particle abrasion treatment on Y-TZP ceramic surfaces can achieve a high bond strength by increasing the roughness of the surface.^{29,31,32} In contrast, Kern and Wegner³⁰ claimed that surface treatment of Y-TZP ceramics with airborne particle abrasion increased the bond strength with Bis-GMA resin cement, but the treatment was not stable for long term storage. In this study, A group treated with airborne particle abrasion showed the lowest bond strength and displayed significantly lower bond strength compared to PC and AC groups which were treated with nano-alumina coating (Table 3). This indicates that airborne particle abrasion can contribute to enhancing mechanical interlocking by roughening the Y-TZP ceramic surface but the process has a significantly lower bonding potential than nano-structured alumina coating treatment, which can create a large micro-retentive area.

In many studies, Blatz *et al.*³² and Amaral *et al.*³³ reported that tribochemical silica coating treatment resulted in stable bond strength even after thermocycling, whereas other treatments exhibited sharp decreases in the bond strength after thermocycling. In addition, Özcan³⁴ reported that there was no significant increase in bond strength after silica coating treatment. The studies conducted by Ernst³⁵, Kern and Wegner³⁰ showed that tribochemical silica coating was ineffective in increasing bond strength. In this study, R group treated with tribochemical silica coating using Rocatec system showed higher bond strength than A group treated with airborne particle abrasion, but lower bond strength than PC and AC groups treated with nano alumina coating (Table 3). This indicates that chemical bonding due to silica-modified alumina particles and silane embedded in the Y-TZP ceramic surface can contribute to enhancing bond strength, though the increase was not significant. In a study by Matinlinna *et al.*,³⁶ it was reported that the amount of silica remaining on the Y-TZP surface is too low for efficient silanization.

PC and AC groups treated with nano alumina coating displayed higher shear bond strength than the other groups. This was due to increased resin bonding resulting from a large micro-retentive area formed on the surface of the Y-TZP ceramic substrate. In a study of Jevnikar *et al.*,⁴ it was reported that nano-sized inter-lamella spaces are open for resin penetration and are filled with resin matrix, forming an intermediate structure designated as the hybrid layer. The formation of the hybrid layer can improve the bond strength of resin cement, which has been shown by the results of this study.

As thermocycling is mainly used to simulate the oral environment, several studies have suggested that resin-bond strength is degraded after thermocycling.^{29,32,37} This was the case with this study; both A group, treated with airborne particle abrasion, and R group, treated with tribochemical silica coating, showed a significant decrease in shear bond strength after thermocycling (Fig. 5). In contrast, PC and AC groups, treated with nano-alumina coating, showed no significant drop in bond strength after thermocycling, which indicates that nano-structured alumina coating is not influenced by thermocycling. This may be ascribed to the formation of a hybrid layer that is stronger than the hydrothermal bond degradation forces during thermocycling.^{38,39}

Group A predominantly displayed an adhesive failure mode (Fig. 6), while R group mainly showed a mixed failure mode (Fig. 7). In contrast, PC and AC groups, treated with nano-alumina coating, showed a predominant cohesive failure mode, and their nano-alumina coating was firmly attached to the surface of the Y-TZP ceramic even after fracture (Fig. 8, Fig. 9). The nano-sized inter-lamella spaces on the alumina coating remaining on the surface of the substrate after fracture were filled with the resin matrix, forming a hybrid layer. This supports the claim that PC and AC groups display a predominantly cohesive failure mode.

There is still debate over the effect of airborne particle abrasion on the bond strength of zirconia. Some studies have reported that airborne particle abrasion can enhance the strength of zirconia,^{40,41} while others reported observing a degradation in the strength of zirconia treated with airborne particle abrasion.⁴² Other studies reported that airborne particle abrasion treatment caused micro cracks, which in turn decreased flexural strength.⁴³ As a non-invasive process which does not create flaws on surfaces, nano-structured alumina coating has an advantage over airborne particle abrasion in that it can enhance the bond strength with resin without causing degradation in the strength of the Y-TZP ceramic.

Further research on standardization options for nano-structured alumina coating on zirconia restoration and study of long term clinical trials are needed.

Conclusion

To determine the influence of nano-structured alumina coating on the bond strength of a resin cement, this study measured the shear bond strength of Y-TZP ceramic substrates with resin cement after each of four different surface treatments. The groups with nanostructured alumina coating treatment displayed significantly higher shear strength than the groups with different treatments and had no statistically meaningful decline in shear bond strength. The application of nano-structured alumina coating can substantially increase the bond strength of resin cement to zirconia and can be a new method for improving the bond strength.

ORCID

Dong-Woon Kim *http://orcid.org/0000-0003-3609-6350* Jung-Jin Lee *http://orcid.org/0000-0002-7381-5230* Kyoung-A Kim *http://orcid.org/0000-0002-2923-5351* Jae-Min Seo *http://orcid.org/0000-0001-5095-4046*

References

- Anusavice KJ. Recent developments in restorative dental ceramics. J Am Dent Assoc 1993;124:72-4, 76-8, 80-4.
- Chevalier J. What future for zirconia as a biomaterial? Biomaterials 2006;27:535-43.
- Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. Biomaterials 1999;20:1-25.
- Jevnikar P, Krnel K, Kocjan A, Funduk N, Kosmac T. The effect of nano-structured alumina coating on resin-bond strength to zirconia ceramics. Dent Mater 2010;26:688-96.
- Denry I, Kelly JR. State of the art of zirconia for dental applications. Dent Mater 2008;24:299-307.
- Della Bona A, Anusavice KJ, Shen C. Microtensile strength of composite bonded to hot-pressed ceramics. J Adhes Dent 2000; 2:305-13.
- Della Bona A, Anusavice KJ. Microstructure, composition, and etching topography of dental ceramics. Int J Prosthodont 2002;15:159-67.
- Brentel AS, Ozcan M, Valandro LF, Alarça LG, Amaral R, Bottino MA. Microtensile bond strength of a resin cement to feldpath-

ic ceramic after different etching and silanization regimens in dry and aged conditions. Dent Mater 2007;23:1323-31.

- Ozcan M, Vallittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. Dent Mater 2003;19:725-31.
- 10. Dérand P, Dérand T. Bond strength of luting cements to zirconium oxide ceramics. Int J Prosthodont 2000;13:131-5.
- 11. Aboushelib MN, Matinlinna JP, Salameh Z, Ounsi H. Innovations in bonding to zirconia-based materials: Part I. Dent Mater 2008;24:1268-72.
- Aboushelib MN, Mirmohamadi H, Matinlinna JP, Kukk E, Ounsi HF, Salameh Z. Innovations in bonding to zirconiabased materials. Part II: Focusing on chemical interactions. Dent Mater 2009;25:989-93.
- Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent 2003;89:268-74.
- Ozcan M, Kerkdijk S, Valandro LF. Comparison of resin cement adhesion to Y-TZP ceramic following manufacturers' instructions of the cements only. Clin Oral Investig 2008;12:279-82.
- Amaral R, Ozcan M, Bottino MA, Valandro LF. Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: the effect of surface conditioning. Dent Mater 2006;22:283-90.
- Heikkinen TT, Lassila LV, Matinlinna JP, Vallittu PK. Effect of operating air pressure on tribochemical silica-coating. Acta Odontol Scand 2007;65:241-8.
- Ozcan M. Evaluation of alternative intra-oral repair techniques for fractured ceramic-fused-to-metal restorations. J Oral Rehabil 2003;30:194-203.
- Valandro LF, Ozcan M, Bottino MC, Bottino MA, Scotti R, Bona AD. Bond strength of a resin cement to high-alumina and zirconiareinforced ceramics: the effect of surface conditioning. J Adhes Dent 2006;8:175-81.
- Xible AA, de Jesus Tavarez RR, de Araujo Cdos R, Bonachela WC. Effect of silica coating and silanization on flexural and composite-resin bond strengths of zirconia posts: An in vitro study. J Prosthet Dent 2006;95:224-9.
- Bottino MA, Valandro LF, Scotti R, Buso L. Effect of surface treatments on the resin bond to zirconium-based ceramic. Int J Prosthodont 2005;18:60-5.
- Ural C, Külünk T, Külünk S, Kurt M, Baba S. Determination of resin bond strength to zirconia ceramic surface using different primers. Acta Odontol Scand 2011;69:48-53.
- Mirmohammadi H, Aboushelib MN, Salameh Z, Feilzer AJ, Kleverlaan CJ. Innovations in bonding to zirconia based ceramics: Part III. Phosphate monomer resin cements. Dent Mater 2010; 26:786-92.
- Wegner SM, Gerdes W, Kern M. Effect of different artificial aging conditions on ceramic-composite bond strength. Int J Prosthodont 2002;15:267-72.
- 24. Akgungor G, Sen D, Aydin M. Influence of different surface treatments on the short-term bond strength and durability between a zirconia post and a composite resin core material. J Prosthet Dent 2008;99:388-99.
- Novak S, Kosmac T. Preparation of alumina ceramics from aqueous suspensions employing the hydrolysis of aluminum ni-

tride. J Mater Res 2002;17:445-50.

- 26. Krnel K, Kocjan A, Kosmac T. A simple method for the preparation of nanostructured aluminate coatings. J Am Ceram Soc 2009;92:2451-4.
- Zhang S, Kocjan A, Lehmann F, Kosmac T, Kern M. Influence of contamination on resin bond strength to nano-structured alumina-coated zirconia ceramic. Eur J Oral Sci 2010;118:396-403.
- Lindgren J, Smeds J, Sjögren G. Effect of surface treatments and aging in water on bond strength to zirconia. Oper Dent 2008;33: 675-81.
- 29. Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. Dent Mater 2007;23:45-50.
- 30. Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. Dent Mater 1998;14:64-71.
- 31. Phark JH1, Duarte S Jr, Blatz M, Sadan A. An in vitro evaluation of the long-term resin bond to a new densely sintered high-purity zirconium-oxide ceramic surface. J Prosthet Dent 2009;101:29-38.
- 32. Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. Quintessence Int 2007;38:745-53.
- 33. Amaral R1, Ozcan M, Valandro LF, Balducci I, Bottino MA. Effect of conditioning methods on the microtensile bond strength of phosphate monomer-based cement on zirconia ceramic in dry and aged conditions. J Biomed Mater Res B Appl Biomater 2008;85:1-9.
- 34. Özcan M, Nijhuis H, Valandro LF. Effect of various surface conditioning methods on the adhesion of dual-cure resin cement with

MDP functional monomer to zirconia after thermal aging. Dent Mater J 2008;27:99-104.

- Ernst CP, Cohnen U, Stender E, Willershausen B. In vitro retentive strength of zirconium oxide ceramic crowns using different luting agents. J Prosthet Dent 2005;93:551-8.
- Matinlinna JP, Heikkinen T, Ozcan M, Lassila LV, Vallittu PK. Evaluation of resin adhesion to zirconia ceramic using some organosilanes. Dent Mater 2006;22:824-31.
- Lüthy H, Loeffel O, Hammerle CH. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. Dent Mater 2006;22:195-200.
- Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, De Stefano Dorigo E. Dental adhesion review: aging and stability of the bonded interface. Dent Mater 2008;24:90-101.
- Yang B, Barloi A, Kern M. Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin. Dent Mater 2010;26:44-50.
- Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. Dent Mater 1999;15:426-33.
- 41. Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. Strength and reliability of surface treated Y-TZP dental ceramics. J Biomed Mater Res 2000;53:304-13.
- 42. Zhang Y1, Lawn BR, Malament KA, Van Thompson P, Rekow ED. Damage accumulation and fatigue life of particle-abraded ceramics. Int J Prosthodont 2006;19:442-8.
- Zhang Y, Lawn BR, Rekow ED, Thompson VP. Effect of sandblasting on the long-term performance of dental ceramics. J Biomed Mater Res B Appl Biomater 2004;71:381-6.

나노구조 알루미나 코팅 처리가 지르코니아 도재와 레진 시멘트 사이 전단 결합강도에 미치는 영향

김동운¹ · 이정진¹ · 김경아² · 서재민^{1*}

'전북대학교 치의학전문대학원 치과보철학교실 및 구강생체과학연구소, '을지대학교 의과대학 치과학교실

목적: 본 연구는 지르코니아 도재 표면의 나노구조 알루미나 코팅이 지르코니아와 레진 시멘트와의 전단결합강도에 미치는 영향을 알아보고자 하 였다.

재료 및 방법: 지르코니아 원판 80개를 표면처리방법(산화알루미늄 분사처리(A), 산화알루미늄 분사 후 Rocatec 처리(R), 연마 후 나노구조 알루미나 코팅(PC), 산화알루미늄 분사 후 나노구조 알루미나 코팅(AC))에 따라 4개의 군으로 나누었다. 알루미나 코팅은 질산 알루미늄을 가수분해시킨 용 액에 침적 후 900°C에서 열처리 하여 시행하였다. 지르코니아 표면 코팅은 주사전자 현미경을 이용하여 관찰하였다. 레진 블럭을 레진 시멘트를 이 용하여 각 실험군의 지르코니아 표면에 합착하고 열순환처리 전, 후의 전단결합강도를 측정하였다.

결과: 알루미나 코팅을 한 지르코니아 표면은 균일하고 치밀한 나노구조 알루미나가 관찰되었다. PC, AC 군은 열순환처리 전과 후 모두 A와 R 군에 비해 현저하게 높은 전단결합 강도를 보였다. A, R 군은 열순환처리 후에 급격한 결합강도의 감소를 보였으나, PC와 AC군은 열순환처리에 의해 유 의할만한 결합강도의 감소를 보이지 않았다.

결론: 지르코니아 표면에 나노구조 알루미나 코팅처리하는 것은 레진시멘트와의 결합강도를 증가시키는 방법이다. (대한치과보철학회지 2016;54:354-63)

주요단어: 지르코니아; 전단결합강도; 알루미나 코팅; 열순환처리; 레진 시멘트

ⓒ 2016 대한치과보철학회
ⓒ 이 글은 크리에이티브 커먼즈 코리아 저작자표시-비영리 3.0 대한민국 라이선스에 따라 이용하실 수 있습니다.