



Effect of liners and primers on tensile bond strength between zirconia and resin-based luting agent

Eun-Hye Jo, Yoon-Hyuk Huh, Kyung-Ho Ko, Chan-Jin Park, Lee-Ra Cho*

Department of Prosthodontics and Research Institute of Oral Science, College of Dentistry, Gangneung-Wonju National University, Gangneung, Republic of Korea

PURPOSE. The effect of silica-based glass-ceramic liners on the tensile bond strength between zirconia and resin-based luting agent was evaluated and compared with the effect of 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-containing primers. **MATERIALS AND METHODS.** Titanium abutments and zirconia crowns (n = 60) were fabricated, and the adhesive surfaces of the specimens were treated by airborne-particle abrasion. The specimens were divided into 5 groups based on surface treatment: a control group, 2 primer groups (MP: Monobond Plus; ZP: Z Prime Plus), and 2 liner groups (PL: P-containing Liner; PFL: P-free Liner). All specimens were cemented with self-adhesive resin-based luting agent. After 24-hour water storage and thermocycling (5,000 cycles, 5°C/55°C), the tensile bond strength was measured using a universal testing machine. Failure mode analysis and elemental analysis on the bonding interface were performed. The data were analyzed using Kruskal-Wallis test, Dunn's post hoc test, and Fisher's exact test. **RESULTS.** The liner groups and primer groups showed significantly higher tensile bond strengths than that of the control group ($P < .05$). PFL showed a significantly higher tensile bond strength than the primer groups ($P < .05$). The percentage of mixed failure was higher in the primer groups than in the control group ($P < .001$), and all the specimens showed mixed failure in the liner groups ($P < .001$). A chemical reaction area was observed at the bonding interface between zirconia and liner. **CONCLUSION.** The application of liner significantly increased the tensile bond strength between zirconia and resin-based luting agent. PFL was more effective than MDP-containing primers in improving the tensile bond strength with the resin-based luting agent. [J Adv Prosthodont 2018;10:374-80]

KEYWORDS: Resin-based luting agent; Methacryloyloxydecyl dihydrogen phosphate (MDP); Liner; Tensile bond strength; Zirconia

INTRODUCTION

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) has

been widely used as a material for prostheses owing to its biocompatibility and excellent mechanical properties.¹ Zirconia prostheses have shown a wide range of survival rate (81 - 100%) in 5-year observation, which was reported to be associated with mechanical complications such as veneer chipping and loss of retention.²⁻⁴ Loss of retention occurs more frequently in Y-TZP than in porcelain-fused metal prostheses.⁵ Ortorp *et al.*³ monitored monolithic zirconia prostheses for 5 years and insisted that loss of retention accounted for the highest percentage of total complications (30%). Most of those prostheses were bonded using resin-based luting agent.

All-ceramic prostheses can form stable chemical bonds with resin-based luting agent through hydrofluoric acid and silane applications.⁶ However, such surface processing is not possible in zirconia due to its polycrystalline structure.⁷ For this reason, researchers have explored various surface treat-

Corresponding author:

Lee-Ra Cho
Department of Prosthodontics and Research Institute of Oral Science,
College of Dentistry, Gangneung-Wonju National University
7 Jukheon-gil, Gangneung, Gangwondo 25457, Republic of Korea
Tel. +82336403153; e-mail, lila@gwnu.ac.kr
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ment methods to improve the bond strength between zirconia and resin-based luting agent, such as airborne-particle abrasion (APA), laser treatment, elective infiltration etching, silica coating, and functional monomer application.⁸ For luting of zirconia prostheses, APA followed by 10-methacryloyloxydecyl dihydrogen phosphate (MDP) or silane application is the most common treatment.⁹⁻¹¹

MDP, a type of acid phosphate monomer, was originally developed to increase the bond strength between metal and resin-based luting agent, and is now used in zirconia prosthesis.¹² It forms phosphate-oxygen-zirconium bonds with zirconia.¹³ To obtain a sufficient bond strength, MDP should be accompanied with APA treatment.¹ However, APA can cause microcracks on zirconia surface, which lead to fracture or damage on the zirconia margin.^{14,15}

Tribochemical silica coating has been reported to effectively improve the bond strength in high-strength ceramics.¹⁶⁻¹⁸ Micromechanical retention and chemical bonding with silica particles can be achieved through tribochemical silica coating.^{11,19} However, these effects may be reduced in zirconia, which has a high surface hardness.²⁰ Matinlinna *et al.*,²⁰ through energy-dispersive X-ray spectroscopy (EDS) analysis, reported that tribochemical treatment of zirconia surface induced insufficient silica coating. It has also been claimed that silica does not chemically bind to zirconia surfaces.²¹ The effect of silica coating can be improved by increasing the blasting pressure during tribochemical silica coating.²² However, the possibility of zirconia surface damage cannot be ruled out.²³

Researchers have proposed another technique in which silica is added to zirconia surfaces instead of the subtraction techniques.²⁴⁻²⁶ Several studies reported that chemical bonding occurred at the bonding interface between zirconia and ceramics.²⁷⁻²⁹ Lung *et al.*²⁵ proposed a sol-gel method for silica-based ceramics. However, this technique resulted in a significantly low shear bond strength with resin-based luting agent compared with that obtained after APA, and the coat-

ing was unstable after thermocycling. Kitayama *et al.*²⁴ proposed an internal coating system (INT), in which the inner surface of zirconia prostheses is partially or fully covered with silica-based ceramics through high-temperature firing. A significant increase in bond strength with resin-based luting agent and compensation of the large internal gap within the zirconia prostheses were achieved with INT.²⁶ However, few studies have compared the INT with different surface processing methods, and have assessed the stability of the coating after thermocycling.

In this study, the effects of silica-based glass-ceramic liners and MDP-containing primers on the tensile bond strength between zirconia and resin-based luting agent following thermocycling were investigated. The null hypothesis was that different surface treatment techniques do not have any effect on the tensile bond strength between zirconia and resin-based luting agent and on the failure mode.

MATERIALS AND METHODS

Sixty titanium abutments and zirconia crowns (12 per group) were fabricated from titanium blocks (commercially pure titanium, grade V) and pre-sintered block (Zirtooth, Hass, Gangneung, Korea) with CAD/CAM program (hyperMiLL, Samwoohyper, Seongnam, Korea). To minimize the effects of mechanical retention, the convergence angle of the titanium abutment was set to 20 degrees on one side, making a total of 40 degrees (Fig. 1). The titanium abutments and intaglio of zirconia crowns were subjected to APA (Basic master, Renfert, Hilzingen, Germany) by vertically spraying alumina (Al₂O₃) particles from a distance of 10 mm at 0.25 MPa. Specimens were divided into 5 groups depending on the applied surface treatment: control group (APA), MP group (APA+Monobond Plus), ZP group (APA+Z Prime Plus), PL group (APA+P-containing Liner), and PFL group (APA+P-free Liner) (Table 1).

The internal gap between the titanium abutment and zir-

Table 1. Materials used

Material	Brand name	Lot No.	Main composition	Manufacturer
Zirconia	Zirtooth	S41FIB901	Zirconium dioxide, hafnium dioxide, yttrium trioxide, inorganic pigment, organic binder	HASS
Resin-based luting agent	RelyX Unicem	3328586	Glass powder, initiator, silica, substituted pyrimidine, calcium hydroxide, peroxy compound, pigment, methacrylate phosphoric ester, dimethacrylate, acetate, stabilizer	3M ESPE
Hydrofluoric acid gel	Porcelain etchant	1700004012	4% Hydrofluoric acid	Bisco Inc
Silane coupling agent	Monobond S	U17000	3-(methacryloxy)propyltrimethoxysilane, water, ethanol	Ivoclar Vivadent
Primer	Monobond Plus (MP)	V33449	3-(trimethoxysilyl)propyl methacrylate, sulphide methacrylate, methacrylated phosphoric acid ester	Ivoclar vivadent
Primer	Z Prime Plus (ZP)	1600002409	Organophosphate monomer, carboxylic acid monomer	Bisco Inc
Liner	P-containing Liner (PL)	EO1D03	Silicon dioxide, lithium oxide, aluminum oxide, phosphorus pentoxide, etc.	HASS
Liner	P-free Liner (PFL)	EO1D11	Silicon dioxide, lithium oxide, aluminum oxide, etc.	HASS

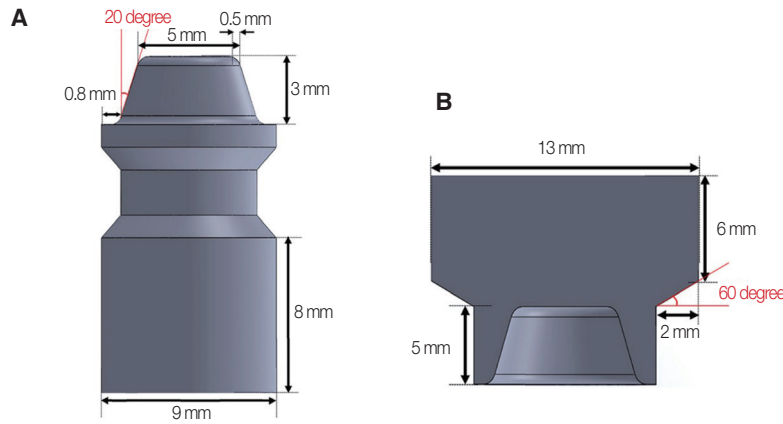


Fig. 1. Titanium abutment and zirconia crown. (A) Schematic diagram of titanium abutment, (B) Schematic diagram of zirconia crown.

conia crown was determined by measuring the mean liner thickness in 10 additional zirconia crowns that were cut using an Accutom-50 cutting machine (Struers, Ballerup, Denmark) (Fig. 2). An internal gap of 20 μm for cement was set in the control and primer groups, while the mean liner thickness (15 μm) was added for the liner group. A spray-type liner was sprayed from a distance of 15 - 20 mm for 2 - 3 seconds and fired for 1.5 minutes at 950°C in a porcelain furnace (Austromat D4, Dekema, Freilassing, Germany). The zirconia crowns were bonded to the titanium abutments using the self-adhesive RelyX Unicem (3M ESPE, Seefeld, Germany). In the liner group, the intaglio of zirconia crown was treated with 4% hydrofluoric acid (Porcelain etchant, Bisco Inc., Schaumburg, IL, USA) for 5 minutes and uniformly coated with silane (Monobond S, Ivoclar Vivadent, Liechtenstein, Germany) for 60 seconds. The bonding interface between the titanium abutment and zirconia specimen was observed at $\times 10,000$ magnification using a scanning electron microscope (SEM, Quanta FEG 250, FEI, Hillsboro, OR, USA). For the liner group, an EDS (Apollo XP, EDAX, Mahwah, NJ, USA) connected to the SEM was used for line scan analysis to observe the elemental distribution between the liner and zirconia.

All specimens were stored in distilled water at 37°C for 24 hours, and then thermocycling was performed for 5,000 cycles (5°C and 55°C, 2 seconds dwelling time; Thermal cyclic tester, R&B Inc., Daejeon, Korea). A tensile force was applied in the axial direction at a crosshead speed of 1.0 mm/min using a universal testing machine (Model 5982, Instron, Norwood, MA, USA). After the tensile bond strength test, the outer surface of the titanium abutment and the intaglio of the zirconia crown were examined at $\times 300$ magnification using an optical microscope (VHX S550E, Keyence, Itasca, TX, USA). Failure modes were categorized into adhesive failure, cohesive failure, and mixed failure. If a mixed failure was observed, the specimen was examined at $\times 100$ and $\times 200$ magnifications under a SEM

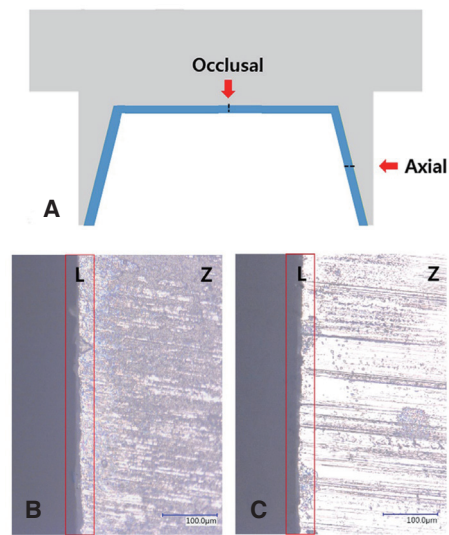


Fig. 2. Liner thickness measurement (optical microscope; original magnification: $\times 500$). (A) Measurement sites, (B) Optical microscopic image to measure occlusal space, (C) Optical microscopic image to measure axial space. L, Liner; Z, Zirconia.

to confirm the failure mode.

The Kruskal-Wallis test and Dunn's post hoc test (IBM SPSS 23.0, IBM, Armonk, NY, USA, $\alpha = .05$) were used to analyze the significant differences among the tensile bond strengths achieved by the different surface treatment techniques. Two-sided P values $< .05$ were accepted as statistically significant. Further, Fisher's exact test was used to determine the significant differences in the failure mode among the groups.

RESULTS

All the experimental groups showed significantly higher tensile bond strengths than that of the control group ($P < .05$). The tensile bond strengths of the PFL group was significantly higher than those of the ZP and MP groups (Fig. 3, $P < .05$).

In the failure mode analysis, significant differences in the failure modes were observed according to the surface treatment (Table 2, $P < .001$ with Fisher's exact test). Cohesive failure was not observed in any specimens. In the control group, adhesive failures were more frequently observed than mixed failures, whereas in the primer groups, mixed failures were more common than adhesive failures. The MP and ZP groups showed similar frequencies of adhesive failures. In both the liner groups (PL and PFL), all specimens presented mixed failures. When closely observed using a SEM, cohesive failures were observed mostly in the resin-based luting agent layers in the occlusal surface, and adhesive failures were observed between the zirconia crown and resin-based luting agent in the axial surface. Similar failure modes were observed in the occlusal surface of the liner group. However, in the axial surface, adhesive failures between the

resin-based luting agent and liner were usually observed, whereas adhesive failure between the liner and zirconia was not observed. In addition, it was seen that the zirconia surfaces were covered with the liner (Fig. 4).

EDS was used to perform line scanning to determine the elemental distribution of the specimens' components (Fig. 5). In the liner group, the intensities of zirconium (Zr) and silicon (Si) ion distributions in a chemical reaction layer changed. Diffusion of Zr into the liner and of Si, main components of the liner, into zirconia were observed. The interdiffusion zone was approximately 1 - 2 μm thick (Fig. 5).

Table 2. Distribution number (%) of failure modes

Group	Adhesive failure	Mixed failure	Total	<i>P</i> value
Control	9 (75%)	3 (25%)	12 (100%)	
MP	3 (25%)	9 (75%)	12 (100%)	< .001
ZP	4 (33.3%)	8 (66.7%)	12 (100%)	
PL	0 (0%)	12 (100%)	12 (100%)	
PFL	0 (0%)	12 (100%)	12 (100%)	

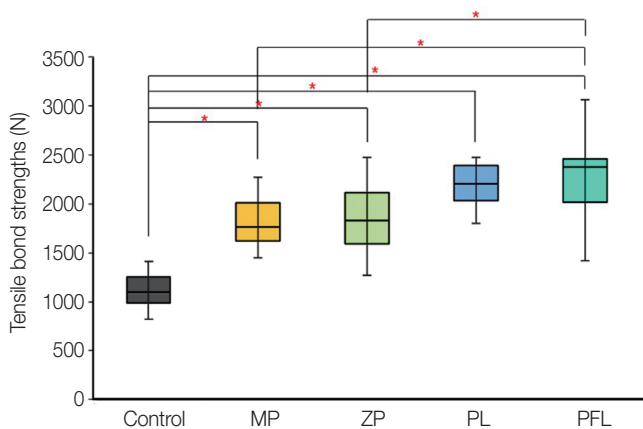


Fig. 3. Box plot for comparison of tensile bond strengths of different groups.

* Indicate statistically significant difference ($P < .05$).

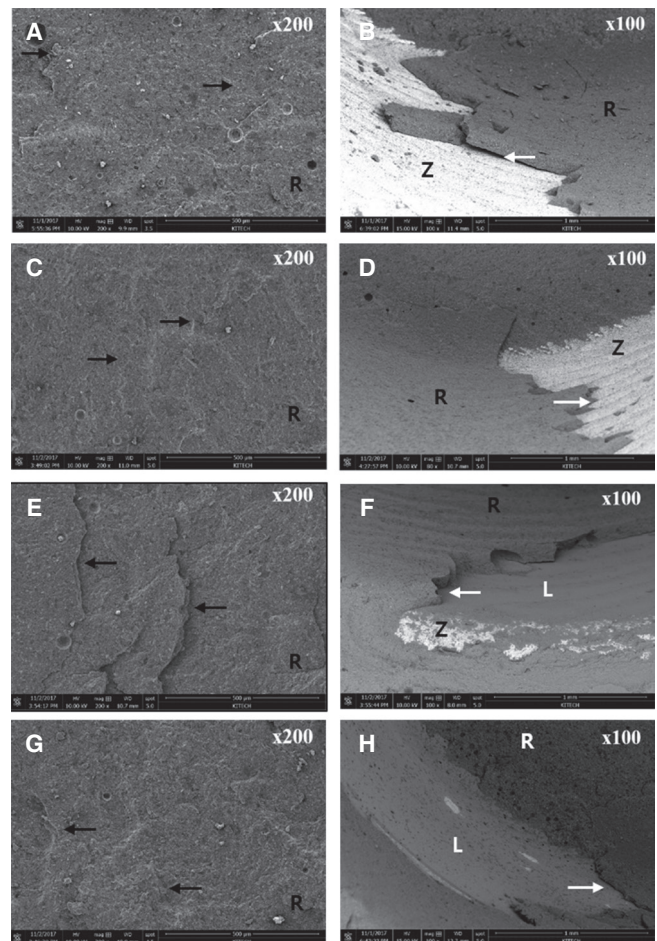


Fig. 4. Scanning electron microscopic (SEM) images of inner surface of zirconia crown after tensile bond test. Black arrow indicates cohesive failure in resin-based luting agent. White arrow indicates adhesive failure between zirconia and resin-based luting agent or liner and resin-based luting agent. (A) MP (occlusal surface), (B) MP (axial surface), (C) ZP (occlusal surface), (D) ZP (axial surface), (E) PL (occlusal surface), (F) PL (axial surface), (G) PFL (occlusal surface), and (H) PFL (axial surface). (A, C, E, G: original magnification $\times 200$; B, D, F, H: original magnification $\times 100$). R, Resin-based luting agent; Z, Zirconia; L, Liner.

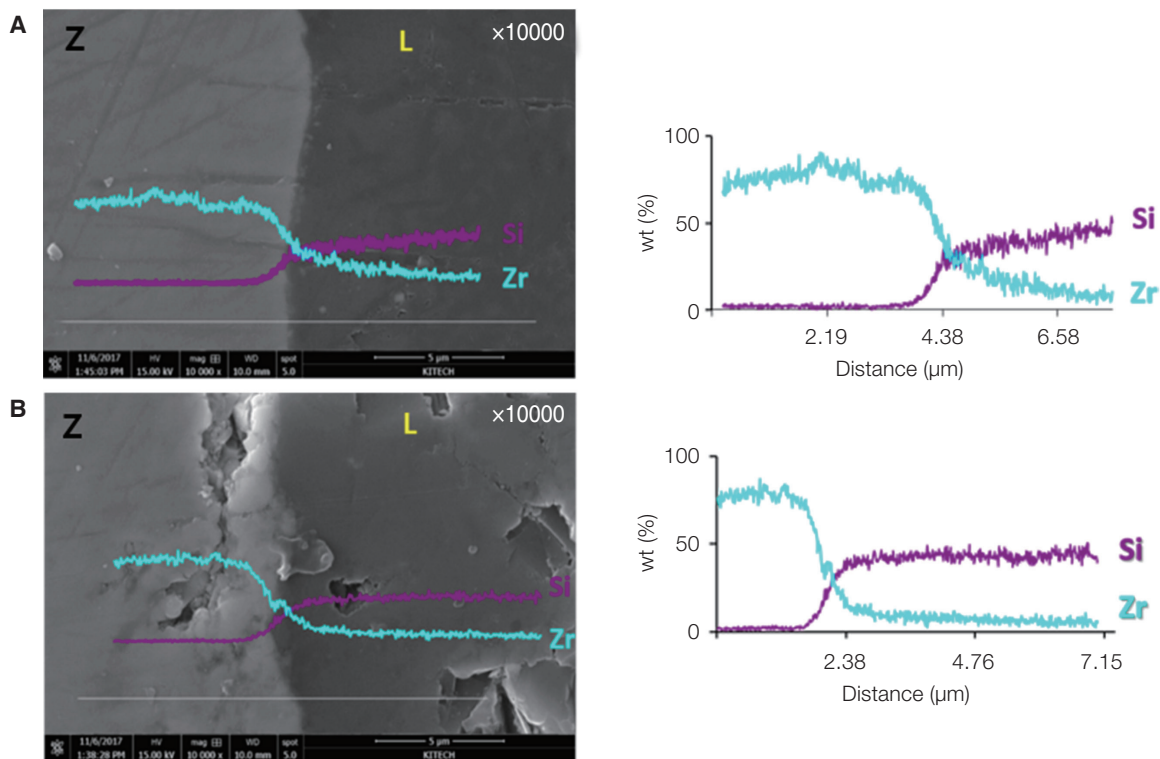


Fig. 5. Line scan analysis (original magnification: $\times 10,000$) of bonding interface between zirconia and liner. Chemical reaction area and diffusion of elements (Si: silicon, Zr: zirconium) observed across the interface. (A) PL, (B) PFL. Z, Zirconia; L, Liner.

DISCUSSION

Based on the measurement results, the null hypotheses of this study were rejected. The primer treatment and liner treatment significantly increased the tensile bond strength between zirconia and resin-based luting agent.

The MP and ZP groups both include MDP. The MP group additionally includes sulfide methacrylate and 3-(trimethoxysilyl) propyl methacrylate (3-MPS) monomers, which are silane monomers, while the ZP group additionally includes carboxylic acid monomer. In this study, there was no significant difference between the tensile bond strengths of the MP and ZP groups. MDP may significantly affect the bonding behavior of zirconia.

The biggest compositional difference between the PFL and PL groups was the presence of phosphorous pentoxide (P_2O_5). P_2O_5 acts as a nucleating agent to induce the formation of bonds between components.³⁰ Therefore, a definite crystal structure can be formed in the presence of P_2O_5 . As the crystal structure becomes clearer, the strength of the liner increases; however, the wettability decreases. This explains the significantly higher tensile bond strength of the PFL group than those of the primer groups, despite of no significant difference between the PL group and the primer groups.

Liner treatment increased the tensile bond strength more effectively than the primer treatment did. This was also confirmed in the failure mode analysis. In the failure mode analysis, the liner groups showed less adhesive failures than the primer groups did, which indicates that more stable and stronger bonds were formed between the zirconia, liner, and resin-based luting agent. Very few adhesive failures were observed between zirconia and liner, and most zirconia surfaces were covered with the liner. It appears that chemical reactions occurred during the liner firing process.²⁷⁻²⁹ Such results are consistent with previous findings. It was reported that the interdiffusion zone within the bonding interface between zirconia and ceramic was observed in EDS analysis.^{27,28} Durand *et al.*²⁹ suggested that the interdiffusion zone is 2 μm in thickness; this value is close to the thickness of the interdiffusion zone observed in this study. Since the liners are crystallized glass-ceramics with silica as the main component, they can form stable siloxane bonds with resin-based luting agent through hydrofluoric acid and silane treatments, which were applied in the conventional ceramic bonding procedures.

In this study, all the bonding surfaces between the titanium abutments and zirconia crowns were subjected to APA. There has been a controversy regarding the optimal blasting pressure and particle size for producing efficient bond strengths

and durability of zirconia. In this study, 50- μm alumina particles were sprayed at 0.25 MPa onto the intaglio of zirconia crown, which was suggested in a previous study.²³ The control, primer, and liner groups were all treated under the same conditions to minimize confounding effects.

Both the primer and liner groups showed higher tensile bond strengths than that of the control group after thermocycling. Studies that investigate the stability of liners after thermocycling procedure are rare, and there is a controversy regarding the stability of primers during thermocycling.¹⁵ The magnitude of the effects of thermocycling can vary depending on the exposure of the bonding interface. In this study, only the margin area was exposed to water. Furthermore, because the zirconia crowns were relatively thick, they may have been less susceptible to the effects of thermocycling. However, the liner may effectively resist thermocycling in which a 1 - 2 μm thick chemical reaction layer was formed in the bonding interface between the zirconia and liner.

There are various methods of liner treatment including dipping and spraying. Although Lee *et al.*²⁶ recommended the use of dipping method, it is a complicated process since it involves the mixing of powder and liquid. In addition, this method leads to a thick accumulation of liner around the line angle from the occlusal to the axial intaglio of the zirconia crown. In this study, the liners produced in the form of a spray were sprayed onto the intaglio of the zirconia crowns. The spraying method is simple and results in comparatively uniform distribution of particles. However, it is technically sensitive as the quantity of sprayed particles, and consequently, the liner thickness can be changed depending on the spraying distance and time.

Further, the tensile bond strength with resin-based luting agent significantly increased for the PFL group compared with the primer groups. However, the liner application has several limitations. First, the liner treatment is inferior in terms of convenience. Liners require high-temperature firing following application and must be treated with hydrofluoric acid and silane for bonding. Second, primer treatment can be performed following internal adjustment of ceramics in the chairside, and the liner layer can disappear during internal adjustment. Third, complete seating may be difficult to achieve if the thickness of the liner is larger than the internal gap of the prostheses previously set. Therefore, further studies are needed to simplify the liner treatment processes and to find methods to sensitively adjust the liner thickness. However, since zirconia prostheses typically have large and various internal gaps compared to other prostheses, the liners may compensate the misfit.²⁴

CONCLUSION

In this study, the inner surfaces of zirconia crowns were treated with primers or silica-based glass ceramic liners, and their tensile bond strengths with resin-based luting agent were measured. Primer treatment led to an increase in the tensile bond strength regardless of the primer types compared with the control group, and led to an increase in the

percentage of mixed failures. Liner treatment led to a significant increase in the tensile bond strength regardless of liner composition compared with the control group, and all specimens showed mixed failures. In addition, the liners without phosphorous compounds exhibited the highest tensile bond strength. Chemical reaction layers and 1 - 2 μm thick interdiffusion zone were observed at the bonding interface between zirconia and liner.

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ORCID

Eun-Hye Jo <https://orcid.org/0000-0001-8375-2563>

Yoon-Hyuk Huh <https://orcid.org/0000-0003-4072-5199>

Kyung-Ho Ko <https://orcid.org/0000-0002-1260-8844>

Chan-Jin Park <https://orcid.org/0000-0003-4734-214X>

Lee-Ra Cho <https://orcid.org/0000-0003-3989-2870>

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