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# Effect of Surface Treatments on the Bond Strengths of Facing Composite Resins to Zirconia Copings

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**Abstract** - *The present study evaluated and compared the bond strength between zirconia and facing composite resin using different surface conditioning methods before and after thermocycling. Four primers, three opaque resins, and two facing composite resins were used, and 10 surface treatment procedures were conducted. The bond strength was measured before and after 4,000 cycles of thermocycling. The mean values of each group were statistically analyzed using one-way analysis of variance (ANOVA). The bond strengths of facing composite resins to zirconia after various treatments varied depending on the primers, opaque resins, body resins, and thermocycling. The application of primers and opaque resins to the zirconia surface after sandblasting is expected to yield strong bond strength of the facing composite resin (Estenia C&B) even after thermocycling.*

KEY WORDS: Zirconia, Bond strength, Facing composite resin, Primer

## INTRODUCTION

Tooth-coloured restorations have been commonly used for permanent prostheses. Patients are currently concerned with treatment options that provide functional as well as aesthetic results. Two of the most popular and reliable materials for aesthetic restorations are porcelain-fused-to-metal crowns and all-ceramic crowns. However, as porcelain is a brittle material and lacks flexural strength, clinicians occasionally report fractures of the veneered porcelain. Recently, the zirconia coping has been introduced for all-ceramic restorations. Zirconium oxide has become particularly popular as a core material in dental restorations and is being used instead of conventional silica-based ceramics, alumina, and metal because of its mechanical strength, toughness, and esthetics[1,2]. In addition, CAD/CAM technologies make it possible to fabricate not only a crown but also a multiple-unit fixed partial denture coping/framework. All-ceramic crowns have shown a high success rate[3-6]; on the other hand, Bona *et al.*[7] have indicated the highest degree of chipping in the posterior region. These complications might be avoided when applying facing composite resins instead of porcelain, which has inherently poor tensile strength.

The introduction of high fillers containing facing composite resin has shown good colour stability, low wear, ease of handling, and a natural appearance[8-11]. A zirconia coping, rather than a metal one, could be used for a composite resin facing crown. Build-up facing composite resins on the coping could contribute to the easy fabrication of crowns with less heat damage to the coping than that resulting from porcelain firing. Problems, such as chipping, which often occur in the posterior region, can be avoided. Furthermore, when chipping occurs, it may be repaired in the mouth directly.

There have been many reports about the bond strength between composite resin cements and zirconia after several surface treatments[12-17]. However, no studies about the bond strength between zirconia and facing composite resin have been conducted. Facing composite resin systems have their pretreatment systems, which include primer, bonding and applying opaque resin before building up resin on the metal coping. The zirconia surface requires pretreatment so that bonding can be ensured. The present study evaluated and compared the bond strength between zirconia and facing composite resin using different surface conditioning methods before and after thermocycling.

The hypothesis was that the application of primers and/or opaque resins to the zirconia surface after sandblasting would yield strong bond strength of the facing composite resin even after thermocycling.

## MATERIALS AND METHODS

Cylindrical pre-sintered yttrium-oxide-partially-stabilized blocks (Lava™ Frame Bridges 60 mm, 3M ESPE, Seefeld, Germany) were sectioned with a diamond disc. Then, each specimen was stabilized using Lava Therm (3M ESPE) with a diameter of 19 mm and a thickness of 5 mm. One hundred specimens on one side were air-abraded with 50 µm $Al_2O_3$ at 0.4 MPa at a distance of 10 mm and then cleaned ultrasonically in distilled water for 5 minutes.

The primers, opaque resins, and facing composite resins used in this study are shown in Table 1. Four primers, three opaque resins, and two facing composite resins were used.

Zirconia specimens were randomly divided into 10 groups consisting of 10 specimens each according to surface conditioning methods (Table 2).

Specimens without an opaque layer (Groups 1 to 4) were fabricated as follows (Figure 1): a piece of polycarbonate ring with a circular hole 4.5 mm in diameter and 0.8 mm in thickness was positioned on the surface of each zirco-

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**Table 1.** *List of materials used; primers and facing composite resins*

| Product | Composition | Manufacturer | Batch number |
|---|---|---|---|
| **Primer** | | | |
| Clearfil Ceramic primer | 3-Methacryloxypropyl trimethoxy silane<br>10-Methacryloyloxydecyl dihydrogen phosphate(MDP)<br>Ethanol | Kuraray Medical Okayama, Japan | SCP-4 |
| Estenia C&B Modeling liquid | Methacrylic acid series monomer<br>Polyurethane methacryl monomer<br>MDP<br>Solvent<br>Others | Kuraray Medical Okayama, Japan | NML-42 |
| Estenia C&B Opaque primer | Methacrylic acid series monomer<br>MDP<br>Solvent<br>Others | Kuraray Medical Okayama, Japan | NOP-156 |
| AZ primer | Acetone<br>6-Methacryloxyhexyl Phosphonoacetate<br>Others | Shofu, Inc., Kyoto, Japan | 030701 |
| **Opaque resin** | | | |
| Estenia C&B Opaque resin | Monomer<br>(BisGMA, Other methacrylic acid series monomer)<br>Filler<br>(Surface-treated quartz powder, Prepolymerized resin filler, Others)<br>Photocuring catalyst<br>Colorant<br>Others | Kuraray Medical Okayama, Japan | O13-EP88 |
| Ceramage Pre-opaque | Urethane Dimethacrylate<br>2-Hydroxyethyl Methacrylate<br>Aluminum Silicate<br>Glass Filler<br>Others | Shofu, Inc., Kyoto, Japan | 110717 |
| Ceramage Opaque | Urethane Dimethacrylate<br>2-Hydroxyethyl Methacrylate<br>Aluminum Silicate<br>Glass Filler<br>Others | Shofu, Inc., Kyoto, Japan | 060824 |
| **Facing composite resin** | | | |
| Estenia C&B Body resin | Monomer<br>(Polyurethane methacrylmonomer Methacrylic acid series monomer)<br>Filler<br>(Surface-treated glass powder, Surface-treated aluminum micro filler)<br>Photocuring catalyst<br>Colorant<br>Others | Kuraray Medical Okayama, Japan | P13-CB9(ODA3)<br>D13-CB55(DA3) |
| Ceramage Opaque dentin | Urethane Dimethacrylate<br>Urethane Diacrylate<br>Zirconium Silicate<br>Others | Shofu, Inc., Kyoto, Japan | 020809(ODA3) |
| Ceramage Body | Urethane Dimethacrylate<br>Urethane Diacrylate<br>Zirconium Silicate<br>Others | Shofu, Inc., Kyoto, Japan | 020809(A3B) |

**Table 2.** *Surface treatment procedures*

| Group |
|---|
| 1 Clearfil Ceramic Primer ⇒Estenia C&B Body resin (ODA3) |
| 2 Estenia C&B Modeling liquid ⇒Estenia C&B Body resin (ODA3) |
| 3 Estenia C&B Modeling liquid ⇒Estenia C&B Body resin (DA3) |
| 4 Estenia C&B Opaque primer ⇒Estenia C&B Body resin (ODA3) |
| 5 Estenia C&B Modeling liquid ⇒Estenia C&B Opaque resin (ODA3) ⇒Estenia C&B Body resin(DA3) |
| 6 AZ Primere ⇒Ceramage Pre-opaque ⇒Ceramage Opaque dentin (ODA3) |
| 7 AZ Primere ⇒Ceramage Pre-opaque ⇒CeramageBody (A3B) |
| 8 AZ Primere ⇒Ceramage Pre-opaque ⇒Estenia C&B Body resin (ODA3) |
| 9 AZ Primere ⇒Ceramage Pre-opaque ⇒Ceramage Opaque (A3O) ⇒Ceramage Body (A3B) |
| 10 AZ Primere ⇒Ceramage Pre-opaque ⇒Estenia C&B Opaque resin (OA3) ⇒Eatenia C&B Body resin (DA3) |

nia specimen. Each primer was applied for 30 seconds and allowed to dry with oil-free air for 3 seconds. Estenia C&B Body resin (ODA3 and DA3) was filled in the ring, and a clear glass plate (1.5×1.5×0.5 cm) was put on it and then irradiated for two minutes for polymerization using a slide projector (Master HILUX-Hr, Philips, Eindhoven, Netherlands). The polycarbonate ring was then carefully removed from the zirconia surface, and Estenia C&B Body resin was heat-polymerized for 15 minutes at a temperature of 110°C using an oven (KL100, Kuraray, Tokyo, Japan).

For Groups 5 to 8, specimens with one layer of opaque resin were fabricated as follows (Figure 2): a piece of tape with a circular hole of 4.5 mm in diameter and 0.15 mm in thickness was positioned on the surface of each zirconia specimen. Each primer was applied for 30 seconds and allowed to dry with oil-free air for 3 seconds except for the AZ primer, and then opaque resin paste was filled and irradiated for two minutes. The tape was removed, and the same polycarbonate ring with a circular hole of 4.5 mm in diameter and 0.8 mm in thickness was fixed on the opaque layer. Estenia C&B Body resin (OA3, DA3, and ODA3) and Ceramage Opaque dentin (ODA3) and Ceramage Body (A3B) were filled in the ring, and a clear glass plate was put on it and irradiated for two minutes. The polycarbonate ring was then carefully removed, and the Estenia C&B groups were heat-polymerized using the same method.

For Groups 9 and 10, specimens with two opaque layers were fabricated as follows (Figure 3): a piece of tape with a circular hole of 4.5 mm in diameter and 0.15 mm in thickness was fixed on the surface of each zirconia specimen. AZ primer was applied for 30 seconds and allowed to dry with oil-free air for 3 seconds. The first opaque resin paste (Ceramage Pre-opaque) was then filled and irradiated with light for two minutes. The second tape, which was the same as the first one, was then fixed on the first tape. The second opaque resin (Ceramage Opaque: A3O and Estenia C&B Opaque resin: OA3) was filled and irradiated for two minutes. After that, two layers of tape were removed, and the same polycarbonate ring with a circular hole of 4.5 mm in diameter and 0.8 mm in thickness was fixed on the opaque layer. Ceramage Body (A3B) and Estenia C&B Body resin (DA3) were filled in the ring, and a clear glass plate was put on it and then irradiated for two minutes. The polycarbonate ring was then carefully removed, and the Estenia C&B groups were heat-polymerized using the same method.

After completion of this process, all specimens were immersed in distilled water at 37°C for 24 hours. The bond strength for each group of 5 specimens was measured, and the remaining 5 specimens were then subjected to thermocycling for 4,000 cycles between 4 and 60°C in each bath. The dwelling time at each temperature was 60 seconds. The shear bond strength was measured to evaluate the durability of the bond.

The specimens were mounted in the jig of a universal testing machine (Autograph AG-I 100kN, Shimadzu, Kyoto, Japan), and shear force was applied to the adhesive interface until failure occurred at a cross-head speed of 1mm/minute (Figure 4). The maximum force to fail was recorded as the bond strength. The failure surfaces were examined under a light microscope to classify the failure mode of the debonded area into adhesive, cohesive, or a combination.

The mean values of each group were statistically analyzed using two-way analysis of variance (ANOVA) to identify the significant differences among surface treatment procedures and before and after thermocycling of each surface treatment procedure. If differences were found, Games-Howellanalysis was used to evaluate significant differences. Allanalyses were conducted at a 95 % confidence level. The software used was SPSS 15.0J (SPSS Inc., Chicago, IL, USA).

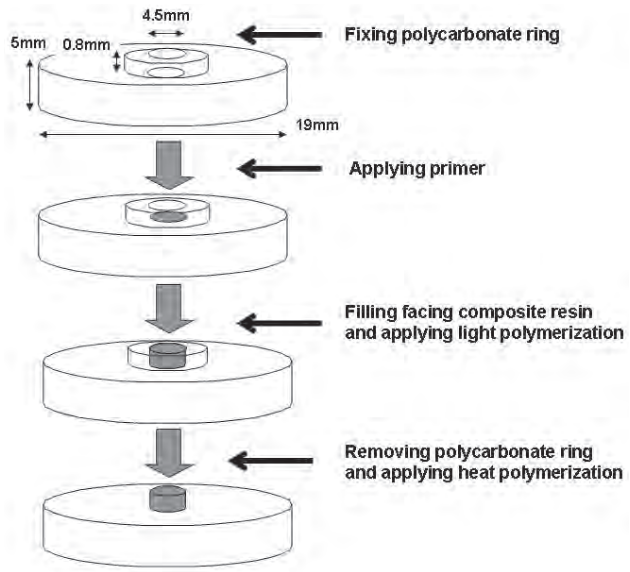


**Figure 1.** *Treatment procedures for specimens without an opaque layer*

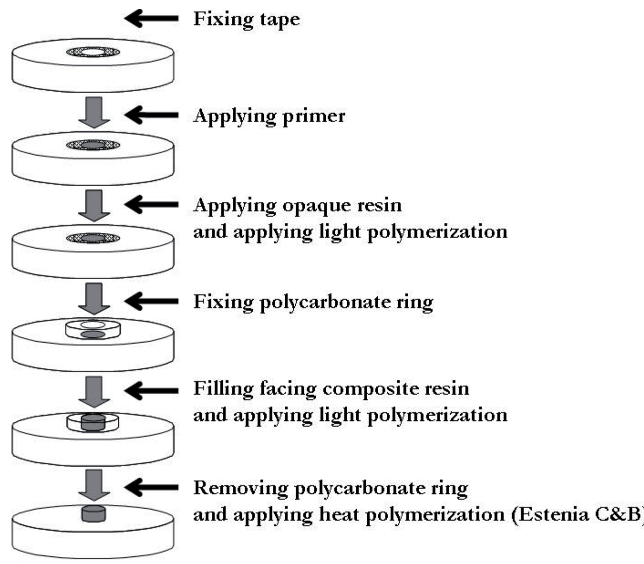


**Figure 2.** *Treatment procedures for specimens with one layer of opaque resin*

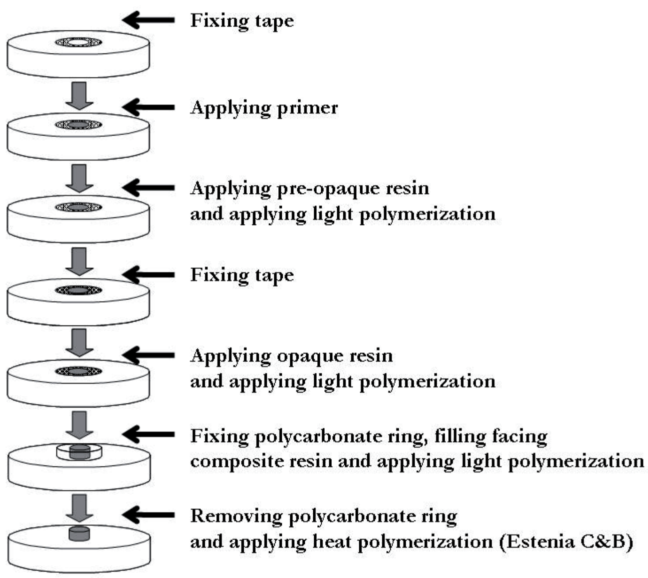


**Figure 3.** *Treatment procedures for specimens with two layers of opaque resins*

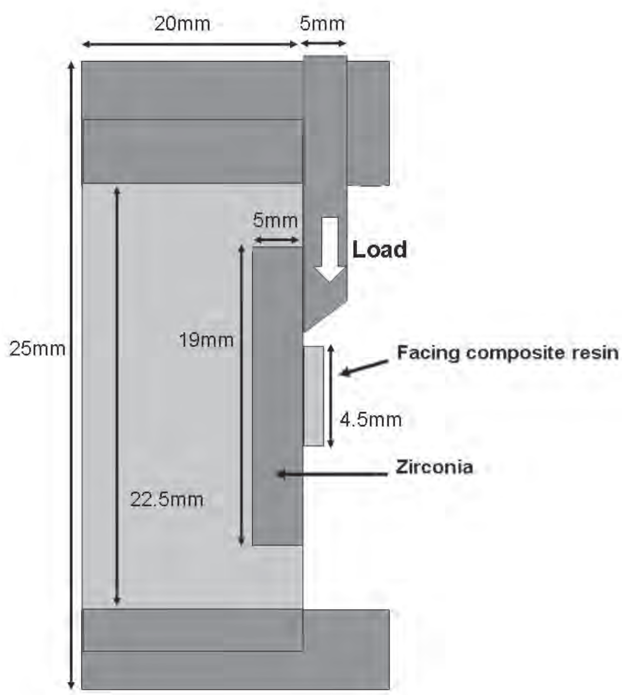


**Figure 4.** *Schematic drawing showing the shear bond strength test in this study*

## RESULTS

The mean bond strengths of each group before and after thermocycling are shown in Figure 5, and results of a multi comparison test are shown in Table 3. The highest mean bond strength was 21.4 MPa for Group 10 before thermocycling, and the lowest one was 3.1 MPa for Group 4 before thermocycling. The bond strength of Group 7 decreased in a statistically significant manner after thermocycling.

The difference in translucency between body and opaque resins did not affect the bond strength before and after thermocycling (Groups 2 and 3 and Groups 6 and 7, respectively).

Among 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer-containing primers (Groups 1 – 5), when using opaque resin (Group 5), the bond strength was statistically significantly high before thermocycling and statistically significantly high after thermocycling in Groups 1, 2, and 4 but not in Group 3.

In a comparison among groups to which the AZ primer had been applied, build-up Estenia C&B showed statistically significantly higher bond strength than Ceramage after thermocycling (Groups 6 and 8). The application of the AZ primer and build-up Ceramage showed more than 10 MPa of bond strength (Groups 6, 7, and 9); however, after

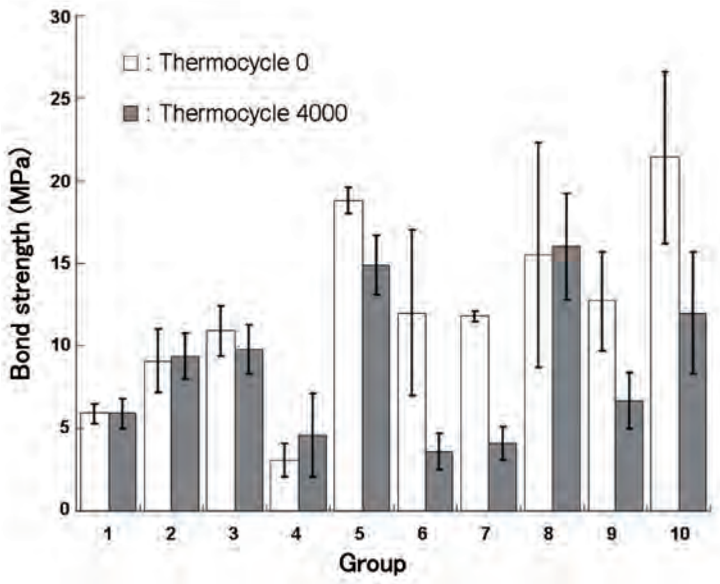


**Figure 5.** *Bond strengths of each group before and after thermocycling*

**Table 3.** *Results of a multi-comparison test of groups 1 to 10 before (Thermocycle 0) and after thermocycling (Thermocycle 4000)*

| | | Thermocycle 0 | | | | | | | | | | Thermocycle 4000 | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| Thermocycle 0 | 1 | | | | | | | | | | | | | | | | | | | | |
| | 2 | | | | | | | | | | | | | | | | | | | | |
| | 3 | * | | | | | | | | | | | | | | | | | | | |
| | 4 | * | * | * | | | | | | | | | | | | | | | | | |
| | 5 | * | * | * | * | | | | | | | | | | | | | | | | |
| | 6 | | | | | | | | | | | | | | | | | | | | |
| | 7 | * | | | * | * | | | | | | | | | | | | | | | |
| | 8 | | | | | | | | | | | | | | | | | | | | |
| | 9 | | | | * | | | | | | | | | | | | | | | | |
| | 10 | * | | | * | | | | | | | | | | | | | | | | |
| Thermocycle 4000 | 1 | | | * | | * | | * | | | * | | | | | | | | | | |
| | 2 | | | | * | * | | | | | | | | | | | | | | | |
| | 3 | | | | * | * | | | | | | | | | | | | | | | |
| | 4 | | | | | * | | | | | * | | | | | | | | | | |
| | 5 | * | | | * | | | | | | | * | * | | * | | | | | | |
| | 6 | | * | * | | * | | * | | * | * | | * | * | | * | | | | | |
| | 7 | | | * | | * | | * | | * | * | | * | * | | * | | | | | |
| | 8 | * | | | * | | | | | | | * | | | * | | * | * | | | |
| | 9 | | | | | * | | * | | | * | | | | | * | | | * | | |
| | 10 | | | | | | | | | | | | | | | | | | | | |

＊: $p$>0.05

thermocycling, the bond strength of Group 7 decreased in a statistically significant manner.

For the failure mode, all specimens of Groups 1 to 5 experienced adhesive failures before thermocycling; after thermocycling, they did so as well, except for 3 specimens in Group 2, which showed a mixed failure. Of the remainder, Groups 6 to 10, to which the AZ primer had been applied, showed adhesive failure between the zirconia surface and the pre-opaque layer before and after thermocycling. Two specimens, Groups 6 and 8, showed cohesive failure of the pre-opaque resin before thermocycling.

## DISCUSSION

The introduction of composite resin facing zirconia crowns instead of conventional resin facing cast crowns, porcelain-fused-to-metal crowns, and all-ceramic crowns might have many advantages for the restoration of natural teeth and implants, such as biocompatibility, fabrication, and repair. When selecting porcelain as a veneering material, porcelain chipping or fracture sometimes occurs after functioning. From these clinical points of view, composite resins, used instead of porcelain, have the potential to avoid such troubles. Furthermore, in the case of chipping, a repair might be easier than that with porcelain. On the other hand, composite resin has inherent disadvantages compared to porcelain; colour change owing to aging, biocompatibility to the soft tissue, and abrasion after functioning. To overcome and confirm these disadvantages, long-term clinical evaluation must be needed as soon as possible.

The effects of conventional surface treatments on the bond strength of a zirconia coping were evaluated as a first step in this study.

Clearfil ceramic primer is a primer for alumina and zirconia crown surfaces that contains a 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) monomer. This method was the easiest pre-treatment before cementation; however, the bond strength was limited (Group 1). According to Kern *et al.*[18], Clearfil ceramic primer showed higher and durable bond strength compared with MDP-none containing primer.

When comparing Groups 2 and 3, the effects of shade differences on the bond strength were evaluated, and the results showed that there were no differences between them. In addition, Groups 2 and 3, to which the Modelling liquid was applied, showed durability; the differences from the ceramic primer might depend on the amount of the MDP monomer in the primer. Thereby, the bond strengths were different with each primer. The Modelling liquid has been generally used for separating a facing composite resin from a spatula; however, this time, it was used as primer.

In contrast, the Opaque primer, which also contains the MDP monomer, lacked sufficient bond strength. However, the application of the Opaque primer and then an opaque resin yielded a strength that was 6 times higher before thermocycling and 3 times higher after thermocycling. This could be the reason that the opaque resin has low viscosity and compatibility with the Opaque primer and that curing of the Opaque primer was accelerated; however, the body resin has high viscosity, which made it more difficult to cure quickly at the interface. Furthermore, the uncured layer of an opaque resin might be compatible with body resin, resulting in a firm bond between the two resins. This means that the opaque resin might act as a binding agent for opaque and body resins. Support for this idea is given by the mode in which most failures occur between zirconia and the pre-opaque interface.

Wolfart *et al.*[19] recommended the use of the MDP-containing composite resin Panavia F on air-abraded zirconia ceramic. This partially agrees with the results obtained here, which demonstrate that, after sandblasting a zirconia surface and applying a primer containing MDP, the use of high-flow resins (opaque resins) would be useful for coping facing. Kern *et al.*[20] demonstrated that the bond strengths of MDP containing resin composites, when compared to those with only a sandblasted zirconia surface, were higher than those of conventional BisGMA resin cement, and they concluded that MDP promotes a water-resistant bond to zirconia, as has been demonstrated for glass-infiltrated alumina[21,22]. Lehmann *et al.*[23] recommended MDP-containing primers on air-abraded zirconia ceramic to improve bonding to zrconia ceramics. Furthermore, Blatz *et al.*[24] showed the same results as Kern *et al.*[18] when using Procera AllZircon specimens. The difference between resin cement and facing composite is the viscosity of the resin; on the other hand, the facing composite has opaque resin to mask the coping colour. From this study, the application of primer and opaque resin was an effective way to obtain durable bond strength between zirconia and facing composite resin.

When two opaque layers were applied, Estenia C&B also showed higher bond strength, which also might depend on the mechanical strength of the body resin.

Comparing the MDP-containing primer with the AZ primer, the AZ primer might have shown a tendency to decrease in bond strength after aging, as occurs with thermocycling. When an AZ primer was applied, Estenia C&B showed higher bond strength than Ceramage before and after thermocycling. The bond strengths obtained from this study were affected by the composite resin itself. When the mechanical strength was higher, the deformation remained, and, as a result, the bond strength was higher. Furthermore, the composite resin used in this study, Ceramage, is polymerized by a light source alone. On the other hand, Estenia C&B is a light- and heat-polymerized resin. In general, heat-polymerized composite resin shows high mechanical strength because of its high degree of polymerization.

Following the manufacturers' instructions, the compressive and bending strengths of Estenia C&B and Ceramage were measured to be 595 and 354 MPa and 190 and 146 MPa, respectively. The both strengths of Estenia C&B were higher than those of Ceramage.

In this study, the effect of sandblasting on the bond strength was not evaluated, and all specimens were sandblasted prior to testing. It is well known that air abrasion with an $Al_2O_3$ particle is a common method to strengthen the surface[19,20,25-27]. In addition, acid etching with solutions of hydrofluoric acid can achieve proper surface texture and roughness.

Sandblasting is an easy and effective method to achieve micromechanical retention on the ceramic surface. On an alumina ceramic surface, a 50-µm $Al_20_3$ particle was used as described in other studies[28,29], and a chemical bond was gained by some special monomers. Some researchers, fearing microcracks, are skeptical of the strength of a material after sandblasting[30]. Kern *et al.*[24] reported that to minimize possible surface damage effects as a result of air-abrasion at a relative high pressure, at lower pressures (0.05 MPa) of air-abrasion and priming with adequate adhesive monomer appears to be effective methods for forming durable bonds between resin composite and zirconia ceramics.

There was a limitation of this study, and could not include other influential parameters, further studies will be needed to examine the best treatment conditions, such as sandblasting or acid etching, for each zirconia.

From a clinical point of view, the bond strength between a zirconia coping and facing composite resin is very important. If the strength is insufficient, microleakage from the margin and facing resin fracture from the coping will occur. To avoid these problems, microretention and a chemical bond will be needed. Microretention will be achieved with sandblasting. As for silica coating, Bona *et al.*[31] compared the bond strength of a glass-infiltrated alumina-based zirconia-reinforced ceramic (Vita In-Ceram zirconia) to a composite resin and concluded that silica coating produced higher values than hydrofluoric acid etching and sandblasting. This may be explained by the fact that silane was bonded to the silica present on the ceramic surface.

In this study, silica coating of a zirconia surface was not conducted. According to Uo *et al.*[12], silica coating did not improve the bond strength. Further research is needed to determine the effect of silica coating on the bond strength of facing composite resin.

The results of this study indicate that different zirconia surface treatments affected the bond strengths not only initially but also after thermocycling. If the primer contains some MDP monomer, it will act as a chemical binder on the zirconia surface. Chemical bonds and micromechanical interlocking would support zirconia facing composite resin surface bonding and its durability. Kern *et al.*[24] evaluated the tensile bond strengths after water storage for 150 days with additional 37,500 thermocycling. As this is a first step, and to evaluate further longevity of the bonding, a long-term thermocycling test should be conducted after selected surface treatments.

A pilot study was conducted on the bond strength between zirconia and cylindrical facing resin composite (Estenia C&B Body resin, ODA3). The sandblasted zirconia surface was etched, the Clearfil ceramic primer was applied to it, and a cylindrical facing resin composite (4.5 mm in diameter and 0.8 mm in thickness) was cemented with Panavia F2.0 (Kuraray Medical, Tokyo, Japan). The bond strengths, initially and after thermocycling, were 10.2 MPa (SD:1.1 MPa) and 9.2 MPa (SD:0.8 MPa), respectively, and there were no significant differences between them. On the basis of these findings, within this study, at least 9.2 MPa shear bond strength might be needed even after thermocycling.

The hypothesis of this study was proved, namely, that the application of a primer, opaque resin, and Estenia C&B provided stronger bond strength even after thermocycling.

Further studies will be required to determine the thickness of facing resin that is necessary to avoid fracture after functioning and discoloration from the cervical area. Bonding to zirconia was conducted in laboratory tests; however, no clinical studies have been conducted. Controlled clinical trials will be required to confirm the finding in this *in vitro* study.

## CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions may be drawn:

The bond strengths of facing composite resins to zirconia after different treatments varied depending on the primers, opaque resins, body resins, and thermocycling.

Without opaque resin, the application of Estenia C&B Modeling liquid and build-up Estenia C&B Body resin resulted in the strongest bond strength before and after thermocycling.

With one layer of opaque resin, the application of the Estenia C&B Opaque primer, Estenia C&B Opaque resin, and build-up Estenia C&B Body resin yielded stronger bond strength than that achieved without the application of the Estenia C&B Opaque primer.

With two layers of opaque resins, both facing composite resins showed relatively strong initial bond strengths; however, after thermocycling, the bond strength of the Ceramage group decreased remarkably.

The application of various primers and opaque resins to the zirconia surface after sandblasting is expected to yield strong bond strength of the facing composite resin even after thermocycling.

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