

Effects of surface treatments on  
shear bond strength of composite resin  
bonded to various ceramic cores  
after thermocycling

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This certifies that the dissertation of  
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## Abstract

### Effects of surface treatments on shear bond strength of composite resin bonded to various ceramic cores after thermocycling

As the popularity of high-strength all-ceramic restorations increase rapidly, studies and clinical guidelines on repair methods for the fractured veneer and core have become a necessity.

The purpose of this study was to evaluate the shear bond strength of composite resin bonded to various high-strength all-ceramic core materials with different surface treatments with and without thermocycling. The hypothesis was that the tribochemical silica coating would be effective in bonding composite resin to high-strength ceramic cores, especially in long term, tested by thermocycling.

Sixty ceramic blocks for each of three ceramic cores(IPS Empress 2, In-Ceram Alumina, Zi-Ceram) and feldspathic ceramic(Duceram Plus) were fabricated and embedded in self curing acrylic resin. Ceramic specimens in each material were randomly divided into three groups for different surface treatments(airborne particle abrasion, acid etching, and tribochemical silica coating). Silane and bonding resin was applied on all 240 specimen surfaces and composite resin was light-cured onto these surfaces. For each surface treatment, 20 specimens were randomly divided into two subgroups, 10 specimens stored in a desiccator at room temperature for 24 hours and the other 10 specimens were thermocycled between 5 °C and 55 °C for 1200 cycles with dwell time of 30 seconds. Shear bond strength was measured using



universal testing machine(Instron 3366, Instron Co., Canton, MA, U.S.A.) with cross head speed of 2.0 mm/min. Shear bond strength values were statistically analyzed with two-way analysis of variance(ANOVA) and Duncan multiple comparison test( $\alpha=0.05$ ).

Regardless of the ceramic material, the highest shear bond strength was produced by silica-coated groups(Dro, Ero, Iro, Zro). After thermocycling, the mean shear bond strength decreased in most of the groups.

From the result of this study, it was concluded that tribochemical silica coating should be recommended for lithium disilicate ceramic or alumina ceramic core. Although further investigation is needed to prove the effectiveness of tribochemical silica coating on zirconia ceramic, this method is more reliable compared to acid etching or airborne-particle abrasion.

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Key words : all ceramic restoration, intraoral repair, tribochemical silica coating, shear bond strength

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## I . Introduction

Recent development in strength and esthetics of high-strength all-ceramic materials has made the metal-free restorations an often-used alternative for metal-ceramic restorations. Since these high-strength all-ceramic materials such as alumina ceramic and zirconia ceramic provide the core part of the veneered all-ceramic restorations, they cannot prevent the fracture of the veneer or the fracture of the core-veneer interface. Previous studies have reported the frequent fractures at the core-veneer interface of layered

all-ceramic fixed partial denture(Kelly *et al.*, 1995; Luthardt *et al.*, 1999; Aboushelib *et al.*, 2005).

Concerning the incidence of the fractures of all-ceramic restoration, there was an in vitro study reporting that the bond of veneering porcelain to a ceramic core is similar to that of the metal ceramic restoration, suggesting that the clinical behavior would be similar(Al-Dohan *et al.*, 2004). Goodacre *et al.*(2003) reported the incidence of the porcelain veneer fracture among the single crown complications to be mean rate of 3%. One study reported porcelain fracture as the second most likely cause for the replacement of restorations following dental caries(Walton *et al.*, 1986). Expecting similar incidence of veneer failure for all-ceramic restorations, studies and clinical guidelines on repair methods for the fractured veneer and core are mandatory.

Repairing the fractured veneer with composite resin is often better than replacing the complete restoration in terms of time and cost(Ozcan, 2003). Intraoral porcelain repair systems for fractured veneering ceramic rely on resin bond strength and methods for creating microretentive surfaces(Latta and Barkmeier, 2000). In cases where the fracture involves the feldspathic porcelain veneer only, bonding is a predictable procedure yielding durable results with commercially available products, mostly involving hydrofluoric acid etching and application of a silane coupling agent(Blatz *et al.*, 2003; Lacy *et al.*, 1988; Denehy *et al.*, 1998). Roughening of ceramic materials by airborne particle abrasion has also been used as a substitute for etching, but detrimental effect on the surfaces of feldspathic porcelain after sandblasting have been reported (Kern and Thompson, 1994; Llobell *et al.*, 1992).

Among various high strength all-ceramic core materials, lithium-disilicate core material(IPS Empress 2, Ivoclar-Vivadent, Schaan, Liechtenstein) demonstrates relatively low flexural strength, but its high translucency kept its popular usage in anterior regions of dentition(Raigrodski, 2004). Since lithium

disilicate glass ceramic can be etched with hydrofluoric acid, its surface treatment for repair does not differ from the procedures for veneers(Della Bona *et al.*, 2000). For high-strength alumina- or zirconia-based ceramics, acid etchants do not provide sufficient micro-irregularity due to low silica content. Alternative roughening procedures have been investigated to enhance microretention of composite resin to these core materials. Airborne particle abrasion with  $\text{Al}_2\text{O}_3$  is proven to be more effective than acid etching for roughening aluminum-oxide ceramic surfaces(Kern and Thompson, 1994). Kern and Thompson(1994) reported that silica coating procedure by Rocatec<sup>TM</sup> system(ESPE, Seefeld, Germany) promotes chemical bond between the alumina ceramic surface and the applied resin. In this system, silicic acid-modified alumina particles are blasted with high energy onto the ceramic surface. The high temperature created by the impact causes components of the blasting abrasive to be incorporated into the surface. This tribochemically coated surface provides not only micromechanical retention but also sites for chemical adhesion(Sun *et al.*, 2000). For zirconium-oxide ceramics, Kim *et al.*(2005) reported that the tribochemical silica coating technique was more effective than other treatments such as airborne particle abrasion and acid etching in terms of tensile bond strength.

Concerning long term durability of ceramic-composite bond, several studies on porcelain repair systems reported decreased bond strength values after thermocycling or long-term water storage(Leibrock *et al.*, 1999; Llobell *et al.*, 1992). But publications on the influence of aging on bonding composite resin to aluminum-oxide and zirconium-oxide ceramic core are very limited.

The purpose of this study was to evaluate the shear bond strength of composite resin bonded to various high-strength all-ceramic core materials with three different surface treatments(airborne particle abrasion, acid etching, and tribochemical silica coating) with and without thermocycling. The

hypothesis was that the tribochemical silica coating would be effective in bonding composite resin to high-strength ceramic cores, especially in long term, tested by thermocycling.

## II. Materials and Methods

Sixty ceramic blocks( $10 \times 10 \times 2$  mm) for each of three ceramic cores(IPS Empress 2, In-Ceram Alumina, Zi-Ceram) and feldspathic ceramic(Duceram Plus) as control, were fabricated(Table I).

All specimens were fabricated according to manufacturers' instructions as follows.

For fabricating IPS Empress 2 specimens, IPS Empress 2 wax patterns(S-U-Dental wax, Schuler Dental, Ulm, Germany) with the specimen size were prepared and invested in IPS Empress 2 Speed-investment. The wax was eliminated in a burnout furnace(EP 500; Ivoclar-Vivadent) and the IPS Empress 2 ingots(shade A2) were automatically pressed into the mold in the furnace at  $1150$  °C. After pressing and cooling to room temperature, the specimens were divested.

Table I. Ceramic materials tested

| Product name     | Ceramic material           | Manufacturer                            | Composition  |
|------------------|----------------------------|---|--|
| Duceram Plus     | Feldspathic ceramic        | Ducera Dental GmbH, Rosbach, Germany    | SiO <sub>2</sub> 60% Al <sub>2</sub> O <sub>3</sub> 20% Na <sub>2</sub> O K <sub>2</sub> O B <sub>2</sub> O <sub>3</sub> ZnO |
| IPS Empress 2    | Lithium-disilicate ceramic | Ivoclar Vivadent, Schaan, Liechtenstein | SiO <sub>2</sub> 60% Li <sub>2</sub> O 15% K <sub>2</sub> O P <sub>2</sub> O <sub>5</sub>                                    |
| In-Ceram Alumina | Alumina ceramic            | Vita Zahnfabrik, Bad Säckingen, Germany | Al <sub>2</sub> O <sub>3</sub> 85% La <sub>2</sub> O <sub>3</sub> SiO <sub>2</sub> CaO                                       |
| Zi-Ceram         | Zirconia ceramic           | Adens, Seoul, Korea                     | ZrO <sub>2</sub> (Y-stabilized) Coloring oxides (<1wt %)   |

For In-Ceram Alumina specimens, the aluminum oxide powder were mixed with a special liquid(Vita In-Ceram Alumina mixing liquid; Vita Zahnfabrik, Bad Säckingen, Germany), and ultrasonicated(Vitasonic II; Vita Zahnfabrik, Bad Säckingen, Germany) for 7 minutes. After shaping the slurry mixture to fit the specimen size, it was fired at 1120 °C in the oven(Inceramat II; Vita Zahnfabrik, Bad Säckingen, Germany) for 10 hours. Glass was infiltrated by coating the aluminum oxide framework with a glass powder(silicate-aluminum-lanthanum)-distilled water mixture and firing in the furnace for 4 hours at 1100 °C.

Zirconium-oxide ceramic specimens were fabricated with zirconia powder(Tosoh Co., Tokyo, Japan) mixed with small quantity of Fe<sub>2</sub>O<sub>3</sub> powder. The mixture was ground to obtain particles that are homeogenous in size and pressed to a block. Pressure of 160 Mpa was applied in vacuum environment and it was sintered at temperature of 850 °C to 1200 °C. After processing it to the shape of specimens, it was re-sintered at 1500 °C.

The feldspathic ceramic specimens were fabricated using a mold created with putty type vinyl polysiloxane(Exafine, GC Co., Tokyo, Japan). Ceramic powder(Duceram Plus; shade DA2; Ducera Dental GmbH, Rosbach, Germany) and liquid(Ducera Liquid; DeguDent GmbH, Hanau-Wolfgang, Germany) were condensed in the space inside the mold and the condensed specimens were fired at 910 °C.

Total of 240 ceramic blocks were embedded in self curing acrylic resin(Orthodontic resin; Dentsply Caulk, Milford, DE, U.S.A.), exposing one ceramic surface for surface treatments(Fig. 1). The ceramic surfaces were polished with 100 grit and 600 grit silicon carbide paper consecutively under water-cooling.

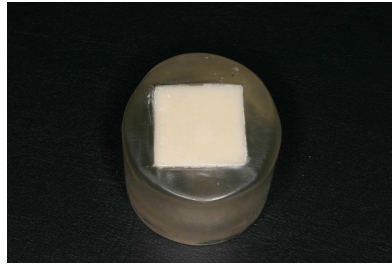


Fig. 1. Test specimen : ceramic block embedded in acrylic resin.

Table II. Summary of surface treatments on ceramic materials

| Ceramics tested  | Group | Surface treatment   |
|------------------|-------|---|
| Duceram Plus     | Dab   | Airborne-particle abrasion                                |
|                  | Dae   | Airborne-particle abrasion / Acid etching                 |
|                  | Dro   | Airborne-particle abrasion / Tribochemical silica coating |
| IPS Empress 2    | Eab   | Airborne-particle abrasion                                |
|                  | Eae   | Airborne-particle abrasion / Acid etching                 |
|                  | Ero   | Airborne-particle abrasion / Tribochemical silica coating |
| In-Ceram Alumina | Iab   | Airborne-particle abrasion                                |
|                  | Iae   | Airborne-particle abrasion / Acid etching                 |
|                  | Iro   | Airborne-particle abrasion / Tribochemical silica coating |
| Zi-Ceram         | Zab   | Airborne-particle abrasion                                |
|                  | Zae   | Airborne-particle abrasion / Acid etching                 |
|                  | Zro   | Airborne-particle abrasion / Tribochemical silica coating |

Sixty samples of each ceramic material were randomly divided into three groups for three different surface treatments (Table II). Specimens for airborne-particle abrasion (ab) group were sandblasted using Microetcher<sup>TM</sup> (Danville Engineering, San Ramon, CA, U.S.A.) with 50  $\mu\text{m}$  grit



alumina particle for 5 seconds at a 10 mm distance with pressure of 40 psi, with the tip perpendicular to the surfaces. Remaining particles on the surface were removed with air/water spray for 10 seconds. Acid etching(ae) group specimens were also sandblasted identically as ab group. Then 4% hydrofluoric acid(Porcelain Etchant; Bisco Inc., Schaumburg, IL, U.S.A.) was applied for 5 minutes and washed with air/water spray for 15 seconds. Rocatec<sup>TM</sup> system treated(ro) groups were sandblasted in the same manner and silicic acid-modified 30  $\mu$ m alumina particle(Rocatec Soft; 3M ESPE, Seefeld, Germany) was applied for 5 seconds with pressure of 40 psi at a distance of 10 mm, perpendicular to the surfaces. Remnant particles were removed by gentle stream of compressed oil-free air. Silane(ESPE Sil; 3M ESPE, Seefeld, Germany) was applied on all 240 specimen surfaces and was left to dry in air for 5 minutes. Bonding resin(One-Step; Bisco Inc., Schaumburg, IL, U.S.A.) was applied on these silane coated surfaces and light-cured for 20 seconds using light curing unit(Elipar<sup>TM</sup> Freelight2 LED Curing Light; 3M ESPE, Seefeld, Germany).

Each specimen was placed on a specially designed holder and a plastic mold with cylindrical hole(2.39 mm in diameter and 3 mm in height) was located on top of the treated surface(Fig. 2). Composite resin(Z100 Restorative; Shade A1; 3M ESPE, St Paul, MN, U.S.A.) was packed into the cylindrical hole incrementally. The first increment was 2 mm in height and the second increment was applied to fill the top of the cylinder. Each increment was light cured with light curing unit(Elipar<sup>TM</sup> Freelight2 LED Curing Light; 3M ESPE, Seefeld, Germany) for 40 seconds. The intensity of the light(650 mW/cm<sup>2</sup>) was checked periodically with Coltolux Light Meter<sup>TM</sup>(Coltene Whaledent Inc., Mahwah, NJ, U.S.A.). Specimens were removed from the holder and the mold.

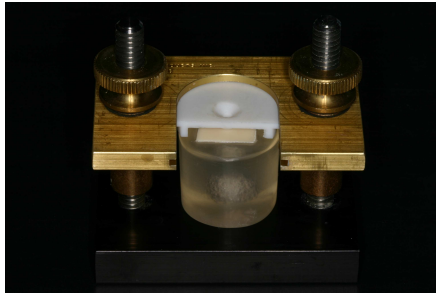


Fig. 2. Specimen placed on a specially designed holder, plastic mold located on top.

For each surface treatment, 20 specimens were randomly divided into two subgroups, 10 specimens stored in a desiccator at room temperature for 24 hours and the other 10 specimens were thermocycled between 5 °C and 55 °C for 1200 cycles with dwell time of 30 seconds. Shear bond strength was measured using universal testing machine(Instron 3366, Instron Co., Canton, MA, U.S.A.) with cross head speed of 2.0 mm/min(Fig. 3).

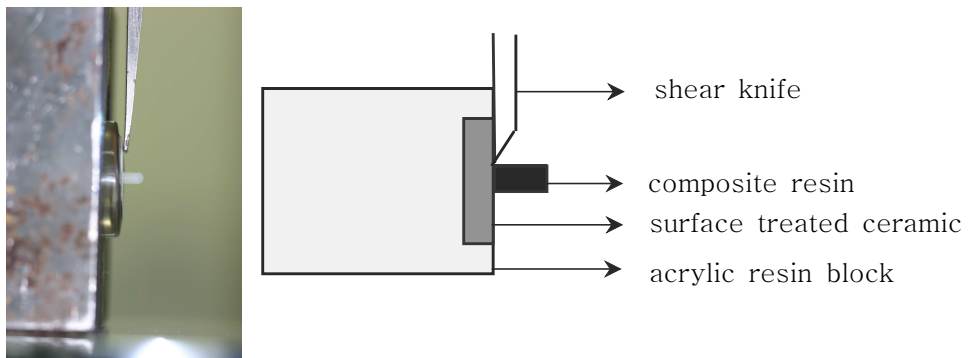


Fig. 3. Specimen positioned on universal testing machine for shear bond strength measurement.

Statistical analysis was carried out using SPSS 12.0 for Windows (SPSS Inc., Chicago, IL, U.S.A.). Shear bond strength values were examined with two-way analysis of variance (ANOVA) and Duncan multiple comparison test ( $\alpha = 0.05$ ). One-way ANOVA, followed by LSD (least significant difference) post hoc test was applied to determine significant differences among the values within each ceramic material groups in dry condition and after thermocycling ( $\alpha = 0.05$ ).

### III. Results

The results of the shear bond test are shown in Table III and figure 4. Regardless of the ceramic material, the highest shear bond strength was produced by Rocatec treated groups(Dro, Ero, Iro, Zro).

Table III. The means and standard deviations of shear bond strengths(MPa) of composite resin to the ceramic materials for three surface treatments in dry/thermocycled condition(n=10), with statistical comparison using one-way ANOVA and LSD post hoc test

| Group | Dry-conditioned |     |                         | Thermocycled |     |                         |
|-------|-----------------|-----|-------------------------|--------------|-----|-------------------------|
|       | Mean            | SD  | *statistical comparison | mean         | SD  | *statistical comparison |
| Dab   | 22.2            | 5.3 | a                       | 16.9         | 3.9 | a                       |
| Dae   | 26.0            | 6.3 | a                       | 19.1         | 5.6 | a                       |
| Dro   | 26.9            | 7.4 | a                       | 21.7         | 6.0 | a                       |
| Eab   | 21.5            | 4.2 | a                       | 14.0         | 3.0 | a                       |
| Eae   | 21.2            | 4.5 | a                       | 16.6         | 4.4 | a                       |
| Ero   | 26.4            | 5.0 | b                       | 23.9         | 5.5 | b                       |
| Iab   | 22.6            | 4.5 | a b                     | 19.0         | 4.4 | a                       |
| Iae   | 20.7            | 4.4 | a                       | 15.2         | 4.6 | a                       |
| Iro   | 26.7            | 4.0 | b                       | 25.9         | 3.0 | b                       |
| Zab   | 20.2            | 4.0 | a                       | 14.2         | 4.7 | a b                     |
| Zae   | 24.3            | 5.2 | a b                     | 10.2         | 3.7 | a                       |
| Zro   | 25.4            | 3.8 | b                       | 17.3         | 5.9 | b                       |

\*Identical letters denote no significant differences among surface treatments in each ceramic material group( $p > 0.05$ ).

Thermocycling had adverse effect on the mean shear bond strength in most of the groups, but the decrease was statistically insignificant in Iro and Ero groups (Table III, figure 4).

Statistical analysis in Table IV showed that the ceramic material, surface treatment, and thermocycling had a statistically significant effect on the shear bond strength ( $p < 0.001$ ). As ceramic+surface combination is shown to have statistically significant effect on shear bond strength ( $p = 0.04$ ), one-way ANOVA, followed by LSD post hoc test was applied to determine differences in the shear bond strength between the surface treatments in each ceramic material (Table III).

Table IV. Statistical analysis of the results of the shear bond strength test by two-way analysis of variance (ANOVA)

| Source                        | df | Sum of square | Mean square | F      | p    |
|-------------------------------|----|---------------|-------------|--------|------|
| Ceramic                       | 3  | 444.936       | 148.312     | 6.240  | .000 |
| Surface                       | 2  | 1503.720      | 751.860     | 31.633 | .000 |
| Thermocycling                 | 1  | 2063.835      | 2063.835    | 86.833 | .000 |
| Ceramic + Surface             | 6  | 320.985       | 53.497      | 2.251  | .040 |
| Ceramic + Thermocycling       | 3  | 296.781       | 98.927      | 4.162  | .007 |
| Surface + Thermocycling       | 2  | 136.657       | 68.328      | 2.875  | .059 |
| Ceramic+Surface+Thermocycling | 6  | 167.887       | 276.981     | 1.177  | .320 |

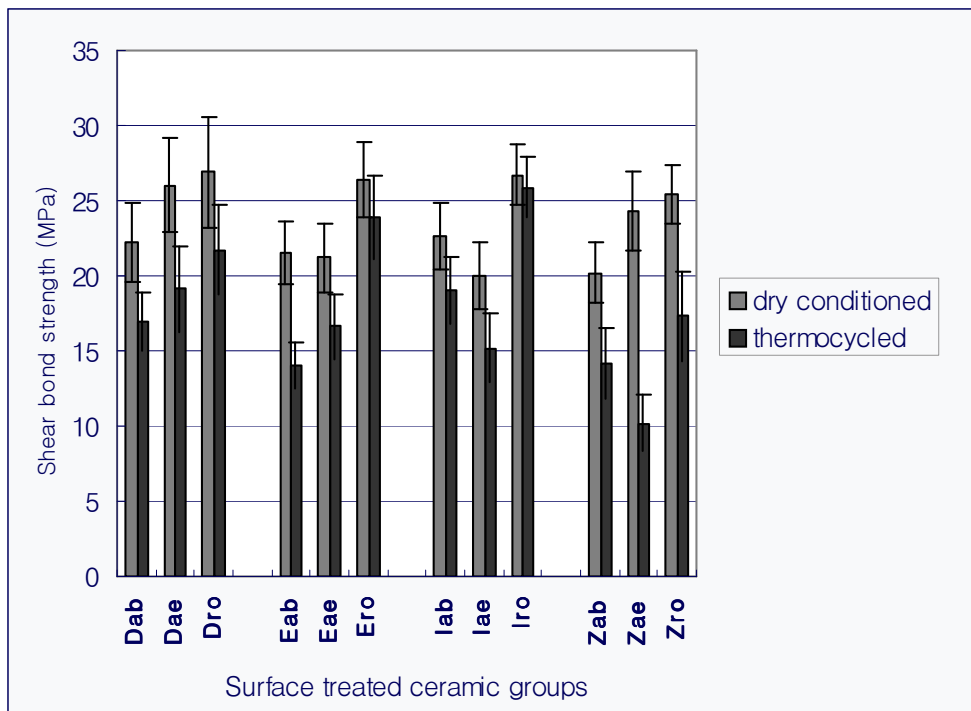


Fig. 4. Shear bond strength for combination of ceramic and surface preparation in dry and thermocycled condition.

## IV. Discussion

In conventional intraoral repair systems for feldspathic ceramic, hydrofluoric acid is applied to dissolve the glass matrix selectively, thus creating physical alteration to promote adhesion of composite resin (Calamia *et al.* 1985; Sheth, *et al.*, 1988; Thurmond *et al.*, 1994). Airborne particle abrasion is another method for roughening surfaces, especially in cases of porcelain fracture that involves metal or ceramic core exposure that is resistant to acid etching (Schneider *et al.* 1992; Lacy *et al.*, 1988; Thurmond *et al.*, 1994).

In this study, feldspathic ceramic was used as control for comparing shear bond strength, since sufficient data regarding bonding methods to feldspathic ceramic has been published and clinical recommendation is given from the previous porcelain repair studies (Kupiec, 1996; Latta, 2000). For feldspathic ceramics, the shear bond strength values among conventional acid etching and airborne particle abrasion and tribochemical silica coating method (Dab, Dae, Dro) did not show significant differences with and without thermocycling, which denoted the validity of these conventional surface treatment methods.

The composition and physical properties of high-strength ceramic materials, such as alumina ceramics and zirconia ceramics, differ substantially from silica-based ceramics and require alternative bonding techniques to achieve a strong and durable resin bond.

The In-Ceram system, which uses high-temperature sintered-alumina glass-infiltrated copings for all-ceramic crown, has flexural strength of 450 MPa (Sehgi and Sorenson, 1995). Alumina ( $\text{Al}_2\text{O}_3$ ) represents 85% by weight of alumina ceramic. It is infiltrated by lanthanum-aluminum-silicate glass containing less than 5% of silica by weight. As the silica phase is the only phase able to be etched by hydrofluoric acid, the etching is considered to be

inefficient(Borges, 2003). Concerning the use of airborne particle abrasion, Borges *et al.* reported that airborne particle abrasion with 50 µm aluminium oxide on the In-Ceram Alumina did not change their morphologic microstructure on SEM. This resistance to airborne particle abrasion seems to be related to the surface hardness of alumina ceramic, since reported hardness of In-Ceram Alumina(9.82 GPa) is significantly higher than that of feldspathic ceramic(5.42 GPa; Seghi *et al.*, 1995). In the study by Kern and Thompson(1994), airborne abrasion with 110 µm-grit particle(Rocatec Pre only) was shown to create microretentive surfaces for In-Ceram ceramic, but the application of the tribochemical silica coating was recommended for creating chemical bond to composite resin. In accordance with the previous studies, the shear bond strength value of Iae was lower than that of Iro in dry condition. After thermocycling, Iab and Iae values remained significantly lower than that of Iro.

The most recent core materials for all-ceramic FPDs are the yttrium tetragonal zirconia polycrystals(Y-TZP)-based materials. In vitro studies of Y-TZP specimens demonstrated a flexural strength of 900 to 1200 MPa(Christel, 1989) and surface hardness of 13 GPa(Guazzato *et al.*, 2004). As with alumina ceramic, previous studies have shown that acid etching and airborne particle abrasion have little effect on the bond strengths. Derand and Derand(2000) examined the surface treatments for zirconia ceramic and showed that hydrofluoric acid etching produced the lowest bond strengths. These authors also observed that sandblasting with aluminum oxide particles did produce an irregular pattern, but with little influence on the bond strength. In the study by Della Bona *et al.*(2007), sandblasting In-Ceram Zirconia surface with aluminum oxide particles revealed significantly higher mean bond strength than hydrofluoric acid etching, yet the values were significantly lower than those of the intraoral tribochemical silica coating system(Cojet<sup>TM</sup> system; 3M



ESPE, Seefeld, Germany) treated group.

In dry condition, the shear bond strength for Zab was significantly lower than Zro group in this study. The reason for the higher value of Zae compared to that of Zab in dry condition can be explained by the surface treatment method, since the specimens were sandblasted prior to acid etching.

Rocatec<sup>TM</sup> system uses tribochemical method for coating silica onto various surfaces. The first step in the Rocatec<sup>TM</sup> system is microblasting with the 110 µm-sized aluminium oxide(Rocatec Pre) to obtain microretentive roughness. Then, 110 µm-sized silicic acid-modified aluminium oxide(Rocatec Plus) is blasted onto the roughened surface. These particles impact the surface, and the resultant heat can reach up to 1200 °C. This rapid momentary rise in the temperature is caused by the transfer of kinetic energy to heat energy. The silica layer formed during this process is immediately embedded into the substrate surface(Guggenberger, 1989). Rocatec Soft, developed for substrates which are highly susceptible to abrasion(e.g. thin electroplated metal edges), has the carrier aluminium oxide whose grain size is reduced to 30 µm. In this study, it was used to simulate intraoral repair of restorations since commercially available in-office silica coating system uses 30 µm blast grit(Cojet<sup>TM</sup> system).

The high shear bond strengths after silica coating observed in the present study can be explained by two mechanisms that improve the bonding to the repair resin composite. Microscopic analysis of the blasted surface reveals a thin and microretentive layer, which should increase the bond strength to resin(Frankenberger, 2000). Simultaneously, the increase in silica content promotes chemical bonding between the composite resin and the silica-coated surfaces(Kern and Thompson, 1994).

The IPS Empress 2 system, using lithium-disilicate glass core material, demonstrates flexural strength of a range of 300-400 MPa(Schweiger, 1999)

and hardness of 5.3 GPa(Guazzato *et al.*, 2004). Studies on the surface treatment for bonding composite resin recommend a combination of airborne-particle abrasion, hydrofluoric acid etching, and silane application as for the feldspathic ceramic(Blatz, 2003). When the surface topography of lithium disilicate ceramic is observed with scanning electron microscopy(SEM) after surface treatments, airborne particle abrasion performed with 50  $\mu\text{m}$  aluminium oxide particles increased the irregularities on IPS Empress 2 surface, whereas elongated crystals and shallow irregularities were observed for the acid etched surface(Borges, 2003). This can be explained by the ability of the acid to remove the glass matrix, thus creating irregularities between the lithium disilicate crystals. This characteristic microstructure is considered to have a significant influence on the fracture resistance of the composite-ceramic adhesive zone(Della bona *et al.*, 2000). In the data of the present study, the shear bond strengths of Eab and Eae group were not significantly different.

The highest value obtained for Ero group can be explained by the combination of micromechanical and chemical bonding effect of tribochemical silica coating. However, it should be noted that in Kim *et al.*'s study(2005), tensile bond strength for acid etched Empress 2 groups was higher than that of tribochemical silica coated group. This difference between tensile bond strength and shear bond strength needs further investigation to provide clinical recommendations for intraoral repair of IPS Empress 2 systems.

For evaluation of long-term durability of surface treatment methods, specimens were thermocycled to determine its suitability in clinical use. Exposure of specimens to thermocycling speeds up the diffusion of water in between the composite resin and the ceramic. Changing the temperature creates stress at the interface of the two materials due to different coefficients of thermal expansion(Ozcan, 2003). The dry conditioned group was used as control for comparing the values. The storage in a dessicator for 24 hours

prior to shear bond testing was to maintain humidity factor as close to the baseline as possible.

Although the mean shear bond strength decreased in most of the groups after thermocycling, the durability of the bond strengths of composite resin to Rocatec system treated In-Ceram and IPS Empress 2 surfaces have been shown to be rather stable. This result was similar to those of previous studies with durable bond to In-Ceram ceramic with another BIS-GMA composite resin(Kern and Thompson, 1995).

In Zi-Ceram group, the shear bond strength value of Zro remained the highest after thermocycling, but there was no significant difference between Zro and Zab group.

It should be noted that the shear bond strength value of Zro after thermocycling was not significantly different from the values of the conventionally treated feldspathic ceramic control group, suggesting that the long term durability of bond between tribochemically silica coated zirconia ceramic and composite resin is comparable to that obtained by conventional porcelain repair systems on feldspathic ceramic.

The results partially confirmed the study hypothesis that tribochemical silica coating produces higher shear bond strength values in certain high-strength all-ceramic core materials(IPS Empress 2, In-Ceram Alumina), in terms of long term durability. Further investigation is needed to prove the effectiveness of tribochemical silica coating for bonding composite resin to zirconia ceramics. Assessment of the bond strength with different measurement methods or analysis of the data in different statistical model(i.e. Weibull statistical analysis) would be helpful in future studies. For a reliable assessment of long-term durability, various mechanical, thermal and hydrolytic loading parameters simulating intra-oral condition should be as realistic as possible.

## V. Conclusion

This study was conducted to evaluate the shear bond strength of composite resin bonded to three high-strength all-ceramic core materials with three different surface treatments (airborne particle abrasion, acid etching, and tribochemical silica coating), and to figure out the surface treatment most suitable for each core material.

Within the limitations of the present in vitro study, it can be concluded :

1. For feldspathic ceramics, the shear bond strength values among conventional acid etching and airborne particle abrasion and tribochemical silica coating method (Dab, Dae, Dro) did not show significant differences with and without thermocycling.
2. Thermocycling did not have significant effect on the shear bond strength values of the Rocatec-system-treated In-Ceram (Iro) and IPS Empress 2 (Ero) groups.
3. In Zi-Ceram group, the shear bond strength value of Zro remained the highest after thermocycling, but there was no significant difference between Zro and Zab group.
4. Bond strength obtained by tribochemical silica coating of Zi-Ceram is comparable to bond strength of conventional porcelain repair systems for feldspathic ceramic after thermocycling.

In cases of porcelain fracture involving high-strength all-ceramic core

material exposure, since no statistical difference was found among the three surface treatments for feldspathic porcelain, the selection of surface treatment may be simplified to choosing the surface treatment most effective for the exposed core material. From the result of this study, tribochemical silica coating could be recommended for lithium disilicate ceramic and alumina ceramic core. Although further investigation is needed to prove the long term stability of tribochemical silica coating on zirconia, this method is more reliable compared to acid etching or airborne-particle abrasion.

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## 국문 요약

# 전부 도재관용 코어 재료의 표면처리 및 thermocycling에 따른 복합레진과의 전단결합강도

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심미적인 이유로 사용이 증가하고 있는 전부 도재관에서 파절된 베니어 도재를 구강 내에서 복합레진으로 수리하는 방법에 대한 임상적인 지침이 필요하게 되었다. 도재 파절로 코어가 노출된 경우 강화형 도재는 알루미나 분사, 불산을 이용한 산부식으로 표면이 충분히 거칠어지지 않아 복합레진과의 결합이 어려운 것으로 알려져 있다.

본 연구에서는 전부 도재 수복물의 코어를 이루는 다양한 강화형 도재에 세 가지 다른 표면처리를 시행하고, 보다 장기적인 결합력 평가를 위하여 thermocycling 전과 후의 전단결합강도를 분석하여 각각의 코어 재료에 적합한 표면 처리 방법을 찾고자 하였다.

리튬 다이실리케이트 도재, 알루미나 도재, 지르코니아 도재와 대조군인 장석형 도재로 각각 60개의 시편을 만들고 여기에 알루미나 분사, 불산 산부식, tribochemical 실리카 코팅 세 가지의 표면처리를 시행하였다. 표면처리된 총 240개의 시편에 실란과 접착레진을 도포하고, 복합레진을 광중합시켜 부착시켰다. 각

표면처리 군을 다시 나누어 한 군은 실온에서 건조기에 24시간 보관하고 다른 군은 5 °C에서 55 °C 사이를 1200회 thermocycling 시행한 후 만능시험기로 전단결합강도를 측정하여 two-way ANOVA와 Duncan multiple comparison test로 통계 분석하였다( $\alpha=0.05$ ).

모든 도재 재료에서 tribochemical 실리카 코팅 시행했을 때 가장 높은 전단결합강도를 보였으며, thermocycling 후에는 대부분의 군에서 평균 전단결합강도가 감소하였다.

본 연구의 결과에서 리튬 다이실리케이트 도재와 알루미늄 도재에서는 tribochemical 실리카 코팅 방법이 복합레진 접착에 가장 효과적인 것으로 나타났다. 지르코니아 도재와 관련해서는 더 많은 연구가 필요하나, tribochemical 실리카 코팅 방법이 다른 두 방법에 비해서는 효과적인 것으로 사료된다.

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핵심어 : 전부도재관용 코어, 구강 내 수리, 트리보케미컬(tribochemical) 실리카 코팅, 전단결합강도