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The Influence of Ceramic Surface Treatments on the Micro-shear Bond Strength of Composite Resin to IPS Empress 2

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Abstract

Purpose: An increasing demand for esthetic restorations has resulted in the development of new ceramic systems, but fracture of veneering ceramics still remains the primary cause of failure. Porcelain repair frequently involves replacement with composite resin, but the bond strength between composite resin and all-ceramic coping materials has not been studied extensively. The purpose of this study was to evaluate the influence of different ceramic surface treatments on the micro-shear bond strength of composite resin to IPS Empress 2 coping material.

Materials and Methods: Sixteen $7 \times 7 \times 1$ mm^3 lithia disilicate-based core ceramic plates were fabricated using the lost wax technique. The plates were divided into eight groups, and eight different surface treatments were performed: (1) no treatment (NT); (2) airborne-particle abrasion with 50-μm alumina particles (Al); (3) acid etching with 9.6% hydrofluoric acid for 1 min (HF); (4) silane coating (S); (5) AlHF; (6) AlS; (7) HFS; and (8) AlHFS. Then, ten composite resin cylinders (0.8-mm diameter $\times$ 0.5-mm height) were light-polymerized onto the ceramic plates in each group. Each specimen was subjected to a shear load at a crosshead speed of 0.5 mm/min until fracture occurred. The fracture sites were examined with scanning electron microscopy (SEM) to determine the location of failure during debonding and to examine the surface treatment effects. One-way analysis of variance (ANOVA) and multiple comparison (Dunnet T3) tests were used for statistical analysis of data.

Results: The mean micro-shear bond strength values (SD) in MPa were—NT: 4.10 (3.06), Al: 7.56 (4.11), HF: 14.04 (2.60), S: 14.58 (2.14), AlHF: 15.56 (3.36), AlS: 23.02 (4.17), HFS: 24.7 (4.43), AlHFS: 26.0 (3.71). ANOVA indicated the influence of surface treatment was significant ($p < 0.0001$). SEM analysis did not reveal entirely cohesive failure in any composite or ceramic.

Conclusion: The micro-shear bond strength of a composite resin to IPS Empress 2 was significantly different depending on the surface treatment method. Among the investigated methods, silane coating after airborne-particle abrasion and etching was the most effective surface treatment in terms of bond strength increase.

Ceramic materials are brittle; therefore, historically, they have been fused to metal copings to increase resistance to fracture.[1] The metal base, however, has esthetic limitations, such as reduced light transmission and discoloration. These disadvantages have prompted the development of all-ceramic systems that do not require metal support.

Most all-ceramic systems have a two layer structure, using a weak veneering ceramic over a strong supporting core.[2,3] Often, failure of all-ceramic restorations occurs when the veneering ceramic fractures, exposing the coping material.[4-6]

Replacement of a failed restoration is not necessarily the most practical solution, considering replacement cost, compromise of additional tooth structure, and additional trauma to the tooth. The repair of a fractured ceramic restoration is a challenging clinical situation and as yet there is little documentation on the clinical performance of the repaired restoration. Suggested clinical procedures for ceramic repair include the preparation of

the damaged surface and the use of ceramic repair materials.[7,8] The material of choice for repair has been resin composite, due to good esthetics, low cost, and easy handling.

To achieve a satisfactory bond between ceramic and resin, several mechanical and chemical retention systems have been developed. Airborne-particle abrasion[9] and acid etching with hydrofluoric acid (HF)[10,11] are commonly used methods to achieve an irregular surface topography of microretentive channels and increased surface area for bonding. Fine alumina oxides under pressure are used in the airborne-particle abrasion method. In this manner, the relatively weaker phases of ceramic are removed, and an irregular rough surface is created.[12] Chemical etching selectively dissolves the glassy matrix in ceramic to generate an irregular surface;[13-16] however, the intraoral airborne-particle abrasion method's use of HF may eventually be harmful to oral tissues because of the very aggressive nature of this acid in the concentrations required for acid etching of the ceramic surface.[17] For clinical techniques, rubber dam and sodium bicarbonate/calcium source acid neutralizing medium have been recommended to protect adjacent teeth and tissues from HF.[18-20]

Silane coupling agents have been extensively used in ceramic repair systems. Establishment of a strong chemical bond between the dental ceramic and resin composite can be achieved by treatment with a silane coupling agent.[21]

The bond strength promoted by mechanical and chemical retention systems can be influenced by ceramic microstructure,[22-25] but most in vitro studies of composite-ceramic bonding are limited to feldspathic ceramics. Therefore, the objective of this study was to evaluate the micro-shear bond strength of a composite resin to IPS Empress 2 (lithium-disilicate ceramic) after different surface treatments. The null hypothesis was that there were no significant differences in the bond strength of composite resin to IPS Empress 2 after different surface treatment methods.

Materials and methods

Sixteen 7 × 7 × 1 mm^3 lithia disilicate-based core ceramic plates (IPS Empress 2, shade 300, lot no. d24552, Ivoclar Vivadent, Schaan, Liechtenstein) were fabricated using the lost wax technique. After removal of the cast ceramic from the investment, the interaction layer was removed from the plates' surface by grit blasting with 80 μm glass beads. The plates were cleaned in 1% hydroflouric acid for 30 minutes. Then, all substrate plates were wet ground with 240-, 400-, and 600-grit silicon carbide abrasive paper for surface standardization. The ceramic plates were cleaned for 10 minutes in an ultrasonic bath containing distilled water and were air-dried. The plates were then assigned to eight groups, which received the following surface treatments:

- Group NT: No surface treatment applied.
- Group Al: Airborne particle abrasion with 50-μm aluminium oxide particles at 35 psi from a distance of approximately 10 mm for 15 seconds and cleaned with compressed air oil-free for 30 seconds.
- Group HF: HF acid (Ultradent porcelain etch 9.6% Buffered, lot no. B1VBC, Ultradent Products, South Jordan, UT) applied for 30 seconds, rinsed for 30 seconds, dried with compressed air oil-free for 30 seconds.
- Group S: The silane (Monobond-S, Ivoclar Vivadent) was applied with a minisponge, allowed to evaporate for 3 minutes, and air-dried for 30 seconds.
- Group AlHF: Airborne-particle abrasion followed by HF application.
- Group AlS: Airborne-particle abrasion followed by silane application.
- Group HFS: HF acid applied, followed by silane application.
- Group AlHFS: Airborne-particle abrasion, HF acid applied, followed by silane application.

The adhesive bonding resin (Heliobond, lot no. F58115, Ivoclar Vivadent) was applied to the surface and light-cured at 450 mW/cm^2 (Optilux, Demetron Research Corporation, Orange, CA) for 20 seconds. Prior to the light-curing step, plastic tubes (Tygon, Norton Performance Plastic Co, Cleveland, OH) 0.8 mm in diameter and 0.5-mm thick were placed on the uncured adhesive surfaces of each plate. These surfaces then polymerized to stabilize the plastic tube on the ceramic surface. Because the tygon cylinder was bonded tightly to the ceramic surface, no flash of resin composite extended onto the ceramic beyond the base of the cylinder.[26,27]

After curing, a resin composite (Tetric Ceram, Lot no. F38857, Ivoclar Vivadent) was placed into the tube iris and cured for 40 seconds. The plates were stored at room temperature (23°C) for 1 hour prior to removal of the tygon tubing. In this manner, a small cylinder of resin composite 0.8 mm in diameter and 0.5 mm in height was bonded to the ceramic surface. Ten specimens were created in each group. The specimens were stored in distilled water at 37°C for 24 hours.

Before the test, all resin cylinders were checked under an optical microscope (30×). The cylinders that showed no apparent interfacial defects or bubble inclusion and no leaking of composite core were tested.

Each ceramic plate was attached to the testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA) with cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA). A wire loop prepared from an orthodontic stainless steel ligature wire (0.2 mm diameter) was wrapped around the bonded assembly so that it was as close as possible to the base of the resin composite. The resin–ceramic interface for the test, the wire loop, and center of the load cell were aligned as straight as possible to ensure the desired orientation in the shear test force. A shear load was applied via a universal testing machine (EZ-test-500N, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min until failure occurred (Fig 1).

Interfacial shear strength was calculated by dividing the maximum load recorded on failure by the circular bonding area in square millimeters and expressed in MPa. Specimens that failed prematurely during handling were assigned zero strength values and were included in the statistical analysis. Statistical analysis was performed using the SPSS statistical package (SPSS 11.5, SPSS, Chicago, IL). One-way analysis of variance (ANOVA) was applied to detect any differences between groups. A Dunnet T3 multiple comparison test was used to determine if a

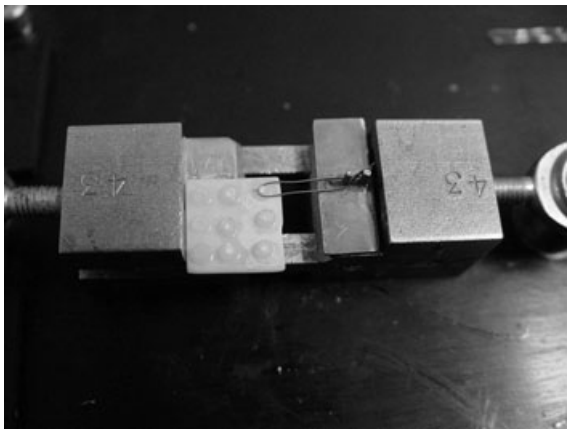

Figure 1 Specimen attached to the test machine.

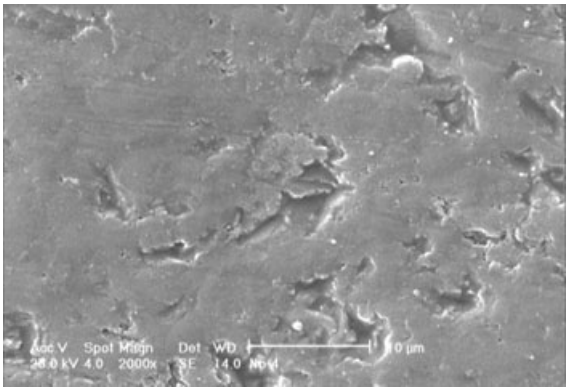

Figure 2 Fracture surface of the no treatment group. The adhesive layer was totally debonded from composite (Mode 1).

significant difference ($\alpha = 0.05$) in load to failure existed among the eight groups.

All debonded specimens were examined under a scanning electron microscope (SEM) (JXA840, JEOL, Tokyo, Japan) to determine the mode of fracture. The failure modes were recorded as:[28]

- Mode 1: adhesive (if one fracture site was at the composite or ceramic surface and the other site remained adhesive only),
- Mode 2: cohesive in adhesive layer (fractures extending through the adhesive),
- Mode 3: cohesive in composite (failure totally within composite) or cohesive in ceramic (failure totally in ceramic), and
- Mode 4: mixed failures (failure including at least two of these materials).

Results

Mean shear bond strength values (± standard deviations) and number of premature failures are shown in Table 1. One-way ANOVA indicated that the micro-shear bond strength was significantly affected by the surface treatment ($F = 45.45$, $p < 0.0001$).

Table 1 Micro-shear bond strength (MPa) and number of prematurely failed specimens

Experimental groups	N	Mean (SD)	Premature failure
NT	10	4.10 (3.06)[a]	3
Al	10	7.56 (4.11)[a]	2
HF	10	14.04 (2.60)[b]	0
S	10	14.58 (2.14)[b]	0
AlHF	10	15.56 (3.36)[b]	0
AlS	10	23.02 (4.17)[c]	0
HFS	10	24.70 (4.43)[c]	0
AlHFS	10	26.00 (3.71)[c]	0

Groups identified by different superscript letters were significantly different ($p < 0.05$).

The Dunnet T3 comparative test revealed that the AlHFS, HFS, and AlS groups had significantly higher bond strength than other groups but were not statistically significant from each other. Bond strength groups S, HF, and AlHF had no statistically significant difference. The group with no surface treatment (NT) had the lowest bond strength but was not statistically different from the airborne-particle abrasion group (Al).

SEM analysis did not reveal entirely cohesive failure in composite or ceramic (Mode 3). Failure mode in the group with no treatment (NT) was predominantly Mode 1. Mechanically-reinforced groups (Groups Al, HF, and AlHF) exhibited failure Modes 1 and 4, but in the silane-coated groups (S, AlS, HFS, and AlHFS) most of the failure modes were 2 and 4 (Figs 2–9).

Discussion

Measurement of bond strength, regardless of the technique chosen, is a controversial topic in dental adhesion.[29] Conventional shear and tensile bond tests have generally been used to evaluate fractured ceramic restorations that were repaired with resin composite; however, the most commonly used shear bond

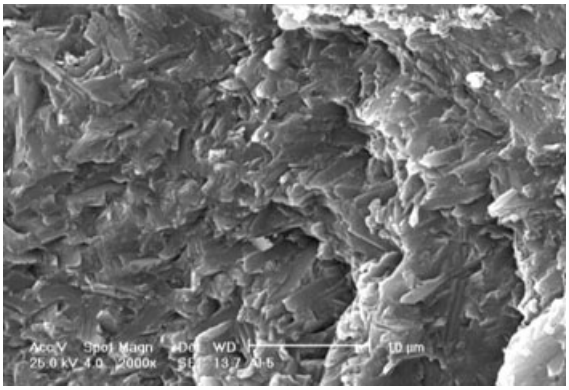

Figure 3 Fracture surface of the airborne-particle abrasion with 50-μm alumina particles group (Al). On the right of the micrograph, composite remains (Mode 4).

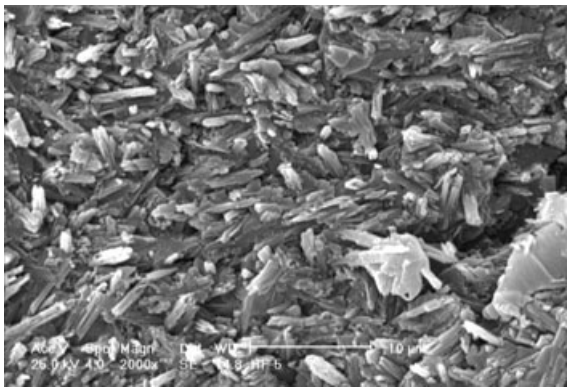

Figure 4 Fracture surface of the acid etching with hydrofluoric acid group (HF). Remains of composite on the right side of micrograph (Mode 4).

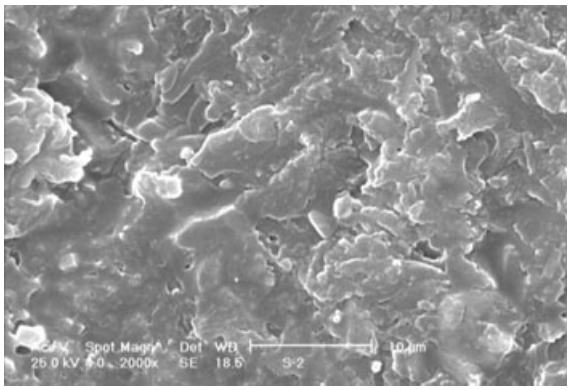

Figure 5 Fracture surface of the silane coating group (S). Fracture occurred in the adhesive layer (Mode 2).

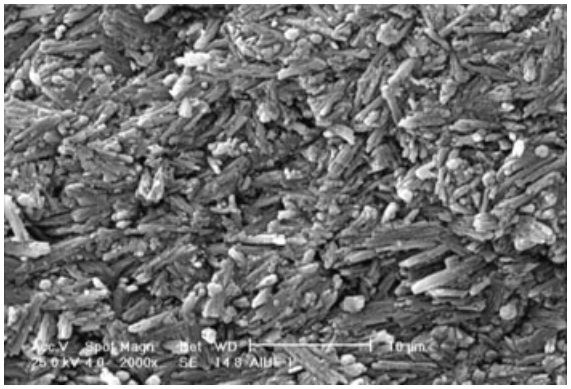

Figure 6 Fracture surface of the group AlHF. The adhesive layer was totally debonded from roughed ceramic surface (Mode 1).

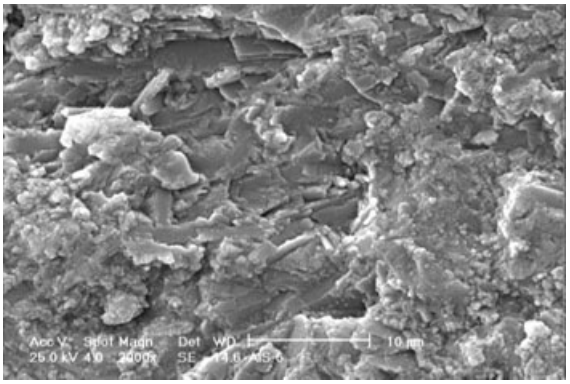

Figure 7 Fracture surface of the group AlS. The remains of composite were on the adhesive layer (Mode 4).

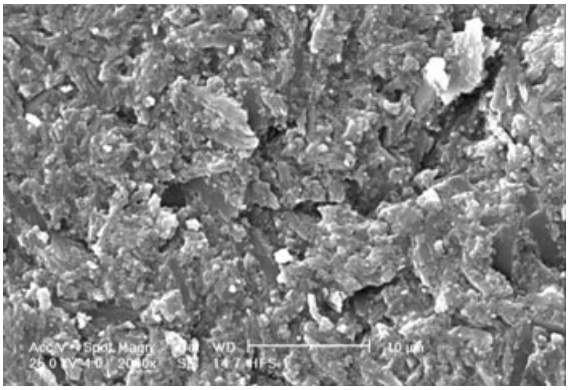

Figure 8 Fracture surface of the group HFS. Failure mode was mixed (Mode 4).

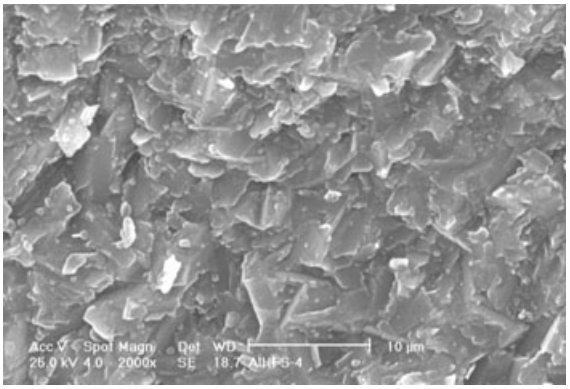

Figure 9 Fracture surface of the group AlHFS. The remains of composite spread on the adhesive layer that attached to roughed ceramic surface (Mode 4).

test often produces fracture away from the adhesion zone.[30-34] Such failures of the substrate prevent measurement of interfacial bond strength and limit further improvements in bonding systems.

Several studies have identified nonuniform stress distributions along bonded interfaces.[30,35,36] The nonuniform interfacial stress distribution generated for conventional tensile and shear bond strength tests initiates fractures from flaws at the interface or in the substrate in areas of high stress concentration. Recently researchers have preferred to use the microtensile method and fracture mechanics to understand the properties of the adhesive interface.[37] Unfortunately, the microtensile bond

test, although an effective method in terms of testing a small area, is difficult to conduct and time-consuming for specimen preparation, especially in the case of glass ceramic samples. Therefore, the micro-shear bond test was designed,[38] because in this method there is no necessity for trimming the specimen, and the bonding surface was intact. In the micro-shear test method, stress distribution is uniform because an ultra small area of bonding interface was tested. Therefore, in the present study micro-shear bond strength testing was used to measure the bond strength.

The results of this study showed that significantly different degrees of adhesion of composite to lithium disilicate substrate can be achieved by different surface treatment methods. The AlHFS group, which involved treatment of lithium disilicate substrate surface with airborne-particle abrasion followed by 9.6% HF acid etching and application of a silane coupling agent, yielded the highest shear bond strength values. In Della Bona et al[39] and Filho et al's[28] studies of microtensile bond strengths of composite resin to lithium disilicate ceramics, silane coating of etched surfaces provided the highest and most durable bond strength values; however, in those studies, airborne-particle abrasion was not included as a separate surface treatment method.

The results of this study were in agreement with Oh et al's[40] study. They showed that airborne-particle abrasion of ceramic surface combined with etching with HF 9.6% creates a significantly higher bond strength than etching of surface alone. But in this study, the higher bond strengths obtained in Group AlHF were not significantly different from the bond strength of Group HF. This may be attributed to the difference in the test method (microtensile vs. micro-shear).

It is known that both airborne-particle abrasion and HF selectively dissolve the weaker glassy phase and exposed lithium disilicate crystals, both of which serve as retentive features. The porous irregular surface facilitates the penetration of the resin into the micro-retentions of the treated ceramic surfaces.[41,42] The only difference that could be observed was the presence of grooves on the ceramic after the airborne-particle abrasion. A possible explanation for this may be that as the surface abrades with alumina particle, microscopic cracks are produced. Therefore, HF acid is able to penetrate and remove the glass matrix along the groove. The enhancement in surface associated with the altered topography caused the stronger bond, because resin could penetrate deeply in micromechanical undercuts.

The definition of the adhesion zone is critical in classification of the mode of failure, which should be an integral component of all failure analysis. A careful microscopic analysis of the fracture surface can produce a more consistent and complete description of the fracture process. Thus, the quality of the bond should not be assessed based on bond strength data alone. The mode of failure could provide important information about the clinical performance limit, which is the ultimate test of any adhesive system. SEM micrographs of fractured surfaces revealed that all fractures occurred in the adhesive interface. In silane-coated groups (S, AlS, HFS, and ALHFS), most fractures occurred totally and some occurred partially in the adhesive bond layer, but in other groups (NT, Al, HF, and AlHF) the adhesive layer separated from the ceramic substrate totally. This showed that the application of a silane coupling agent to the ceramic surface provides a chemical covalent and hydrogen bond of resin systems to ceramic[43,44] and is a significant factor for a sufficient resin bond to ceramics. These results are consistent with previous studies.[13,45,46] On the other hand, the silica content of lithium disilicate ceramics is approximately 60 wt%, as reported by the manufacturer, and is enough to obtain a reliable bond strength between the composite resin and ceramic without the silica-coating technique.

This study had limitations in its ability to simulate clinical loading forces on restorations and oral environmental changes. The loading was monotonic instead of cyclic fatigue, and the temperature and moisture of the oral cavity were not simulated. Specimens were not thermal cycled. Furthermore, only one composite resin was tested. Future studies with a model that more closely resembles the oral environment and simulates clinical loading conditions are indicated.

Conclusions

Within the limitations of this in vitro study, there was significant difference in the fracture load of Empress 2 depending on the surface treatment method. Airborne particle abrasion followed by 9.6% HF acid etching and application of a silane coupling agent yielded the highest shear bond strength values.

Acknowledgment

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