Influence of Surface Conditions and Silane Agent on the Bond of Resin to IPS Empress 2 Ceramic

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Purpose: The aim of this study was to evaluate the effect of different ceramic surface treatments on the tensile bond strength between IPS Empress 2 ceramic framework and Rely X adhesive resin cement, with or without the application of a silane coupling agent. **Materials and Methods:** One hundred twenty disks were made, embedded in resin, and randomly divided into six groups: group 1 = sandblasting (100 μm), no silanation; group 2 = sandblasting (100 μm), silane treatment; group 3 = sandblasting (50 μm), no silanation; group 4 = sandblasting (50 μm), silane treatment; group 5 = hydrofluoric acid etching, no silanation; and group 6 = hydrofluoric acid etching, silane treatment. The disks were bonded into pairs with adhesive resin cement. All samples were stored in distilled water at 37°C for 24 hours and then thermocycled. The samples were submitted to tensile testing. **Results:** The use of silane improved the bond strength in relation to the groups in which silane was not applied ($P < .05$). The most effective surface treatment was etching with 10% hydrofluoric acid, both with (25.6 MPa) and without silane application (16.4 MPa); these values showed a statistically significant difference compared to sandblasting with 50- and 100-μm Al_2O_3. Sandblasting with 50-μm Al_2O_3, with (11.8 MPa) and without silane (5.4 MPa), demonstrated significantly higher tensile bond strength than sandblasting with 100-μm Al_2O_3, with (8.3 MPa) and without silane (3.8 MPa). **Conclusion:** Combined application of 10% hydrofluoric acid and silane enhanced the bond strength between the IPS Empress 2 ceramic framework and resin agent. *Int J Prosthodont 2003;16:277–282.*

Ceramics as dental materials have excellent properties, such as low thermal conductivity, thermal diffusivity, and electrical conductivity.[1] However, they are brittle, a quality attributed to surface and bulk defects.[2,3] Metal-backed ceramics were developed with the objective of improving the mechanical properties of the overall restoration,[4] but the metal core can affect esthetics.

In the last few years, systems such as Dicor (Dentsply),[5] Optec-HSP (Jeneric/Pentron),[6] In-Ceram (Vita),[7] IPS Empress (Ivoclar),[8] and Duceragold (Degussa-Huls) have been introduced. Recently, IPS Empress 2 (Ivoclar) was developed. It is a multiphase glass ceramic with a high degree of crystallinity, which improves the mechanical properties; this material is indicated for crowns and fixed partial dentures. The veneering material consists of an apatite glass ceramic.[9–11]

The clinical success of the ceramic restoration depends on a number of factors, such as the cementation procedure. The framework of IPS Empress 2 may be conventionally or adhesively cemented. However, when the retentive area is small, retention may be inadequate,[12] and an adhesive luting agent is desirable. Bonding of the resin cement to the tooth is ensured by acid etching of enamel and/or dentin, and by the use of a dentin adhesive.[13] Bonding to the feldspathic ceramic side of the joint is normally obtained by etching the ceramic with hydrofluoric acid to create a rough surface, favoring adherence by mechanical

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interlocking.[14] The bond may also be improved by the application of silane coupling agents that are capable of forming chemical bonds with both the inorganic and organic surfaces.[15,16]

A potential limitation to clinical use of some core ceramics, In Ceram for example, has been the inability to etch fit surfaces.[17,18] For this reason, a number of methods and materials have been suggested to enhance resin cement adhesion to this core material, such as the use of a tribochemical silica-coating system (Rocatec, ESPE) or sandblasting and use of a resin cement modified with a phosphate monomer (Panavia 21, Kuraray).[19] With respect to IPS Empress 2, it is also important to identify the type of surface treatment that may enhance the bond strength between the lithium disilicate framework and the adhesive luting agent.

It was the hypothesis of this study that surface treatment combined with the application of a silane coupling agent could significantly improve the bond strength between IPS Empress 2 ceramic framework and Rely X (3M) adhesive resin cement.

Materials and Methods

Wax patterns for the IPS Empress 2 ceramic framework were made using a cylindric, internally tapered stainless steel mold. Molten wax was applied to the stainless steel mold to produce patterns 5.3 mm in diameter at the top and 7.0 mm at the bottom, with a thickness of 2.5 mm. The wax patterns were sprued and attached to a muffle base with a surrounding paper cylinder. The IPS Empress 2 wax patterns were invested, and the wax was eliminated in a burnout furnace. One hundred twenty specimens were pressed using IPS Empress 2 ingots (shade A2) in an automatic press furnace (EP 500, Ivoclar) using the manufacturer's instructions and 5-bar pressure. After cooling, the samples were divested, sprues were removed and ground, and samples were cleaned with water in an ultrasonic unit (Thornton).

An adaptor to centralize the position of the ceramic disk on a PVC tube was made, and the ceramic disk was attached to the tube with acrylic resin, leaving 1 mm of the ceramic surface exposed. The acrylic resin cylinders with the ceramic disks were positioned in a stainless steel holder and wet ground with 400- and 600-grit SiC paper to obtain a flat area with a diameter of 5.5 mm, which was controlled with the use of a digital caliper rule (Mitutoyo). All ceramic disks were then sandblasted with 100-µm aluminum oxide at 2-bar pressure for 5 seconds and cleaned ultrasonically in water for 20 seconds. The specimens were randomly divided into six groups, and each group was submitted to a different surface treatment:

- Group 1 = sandblasting with 100-µm Al_2O_3 particles for 5 seconds under a pressure of 2 bars, with a sandblasting device (Manfredi) held at a distance of 10 mm from the ceramic surface. Sample were then rinsed, cleaned ultrasonically in water for 20 minutes, and dried.
- Group 2 = sandblasting with 100-µm Al_2O_3 for 5 seconds as described for group 1. One coat of Scotchbond Ceramic Primer (3M) was applied and allowed to air dry for 2 minutes.
- Group 3 = same as group 1, except the Al_2O_3 particles were 50 µm.
- Group 4 = sandblasting with 50-µm Al_2O_3 for 5 seconds as described for group 3, followed by silane application as described for group 2.
- Group 5 = etching with 10% hydrofluoric acid for 20 seconds, followed by rinsing for 1 minute. Samples were then cleaned ultrasonically with water for 20 minutes and dried.
- Group 6 = etching with 10% hydrofluoric acid for 20 seconds as described for group 5, and silane application as described for group 2.

One coat of Single Bond Adhesive (3M Dental) was applied, air dried for 5 seconds, and polymerized for 10 seconds. A resin luting agent (Rely X ARC, 3M Dental) was manipulated according to the manufacturer's instructions and applied to the ceramic surface. The ceramic disks were then joined in pairs, and a 500-g static load was applied for 1 minute. The excess cement was removed with a brush before light curing for four 40-second periods at right angles to each other. The specimens were stored in distilled water at 37°C for 24 hours, followed by 500 thermocycles between 5 and 55°C.

The tensile strength testing was performed using an Instron universal testing machine at a cross-head speed of 1.0 mm/minute until failure occurred. The tensile bond strength was calculated by dividing the maximum load by the cross-sectional area under test to give results in MPa. A total of 10 specimens were made and tested for each group. The results were subjected to analysis of variance (ANOVA) and Tukey's test at the 95% significance level.

The fractured surfaces of the specimens were examined with a stereomicroscope (Olympus) at 20× to classify the type of failure that occurred during the debonding procedure. Failure was classified as: *(1)* adhesive, when the fracture occurred at one of the ceramic interfaces; *(2)* cohesive, when the resin cement was fractured; and *(3)* mixed, with a combination of adhesive and cohesive failures. The surfaces treated by different methods were coated with gold and examined by scanning electron microscopy (SEM; LEO 435 VP).

Table 1 Mean Tensile Bond Strength (MPa) with and without Silane Application

Surface treatment	Mean with silane	Standard deviation	Mean without silane	Standard deviation
10% hydrofluoric acid	25.6	1.2	16.4	1.4
50-µm sandblasting	11.8	1.0	5.4	0.3
100-µm sandblasting	8.4	0.9	3.8	0.3

Table 2 Failure Mode Analysis of Debonded Specimens

Surface treatment	Adhesive	Mixed	Cohesive
100-µm sandblasting (without silane)	3	7	—
100-µm sandblasting (with silane)	—	10	—
50-µm sandblasting (without silane)	3	7	—
50-µm sandblasting (with silane)	—	10	—
10% hydrofluoric acid (without silane)	—	10	—
10% hydrofluoric acid (with silane)	—	8	2

Results

Following silane application, the mean tensile bond strength for the samples etched with 10% hydrofluoric acid was significantly higher than for the samples sandblasted with 50- and 100-µm Al_2O_3 (Table 1; $P < .05$). Sandblasting with 50-µm Al_2O_3 produced significantly higher tensile bond strengths than sandblasting with 100-µm Al_2O_3 ($P < .05$). Similar results were observed in the absence of silane application (Table 1).

Steromicroscopic examination demonstrated that the majority of failures were mixed in all surface treatments both with and without silane application (Table 2). Figures 1 to 4 show sample specimens.

Discussion

The prosthetic elements made with IPS Empress 2 ceramic framework can be luted to the dental preparation using adhesive or conventional techniques. In this study, the influence of three ceramic surface treatments was evaluated, as was the application of silane using Single Bond adhesive and Rely X resin cement. The etching procedure with 10% hydrofluoric acid resulted in the highest mean tensile bond strength, with a statistically significant difference in relation to sandblasting with 50- and 100-µm Al_2O_3. Etching with 10% hydrofluoric acid resulted in the glassy matrix of the samples dissolving from the surface to the depth of a few microns and, as a result, the lithium disilicate crystals protruded from the glassy matrix (Figs 3 and 4). This treatment significantly changed the surface morphology, increasing the surface area, which favored infiltration and retention of adhesive materials and made the ceramic surface more retentive. Etching with hydrofluoric acid has also been mentioned as an efficient surface treatment for feldspathic ceramics.[20–23] The failure modes were predominantly of the mixed variety, probably because of a more retentive surface.

Lithium disilicate crystals measure 0.5 to 4.0 µm in length and represent the main crystal phase of the glass ceramic.[24] A second crystalline phase, present at low levels, is lithium orthophosphate. These crystals measure 0.1 to 0.3 µm in diameter and are located in the glassy matrix and on the surface of the lithium disilicate crystals. However, the lithium orthophosphate crystals are no longer discernible because of the acid etching, and voids are visible, indicating the probable location of the previous crystal precipitation.

The lowest mean tensile bond strength was obtained for samples sandblasted with 100-µm Al_2O_3, a treatment that was performed with the objective of reproducing the ceramic surface topography produced by the laboratory; this was confirmed by SEM (Fig 2). SEM also gave the explanation for the low bond strength, showing evidence of a roughness that did not produce as good a retentive surface as the one obtained with etching. However, sandblasting with 50-µm Al_2O_3 increased the ceramic surface roughness (Fig 1), which promoted a significant increase in the bond strength compared with sandblasting with 100-µm Al_2O_3.

Another factor evaluated in this study was the influence of silane on the bond strength between the IPS Empress 2 framework and resin cement. Several silane coupling agents are available on the market. Those supplied as a single-component system are already hydrolyzed. In the ones supplied as two separate components, the hydrolysis occurs moments before application, after mixing the two components.

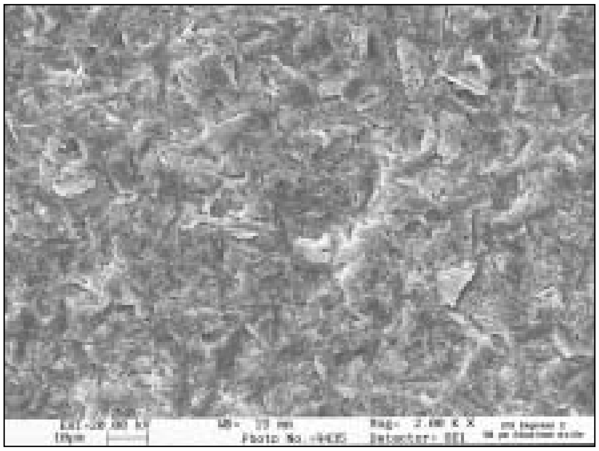

Fig 1 SEM of surface treated with sandblasting with 50-µm Al_2O_3 particles.

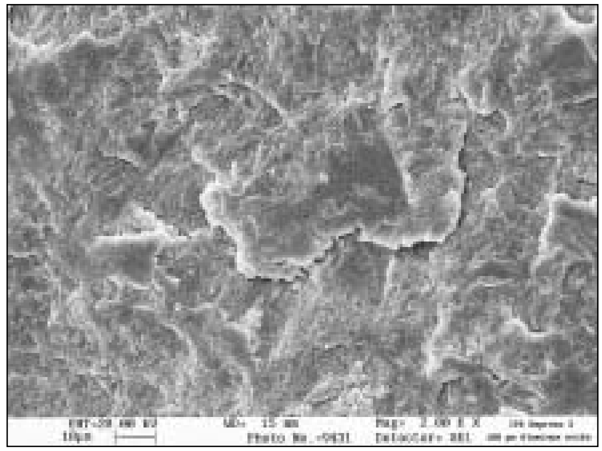

Fig 2 SEM of surface treated with sandblasting with 100-µm Al_2O_3 particles appears quite similar to Fig 1, but perhaps marginally less rough.

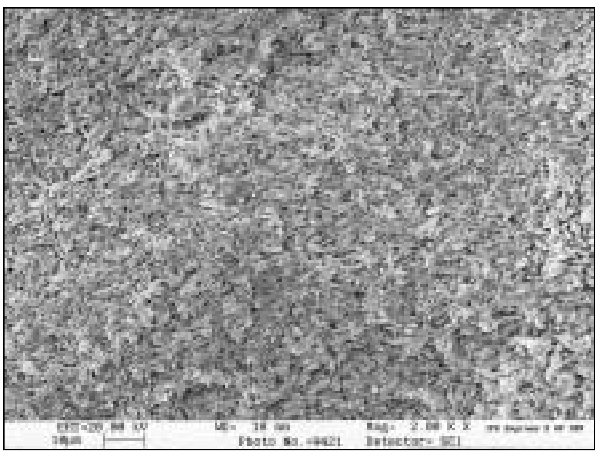

Fig 3 Specimen treated with 10% hydrofluoric acid etching clearly shows the very rough surface.

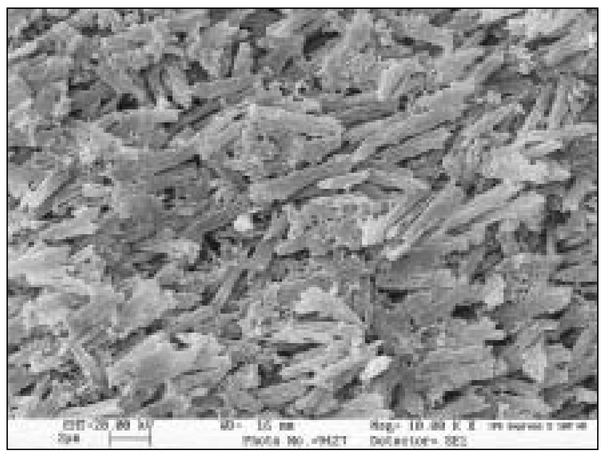

Fig 4 Higher magnification of specimen in Fig 3 shows the developed porosity penetrating the surface.

Although the literature demonstrates that different results are obtained according to the kind of silane used,[25,26] the present study did not intend to compare the efficiency of different kinds of silanes. Scotchbond Ceramic Primer was chosen because it is ready for immediate use as supplied by the manufacturer.

Independent of the treatment applied to the ceramic surface, there was always an increase in the bond strengths for the groups that received the silane application; the same behavior has been observed for feldspathic ceramics.[27–30] Silane coupling agents are usually monomeric species in which silicon is linked to reactive organic radicals and hydrolyzable ester groups. The reactive organic groups become chemically bonded to the resin molecules, such as HEMA, found in the Single Bond adhesive as well as in the Rely X ARC resin cement. Hydrolyzable monovalent groups bond chemically to silicon contained in the glassy matrix and lithium disilicate crystals. Another important factor is the capacity of silane to improve surface wettability,[31] causing better contact and infiltration of the adhesive in irregularities on the ceramic.[32]

Silane priming minimizes leakage at the resin-ceramic interface after thermocycling.[33] The effect of thermocycling on bond strength has not been determined. However, several studies have demonstrated that, depending on the type of silane used, thermocycling may have a significant effect on bond strength reduction between the resin-ceramic interface.[34–36] The difference between the silane products and their efficacy is related to the degree of hydrolysis; the higher the degree of hydrolysis, the better the bond provided by the silane coupling agent.[37] In addition, certain silanes have a greater capacity to wet the ceramic surface.[38] The permeability of the silane, that is, the hydrolysis of the Si-O bonds at the porcelain-silane interface because of the ingress of water, may also be responsible for the level of degradation and the bond strength between the ceramic-resin interface.[39] The Scotchbond Ceramic Primer used in this study showed its effectiveness in the thermocycling regimen, since the predominant mode of failure was mixed or cohesive in the resin cement and not at the cement-ceramic interface.

Etching the IPS Empress 2 ceramic framework surface with 10% hydrofluoric acid, along with silane application, produced higher tensile bond strengths. This may explain the occurrence of cohesive failures in the resin cement of two specimens. In the case of feldspathic ceramics, etching with hydrofluoric acid and silane application also gave higher bond strengths.[40–43] Some studies indicate that acid etching of ceramic could be eliminated, resulting in a reduction in operating time and elimination of the hazard of storing hydrofluoric acid. Only the application of silane would give bond strengths comparable with acid etching and surpass the ceramic's own cohesive strength.[44,45] This may not be necessary for the IPS Empress 2 ceramic framework, since the ceramic retentive surface promoted by acid etching was sufficient to obtain the highest bond strengths. Another factor to be considered is the higher cohesive strength of the IPS Empress 2 ceramic framework in comparison to feldspathic ceramics. In spite of the high values of bond strength obtained during the tensile test, no cohesive failure in the ceramic was observed.

Conclusions

1. The application of silane was effective in increasing the bond strength between the IPS Empress 2 ceramic framework and the resin agent.
2. Etching with 10% hydrofluoric acid increased the tensile bond strength.
3. Combined application of 10% hydrofluoric acid and silane provided the strongest bonding of the resin agent to the IPS Empress 2 ceramic framework.

Acknowledgment

The authors acknowledge the support of NAP/MEPA for this study.

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Literature Abstract

Corono-radicular reconstruction of pulpless teeth: A mechanical study using finite element analysis.

This study analyzed the effect of different reconstruction methods on stress transmission in endodontically treated teeth; 3-D models of a tooth embedded in bone were involved. Two levels of coronal tissue loss were studied: total loss of the coronal dentin, and partial loss of coronal dentin with 2-mm dentin walls. Teeth were reconstructed by four different modalities: cast Ni-Cr post and core, Ni-Cr post and composite core, carbon-fiber post and composite core, and composite restoration without post. An Ni-Cr crown covered each of the models and was subjected to a 30-degree off-axial load at 100 N. The stress levels were computed. The greatest stress was observed in the cervical region in all models, cervical tensile stresses exceeded 230 Pa without a ferrule, and cervical tensile stresses were less than 140 Pa with a ferrule. Without a ferrule, the Ni-Cr post and composite core generated greater cervical stress (254 Pa) than the cast post and core (235 Pa); with a ferrule, the Ni-Cr post and composite core generated slightly higher cervical stress (92 Pa) than the cast post and core (90.5 Pa). With a ferrule, the tensile stress generated by the composite restoration without a post (139 Pa) was 51% greater than that generated by the Ni-Cr post and composite core and 26% greater than that generated by the carbon-fiber post and composite core. This finite element study demonstrated that cervical stresses were lower with a post; the higher the elasticity modulus of the post, the lower is the stress; a ferrule in the tooth reduced stress; and posts reduced cervical stress to a lesser extent than ferrules.

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