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Shear Strength of the Composite Bond to Etched Porcelain

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The shear bond strength of composite resin to porcelain was investigated to optimize variables for bonding porcelain laminate veneers. Scanning electron microscopy was initially used to examine the surface configuration of porcelain prepared under various conditions. A factorial experiment was undertaken to determine the effects of three different bonding methods on both etched and non-etched porcelain. Composite resin was bonded to the porcelain groups using (a) unfilled resin, (b) silane, and (c) silane with dentin adhesive. The results indicated a significant difference in shear bond strength for the three bonding groups, depending on the porcelain surface condition. For the unetched samples, significant differences in bond strength were obtained for all three bonding conditions. However, for the etched group, there were no differences between the silane and silane-with-dentin-adhesive groups. Porcelain etching significantly increased bond strength across all three bonding methods and was the main contributor to the obtained values.


Introduction.

Traditionally, cosmetic or functional restoration of anterior teeth has often necessitated the use of full-coverage restorative procedures. However, more conservative methods of restoration have been presented within the past decade. Mouradian et al. (1976) first discussed the use of pre-fabricated acrylic facings bonded to etched enamel as a means of masking tetracycline stains. Others have since described additional veneering techniques. These have included the use of hollow ground denture teeth (Faunce and Myers, 1976), composite resins veneered directly to enamel (Jordon et al., 1977), and pre-formed plastic veneers (Avery, 1980; Chalkley, 1980; Paterson and Anson, 1980), as well as custom-fabricated laminates used with laboratory models (Barkley et al., 1979; Ronk, 1981; Cheung et al., 1982).

Problems inherent in plastic veneers became evident with their widespread use. Color instability, surface stain, leakage at the laminate-composite interface, fracture, and loss resulting from limited bond strength of laminates to composite resins have been described by various authors (Perez et al., 1980; Avery, 1980; Boyer and Chalkley, 1982). Within the past several years, porcelain veneers have been introduced as a means of overcoming some of these deficiencies (Horn, 1983).

Procedures advocated by Horn for the fabrication and bonding of porcelain veneers include the firing of porcelain on a platinum foil matrix, etching with hydrofluoric acid, and bonding with silane and a composite resin. Simonsen and Calamia (1983), in examining some of these variables, correlated porcelain etching time and increased bond strength when using a 7.5% hydrochloric acid solution. Calamia and Simonsen (1984) confirmed that the use of silane increased the bond strength of composite resin to etched porcelain.

Since the use of porcelain laminate veneers requires considerable support from the underlying composite and enamel in order to resist stress in the oral environment, the present study was undertaken to optimize the bond strength of composite resin to porcelain by varying porcelain manipulation and bonding procedures.

Materials and methods.

A two-phase study was undertaken that initially examined surface configuration of porcelain for the bonding of composite resin. Using an orthoclave feldspathic porcelain containing leucite crystals (Microbond Natural Ceramic, Aesthetic Products, Chicago, IL), we fired equal numbers of buttons (measuring 10 mm in diameter and 2 mm in height) on both platinum foil and investment material. One surface of each sample was abraded with glass beads for 20 seconds and then randomly subjected to one of the following conditions: (a) no etching, (b) etched with 52% (w/w) hydrofluoric acid for 90 seconds, and (c) etched with 20% (w/w) hydrofluoric acid (Micro-etch Solution, Aesthetic Products) for 2.5, 5, 10, and 20 minutes. After being etched, all specimens were examined with scanning electron microscopy (SEM) for determination of surface morphology. Methods of porcelain preparation used in the second part of the study were determined from this examination.

The second phase of the experiment examined the effects of various bonding conditions on shear bond strength of composite resin to etched porcelain. Forty-two porcelain buttons were fired on investment material and randomly divided into two equal groups. One group remained unetched, while the second group was etched with 20% hydrofluoric acid for 2.5 minutes. Specimens from each of these groups were further randomly divided into three equal subgroups (A,B,C and D,E,F), each consisting of seven specimens. Each specimen was mounted in a cylindrical plastic mold with a flat surface protruding 0.5 mm above the ring. In groups A and D, porcelain surfaces were coated with light-polymerizable unfilled resin (Visar Seal, Den-Mat, Santa Maria, CA). A celluloid capsule (5 Gelatin, Eli Lilly Co., Indianapolis, IN) was filled with composite resin (Ultra-Bond, Den-Mat), positioned over the center of the porcelain, and polymerized by exposure to a visible-light source for one minute. In groups B and E, porcelain surfaces were treated with a silane solution (Porcelain Bonding Agent, Den-Mat) before placement of composite. In groups C and F, porcelain surfaces were treated with silane followed by application of a dentin adhesive (Creation Bond, Den-Mat). Composite resin was placed over the center of each porcelain sample and polymerized by visible light for one minute. After being processed, all specimens were immersed in water at room temperature for seven days. Each sample was subjected to a shear load in a universal testing machine (Instron Corporation, Canton, MA) at a cross-head speed of 0.2 cm/min. The force was applied parallel to the porcelain surface close to the bonded area and a recording made of the shear load at the point of failure.

Representative samples from each of the groups were examined by SEM after mechanical testing in order to establish

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modes of failure. Additionally, the dimension of the contraction gap at the porcelain-composite interface was determined in order that the relationship between polymerization shrinkage and bond strength be explored. Using SEM at 1000 × magnification, we recorded measurements at 11 standardized points along the gap junction of a randomly selected specimen from each of the groups. Linear regression was used to correlate these data to bond strength and establish the relationship.

Analysis of data.—All data were analyzed by means of a statistical software package (SAS, Cary, NC). Homogeneity of variance was determined by Bartlett’s test. A two-way analysis of variance was performed to analyze the main effects of bonding method (unfilled resin, silane, and silane/dentin adhesive), surface condition (unetched and etched), and their interaction. Multiple comparisons were made by means of Duncan’s Test after it was determined that interaction was not significant. For all analyses, “highly significant” was defined as p<0.01, while significance was defined as p<0.05.

Results.

SEM examination of the etched porcelain groups revealed that surface configuration varied with the acid concentration, firing condition, and etching time. The porcelain fired on investment material demonstrated a relatively rougher surface texture compared with the porcelain fired on platinum foil. When exposed to 52% hydrofluoric acid for 90 seconds, the etch patterns appeared uniformly crystalline and were homologous for both porcelain firing conditions (Figs. 1A and 1B). For the porcelain fired on investment material and etched with 20% hydrofluoric acid for 2.5 minutes, an amorphous microstructure containing numerous large porosities was noted (Fig. 1C). These characteristics were not observed when the porcelain was fired on platinum foil and exposed to the same etch conditions. Rather, the surface was characterized by pores and intact islands on a relatively flat surface (Fig. 1D), suggesting less surface degradation than with the preceding condition. When the exposure to 20% hydrofluoric acid was increased to five minutes, the etch morphology of the investment fired porcelain remained amorphous. However, the large porosities observed with the shorter etching time were less frequently seen (Fig. 1E). When fired on platinum foil and with an increase in the etch time (to 20 minutes), the microstructure once again appeared amorphous, with few of the described spaces being noted (Fig. 1F). The surface configuration of the investment-fired porcelain exposed to 20% hydrofluoric acid for 20 minutes appeared similar. Based on these observations, we fired the etched porcelain used in this study on investment material and etched it for 2.5 minutes with 20% hydrofluoric acid.

For the shear bond testing, the mean values and standard deviations for all groups are reported in Table 1. Bartlett’s test was found to be significant. Two-way analysis of variance revealed that the main effects of porcelain treatment and bonding condition were highly significant, while no significance was found for their interaction (Table 2). Duncan’s test indicated highly significant differences between means of groups A through E and no differences between means of groups E and F.

The etching of porcelain significantly increased shear strength across all three bonding groups. The use of a silane coating on porcelain, when compared with unfilled resin, significantly increased the bond strength of composite in both the unetched and etched conditions. The addition of dentin adhesive to silane, however, increased bond strength only in the unetched condition. The highest bond strengths were developed when etched porcelain with silane alone or silane with dentin adhe-

sive was used. Bonds obtained with this combination exceeded the intrinsic strength of the porcelain.

Failure modes varied with the individual groups, as evidenced by SEM examination of the bonding sites after shear testing. Groups A and B consistently failed at the porcelain-composite interface. Failures in group C were primarily adhesive, with some retention of porcelain at the bonding sites. The mode of failure for Group D was generally mixed, being half cohesive within porcelain and the remainder being primarily adhesive (Fig. 2). Groups E and F had failures that were completely cohesive within porcelain (Fig. 3). These findings were entirely consistent with the bond strength developed by each of the groups.

Views of the porcelain-composite interface of groups A through F can be seen in Fig. 4. Measurement of the polymerization gap for each of the groups revealed that dimension decreased as shear bond strength increased, with this relationship being exponential (Fig. 5). Regression analysis of the measurement data indicated moderately good correlation between decreasing gap size and increasing shear bond force (r² = 0.76 at the 95% confidence interval).

Discussion.

The selection of the manipulation variables for producing the porcelain in the bond strength tests was empirically determined by high-power SEM examination of their etched surfaces. The porcelain group selected demonstrated a microstructure that appeared most conducive to the development of high bond strengths as a function of the number of large porosities contained within its amorphous surface. Clearly, resin penetration of these spaces would enhance micromechanical bonding. Although other groups may also have been able to generate high strength values, this was not specifically tested.

The distinct differences in microstructure observed with each of the concentrations of hydrofluoric acid suggest an associated preferential dissolution of one of the phases of porcelain. The glassy phase would appear to be dissolved preferentially with the 52% concentration, while the 20% form would seem to dissolve the crystalline phase preferentially.

For the 2.5-minute exposure to the 20% hydrofluoric acid, the dissolution rates for each of the porcelain groups appeared to vary with their surface configuration prior to the acid treatment. This may be related to the greater initial surface roughness of the porcelain fired on investment material, resulting in a subsequently increased physical area available for reaction. The porosities observed in the microstructure of the two types of porcelain indicate the effect of the glass-bead-abraded layer at this early time. As the etch time was increased, this layer was dissolved, resulting in the loss of these characteristics and the development of similarity in microstructure of each of the porcelain groups. This transition occurred with the five-minute acid exposure. These observations clearly suggest the importance of initial surface morphology in the development of an appropriate microstructure for bonding.

The SEM observations indicating a more optimal microstructure for the bonding of composite to porcelain using a 2.5-minute etch were consistent with the work of Calamia et al. (1985). The shear strength of composite bonded to porcelain etched for 2.5 minutes was two to three times greater than that of porcelain etched for 20 minutes. The values obtained for the shorter etching time ranged from 17.1 to 21 MPa, and were in the order of magnitude of the shear bond strengths obtained in this study.

As expected, the etching of porcelain was the most significant factor in producing high bond strengths. This can be
Fig. 1 — Micrographs of etched porcelain surfaces. A — Porcelain fired on investment, etched with 52% hydrofluoric acid for 90 seconds. B — Porcelain fired on platinum foil, etched with 52% hydrofluoric acid for 90 seconds. C — Porcelain fired on investment, etched with 20% hydrofluoric acid for 2.5 minutes. D — Porcelain fired on platinum foil, etched with 20% hydrofluoric acid for 2.5 minutes. E — Porcelain fired on investment, etched with 20% hydrofluoric acid for five minutes. F — Porcelain fired on platinum foil, etched with 20% hydrofluoric acid for 20 minutes. White bar = 100 μm.
TABLE 1
TREATMENTS, MEANS AND STANDARD DEVIATIONS (MPa) FOR SIX GROUPS

<table>
<thead>
<tr>
<th>Group</th>
<th>Treatment</th>
<th>Unfilled</th>
<th>Resin</th>
<th>Silane</th>
<th>Adhesive</th>
<th>Mean</th>
<th>SD</th>
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<tbody>
<tr>
<td>A</td>
<td>Etched</td>
<td>-</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>3.02</td>
<td>0.75</td>
</tr>
<tr>
<td>B</td>
<td>-</td>
<td>+</td>
<td>-</td>
<td>+</td>
<td>-</td>
<td>5.24</td>
<td>2.11</td>
</tr>
<tr>
<td>C</td>
<td>-</td>
<td>+</td>
<td>-</td>
<td>+</td>
<td>+</td>
<td>8.80</td>
<td>1.31</td>
</tr>
<tr>
<td>D</td>
<td>+</td>
<td>-</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>15.58</td>
<td>0.89</td>
</tr>
<tr>
<td>E</td>
<td>-</td>
<td>+</td>
<td>+</td>
<td>-</td>
<td>+</td>
<td>18.68</td>
<td>1.83</td>
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<tr>
<td>F</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>19.37</td>
<td>1.29</td>
</tr>
</tbody>
</table>

Fig. 2 — Composite resin stub from group D after failure from shear test. A portion of the porcelain substrate can be observed attached to the bonding site. SEM × 25.

Fig. 3 — Composite resin stub from group F after failure from shear test. Porcelain substrate remains attached to the entire surface of the composite. SEM.

Table 2
ANOVA SUMMARY

<table>
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<tr>
<th>Source</th>
<th>DF</th>
<th>SS</th>
<th>F</th>
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</tr>
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<td>Bonding Method</td>
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<td>160.9</td>
<td>33.0*</td>
</tr>
<tr>
<td>Interaction</td>
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</tr>
<tr>
<td>Error</td>
<td>36</td>
<td>87.7</td>
<td>33.9</td>
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</tbody>
</table>

*p<0.01.

noted from an examination of the two-way ANOVA, where the sum of squares for the surface condition is significantly greater than that of the bonding method. This clearly implicates micromechanical retention as the more significant mediator of the bond of composite resin to etched porcelain.

The use of silane as an organosilane coupler between the components of the resin-composite system is well known (Bjorksten and Jaeger, 1952; Vanderbilt and Simko, 1960; Pleudemann et al., 1962; Paffenbarger et al., 1967; Semmelman and Kulp, 1968; Pleudemann, 1982; Ferrando et al., 1983; Newman et al., 1984; Wood et al., 1986). Thus, the increase in composite resin bond strength resulting from silane use with both unetched and etched porcelain is not surprising. Although intended to work as a monolayer without adding film thickness, silane is more likely to form several monolayers which then condense and form oligomeric siloxanols. These can ultimately condense to cross-linked structures such as polymers (Pleudemann, 1982). The further increase in bond strength in the unetched porcelain group (resulting from the additional use of a dentin adhesive) may be attributable to increased wetting at the interface, thus promoting condensation of silane to the composite. The bond strength improvement produced by this combination was not observed in the etched porcelain sample, its possible effect being masked by cohesive failure of the porcelain.

The decreased dimension of the contraction gap at the porcelain composite interface for groups A to F can be related to the magnitude of bond strength observed in each of the groups. These findings are consistent with the work of Davidson et al. (1984) who, though working with dentin-composite bonds, demonstrated that polymerization contraction can be restricted by bonding forces in a unidimensional system. It would be consistent with these data to conclude that for a given unidimensional bonding system, the contraction gap would be diminished as bonding force increased. This was observed in this study. It was of further interest to note that the variance as well as the mean dimensions of the gap decreased as bond strength increased.
The results of this study indicate that composite resin bonded to etched porcelain with both silane and silane with dentin adhesive produced high shear bond strengths. Inasmuch as these values generally exceed the shear bond strength of composite to enamel, a high probability exists that these strengths will contribute to the successful clinical use of porcelain laminate...
veneers. Factors to consider in production of these high-strength values include the method of porcelain preparation, the etching procedure, and the bonding protocol.

REFERENCES


