Influence of post-etching cleaning and connecting porcelain on the microtensile bond strength of composite resin to feldspathic porcelain

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Statement of problem. There are different methods to fabricate layered feldspathic porcelain restorations, including the refractory die technique and the hot-press technique. Standard adhesive protocol for such restorations requires etching and silanating the fitting surface of the porcelain. Variations in bond strength between porcelain and composite resin might result from the different fabrication methods for porcelain restorations.

Purpose. The purpose of this study was to determine differences in microtensile bond strength between composite resin (used as a luting agent) and feldspathic porcelain generated from the refractory die technique using 2 different connecting porcelains and the hot-press technique. The effect of post-etching cleaning was also investigated.

Material and methods. Sixteen pairs of ceramic blocks (7 × 8 × 4 mm) were fabricated. Eight pairs were fabricated using feldspathic porcelain (D-B4) on refractory dies. For 4 pairs, the refractory dies were coated with a paste (Ducera Lay Connector Paste; group CON) as a connecting porcelain, and the other 4 pairs were coated with a clear translucent porcelain (CL-O; group CLO). Another 8 block pairs were fabricated using the hot-press technique (Authentic porcelain SL B00+; groups AUTH and AUTH-N). Surface conditioning of the ceramic blocks included airborne-particle abrasion followed by hydrofluoric acid etching (all groups), and post-etching cleaning with a brush/H_3PO_4 and ultrasonic bath immersion (cleaning not applied to group AUTH-N). All specimens were then silanated/heat dried, and blocks of the same porcelain were bonded to each other using an adhesive resin (Optibond FL) and a light-polymerizing composite resin (Z100). Specimens were stored in water for 24 hours. A nontrimming microtensile bond strength test was applied. Ten beams (0.9 × 0.9 × 8 mm) from each pair of blocks were selected for testing. Bond strength data (MPa) were analyzed with a Kruskal-Wallis test, and post hoc comparison was done using the Mann-Whitney U test (α=.05). Additional specimens (1 block per group) were also evaluated for the effect of conditioning steps and mode of fracture using optical microscopy and scanning electron microscopy (SEM) analysis.

Results. The mean microtensile bond strengths of CLO and AUTH groups were not significantly different from one another at 46.3 and 49.7 MPa, respectively. For both CON and AUTH-N groups, the mean bond strengths at 37.9 MPa and 24.1 MPa, respectively, were significantly different ($P<.05$) from the other 3 groups. Optical microscopy revealed a significant amount of white residue for all groups as a result of hydrofluoric etching. Cleaning with a microbrush and 37.5% phosphoric acid for 1 minute resulted in the removal of the crystalline debris. The SEM analysis of specimens cleaned by phosphoric acid brushing alone revealed microscopic deposits still contaminating the etched surface; those were efficiently removed after ultrasonic cleaning. The SEM analysis of fractured beams demonstrated a trend for more mixed-type failure in CON and AUTH-N specimens involving both the composite resin and the surface of the porcelain, whereas CLO and AUTH fractured surfaces were primarily confined to the composite resin.

Conclusions. Increased resin bond strength to refractory-generated porcelain is obtained with CLO as the connecting porcelain compared to the CON paste. The AUTH porcelain exhibited the highest mean bond strength, but omission of post-etching cleaning resulted in the lowest bond strength. (J Prosthet Dent 2006;96:354-61.)

CLINICAL IMPLICATIONS

Using a standard bonding protocol (hydrofluoric etching/post-etching cleaning/silanization/heat drying), resin-porcelain bond strength data indicate that the use of a wash of translucent porcelain to seal the refractory dies (connecting porcelain) is recommended rather than connecting paste. The leucite-reinforced heat-pressed porcelain exhibited the highest mean bond strength. Omission of a specific post-etching cleaning regimen resulted in the lowest bond strength, because hydrofluoric etching generates a significant amount of crystalline debris, thus contaminating the porcelain surface.

There are different methods used to fabricate metal-free layered feldspathic porcelain restorations, including the oldest and the most widely used—the refractory die[1]

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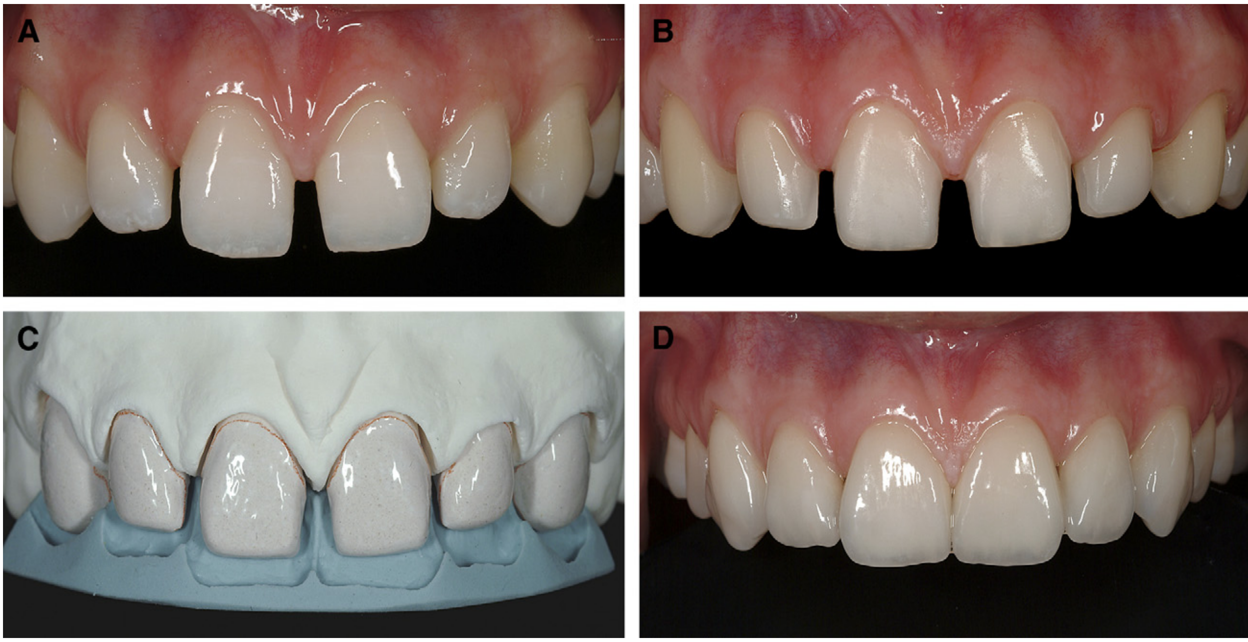

Fig. 1. A, Typical smile redesign situation requiring diastema closure. **B,** Because teeth were intact, ultraconservative tooth preparations were achieved. Refractory die technique is indicated to allow fabrication of fully layered porcelain veneers in range of 0.4- to 0.6-mm thickness. **C,** Porcelain layering is preceded by application and firing of special connecting porcelain onto removable refractory dies and careful examination of clearance with different silicone indexes from diagnostic waxing. **D,** Definitive restoration (ceramic by Michel Magne, CDT, University of Southern California, Los Angeles, Calif).

(Fig. 1), the platinum foil,[2-4] and the more recent heat-pressed ceramic techniques.[5] The primary advantages of the refractory die technique are (1) the realistic effects of color and translucency that can be obtained through a full-thickness layering technique[6-8]; (2) the reliable resin bond that can be developed through etching and chemical coupling[9]; and (3) lack of any need for special equipment. Prior to applying the porcelain, refractory dies are conditioned by surface sealing with fine-grain porcelain in the form of a paste or a wash of translucent porcelain (Fig. 1). The connecting porcelain is applied to the refractory material and fired before the porcelain is added.

Although described more than 30 years ago,[10] heat-pressed ceramics became popular during the 1990s due to the initial works of Arnold Wohlwend and Peter Scharer.[5] To obtain the most effective bond of feldspathic porcelains to tooth structure, inlays, onlays, and veneers fabricated using the refractory die technique or heat-pressed ceramics are generally placed using the same standard adhesive protocol, which is the combination of micromechanical interlocking (hydrofluoric etching) and chemical coupling (silanization).[11-13]

There are, however, less commonly described steps involved in the optimization of porcelain etching and silanization. It was demonstrated that hydrofluoric (HF) etching generates a significant amount of crystalline debris, contaminating the porcelain surface (Fig. 2), and that post-etching cleaning using an ultrasonic bath proved to be essential to enlarge and enhance access to the undercuts generated by etching.[14-16] The effect of this cleaning on microtensile bond strength (MTBS) has not been investigated. Another significant optimization step is drying the silane layer with the application of heat.[9,17,18] This procedure is not always included in the standard adhesive protocol, even though it has been shown to significantly increase the bond strength between resin cement and porcelain.[9,17,18]

The purpose of this study was to determine whether there were differences in MTBS between composite resin cement and feldspathic porcelain generated from the refractory die technique using 2 different connecting porcelains (paste versus regular translucent porcelain) and the hot-press technique. The effect of a 2-stage post-etching cleaning was also measured and observed under optical and scanning electron microscopes.

MATERIAL AND METHODS

Porcelain specimen fabrication

Sixteen pairs of ceramic blocks ($7 \times 8 \times 4$ mm) were fabricated (Fig. 3). Eight block pairs were fabricated using feldspathic porcelain (D-B4, Creation; Jensen Industries, North Haven, Conn) on refractory dies (Orbit Vest; GC America, Alsip, Ill). The dies were obtained by duplicating stone blocks with silicone (Elite Double 22; Zhermack, Eatontown, NJ). For 4 block

Fig. 2. A, Hydrofluoric acid etching of feldspathic porcelain typically generates white residue, contaminating etched surface, which may be confused with well-etched porcelain. **B,** SEM analysis (original magnification ×1200) reveals that deposits may potentially prevent penetration of bonding resin into ceramic undercuts.

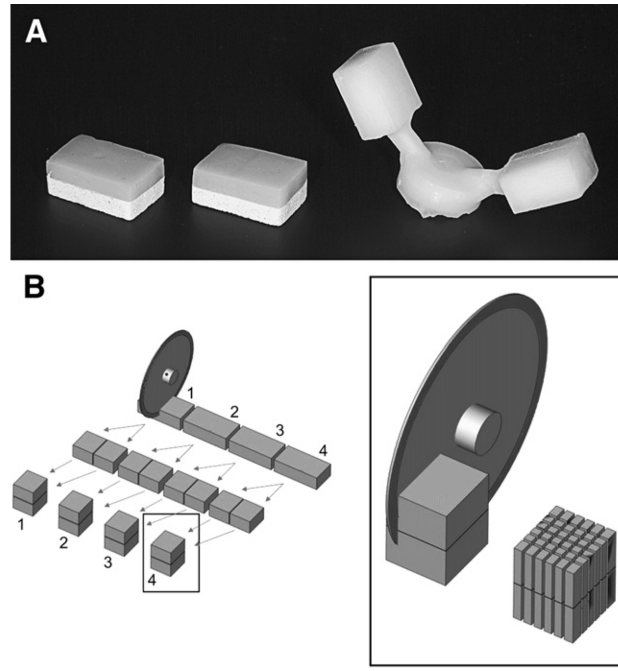

Fig. 3. A, Ceramic blocks on original refractory dies *(left)* and as obtained after removing investment material in hot-press technique *(right)*. **B,** Four blocks were fabricated for each experimental group, each block producing a bonded pair *(left)*, and each bonded pair being vertically sectioned into 0.9-mm-thick beams *(right)*.

pairs (Group CON), the refractory dies were coated with a paste (Ducera Lay Connector Paste; Dentsply Ceramco, Burlington, NJ) as the connecting porcelain, and the other 4 pairs of blocks (Group CLO) were treated with a clear translucent porcelain (CL-O, Creation; Jensen Industries). Another 8 pairs (4 pairs each for groups AUTH and AUTH-N) were fabricated using a hot-press ceramic (SL B00+, Authentic; Ceramay, Stuttgart, Germany) and the lost-wax technique. The firing schedule followed the manufacturers' recommendations. For both refractory and hot-press techniques, the blocks were cleaned by removing the refractory material with airborne-particle abrasion with 80-μm glass beads at 22 psi.

Bonding procedures

Surface conditioning for all groups included airborne-particle abrasion with 30-μm aluminum oxide at 22 psi, followed by 9% hydrofluoric acid etching (Porcelain Etch; Ultradent, South Jordan, Utah) for 90 seconds, and rinsing with water for 20 seconds. Specimens were then subjected to post-etching cleaning using a microbrush and 37.5% phosphoric acid (Ultra-Etch; Ultradent) with a gentle brushing motion for 1 minute, followed by rinsing with water for 20 seconds. Cleaning was completed by immersion in distilled water in an ultrasonic bath for 5 minutes. The post-etching cleaning was not applied to group AUTH-N.

Following thorough oil-free air drying, all surfaces were then silanated (Silane; Ultradent) and dried at 100°C for 5 minutes in an oven (DI500; Coltene/Whaledent, Cuyahoga Falls, Ohio). Four pairs of blocks of the same type of porcelain were bonded together using a coat of adhesive resin (Optibond FL Adhesive; Kerr Corp, Orange Calif) on each fitting surface and a light-polymerizing composite resin (Z100; 3M ESPE, St. Paul, Minn) as a luting agent. A constant force of 1 kg was applied to the blocks for 10 seconds before light polymerizing for 160 seconds (40 seconds per surface, 4 times) at 600 mW/cm^2 (Demetron LC; Kerr Corp). The summary of group characteristics is presented in Table I. Four additional blocks (1 block for each group) were fabricated to study the effect of etching and post-etching cleaning procedures with optical and scanning electron microscopy (SEM).

Table I. Mean (SD) and median and interquartile ranges (IQR) for microtensile bond strength of Optibond FL/Z100 to porcelains with varying fabrication techniques and precementation treatments

Group	Fabrication technique	Connecting porcelain	Post-etching cleaning procedure*	Mean MTBS for each block pair†	MTBS Mean (SD)	MTBS Median (IQR)
CON‡	Refractory die (Orbit Vest; GC America)	Connecting paste (Connector; Dentsply Intl)	Brush with H_3PO_4 + ultrasonic bath	1: 33.7 2: 34.7 3: 41.2 4: 42.2	37.9 (4.4)^b	38.0 (7.0)
CLO‡	Refractory die (Orbit Vest; GC America)	Clear translucent (CL-O; Creation)	Brush with H_3PO_4 + ultrasonic bath	1: 47.0 2: 45.8 3: 43.8 4: 48.6	46.3 (2.0)^a	46.4 (2.1)
AUTH	Heat-pressed (Authentic; Ceramay)	Not applicable	Brush with H_3PO_4 + ultrasonic bath	1: 56.2 2: 62.5 3: 44.2 4: 35.9	49.7 (11.9)^a	50.2 (15.7)
AUTH-N	Heat-pressed (Authentic; Ceramay)	Not applicable	None	1: 27.8 2: 25.6 3: 25.7 4: 17.3	24.1 (4.6)^c	25.6 (2.7)

Mean values with different superscript letter are significantly different at $P<.05$.
*All specimens HF etched.
†All specimens silanated and heat dried before bonding.
‡Build-up porcelain = D-B4, Creation.

Preparation for microtensile bond strength testing

All bonded specimens were stored in distilled water at room temperature for 7 days before testing. Each specimen was individually secured with sticky wax (GEO Cervical; Renfert, St. Charles, Ill) to a transparent plastic sectioning block. Using the nontrimming technique developed by Shono et al[19] (Fig. 3, *B*), multiple beams were prepared, with the ceramic-to-ceramic adhesive interface in the center of the beam. To do so, specimens were vertically sectioned into 0.9-mm-thick slabs using a low-speed diamond saw (Isomet; Buehler Ltd, Lake Bluff, Ill). The slabs were sectioned again into beams with approximately 0.81-mm^2 cross-sectional areas. Forty beams were prepared from each experimental group by selecting 10 beams per block pair. The specimens were attached to a table-top material tester (The Micro Tensile Tester; Bisco, Schaumburg, Ill) using cyanoacrylate (Zapit; Dental Ventures of America, Corona, Calif) and subjected to microtensile testing at a crosshead speed of 5.4-kg force per minute. After testing, the failure mode of each beam was determined under a stereoscopic microscope (×80). Failures were classified as an interfacial failure if the fracture site was located entirely between the adhesive and porcelain or if the fracture site continued from the adhesive into either the composite resin or porcelain, and as a substrate failure if the fracture occurred exclusively within the porcelain. Bond strength data obtained from the 4 experimental groups were analyzed with a Kruskal-Wallis test, with each block pair (mean MTBS from the 10 beams) used as a single measurement, yielding 4 measurements per group (Table I). The use of the nonparametric Kruskal-Wallis test was justified by the small sample size. Statistical significance was set in advance at α=.05. Post hoc comparisons were performed using the Mann-Whitney U test and were performed without adjusting for the multiple comparisons; thus, the potential for indicating that a difference exists, when in fact there is no difference, increased (type I error). Adjusting for the multiple comparisons using the Bonferroni correction, however, would have increased the chance of a type II error (no effect or difference declared, when in fact there is an effect).

Table II. Distribution of failure modes as observed by optical microscopy

	CON	CLO	AUTH	AUTH-N
Interfacial (%)	100	55	90	100
Ceramic substrate (%)	0	45	10	0

Optical and scanning electron microscopy

The failed surfaces of 4 fractured beams (interfacial failure) from each group were air dried, sputter coated with gold/palladium (Ernest Fullam, Schenectady, NY), and examined using SEM (Cambridge 360; Carl Zeiss, Thornwood, NY). One block from each group was also used for surface analysis of the different conditioning steps (hydrofluoric etching, cleaning) with optical microscopy and SEM.

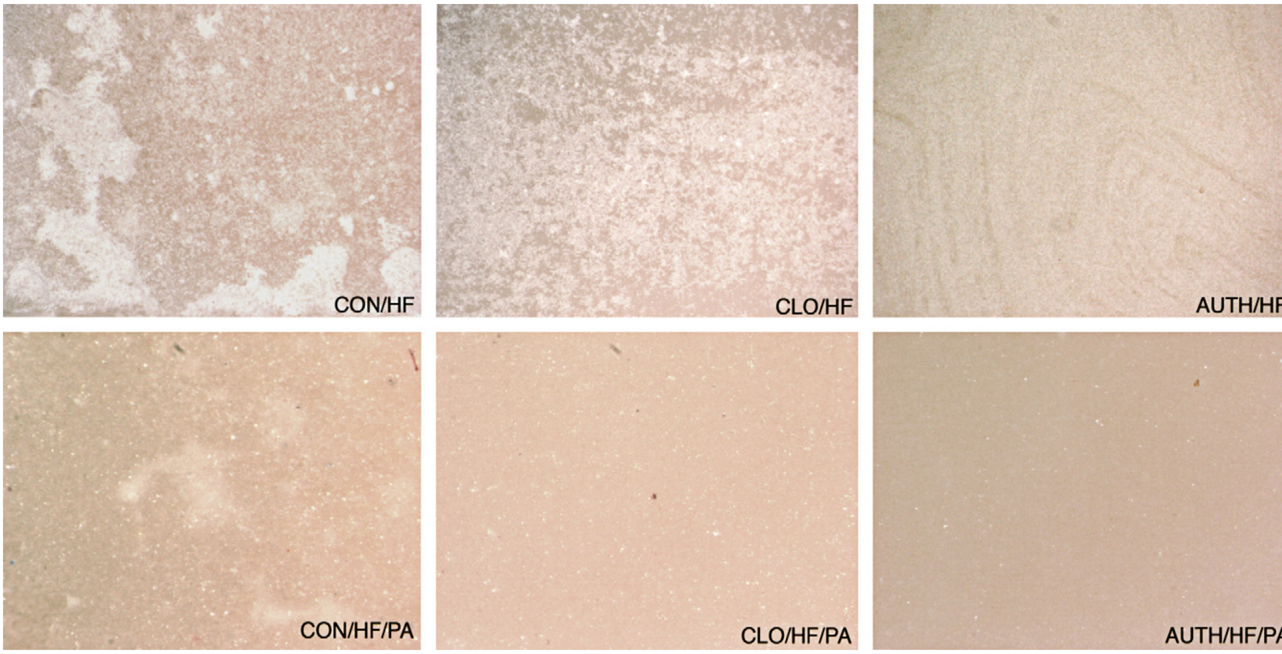

Fig. 4. Typical optical microscope views (original magnification ×100) of porcelain surfaces following HF etching *(upper row)*. Note intense agglomerate-contaminating specimen CON *(left)* compared to more diffuse residues left on specimen CLO *(center)* and even more diffuse and less visible chalky layer on specimen AUTH *(right)*. Whitish remnants cleaned by brushing with 37% phosphoric acid for 1 minute *(bottom row)*. Note, however, some remaining lighter areas on specimen CON *(left)*.

RESULTS

Microtensile bond strength

Table I lists the MTBS of Optibond FL (Kerr Corp) and Z100 (3M ESPE) to porcelain in the 4 experimental groups. The mean MTBS varied from 24 to 49 MPa. The Kruskal-Wallis test indicated a significant difference among the 4 groups (P=.008). The Mann-Whitney U test applied to the CLO and AUTH groups did not show a significant difference (P=.386). The mean bond strength of the group CON (37.9 MPa) was significantly lower (P=.010) than that of groups CLO and AUTH (46.3 and 49.7 MPa, respectively), and the mean bond strength of group AUTH-N (24.1 MPa) was significantly lower (P=.010) than that of all other groups.

Optical and scanning electron microscopy

Results of the failure modes determined by optical microscopic evaluation are shown in Table II. Failures were either interfacial or cohesive for the CLO group, whereas almost all failures in the other groups were interfacial. Optical microscopy revealed a significant amount of white residue for all groups as a result of HF etching (Fig. 4, *top row*). The residue, however, occurred systematically with a specific pattern related to the connecting porcelain: dense agglomerates for CON, diffuse agglomerates for CLO, and diffuse layer with a wave pattern for AUTH. Cleaning with a microbrush and 37.5% phosphoric acid for 1 minute resulted in the removal of the white residue, except in CON group, where whitish areas were still visible after cleaning (Fig. 4, *bottom row*). Even when all white residues were removed, SEM analysis of specimens cleaned only with phosphoric acid brushing revealed microscopic deposits (approximately 100 nanometers) still contaminating the etched surface (Fig. 5, *top row*). Such remnants were efficiently removed after ultrasonic cleaning (Fig. 5, *bottom row*). The SEM analysis of fractured beams demonstrated a trend for more mixed-type failure in CON and AUTH-N specimens, involving both the composite resin and the surface of the porcelain (Fig. 6, *top*), whereas CLO and AUTH fractured surfaces were primarily confined to the composite resin (Fig. 6, *bottom*).

DISCUSSION

The use of the refractory die technique, rather than the platinum foil technique, in the present study was motivated by the inherent versatility and practicality of this method.[8] Data from the early 1990s[2-4] have shown the superior marginal fidelity of platinum foil veneers. These results might have lost their relevance since the introduction of improved refractory materials, such as Orbit Vest (GC America) and use of smaller individual dies.[6-8] The disadvantage of the platinum foil technique is that casts must be prepared by previous trimming of

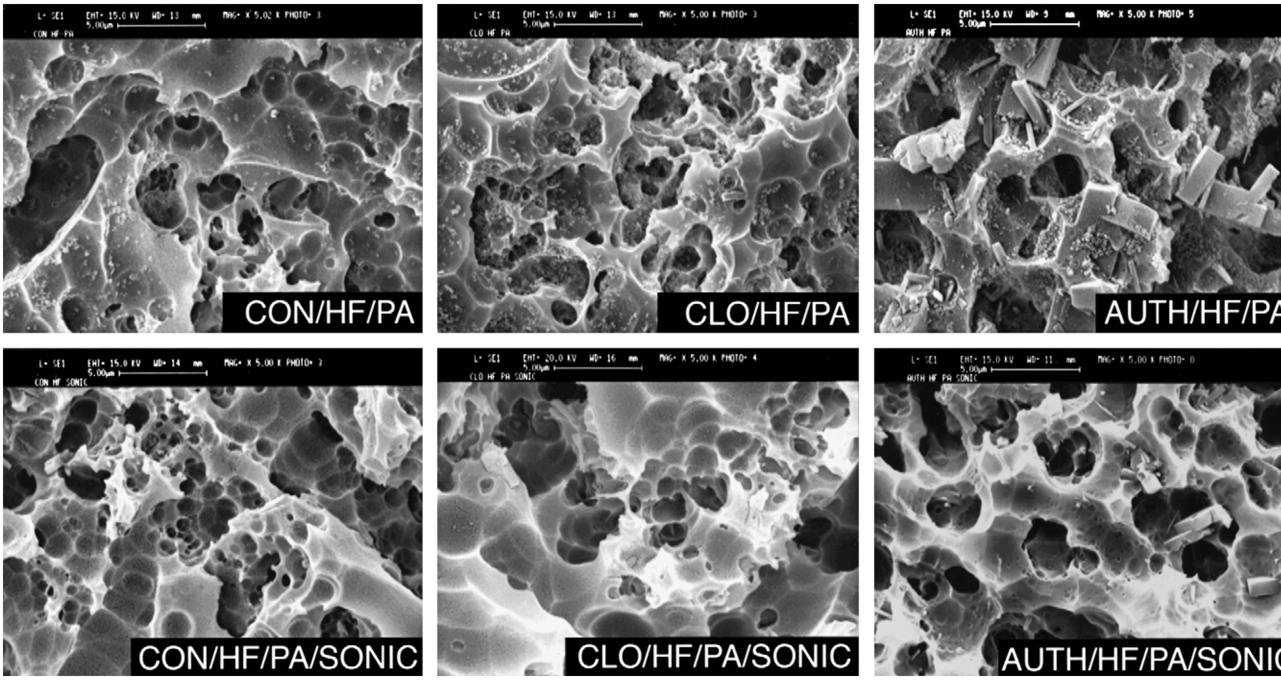

Fig. 5. Typical SEM micrographs (original magnification ×5000) of porcelain surfaces following HF etching and phosphoric acid cleaning *(upper row)*. Note small remnants still contaminating etched surface. All residue was removed following ultrasonic immersion in distilled water for 5 minutes *(lower row)*.

the gingival portion of the stone cast. Newer methods with refractory dies allow maintaining the gingival stone (Fig. 1), which is a major element to guide the stratification and elaboration of fine ceramic contours and gingival emergence profile.

Bonding procedures are applied to the fitting surface of the porcelain. In the refractory die technique, the fitting surface is represented by the connecting porcelain used to seal the dies before the addition of ceramic. According to the present study, the latter seems to be a critical element for bond strength, as demonstrated by the difference between the 2 connecting materials: the connecting paste did not perform as well (37.9 MPa) as the traditional translucent porcelain (46.3 MPa). Figure 6 suggests the intrinsic weakness of the paste used in group CON (interfacial ceramic-adhesive failure Mode 2, according to Della Bona et al[20]), whereas failures in groups CLO and AUTH involved primarily the luting composite (interfacial ceramic failure Modes 3-5, according to Della Bona et al[20]). Although the present study shows that the regular translucent porcelain can be recommended because of its improved bond strength, the application of the premixed "ready-to-use" connecting paste appears to be more versatile, less technique sensitive, and allows better control of thickness.

In heat-pressed ceramics, the fitting surface does not involve the use of a connecting material but consists of the core material itself. This substrate can be efficiently conditioned by both acid etching and silanization, as demonstrated by the high bond strength of group AUTH at 49.1 MPa. The present work provides the first data collection regarding the resin bond strength to the Authentic leucite-reinforced porcelain (Ceramay). It is interesting to note that these results do not match the bond strength obtained by Della Bona et al[20,21] with another brand of leucite-based heat-pressed ceramic (IPS Empress 1; Ivoclar Vivadent, Schaan, Liechtenstein) at a maximum of 27.2 MPa. Resin bonding to IPS Empress 1 proved to be dominated by chemical coupling alone (silane) and was significantly weakened by the acid application. In the same study, the heat-pressed lithia-based ceramic (IPS Empress 2; Ivoclar Vivadent) demonstrated an MTBS of 56.1 MPa by combining HF etching and silane. Interestingly, HF acid conditioning is unable to generate a retentive surface on highly crystalline ceramics with reduced glassy content such as InCeram (VITA Zahnfabrik; Bad Sackingen, Germany) or Procera (Nobel Biocare, Yorba Linda, Calif), or pure noncrystalline ceramics such as Duceram LFC hydrothermal glass (Dentsply Intl, York, Pa).[22] Additional steps are required for some of these products to generate a positive mechanical interlocking, such as the sintering of silica particles.[22] All of the aforementioned examples emphasize the fact that the tensile fracture resistance of the resin-ceramic adhesion zones is controlled primarily by microstructure and surface treatment of the ceramic. Each new material or product must, therefore, be studied individually to define the optimal bonding protocol.

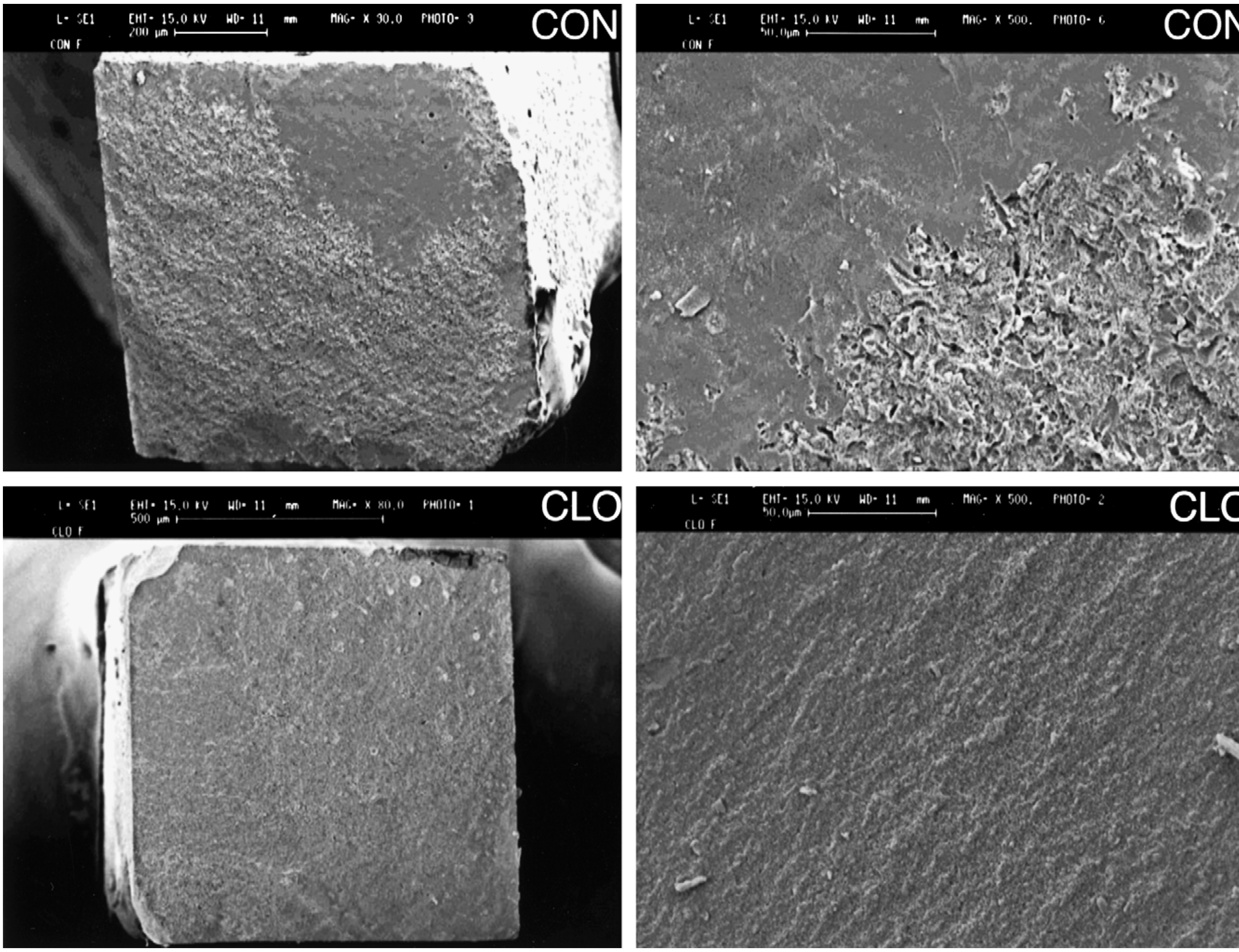

Fig. 6. Typical SEM micrograph of fractured beam from CON group *(upper row),* which failed at 37.8 MPa. Note mixed interfacial failure, primarily involving porcelain and, in part, filled adhesive/composite resin *(top left,* original magnification ×90). Higher magnification of porcelain-composite transition area *(top right,* original magnification ×2000). Typical SEM micrograph of fractured beam from CLO group *(lower row),* which failed at 62.14 MPa. Note clean and uniform interfacial failure mainly involving composite resin *(bottom left,* original magnification ×90). Higher magnification *(bottom right,* original magnification ×2000).

Another significant finding of the present study is related to the post-etching cleaning regimen, tested for the first time using the microtensile test. The absence of post-etching cleaning resulted in a reduction of bond strength of approximately 50% (24.1 MPa for AUTH-N versus 49.7 MPa for AUTH). The energy dispersive spectroscopy analyses have shown that the crystalline precipitates on the etched surfaces, which were not readily soluble in water (Fig. 4), were the reaction products of Na, K, Ca, and Al.[16] It has been reported that the precipitates remain on the surface after acid application and can only be removed by ultrasonic cleaning and not by rinsing.[16] Optical microscopy showed that most of these deposits can also be eliminated by brushing with phosphoric acid alone (Fig. 4). However, residue remained, contaminating the retentive structure (Fig. 5, *top row*). This can be eliminated by placing the restoration in distilled water or 95% alcohol (or acetone) in an ultrasonic bath for 4 to 5 minutes (Fig. 5, *bottom row*).[14] To avoid further problems of contamination, the final trial insertion of the restoration must always precede HF etching and silanization.[23] The emphasis should be on HF cleaning and silanization immediately prior to cementation.

In all experimental groups, the etched and cleaned porcelain was treated with a silane solution. Because of the silica content of feldspathic porcelains, a chemical bond can be potentially achieved between the porcelain and the luting resin. This bond requires coupling molecules, such as γ-methacryloxypropyl trimethoxysilane, also called *organofunctional silanes.*[12] They are typically used as adhesion promoters between inorganic substrates and organic polymers. Silane-treated porcelain offers an improved wettability as well as methacrylate groups that can form a bond with the methacrylate groups in the resin.[12] Drying the restoration by applying heat to evaporate the silane solvents proved to significantly enhance the bond strength.[9,17,18] That is the reason this procedure was included in the standard bonding protocol for all groups in the present study.

Further studies are indicated to develop a connecting porcelain for the refractory die technique that would combine bond strength, versatility, and ease of use. Meanwhile, the use of regular translucent porcelain such as the C-LO (Creation) as a refractory die sealer can be recommended because of its superior bond strength over the existing connecting paste (Ducera Lay Connector Paste; Dentsply Ceramco).

CONCLUSIONS

The present study determined that there were significant differences in MTBS between composite resin and feldspathic porcelain generated from the refractory die technique using 2 different connecting porcelains (paste vs regular translucent porcelain) and the hot-press technique. The following conclusions reflect the products tested in this study.

Using a standard bonding protocol (HF etching/post-etching cleaning/silanization/heat drying), the results indicate the following:

1. To maximize the bond strength to refractory generated porcelain, it is recommended a wash of translucent porcelain be used to seal the refractory dies (connecting porcelain) rather than connecting paste.
2. The leucite-reinforced heat-pressed porcelain exhibited the highest mean MTBS (49.7 MPa). Omission of the post-etching cleaning regimen resulted in more than 50% loss of bond strength (24.1 MPa) because HF etching generates a significant amount of contaminating debris. Both brushing with phosphoric acid and immersion in the ultrasonic bath proved necessary to remove the crystalline debris from the etched surface.

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