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Laboratory Research

The Influence of Surface Standardization of Lithium Disilicate Glass Ceramic on Bond Strength to a Dual Resin Cement

R Brum • R Mazur • J Almeida
G Borges • D Caldas

Clinical Relevance

Laboratory bond strength tests using standardized ground flat ceramic surfaces can be predictive of clinical situations where ceramic surfaces are irregular.

SUMMARY

In vitro **studies to assess bond strength between resins and ceramics have used surfaces that have been ground flat to ensure standardization; however, in patients, ceramic surfaces are irregular. The effect of a polished and unpolished ceramic on bond strength needs to be investigated. Sixty ceramic specimens (20×5×2 mm) were made and divided into two groups. One group was ground with 220- to 2000-grit wet silicon carbide paper and polished with 3-, 1-, and ¼-μm diamond paste; the other group was neither ground nor polished. Each group was divided into three subgroups: treated polished controls (PC) and untreated unpolished controls (UPC), polished (PE) and unpolished specimens (UPE) etched with hydrofluoric acid, and polished (PS) and unpolished specimens (UPS) sandblasted with alumina. Resin cement cylinders were built over each specimen. Shear bond strength was measured, and the fractured site was analyzed. Analysis of variance (ANOVA) and Tukey *post hoc* tests were performed. PE (44.47 ± 5.91 MPa) and UPE (39.70 ± 5.46 MPa) had the highest mean bond strength. PS (31.05 ± 8.81**

*Rafael Torres Brum, MSc, School of Dentistry, Graduate Program, Pontiphical Catholic University of Paraná, Curitiba, Brazil

Rui Fernando Mazur, PhD, School of Dentistry, Graduate Program, Pontiphical Catholic University of Paraná, Curitiba, Brazil

Janaína Bertoncelo de Almeida, PhD, School of Dentistry, Graduate Program, Pontiphical Catholic University of Paraná, Curitiba, Brazil

Gilberto Antonio Borges, PhD, School of Dentistry, Graduate Program, University of Uberaba, Uberaba, Brazil

Danilo Biazetto de Menezes Caldas, PhD, School of Dentistry, Graduate Program, Pontiphical Catholic University of Paraná, Curitiba, Brazil

*Corresponding author: 1155, Prado Velho, Curitiba, Paraná, Brazil 80-915-901; e-mail: rafaeltbrum@yahoo.com.br

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MPa), UPC (29.11 ± 8.11 MPa), and UPS (26.41 ± 7.31 MPa) were statistically similar, and PC (24.96 ± 8.17 MPa) was the lowest. Hydrofluoric acid provides the highest bond strength regardless of whether the surface is polished or not.

INTRODUCTION

Ceramic restorations have a high degree of crystallization to enhance their mechanical properties.[1,2] Pressable lithium disilicate-based glass ceramic is a partially crystallized glass obtained from the controlled growth and nucleation of crystals in the glassy phase. It can be defined as a ceramic composed of one glassy phase and at least one crystalline phase.[3] It has been shown to possess remarkable mechanical properties and is indicated for use in veneers, inlays, onlays, full crowns, and three-unit bridges for the anterior and premolar region.[4,5]

The internal surface of an adhesive ceramic restoration must be treated to promote the bond with the resin cement. Two adhesive procedures are commonly described: 1) mechanical adhesion promoted by acid etching or abrasion of the ceramic surface by means of airborne particles and 2) chemical bonding promoted by a silane agent.[6]

Hydrofluoric acid etching changes the surface of the glass ceramic by dissolving its glassy phase.[7] This process creates irregularities on the surface and increases the contact area between the adhesive system and the glass ceramic. Rough surfaces increase mechanical retention by enabling the adhesive to interlock with the surface irregularities created by the hydrofluoric acid.[8] Airborne particle abrasion with 50-μm aluminum oxide particles changes the ceramic surface, increasing mechanical retention.[9]

The surfaces of pressable glass-ceramic restorations are rough and thus provide a degree of inherent mechanical retention. The standardization of the surfaces of ceramic materials with silicon carbide paper (SiC) and diamond pastes in many previous studies[4,10-18] was intended to remove any preexisting mechanical retention that could interfere with bond-strength tests. This treatment produced a flat, polished ceramic surface that enabled the conditioning effect of hydrofluoric acid and the abrasive effect of sandblasting with 50-μm aluminum oxide on the bond strength between the resin cement and glass ceramic to be determined separately. However, a completely flat and polished ceramic surface does not reflect the actual clinical situation in the dental office, in which a glass ceramic is adhesively luted to a prepared tooth. Further studies are required to establish the correlation between the findings of laboratory tests of surface treatments of glass ceramics and their clinical application.

The aim of this study was therefore to evaluate the effect of different surface treatments on the bond strength between a heat-pressed lithium disilicate glass ceramic, both polished and unpolished, and a dual-polymerizing resin cement. The tested null hypothesis was that surface treatment would not lead to any significant difference in bond strength between glass ceramic and resin cement.

MATERIALS AND METHODS

Sixty rectangular glass ceramic specimens 20 mm long, 5 mm wide, 2 mm thick, and shade 300 were made with the IPS Empress 2 system (Ivoclar Vivadent, Schaan, Liechtenstein; basic composition: SiO_2 [57%-80%], Al_2O_3 [0%-5%], La_2O_3 [0.1%-6%], MgO [0%-5%], ZnO [0%-8%], K_2O [0%-13%], Li_2O [11%-19%], and P_2O_5 [0%-11%]) using the lost-wax technique in accordance with the manufacturer's instructions.

Half the specimens were not polished, and the other half were ground sequentially on one of their sides with 220-, 320-, 360-, 400-, 500-, 600-, 1200-, 1500-, and 2000-grit wet silicon carbide paper and then polished with 3-, 1-, and ¼-μm[4,6,7,11] diamond paste (Arotec, São Paulo, SP, Brazil) under continuous and abundant water cooling.

The specimens were embedded in an autopolymerizing acrylic resin (Jet, Artigos Odontológicos Clássico, São Paulo, SP, Brazil) using a plastic mold that left only the surface to be tested exposed. Adhesive tape (3M, Sumaré, SP, Brazil) with three circular holes 5 mm apart and an internal area of 1.32 mm^2 each was placed on the exposed surface. This method ensured that the area to which the adhesive was applied was limited to the inside of the circular holes, preventing possible interference from excess adhesive beyond the edge of the bond area, which could lead to artificially high adhesive values.[11]

The polished (P) and unpolished (UP) groups were divided into a total of six subgroups, two of which constituted the positive and negative controls. The polished control (PC) and unpolished control (UPC) groups did not receive any additional surface treatment. The polished etched (PE) and unpolished

etched (UPE) groups were etched with 10% hydrofluoric acid (FGM, Joinville, SC, Brazil) in the exposed areas for 20 seconds, washed with air/water spray for 60 seconds, and air-dried. The polished sandblasted (PS) and unpolished sandblasted (UPS) groups were subjected to abrasion using 50-μm aluminum oxide airborne particles at a pressure of 4 bar for 5 seconds and a distance of 10 mm, adjusted with a special device made with condensation silicone (Zetaplus, Zhermack, Badia Polisene, Rovigo, Italy), and then washed with an air/water spray for 60 seconds and dried completely with an air spray. Table 1 summarizes the treatments for the groups and subgroups.

Following this, a silane coupling agent (Monobond S, Ivoclar Vivadent) was applied to the ceramic surfaces for 60 seconds and dried with an air spray. Scotchbond Multi-Purpose hydrophobic adhesive (3M/ESPE Dental Products Division, St Paul, MN, USA) was then applied to the ceramic surfaces inside the three holes and light-cured for 20 seconds with a curing light with an output of 400 mW/cm^2 (Optilux 500, Demetron, Sybron Dental Specialties Inc, Orange, CA, USA). The power density was checked with a built-in radiometer while the specimens were being prepared. The ceramic specimens were then ready to receive the resin cement cylinders (Variolink II, Ivoclar Vivadent).

Plastic colorless tubes 1.3 mm in diameter and 1 mm high were used to produce the resin cement cylinders. These were positioned before the hydrophobic adhesive was light-activated so that their walls could be aligned to the three holes in the adhesive tape. After the hydrophobic adhesive had been cured, each tube was kept in position until the resin cement had been inserted with a Centrix Snap-Fit^{TM} syringe (Centrix, Shelton, CT, USA) and light-cured for 60 seconds following the manufacturer's instructions. Each ceramic surface was thus bonded at three different locations with cylinders made out of adhesive cement. The ceramic/resin luting agent assemblies were stored at room temperature (23°C ± 2°C) for 24 hours, after which the plastic tubes were removed and the specimens immersed in distilled water at 37°C for 24 hours. The diameters of each of the 180 resin cement cylinders were then inspected using a digital caliper (model CD6 CS, Mitutoyo, Kanagawa, Japan). The average area measured was 1.3 ± 0.1 mm.

All the resin cement cylinders (180) were analyzed using an optical microscope with a magnification of 30× to identify whether there were any interfacial gaps, bubbles, or other defects in the ceramic/resin luting agent assemblies.[19,20] Specimens with defects were replaced. All the specimens were then thermally cycled 500 times in 5°C ± 1°C and 55°C ± 1C° water baths, with a dwell time of 60 seconds in each bath and a 15-second transfer time. The shear bond test was carried out on 30 ceramic/resin luting agent assemblies for each subgroup tested (n=30). The specimens were positioned using a special device in a universal testing machine (DL3000, EMIC, São José dos Pinhais, PR, Brazil), and a thin 0.30-mm-diameter wire (Morelli, Sorocaba, SP, Brazil) was looped around the resin cement cylinder so that it made contact along half its circumference and gently held flush against the resin/ceramic interface. A shear force was applied to each specimen at a crosshead speed of 1 mm/min until failure occurred. The resin/ceramic interface, wire loop, and center of the load cell (20 kgf) were aligned as straight as possible to ensure the desired orientation of the shear test force.[20]

Finally, all the specimens were gold-coated with a sputter coater (SCD 050, Balzers, Balzers Union Aktiengeselischaft Fuürstentun, Liechtenstein) for 180 seconds at 40 mA and examined by the same operator in a scanning electron microscope (SEM; (JSM-6360-LV, Jeol, Tokyo, Japan). The areas (pixels) for each type of failure based on images taken at the same magnification (50×) were measured using the ImageTool program (Department of

Table 1: *Groups and subgroups according to surface treatment*

Groups	**Subgroups**	**Surface Treatment**
Polished (P)	Control (PC)	Without additional treatment
	Etched hydrofluoric acid (PE)	10% hydrofluoric acid for 20 seconds
	Sandblasted aluminum oxide (PS)	Abrasion with 50-μm alumina
Unpolished (UP)	Control (UPC)	Without additional treatment
	Etched hydrofluoric acid (UPE)	10% hydrofluoric acid for 20 seconds
	Sandblasted aluminum oxide (UPS)	Abrasion with 50-μm alumina

Table 2: *Mean bond strength (MPa) according to the initial state of the surface*

Surface treatment	n	Mean (MPa)[a]	Standard Deviation
Polished	85	33.99 A	11.16
Unpolished	90	31.74 A	9.05

[a] *Five pretest failures occurred during thermal cycling in the polished group. The same letters represent statistically similar groups.*

Table 3: *Ranking of bond strength values (MPa)*

Surface treatment	n	Mean (MPa)[a]	Standard Deviation
Polished etched (PE)	30	44.47 A	5.91
Unpolished etched (UPE)	30	39.70 A	5.46
Polished sandblasted (PS)	30	31.05 B	8.81
Unpolished control (UPC)	30	29.11 B,C	8.11
Unpolished sandblasted (UPS)	30	26.41 B,C	7.31
Polished control (PC)	25	24.96 C	8.17

[a] *Five pretest failures occurred during thermal cycling in the polished group. The same letters represent statistically similar groups.*

Table 4: *Percentage failures by failure type as analyzed by SEM*[a]

Surface treatment	Type A (%)	Type B (%)	Type C (%)
Polished etched (PE)	15.70	6.80	77.50
Unpolished etched (UPE)	13.40	7.57	79.03
Polished sandblasted (PS)	58.03	4.00	37.97
Unpolished sandblasted (UPS)	59.23	4.67	36.10
Polished control (PC)	91.03	8.97	0.00
Unpolished control (UPC)	57.93	11.50	30.57

[a] *Type A, adhesive failure between the glass ceramic and resin cement; type B, cohesive failure in the resin cement; type C, cohesive failure in the glass ceramic.*

Dental Diagnostic Science, University of Texas Health Science Center, San Antonio, Texas, USA) and identified as 1) adhesive failure between the glass ceramic and the resin cement, 2) cohesive failure in the resin cement, and 3) cohesive failure in the glass ceramic. Two additional specimens—one polished and one unpolished—were made for each of the six subgroups. These were then gold-coated and observed in the SEM to evaluate the effect of each surface treatment. The data were analyzed using two-way ANOVA, and multiple comparisons were made using the Tukey test. Statistical significance was set at α=0.05.

RESULTS

The mean bond-strength values for the polished and unpolished ceramic surfaces are shown in Table 2. Two-way ANOVA revealed that there were no significant differences in bond strength between the polished and unpolished ceramic surfaces but that there was a significant difference in bond strength between the two surface treatments (etching and air abrasion). Power analysis for the groups yielded values of 1.00 and 0.913, respectively. Multiple comparisons for the Tukey HSD test are shown in Table 3. The percentages of each type of failure are given in Table 4. There was a high percentage of type C failures among the ceramic surfaces etched with hydrofluoric acid, and a high percentage of type A failures among the ceramic surfaces submitted to the other surface treatments.

DISCUSSION

A critical question that remained unanswered before this study was carried out was whether an extremely smooth surface on glass ceramic could affect the strength of the bond between this material and adhesive cement. Figure 1 shows the polished ceramic surface without any additional mechanical retention, while Figure 2 shows the unpolished ceramic with mechanical retention similar to that achieved at the end of the laboratory procedures visible on the surface.

In view of the statistical similarity between the bond strength in the polished group and that in the unpolished group, the first part of the null hypothesis was confirmed. This result suggests that polishing the ceramic surface does not affect bond strength and is therefore a suitable procedure for eliminating any mechanical interference caused by the ceramic surface in bond-strength tests. Despite the absence of a polished dental surface in a clinical

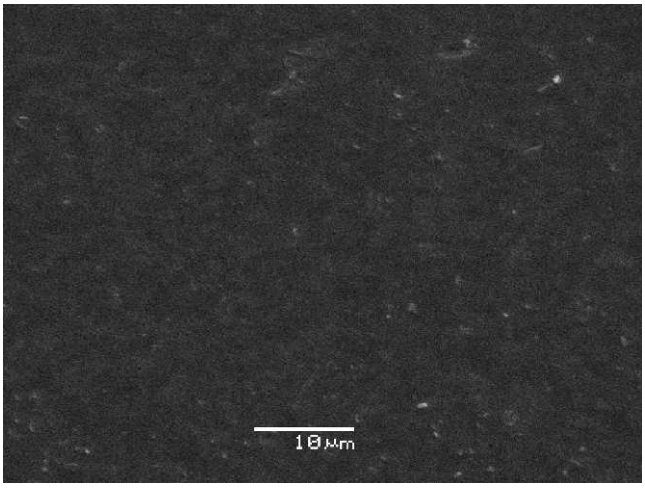

Figure 1. *SEM. Polished control surface. 2000×. No micromechanical retention.*

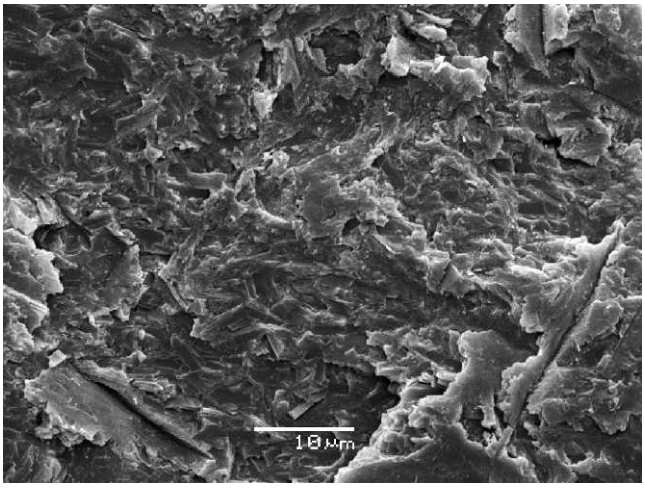

Figure 2. *SEM. Unpolished control surface. 2000×. Surface irregularities providing limited micromechanical retention.*

setting, these results can be usefully applied in clinical practice.

The high degree of crystallization found in glass ceramic results in improved mechanical properties.[1-3] According to the manufacturer's literature, IPS Empress 2 (Ivoclar Vivadent) is based on one main crystalline phase with elongated lithium disilicate crystals, a secondary crystalline phase with lithium orthophosphate, and a glass phase surrounding the crystalline phase. Hydrofluoric acid reacts with the glass and secondary crystalline phases, creating surface irregularities and increasing the microretentive surface area into which the bonding agent can flow, thus optimizing the interaction between the resin bonding agent and glass ceramic.[6,8] In Figures

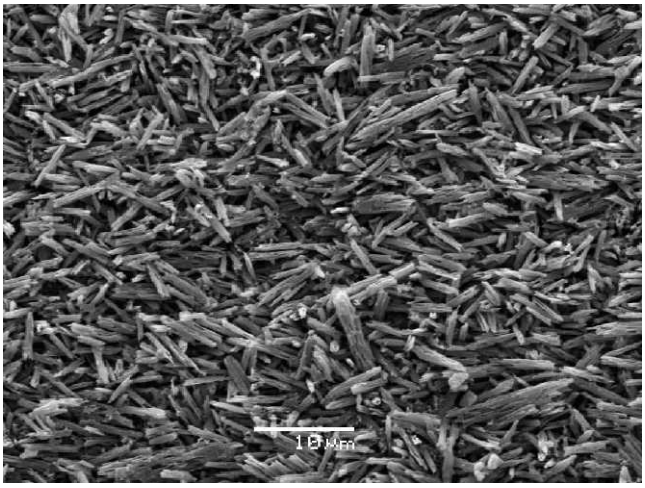

Figure 3. *SEM. Polished and etched surface. 2000×. Exposed lithium disilicate crystals with a regular appearance. The spaces between the crystals were filled with the glassy matrix that was removed by hydrofluoric acid.*

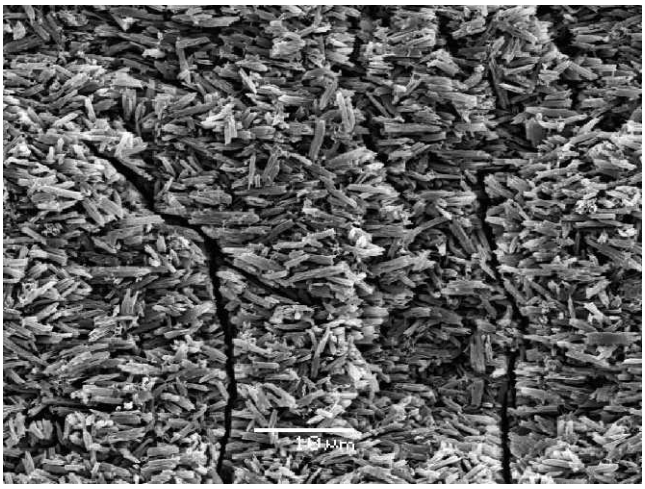

Figure 4. *SEM. Unpolished and etched surface. 2000×. Exposed lithium disilicate crystals with an irregular appearance. Note the presence of cracks and irregularities in the arrangement of the lithium disilicate crystals.*

3 and 4, the lithium disilicate crystals exposed by the hydrofluoric acid attack on the glass phase and secondary crystalline phase can be clearly seen. There is little difference between the surfaces after the application of hydrofluoric acid, with the exception of the brittle appearance of the lithium disilicate crystals and the presence of cracks on the unpolished surface. These may be associated with the impact of the aluminum oxide particles on the glass ceramic surface during air abrasion that is necessary to remove the investment after the ceramic pressing procedure. Such procedure is in accordance with manufacturer's instructions to produce ceramic

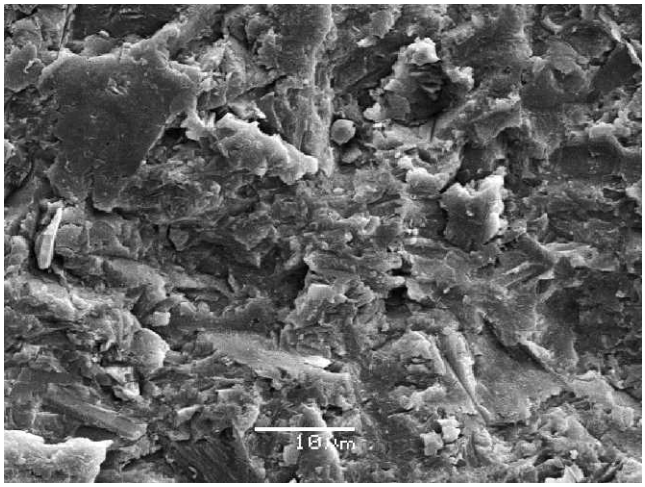

Figure 5. *SEM. Polished and sandblasted surface. 2000×. Sandblasting has changed the surface morphology, creating irregularities that provide micromechanical retention.*

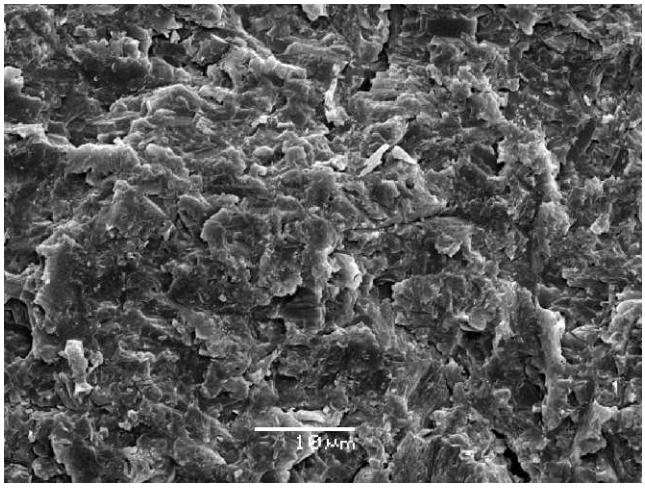

Figure 6. *SEM. Unpolished and sandblasted surface. 2000×. Comparison with the unpolished control group shows that the additional sandblasting led to flattening of some larger, more prominent surface irregularities.*

restorations. Borges and others[9] reported that hydrofluoric acid attack exposes the lithium disilicate crystals because of dissolution of the silica components surrounding these crystals. Consequently, the acid removed the glass phase and exposed the cracks of the UPE surface. In a clinical procedure, that is the surface that receives the bonding procedures. In the PE group, the cracks were removed by the polishing procedure.

Airborne particle abrasion using 50-µm aluminum oxide particles led to an increase in surface irregularities on the polished surface compared with the baseline state, as can be seen from a comparison of Figures 1 and 5. In the specimens with unpolished

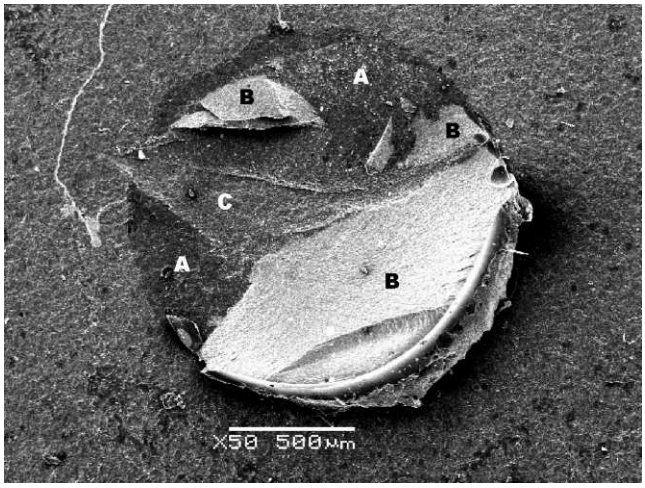

Figure 7. *SEM for failure analysis. The area corresponding to each type of failure was measured. (A): Adhesive failure. (B): Cohesive failure in the resin cement. (C): Cohesive failure in the glass ceramic.*

surfaces, airborne particle abrasion apparently resulted in flattening of preexisting irregularities, as can be observed from a comparison of Figures 2 and 6. Borges and others[9] reported that airborne particle abrasion using 50-µm aluminum oxide particles modified the ceramic surface but that the irregularities were shallow and superficial and similar to the unsandblasted control surface.

Multiple authors [4,7,11,16,18] have evaluated the bond strength between lithium disilicate–based glass ceramic and composite resin or resin cements using microtensile, tensile, shear, and microshear mechanical tests and concluded that the combination of hydrofluoric acid surface treatment and silane coupling agent provides high bond strength, a finding that agrees with the results of the present study.

Subgroups PE and UPE had the highest bond strength values, and there was no statistically significant difference between the results for the two groups. Part of the null hypothesis was therefore rejected.

Etching is used when the matrix contains silica or silicates. First, silicon tetrafluoride is formed. This combines with hydrofluoric acid to form a soluble complex ion (hexafluorosilicate), which in turn reacts with hydrogen protons to form tetrafluorosilicic acid, a product that can be rinsed off with water.[3] This reaction produces a microretentive surface where the hydrophobic resin can penetrate and polymerize, forming a mechanical bond with the ceramic.[3]

The results for subgroups PS, UPC, and UPS were statistically similar. Borges and others[9] reported that additional airborne particle abrasion using 50-μm aluminum oxide particles after the laboratory procedures can change the surface and produce a greater number of pits per unit area than the control treatment. However, the results of the present study showed that an additional step involving sandblasting did not change the glass ceramic surface sufficiently to produce higher bond strength values, as can be seen in subgroups UPC and PS. These results are supported by Soares and others[1] and Blatz and others,[6] who reported that sandblasting alone is not sufficient to produce adequate bond strength and that, as excessive use of this method can cause chipping or additional loss of ceramic material, it is not recommended.

The effect of the chemical bond promoted by the silane agent can be observed in the bond-strength values for subgroup PC, which were statistically similar to those for subgroups UPS and UPC. This confirms the effectiveness of the siloxane bond between the silica compounds in the glass phase of the ceramic and the methacrylate groups in the cement.[3,5,6,14,20] Nevertheless, the only subgroup that did not have a normal data distribution was the PC subgroup. It is possible that use of the silane agent alone is not sufficient to achieve reliable values of bond strength between the ceramic surface and resin cement, a hypothesis that is supported by the findings of a study by Matinlinna and others.[3]

There was a statistically significant difference between the results for subgroup PS and those for subgroup PC, suggesting that airborne abrasion with aluminum oxide changed the surface morphology and increased the irregularities, promoting bond strength. This finding confirms the importance of mechanical retention for bond strength between glass ceramic and resin cement. However, the mechanical retention produced by etching with 10% hydrofluoric acid greatly outweighed that produced by airborne particle abrasion using 50-μm aluminum oxide particles.

The prevalence of cohesive failure in the glass ceramic treated with hydrofluoric acid may show that this material has a lower resistance to crack propagation when subjected to surface treatment with hydrofluoric acid.[12] The nonhomogeneous stress distribution generated by shear testing at the bonded interface may also be linked to this type of failure (Figure 7).[17] We chose to use the shear bond test because the samples do not need to be trimmed after bonding, the preparation of the specimens is relatively straightforward, and multiple samples—even those using brittle materials—can be easily produced.[20]

CONCLUSIONS

Notwithstanding the limitations of this *in vitro* study, the following conclusions can be drawn: flattening and polishing of the glass ceramic surface (for the purpose of standardizing the specimens in the laboratory) did not yield different bond-strength results from those obtained using untreated ceramic surfaces (such as those found in the clinical setting). Etching with 10% hydrofluoric acid was the most effective treatment in terms of bond strength between glass ceramic and resin cement regardless of the initial state of the ceramic surface. The results obtained when sandblasting with aluminum oxide was used differed from those obtained without sandblasting only for specimens that were already polished.

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