©*Operative Dentistry, 2013,* **38-2,** 208-217

Effects of Surface Treatments, Thermocycling, and Cyclic Loading on the Bond Strength of a Resin Cement Bonded to a Lithium Disilicate Glass Ceramic

GB Guarda • AB Correr • LS Gonçalves
AR Costa • GA Borges • MAC Sinhoreti
L Correr-Sobrinho

Clinical Relevance

The current results indicate that acid etching is a better option than air abrasion when bonding IPS e.max Press ceramic to dual-cure RelyX ARC resin cement. However, both fatigue and thermocycling decrease the bond strength.

SUMMARY

Objectives: The aim of this present study was to investigate the effect of two surface treatments, fatigue and thermocycling, on the microtensile bond strength of a newly introduced lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent) and a dual-cured resin cement.

Methods: A total of 18 ceramic blocks (10 mm long × 7 mm wide × 3.0 mm thick) were fabricated and divided into six groups (n=3): groups 1, 2, and 3—air particle abraded for five seconds with 50-μm aluminum oxide particles; groups 4, 5, and 6—acid etched with 10%

Guilherme Bottene Guarda, DDS, MDS, PhD student, Department of Restorative Dentistry, Dental Materials Division, Piracicaba Dental School, State University of Campinas – UNICAMP, Piracicaba, SP, Brazil

Américo Bortolazzo Correr, DDS, MDS, PhD, professor

Luciano Sousa Gonçalves, MDS, DDS, PhD, professor

Ana Rosa Costa, DDS, MDS, PhD student

Gilberto Antonio Borges, DDS, MDS, PhD, professor

Mário Alexandre C Sinhoreti, DDS, MDS, PhD, professor

*Lourenço Correr-Sobrinho, DDS, MDS, PhD, professor

*Corresponding author: UNICAMP, Dental Materials Division, Av Limeira 901, Piracicaba, SP 13414–903, Brazil. E-mail: sobrinho@fop.unicamp.br

DOI: 10.2341/11-076-L

hydrofluoric acid for 20 seconds. A silane coupling agent was applied onto all specimens and allowed to dry for five seconds, and the ceramic blocks were bonded to a block of composite Tetric N-Ceram (Ivoclar Vivadent) with RelyX ARC (3M ESPE) resin cement and placed under a 500-g static load for two minutes. The cement excess was removed with a disposable microbrush, and four periods of light activation for 40 seconds each were performed at right angles using an LED curing unit (UltraLume LED 5, Ultradent) with a final 40 second light exposure from the top surface. All of the specimens were stored in distilled water at 37°C for 24 hours. Groups 2 and 5 were submitted to 3,000 thermal cycles between 5°C and 55°C, and groups 3 and 6 were submitted to a fatigue test of 100,000 cycles at 2 Hz. Specimens were sectioned perpendicular to the bonding area to obtain beams with a cross-sectional area of 1 mm^2 (30 beams per group) and submitted to a microtensile bond strength test in a testing machine (EZ Test) at a cross-head speed of 0.5 mm/min. Data were submitted to analysis of variance and Tukey post hoc test ($p \leq 0.05$).

Results: **The microtensile bond strength values (MPa) were 26.9 ± 6.9, 22.2 ± 7.8, and 21.2 ± 9.1 for groups 1–3 and 35.0 ± 9.6, 24.3 ± 8.9, and 23.9 ± 6.3 for groups 4–6. For the control group, fatigue testing and thermocycling produced a predominance of adhesive failures. Fatigue and thermocycling significantly decreased the microtensile bond strength for both ceramic surface treatments when compared with the control groups. Etching with 10% hydrofluoric acid significantly increased the microtensile bond strength for the control group.**

INTRODUCTION

Ceramics are used for dental restorations and have excellent properties, such as chemical stability, biocompatibility, low thermal conductivity, high compressive strength, thermal diffusivity, translucence, fluorescence, and a coefficient of thermal expansion similar to that of tooth structure.[1–3] However, ceramic is brittle, a property that can be attributed to the presence of microcracks on the surface, making the material susceptible to fracture during luting and under occlusal forces.[2,4] To compensate for brittleness, ceramic has been associated with a metal substructure, and the system has served dentistry for approximately 50 years. Despite the high fracture resistance of traditional metal-ceramic crowns,[5] limitations are imposed on the systems because the metal core can reduce the translucency and affect the esthetics of restorations. Several technological advances have recently led to the development of new materials that possess high strength, such as glass-infiltrated, heat-pressed, and copy-milled ceramics.

The clinical success of ceramic restorations depends on a number of factors, such as the cementation procedure and composition of the ceramic material. Different ceramic surface treatments have been introduced to improve resin bonding to ceramic. A newly introduced lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) may be adhesively cemented, but when the retentive area is small, retention may be inadequate. Bonding of the resin cement to the tooth is aided by acid etching of the enamel or dentin and by the use of a dentin adhesive.[6] Techniques for bonding to ceramic IPS e.max Press take advantage of the formation of chemical bonds and micromechanical interlocking at the resin-ceramic surface. Etching with hydrofluoric acid is used to create a rough surface on the bonding area of the ceramic material to enhance bonding between the ceramic and resin cement. Hydrofluoric acid removes the glass matrix and the second crystalline phase, thus creating irregularities within the lithium disilicate crystals of the IPS e.max Press for bonding.[3,7–8] Another treatment recommended for ceramic surfaces involves airborne particle abrasion with 50-μm aluminum oxide (Al_2O_3) particles to aid in mechanical retention.[7–11] After air abrasion, the ceramic surface must be coated with a suitable silane, which forms chemical bonds between the inorganic phase of the ceramic and the organic phase of the resin cement.[8,12–14]

Clinically, when ceramic restorations are cemented and exposed to the oral environment, factors that could result in fatigue may influence their physical and mechanical properties. Fatigue fracture is a form of failure that occurs in structures with microscopic cracks subjected to dynamic and fluctuating stresses.[15] Continued loading during mastication results in stress concentration, whereas thermal variations induce fatigue, and these cracks propagate and weaken the restoration. Catastrophic fracture results from a final loading cycle that exceeds the load-bearing capacity of the remaining sound portion of the material.[16–17] Thermal variations and the evaluation of fatigue resistance of dental ceramics could provide a more detailed understanding of clinical failures.[18]

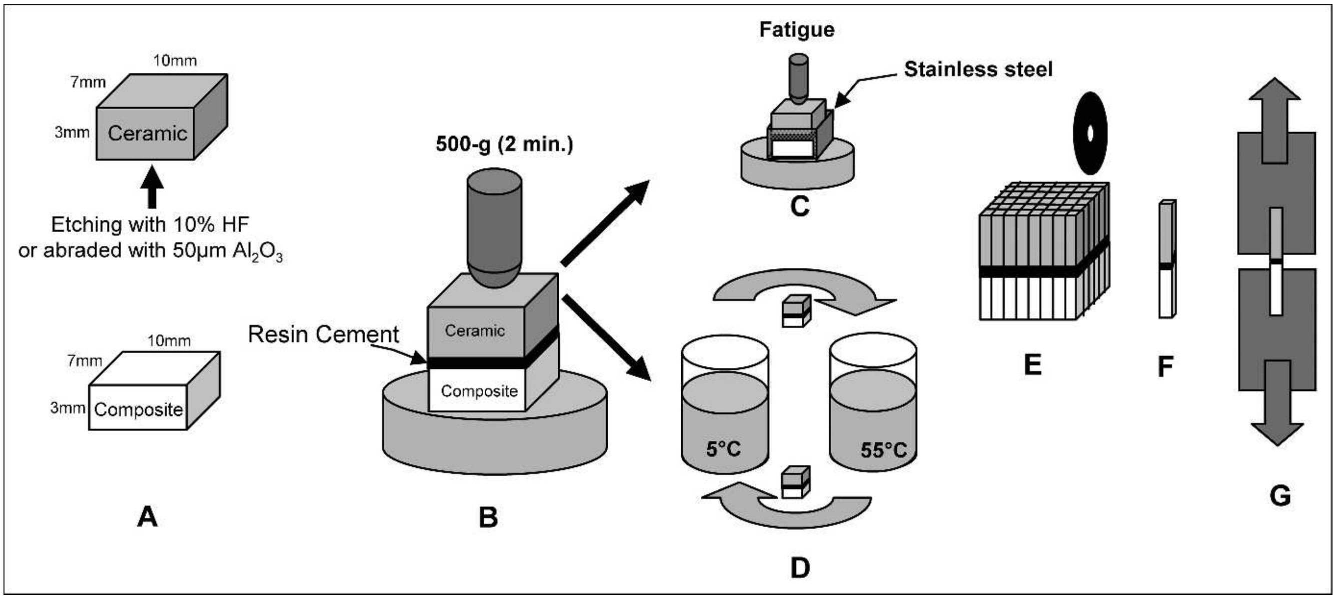

Figure 1. *Experimental setup of the study. (A): The surfaces of ceramic blocks were abraded with 50-µm Al_2O_3 particles (AOP) or etched with 10% hydrofluoric acid (HF) and one layer of silane was applied. (B): The ceramic blocks were bonded to a block of the composite under a 500-g static load for two minutes and light activated. (C): The specimens were put into a stainless steel box with a layer of elastomer and then submitted to a fatigue test of 100,000 cycles with an 8-mm-diameter stainless ball. (D): The specimens were submitted to 3,000 thermal cycles between 5°C and 55°C. (E): Beams obtained after perpendicular sectioning. (F): Beam with bonding area of 1 mm^2. (G): Beam was positioned on the testing machine and submitted to a microtensile bond test.*

Therefore, the aim of this present study was to investigate the effect of two surface treatments, fatigue and thermocycling, on the microtensile bond strength of the ceramic IPS e.max Press luted with a dual-cured resin cement. The hypotheses tested were 1) the surface treatments do not affect the microtensile bond strength of the ceramic; and 2) the fatigue and thermocycling do not affect the microtensile bond strength of the ceramic.

MATERIALS AND METHODS

Ceramic Blocks

A total of 18 rectangular blocks (10 mm long × 7 mm wide × 3.0 mm thick) of IPS e.max Press ceramic (Ivoclar Vivadent), shade LT D3, were fabricated (Figure 1A) in accordance with the manufacturer's instructions. Rectangular wax patterns were made, sprued, and attached to a muffle base with a surrounding paper cylinder. The wax patterns were invested with phosphate-based material (IPS PressVest Speed, Ivoclar Vivadent), and the wax was eliminated in an automatic furnace (Vulcan A-550, Degussa-Ney, Yucaipa, CA, USA) at 850°C for one hour. The IPS e.max Press ceramic ingots were pressed into the molds in an automatic press furnace (EP 600, Ivoclar Vivadent). After cooling, the specimens were divested and submitted to wet polishing with 600- and 1200-grit silicon carbide abrasive papers (Norton SA, São Paulo, Brazil) to obtain a flat surface.

Composite Blocks

A total of 18 rectangular blocks (10 mm long × 7 mm wide × 3.0 mm thick) of composite Tetric N-Ceram (Ivoclar Vivadent, shade A3) were fabricated (Figure 1A). The composite (Tetric N-Ceram) was bulk inserted into an elastomeric mold (Express STD, 3M ESPE, St Paul, MN, USA), and a Mylar strip was placed on the composite surface and manually pressed using a microscope slide to remove excess composite. The composite was light activated for 80 seconds from the top surface using an LED source (UltraLume 5, Ultradent, South Jordan, UT, USA) with an irradiance of 1100 mW/cm^2.

Surface Treatments of the Ceramic Blocks

The 18 ceramic blocks were randomly divided into six groups (n=3). In groups 1, 2, and 3, the test surfaces of the ceramic blocks were air particle abraded with 50-µm Al_2O_3 particles (AOP) (Bioart, São Carlos, Brazil) for five seconds under two bars of pressure using a sandblasting device (Microetch, Bioart) held at a distance of 10 mm and perpendicular to the ceramic surface (Figure 1A). Specimens were then rinsed, ultrasonically cleaned in distilled water for 20 minutes, and dried with compressed air. The test surfaces of groups 4, 5, and 6 were etched with 10% hydrofluoric acid (HF) (Dentsply, Petrópolis, Brazil) for 20 seconds, followed by rinsing with distilled water for one minute (Figure 1A). The specimens were then rinsed, ultrasonically cleaned in distilled water for 20 minutes, and dried with compressed air. One layer of a silane coupling agent (RelyX Ceramic Primer, 3M ESPE) was applied onto all ceramic specimens and allowed to air dry for five seconds.

Cementing the Composite to the Ceramic

One coat of Single Bond Adhesive (3M ESPE) was applied to all composite blocks, air dried for five seconds, and light activated for 10 seconds (UltraLume 5, Ultradent). A dual-cured resin luting agent (RelyX ARC, 3M ESPE), shade A3, was manipulated according to the manufacturer's instructions and applied to the ceramic surface. The ceramic blocks were bonded to a block of the composite Tetric N-Ceram and placed under a 500-g static load for two minutes (Figure 1B); the excess cement was removed with a disposable microbrush. Light-activation was performed for 40 seconds at right angles to each of the IPS e.max Press/Tetric N-Ceram margins (four activations) using an LED source (UltraLume LED 5, Ultradent), with a final 40-second light exposure from the top surface.

Conditioning the Specimens

All specimens were stored in distilled water at 37°C for 24 hours. The specimens of groups 2 and 5 were submitted to 3000 thermal cycles (MSCT 3, Marnucci ME, São Carlos, Brasil) between 5°C and 55°C (dwell time of 30 seconds) (Figure 1D). The specimens of groups 3 and 6 were put into a stainless steel box with a layer of polyether impression material (Impregun F, 3M ESPE, Seefeld, Germany) with 1 mm thickness. This layer was placed on the bottom as well as on the sides to stabilize the specimen. Then, the specimen was submitted to a fatigue test of 100,000 cycles (ER37000, ERIOS, São Paulo, Brazil) that consisted of cyclic loading with an 8-mm-diameter stainless steel ball applied on the central area of the ceramic side of the specimen with load of 80 N in a wet environment prior to microtensile bond testing. The cyclic loading had a force profile in the form of a sine wave at 2 Hz (Figure 1C).

Microtensile Bond Strength Testing

After the experimental procedures, the specimens were sectioned perpendicular to the bonding interface area (Figure 1E) to obtain beams with a bonding area of 1 mm^2 (Figure 1F) using a water-cooled diamond blade (EXTEC Corporation, Enfield, CT, USA) in a low-speed saw machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). The cross-sectional area of the bond interface of each beam was measured using a digital caliper (Mitutoyo Corporation, Tokyo, Japan). Each beam was fixed to the grips of a microtensile device using a cyanoacrylate adhesive (Zapit, Dental Ventures of America Inc, Corona, CA, USA), and the microtensile bond test was conducted in a testing machine (EZ Test, EZS, Shimadzu, Tokyo, Japan) at a crosshead speed of 0.5 mm/min until failure (Figure 1G).

Statistical Analysis

Bond strength values were calculated and the data supplied in megapascals. The experimental unit was the ceramic/composite block. Each group contained three blocks, and each block generated an average of 10 beams, for a total of 30 beams per group. Thus, the mean of the bond-strength values in each group represented the sum of the three experimental units. Microtensile bond-strength data were submitted to two-way analysis of variance, and multiple comparisons were performed using the Tukey *post hoc* test ($p<0.05$).

Failure Analysis

The fractured specimens were observed under optical microscopy (Olympus Corp, Tokyo, Japan) at 40× magnification. The mode of failure was classified as follows: adhesive (mode 1); cohesive within ceramic (mode 2); cohesive within composite (mode 3); and mixed, involving cement, ceramic, and composite (mode 4). The specimen surfaces were gold coated with a sputter coater (Balzers-SCD 050, Balzers Union, Aktiengeselischaft, Fürstentun, Liechtenstein) for 180 seconds at 40 mA. The specimens were then mounted on coded brass stubs and examined using scanning electron microscopy (SEM; LEO 435 VP, Cambridge, England), operated at 20 Kv, by the same operator.

RESULTS

Bond Strength Testing and Failure Analysis

The mean values of the microtensile bond strength testing are shown in Table 1. For the control group, the bond strength of the specimens etched with 10% HF was significantly higher than the specimens that received AOP ($p\leq0.05$). For the fatigue-tested group, no statistical difference was found between the specimens etched with HF and AOP ($p\geq0.05$).

Table 1: *Microtensile Bond Strength Means ± Standard Deviations (MPa) for All Groups*[a]

Surface Treatments	**Bond Strength (MPa)**		
	Control Group	**Fatigue Tested**	**Thermocycled**
10% hydrofluoric acid	35.0 (9.6) A,a	23.9 (6.3) B,a	24.3 (8.9) B,a
50 µm Al_2O_3	26.9 (6.9) A,b	21.2 (9.1) B,a	22.2 (7.8) B,a

[a] *Means followed by different capital letters in the same row and/or small letters in the column are significantly different at* $p < 0.05$ *(Tukey test).*

Similar results were observed for the thermocycled groups.

The microtensile bond strength of the control group was significantly higher than that for the fatigue-tested and thermocycled groups ($p \leq 0.05$). No statistical difference was found between the fatigue-tested and thermocycled groups ($p \geq 0.05$).

The distribution of failure modes is shown in Table 2. The control, fatigue-tested, and thermocycled groups showed a predominance of failure mode 1 for the HF surface treatment and modes 1 and 4 for the AOP surface treatment.

The SEM micrographs showed that the control group (Figure 2) had fewer irregularities. On the other hand, the acid-etched group (Figure 3) presented more retentive irregularities when compared with the air-abraded group (Figure 4).

DISCUSSION

The clinical success of a ceramic restoration depends on the quality and durability of the bond between the ceramic and the resin cement. The quality of this bond is determined by the bonding mechanisms, which are controlled in part by the specific surface treatment used to promote micromechanical or chemical retention to the ceramic substrate.[19] The micromechanical retention of the ceramic surface plays an important role in bonding with a resin luting cement. Morphology modification on the ceramic surface may be performed to promote a better bond strength.[7] After surface treatment, the resin cement is applied on the ceramic surface, and the penetration of the cement and its polymerization is responsible for bonding.[20–21]

Table 2: *Failure Modes Analysis of the Debonded Specimens (%) Among Groups*[a]

Groups	**Mode 1**		**Mode 2**		**Mode 3**		**Mode 4**	
	HF	**AOP**	**HF**	**AOP**	**HF**	**AOP**	**HF**	**AOP**
Control	47	53	5	0	24	0	24	47
Fatigue tested	52	73	0	18	11	0	37	9
Thermocycled	80	91	0	9	5	0	15	19

Abbreviations: HF, hydrofluoric acid; AOP, aluminum oxide particles.
[a] *Mode 1: adhesive; Mode 2: cohesive within ceramic; Mode 3: cohesive within composite and Mode 4: mixed, involving cement, ceramic, and composite.*

In the present study, two ceramic surface treatments were evaluated. The results indicated that the first hypothesis was partly accepted. The etching procedure with HF resulted in the highest tensile bond strength with a statistically significant difference when compared with the AOP group or the control group. No statistical difference was found after fatigue and thermocycling.

The difference in tensile bond strength can be explained on the basis of morphology created on the AOP and HF specimens (Figures 2–4). Etching the ceramic surface with 10% HF promoted dissolution in the glassy matrix of the specimens to the depth of a few microns, enabling the lithium disilicate crystals to protrude from the glass matrix. Elongated crystals and shallow irregularities were clearly observed (Figure 3). The change in the surface morphology treated with 10% HF increased the surface area and facilitated the penetration and retention of resin cement into the microretentions of the treated surface.[7,8,22] It has been shown that this treatment has an efficient result for other kinds of dental ceramics.[3,7,8,12,19–20,23] The lowest mean tensile bond strength was obtained for the specimens that were air abraded with 50-µm Al_2O_3. The present results indicate that this treatment does not provide a mechanically retentive surface that is as efficient as etching with HF. This treatment promoted morphologic alterations of the ceramic surface, resulting in an increase in the number of potential

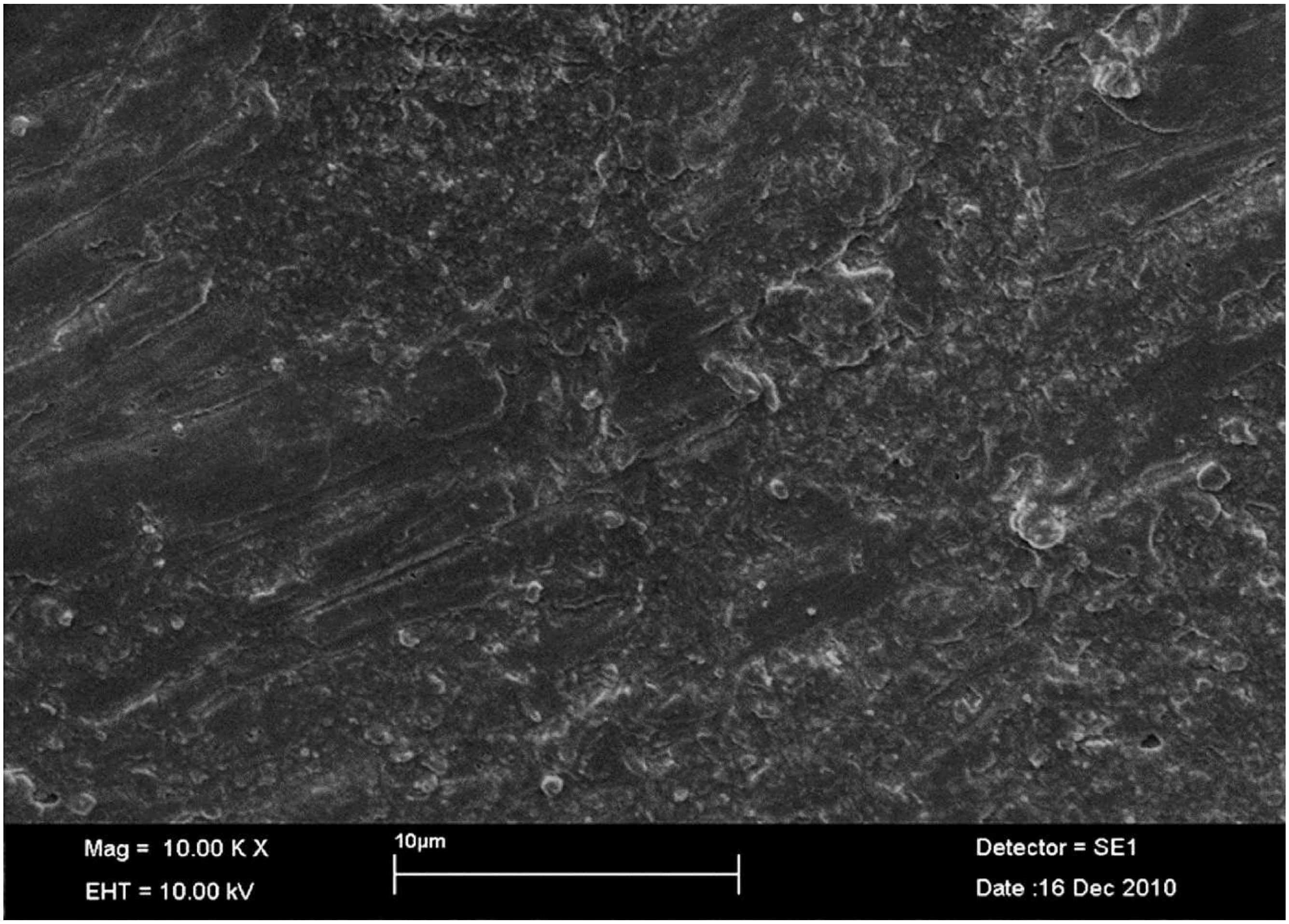

Figure 2. *SEM morphological aspect of a ceramic surface without additional treatment (10,000×).*

retention areas and surface area (Figure 4). Salvio and others,[7] Spohr and others,[8] Ayad and others,[20] and Attia[23] also demonstrated that there is a decrease in shear bond strength when the ceramic surface was air abraded with 50-µm Al_2O_3 when compared with a ceramic surface etched with 10% HF. However, Panah and others[24] showed no significant differences between a ceramic surface etched with 10% HF and one air abraded with 50-µm Al_2O_3.

A variable that can contribute to failure of a ceramic is the oral environment. It is known that the oral environment is able to induce physicochemical alterations in dental materials.[25] Mechanical fatigue and temperature alterations of materials provide conditions for their degradation in an aqueous environment.

In the present study, fatigue testing and thermal cycling were evaluated. The results indicated that the second hypothesis was not accepted. The groups etched with 10% HF or air abraded with 50-µm Al_2O_3 and submitted to fatigue testing or thermal cycling decreased in tensile bond strength, with statistically significant differences when compared with the control groups. It is known that fatigue results in material alterations in areas of stress concentration.[17] It is possible that when the specimens were submitted to a cyclical loading under wet conditions, the propagation of small cracks in the interface between the ceramic surface and the resin luting agent might have significantly reduced the bond strength.

In this present study, the effect of thermocycling on microtensile bond-strength testing was determined. Kamada and others[13] related that the durability of the bond strength between a silane-treated ceramic surface and the resin cement decreased with thermocycling and water storage. Oyafuso and others[25] showed a decrease in resistance to fracture after both ceramic-gold and ceramic-commercially pure titanium combinations were

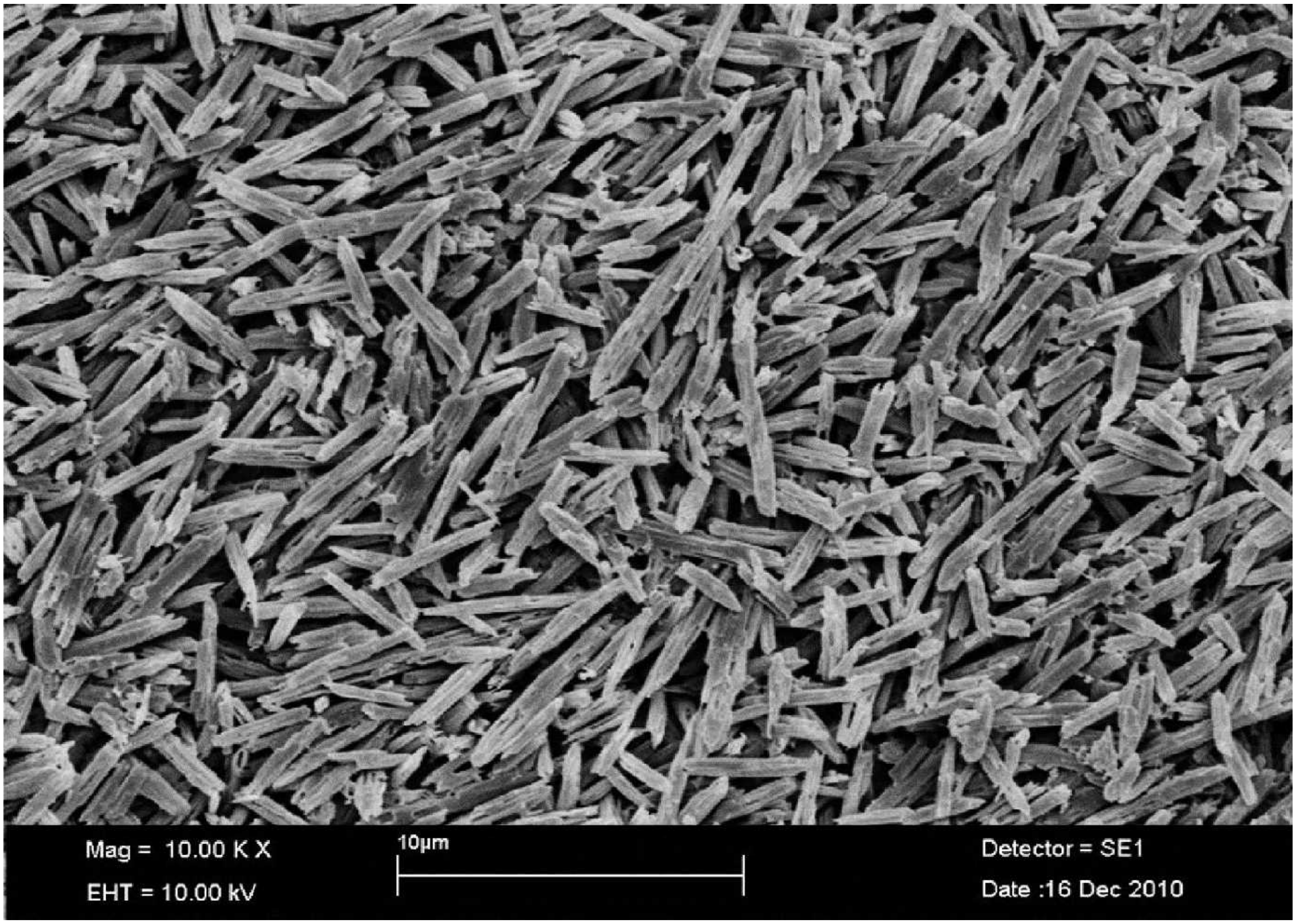

Figure 3. *SEM morphological aspect of a ceramic surface treated with 10% HF for 20 seconds (10,000×).*

subjected to thermocycling and mechanical cycling. Different studies have demonstrated that, depending on the silane used, thermocycling might have a significant effect on a reduction of bond strength between the resin and ceramic interface.[27–31] A degree of hydrolysis is responsible for the efficacy of the silane product; the higher the degree of hydrolysis, the better the bond provided by the silane coupling agent.[32] The permeability of the silane results from hydrolysis of the silicon-oxygen bonds at the ceramic-silane interface through water absorption.[33] However, this may also be responsible for the level of degradation of the bond strength between the ceramic-resin interface during thermocycling. Salvio and others[7] reported that the application of the Monobond S silane (Ivoclar Vivadent) followed by RelyX ARC resin cement was partially effective in water storage conditions, given that the mean bond strength decreased after 1 year. On the other hand, Spohr and others[8] showed that Scotchbond Ceramic Primer (3M ESPE), when used with the Single Bond adhesive system and Rely X resin cement, was effective after thermocycling procedures. However, in that study, the specimens were submitted to only 500 thermal cycles.

Another possible factor for the reduced bond strength seen in this current study is the reduction in the mechanical properties of the resin cement when submitted to fatigue testing and thermocycling. The reduction of mechanical properties of the resin cement is probably a result of a continuous action of water on the interface of the ceramic-resin cement. The mechanism of water transport and its effects on the mechanical properties of polymers depend on several factors.[34–35] Monomer ratio and composition vary according to the specific applications and the manufacturer's goals,[36] and variability will define the chemical stability of a resin in a specific environment.[37] The sensitivity of resin-based materials to water depends on the degree of monomer conversion,[38] degree of polymer cross-linking, volume fraction of intrinsic nanometer-sized

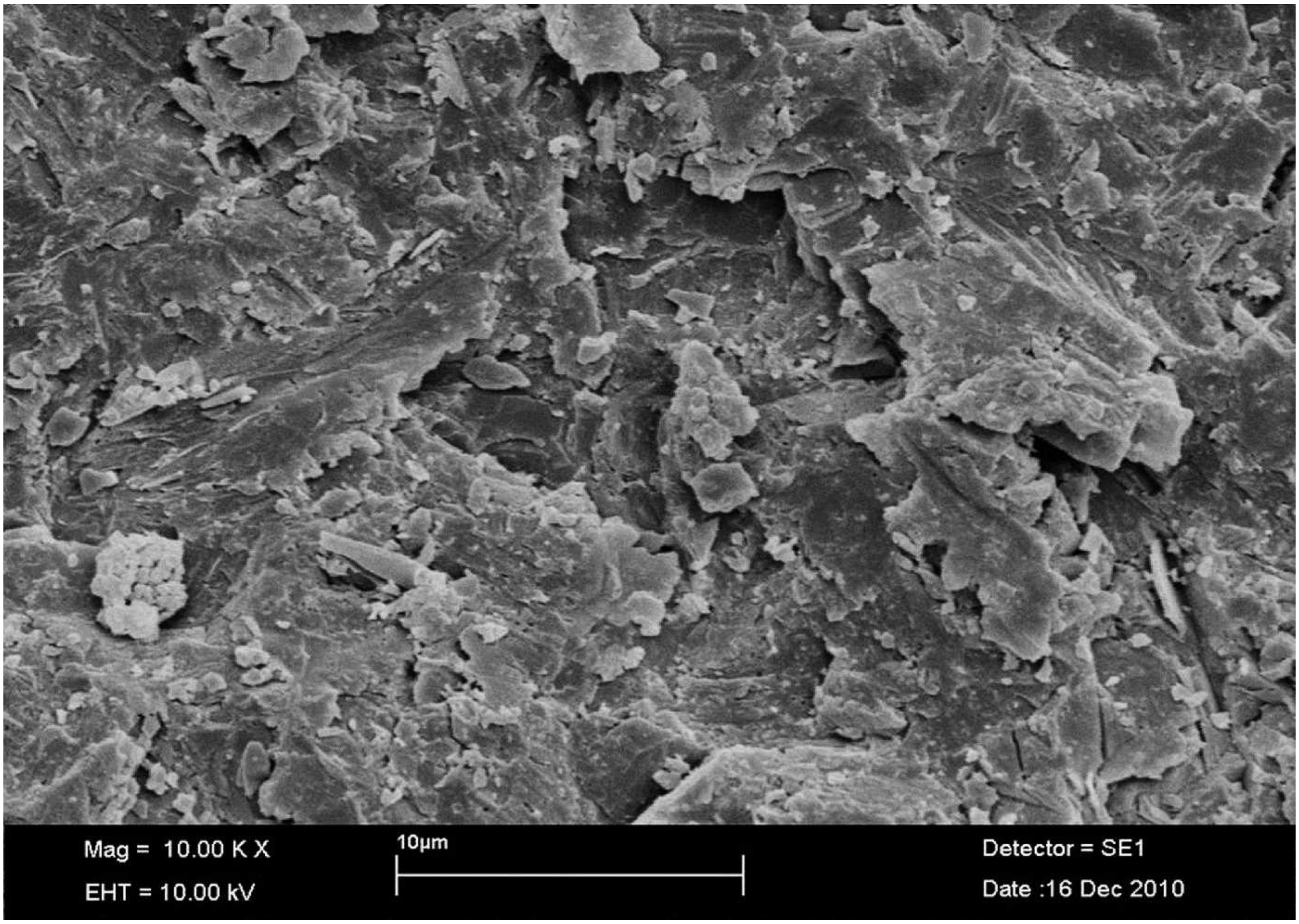

Figure 4. *SEM morphological aspect of a ceramic surface treated with air particle abrasion for five seconds with 50-μm aluminum oxide particles (10,000×).*

pores, and the quantity and presence of fillers.[34] One study found that by increasing the ratio of triethyleneglycol dimethacrylate (TEGDMA) and urethane dimethacrylate to bisphenol-A-glycidyl dimethacrylate, an increase in water sorption was observed.[39–40] It is possible that the presence of TEDGMA in the resin cement used in the present research contributed to the acceleration of water sorption and affected the mechanical properties of the resin cement after fatigue testing and thermocycling.

The mode of failure did not correlate directly with the bond-strength results, as can be seen in Table 2. Even though the control group showed bond strengths that were higher than the fatigued and thermocycled specimens for both treatments, the adhesive mode failure was predominant. This could be explained by the fact that the microtensile test evaluates a small area and the resin cement was much more strongly bonded with the resin composite than the ceramic, even though the bond with the ceramic material is a chemical union. Hence, more detailed studies would clarify the failures at microscopic levels. Thus, a closer evaluation using a SEM and an energy-dispersive spectrometer of the debonded surface could obtain more definitive information.

The present study evaluated one silane agent and one resin cement with a silicate-based ceramic. Further studies should be conducted to evaluate different materials. The current results showed the efficacy of surface treating a disilicate ceramic with 10% HF or with air abrasion using 50-μm Al_2O_3 particles. These results may be clinically useful when choosing a specific surface treatment of disilicate ceramics prior to luting with a resin cement.

CONCLUSION

Within the limitations of the present study, the following conclusions can be drawn:

1. Fatigue and thermal cycling significantly de-

creased the microtensile bond strength for both ceramic surface treatments when compared with the control groups.

2. For the control group, the 10% HF surface treatment showed higher microtensile bond strength values when compared with the specimens treated by sandblasting with 50-µm Al_2O_3 particles.

Acknowledgements

This study was supported by Conselho Nacional de Desenvolvimento Científico e tecnológico–CNPq (Grant 303928/2009–3) and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES). Thanks to Dr EW Kitajima, Dr FAO Tanaka, and RB Salaroli (Núcleo de Apoio a Pesquisaem Microscopia Eletrônica, NAPME/ESALQ/USP, Brazil) for SEM equipment support.

Conflict of Interest Declaration

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 30 May 2012)

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