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Fatigue Behavior of Monolithic Zirconia-Reinforced Lithium Silicate Ceramic Restorations: Effects of Conditionings of the Intaglio Surface and the Resin Cements

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Clinical Relevance

Clinical longevity with the zirconia-reinforced lithium silicate glass-ceramic dental restoration may be influenced by the intaglio surface treatment and the resin cement used. Hydrofluoric acid etching plus self-adhesive resin cement provided better fatigue results.

SUMMARY

Objective: This study assessed the effect of conditioning of the intaglio surface and resin cements on the fatigue behavior of zirconia-reinforced lithium silicate ceramic (ZLS) restorations cemented to a dentin analogue.

Methods: ZLS ceramic (Ø=10 mm, thickness=1.5 mm) and dentin analogue (Ø=10 mm, thickness=2.0 mm) discs were produced and allocated according to the study factors, totaling nine study groups: ceramic surface treatment (three levels: hydrofluoric acid etching [HF]; self-etching ceramic primer [EP]; tribochemical silica coating [TBS]) and resin

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cement (three levels: 10-methacryloyloxydecyl dihydrogen phosphate [nMDP]; MDP–containing conventional resin cement [MDP]; self-adhesive resin cement [SA]). The ceramic bonding surfaces were treated and cemented on the dentin analogue, and all the specimens were aged for 5000 thermal cycles (5°C-55°C) prior to fatigue testing. The stepwise fatigue test (20 Hz frequency) started with a load of 400 N (5000 cycles) followed by steps of 500, 600, and up to 1800 N (step-size: 100 N) at a maximum of 10,000 cycles each step. The specimens were loaded until failure (crack), which was detected by light transillumination and visual inspection at the end of each step. The fatigue failure load and number of cycles for failure data were analyzed by the Kaplan-Meier (log-rank test; α=0.05). Topographic and fractographic analyses were also performed.

Results: HF- (973.33-1206.67 N) and EP- (866.67-1066.67 N) treated specimens failed at statistically similar loads and higher than TBS (546.67-733.33 N), regardless of the cement used. All the fractographical inspections demonstrated failure as radial crack.

Conclusion: The HF and EP treatments promoted better mechanical fatigue behavior of the ceramic restoration, while tribochemical silica coating induced worse fatigue results and should be avoided for treating the ZLS surface prior to bonding.

INTRODUCTION

The zirconia-reinforced lithium silicate glass-ceramic (ZLS) is composed of a glassy matrix reinforced by lithium silicate crystals and dissolved zirconia (56%-64% silicon dioxide, 15%-21% lithium oxide, 8%-12% zirconium oxide), among other components.[1,2] The reduction in the glass content and the crystalline microstructure (0.5-0.7 μm) of the lithium silicate creates a material with high strength and easy machining and polishing, associated to good esthetic properties.[3,4] Due to its glassy content, this ceramic is considered acid-sensitive, being the hydrofluoric acid (HF) etching followed by the silane coupling agent application is the most recommended protocol for its surface conditioning and adhesive bonding to resin cements.[5,6]

The HF acid selectively dissolves the glassy matrix, inducing pull-out of some lithia crystals without glassy support from the surface, revealing the microstructure of the ceramic surface and creating a micromechanical pattern that, associated to the siloxane bonds provided by the silane between the exposed silica and methacrylate group of the resin cement,[7] results in bond improvements.[6,8] However, if all the microporosities and defects created by the HF are not completely filled by the cement, the restoration could be weakened by reducing its resistance to intermittent masticatory loading over time.[9] In addition, the HF acid is considered to be potentially toxic to human health and should be used with caution or even avoided.[5,10] Therefore, it is important to understand this mechanism and to consider other safer alternatives for surface treatment.[11-13]

A promising possibility for dental glass-ceramic treatment is the use of a self-etching ceramic primer, which is a single-step ceramic initiator that facilitates handling and reduces the clinical time and the technique sensitivity when compared to conventional HF etching. However, there is little information available in the literature about the efficacy of these products.[11,12] Wille and others[11] showed that the self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent) provided bond strength to the lithium disilicate ceramic comparable to the well-established HF etching plus silane application, indicating that this surface treatment may be promising for use in glass ceramics. However, in a study by Strasser and others,[13] the use of this self-etching ceramic primer did not cause significant changes in the surface of a zirconia-reinforced lithium silicate ceramic when compared with 5% HF acid etching.

Another alternative for treating the ZLS ceramics is to apply the tribochemical silica coating (silica coating by air abrasion with silica-coated aluminum oxide particles followed by the silane application). Al-Thagafi and others[14] observed the application of tribochemical silica coating provided higher bond strength values compared to 5% HF etching plus silane in a zirconia-reinforced lithium silicate ceramic. The tribochemical silica coating consists of a silica deposition into the ceramic surface through an air-abrasion process, followed by the application of a silane-based coupling agent. The CoJet system (SiO_2-coated Al_2O_3, 30 μm particles, CoJet Sand, 3M ESPE, Seefeld, Germany) is a versatile and portable option for clinical use, which creates chemical bonds by applying mechanical energy.

The increase in bond strength is also directly influenced by the cement characteristics such as viscosity, elastic modulus, and its ability to fill the defects created by the surface treatments.[9,15] There is a high tendency to simplify and reduce steps of the adhesive bonding process in dentistry; therefore, different simplified resin cements were created (e.g., self-adhesive) to chemically interact with the dentin without the need of previous treatment, but the ceramic surface treatment is still required.[16] In addition, they exhibit a lower polymerization shrinkage, resulting in less tooth/restoration interface failure, presenting bond strength values similar to conventional methacryloyloxydecyl dihydrogen phosphate (MDP)–containing cements when used with hybrid ceramics.[17]

On the other hand, resin cements containing phosphate monomers, like the 10-MDP, are composed of bifunctional molecules that directly bond to the oxides of the ceramic surface, through their phosphate ester group, and to the resin matrix of the cement, through their methacrylate groups, providing a better bond strength between the components.[18] Gundogdu and Aladag[19] obtained better values of bond strength when a zirconia ceramic was cemented with an MDP-containing conventional resin-cement compared with a self-adhesive resin cement. However, there is no data in the literature about its real effect on zirconia-reinforced glass ceramics, such as ZLS.

Furthermore, the data presented in the literature evaluating the ZLS related to their different surface treatments are scarce, especially regarding the effects on their fatigue behavior, therefore, some questions remain. Since surface treatments promote distinct topographic changes (different shape, size, and population of defects) on the ZLS ceramic surface, would this affect the fatigue behavior of such restorations? Different resin cements might promote different adhesion potential, therefore, could this affect the fatigue behavior of such restorations?

METHODS AND MATERIALS

The materials used in the study are described in Table 1.

Specimen Preparation

Ceramic—

Prefabricated ZLS blocks (18 × 14 × 12 mm^3; VITA Suprinity, VITA Zahnfabrik, Bad-Säckingen, Germany) were ground into cylinders (Ø=10 mm) using #150-grit size silicon carbide paper (SiC) (Norton Abrasives, Saint-Gobain, São Paulo, SP, Brazil) in a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, IL, USA). After, 1.7-mm-thick discs were obtained in a diamond saw machine (Isomet 1000, Buehler) under constant water cooling. For the removal of any cutting irregularities, the discs were polished with SiC papers of #400, #600, and #1200 grit (Norton Abrasives, Saint-Gobain) on both sides up to the final thickness of 1.5 mm. To simulate the roughness produced by the computer-aided design/computed-aided manufacturing (CAD/CAM) milling process, the cementation surface was ground with #60-grit size SiC paper (Norton Abrasives, Saint-Gobain) 15 times on each axis, *x* and *y*, providing an initial roughness (mean Ra=2.11 μm and mean Rz=12.77 μm) similar to that of CAD/CAM milled restorations.[20]

The specimen's dimensions were based on literature data[21-23] considering the mean diameter of the occlusal surface of the first permanent molars (Ø=10 mm)[24] and their mean thickness based on the distance between the occlusal surface and the dental pulp chamber roof.[25,26] After, the discs were washed in an ultrasonic bath (1440D, Odontobras, Ind and Com Equip Med Odonto, Ribeirão Preto, SP, Brazil) with distilled water for 10 minutes, dried with air-spray, and crystallized in a specific furnace (VACUMAT 6000MP, VITA Zahnfabrik) according to the manufacturer's instructions (840°C, eight-minute vacuum).

Dentin Analogue—

Dentin analogue discs (2.5 mm) were obtained from an epoxy resin plate (elastic modulus of 18 GPa; Carbotec GmbH & Co KG, Königs Wusterhausen, Germany) using a cylindrical diamond drill (Ø=10 mm) under constant water cooling and then ground (SiC #400-, #600- and #1200-grit sizes; Norton Abrasives, Saint-Gobain) until a final thickness of 2.0 mm.

The ceramic and dentin analogue discs were randomly distributed (www.randomizer.org) in nine groups (n=15) according to the factors under study, as shown in Table 2.

Surface Treatments

Ceramic—

Hydrofluoric acid etching. HF (5% IPS Ceramic Etching Gel, Ivoclar Vivadent) was applied and scrubbed on the ceramic surface with a microbrush and kept to react for 20 seconds, as recommended by the manufacturer. After, the specimens were washed with air/water-spray for 30 seconds, gently air dried, subjected to the ultrasonic bath (1440D, Odontobras, Ind and Com Equip Med Odonto) with distilled water for 5 minutes and air-spray dried for 30 seconds. The silane coupling agent (Prosil, FGM, Joinville, SC, Brazil) was then actively applied for 15 seconds with a microbrush, left to react for 60 seconds, and air dried for 15 seconds.

Etch & prime ceramic primer (EP). The self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent) was actively applied on the ceramic surface with a microbrush for 20 seconds, left to react for 40

Table 1: *Materials Used and Their Respective Characteristics: Commercial Name, Manufacturer and Composition*

Material and Manufacturer	Composition
Zirconia-reinforced lithium silicate glass-ceramic, VITA Suprinity, VITA Zahnfabrik	SiO_2; Li_2O; K_2O; P_2O_5; ZrO_2; Al_2O_3; CeO_2; pigments
Hydrofluoric acid 5% (IPS Ceramic Etching-gel), Ivoclar Vivadent	Hydrofluoric acid < 5%
Hydrofluoric acid 10% (Condac Porcelana), FGM	Hydrofluoric acid 10%, water, thickener, surfactant and coloring
Monobond Etch &Prime, Ivoclar Vivadent	Tetrabutyl ammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, trimethoxysilylpropyl methacrylate, alcohol, water
CoJet Sand, 3M ESPE	Aluminum oxide; free amorphous synthetic silica
MDP-free conventional resin cement (Multilink Automix), Ivoclar Vivadent	Base: ytterbium trifluoride, ethoxylated bisphenol A dimethacrylate, Bis-GMA, 2-HEMA, 2-dimethylamanoethyl methacrylate Catalyst: ytterbium trifluoride, ethoxylated bisphenol A dimethacrylate, urethane dimethacrylate, 2-HEMA, dibenzoyl peroxide and silica filler (68 wt%), pigments
MDP-containing conventional resin cement (Panavia F 2.0), Kuraray Noritake)	10-methacryloxydecyl dihydrogen phosphate, bisphenol-A-polyethoxy dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic methacrylate, silanated silica filler, silanated barium glass filler (78 wt%), sodium fluoride
Self-adhesive resin cement (RelyX U200, (3M Oral Care)	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers (72 wt%), initiator components, stabilizers, pigments, rheological additives.
Silane (Prosil), FGM	3-methacryloxypropyl trimethoxy silane (< 5%); ethanol (> 85%); water (< 10%)

Abbreviations: Bis-GMA, glycidyl methacrylate; HEMA, hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

seconds, and then washed with air/water-spray for 20 seconds.

Tribochemical silica coating (TBS). The TBS was performed with 30 μm silica-coated aluminum trioxide particles (CoJet Sand, 3M ESPE) on the intaglio ceramic surface using a micro-etcher (DENTO-PREP microblaster, Ronvig, Daugaard, Denmark) at a distance of 15 mm from the blast nozzle to the ceramic surface, with a blast pressure of 2.5 bar,[27] in oscillatory movements for 15 seconds.[14] After, a light air-spray was applied to remove loose particles, and a silane coupling agent (Prosil, FGM) was actively applied for 15 seconds, left to react for 60 seconds, and air-spray dried for 15 seconds.

Dentin analogue. To simulate, as close as possible, a clinical setting, dentin analogue discs were used to mimic the hydrated human dentin, being similar in terms of bond strength and elasticity.[22] Prior to cementation, the dentin analogue discs were cleaned in an ultrasonic bath (1440D, Odontobras, Ind And Com Equip Med Odonto) with distilled water for five minutes and air-spray dried. After, they were acid etched with 10% HF (Condac Porcelana, FGM) for one minute, removed with air/water-spray for 30 seconds, and washed in an ultrasonic bath with distilled water for five minutes and air-spray dried for 15 seconds.

Cementation

MDP-free Conventional Resin Cement (nMDP; Multilink Automix)—

The primers A and B of the Multilink Automix system were mixed (1:1) and applied in the dentin analogue surface for 30 seconds with a microbrush, and a light air-spray was applied to create a thin and uniform layer. The base and catalyst pastes of the resin cement were

Table 2: *Study Design and Study Groups*

Resin Cements	Ceramic Surface Treatments	Groups
Multilink Automix – nMDP MDP-free conventional resin cement	HF → 5% hydrofluoric acid etching (IPS Ceramic etching-gel) + Silane (Prosil) EP → Self-etching ceramic primer (Monobond Etch & Prime) TBS → Tribochemical silica coating (CoJet Sand) + Silane (Prosil)	nMDP+HF
		nMDP+EP
		nMDP+TBS
Panavia F 2.0 – MDP MDP-containing conventional resin cement		MDP+HF
		MDP+EP
		MDP+TBS
RelyX U200 – SA Self-adhesive resin cement		SA+HF
		SA+EP
		SA+TBS

Abbreviations: MDP, 10-methacryloyloxydecyl dihydrogen phosphate; SA, self-adhesive resin cement.

dispensed from the double-push syringe, mixed for 20 seconds, and applied on the dentin analogue surface.

MDP-containing Conventional Resin Cement (Panavia F2.0)—
ED primers liquid A and liquid B were mixed (1:1) and applied on the dentin analogue surface for 60 seconds with a microbrush and dried with a light air-spray. The base and catalyst pastes of the resin cement were dispensed from the syringes, mixed for 20 seconds, and applied on the dentin analogue surface.

Self-adhesive Resin Cement (RelyX U200)—
The base and catalyst pastes of the resin cement were dispensed from the double-push syringe, mixed for 20 seconds and applied on the dentin analogue surface.

After the resin cements were placed in the treated dentin analogue, the ceramic disc was seated over the cement and a constant load of 2.5 N was applied on the ceramic surface to promote a uniform cement spreading, standardizing its thickness.[28] After removing the cement excesses with a microbrush, the light curing was performed (LED light, 1200 mW/cm^2, 440-480 nm, Radii-cal, SDI Limited, Bayswater, Australia) for 40 seconds on the occlusal surface of the ceramic followed by 20 seconds on each lateral side of the interface (0°, 90°, 180° and 270°).

Aging —Thermocycling

After bonding, the specimens were stored for 24 hours submerged in distilled water at 37°C in a laboratory incubator. Then, the specimens underwent intermittent 5000 thermal cycles (model 521-6D, Ethik Technology Limited, Vargem Grande Paulista, SP, Brazil) with temperatures ranging from 5°C to 55°C with 30 seconds of dwell time at each temperature and 4 seconds of transfer time.[29]

Fatigue Testing—Stepwise Method

The specimens (n=15) were tested submerged in distilled water in an electrodynamic testing machine (Instron ElectroPuls E3000, Instron Corp, Norwood, MA, USA) over a flat metal base, and the load was applied on the ceramic surface with a 40-mm-diameter stainless steel hemispherical piston.[22] Prior to testing, an adhesive tape (110 μm) was placed between the piston and the ceramic to improve the contact between them and to avoid contact damage.[28] The test started with a load of 200 N at a frequency of 20 Hz per 5000 cycles (preconditioning phase to ensure predictable positioning of the piston with the sample), followed by steps of 400 N, 500 N, 600 N, and so on with increments of 100 N to 1800 N to a maximum of 10,000 cycles each step. At the end of each step, the specimens were analyzed by transillumination to check the presence of a crack. The specimens were tested until fracture (presence of a minimal radial crack) or until all steps were complete.

Topographic Analysis

Micrographs of the ceramic surface at control (CAD/CAM milling simulation) and after the surface treatments were analyzed (100× magnification) to inspect the topographical changes (micromechanical patterns). Representative specimens (n=2) were ultrasonically cleaned, gold sputtered, and analyzed in a scanning electron microscope (SEM) (Secondary electrons, 20kV; VEGA3 Tescan, Brno-Kohoutovice, Czech Republic).

Fractographic Analysis

After the fatigue testing, the specimens were analyzed in a stereomicroscope (100× magnification; Stereo Discovery V20, Carl-Zeiss, Göttingen, Germany) to identify their crack path. Representative specimens were cut under water-cooling (Isomet 1000, Buehler) in the middle and perpendicular to the crack, ultrasonically cleaned in distilled water for 10 minutes, sputtered with a gold-palladium alloy, and analyzed in the SEM (500× magnification; Secondary electrons, 20kV, VEGA3 Tescan) to identify the crack features.

Statistical Analysis

The fatigue failure load and number of cycles for failure data were recorded and analyzed by the Kaplan-Meier and Mantel-Cox (log-rank) tests at α=0.05.

RESULTS

Kaplan-Meier analysis showed that surface treatments with HF and EP presented loads to failure and number of cycles for failure statistically similar to each other and higher than TBS, regardless of the type of cement (Table 3). When only the cement was considered, the self-adhesive cement obtained statistically greater results for the HF and TBS treatments, and no difference among cements was found when applying EP (Table 3). Regarding the survival rate, the similar findings could be noticed: for instance, at 900 N, all the specimens in the TBS group had already failed, whereas HF and EP samples had more than 47% and 27% of survival chance, respectively (Table 4).

The topographic analysis showed distinct ceramic surface patterns depending on the surface treatment. Whereas the EP seems to not alter the surface created during CAD/CAM milling simulation (in the section Specimens Preparation: Ceramic), HF etching seems to soften the roughness created during this process, and TBS created a more irregular surface than the other treatments (Figure 1).

The fractographic analysis showed that the failure pattern was similar for all the groups. The origin of the radial cracks seemed to be located at the ceramic intaglio surface, and it propagated toward the surface of the load application (ceramic occlusal side) (Figure 2). In addition, no failures due to contact damage (Hertzian cone cracks) were observed.

DISCUSSION

The results of the present study show that the surface treatment and the type of cement used influenced the fatigue failure load, number of cycles for failure, and the survival rates of the ZLS (VITA Suprinity), and the study hypotheses were rejected. As a glass-ceramic, the most recommended surface treatment for ZLS is the etching with 5% HF followed by the silane agent application.[30] This treatment promotes selective removal of the glassy matrix, cleans the surface, and creates microretention, increasing the surface wettability,[13,27] besides removing and/or smoothening the surface defects resulting from CAD/CAM process.[27,31] Strasser and others[13] stated that the HF etching followed by the silane agent application provided a greater flexural strength when compared to the TBS followed by the silane application in a ZLS, corroborating our results (Table 3).

During the TBS with silica-coated alumina particles, the high energy generated by the impact of the particles onto the surface leads to incrustation and coating of the silica layer to the ceramic surface,[27,30] modifying it chemically.[32] However, the impact of the particles also

Table 3: *Results for Fatigue Failure Load (FFL) and Number of Cycles for Failure (CFF)*[a]

Groups	FFL (N) - Mean (CI)	CFF - Mean (CI)
nMDP+HF	973.33 (891-1055) B	72,333 (64,113-80,553) B
nMDP+EP	866.67 (759-974) B	61,666 (50,902-72,430) B
nMDP+TBS	546.67 (514-579) D	29,666 (26,428-32,905) D
MDP+HF	986.67 (920-1052) B	75,000 (67,843-82,156) B
MDP+EP	1066.67 (993-1139) AB	81,666 (74,341-88,991) AB
MDP+TBS	546.67 (520-572) D	29,666 (27,053-32,280) D
SA+HF	1206.67 (1086-1326) A	95,666 (83,650-107,683) A
SA+EP	1026.67 (942-1111) AB	77,666 (69,227-86,105) AB
SA+TBS	733.33 (702-764) C	48,333 (45,209-51,456) C

Abbreviations: CI, confidence interval; EP, etch & prime ceramic primer; HF, hydrofluoric acid; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; SA, self-adhesive resin cement; TBS, tribochemical silica coating.
[a]*Different uppercase letters in each column indicate significant statistical difference based on the Kaplan-Meier and Mantel-Cox (log-rank) tests (α=0.05).*

Table 4: *Survival Rates (Probability of the Specimens to Exceed the Respective Fatigue Failure Load and Number of Cycles for Failure Without Fail) of the Study Groups and Their Respective Standard Error Measurements[a]*

Fatigue Failure Load (N) / Number of Cycles (×10³) on the Respective Step[b]	Groups								
	nMDP+HF	nMDP+EP	nMDP+TBS	MDP+HF	MDP+EP	MSP+TBS	SA+HF	SA+EP	SA+TBS
400 / 15×10³	1	1	1	1	1	1	1	1	1
500 / 25×10³	1	1	0.40 (0.12)	1	1	0.47 (0.13)	1	1	1
600 / 35×10³	1	1	0.06 (0.06)	1	1	0.00 (0.00)	1	1	0.93 (0.06)
700 / 45×10³	1	0.67 (0.12)	0.00 (0.00)	0.93 (0.06)	1	—	1	1	0.40 (0.12)
800 / 55×10³	0.73 (0.11)	0.27 (0.11)	—	0.87 (0.08)	0.93 (0.06)	—	1	0.87 (0.09)	0.00 (0.00)
900 / 65×10³	0.47 (0.13)	0.27 (0.11)	—	0.73 (0.11)	0.80 (0.10)	—	1	0.60 (0.12)	—
1000 / 75×10³	0.27 (0.11)	0.20 (0.10)	—	0.20 (0.10)	0.53 (0.12)	—	0.80 (0.10)	0.40 (0.12)	—
1100 / 85×10³	0.20 (0.10)	0.13 (0.09)	—	0.13 (0.09)	0.27 (0.11)	—	0.47 (0.13)	0.27 (0.11)	—
1200 / 95×10³	0.07 (0.06)	0.07 (0.06)	—	0.00 (0.00)	0.13 (0.09)	—	0.20 (0.10)	0.13 (0.09)	—
1300 / 105×10³	0.00 (0.00)	0.00 (0.00)	—	—	0.00 (0.00)	—	0.13 (0.09)	0.00 (0.00)	—
1400 / 115×10³	—	0.00 (0.00)	—	—	—	—	0.13 (0.09)	—	—
1500 / 125×10³	—	—	—	—	—	—	0.13 (0.09)	—	—
1600 / 135×10³	—	—	—	—	—	—	0.13 (0.09)	—	—
1700 / 145×10³	—	—	—	—	—	—	0.06 (0.06)	—	—
1800 / 155×10³	—	—	—	—	—	—	0.00 (0.00)	—	—

Abbreviations: EP, etch & prime ceramic primer; HF, hydrofluoric acid; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; SA, self-adhesive resin cement; TBS, tribochemical silica coating.
[a] *"—" indicates absence of specimen being tested on the respective step.*
[b] *These values are approximated.*

causes a certain amount of abrasion, creating sharper defects and cracks at the ceramic surface,[13] as observed in the present study (Figure 1), which could be potential sites for the crack growth during function. According to Griffith's weakest link theory,[33] the greater the number of critical defects that exist, the greater the chance an earlier crack growth when the material is subjected to intermittent loading, as occurs in the oral environment during chewing. This is corroborated by others[9,34,35] who have shown the deleterious effect of a rougher ceramic surface when the defects are not properly filled by the adhesive bonding.

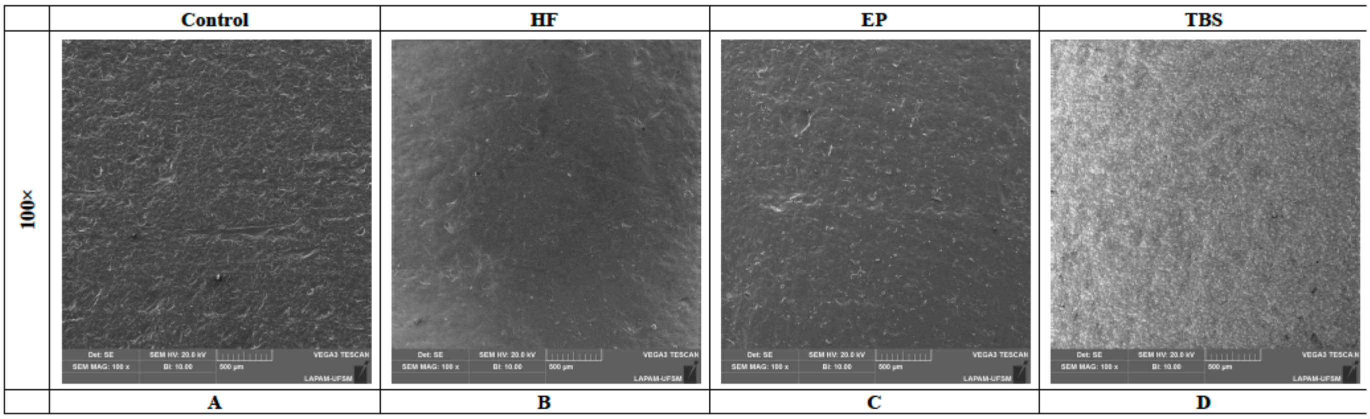

Figure 1. *Topographic images on scanning electron microscopy (100× magnification) of the ceramic surface on control (CAD/CAM milling simulation) (A) and after the surface treatments: (B) 5% hydrofluoric acid etching (HF); (C) Monobond Etch & Prime application (EP); (D) tribochemical silica coating (TBS).*

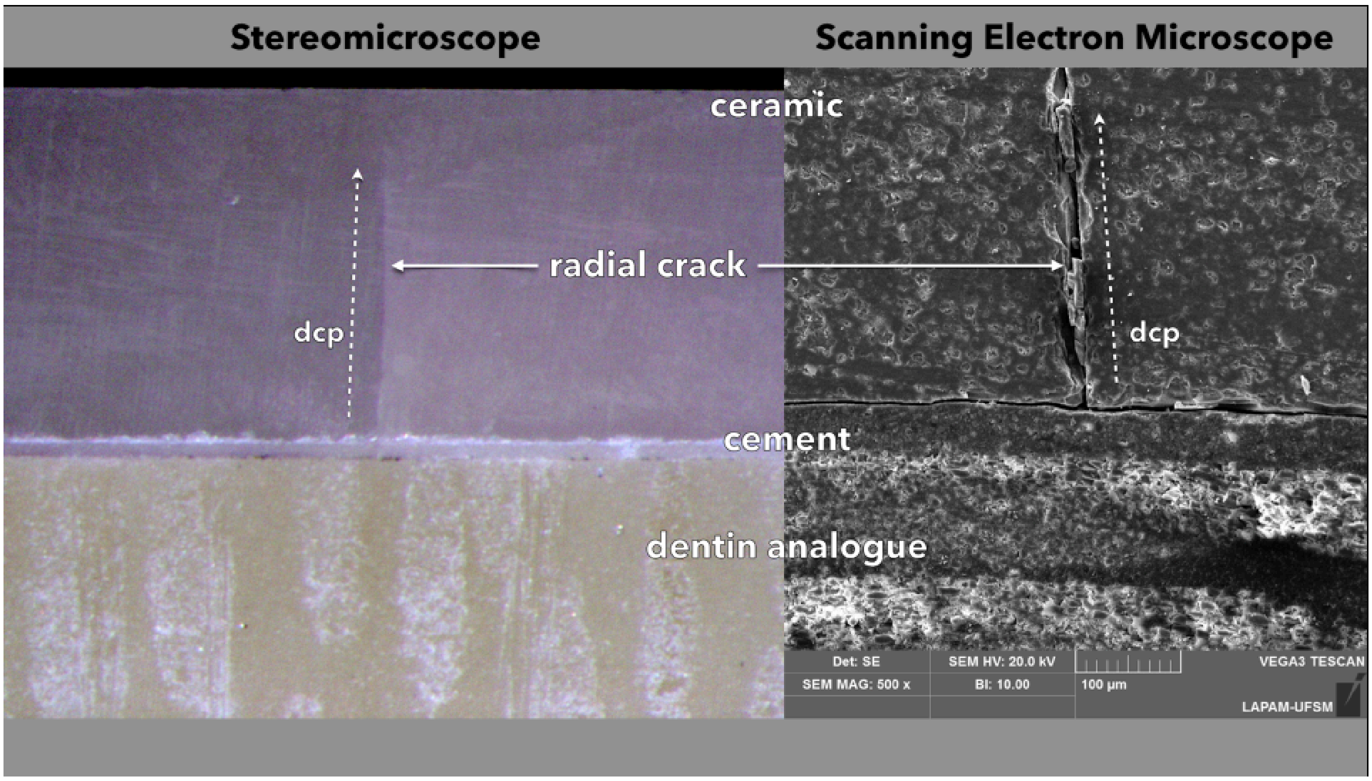

Figure 2. *Images on stereomicroscope (100× magnification) and scanning electron microscopy (500× magnification) of a representative failed specimen showing the crack characteristics after failure during the fatigue test. It seems that the failure origin is located at the intaglio ceramic surface, propagating toward the ceramic occlusal side. Abbreviation: dcp, direction of crack propagation.*

Another alternative for surface conditioning of vitreous ceramics is the application of a self-etching ceramic primer, such as the Monobond Etch & Prime (Ivoclar Vivadent), which consists of a combination of ammonium polyfluoride and silane (trimethoxysilypropyl methacrylate) in a single bottle, simplifying the conditioning technique.[36] Studies have shown a poor ability of such ceramic primer to properly modify the surface when compared to 5% HF etching in a lithium disilicate ceramic and in a ZLS ceramic.[12,13,37] Our study corroborates those findings since EP was not able to significantly modify the surface created during the CAD/CAM milling simulation (Figure 1). In the study of Prado and others,[36] the use of a self-etching ceramic primer, although providing lower bond strength values than HF etching followed by the silane application, promoted more stable long-term results. According to our results, the surface treatment with

EP was as effective as the HF, regardless of the cement applied; therefore, it could be a viable option for the ZLS ceramic surface treatment from the mechanical fatigue performance viewpoint.

The use of resin cements to bond the vitreous ceramic restorations improves their mechanical performance.[9,34,38,39] This finding has been related to the ability of resin cements to seal and modify the ceramic surface defects, possibly by creating compressive forces at the crack tips or simply by sealing the critical defects.[39] However, the explanation for this strengthening mechanism has not yet been fully clarified and requires further investigation.[39] The characteristics of the bonding systems can determine their ability to fill these defects, and the type, size, and content of the cement filler particles,[40] as well as the presence of bisphenol-A-diglycidylether dimethacrylate (Bis-GMA),[41] directly influence such ability. An increase in the filler content and the presence of Bis-GMA result in an increase in their viscosity (Table 1).[41,42] In this sense, a low viscosity cement is preferable since it generates a better intimacy between the ceramic surface defects and the infiltrating bonding system, resulting in a greater capacity of filling the defects.[43,44] Furthermore, it also provides a thinner cement thickness, resulting in lower shrinkage stress, reducing the possibility of gaps and resin cement sorption and solubility.[40,45]

The results of our study show a difference between the cements used when the ceramic surface was treated with HF or TBS, and the self-adhesive resin cement presented better fatigue performance than the other cements in such groups (Table 3). Although MDP-free conventional resin cement (Multilink N) has a lower filler particle content,[43] which would decrease its viscosity in relation to the other resin cements, the presence of a Bis-GMA monomer in its composition is responsible for increasing its viscosity (Table 1).[46] Thus, even with a higher filler content (Table 1), the self-adhesive resin cement (RelyX U200)[44] seems to be more effective in penetrating deeper into the irregularities of the glass ceramic surface and improving the adhesive bonding and consequently its fatigue performance.[40] Also, the methacrylate monomers with phosphoric ester functional groups in the self-adhesive resin cement are able to improve adhesion by creating hydrogen bonds with the ceramic surface.[45] However, no difference between the cements was observed when the surface was treated with the ceramic primer (EP) (Table 3), which may be explained since the EP did not create an irregular surface and all the cements had the same capacity for filling the defects created on such surfaces.

Although laboratory tests are important in determining properties and characteristics of materials, they do not fully simulate *in vivo* conditions. Simplified restoration, besides providing a more standardized approach, allows for a less complex stress distribution, and the factors under study are better evaluated. The present study has some limitations, such as it does not apply the load with sliding contact, from which ceramic prostheses are subjected under normal and tangential loads during chewing, and it can generate surface damage accumulation, consequently reducing the load-bearing capacity.[47] In addition, as an accelerated fatigue test method, caution is required when evaluating the results.

CONCLUSION

1. Different surface treatments and resin cements directly influence the fatigue behavior of a bonded simplified ZLS.
2. The hydrofluoric acid etching and the self-etching ceramic primer provided better fatigue behavior than TBS, regardless of the cement used.
3. HF etching followed by the use of the self-adhesive resin cement created better fatigue results, being only equal to EP-treated ceramic when bonded with self-adhesive resin cement A or MDP cements.

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Conflicts of Interest

The authors 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.

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