The effect of silane applied to glass ceramics on surface structure and bonding strength at different temperatures

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PURPOSE. To evaluate the effect of various surface treatments on the surface structure and shear bond strength (SBS) of different ceramics. **MATERIALS AND METHODS.** 288 specimens (lithium-disilicate, leucite-reinforced, and glass infiltrated zirconia) were first divided into two groups according to the resin cement used, and were later divided into four groups according to the given surface treatments: G1 (hydrofluoric acid (HF)+silane), G2 (silane alone-no heat-treatment), G3 (silane alone-then dried with 60°C heat-treatment), and G4 (silane alone-then dried with 100°C heat-treatment). Two different adhesive luting systems were applied onto the ceramic discs in all groups. SBS (in MPa) was calculated from the failure load per bonded area (in N/mm^2). Subsequently, one specimen from each group was prepared for SEM evaluation of the separated-resin–ceramic interface. **RESULTS.** SBS values of G1 were significantly higher than those of the other groups in the lithium disilicate ceramic and leucite reinforced ceramic, and the SBS values of G4 and G1 were significantly higher than those of G2 and G3 in glass infiltrated zirconia. The three-way ANOVA revealed that the SBS values were significantly affected by the type of resin cement ($P<.001$). FIN ceramics had the highest rate of cohesive failure on the ceramic surfaces than other ceramic groups. AFM images showed that the surface treatment groups exhibited similar topographies, except the group treated with HF. **CONCLUSION.** The heat treatment was not sufficient to achieve high SBS values as compared with HF acid etching. The surface topography of ceramics was affected by surface treatments. ***[J Adv Prosthodont 2016;8:75-84]***

KEY WORDS: AFM; Silane treatment; Heat treatment; Bond strength; Surface treatment; SEM analysis

INTRODUCTION

New ceramic systems for dental application include ceramic cores reinforced with leucite[1-5] and zirconium dioxide (zirconia, ZrO_2).[6,7] The success of all-ceramic restorations depends on the cementation procedures, which are related to the type of ceramic materials, surface-conditioning techniques, and cementing agents.[8-10] Different surface-conditioning methods are used on surfaces of the different types of ceramics, which have different chemical compositions.[7,10]

Different surface treatments, such as grinding with diamond burs, air-particle abrasion, and acid etching, have been studied *in vitro* to improve the bond strength of composite resins to ceramics.[11-13] The surfaces of glass-infiltrated ceramics and lithium-disilicate-based ceramics, namely acid-sensitive materials, are modified by hydrofluoric acid (HF) gel.[10] Acid etching followed by the application of a silane coupling agent is a well-accepted and recommended method for increasing bond strength.[7,14] Hydrofluoric acid dissolves the glassy surface on the ceramic matrix, creating surface pits.[15] Surface etching with hydrofluoric acid and the subsequent application of silane increase the wettability and form covalent bonds between the ceramic and the resin cement.[15] Silane coupling agents establish adhesion, which occurs between the inorganic phase of the ceramic and the

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organic phase of the bonding agent applied to the ceramic surface, by forming a siloxane bond correspondingly to an increase in the surface energy of the ceramics and the wettability of the cement,[10] resulting in microscopic interactions between both parts.[10]

Despite the functions of hydrofluoric acid, the HF-etching step is not required and can be removed from the ceramic-restoration procedure for the following reasons: (1) Hydrofluoric acid is highly toxic and can pose severe health hazards.[16] (2) It has been stated that etching silica-based ceramics with hydrofluoric acid produces insoluble by-products consisting of silica fluoride salts on the surface,[15,17] and the remaining by-products can disrupt the bond strength of the resin.[18] (3) Hydrofluoric acid etching is not suitable for some recently developed glass ceramics with high crystalline structures. Therefore, it would be extremely advantageous to remove this step for those ceramics, and it would be possible to do so only if a strong enough silane bond can be otherwise established.[15]

If glass is infiltrated into zirconia ceramics, the formation of a ceramic matrix occurs as they are melted together at high temperatures.[7] Strong covalent bonds occur between the chemical constituents of the ceramics (traces, such as Li_2O, Na_2O, K_2O, CaO, and MgO) and functional groups at the surface of the ceramic material.[18] Acid etching creates more hydroxyl groups on the surface and increases micromechanical retention.[7] In addition, the formation of silanol groups occur when the methoxy groups of silane react with water, which after turns into a form of siloxane network as the methoxy groups of silane react with the surface of hydroxyl groups.[7]

Heat treatment improves silane performance by eliminating alcohol, water, and other by-products on the ceramic surface.[10,19,20] To improve the effectiveness and stabilization of reaction, heat treatment was made between silane and ceramic and their interface by completing the condensation.[10,19,20] Thus, eliminating the use of hazardous HF acid gel during the cementation phase is made possible with this stable silane reaction.[10]

Resin-based cements are the materials of choice for the luting cement of ceramic restorations.[21] When compared to other luting cements, resin cements show low solubility under oral conditions and bond effectively to different dental or ceramic surfaces.[10,22] Resin-based luting cements suitable for glass-infiltrated ceramics are usually chemically formed from a urethane dimethacrylate (UEDMA) or bisphenol A-glycidyl methacrylate (bis-GMA) matrix integrated with other monomers of lower molecular weight, such as triethylene glycol dimethacrylate (TEGDMA).[10]

It has been demonstrated that improving the condensation reaction of the silane results in the higher bond strength of resin to ceramic.[15,17,23] In this study, the effects of heat treatment, silane, and the hydrofluoric acid etching step were investigated. Our objective was to determine whether hydrofluoric acid etching is necessary with post-silanization heat treatment to improve the shear bond strength (SBS) of the resin cement to the ceramic. The null hypothesis was that different surface treatments would not affect the SBS of the resin cement to the ceramic.

MATERIALS AND METHODS

This *in vitro* study was approved by the ethics committee of the University of Selcuk. The study was designed to have a power of 99% at $\alpha = 0.05$. In this study, 312 all-ceramic specimens were fabricated from (1) IPS Empress e-max (IEX), a lithium-disilicate glass ceramic (Ivoclar Vivadent AG; Schaan, Liechtenstein); (2) Finesse All-Ceramic (FIN), a leucite reinforced ceramic (Dentsply; New York, NY, USA); and (3) In-Ceram zirconia (ICZ), a glass-infiltrated zirconia (Bad Säckingen, Germany), in accordance with the manufacturers' instructions (Table 1). Of the 312 specimens, 288 were first divided into two groups, with one for each of the resin cements. Then, each group of 144 specimens were divided into three subgroups, with one for each ceramic system; these specimens were subjected to different surface treatments (n = 12). The remaining 24 specimens were examined under atomic force microscopy (AFM) and

Table 1. Types of ceramics with codes, and manufacturing company names

Ceramics	Structure of ceramics	Suggesting surface treatment	manufacturer	Lot number
IPS e.max Press	Including crystalized pin point litium disilicate within 70% glass matrix. Additional contents: Li_2O, K_2O, MgO, ZnO, Al_2O_3, P_2O_5 and other oxides	Etching with 5% hydrofluoric acid for 20 s	Ivoclar Vivadent, Schaan, Liechtenstein	N68326
Finesse All Ceramic	Feldspathic glass ceramic system reinforced with 8-10% leucite crystals	Etching with 5% hydrofluoric acid for 60 s	Dentsply, Newyork, USA	291106
In-Ceram Zirconia	In-Ceram Zirconia powder includes 69.2% Al_2O_3, approximately 30.8% t-ZrO_2 (Ce-stabilize) by weight and then glass is infiltrated	Etching with 5% hydrofluoric acid for 20 s	Bad Sackingen, Germany	25730 16900

Table 2. Types of resin cements with codes, and manufacturing company names

Resin luting cement systems	Cement-silane bonding agent	Composition	Manufacturer	Lot number
Variolink N	Variolink N Base	Monomer matrix: Bis-GMA*, UDMAα and TEG-DMAβ Inorganic fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass catalysts, stabilizers and pigments	Ivoclar Vivadent, Schaan, Liechtenstein	R32733 P44912
	Monobond-S	3-MPSΦ (1 wt%) water-ethanol solution containing acetic acid at pH 4 (99 wt%)		P20956
	Heliobond	Bis-GMA* (60 wt%) Triethylene glycol dymethacrylate (40 wt%)		R23145
Clearfil Esthetic Cement	Clearfil Esthetic Cement Base	BPEDMAθ, MDPλ, DMAэ, 78% fillers	Kuraray, Osaka, Japan	033AAA
	Clearfil Ceramic Primer	Phosphate monomer MDP		00019D

scanning electron microscopy (SEM) (see below). Light-cured (Clearfil Esthetic (CE), Kuraray Medical Inc., Osaka, Japan) and dual-cured (Variolink N (VN), Ivoclar Vivadent AG, Schaan, Liechtenstein) resin cements were used in the study (Table 2). The IEX and FIN ingots were used to produce porcelain discs by the lost-wax method with metal molds according to manufacturer's recommendations. The specimens were also obtained from the ICZ ingots with a precision saw to form discs with dimensions of 8 × 10 × 1.5 mm. The acquired ICZ specimens were infiltrated with glass according to the manufacturer's instructions. Before the bonding procedure, the specimens were embedded in clear, chemically polymerizing acrylic resin (SC self-cure acrylic, Imicryl, Konya, Turkey) using a PVC ring measuring 20 mm in height and 25 mm in diameter, leaving one surface of the disc uncovered for bonding. Four surface-conditioning techniques for the ceramic materials were explored with different storage conditions, namely dried and thermocycled. The surface of each specimen was ground with a silicone carbide abrasive (RotoPol-11, Struers A/S, Rødovre, Denmark) and then final-finished using a grit size of 800 grit. An ultrasonic bath (Quantrex 90 WT, L&R Manufacturing, Inc., Kearny, NJ, USA) with acetone was used for cleaning for 5 minutes, after which the specimens were air-dried.

After completing specimens, surface contioning methods were started;

- Group 1: The ceramic specimens were etched with 5% hydrofluoric acid gel (IPS Ceramic Etching gel, Ivoclar Vivadent AG, Schaan, Liechtenstein), which was applied for 20 seconds on IEX and 60 seconds on FIN and ICZ. The specimens were then rinsed for 60 seconds and air-dried for 60 seconds. Silane coupling agents (Monobond S for Variolink N; Clearfil Ceramic Primer for Clearfil Esthetic) were applied according to the manufacturers' instructions and air-dried for 30 seconds.
- Group 2: Silane coupling agents were applied according to the manufacturers' instructions for 60 seconds and air-dried with compressed oil-free air for 30 seconds.
- Group 3: Silane coupling agents were applied for 60 seconds, after which heat treatment was applied in a furnace (FN 400, Nüve, Ankara, Turkey) at 60°C for 60 seconds.
- Group 4: Silane coupling agents were applied for 60 seconds, after which heat treatment was applied in a furnace (FN 400, Nüve, Ankara, Turkey) at 100°C for 60 seconds.

Following steps were made after the surface conditioning for the bonding procedure. All materials were mixed and applied by the same operator for consistency. Both of the resin cements were mixed according to the manufacturers' instructions and injected into tubes, and the bonding process was also performed as suggested by the manufacturers. The resin cements were photopolymerized for 20 seconds from the top surface using a light-emitting diode unit (Bluephase G2, Ivoclar-Vivadent, Schaan, Liechtenstein), which emitted radiation at a wavelength of 380-515 nm and intensity of 900 mW/cm^2; these values were measured with a radiometer (Bluephase Meter, Ivoclar-Vivadent). After the polyethylene molds were removed, the resin cements were light-cured for 40 seconds on each side.

The ceramic-cement assembly was then washed with an air-water spray and kept in distilled water at 37°C for 24 hours. All of the groups were subjected to thermocycling (Thermal Cycler Tester, Dental Teknik, Konya, Turkey) for 10,000 cycles between 5°C and 55°C in deionized water. The dwelling time at each temperature was 20 seconds. The transfer time from one bath to the other was 10 seconds. The SBS test was applied to the adhesive interface with a universal testing machine (Shimadzu AGS-X, Shimadzu Corporation, Tokyo, Japan) at a crosshead speed of 0.5 mm/min until bonding failure occurred (Fig. 1).

After the SBS test, the ceramic bonding areas were

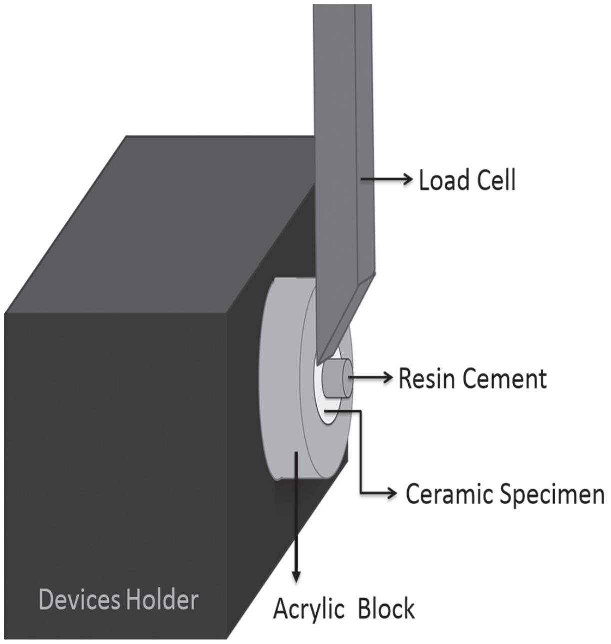

Fig. 1. Scheme of the test sample.

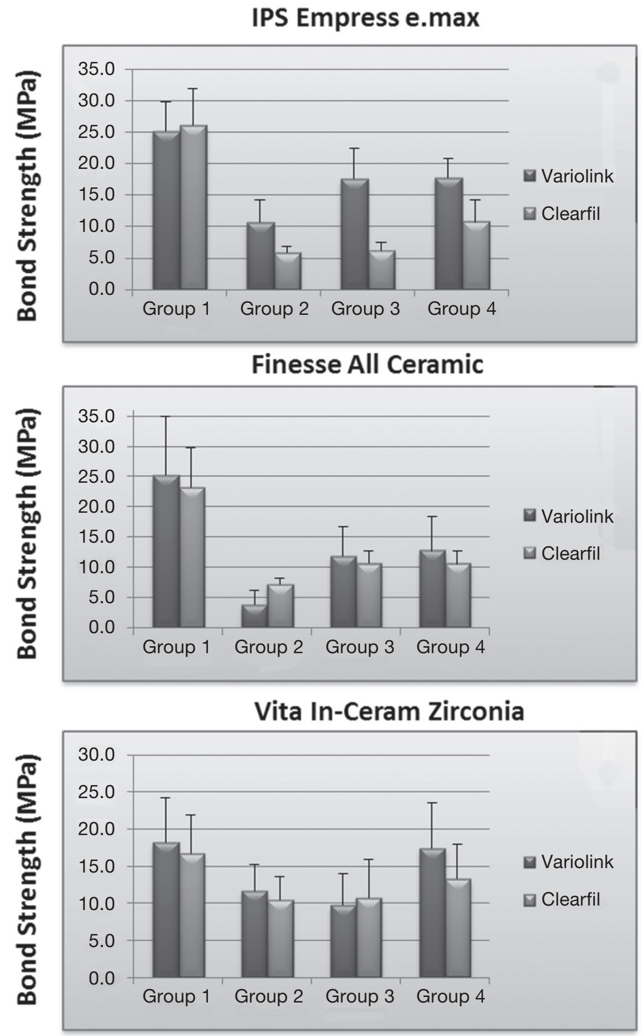

Fig. 2. The diagrams of the SBS values of the IEX, FIN and ICZ specimens subjected to the different surface treatments.

observed with a stereomicroscope (CX41, ×40, Olympus, Tokyo, Japan) to characterize the mode of fracture. The fractured surface was classified as one of four types: Type 1, adhesive failure between the ceramic surface and the resin cement; Type 2, cohesive failure in the ceramic; Type 3, cohesive failure in the resin cement; Type 4, mixed failure in the resin cement and the ceramic.

One specimen from each group was evaluated by AFM (NT-MDT, NTEGRA Solaris, Moscow, Russia); the digital images were captured in air. A gold-doped silicon tip (40 µm) with resistivity of 0.01-0.025 Ω cm was used in the non-contact mode. The height of the image was defined by the changes in the vertical position, which were recorded as bright and dark regions. A constant tip sample "tap" was supplied by using constant oscillation amplitude (set-point amplitude). Twenty-four 25 × 25 µm digital images were obtained for each surface, and they were recorded at a slow scan rate of 1 Hz.

Final analysis was made by scanning electron microscopy. One specimen from each group was prepared for SEM (JSM-5600; JEOL Ltd., Tokyo, Japan) evaluation of the debonded-resin-ceramic interface. After debonding, the specimens were sputter-coated (Polaron SC500 Sputter Coater, VG Microtech, E. Sussex, England) with a gold-palladium alloy under vacuum. The photomicrographs were obtained and the ceramic surface was examined by SEM at ~×20 magnification.

Statistical analysis was performed using SPSS 20.0 software for Windows (SPSS/PC version 20.0, SPSS, Chicago, IL, USA). The data were analyzed by three-way analysis of variance (ANOVA) and Tukey's honestly significant difference (HSD) tests for paired comparisons among the groups ($P < .05$). P values below 0.05 were considered to be statistically significant in all tests.

RESULTS

The results of the SBS test for the treated groups are shown in Fig. 2. As ANOVA demonstrated, the type of ceramic had a significant effect on the shear bond strength ($P = .0001$). On the other hand, different methods of surface conditioning showed smaller differences (Table 3, Table 4).

The highest SBS values for HF acid etching were obtained with glass ceramics (FIN and IEX) in all surface-treatment groups, with the results varying between 18.2 and 26.1 MPa. The lowest SBS values for all of the ceramics groups were obtained in group 2 (silane etching only and no heat treatment), with the results varying between 3.8 and 11.7 MPa.

Table 3. Results of three way ANOVA

	Type III SS	df	MS	F	Sig.
Ceramic	183.474	2	91.737	4.293	.015
Surface	8069.810	3	2689.937	125.878	.000
Resin cement	447.728	1	447.728	20.952	.000
Ceramic * Surface	1312.381	6	218.730	10.236	.000
Ceramic * Resin cement	339.409	2	169.704	7.941	.000
Surface * Resin cement	193.692	3	64.564	3.021	.030
Ceramic * Surface * Resin cement	470.917	6	78.486	3.673	.002

Table 4. Shear bond strengths (SBSs) of the various groups (in MPa)

Ceramic	IEX		FIN		ICZ	
Resin	VN	CE	VN	CE	VN	CE
Group 1 (hydrofluoric acid + silane)	25.16 ± 4.61	26.07 ± 8.94	25.20 ± 9.68	23.25 ± 6.50	18.23 ± 6.02	16.69 ± 5.25
Group 2 (silane alone, no heat treatment)	10.67 ± 3.55	5.91 ± 0.90	3.79 ± 2.50	7.13 ± 1.16	11.71 ± 3.54	10.49 ± 3.11
Group 3 (silane alone then dried with heat treatment at 60°C)	17.55 ± 4.81	6.27 ± 1.22	11.81 ± 5.00	10.66 ± 2.09	9.76 ± 4.17	10.75 ± 5.11
Group 4 (silane alone then dried with heat treatment at 100°C)	17.69 ± 3.08	10.78 ± 3.41	12.80 ± 5.65	10.62 ± 2.07	17.45 ± 6.11	13.26 ± 4.72

The silane combined with heat treatment at 60 or 100ºC did not increase the results in the etched groups ($P > .05$).

The three-way ANOVA revealed that the SBS values were significantly affected by the type of resin cement ($P < .001$). The IEX ceramic that was etched with HF and bonded with CE resin cement showed the highest shear bond strength (26.1 MPa).

The microscopic examination of the failures demonstrated that the IEX ceramics had the highest percentage of adhesive failures along the ceramic surface in the specimens from group 2 (96%), group 3 (58%), and group 4 (79%). On the other hand, group 1 specimens that were etched with HF showed mixed cohesive failures (50%), with a thin layer of resin cement remaining on the ceramic surface (Fig. 3). The FIN ceramics had the highest percentage of cohesive failures on the ceramic surfaces (group 1, 100%; group 3, 79%; group 4, 83%), while group 2 specimens (etched with silane and no heat treatment) had adhesive failures (79%). Finally, the ICZ ceramics had the highest percentage of adhesive and mixed failures on the ceramic surfaces, with no cohesive failure in the ceramic structure.

Fig. 4 shows the representative AFM images of the three ceramic groups that were treated by the different surface-conditioning methods and bonded with two different resin cements. The surface-treated specimens exhibited similar topographies, except those from the group treated with HF. The heat-treated groups exhibited moderate irregularity and less roughness than the group treated with HF acid (group 1). The IEX and FIN ceramics that were etched with HF had the most distinct sharp peaks.

DISCUSSION

The cementation of restorative material, ceramic material, and tooth structure is influenced by several factors such as the luting cement type,[24,25] the ceramic type, and tooth enamel or dentin structure.[7,15] Two factors should be considered in the bonding of ceramics to the tooth: the resin-ceramic interface and the resin-tooth interface.[7,15] According to the results of this study, the heat treatment of silane did not increase the SBS values; thus, the null hypothesis was accepted.

Several test methods have been reported for the evaluation of SBS.[15] Measuring the SBS is a currently acceptable testing method for resin-cement luting systems, and it was used in the present study.[26] The two materials are connected with an adhesive agent in the SBS testing method, and they

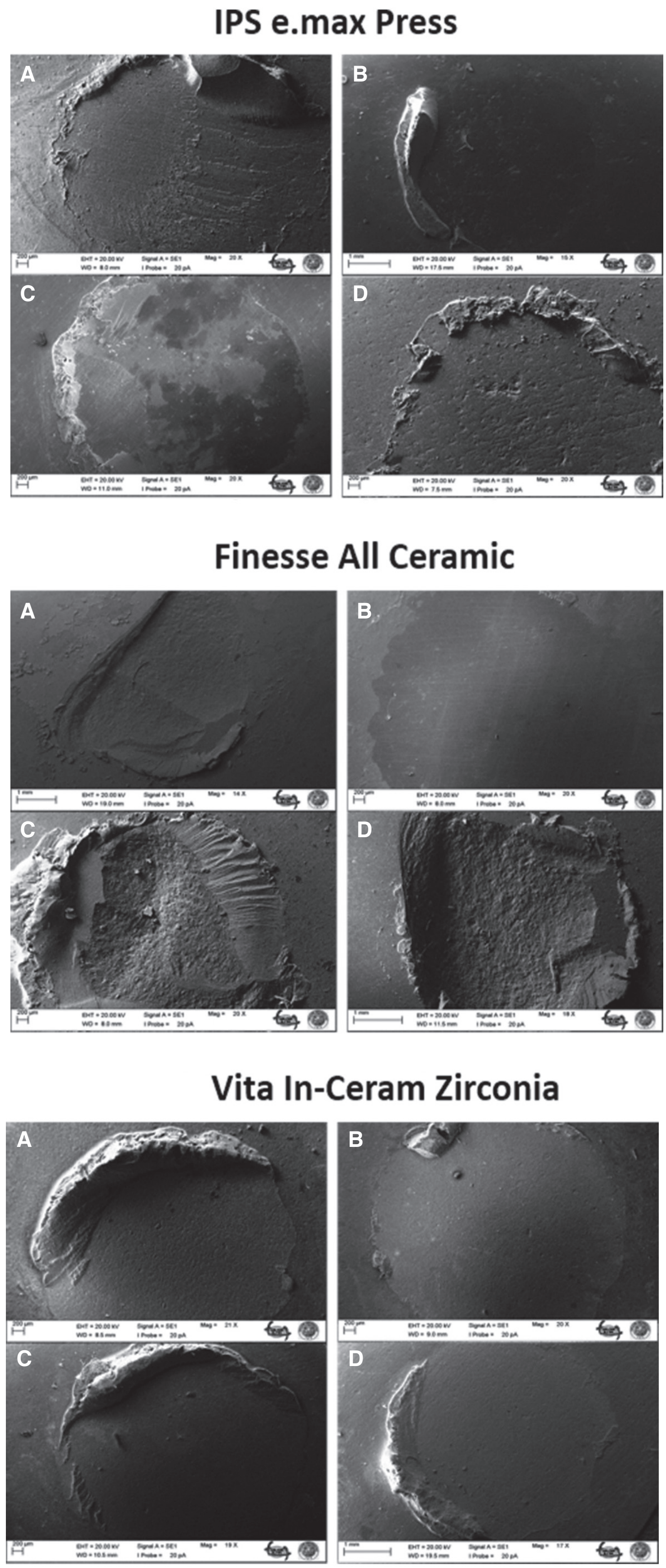

Fig. 3. SEM images of the IEX, FIN and ICZ specimens subjected to different surface treatments, which are the representative images of failure types after SBS test. (A) hydrofluoric acid (HF)+silane, (B) silane alone-no heat-treatment, (C) silane alone-then dried with 60°C heat-treatment, and (D) silane alone-then dried with 100°C heat-treatment.

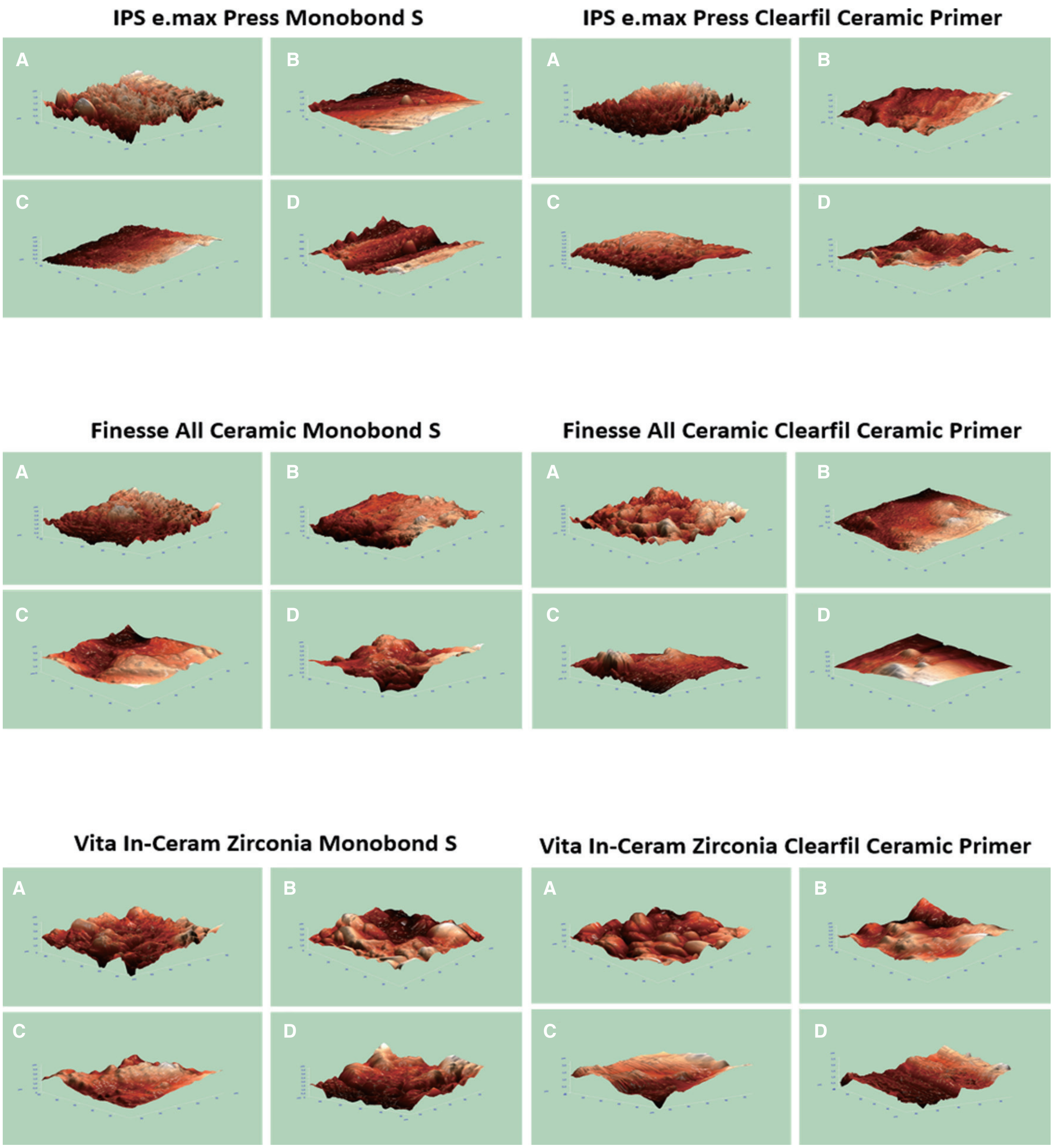

Fig. 4. AFM images of the IEX, FIN and ICZ specimens subjected to different surface treatments. (A) hydrofluoric acid (HF) + silane, (B) silane alone-no heat treatment, (C) silane alone-then dried with 60°C heat treatment, and (D) silane alone- then dried with 100°C heat treatment.

are loaded under shear stress until separation occurs.[26,27] In the present study, this test was used for several reasons such as easy specimen preparation, simple application of test protocol, and the ability to rank different products according to the bond strengths.[28] Besides the easy standardization of the prepared specimens, the advantage of the SBS test includes an easily observed cross-sectional surface, and the clinical preference for the SBS test is related to the rate of loading.[28]

Applying the silane coupling agent with heat eliminates alcohol, water, and other by-products from the surface of the ceramic.[10,29] In addition, the heat treatment helps by the completion of the silane-ceramic condensation reaction, making the covalent bond more effective and resistant.[10,14,19] In the present study, the heat treatment was applied by using two different silane agents on three different glass ceramic surfaces. In the previous studies, the heat treatment of silane was performed in different ways. Fabianelli *et al.*[15] applied hot air at 100°C for 1 minute and then at 50°C for 15 seconds. Moharamzadeh *et al.*[30] attempted heat treatment in a furnace at 100°C for 2 minutes. In the present study, the application of silane with heat treatment was performed with the heat treatments at 100°C and 60°C for only 1 minute each. The results of the present study did not confirm the results of Fabianelli *et al.*[15] The discrepancy of the results can be explained by the difference in test methods. Besides, no difference was observed between shear strength, neighter in this present study nor in the study of Carvalho *et al.*[10] Moreover, hot air was used in study of Fabianelli *et al.*,[15] while a heated oven was used in our study. The differences in the results of two studies can also be explained by the use of different ceramic systems and resin cements.

In vitro testing of luting cements is very important for improvement of the new resin-cement systems and for proving the reliability of the products. The longevity of a ceramic restoration depends on the durable bonding between the ceramic and the resin cement, which is provided by surface conditioning.[10,22] In our study, for both resin cements, using a combination of hydrofluoric acid etching and conventional silane treatment yielded significantly higher SBSs than those of all three different ceramic systems that were treated with silane without hydrofluoric acid etching. In the drying step of silane appliance, there were no significant effects on the SBS values in any group during heat treatment. Hexafluorosilica was formed by the selective reaction of hydrofluoric acid with the silica that was present on the microstructure of the ceramics. As a result of this reaction, the surface became irregular and porous with the dissolution of the glassy phase, which increased the surface area and enabled the bonding agent to penetrate the micro-spaces of the acid-conditioned ceramic surface.[10,15,31] However, the bond strengths of the ICZ ceramic groups were low compared to those of the other ceramic groups, which can be explained by a poor glassy phase.

When the two different resin cement systems were compared in group 1, the VN resin cement showed higher bond strengths than the CE resin cement for the FIN and ICZ ceramics. In general, although the same HF acid agent was used, the VN resin cement exhibited a higher bond strength than the CE resin cement, which can be explained by the difference in the recommended silane content. The ceramic-resin interface was bonded by a silane coupling agent, unfortunately this layer could be unstable and cause hydrolytic degradation between adhesive interface (Bis-GMA).[32] However; to prevent this Clearfil Ceramic Primer was used in this study, which contains a phosphate monomer (MDP), so that we provided a stable chemical union that was resistance to hydrolytic degradation.[29] this could explain the resistance values obtained in the group that did not recieve effective heat treatment of the silane.[29] The silane coupling agent reaction may vary with other resin cement systems containing methacrylate monomers.[10]

The IEX and FIN ceramic groups, which were not treated with HF etching, showed almost 50% lower mean bond strengths. These results show that the SBS values of etching-and-silane-application are higher than the silane-and-heat treatment's values. On the other hand, silane coupling agents are important in enhancing the bond strength of composite resins to silica-based ceramics.[10,11,23,33] As silane agents are considered organic bifunctional molecules, they promote chemical bonding between the silicone dioxide and the OH groups on the ceramic surfaces.[34] In addition, silane agents have a degradable functional group that copolymerizes with the organic matrix of the resin cement.[35,36] The use of silane also enhances the wettability of the ceramic surface.[33]

The possible effect of thermocycles during experimental studies must be evaluated.[7] The application of thermocycles usually decreases the bond strength.[7,23,37] However, some other researchers reported no such decrease.[38] The differences in the results might be explained by variations in the experimental set-ups since *in vitro* studies are designed to simulate different clinical situations.[7] In this study, the SBS test was performed after 10,000 thermocycles.

The data on the mode of failure demonstrated that both types of resin cement (VN and CE) in our study had the highest frequency of adhesive failures along the resin cement surface, while the FIN ceramics had mostly mixed and cohesive failures with a thin layer of resin luting cement remaining on the ceramic surface. The cohesive fractures of the FIN ceramics might be explained by the lower cohesive strength of the material. On the other hand, because zirconia has a very solid structure compared with other groups, there were no cohesive or mixed failures in the ICZ groups for the two types of resin cements.

This *in vitro* study had limitations in its ability to simulate clinical loading forces on resin cements and changes in the oral environment.[39] In the SBS tests, the loading was monotonic instead of being representative of the cyclic fatigue in the oral cavity. These important aspects should be added to future studies, as many factors affect the SBSs of the resin cements used for the ceramics.[39,40] Future studies should resemble the oral environment and simulate clinical loading conditions[39] for the different types of resin cements and silane coupling agents. At the same time, it is recommended that silane is not only heated in an oven but also dried with hot air, and results of the two groups with and without the drying should be compared with each other. Comparing the shear method, used in this study, with other test methods should also be conducted.

CONCLUSION

Within the limitations of this *in vitro* study, the main conclusions are as follows.

The heat treatment on the silane coupling agent did not yield the sufficient SBS values and did not create physical changes in the surface topography when compared with HF acid etching. HF acid etching followed by the application of silane coupling agent is a recommended method for the IEX bonded with CE resin cement.

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