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Effect of Different Surface Treatments on Porcelain-Resin Bond Strength

Tevfik Yavuz, DDS, PhD,[1] Özgün Yusuf Özyılmaz, DDS, PhD,[2] Erhan Dilber, DDS, PhD,[3] Elif Sümeyye Tobi, DDS,[4] & Hamdi Şükür Kiliç, PhD[5]

[1]Department of Prosthodontics, Faculty of Dentistry, Abant Izzet Baysal University, Bolu, Turkey
[2]Department of Prosthodontics, Faculty of Dentistry, İstanbul Medipol University, İstanbul, Turkey
[3]Department of Prosthodontics, Faculty of Dentistry, Sifa University, Izmir, Turkey
[4]Department of Prosthodontics, Faculty of Dentistry, Selcuk University, Konya, Turkey
[5]Department of Physics, Faculty of Science, Selcuk University, Konya, Turkey

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Correspondence
Erhan Dilber, Department of Prosthodontics, Faculty of Dentistry, Sifa University, Mansuroğlu Mah. 293/1 No: 14, 35100, Bayrakli, Izmir, Turkey.
E-mail: dilberhan@gmail.com

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Abstract

Purpose: The aim of this study was to evaluate the effects of various surface treatments on the surface structure and shear bond strength (SBS) of different ceramics.
Materials and Methods: A total of 192 disk-shaped cores were prepared using two all-ceramic systems, of which 168 were submitted to SBS tests, and 24 were investigated by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The ceramics used were IPS Empress e.max (EX) lithium glass-ceramic and Vita In-Ceram Zirconia glass-infiltrated zirconia (ICZ). The specimens were randomly divided into seven groups ($n = 12$) on the basis of the surface treatment used: control; SB—sandblasting with 50 μm Al_2O_3 particles; CJ—chairside silica coating with 30 μm SiO_2 particles and silanization (Clearfil Porcelain Bond Activator); HF—etching in 5% hydrofluoric acid and silanization; ER—etching with an Er:YAG laser (10 W); ND—Nd:YAG laser etching (0.8 W); and FS—etching with a femtosecond laser (860 mW). A luting cement (Clearfil Esthetic) was photopolymerized on each treated ceramic disk. After subjecting the specimens to thermocycling (1000 cycles, 5°C to 55°C), SBS tests were performed using a universal testing machine. The data were analyzed with two-way ANOVA and Tukey's tests using a significance limit of 5%.
Results: Among the EX ceramics, the CJ (29.10 MPa) and HF (26.07 MPa) specimens had statistically higher SBS values. For the ICZ ceramics, the highest value (28.08 MPa) was obtained for the CJ specimens.
Conclusions: Silanization after coating with silica improves the bond strengths of both EX and ICZ specimens, while HF etching is favorable only for the EX specimens.

The pleasing esthetics of all-ceramic restorations explains their increasing popularity in cosmetic dentistry. In addition, the biological properties of currently available all-ceramic systems render them suitable for tooth reconstructions and high-quality esthetic restorations for many clinical indications.[1,2] Furthermore, owing to improvements in their mechanical properties, all-ceramics can now also be used to restore both single and multiunit tooth defects.[3]

A number of surface pretreatments have been developed that mechanically facilitate the bonding between resin luting cements and ceramic surfaces.[2] The bonding of resin cements to feldspar, leucite, and lithium disilicate-based ceramics has been studied extensively.[4] The recommended method for conditioning the surfaces of ceramic restorations is to treat them with hydrofluoric acid[5] and subsequently apply a silane-coupling agent to ensure strong bonding.[6] Hydrofluoric acid creates a retentive surface for micromechanical bonding via the preferential dissolution of the glassy phase, and the applied silane-coupling agent increases the wettability of the surface and promotes the formation of covalent bonds between the methacrylate groups of the resin and the silica in the ceramic.[7,8]

Airborne-particle abrasion is a conventional surface treatment method that creates a rough, irregular surface and improves micromechanical retention by increasing the surface area and the adhesion energy of resin cements to all-ceramics.[9] Abrasion performed with aluminum oxide particles under pressure decreases the surface tension and improves the wettability of silane-coupling agents on ceramic surfaces.[10] During the last two decades, numerous alternative methods for treating ceramic surfaces have been developed.[11] One of the most popular techniques is the particle-deposition method, whereby silanization is preceded by chairside abrasion with 30 μm silica-coated aluminum oxide (Al_2O_3) particles,[11-13] which both roughen and chemically activate the surfaces of all-ceramics. Covalent bonds then form during silanization between the silica particles and the methacrylate groups of the resin cements.[14] Elsewhere, Bottino et al[15] found that tribochemical systems increased the bond strength between Vita In-Ceram zirconia and Panavia F resin cement.

Lasers have been used in dental clinical practices since the 1960s, but a number of new applications have recently come to the fore. Neodymium-doped yttrium aluminum garnet (Nd:YAG) lasers have been used to roughen the surfaces of solid ceramics to attach veneering porcelain or resin cements,[16,17] for tooth bleaching,[18] to reduce tooth sensitivity,[19] and to remove caries.[20] Akın et al[21] reported that etching the surface of zirconia with Nd:YAG and erbium-doped YAG (Er:YAG) lasers strengthened the subsequent bonding more than sandblasting did. Er:YAG lasers have also been used to modify ceramic surfaces, prepare cavities, and remove caries.[22] They also increase the bond strength between ceramic materials and resin luting cements, allowing for indirect restorations and the use of lithia-based ceramics.[23] Furthermore, the high optical penetration depth of Er:YAG radiation[24] means that it can be used directly on the solid surfaces of ceramics, making it an attractive alternative to other surface treatment methods.

Ultrashort-pulse laser systems (UPLS's) are used routinely in medicine, biology, and industry, notably for cataract surgery, nonlinear spectroscopic and fluorescence techniques, and the micromachining of materials. For the latter application, in particular, UPLS's cut with minimal collateral damage, allowing the ablation of thin layers with extreme precision and high reproducibility without any thermal side effects.[25] Lorenzo et al[26,27] reported that femtosecond laser treatment improved the shear bond strength (SBS) of brackets attached to enamel surfaces. This kind of treatment should therefore also be suitable for all-ceramics.

Although numerous studies have reported the advantages and disadvantages of different types of surface treatments applied to different ceramics,[3,11,14,23,28,29] a consensus on the optimal treatment to strengthen bonding between resin cements and ceramic surfaces has yet to be found. The present study was therefore conducted to evaluate the effects of various surface treatments on the surface structure of resin cements and on their bonding to different ceramics via scanning electron microscopy (SEM) and atomic force microscopy (AFM). The null hypothesis was that the different surface treatments would not affect the SBS of the resin cements and ceramics.

Materials and methods

Specimen preparation

In this study, 192 completely ceramic specimens were fabricated from IPS Empress e.max, a lithium-disilicate glass-ceramic (EX; Ivoclar, Schaan, Liechtenstein) and In-Ceram zirconia, a glass-infiltrated zirconia (ICZ; Vita Zahnfabrik, Bad Sackingen, Germany), in accordance with the manufacturers' instructions. The disk-shaped specimens were 10 mm in diameter and 2 mm thick. Of the 192 specimens, 168 were divided into two groups, one for each of the ceramic systems; these specimens were subjected to different surface treatments (n = 12). The remaining 24 specimens were examined by AFM and SEM. Prior to surface treatment, the specimens were embedded in acrylic resin blocks while ensuring that the bonding surface remained exposed. The bonding surfaces were polished using metallographic paper (600 to 1200 grit) and a polishing machine (Minitech 233; Presi, Grenoble, France) with water cooling, and then cleaned ultrasonically (Biosonic UC 50; Coltene Whaledent Inc., Cuyahoga Falls, OH) in distilled water for 10 minutes to remove any surface contaminants.

Surface treatment and cementation of ceramic blocks

Control

No additional surface treatment was performed.

Group SB (Sandblasting)

The ceramic surfaces were air-abraded with 50 μm Al_2O_3 particles (Korox; Bego, Bremen, Germany) from a distance of approximately 10 mm at a pressure of 2.7 atm for 20 seconds.

Group CJ (tribochemical silica coating)

Abrasion was performed using an airborne-particle abrasion device (Cojet System; 3M ESPE, Seefeld, Germany) filled with 30 μm alumina particles coated with silica (Cojetsand; 3M ESPE). The abrasive particles were blasted at a distance of 10 mm perpendicular to the specimen surface at a pressure of 2.7 atm for 15 seconds. The specimen was then washed and dried. After the surface treatment, air was gently blown on the specimen to remove any remnant particles.

Group HF (hydrofluoric acid)

The ceramic specimens were etched with a 5% hydrofluoric acid gel (IPS Ceramic Etching gel, Ivoclar) for 60 seconds, rinsed for 60 seconds, and then dried in air for 60 seconds.

Group ER (Er:YAG laser)

An Er:YAG laser (Fotona; AT Fidelis, Ljubljana, Slovenia) was used to irradiate the ceramic specimens. A contact handpiece (R14, 1.3 mm in diameter) with an integrated spray nozzle was placed perpendicular to the ceramic surface at a distance of 1 mm, and the entire surface of the specimen was scanned manually with the laser while being cooled with water. The laser-treatment parameters were as follows: pulse energy: 500 mJ; power: 10 W; pulse mode: medium short (100 μs pulse duration); pulse rate: 20 Hz; energy density: 37.68 J/cm^2.

Group ND (Nd:YAG laser irradiation)

A Nd:YAG laser (Fotona) was used to irradiate the ceramic surfaces. The optical fiber of the laser (320 μm in diameter) was placed perpendicular to the surface of the ceramic specimen at a distance of 1 mm; during the irradiation process, the entire specimen surface was kept under water and cooled with air using an adjustable air and water spray. The laser parameters used were as follows: pulse energy: 100 mJ; pulse rate: 20 Hz; power setting: 2 W; energy density: 141.54 J/cm^2; pulse duration: 150 μs.

Group FS (femtosecond laser)

A system based on a titanium:sapphire oscillator (Millenia, Spectra Physics, Santa Clara, CA), which produces 90 fs, 750 mW pulses at a wavelength of 810 nm. A pulse rate of 1 kHz was used. The femtosecond laser beam was delivered to the polished surfaces of the specimens using a laser marker system, which has a lens adjustable along the x and y axes. A spiral shape was designed using the laser marker system software and then etched into the polished surface.

A silane-coupling agent (Clearfil Ceramic Primer; Kuraray Medical Inc., Okayama, Japan) was applied with a brush on the surface of each surface-treated specimen. The specimens were then dried in air using oil-free compressed air. To apply the resin, the treated ceramic specimens were placed in a silicone mold. The resin cement (Clearfil Esthetic Cement, Kuraray Medical Inc.) was mixed as per the manufacturer's instructions and injected into Teflon tubes 4 mm in diameter and 3 mm thick. The bonding process was also performed as stipulated by the manufacturers. Once the cementation process was complete, a constant load of 750 g was applied using a universal test machine (TSTM 02500; Elista Ltd Sti, Istanbul, Turkey), to standardize the pressure exerted on the specimens and the thickness of the cement layer. Any excess cement was removed before it could harden. The resin cement was photopolymerized for 40 seconds from the top of the bonding surface using a light-emitting diode unit (Bluephase G2; Ivoclar-Vivadent), which emitted 380 to 515 nm radiation at 900 mW/cm^2; these values were measured with a radiometer (Bluephase Meter; Ivoclar-Vivadent). An oxygen-inhibiting gel (Oxyguard II; Kuraray Medical Inc.) was applied on the exposed surfaces. Having removed the Teflon molds, the composite resin cylinders obtained were light cured for 40 seconds on each side. The bonded specimens were stored in distilled water at 37°C for 24 hours and subsequently thermocycled (Thermal Cycler Tester; DentalTeknik, Konya, Turkey) for 1000 cycles between 5°C and 55°C; the dwell and transfer times were 30 seconds and 10 seconds, respectively. Shear bond strengths were measured using a universal testing machine (TSTM 02500) at a 0.5 mm/min crosshead speed via a knife-edge rod. The failure loads were converted into MPa equivalents. The failure modes were analyzed under a stereomicroscope (Olympus SZ40; Olympus Optical Co., Tokyo, Japan) at 22× magnification. The failures were classified either as an adhesive failure between the resin cement and the ceramic or as one of two types of mixed failures, namely cohesive failure in the resin cement and adhesive failure at the core/veneer interface, or cohesive failure in the veneering ceramic and adhesive failure at the core/veneer interface.

AFM and SEM observations

Next, 28 surface-treated EX and ICZ specimens (one specimen for each group) were examined by AFM (NTEGRA Solaris; NTMDT, Moscow, Russia) and SEM (Evo LS10; Carl Zeiss, Oberkochen, Germany). For the AFM, a gold-doped silicon tip (40 μm; 0.01 to 0.025 Ω·cm) was used in noncontact mode. Changes in the vertical position of the tip were registered as bright and dark regions, yielding the surface topography of the specimens. The tip was kept in tapping mode at a constant oscillation amplitude (set point amplitude). Digital images of 25 μm × 25 μm were acquired at a low scanning frequency (1 Hz) for each specimen surface.

For the SEM analysis, the ceramic specimens were first sputter coated with gold-palladium particles (Cressington Sputter Coater 108Auto, Cressington MTM-20; Elektronen-Optik-Service, Dortmund, Germany) for 15 seconds to obtain a 90 Å thick layer. The surfaces were then observed at 1000 to 10,000× magnification.

Table 1 Means and standard deviations of the SBS values (MPa)

Surface treatment	IPS e.max	In Ceram zirconia
Control	7.61 ± 3.69 cdA	7.62 ± 4.26 cA
HF acid	26.07 ± 5.84 aA	16.72 ± 3.39 bB
Sandblasting	9.80 ± 3.65 bcA	19.94 ± 5.04 bB
ER:YAG laser	4.92 ± 2.53 dA	9.47 ± 3.47 cA
ND:YAG laser	5.30 ± 2.13 dA	9.08 ± 2.97 cA
Cojet sandblasting	29.10 ± 4.01 aA	28.08 ± 5.30 aA
Femtosecond laser	12.66 ± 1.55 bA	15.12 ± 2.67 bA

[a]Different uppercase letters in the rows and lowercase letters in the columns indicate statistically significant differences ($p < 0.05$).

Statistical analysis

Two-way ANOVA was used to evaluate the effects of the ceramic type and the surface treatments on the SBS. A pairwise multiple-comparison Tukey's test was used to identify significant differences between specimens with different surface treatments, within each ceramic group and between the different ceramics within each surface treatment group. All the tests were performed at a significance level of 0.05.

Results

The two-way ANOVA tests revealed that the SBS was affected both by the surface treatment and the type of ceramic used ($p < 0.05$). The SBS values, standard deviations, the statistical groupings identified using Tukey's honest significance test, and the box-plot diagrams of the two ceramic groups for each surface treatment are shown in Table 1 and Figure 1. The SBS values for the ICZ-CJ, EX-CJ, and EX-HF specimens were significantly higher than those of the other groups ($p < 0.05$). Furthermore, the SBS's of the EX-FS and ICZ-FS specimens were statistically higher than those of the EX-ER, EX-ND, ICZ-ND, and ICZ-ER.

Representative AFM images of specimens of the two ceramic groups subjected to different surface treatments are shown in Figures 2 and 3. The highest features are observed for the EX-HF surface (Fig 2a). For the SB- and CJ-treated specimens of both ceramic types, a moderately high number of irregularities are visible, such as peaks and valleys, which are favorable for adhesion (Figs 2b,e and 3b,e). In contrast, no surface texturing can be seen for the ER- and ND-treated specimens

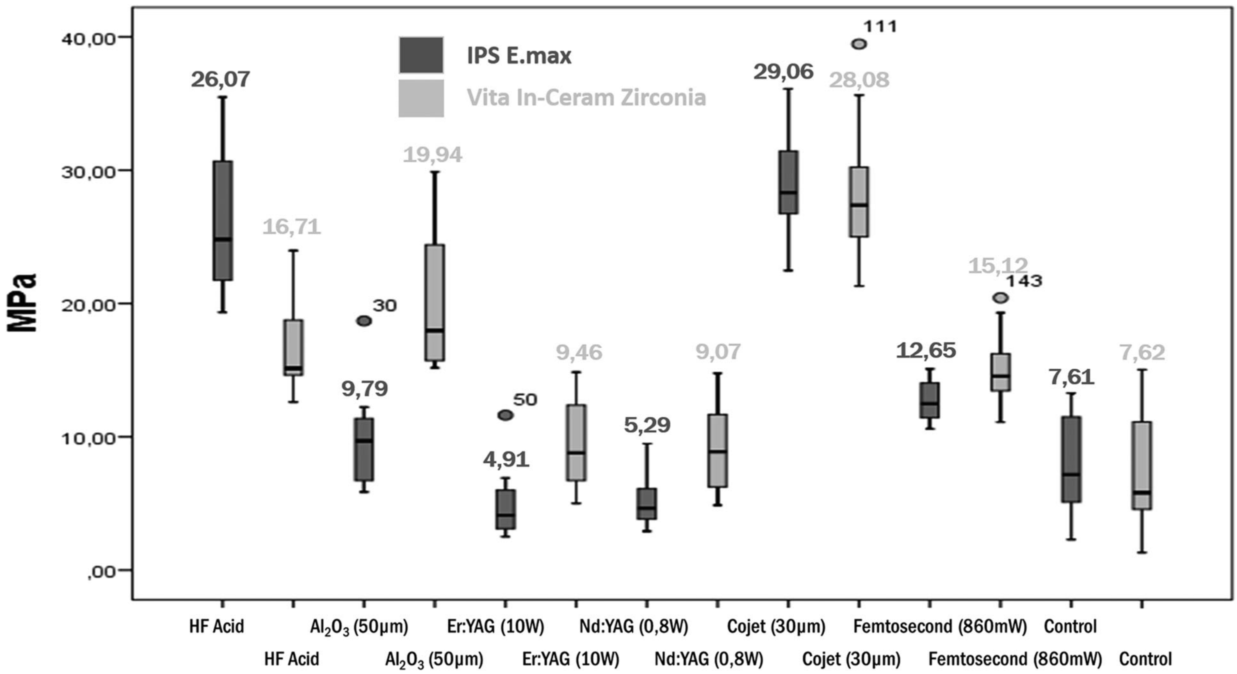

Figure 1 Box-plot diagrams of the shear bond strengths measured for IPS e.max and Vita In-Ceram zirconia specimens subjected to different surface treatments. The median is represented by a horizontal line within each box. The upper and lower strokes represent the minimum and maximum values, respectively. The symbol (O) denotes outliers.

(Figs 2c,d and 3c,d). The surface of EX-FS and ICZ-FS specimens could not be recorded due to deep surface topography.

The SEM images of the SB-, CJ-, and HF-treated surfaces reveal important information regarding the surface porosity of the two ceramic materials. The surface of the EX-CJ specimens seems to hold a greater number of silica particles than those of the ICZ-CJ (Figs 4 and 5). The SB- and CJ-treated surfaces are similarly porous, whereas those of the specimens subjected to ER or ND treatments are smoother. The surfaces of the EX-FS specimens show a few cracks, while mesh-like microporous structures are visible for the ICZ specimens.

Adhesive failure predominated for the EX and ICZ specimens subjected to SB (91%), ER (100%), and ND (100%) treatments. Those subjected to FS, HF, and CJ treatments exhibited mixed-mode failure, that is, cohesive failure in the veneering ceramic and adhesive failure at the core/veneer interface for the EX and ICZ specimens. The failure of the ICZ-CJ specimens also occurred in mixed mode, with cohesive failure in the resin cement and adhesive failure at the core/veneer interface (Table 2). None of the ICZ specimens exhibited cohesive fracture in the core material.

Discussion

The surface treatments of the ICZ and EX specimens had a significant effect on their bonding with resin cement; the null hypothesis (no effect) is therefore rejected. Researchers have found that sandblasting and the use of a monomer-phosphate-based resin cement results in durable bonds in the case of yttria-partially stabilized zirconia ceramics.[15] The surface characteristics of porcelain have a major influence on the quality of the contact between the adhesive and the solid ceramic surface. The adhesive wets and spreads over the surface, penetrating into the pits formed by roughening or chemical etching. Hydrofluoric acid treatment removes the glass matrix and the second crystalline phase, while silane-coupling agents increase the wettability of the surface, forming structural layers that promote bonding.[30] The type of porcelain used (leucite, lithia disilicate glass, glass infiltrated, or densely sintered aluminum oxide and zirconium oxide) affects the composition and physical properties of the ceramic and determines the type of adhesion layer formed on the surface. Indeed, in the present study, the AFM images show that the surfaces of the EX-HF specimens (Fig 2a) were rougher than those of ICZ-HF (Fig 3a), as expected from the different compositions and microstructure of the two glass-ceramics.

In the present study, some of the ICZ and EX specimens were irradiated with an Er:YAG laser to enhance the adhesion of resin cement to their surface. Er:YAG laser treatments smooth surfaces by removing particles through microexplosions and by ablation through vaporization. Local temperature changes in ceramics create internal tensions that can be damaging,[31] as observed here for the ICZ specimens, the variations possibly inducing phase transformations.[32] Poosti et al[16] determined the SBS of brackets attached to porcelain following treatment with Nd:YAG (0.8 W), Er:YAG (2 W), and Er:YAG (3 W) lasers, obtaining mean values of 6.9 ± 2.7, 2.3 ± 1.1, and 3.7 ± 2.3 MPa, respectively. In keeping with the former study, the

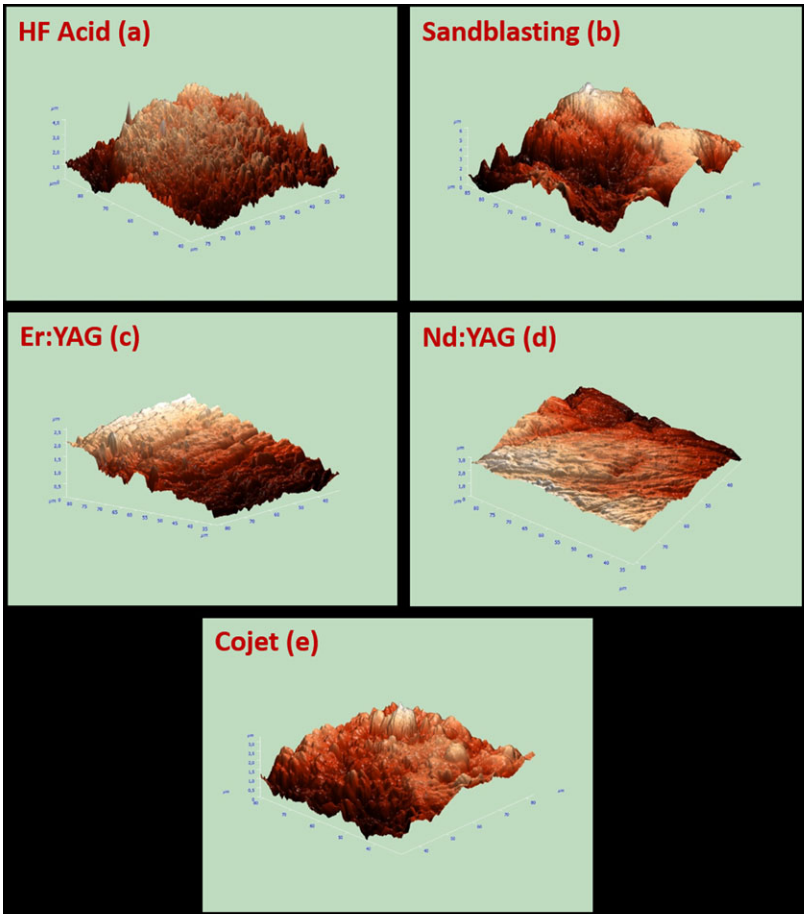

Figure 2 Atomic force micrographs of IPS e.max specimens subjected to different surface treatments.

Table 2 Types of failures observed

	IPS e.max			In Ceram zirconia		
Surface treatment	Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed
Control	12	-	-	12	-	-
HF acid	7	-	5	10	-	2
Sandblasting	11	-	1	9	-	3
ER:YAG laser	12	-	-	12	-	-
ND:YAG laser	12	-	-	12	-	-
Cojet sandblasting	5	-	7	6	-	6
Femtosecond laser	10	-	2	10	-	2

mean SBS's measured here ranged from 4.91 to 9.46 MPa. Thus, the Er:YAG and Nd:YAG laser treatments did not increase the SBS of these specimens; in other words, these treatments do not strengthen porcelain bonding. This conclusion is supported by the SEM and AFM images of the EX-ER, EX-ND, ICZ-ER, and ICZ-ND specimens, whose surface texture was not significantly improved.

The tribochemical silica sandblasting and silanization of zirconia surfaces is an "active" method, in contrast to conventional alumina powder blasting, and is one of the most effective mechanical pretreatments for zirconia surfaces.[14] However, sandblasting has been reported to produce microcracks, which lower the resistance of zirconia against low-temperature degradation and thus decrease its strength and longevity.[14,33] Zirconia surfaces should preferably be sandblasted at low pressures (1 to 2 bar) using powders with particles smaller than 50 μm.[34] Atsu et al[35] reported that the use of a silica coating and the application of a methacryloyloxydecyl dihydrogen phosphate (MDP)-containing bonding/silane-coupling agent mixture increased the SBS of a zirconia (Cercon) ceramic and a

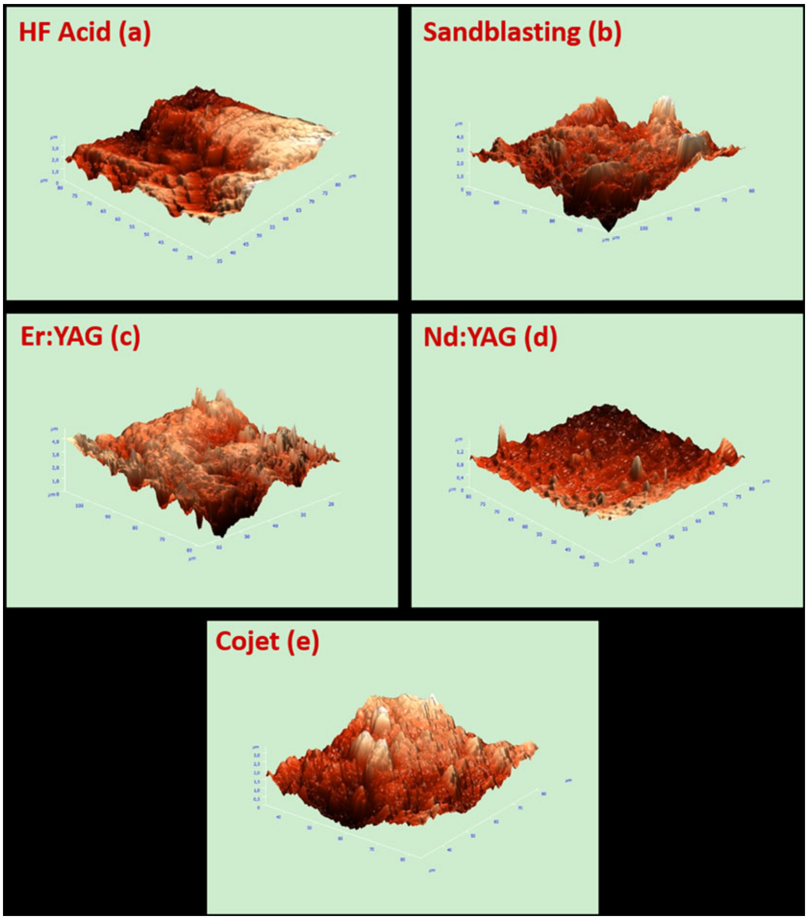

Figure 3 Atomic force micrographs of Vita In-Ceram zirconia specimens subjected to different surface treatments.

resin-luting cement. Elsewhere, Saker et al[29] stated that the tensile bond strength of zirconia ceramics treated with a chairside silica coating and an alloy primer containing MDP was higher than that of ceramics subjected to a silica coating and subsequent silanization. These studies suggest that MDP acts as a coupling agent and plays an important role in the bonding process. In the present study, the ceramic primer Clearfil was applied after tribochemical silica coating, increasing the SBS of both the ICZ (28.08 MPa) and EX specimens (29.06 MPa). Glass-infiltrated alumina-zirconia ceramics such as ICZ have high crystal contents (aluminum oxide ~67 wt%, zirconium oxide ~13 wt%, and vitreous phases and lanthanum aluminum silicate ~20 wt%), making surface conditioning difficult.[36] Bottino et al[15] suggested that ICZ is better suited for silica coatings than are ceramics that do not contain vitreous phases, such as yttria-partially stabilized zirconia or densely sintered alumina ceramics. The SBS (26.8 MPa) reported by these authors for ICZ and resin cement is similar to the value obtained in the present study (28.08 MPa).

Ultrashort-pulse laser systems are not suitable for direct clinical use; however, many studies have reported on the bond strength of dental tissues subjected to treatments using a UPLS.[26,37-39] Lorenzo et al[26] reported stronger bonding of brackets to enamel treated with a UPLS rather than other laser systems. The ablation from UPLS's is induced by a plasma, which imparts less thermal damage than do the longer pulses of ER:YAG lasers. The bonding of resin cement to porcelain treated with a UPLS has seldom been studied. A large number of parameters determine the suitability of modern UPLS's (with scanning systems) for dental applications. Nevertheless, the ablation rates afforded by these systems make them promising for dental applications.[37] Pedrazzi et al[40] evaluated the SBS of repairs in porcelain conditioned with a UPLS (760 mW and 900 mW) for 10 seconds or with 10% HF acid for 2 minutes. The UPLS significantly altered the irradiated area. The SBS values measured for repairs in feldspathic porcelain conditioned with a UPLS at 760 mW and 900 mW, and with HF acid were 11.25, 14.02, and 12.74 MPa, respectively. Here, the surfaces of the EX and ICZ ceramics were conditioned with a 860 mW UPLS, resulting in desirable SBS's (12.65 and 15.12 MPa, respectively). Note that these samples could not be observed using AFM because the dimensions of the peaks and valleys were greater than the diameter of the gold-doped silicon tip (40 μm).

Shear bond tests were used to evaluate the resin-cement-to-ceramic bond strength. The fracture types observed were consistent with the corresponding SBS's. While mixed-mode

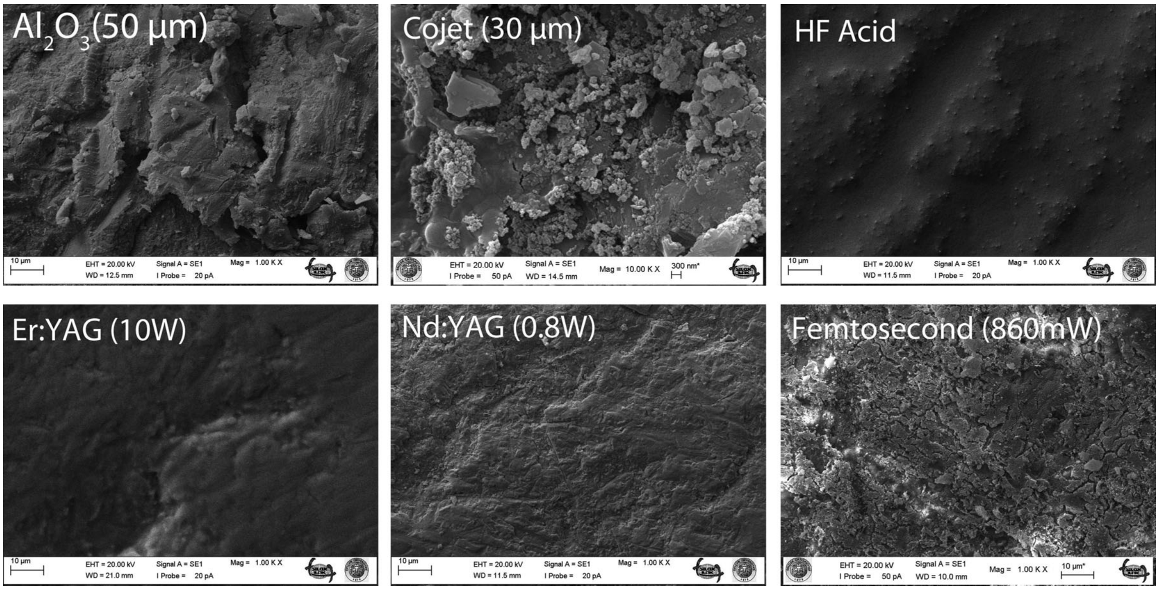

Figure 4 Scanning electron micrographs of IPS e.max specimens subjected to different surface treatments.

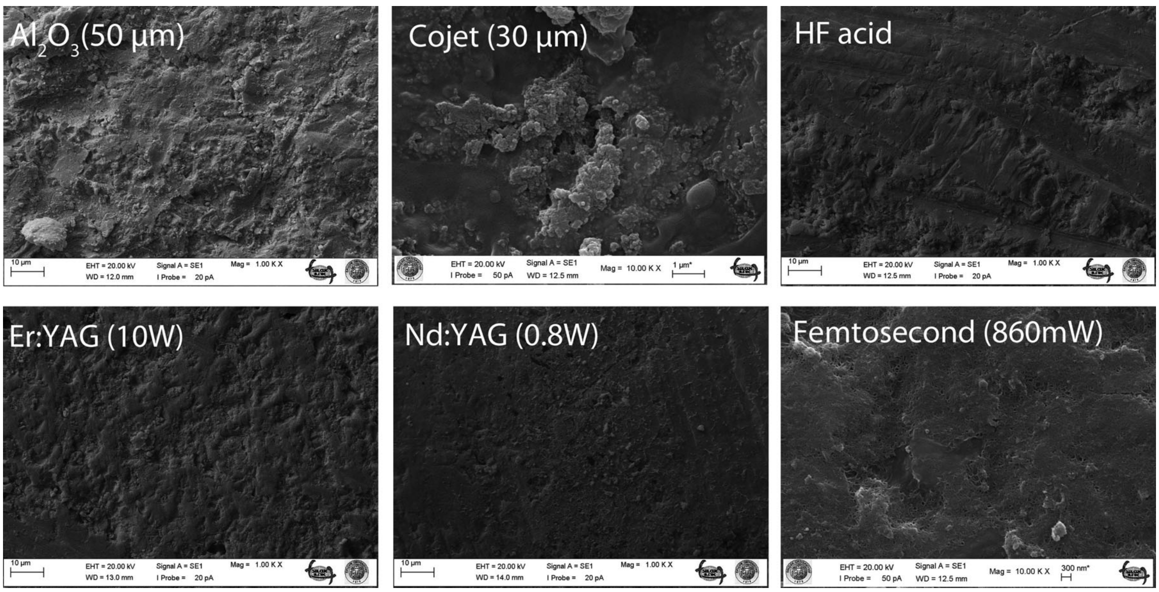

Figure 5 Scanning electron micrographs of Vita In-Ceram zirconia specimens subjected to different surface treatments.

failures were observed for the specimens with significantly higher SBS values, those with significantly lower values failed adhesively. No cohesive failures were observed either in the ceramic or on the composite surface. Although a few researchers have reported that thermocycling and long-term water storage do not affect the bonding effectiveness of zirconia, we subjected the ICZ and EX specimens to 1000 thermocycles to assess the effect on bond durability. Further similar studies are needed to clarify the effects of several other parameters (namely the laser frequency, resin cement type, and composition of the ceramic primer) on the long-term durability of the bonds between resin cements and glass-infiltrated zirconia as well as those between resin cements and lithium-disilicate-based glass ceramics.

Conclusions

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

1. Silanization after silica coating improves the bond strengths to both EX and ICZ surfaces, while HF etching is similarly favorable only for EX surfaces.
2. Treatment with a UPLS seems to be a suitable conditioning method to strengthen the bonding of resin cements to ceramic surfaces. Further investigations are nonetheless required, notably into the potential effects of various instrument parameters.

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