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Laboratory Research

Surface Roughness of Ceramic-Resin Composites After Femtosecond Laser Irradiation, Sandblasting or Acid Etching and Their Bond Strength With and Without Silanization to a Resin Cement

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Clinical Relevance

The femtosecond laser irradiation and silanization seem to be a promising treatment for improving the bond strength of resin cement to Vita Enamic and Lava Ultimate. It is expected that dental configurations of ultra short pulse lasers such as femtosecond lasers will reduce the drawbacks of available dental lasers used for roughening restorative materials.

SUMMARY

Objectives: The aim of this study was to investigate the effects of femtosecond laser irradiation, sandblasting, or acid etching treatments on the surface roughness of ceramic-resin composites and also shear bond strength (SBS) with and without silanization to a resin cement.

Methods: Samples of Vita Enamic (VE; Vita Zahnfabrik, Bad Säckingen, Germany) and Lava Ultimate (LU; 3M ESPE, Seefeld, Germany) were classified into control (no treatment), sandblasting, hydrofluoric acid, and femtosecond laser groups (n=30). Surface roughness was determined using two-dimensional contact profilometry. Surface topography was evaluated using a three-dimensional contact profilometer and a scanning electron microscope. Then groups were divided into two subgroups with similar surface roughness values, including control (C), control + silane (C-S), sandblasting (SB), sandblasting + silane (SB-S), hydrofluoric acid (HF), hydrofluoric acid + silane (HF-S), femtosecond laser (FS), and femtosecond laser + silane (FS-S) groups (n=15). Panavia F 2.0 resin cement was applied

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to the sample surfaces using an SDI SBS rig (SDI Limited, Bayswater, Australia). The SBS test was performed after water storage (24 h, 37°C) and thermocycles (2000 cycles, 5°C to 55°C), and failure modes were evaluated.

Results: The highest surface roughness was observed in the FS group, and the highest SBS was observed in the FS-S group for both VE and LU (p<0.001). Silanization improved the SBS of VE significantly (p<0.001) in all surface treatments but did not improve that of LU except in the FS group (p=0.004). There was a significantly moderate negative correlation in the VE/SB group (p=0.012) and a moderate positive correlation in the VE/HF group (p=0.049).

Conclusions: Femtosecond laser irradiation was found to be more effective than sandblasting or acid etching in increasing the surface roughness, and it was also the most effective surface treatment with silanization on the SBS of a resin cement to the ceramic-resin composites.

INTRODUCTION

Ceramics and composite resins are leading restorative materials that can be used with computer-aided design/computer-aided manufacturing (CAD/CAM) systems. Despite their superior mechanical and esthetic characteristics, ceramics have disadvantages, such as antagonist tooth wear and brittleness. On the other hand, composite materials do not have these disadvantages but cause polymer shrinkage and possess relatively weak mechanical properties.[1] Manufacturers have been improving new concept restorative materials combining the advantageous properties of ceramics and composites.[2] Thus, these materials, which could show similar mechanical properties to dentin or enamel, have contributed to the manufacturing of biomimetic materials.[3,4]

Lava Ultimate (LU) is a nanoceramic resin containing nanoceramic particles embedded in a highly cross-linked resin matrix. LU contains (by weight) 80% nanoceramic particles and 20% resin matrix,[5] and its composition was based on the Filtek Supreme Ultra (3M ESPE, Seefeld, Germany) resin-based composite.[6] It is different from resin-based composites in that it uses a high-temperature polymerization process for its manufacturing,[7] presenting more advanced physical, mechanical, and optical properties than the conventional manufacturing process of resin-based composites.[8-11] Vita Enamic (VE) is a hybrid material with a dual-network structure manufactured by infiltrating polymer into a porous feldspar ceramic enriched with aluminum oxide. VE is (by weight) 86% ceramic and 14% polymer.[12] Both these materials combine the positive characteristics of ceramic and composite materials because of their dual-network structure, and they are classified as ceramic-resin composites (CRC).[13,14]

Micromechanical retention, stability, and wettability are indispensable to facilitating a durable and reliable bond between the adhesive cement and restorative material.[15,16] Surface treatments are performed to enhance surface energy between the cement and the restorative material by increasing the surface roughness. Hydrofluoric acid etching selectively removes the glass matrix within the ceramic base and exposes crystal particles, thereby creating microporosities on the surface.[17] Sandblasting with aluminum oxide (Al_2O_3) both cleans the material surface and increases the surface area.[15] However, long sandblasting times can lead to cracks and volume loss.[18] Laser irradiation increases surface roughness and mechanical retention by melting the material's surface.[19] Silanization facilitates chemical bonding of two different organic and inorganic materials on ceramic surfaces.[20] Sandblasting and/or acid etching are commonly used methods to enhance micromechanical bonding, and silanization is a commonly used method to enhance chemical bonding.[21] These methods can be combined to enhance both micromechanical and chemical bonding.[18,22]

Studies using various laser irradiation applications to modify dental ceramics[19,23] and indirect composites[24,25] have reported promising results; however, these studies have some limitations. Yucel and others[26] and Ersu and others[27] observed crack formation in ceramics associated with thermal damage using Nd:YAG laser and CO_2 laser, respectively. However, Moezizadeh and others[28] reported that Er,Cr:YSGG laser may reduce bonding by increasing thermal destruction in indirect composites. Such disadvantages of long-pulsed lasers brought ultrashort-pulsed femtosecond (FS) lasers to the forefront. As FS lasers have a short interaction time, they limit temperature distribution in the dental tissue and reduce energy loss on the surface. This feature not only provides a much higher surface energy than other laser systems but also causes minimal thermal and mechanical damage.[29] FS lasers can create repeated microvoids in different

Table 1: *Brand Names, Manufacturers, Compositions, and Lot Numbers of Materials Used in the Present Study*

Material	Brand Name	Manufacturer	Composition	Lot Numbers
Hybrid ceramic	Vita Enamic	Vita Zahnfabrik (Bad Säckingen, Germany)	86 wt% feldspar ceramic, 14 wt% polymer	51100
Resin nanoceramic	Lava Ultimate	3M ESPE (Seefeld, Germany)	80 wt% nanoceramic, 20 wt% resin	N606702
Dual-cure resin cement	Panavia F 2.0	Kuraray Medical Inc (Okayama, Japan)	Paste A: MDP, DMA, silanated silica, camphorquinone, catalysts, photoinitiator Paste B: DMA, silanated barium glass, sodium fluoride, catalysts, accelerator	990076 980014
Silane	Clearfil Ceramic Primer	Kuraray Medical Inc	3-MPS, MDP, ethanol	940008
Hydrofluoric acid gel	Vita Ceramics Etch	Vita Zahnfabrik	5% hydrofluoric acid	37160
Al_2O_3 powder	Korox 50	BEGO (Bremen, Germany)	99.6% Al_2O_3 (50μm)	14 361781112

Abbreviations: MDP, 10-methacryloxydecyldihydrogenphosphate; DMA, dimethacrylate; MPS, methacryloxypropyltrimethoxysilane; Al_2O_3, aluminum oxide.

shapes and depths by using software. FS laser irradiation has become more widely used because this procedure provides sensitive and controlled roughening without altering the surface characteristics of the material.[29-32]

To date, only a few studies have examined surface treatments for CRC materials and their bond strength with resin cement. The aim of this study is to evaluate the effects of various surface treatments on the surface roughness and bond strength of resin cement to CRC CAD/CAM blocks. The null hypotheses (H_0) of this study are as follows: 1) femtosecond laser irradiation would not enhance surface roughness more than aluminum oxide sandblasting and hydrofluoric acid etching, 2) femtosecond laser irradiation would not enhance the bond strength to resin cement more than aluminum oxide sandblasting and hydrofluoric acid etching, and 3) silanization would not enhance the bond strength to resin cement.

METHODS AND MATERIALS

Experimental Design

All the materials used in the present study are listed in Table 1. Two hundred and forty-eight samples (5×5×2 mm) were prepared from VE and LU CAD/CAM blocks using a low-speed cutting saw (Micracut 201, Metkon, Bursa, Turkey). The samples were polished under water using 240-, 800-, and 1200-grit silicon carbide abrasive papers (Minitech 233, Presi, Grenoble, France) to obtain smooth and standard sample surfaces. After polishing, all samples were cleaned by keeping them for five minutes in an ultrasonic cleaner (Eurosonic Energy, Euronda, Vicenza, Italy) containing distilled water. The VE and LU material groups (n=124) were first divided into the following four groups (n=31): control, sandblasting, hydrofluoric acid, and femtosecond laser groups. The relevant surface treatment methods were used for each group. After measuring surface roughness, the sample with a roughness value closest to the average value was selected from each group for surface analysis. Then each group (n=30) was divided into two subgroups with similar average roughness values. At this stage, silane was applied to one of the groups, whereas no additional procedure was performed in the other groups. Thus, the following eight surface treatment groups were formed: control (C), silane (C-S), sandblasting (SB), sandblasting + silane (SB-S), hydrofluoric acid (HF), hydrofluoric acid + silane (HF-S), femtosecond laser (FS), and femtosecond laser + silane (FS-S).

- Group C: No surface treatment was applied.
- Group C-S: Silane coupling agent (Clearfil Ceramic Primer, Kuraray Medical Inc, Okayama, Japan) was applied to the surface for five minutes.
- Group SB: The sample surfaces were sandblasted with 50 μm of Al_2O_3 at 2 bar for five seconds at a distance of 10 mm (BEGO Easyblast, Bremen, Germany). A specifically designed holder was used to standardize the distance between the sample surface and the nozzle. After sandblasting, the samples were again ultrasonically cleaned in distilled water for five minutes.

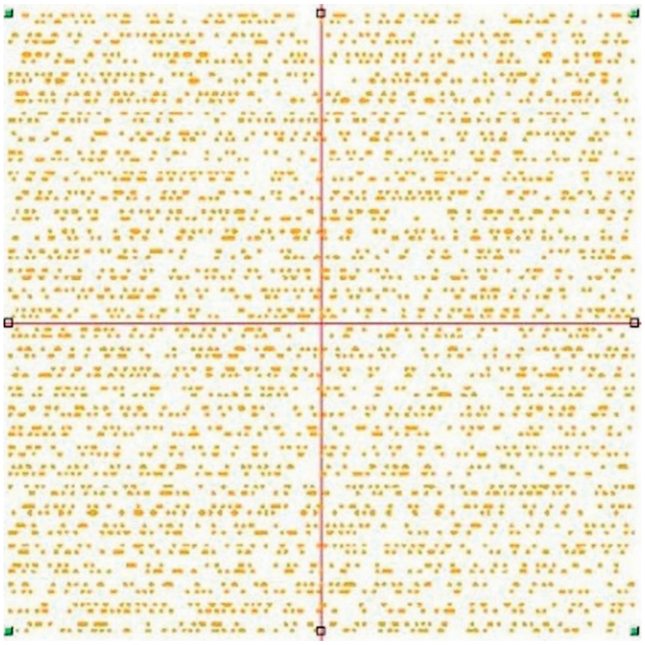


Figure 1. *The standard roughening pattern used by femtosecond laser.*

- Group SB-S: Silane was applied after sandblasting.
- Group HF: The sample surfaces were etched with 5% HF (Vita Ceramic Etch, Vita, Bad Säckingen, Germany) for 60 seconds, rinsed with water for 20 seconds, and then dried using oil-free airflow.
- Group HF-S: Silane was applied after etching with HF acid.
- Group FS: FS laser (Integra-C-3.5, Quantronix, New York, NY, USA) was used with an output power of 300 mW per pulse, an 800-nm wavelength, a 90-femtosecond pulse duration, a 1-kHz repetition frequency, and an energy density of 10.62 J/cm^2. Then laser pulses with a diameter of approximately 60 μm were focused on the sample surfaces; this was done by passing the beams through an f-theta lens (Q-Mark, Quantronix) with a focal length of 11 cm. The samples were roughened using a standard roughening pattern (Figure 1).
- Group FS-S: Silane was applied after irradiating with FS laser.

Surface Roughness Analysis

The average roughness values (R_a) of the samples were determined using a two-dimensional contact profilometer (Perthometer M2, Mahr, Göttingen, Germany) with a measurement length of 1.75 mm and a cutoff value of 0.25 mm. The roughness value (μm) was calculated by taking the average of the values obtained from five different regions of the samples. The profilometer was calibrated using a reference block after every 10 measurements.

Surface Topography Analysis

The surface topography of the samples was evaluated using a three-dimensional (3D) contact profilometer (Nanomap500LS, AEP Technology, Saratoga, CA, USA). The vertical dynamic range was set to 500 μm, the scan range was set to 2 × 2 mm, and the stylus loading force was set to 12 mg. The obtained images were interpreted using a color scale and graphics. Each color in the images represents a different value; the negative values represent pits, and the positive values represent peaks.

Scanning Electron Microscopic Analysis

Two-dimensional surface morphology of the samples was analyzed using a scanning electron microscope (SEM; JSM-5600 LV, JEOL, Tokyo, Japan). Each sample was coated with gold-palladium, and the roughness images of the samples were recorded 1000× magnification except FS samples (250×).

Bonding Procedure

An SDI SBS rig (SDI Limited, Bayswater, Australia) was used to prevent overflow of the cement from the interface and to provide a standard bonding area. Panavia F 2.0 resin cement (Kuraray, Osaka, Japan), which was mixed in accordance with the manufacturer's instructions, was layered in four steps into the rigs that were fixed onto the sample surface. Each layer was polymerized using Elipar S10 (3M ESPE, Seefeld, Germany) with a light intensity of 1200 mW/cm^2 for 20 seconds. After the polymerization of the final layer, oxygen inhibition gel was applied onto the cement layer for 10 minutes. The samples that were stored in 37°C distilled water were thermocycled for 2000 cycles in water between 5°C and 55°C with a dwell time of 30 seconds and transfer time of five seconds.

Shear Bond Strength Test

The shear bond strength (SBS) of a CRC to resin cement was tested with a knife-edge chisel by using a universal testing machine (Shimadzu AGS-X, Shimadzu Corp, Tokyo, Japan) at a crosshead speed of 0.5 mm/min until fracture. The maximum stress of the CRC-resin cement was calculated by the following formula: Stress (MPa) = Load (N)/Area (mm^2). The standard bond area (9.62 mm^2) was obtained using the SDI rig stainless-steel mold, which has an internal diameter of 3.5 mm.

Table 2: *Mean (Standard Deviation) of the Average Surface Roughness (Ra/μm) of Ceramic-Resin Composites According to Different Surface Treatments*[a]

Surface Treatments	Vita Enamic	Lava Ultimate
C	0.07 (0.02) Aa	0.05 (0.02) Ba
SB	1.77 (0.15) Ab	2.00 (0.19) Bb
HF	0.65 (0.05) Ac	0.15 (0.02) Bc
FS	6.88 (0.71) Ad	6.65 (0.83) Ad

Abbreviations: C, control; SB, sandblasting; HF, hydrofluoric acid; FS, femtosecond laser.
[a] *Same uppercase letters in each row indicate no significant difference, while same lowercase letters in each column indicate no significant difference according to the* post hoc *Tamhane test* ($p>0.05$).

Failure Mode Analysis

Failed surfaces were examined using a stereomicroscope (NZ.1902-P, Euromex, Arnhem, Netherlands) at 40× magnification. Failure modes were classified as adhesive (between cement and ceramic), cohesive (within CRC), and mixed (simultaneous adhesive and cohesive failure).

Statistical Analysis

The data were analyzed using the statistical software (IBM SPSS Statistics 20, SPSS Inc and IBM Corp, New York, NY, USA) at a significance level of 0.05. The normality of the data was determined using the Kolmogorov-Smirnov test (normal, $p>0.05$). The surface roughness values were analyzed using one-way analysis of variance and the *post hoc* Tamhane test. The SBS values were analyzed using the Kruskal-Wallis test and the Mann-Whitney U-test. The relationship between the surface roughness and SBS values was analyzed using Spearman's correlation test.

RESULTS

Surface Roughness Analysis

There was a significant difference between subgroups of VE and subgroups of LU (both $p<0.001$). The FS group showed the highest values for VE and LU (R_a, 6.88±0.71 μm; R_a, 6.65±0.83 μm, respectively), followed by the SB, HF, and C groups in descending order. However, the VE/C (R_a, 0.07±0.02 μm) and VE/HF (R_a, 0.65±0.05 μm) groups showed significantly higher roughness values than the LU/C (R_a, 0.05±0.02 μm) and LU/HF (R_a, 0.15±0.02 μm) groups, and the LU/SB (R_a, 2.00±0.19 μm) group showed significantly higher values than the VE/SB (R_a, 1.77±0.15 μm) group (all $p<0.001$) The surface roughness results are displayed in Table 2.

Surface Topography Analysis

While the LU/C group showed a higher roughness value, the VE/C and LU/C groups showed a smooth surface structure (Figure 2A,E). While the LU/SB group showed higher roughness, the VE/SB and LU/SB groups had rough surfaces with irregular peaks and valleys (Figure 2B,F). While the VE/HF group showed a very indented surface topography, the LU/HF group showed a less indented and rougher surface topography (Figure 2C,G). The VE/FS and LU/FS groups, on the other hand, showed similar surface topographies with deep parallel pits (Figure 2D,H).

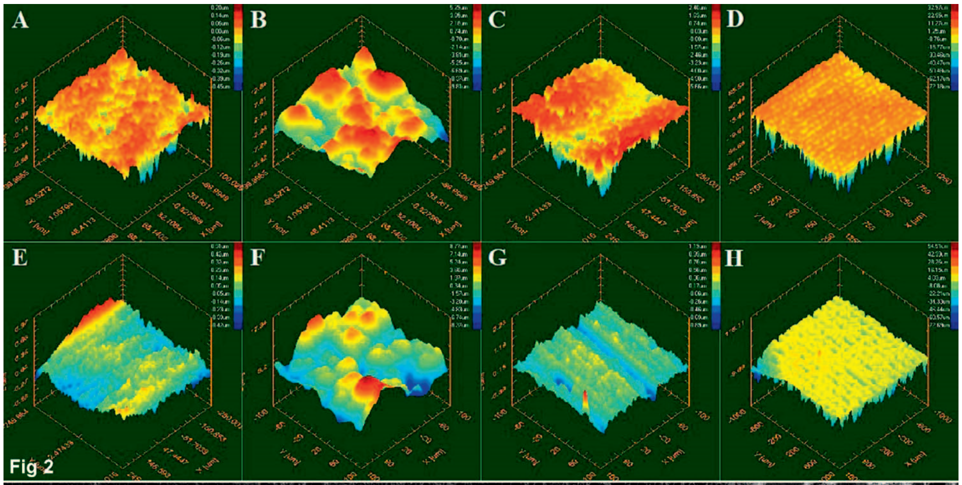
SEM Analysis

Although microstructural changes were observed in the images, no defects or microcracks were observed. A smooth surface with irregular micropores was observed in the VE/C group; the polymer phase was dark gray, and the ceramic phase was light gray (Figure 3A). In the LU/C group, on the other hand, a more smooth and homogenous surface with smaller micropores was observed (Figure 3E). Prominent peaks and pits with crevices were observed in the VE/SB group (Figure 3B). In the LU/SB group, however, there were less prominent peaks and pits (Figure 3F). In the VE/HF group, it was observed that the ceramic phase was partially resolved and there were many micropores, which is suitable for micromechanical bonding (Figure 3C). In the LU/HF group, however, shallower micropores were observed (Figure 3G). The VE/FS and LU/FS groups showed deep pits that were distributed in accordance with the standard roughening pattern, whereas finer-grained particles were observed in the LU/FS group (Figure 3D,H).

SBS Analysis

There was a significant difference between the groups in both materials (both $p<0.001$). The VE/SB-S (18.64 MPa), VE/HF-S (22.99 MPa), and VE/FS-S (25.55 MPa) groups showed significantly higher SBS than the VE/SB (10.47 MPa), VE/HF (9.86 MPa), and VE/FS (12.45 MPa) groups, respectively (all $p<0.001$). However, the VE/FS-S and VE/HF-S groups showed significantly higher SBS than the VE/SB-S group ($p=0.012$ and $p=0.031$, respectively), and the VE/SB-S group showed significantly higher SBS than the VE/SB, VE/HF, and VE/FS groups (all $p<0.001$).

The LU/FS-S (18.14 MPa) group showed significantly higher SBS than the LU/FS (11.59 MPa) group ($p=0.004$). However, the LU/FS-S group



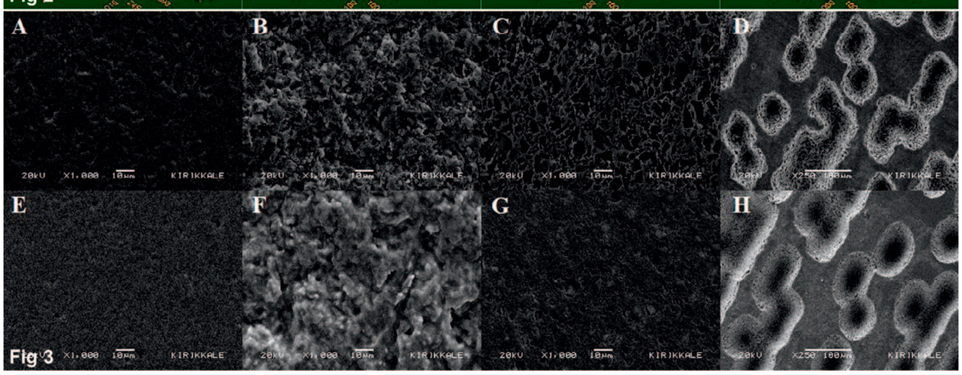


Figure 2. *3D profilometer images. (A): Vita Enamic/Control-VE/C. (B): Vita Enamic/Sandblasting-VE/SB. (C): Vita Enamic/Hydrofluoric Acid-VE/HF. (D): Vita Enamic/Femtosecond Laser-VE/FS. (E): Lava Ultimate/Control-LU/C. (F): Lava Ultimate/Sandblasting-LU/SB. (G): Lava Ultimate/Hydrofluoric Acid-LU/HF. (H): Lava Ultimate/Femtosecond Laser-LU/FS.*

Figure 3. *SEM images. (A): Vita Enamic/Control-VE/C. (B): Vita Enamic/Sandblasting-VE/SB. (C): Vita Enamic/Hydrofluoric Acid-VE/HF. (D): Vita Enamic/Femtosecond Laser-VE/FS. (E): Lava Ultimate/Control-LU/C. (F): Lava Ultimate/Sandblasting-LU/SB. (G): Lava Ultimate/Hydrofluoric Acid-LU/HF. (H): Lava Ultimate/Femtosecond Laser-LU/FS.*

showed significantly higher SBS than the LU/HF (11.33 MPa) and LU/HF-S (12.20 MPa) groups (p=0.004 and p=0.017, respectively), whereas these groups showed significantly higher SBS than the LU/SB (6.02 MPa) and LU/SB-S (6.32 MPa) groups (all p<0.001).

The VE/SB, VE/SB-S, VE/HF-S, and VE/FS-S groups showed significantly higher SBS than the LU/SB, LU/SB-S, LU/HF-S, and LU/FS-S groups (p=0.002, p<0.001, p<0.001, and p=0.005, respectively). The SBS results are displayed in Table 3.

Failure Mode Analysis

The failure modes observed in the VE groups were as follows: completely adhesive failures in the VE/C group, mostly adhesive in the VE/C-S (93%) and VE/SB (60%) groups, mostly cohesive in the VE/HF-S (60%) and VE/FS-S (53%) groups, and mostly mixed in the VE/SB-S (53%), VE/HF (53%), and VE/FS (93%) groups. The failure modes observed in the LU groups were as follows: completely adhesive in the LU/C, LU/ C-S, LU/SB-S, and LU/HF-S groups, mostly adhesive in the LU/SB (87%) and LU/HF (80%) groups, completely mixed in the LU/FS group, and mostly mixed in the LU/FS-S (87%) group. The most frequently observed failure mode for VE was mixed failure (42%), followed by adhesive (37%) and cohesive (21%) failures. The most frequently observed failure mode for LU, on the other hand, was adhesive failure (71%), followed by mixed (27%) and cohesive (2%) failures (Figure 4).

Table 3: *Median (Min-Max) of the Shear Bond Strengths (MPa) of Ceramic-Resin Composites According to Different Surface Treatments*[a]

Surface Treatments	Vita Enamic		Lava Ultimate	
	Median	Min-Max	Median	Min-Max
C	0.00 Aa	0.00-0.65	0.00 Aa	0.00-0.00
C-S	0.20 Aa	0.00-1.79	0.00 Aa	0.00-0.51
SB	10.47 Ab	3.24-18.43	6.02 Bb	2.66-10.63
SB-S	18.64 Ac	11.60-25.36	6.32 Bb	2.62-12.04
HF	9.86 Ab	4.94-18.41	11.33 Ac	7.96-17.44
HF-S	22.99 Ad	14.02-32.64	12.20 Bc	9.12-20.24
FS	12.45 Ab	3.38-19.32	11.59 Ac	5.75-18.04
FS-S	25.55 Ad	9.06-35.30	18.14 Bd	4.26-31.49

Abbreviations: C, control; C-S, silane; SB, sandblasting; SB-S, sandblasting + silane; HF, hydrofluoric acid; HF-S, hydrofluoric acid + silane; FS, femtosecond laser; FS-S, femtosecond laser + silane.

[a] *Same uppercase letters in each row indicate no significant difference, while same lowercase letters in each column indicate no significant difference according to Mann-Whitney* U*-test (*p*>0.05).*

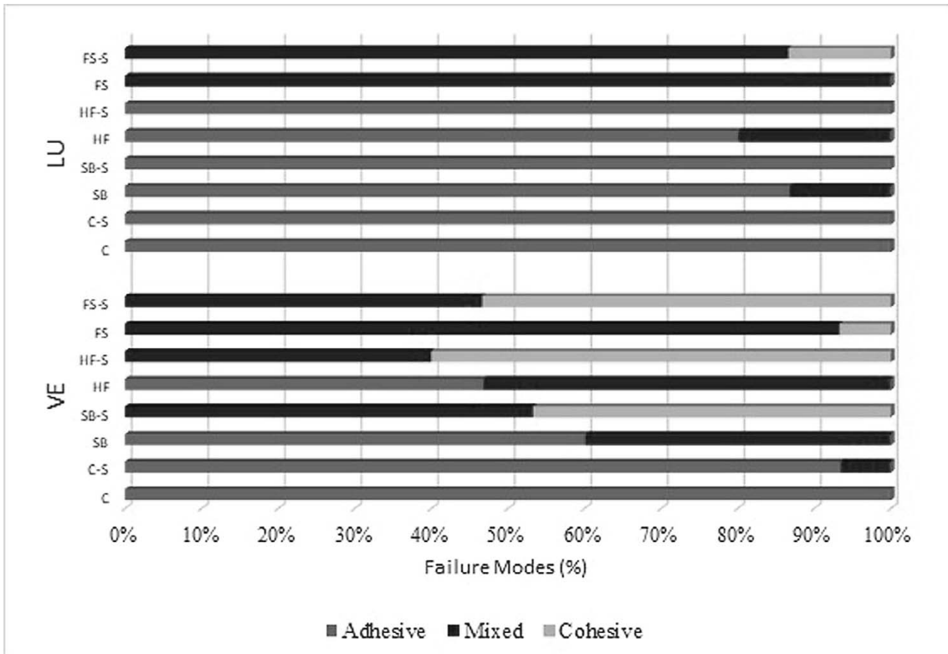


Figure 4. *Failure mode distributions according to surface treatment groups of Vita Enamic and Lava Ultimate.*

Correlation Analysis

There was a significant moderately negative correlation between surface roughness and SBS values in the VE/SB group (r=−0.63, p=0.012) and a moderately positive correlation in the VE/HF group (r=0.51, p=0.049); however, no significant correlation was observed in the other groups (p>0.05). On the other hand, prefailure was observed during the thermal cycle in the LU/C group; therefore, this group could not be evaluated in terms of correlation. The correlation results are displayed in Table 4.

DISCUSSION

The present study evaluated the effects of SB, HF, and FS treatments on the surface roughness of CRC materials and the effects of these treatments with and without silane coupling agent application on the bond strength. The first null hypothesis was rejected because FS treatment significantly enhanced surface roughness more than SB and HF treatments. The second null hypothesis was accepted for VE and partially accepted for LU because FS treatments did not enhance the SBS significantly except in the LU/SB group. Silanization increased the SBS significantly in the VE groups but did not increase it in the LU groups except the FS subgroup. Therefore, the third null hypothesis was rejected for VE and partially accepted for LU.

Cekic-Nagas and others[33] reported that resin cement and MDP-containing silane enhance the bond strength of CRC materials. In addition, MDP-containing resin cements reduce susceptibility to technical drawbacks and increase bond strength.[34] MDP-containing Panavia F 2.0 resin cement, which was used in the present study, was accepted as the gold standard by Behr and others,[35] and Kitayama and others[36] reported further improvement in the bond strength when it was combined with MDP-containing silane. Since the bond strength between ceramics and resin cement was evaluated in the present study, resin cement was applied directly to sample surfaces using an SDI SBS rig, as reported in previous studies.[26,37] Thus, the CRC cement interface was evaluated more reliably. Although the SBS test may cause cohesive failure with regard to nonuniform stress distribution and this may lead to faulty interpretations, it was preferred in the

Table 4: *Correlation Coefficients (r) and p-Values of the Relationship Between the Average Surface Roughness (Ra/μm) of the Shear Bond Strengths (MPa) of Ceramic-Resin Composites According to the Spearman Correlation Test*

Surface Treatment/ Shear Bond Strength	Vita Enamic		Lava Ultimate	
	r	*p*	*r*	*p*
C	0.27	0.329	—	—
SB	−0.63	0.012	0.23	0.405
HF	0.51	0.049	−0.39	0.152
FS	0.50	0.058	0.41	0.125

Abbreviations: C, control; SB, sandblasting; HF, hydrofluoric acid; FS, femtosecond laser.

present study because it can be easily and quickly applied with specific jigs standardizing the bonding area.[38] Similar to previous studies,[33,37,39] the SBS of the samples with prefailure during thermal cycling was considered to be 0 MPa in the present study.

Consistent with surface roughness values, the results of SEM images show that the surface texture of the VE/C group was rougher than that of the LU/C group. This result can be attributed to the fact that while VE contains microstructured silica particles, LU contains nanostructured silica and zirconia particles; moreover, it can be attributed to the differences in manufacturing processes and compositions of the materials. Although LU/SB showed higher roughness than VE/SB, LU/SB showed more irregular crater-like areas on the surface because of zirconia-silica particle networks. Consistent with its roughness values, VE/HF showed a more indented surface morphology than LU/HF. These results are consistent with those of previous studies.[40,41] Duzyol and others[42] reported that zirconia fillers and the resin matrix in the LU material were not affected by HF. On the other hand, the VE/FS and LU/FS groups showed similar surface textures, consistent with their roughness values. However, VE/FS exhibited a more porous surface morphology than LU/FS. The obtained images can be explained by the fact that LU has a resin matrix and VE a feldspathic ceramic matrix.

Because of its larger scanning area and versatility, a 3D profilometer can visualize surfaces where atomic force microscopy (AFM) proves to be ineffective.[43,44] The fact that the peak heights and pit depths observed in the FS groups are too deep and too high to be detected by AFM analysis justifies the use of 3D profilometry on surfaces with high roughness.[32,45] Akpinar and others[32] obtained peaks and pits with a range of 180 to 201 μm in zirconia ceramics using FS lasers with a 750-mW pulse power. The low heights and pits obtained in the present study can be attributed to the use of a low pulse power. Lorenzo and others[45] reported that pits of 15 to 90 μm provided higher bond strengths than pits of 120 to 180 μm. The pits of the laser groups in the present study were within the ideal range specified for bond strength.

Manufacturers recommend 5% HF and/or silane for VE[12] and SB (<50 μm Al_2O_3, <2 bar) and/or silane for LU.[5] Elsaka and others[40] reported that SB with 110 μm of Al_2O_3 or etching with 9% HF increases the bond strength and surface roughness of CRC and that silane further improves the bond strength of VE. On the other hand, Cekic-Nagas and others[33] reported that 10% HF did not increase the bond strength of CRC. In the present study, the SB treatment provided a higher surface roughness than the HF treatment for both materials; moreover, the surface roughness produced by the SB treatment was higher in the VE group than in the LU group, and the effectiveness of the SB, HF, and silane treatments was found to be consistent with the study by Elsaka.[40] The application of silane was found to be effective after HF for Vita VM7[46] and after HF and SB for IPS Empress 2.[21] Similar to the present study's finding, Elsaka[40] found the application of silane after SB and HF treatments to be effective only for VE and concluded that the differences in the results were due to different microstructural features and silica contents of the VE and LU materials. The effectiveness of silane application after SB and HF for VE can be attributed to the fact that the material has a silica-containing feldspathic matrix and that the selective removing effect of HF treatment removes the glassy phase in the ceramic and reveals more silica particles. In composite resins, the application of silane was not found to be effective after HF[47] and SB.[48] In the present study, in agreement with the above-mentioned results, the ineffectiveness of silane application after the SB or HF treatments for LU was attributed to the fact that the material has a composite matrix with resin content. The effectiveness of silane application after FS for LU can be attributed to the fact that more filler silica particles are released as a result of the removal of the resin structure by ablation. In this study, VE showed generally higher bond strength than LU. Thornton and Ruse[49] reported that VE exhibited superior mechanical properties and was less affected by storage in water than LU. Flury and others[50] reported that the SBS results for VE cemented with Panavia F 2.0 did not change even after storage for six months (37°C, 100% humidity); however, the SBS values for LU decreased. Cekic-Nagas and others[33] reported that thermal cycling reduced the bond strength of both materials and that the higher bond strength of VE was attributed to the fact that it absorbs less water because of its interpenetration phase. Thus, the hydrolytic stability of VE can be considered an important reason for the obtained results.

No studies or recommendations on CRC materials were identified in terms of FS or other laser surface irradiation. In this study, the FS treatment provided the highest roughness values, and the FS-S treatment yielded the highest SBS values for both materials. However, FS lasers have been reported to produce homogeneous roughness without causing structural changes and thermal damage on the material surface by avoiding heat transfer on the surface using ultrashort laser pulses.[30] Akpinar and others[31] reported that FS laser provides homogeneous and regular surface roughness without generating cracks on the feldspar ceramic surface and that ablation patterns can also be controlled during laser irradiating using software. Although *in vitro* studies anticipate beneficial results about the prognosis of FS lasers since they compensate for negative properties of dental lasers, FS lasers have financial and dimensional disadvantages.[51,52] These disadvantages of FS lasers do not allow chairside applications in dentistry, whereas it is currently used in medicine and research laboratories. However, FS lasers in different configurations are expected to be introduced to dentistry and to increase in dental applications with FS lasers by reducing the cost of investment and simplifying their complex structure.[52-54] The effects on monomer chemistry and microcrack propagation of FS laser beams within the restorative materials were not encountered in the dental literature, and it seems that there is insufficient evidence for these issues.

Atsu and others[22] observed adhesive failures at lower bond strengths and mixed and cohesive failures at higher bond strengths. Oyague and others[55] also stated that mixed and cohesive failures are clinically preferable, as they maintain higher bond strengths compared to adhesive failures. In the present study, higher SBS (17 to 25 MPa) mostly exhibited mixed failure and rarely exhibited cohesive failure. Moreover, VE/HF, VE/FS, and LU/FS, which had lower SBS, showed mixed failures. These results may be due to the retention pattern of related surface treatments. In this study, cohesive failures were not observed in resin cement, as it was contained in the SDI SBS rig molds. Silane application increased the cohesive failure rate by increasing the bond strength in the VE material. In a similar study, Elsaka[40] most frequently observed mixed failure in both materials. On the other hand, Cekic-Nagas and others[33] most frequently observed adhesive failure in both materials. In the present study, adhesive failures (54%) were the most frequently observed failure mode.

In the present study, the surface roughness showed significant differences in the VE/SB, VE/HF, and VE/FS groups; however, their bond strengths were not significantly different. Although the LU/SB group showed higher roughness than the LU/HF group, its SBS value was significantly lower than that of the LU/HF group. This was attributed to the nonselective abrasive effect caused by SB treatment on LU because it may remove ceramic filler particles, which can be useful for bonding on composite surfaces. Although the LU/FS group showed higher roughness than the LU/HF group, their SBS values were similar. These results confirm the results of studies suggesting that the bond strength does not increase with surface roughness.[23,27,40] Oyague and others[55] reported that the type of resin cement has a higher impact on bond strength when compared to surface treatment methods. In the present study, surfaces with a lower roughness exhibited SBS values as high as those on surfaces with a higher roughness; this suggests that MDP-containing silane and cement increase SBS values of surfaces with a low roughness.

The limitations of the present study include the inability to fully simulate the mouth conditions and the inability to measure volume loss caused by surface treatments. Further studies are required to evaluate the effects of surface treatments on fracture resistance and discoloration, the effects of surface treatments with different sandblasting and femtosecond laser irradiation parameters and cements on bond strength, and the outcomes in the long-term clinical follow-up.

CONCLUSIONS

According to the results obtained in this study, the following conclusions were drawn:

1) FS surface treatment was the most effective method for both VE and LU in terms of surface roughness and bond strength to resin cement.
2) Although there was no significant difference between the surface roughness values of VE and LU, the bond strength to resin cement for VE was higher.
3) Silanization after surface treatments significantly increased the bond strength to resin cement for VE; however, it did not increase the bond strength to resin cement for LU except in the FS.
4) FS-S, HF-S, and SB-S surface treatments are recommended to condition the surface of VE restoration, and FS-S is recommended to condition the surface of LU restoration.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the University of Kirikkale.

Conflict of Interest

The authors of this article 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.

Note

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