

Shear bond strength of resin cement to an acid etched and a laser irradiated ceramic surface

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PURPOSE. To evaluate the effects of hydrofluoric acid etching and Er,Cr:YSGG laser irradiation on the shear bond strength of resin cement to lithium disilicate ceramic. **MATERIALS AND METHODS.** Fifty-five ceramic blocks (5 mm × 5 mm × 2 mm) were fabricated and embedded in acrylic resin. Their surfaces were finished with 1000-grit silicon carbide paper. The blocks were assigned to five groups: 1) 9.5% hydrofluoric-acid etching for 60 s; 2-4), 1.5-, 2.5-, and 6-W Er,Cr:YSGG laser applications for 60 seconds, respectively; and 5) no treatment (control). One specimen from each group was examined using scanning electron microscopy. Ceramic primer (Rely X ceramic primer) and adhesive (Adper Single Bond) were applied to the ceramic surfaces, followed by resin cement to bond the composite cylinders, and light curing. Bonded specimens were stored in distilled water at 37°C for 24 hours. Shear bond strengths were determined by a universal testing machine at 1 mm/min crosshead speed. Data were analyzed using Kruskal–Wallis and Mann–Whitney U-tests ($\alpha=0.05$). **RESULTS.** Adhesion was significantly stronger in Group 2 (3.88 ± 1.94 MPa) and Group 3 (3.65 ± 1.87 MPa) than in Control group (1.95 ± 1.06 MPa), in which bonding values were lowest ($P<.01$). No significant difference was observed between Group 4 (3.59 ± 1.19 MPa) and Control group. Shear bond strength was highest in Group 1 (8.42 ± 1.86 MPa; $P<.01$). **CONCLUSION.** Er,Cr:YSGG laser irradiation at 1.5 and 2.5 W increased shear bond strengths between ceramic and resin cement compared with untreated ceramic surfaces. Irradiation at 6 W may not be an efficient ceramic surface treatment technique. [*J Adv Prosthodont 2013;5:98-103*]

KEY WORDS: Ceramic; Resin cement; Er,Cr: YSGG Laser; Shear bond strength

INTRODUCTION

Interest in the use of ceramic laminate veneers, inlays, and onlays has grown considerably in recent years due to their excellent esthetic properties.^{1,2} However, ceramic restorations are very brittle and have limited flexural strength; thus, adhesive cementation increases the risks of fracture³⁻⁶ and debonding.^{7,8} Successful treatment depends in part on

the strength of the bond between the composite resin and the ceramic restoration.⁹ In addition, the repair of ceramic restoration fractures caused by inadequate design or occlusal forces requires surface preparation.¹⁰⁻¹² The ceramic surface must be roughened to achieve successful chemical bonding and micromechanical interlocking with the resin cement.¹³⁻²⁰

Various techniques can be used to optimize bond strength at the ceramic–cement interface. Common treatment options include abrasion with a diamond rotary cutting instrument^{18,19} or aluminum oxide particles,^{21,22} as well as acid etching.^{17,23,24} Etching with a hydrofluoric (HF) acid solution can achieve proper ceramic surface texture¹³⁻¹⁷ by dissolving the glass matrix to expose the crystalline phase.^{25,26} However, this method is hazardous for both the operator and patient. The operator must be protected from skin and eye damage, and any remaining HF acid must be removed completely with an ultrasonic cleaner before cementation. Given these limitations, HF acid etching cannot be used in some cases, such as ceramic restoration

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repair;²⁷ and so more biofriendly techniques are needed. Relatively safe and easy methods of laser irradiation for surface modification have been proposed.²⁸⁻³³ Studies have examined the use of several laser types, including neodymium: yttrium aluminum garnet (Nd:YAG), erbium (Er):YAG,³⁴ erbium, chromium: yttrium, scandium, gallium, garnet (Er,Cr:YSGG),³⁵ and carbon dioxide³⁶ lasers. The use of pulsed erbium lasers, such as Er:YAG and Er,Cr:YSGG lasers, has been considered for surface treatment. Previous reports have shown that the Er,Cr:YSGG laser is capable of producing surface roughness comparable to that produced by acid etching of enamel and dentin surfaces.^{37,38} Many studies have evaluated the effects of different surface treatments,^{12,15,39-41} including laser treatments,^{29,34,42-44} on the shear bond strength of resin cement to ceramic restorations. Shear bond strengths in Nd:YAG and Er:YAG laser-irradiated ceramic surfaces have also been investigated,⁴⁴ but not in those treated with a Er,Cr:YSGG laser. In addition, no consensus on the appropriate power settings for particular laser types has been reached.^{29,34,43}

Given the lack of information regarding the effects of different Er,Cr:YSGG laser power settings on ceramic surface roughness, we aimed to compare the effects of different ceramic surface modification techniques (HF acid etching and Er,Cr:YSGG laser irradiation) and laser power settings on the shear bond strength of resin cement to lithium disilicate ceramic.

MATERIALS AND METHODS

Fifty-five lithium disilicate ceramic blocks (5 mm × 5 mm × 2 mm; IPS Empress 2, Ivoclar Vivadent, Schaan, Liechtenstein) were fabricated using the hot pressing technique according to the manufacturer's instructions and embedded in acrylic resin. All specimen surfaces were wet ground with 300-1000 grit silicon carbide paper (English Abrasives & Chemicals Ltd., London, UK) on a grinding device (Phoenix Beta, Buechler, IL, USA) and ultrasonically cleaned (CD-4800 Digital Ultrasonic Cleaner; Jeken, Dongguan, China) in distilled water for 180 seconds to remove contaminants before surface treatment. They were then dried with oil-free air and assigned to five groups (n=11).

Specimens in Group 1 were subjected to 9.5% HF acid etching (buffered porcelain etch; Ultradent Products, South Jordan, UT, USA) for 60 seconds, rinsing for 5 minutes to remove residual acid, ultrasonic cleaning for 180 seconds, and then air drying. The ceramic surfaces of specimens in groups 2-4 were subjected to Er,Cr:YSGG laser irradiation (Millenium; Biolase Technology, Inc., San Clemente, CA, USA) with a 2.78 μm wavelength, pulsed laser-powered hydrokinetics, and energy parameters of 300 mJ at 1.5, 2.5, and 6 W, respectively. The air and vapor were adjusted to 50% of the laser unit. The optical fiber of the laser (400 μm diameter, 4 mm length) was aligned perpendicular to each specimen at a distance of 1 mm and moved manually in a sweeping fashion over the entire area during a 60 seconds exposure period. The laser-irradiated specimens were

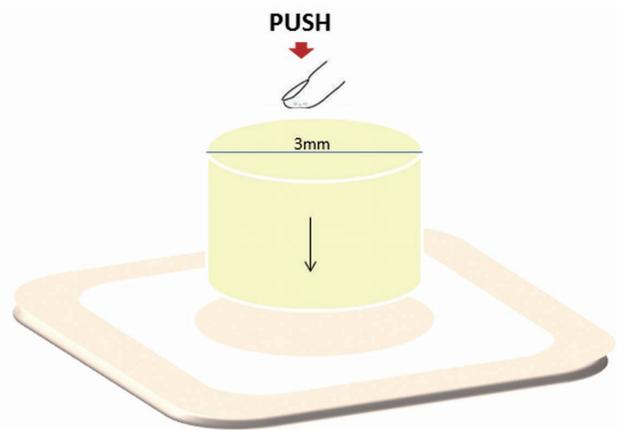


Fig. 1. Demonstration of the composite cylinder bonding to the ceramic.

then cleaned ultrasonically for 180 seconds and air dried. Specimens in Group 5 received no surface treatment and served as controls. One specimen from each group was selected for scanning electron microscopic (SEM) evaluation.

After surface treatment, a ceramic primer (Rely X; Lot #2721; 3M ESPE, St. Paul, MN, USA) was applied to the ceramic surfaces allowed to evaporate for 3 minutes and air-dried for 30 seconds. Then, an adhesive (Adper Single Bond; Lot #1122; 3M ESPE, St. Paul, MN, USA) was applied to the ceramic surfaces and air dried to achieve thinning without light curing, according to the manufacturer's instructions. To standardize the areas subjected to resin cement bonding, a perforated sticker (3 mm diameter) was placed in the center of each specimen (Fig. 1). Cylindrical composite resin blocks (3 mm diameter, 4 mm length; Filtek Z250, Lot #6020A2; 3M ESPE, St. Paul, MN, USA) were created using a transparent rubber ring mold and then bonded to the ceramic surfaces under consistent pressure by the same operator with a resin luting cement (Rely X ARC, Lot #CPCY; 3M ESPE, St. Paul, MN, USA). Care was taken to position the resin blocks on the intact ceramic surfaces, and excess resin cement was removed with an explorer before light curing for 40 seconds (Elipar Freelight 2; 3M ESPE, St. Paul, MN, USA). The stickers were then removed carefully from the ceramic surfaces. Polymerization was performed for 20 seconds and distributed equally around the circumference of each composite resin cylinder. The bonded specimens were stored for 24 hours at 37°C in distilled water in an incubator (UM 400; Memmert GmbH, Schwabach, Germany). Shear bond strength was then tested using a universal testing machine (Model 3345; Instron, Norwood, MA, USA) at 1 mm/min crosshead speed. One specimen from each group was examined by SEM (JSM-6335; JEOL, Tokyo, Japan) to assess the surface texture.

The data were analyzed by Kruskal-Wallis and Mann-Whitney U-tests using the SPSS software (version 15.0 for Windows; SPSS Inc., Chicago, IL, USA). *P* values <.05 were considered to indicate statistical significance.

RESULTS

Bond strength was significantly higher in Group 1 (HF acid etching; 8.42 ± 1.86 MPa) than in other groups ($P < .01$). Adhesion was significantly stronger in Group 2 (3.88 ± 1.94 MPa) and 3 (3.65 ± 1.87 MPa), which were laser-irradiated, than in Group 5 (1.95 ± 1.06 MPa; $P < .01$). There was no significant difference between Group 4 (3.59 ± 1.19 MPa), which was subjected to laser irradiation, and the control group ($P > .05$; Fig. 2).

SEM evaluation of the Er,Cr:YSGG laser-treated lithium disilicate ceramic surfaces revealed that surface structure depended on the surface treatment method and laser parameter. Lithium disilicate crystals were visible on the

specimen from Group 1 (Fig. 3). More surface irregularities were observed on the specimen from Group 2 than on the control specimen (Fig. 4). In turn, more surface irregularities were observed in Group 3 than in Group 2, despite the absence of a significant difference in shear bond strength values (Fig. 5). The specimen from Group 4 (6 W laser irradiation) also exhibited increased surface irregularity, as well as severely affected lithium disilicate crystals and over destruction of the surface (Fig. 6). The specimen from Group 5 had a typical untreated ceramic surface (Fig. 7).

Laser irradiation increased shear bond strengths compared with the control group. Higher laser power settings reduced bonding strengths between all-ceramic restorations and resin cement.

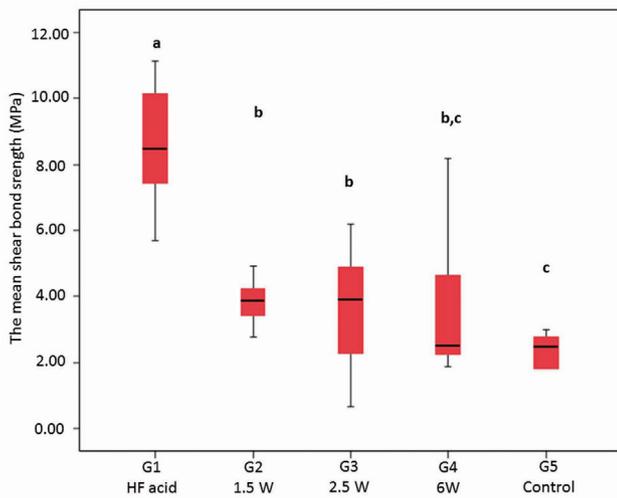


Fig. 2. The means and the standard deviations of the shear bond strength. Same lowercase letters indicate groups that were not statistically different ($P > .05$).

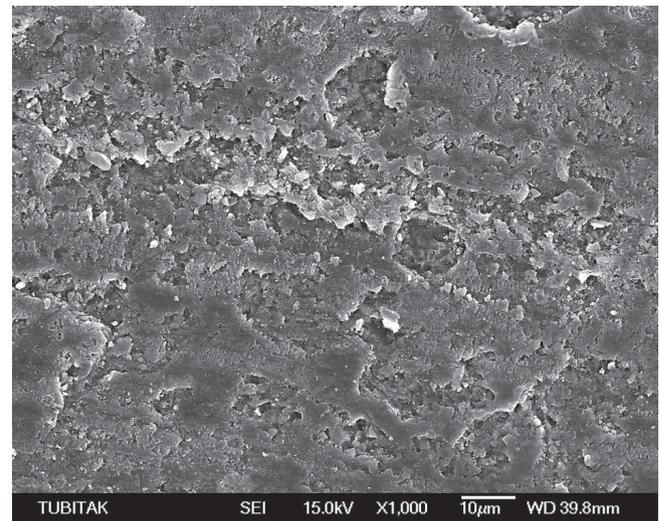


Fig. 4. Scanning electron microscopic image of 1.5 W laser irradiated surface ($\times 1,000$ magnification).

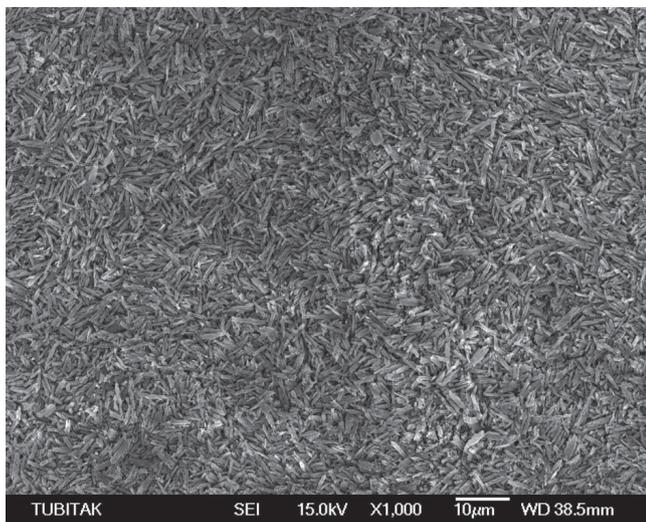


Fig. 3. Scanning electron microscopic image of hydrofluoric acid etched surface ($\times 1,000$ magnification).

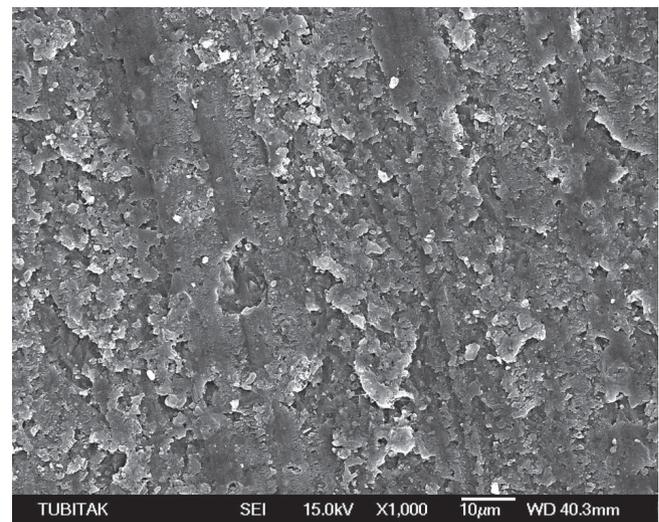


Fig. 5. Scanning electron microscopic image of 2.5 W laser irradiated surface ($\times 1,000$ magnification).

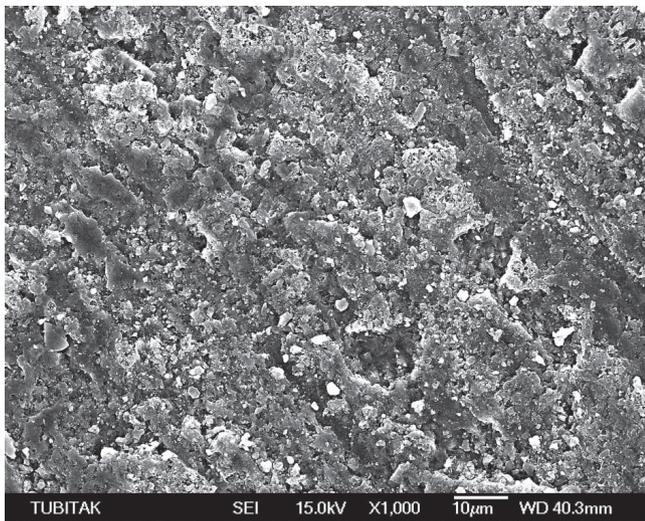


Fig. 6. Scanning electron microscopic image of 6 W laser irradiated surface ($\times 1,000$ magnification).

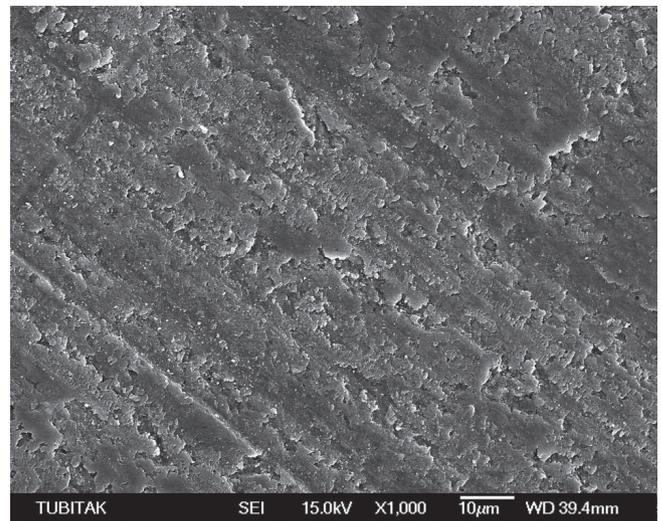


Fig. 7. Scanning electron microscopic image of no treated surface ($\times 1,000$ magnification).

DISCUSSION

In the current study, bond strength between resin cement and ceramic restorations was evaluated by applying different surface treatment methods. Modification of the lithium disilicate ceramic surfaces affected shear bond strength.

Dental materials are influenced by laser ablation rates. The effectiveness and safety of laser treatment, and thus bond strength, are related to the use of adequate parameters, such as the duration, frequency, and power of irradiation.⁴²

Er:YAG, Nd:YAG, and Er,Cr:YSGG lasers have been used to condition the surfaces of dental materials.^{34,35} Because no previous study has evaluated ceramic-surface conditioning using a Er,Cr:YSGG laser and because the wavelengths of Er:YAG (2.94 μm) and Er,Cr:YSGG (2.78 μm) lasers are similar, the results of the present study are compared to those using the Er:YAG laser.

Many *in vitro* studies have assessed shear bond strengths after surface treatment of ceramic restorations using different laser power settings. One study⁴⁴ used a 3 W power setting for Er:YAG laser irradiation, while another²⁹ assessed surfaces treated with 6, 12, and 18 W settings. Shiu *et al.*⁴³ evaluated Er:YAG laser treatment of feldspathic ceramic using 500 mJ pulses at a frequency of 4 Hz, and da Silva Ferreira *et al.*³⁴ applied Al_2O_3 using the same parameters. These studies illustrate the lack of consensus on appropriate power settings for laser irradiation.

Akyil *et al.*⁴⁴ compared the shear bond strengths of feldspathic ceramics repaired with composite resin after six surface treatments, and found lower bond strengths in specimens treated with 3 W Er:YAG laser irradiation than in control specimens that received no surface treatment.

In the present study, all laser treatments were performed using 300 mJ pulses, but the power settings differed; 1.5

and 2.5 W laser irradiation enhanced shear bond strengths compared with the control group, but 6 W irradiation did not produce a significant difference. These findings differ from those previous study,⁴⁴ perhaps due to the use of different ceramic materials, initial surface treatments (grinding in the present study), laser types, and/or ablation rates.

Gökçe *et al.*²⁹ subjected Empress 2 ceramic specimens to various surface treatments: 9.5% HF acid etching for 30 seconds and Er:YAG laser treatment at 20 Hz with 300, 600, and 900 mJ pulses. Bond strength values were highest in the 300 mJ laser treatment group. Based on this finding, a 300 mJ 20 Hz (6 W) treatment was included in the present study. In the same study, bond strength was found to decrease with increasing energy per pulse. The authors attributed the low bond strengths in the 600 and 900 mJ groups to the inadequacy of microdepths created by the Er:YAG laser, and assumed that they might be due to overdestruction of the crystal and/or matrix phases or creation of a heat-damaged layer.²⁹ In the present study, SEM analysis revealed overdestruction and weakening of the surface, rather than heat damage, in the specimen from Group 4 (6 W laser irradiation). No heat damage could be attributed to the hydrokinetic system of the Er,Cr:YSGG laser; the absorption of laser energy by water microdroplets is believed to be partially responsible for its hard tissue-cutting effects.^{45,46}

In the present study, testing of the same ceramic material and power setting (6 W) used by Gökçe *et al.*²⁹ yielded different results, perhaps due to differences in the laser type and timing of irradiation. Lower power settings resulted in significantly higher bond strengths than the control group and with the 6 W power setting. These higher bond strengths were associated with the creation of surface irregularities, whereas overdestruction of the surface was observed in the 6 W group. Increased output power created

rough surfaces. Increase of shear bond strength was expected as a result of increased surface roughness. However, bonding ability decreased on the contrary. Therefore, it is supposed that despite an increase in penetration of the cement, bonding between the ceramic and cement decreases because of the overdestructive effect of the high power laser. As a result, it is speculated that high power laser application may decrease bonding ability of the ceramic material. In addition, HF acid etching was more effective than all laser treatments. These findings suggest that shear bond strength decreased with increasing output power; various energy settings should be evaluated in future studies.

A previous study comparing the effects of 10 surface treatments (Er:YAG laser irradiation and HF acid etching) on bonding strength between feldspathic ceramic and resin cement found that bond strength values did not differ significantly between laser-treated groups and the control group.⁴³ In contrast, 1.5 and 2.5 W laser treatments resulted in bond strengths that differed significantly from those of the control group in the present study, although 6 W laser treatment did not. This difference may be related to the use of different laser and ceramic types and power settings.

HF acid etching was found to result in superior bond strengths in the present study (8.42 MPa); it achieves proper ceramic surface texture by dissolving the glass matrix to expose the crystalline phase and several other studies have also demonstrated that HF acid etching is the most effective surface treatment.^{22,24} Akyil *et al.*⁴⁴ found the bond strength of HF acid etching higher than the present study, the difference can be attributed to the different etching time. However, HF acid etching cannot be used intraorally;⁴⁷ thus, laser irradiation at low-power settings may be used to obtain the highest possible shear bond strength values.

Only one type of laser with three power settings and one type of ceramic material were used in this study. Various laser treatments and parameters are known to affect ceramic materials differently.²⁹ Thus, evaluation of other ceramic and laser types and energy parameters may yield different results; therefore, further studies are required.

CONCLUSION

The application of 1.5 and 2.5 W laser irradiation increased shear bond strengths between lithium disilicate ceramic and resin cement compared with untreated surfaces. The use of an Er,Cr:YSGG laser at a 6 W power setting may not be an efficient ceramic surface treatment technique. HF acid etching increased the shear bond strength between resin cement and ceramic surfaces more effectively than any laser treatment.

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