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Bond Strengths of All-Ceramics: Acid vs Laser Etching

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Clinical Relevance

For surface modification of the inner surfaces of ceramic restorations, the etching pattern obtained by dental lasers could be an alternative to conventional acid etching.

SUMMARY

Various applications of dental lasers on dental materials have been proposed for surface modifications. This study evaluated whether laser etching could be an alternative to hydrofluoric acid (HF) etching. One hundred and ten lithia-based all-ceramic specimens (Empress 2) (R: 4 mm, h: 4 mm) were prepared and divided into five groups (n=22/group). The untreated specimens served as the control, while one of the experimental groups was treated with 9.5% HF for 30 seconds. Three remaining test groups were treated with different laser (Er:YAG laser wavelength:2940 nm, OpusDent) power settings: 300 mJ, 600 mJ and 900 mJ. Ten specimens in each group were luted to the other 10 specimens by a dual-curing cement (Variolink II), and shear-bond strength (SBS) tests were performed (Autograph, crosshead speed: 0.5 mm/minute). The results were statistically analyzed (Kruskal Wallis and Mann Whitney-U, α=.05). Mean SBS (MPa) were 31.9±4.0, 41.4±4.3, 42.8±6.2, 29.2±4.5 and 27.4±3.8 for the control and HF, 300, 600 and 900 mJ groups, respectively. SEM evaluations revealed different surface morphologies depending on the laser parameters. The differences between HF acid and 300 mJ, when compared with the control, 600 and 900 mJ groups, were significant (*p*<.05). The 300 mJ laser group exhibited the highest shear-bond strength values, indicating that laser etching could also be used for surface treatments.

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INTRODUCTION

Advances in adhesive dentistry have resulted in the recent introduction of modern surface conditioning methods in order to achieve high bond strengths.

Several conditioning methods have been suggested for ceramic surface pretreatment, such as sandblasting, chemical etching and silica coating.[1] Among the chemical etchants, hydrofluoric acid (HF), acidulated phosphate fluoride and ammonium bifluoride were reported to promote micromechanical retention.[1,10] The use of an acid-based gel for ceramic etching has the advantage of ease of chairside use and has been shown to provide better retention.[3]

Etching the inner surface of a restoration with different concentrations of HF acid, followed by the application of a silane coupling agent, is a well known and recommended method to increase bond strength.[4]

In addition to the currently used ceramic surface conditioning methods, processes for laser-induced modification of ceramic materials have also been investigated. However, there is little in the existing literature about the laser treatment of dental ceramics.[5] During laser treatment, steep local temperature changes in the heating and cooling phases create internal tensions that can damage the materials. Therefore, it is necessary to use appropriate laser operating parameters.[6]

Various applications of dental lasers on dental materials (CO_2, Nd:YAG lasers) have also been proposed for surface modifications, such as forming a glazed surface layer on ceramics, the removal of resin composite filling materials, laser welding of ceramics and metal alloys, including titanium, and increasing the corrosion resistance of metal alloys.[2] Among the several applications of dental lasers, enamel and dentin etching have been reported to be most frequently performed by Er:YAG lasers.[7] However, their roughening capacity of the inner surfaces of all-ceramics for adhesive luting procedures is unknown. Therefore, this study evaluated whether laser etching could be an alternative to HF acid etching for ceramic surfaces.

METHODS AND MATERIALS

One hundred and ten lithia-based all-ceramic specimens (Empress 2, Ivoclar, Schaan, Liechtenstein) (diameter: 4 mm, height: 4 mm) were prepared according to the lost wax technique recommended by the manufacturer and ultrasonically cleaned for 15 minutes in ethanol and deionized water. They were then divided into one control and four experimental groups (n=22/group). Untreated specimens served as the control. One of the experimental groups was treated with 9.5% HF acid for 30 seconds. The other three experimental groups were treated with different laser power settings (300, 600 and 900 mJ), with a repetition rate of 20 Hz and an adjustable air and water spray (Er:YAG laser, wavelength: 2940 nm, pulse duration of 250 microsec, OpusDent, Tel Aviv, Israel). Laser energy was delivered through a hollow wave-guide system to a sapphire tip terminal 10 mm long and 1 mm in diameter.

Table 1: *Energy and Power Densities for Each Laser Group*

Power Settings	Power Density (W/cm^2)	Energy Density (J/cm^2)
300 mJ	191.08	9.5
600 mJ	382.16	19.1
900 mJ	573.25	28.7

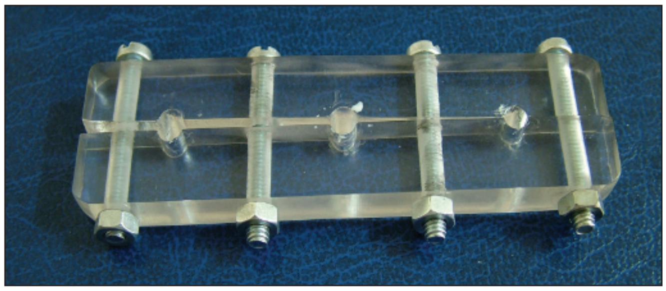

Figure 1: *Plexiglass mold for the luting procedure.*

Average power output varied from 4 to 10 W, depending on the laser energy. The energy and power densities for each laser group are listed in Table 1. The air and the water spray of the handpiece was adjusted to “50” scale of the laser unit. The beam was aligned perpendicular to the specimens at a distance of 1 mm and moved in a sweeping fashion by hand during a 20 second exposure period over the entire area. The irradiated specimens were dried with an oil-free air source for 15 seconds.

Two specimens from each group were separated and evaluated for surface characteristics under SEM (Jeol JSM-5200, Tokyo, Japan).

Then, all specimens were silanated (Monobond-S, Ivoclar, Schaan, Liechtenstein) for 60 seconds and dried gently, and the bonding agent (Heliobond, Ivoclar, Schaan, Liechtenstein) was applied.

Ten of the ceramic specimens in each group were luted to the remaining 10 specimens using a dual-curing cement (Variolink II, Ivoclar, Schaan, Liechtenstein) applied on both opposing ceramic surfaces according to the manufacturer’s instructions and were light cured for 160 seconds circumferentially with a light-curing unit (Elipar Trilight 3M, ESPE, Germany) with an energy output exceeding $500mW/mm^2$. A plexiglass mold (Figure 1) was used for cementation under a 600 gr load.

After storing the luted specimens in deionized water at 37°C for 24 hours, the shear-bond strength (SBS) tests were performed with a Shimadzu universal testing machine (Autograph, Shimadzu Corp, Japan) at a crosshead speed of 0.5 mm/minute. The shear bond test results were statistically analyzed by Kruskal Wallis and Mann Whitney-U tests for all groups (SPSS 11.0 for Windows, Chicago, IL, USA). After the SBS tests, some representative specimens were also evaluated under SEM to determine the pattern of debonding.

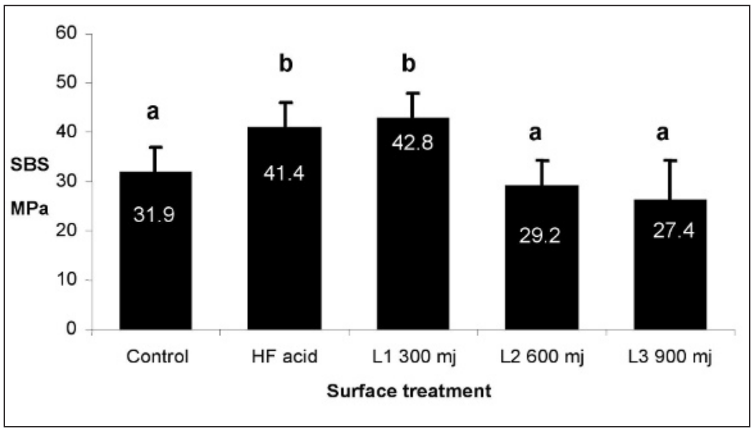

Figure 2: *Mean SBS values (MPa) of the test groups. *No significant differences are present among the groups with the same letters.*

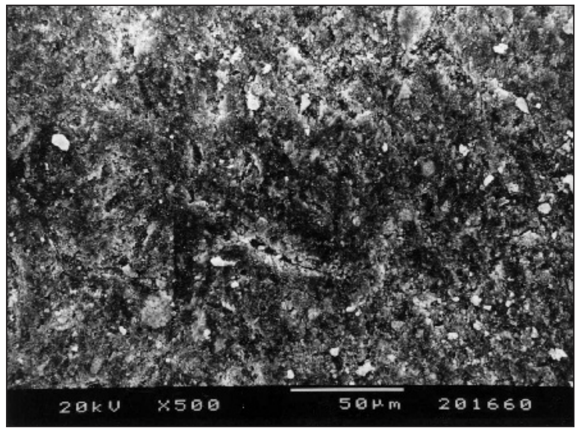

Figure 3: *SEM view of the untreated control group showing the intact glassy phase.*

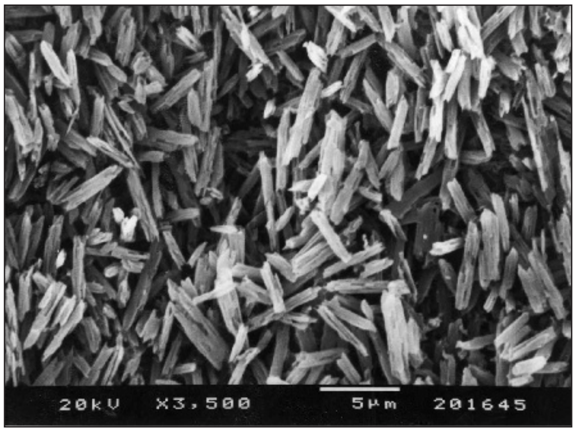

Figure 4: *SEM view of the HF acid etched group. Note the lithium disilicate crystals.*

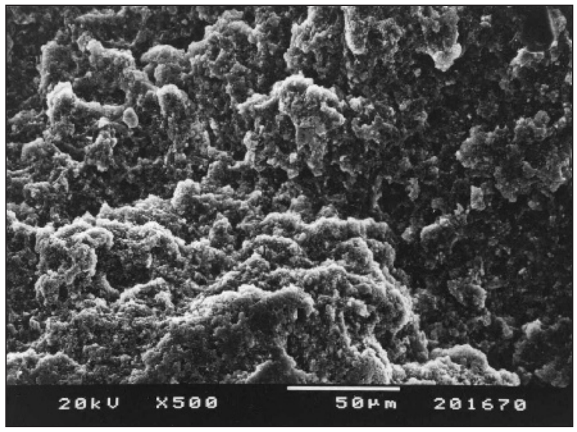

Figure 5: *SEM view of the 300 mJ lased group showing irregular lithium disilicate crystals in smaller sizes.*

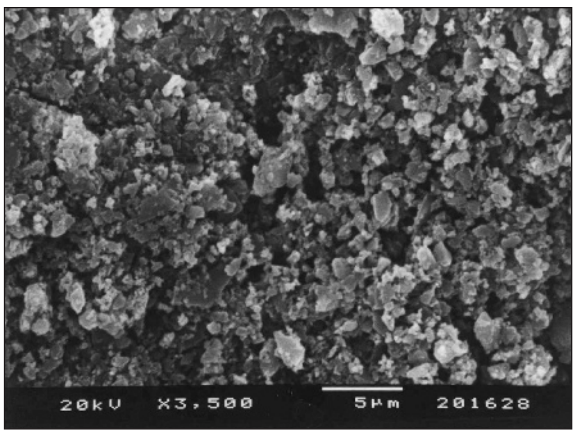

Figure 6: *SEM view of the 600 mJ lased group. Increased surface irregularities with severely affected and disassociated lithium disilicate crystals.*

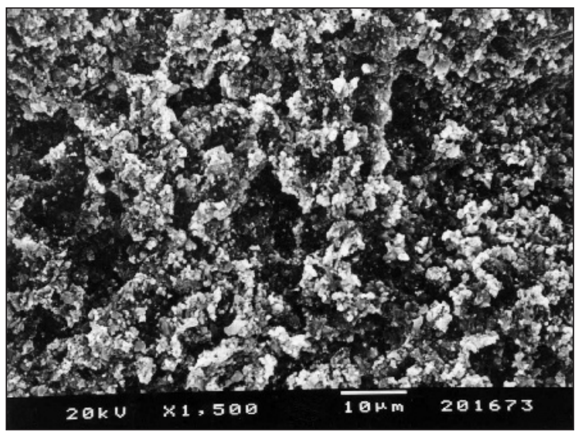

Figure 7: *SEM view of the 900 mJ lased group. Note the severely affected and disassociated lithium disilicate crystals.*

RESULTS

The mean shear-bond strengths are presented in Figure 2. Among the conditioning groups, significantly higher SBS values ($p<.05$) were obtained for the 300 mJ lased and HF acid applied groups. However, differences between the HF acid and 300 mJ lased groups were

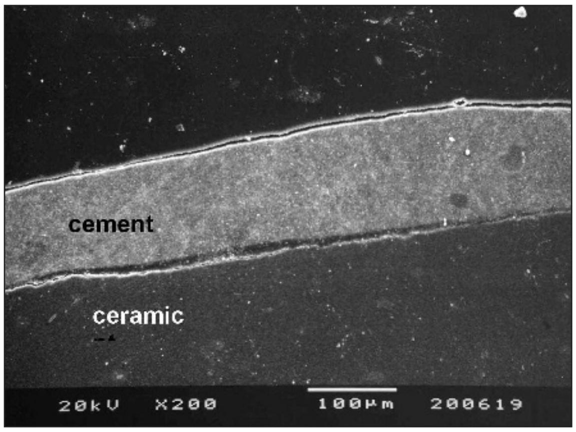

Figure 8: *Adhesive failure between the ceramic and the cement. No rough surfaces were noted on the ceramic.*

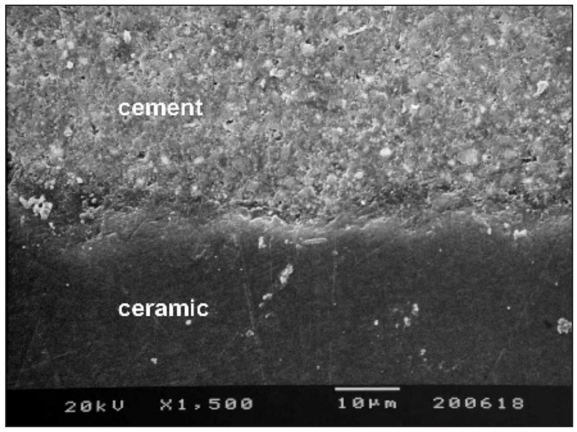

Figure 9: *Good adhesion at the cement-ceramic interface with increased surface roughness.*

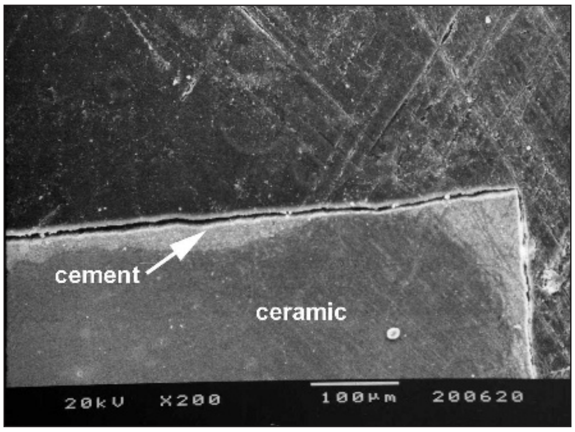

Figure 10: *No visible cement on the margins, while a cement remnant at the center of the specimen is observed for 300 mJ lased specimen.*

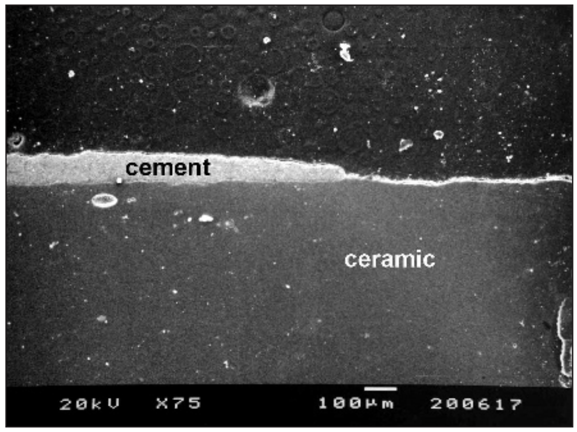

Figure 11: *Partially delaminated cement surfaces can be observed on the 600 mJ lased ceramic.*

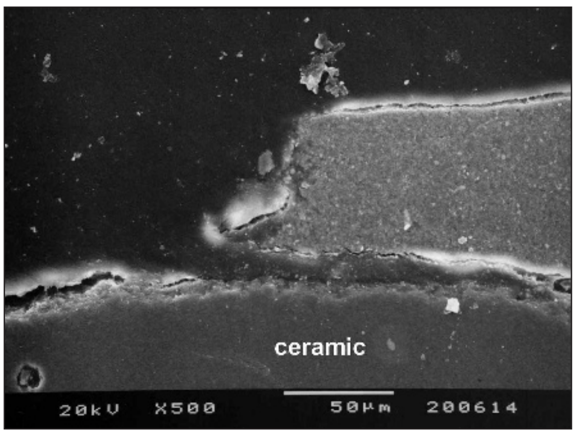

Figure 12: *Decreased irregularities on the ceramic surface of 900 mJ lased specimen.*

insignificant (p>.05). In addition, no significant differences among the control, 600 and 900 mJ groups were observed (p>.05).

SEM Evaluations

SEM evaluations revealed different surface morphologies, depending on the surface conditioning methods. The untreated control specimen did not exhibit any alterations in surface morphology (Figure 3). HF acid etching resulted in visible lithium disilicate crystals showing the appropriate etching pattern and surface for adhesive cementation (Figure 4). While the 300 mJ laser application exhibited irregular lithium disilicate crystals (Figure 5), the irregularities increased after 600 mJ and 900 mJ laser applications, where the crystals were severely affected and disassociated (Figures 6 and 7). After SBS tests, the untreated control specimen exhibited adhesive failure between the ceramic and cement (Figure 8). The HF acid applied specimen revealed increased surface roughness with irregularities on the ceramic surface (Figure 9), demonstrating predominantly cohesive failures in the cement. Three hundred mJ lased specimens showed combined adhesive+cohesive and predominantly cohesive failures (Figure 10) within the cement, while the 600 and 900 mJ lased specimens showed predominantly adhesive failures (Figures 11 and 12).

DISCUSSION

This study demonstrated an alternative ceramic etching pattern by laser treatment in comparison to conventional HF conditioning. Although HF acid was reported to be efficient in roughening feldspathic ceramic for bonding resin composite,[8-9] neither etching with these solutions, nor adding silane had been demonstrated to result in an adequate resin bond to some new ceramics.[10] However, HF acid etching has been shown to increase the shear-bond strength of ceramics.[1]

Earlier studies have reported superior bond strength using HF acid with silane.[10] The results of this study revealed that the SBS values obtained from the 300 mJ lased group were higher than the HF acid group; however, the difference was insignificant. This effect might be attributed to the irregular surface produced as a result of the effective removal of the glassy phase and the preservation of lithium disilicate crystals, while higher power settings might have resulted in over destruction (disassociation) of the crystal and/or matrix phases. According to the authors of this study, one theory on low bond strengths after high laser power settings could be that higher laser power settings might cause a heat damaged layer. This layer might be poorly attached to the infra layers of the substrate, while the outermost layer of the substrate still strongly bonded to the silane and luting agents. In contrast, it has been suggested that the reason for the low SBS values could be disintegration of the ceramic crystals.

Silane treatment has been demonstrated to be essential for achieving chemical adhesion between the ceramic and resin composite,[1,4] but the possible effect of thermocycyling weakening the silane bond after HF acid etching should be considered.[4] Although no thermocycling was applied in this study, it is well known that thermocycling has a significant effect on bond strength; bond strength values decrease in comparison with studies in which no thermocycling was applied.[2,4]

Developments in laser technology have steadily been made in order to reduce heat related structural changes and damage to the surrounding dental tissues. Today, more than 10 different laser types are used in dental research.[5-6] Their effects on dental hard tissues and dental materials are under investigation. The CO_2 laser is well suited for the treatment of ceramic materials, since its emission wavelength is almost totally absorbed by ceramics. During the process of heat induction of ceramic surfaces with a focused CO_2 laser, conchoidal tears appear, which are typical effects of warming.[5]

The best known laser effect in dentistry is thermal vaporization of the substrate by absorbing laser light. Laser energy is converted to thermal energy. The Er:YAG laser has been reported to create thermomechanical effects on substrates, but there is still no common agreement about the possible benefit of this laser type.[5] However, this study demonstrated that high bond strengths can be obtained with the Er:YAG laser etching of ceramic luting surfaces, particularly at lower power settings.

The optimal etching pattern in this *in vitro* study was obtained by low laser power settings, indicating that it could be an alternative ceramic surface treatment method. However, the chairside use of a laser may not be as practical as the conventional gel form HF acid treatment. Hydrofluoric acid in gel form facilitates homogenous and controlled surface treatment, which can not be obtained with laser applications.

In this study, no cohesive failures within ceramic samples were observed; whereas, predominantly adhesive failures were observed between the ceramic and resin cement interface in control, 600 mJ and 900 mJ lased groups. On the other hand, combined (adhesive and cohesive) failures within resin cement were obtained both in the HF acid and 300 mJ lased groups. Increased surface roughness by the acid etching effect of HF treatment resulted in good adhesion between the ceramic and cement, while 600 and 900 mJ lased specimens exhibited poor adhesion patterns. These results could be explained by insufficient micro depths of the irregularities formed by high Er:YAG laser power settings, which resulted in limited penetration of silane and low SBS.

CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn:

1. Acid etching (9.5% HF) and 300 mJ laser application increased shear bond strength.
2. As the laser power setting increased, the mean SBS values decreased.
3. Further studies with different power settings and lasers might be required.

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