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The Effect of Laser Treatment on Bonding Between Zirconia Ceramic Surface and Resin Cement

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Abstract

Objective. The purpose of this *in-vitro* study was to evaluate and compare the effects of different surface treatments and laser irradiation on the shear bond strength of resin cement to zirconia-based ceramic. ***Material and methods.*** Forty zirconia core specimens (10-mm diameter, 2-mm thickness) were produced and embedded in the centers of autopolymerizing acrylic resin blocks. Subsequently, specimens were randomly divided into four groups, each containing 10 specimens, for different surface treatment methods. The details of the groups are as follows: Group C, no treatment applied (control); Group SB, bonding surfaces of ceramic disks were airborne particle-abraded with 110-μm alumina oxide particles; Group HF, bonding surfaces of ceramic disks were etched with 9.6% hydrofluoric acid; and Group L, bonding surfaces of ceramic disks were irradiated by a CO_2 laser. A total of 40 composite resin disks were fabricated and cemented with an adhesive resin cement to the specimen surfaces. A universal test machine was used for the shear bond strength test at a crosshead speed of 1 mm/min. ***Results.*** The highest shear bond strength values were obtained with Group L (20.99 ± 3.77 MPa) and the lowest values with Group C (13.39 ± 3.10 MPa). Although there was no significant difference between Groups C, HF and SB ($P > 0.05$), Group L showed a significant difference from all other groups ($p < 0.05$). ***Conclusion.*** All surface treatment methods improved the bond strength between resin cement and the zirconium oxide ceramic surface. CO_2 laser etching may represent an effective method for conditioning zirconia surfaces, enhancing micromechanical retention and improving the bond strength of resin cement on zirconia ceramic.

Key Words: *Airborne particle abrasion, laser etching, surface treatment, zirconium oxide*

Introduction

For many years the most predictable and durable esthetic correction of anterior teeth has been achieved by the preparation of complete crowns [1]. With the increase in the aluminum oxide (Al_2O_3) content of feldspathic ceramics, there has been a significant improvement in the mechanical properties of these materials, allowing metal-free restorations to be used more predictably [2]. One of the most commonly used all-ceramic core materials for conventional and resin-bonded fixed partial dentures and complete coverage crowns is yttrium tetragonal zirconia polycrystal (Y-TZP; zirconia) [3–6]. These ceramics with high crystalline content (aluminum and/or zirconium oxides) have been shown to demonstrate clinical success rates higher than or comparable to those of feldspar, leucite and lithium disilicate-based ceramics [7–9]. The introduction of zirconia frameworks extended the design and application limits of all ceramic restorations, leading to improved success and reliability [10]. With the development of computer-aided design–computer-aided manufacturing (CAD-CAM) technology, the design and production of zirconia frameworks could be achieved using a digital process [11,12]. Therefore, restorations using a zirconia framework became more practical [11].

Success with resin-bonded all-ceramic restorations is highly dependent on obtaining a reliable bond, which has to integrate all parts of the system into one coherent structure [13]. Resin bonding between a tooth and a restoration is advocated for improving the retention, marginal adaptation, fracture resistance and bond strength of restorations [14,15]. Obtaining adhesion between a luting agent and a ceramic surface requires surface pretreatment [16,17]. These surface treatment methods comprise grinding, abrasion with diamond rotary instruments, airborne particle

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abrasion with Al_2O_3, silicate coating (CoJet, Rocatec), acid etching with hydrofluoric acid, coupling with silane and combinations of any of these methods [4,18,19]. Etching with hydrofluoric acid is recommended only for surfaces with a glassy component, but it has been reported that such a procedure has no influence on zirconia ceramics, where no micro-grooves will be created [20]. However, Chaiyabutr et al. [18] stated that acid etching on a zirconia ceramic surface produced a significant difference in the surface roughness. Airborne particle abrasion with Al_2O_3 abrasive particles has been identified as an effective means of achieving a stable, durable bond for alumina- and zirconia-based ceramics [12].

Since the development of the ruby laser by Maiman in 1960, lasers have become widely used in medicine and dentistry [19]. CO_2 and Nd:YAG lasers are the most generally used instruments for both intraoral soft tissue surgery and hard tissue applications [21]. Only a few studies have been performed on the laser treatment of zirconium oxide ceramics [19,22]. During the process of heat induction of ceramic surfaces with a focused CO_2 laser, conchoidal tears—typical effects of surface warming—appear. These tears are believed to provide mechanical retention between resin composite and ceramics [19]. The disadvantages of lasers include stepped local temperature changes during the heating and cooling phases, which could create internal tensions damaging to teeth and dental materials [19].

The CO_2 laser is well suited to the treatment of ceramic materials because its emission wavelength is almost totally absorbed by the ceramic. Its effect on zirconia-based ceramics has not been well established. Therefore, the purpose of this *in-vitro* study was to evaluate and compare the effects of different surface treatments and laser irradiation on the shear bond strength of resin cement to zirconia-based ceramic. Our research hypothesis was that CO_2 laser treatment would not increase the bond strength more than other surface treatment methods.

Material and methods

The materials used, compositions and manufacturers' details are presented in Table I.

Forty zirconia core specimens (10-mm diameter, 2-mm thickness) were produced by a copy-milling system (Zirconzahn, Bruneck, Italy) using prefabricated blanks of zirconia (ICE Zircon Translucent; Zirconzahn) and then sintered according to the manufacturer's instructions. The surfaces were cleaned with ethanol and carefully air-dried before surface treatment (Branson 2210; Branson Ultrasonics Corporation, Danbury, CT). Zirconia cores were embedded in the centers of autopolymerizing acrylic resin blocks (Meliodent; Heraeus Kulzer, Armonk, NY). Zirconia core surfaces were ground-finished with a 600-grit silicon carbide abrasive (3M ESPE, St. Paul, MN) under running water on a polishing machine (Buehler Metaserv, Buehler, Germany) and ultrasonically cleaned for 3 min in ethanol and deionized water and then air-dried.

Subsequently, specimens were randomly divided into four groups, each containing 10 specimens, for the following different surface treatment methods.

Group C: untreated. No treatment was applied to the zirconia ceramic surfaces, and thus this group served as a control.

Group SB: airborne particle-abraded. Bonding surfaces of ceramic discs were airborne particle-abraded with 110-µm Al_2O_3 particles (Korox 110; BEGO, Bremen, Germany) applied perpendicular to the surface at a pressure of 120 psi for 10 s at a distance of 2–3 mm. After abrasion, the discs were thoroughly rinsed with a water spray for 30 s to clean the surface residual Al_2O_3 particles, and then dried with oil-free air.

Group HF: hydrofluoric acid-etched. Bonding surfaces of ceramic discs were etched with 9.6% hydrofluoric acid (Ultradent Porcelain Etch; Ultradent, South Jordan, UT) for 60 s. The gel was rinsed off with water for 20 s and then dried with oil-free air.

Table I. Materials used in the study.

Material	Type of material	Manufacturer
Ice Zircon	Yttrium partially stabilized with tetragonal polycrystalline structure (Y-TZP)	Zirconzahn SRL, Bruneck, Italy
Korox 110	99.6% 110-µm Al_2O_3 particles	BEGO, Bremen, Germany
Panavia F 2.0	Dual-polymerized adhesive resin cement (BPEDMA, MDP, DMA, Ba–B–Si glass, silica, chemical and photo-initiators)	Kuraray, Okayama, Japan
Ceramic Etching Gel	9.6% Hydrofluoric acid	Ivoclar Vivadent, Schaan, Liechtenstein
Filtek Z250	Composite resin	3M ESPE, Germany
Smart US20D	CO_2 laser	Deka, Florence, Italy

BPEDMA = Bisphenol-A-polyethoxy dimethacrylate; MDP = 10-methacryloyloxy-decyl-dihydrogenphosphate; DMA = Aliphatic dimethacrylate.

Group L: CO_2 laser irradiation. Bonding surfaces of ceramic discs were irradiated by a CO_2 laser (Smart US20D; Deka, Florence, Italy). Laser energy was delivered in a pulse mode with a wavelength of 10.6 μm, a pulse repetition rate of 1000 Hz and a pulse duration of 160 ms at an average power setting of 3 W.

Sixty composite resin discs (Filtek Z250; 3M ESPE, Germany) were fabricated by compacting the material into a polytetrafluoroethylene mold (Isoflon; Diemoz, France) with a hole in the center (6-mm diameter, 2-mm thickness). Composite resin was incrementally condensed into the mold to fill up the mold and each layer was light-polymerized for 40 s at a distance of 1 mm using a light-polymerizing unit (Astralis 3; Ivoclar Vivadent, Schaan, Liechtenstein) with an output power of 600 mW/cm^2. One composite resin block was fabricated for each specimen.

Composite resin discs were cemented to the specimen surfaces with dual-polymerized adhesive resin cement (Panavia F 2.0; Kuraray Co Ltd; Osaka, Japan) containing the adhesive phosphate monomer 10-Methacryloyloxydecyl dihydrogen phosphate (MDP). For cementation, equal amounts of a dual-polymerized resin-luting agent paste base and catalyst were mixed and applied to the composite resin block with a plastic spatula. Each composite disc was bonded to a zirconia core specimen under finger pressure. The excess resin cement was removed by means of a brush. The resin cement was then light-polymerized for 20 s with a curing light (Astralis 3). A glycerin gel (Oxyguard II; Kuraray Co Ltd, Osaka, Japan) was applied to the cement layer for 10 min. The specimens were washed with an air–water spray and were then stored in distilled water at 37°C for 24 h before the shear bond strength test.

A universal test machine (Lloyd LRX; Lloyd Instruments PLC., Fareham, UK) was used for the shear bond strength test at a crosshead speed of 1 mm/min. Each specimen surface was parallel to the direction of the force during the shear strength test. Force was applied to the zirconium–composite interface. The shear bond strength values were calculated in megapascals (MPa) by dividing the failure load (N) by the area of the composite resin (πr^2). Data were statistically analyzed. The Kolmogorov–Simirnov test showed that the data followed a normal distribution ($P > 0.05$). A homogeneity of variance test was done using Levene's test ($F = 0.475$, $P > 0.05$). Means and standard deviations (SDs) of bond strengths were calculated and mean values were compared by one-way ANOVA (SPSS 12.0; SPSS Inc., Chicago, IL), followed by a multiple comparisons test performed using a post-hoc Tukey test ($\alpha = 0.05$).

To evaluate the effects of different surface treatment methods on the surface morphology of zirconia core ceramic, four additional samples were treated with the same experimental protocol as described previously. All specimens were coated with gold using a sputter coater (S150B; Edwards, Crawley, UK) and examined under a field emission scanning electron microscope (JSM-6335F; JEOL, Tokyo, Japan) at 15 kV. The scanning electron microscopy (SEM) images were developed at a magnification of ×500 for visual inspection (Figure 1).

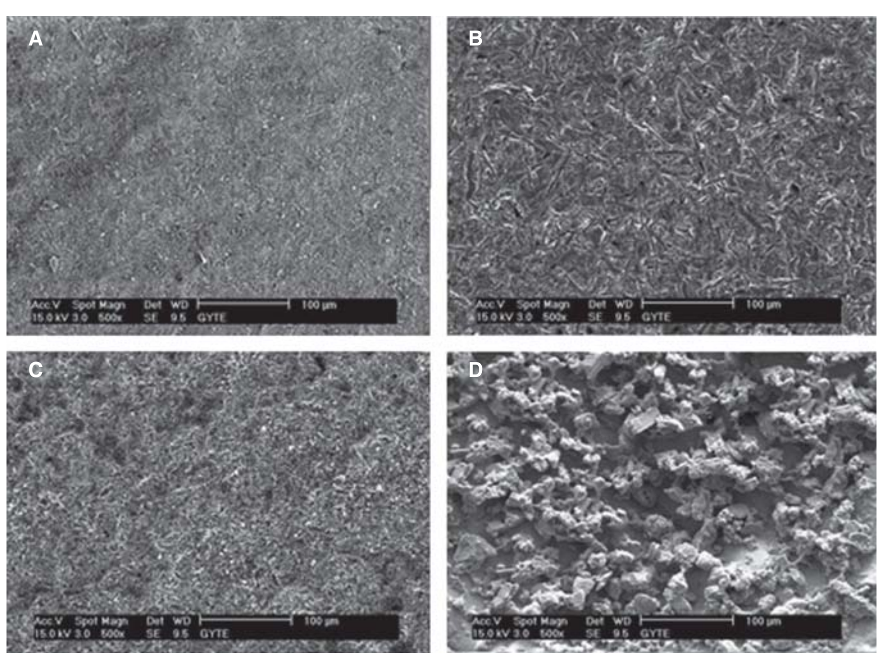

Figure 1. SEM images of zirconium oxide ceramic surfaces: (A) untreated; (B) airborne particle-abraded; (C) hydrofluoric acid etching; (D) CO_2 laser irradiation. Original magnification ×500.

Results

The shear bond strength values of the untreated group were compared with the values after the surface treatments for each of the treated groups. The results of the one-way ANOVA are presented in Table II.

The mean shear bond strength values and SDs are listed in Table III. The highest shear bond strength values were obtained with Group L (20.9 ± 3.77 MPa) and the lowest values were obtained with Group C (13.4 ± 3.10 MPa). While there were no significant differences between Groups C, HF and SB ($P > 0.05$), Group L showed significant differences from all other groups ($P < 0.05$). The greatest number of adhesive failures occurred in Group C and the greatest number of cohesive failures in Group L.

SEM images of treated zirconia core surfaces are shown in Figure 1. When the SEM images were evaluated, the airborne particle-abraded surface of the zirconia core showed irregularities (Figure 1B). Treatment with hydrofluoric acid did not change the superficial structure when compared with the airborne particle-abraded surface (Figure 1C). The laser-irradiated porcelain surface exhibited significant irregularities and retentive areas (Figure 1D).

Discussion

The data support rejection of the hypothesis that laser treatment would not increase the bond strength more than other surface treatment methods. Laser treatment increased the shear bond strength values significantly ($P < 0.05$).

Adhesive resin cements are recommended for all ceramic restorations for their longevity and for success of the restoration. Various *in-vitro* studies have shown that airborne particle abrasion with Al_2O_3 particles is an essential step in achieving a durable bond to high-strength ceramics [23–27]. Different sizes of abrasive Al_2O_3 particles, between 50 and 110 μm, are generally used [23–25]. However, differences in the size of particles and the application time may induce discrepancies in the achieved results: excessively high pressure during blasting may initiate phase transition, and expedite the formation of micro-cracks, thus reducing the mechanical properties of zirconia [28,29].

As expected, in the present study, the application of airborne particle abrasion to a zirconia ceramic surface resulted in a increase in shear bond strength values. However, there was no significant difference between Groups SB and C. In this study we preferred 110-μm Al_2O_3 particles for airborne particle abrasion. No direct comparisons between the influence of different particle sizes and shear bond strength to machined zirconia ceramic have been identified.

Table II. Results of one-way ANOVA test.

	Sum of squares	df	Mean square	F	P
Between groups	352.148	3	117.383	8.130	0.000
Within groups	519.795	36	14.439		
Total	871.942	39			

Table III. Mean and SD of shear bond strength values.

		Shear bond strength (MPa)	
Group	Surface treatment	Mean	SD
C	Control	13.4[a]	3.1
HF	Hydrofluoric acid	14.1[a]	4.7
SB	Sandblasting	16.4[a]	3.4
L	CO_2 laser	20.9[b]	3.7

[a,b]Identical letters indicate no statistically significant difference ($P > 0.05$).

Hydrofluoric acid has been routinely applied for etching ceramics and has achieved proven bond strength because of the glassy matrix of such materials [20]. However, its effectiveness on zirconia is limited, due to the low amount of silica-based phase in the material composition; acid application did not produce any morphological change or improvement in average surface roughness and it is not recommended for etching high-strength zirconia cores [30]. Also in our research, Group HF did not show a significant improvement in zirconia ceramic–cement bonding. The increased shear bond strength values obtained after application of hydrofluoric acid may be explained by the increased wettability of the zirconium oxide ceramic surfaces.

A 3W CO_2 laser in superpulse mode was found to be appropriate for achieving a strong resin–ceramic bond as laser-treated surfaces reached a shear bond strength value higher than that of our Groups C, HF, and SB in a recent study. Akova et al. [22] reported that the laser etching of porcelain fused to metal crowns showed improved bonding results. When a laser is applied to a tooth surface, the major concern is the risk of pulpal damage from thermal effects. The adverse effects of excessive heat on pulpal tissue have been well described [22]. However, Obata et al. [31] reported that when the enamel surface was etched with a 3-W laser, the dental pulp showed a temperature increase of 3.5°C, which is within safety limits.

Different reasons for micro-crack formation on ceramics after CO_2 laser irradiation have been proposed. According to Großmann et al. [19], during the heating of the ceramic surface caused by the absorption of the laser radiation, superficial emission of ions, electrons, and atoms takes place. Owing to the characteristic photo-ionization caused by the radiation, a physical plasma emerges. Its formation is accompanied by the development of an extremely high pressure and fluctuations in temperature in the range 10,000–50,000 K. This may cause extreme physical stress in the rehardening ceramic surface [19].

Chemical etching is commonly used for comparison when discussing the bond strength of laser etching. Obata et al. [31] reported that laser etching produces a lower bond strength when compared with chemical etching. However, in our study, the laser-etching procedure produced higher bond strength when compared with other surface treatments. This can be attributed to the power levels of the CO_2 laser. An increase in laser power levels or the irradiation time may cause higher adhesive properties. The highest bond strength value in Group L may also be attributed to the micro-cracks created by the laser irradiation, which may have caused microretentive areas [19]. Furthermore, when the SEM image was evaluated the retentive areas could be observed (Figure 1D).

Several testing methodologies, namely shear, tensile, and microtensile tests, have been suggested for bond strength evaluation of resin-based materials to dental ceramics [19]. These test methods are based on the application of a load in order to generate stress at the adhesive joints until failure occurs [19]. In the present study, shear bond tests have been used; this commonly used bond strength test is fast and easy to perform and also reflects the clinical situation. It can be questioned whether tension tests are more appropriate for evaluating the adhesive capabilities of resin agents to ceramics [17]. However, our study did not intend to determine absolute bond strength values but to evaluate whether the pretreatments used showed dramatic differences in bond strength. In this experimental study, early shear bond strength was tested; the effects of thermal cycling and long-term storage on bond strength were not evaluated and these may thus be considered limitations of this study.

Conclusions

Within the limitations of this study, it can be concluded that CO_2 laser etching may represent an effective method for conditioning zirconia surfaces, enhancing micromechanical retention and improving the bond strength of resin cement on zirconia ceramic. Laser irradiation is recommended as an alternative surface treatment technique for bonding resin cement on zirconia ceramic surfaces, but further investigation is required to refine the technique.

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