# Effect of Surface Treatment with Carbon Dioxide (CO<sub>2</sub>) Laser on Bond Strength between Cement Resin and Zirconia

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# Abstract:

**Introduction:** Since it is not possible to form an adequate micromechanical bond between resin cement and zirconia ceramics using common surface treatment techniques, laser pretreatment has been suggested for zirconia ceramic surfaces. The aim of this study was to evaluate the effect of Carbon Dioxide (CO<sub>2</sub>) Laser treatment on shear bond strength (SBS) of resin cement to zirconia ceramic. **Methods:** In this in vitro study thirty discs of zirconia with a diameter of 6 mm and a thickness of 2 mm were randomly divided into two groups of 15. In the test group the zirconia disc surfaces were irradiated by CO<sub>2</sub> laser with an output power of 3 W and energy density of 265.39 j/cm<sup>2</sup>. Composite resin discs were fabricated by plastic molds, measuring 3 mm in diameter and 2 mm in thickness and were cemented on zirconia disk surfaces with Panavia F2.0 resin cement (Kuraray Co. Ltd, Osaka, Japan). Shear bond strength was measured by a universal testing machine at a crosshead speed of 0.5 mm/min. The fracture type was assessed under a stereomicroscope at ×40. Surface morphologies of two specimens of the test group were evaluated under SEM before and after laser pretreatment. Data was analyzed by paired t-test (p value < 0.05).

**Results:** The mean SBS values of the laser and control groups were  $12.12 \pm 3.02$  and  $5.97 \pm 1.14$  MPa, respectively. Surface treatment with CO<sub>2</sub> laser significantly increased SBS between resin cement and zirconia ceramic (p value = 0.001).

**Conclusion:** Under the limitations of this study, surface treatment with  $CO_2$  laser increased the SBS between resin cement and the zirconia ceramic.

Keywords: CO<sub>2</sub> laser; resin cements; zirconium oxide.

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### Introduction

Pure zirconia is an unstable combination of zirconium dioxide, and its tetragonal crystalline phase stabilized by yttrium oxide (Yttrium-tetragonal zirconia polycrystal) Y-TZP is also used in dentistry <sup>1</sup>. Due to its exceptional mechanical properties, such as bending strength superior to 1000 MPa, chemical stability, biocompatibility and adequate light properties <sup>2,3</sup>, this composition becomes a suitable substructure for all ceramic crowns and long

span fixed prosthesis 4-7.

Numerous studies showed that in order to have long term efficiency, indirect whole ceramic restorations need a stable bonding between cement and ceramic <sup>8,9</sup>. Using resin cements as bonding agent between tooth and restoration improves retention, restoration shear resistance and marginal adaptation, in addition to restoration bond stability <sup>10,11</sup>. If there is lack of adequate bond between tooth and restoration will be reduced; and the reduction of marginal adaptation increases the possibility of having secondary caries <sup>12</sup>.

To obtain a good micromechanical bond between resin cement and zirconia ceramic, you need to create roughness on the internal surface of the ceramic, for enhancing available surface for resin penetration. Regarding Y-TZP ceramics, conventional surface preparation methods such as hydrofluoric acid etching, are mostly considered ineffective, due to absence of glass phase in this material's composition <sup>8,9,12</sup>. Nevertheless, other methods such as surface abrasion, abrasion via diamond burs, air abrasion with alumina particles or silica covering, and selective infiltration etching have been suggested for these ceramics preparation <sup>13,14</sup>. A few studies have suggested application of Er: YAG (Erbium- doped yttrium aluminum garnet), CO2 (Carbon Dioxide) Laser and Nd: YAG (Neodymiumdoped yttrium aluminum garnet) for changing zirconia ceramics' surface, to improve it's bonding to dental structure 13,15,16.

Laser application has been taken in consideration in medical science and dentistry after 1960, by Convissar and Goldstein <sup>17</sup>. Recently, with laser technology advancement, laser is used to change surface in materials in order to improve the bonding to dental structure <sup>13,14</sup>. Some studies have reported that in comparison with control group and the application of Er:YAG Diode laser, using CO<sub>2</sub> laser increases bond strength between resin cement and zirconia ceramic <sup>16,18</sup>. Whereas some other studies demonstrated that in comparison to other zirconia surface treatment methods such as sandblasting and Er:YAG, Nd:YAG laser application, this method was not efficient for increasing bond strength or even decreased it <sup>19,20</sup>.

Zirconia ceramic has a high potential of absorbing  $CO_2$ laser wavelength. The increase of temperature and surface destruction due to laser absorption by ceramic creates some porosity on the surface, inducing an augmentation of micromechanical retention of resin cement to zirconia ceramic <sup>16</sup>.

Considering the esthetic quality and strong stability

of zirconia ceramic, and its growing use in aesthetic dentistry, creating bond between zirconia ceramic and tooth increases shear resistance and longevity of the restoration. It also provides the possibility of cementing zirconia posts via resin cements for highly deteriorated teeth.

The aim of this study was to determine the effect of  $CO_2$  laser on shear strength of resin cement to zirconia ceramic base. According to hypothesis zero of the study, surface preparation with  $CO_2$  laser does not increase shear bond resistance of resin cement to zirconia ceramic.

## Methods

In this experimental in-vitro study, 30 Y-TZP discs (Ceramill Zi; Amann Girrbach AG, Koblach, Austria) were used. Discs of 6 mm diameter and 2 mm thickness, were made by a milling machine by copy milling technic, (Ceramil mulltix; Amann Girrbach AG, Koblach, Austria), following the company's instructions. For this purpose, first of all, a brass mold of 6 mm diameter and 2 mm thickness was made, and then cemented to the Copy part of milling machine by cyanoacrylate adhesive. The brass model was analyzed by Tester bar of the machine, and in the other part of machine, presintered zirconia cylinders was prepared at the same dimensions by bur. After samples heat treatment, their surface was polished with 600 grit silicon carbide polishing paper. For 3 minutes, all samples were cleaned in isopropanol dissolvent with ultrasonic cleaning apparatus, and dried with air-syringe before surface preparation. Samples having bubbles or fissures were replaced by perfect samples. Samples were randomly divided into two groups of 15 each. First group: No preparation was done, taken as control group. Second group: Zirconia discs surface were treated with CO<sub>2</sub> laser (Smart us20D; Deka, Florence, Italy). Considering to the previous studies, the most efficient parameters and intensity of CO<sub>2</sub> laser were determined and were used to have effect on zirconia surface<sup>13,16,19,20</sup>. The laser wavelength was 10,6 micrometer, pulse repetition 100 Hz, pulse duration 160 ms, the ceramic hollow tube diameter 600 micrometer, output power 3W, energy density 265/39J/cm<sup>2</sup> and the laser diameter spot size at application site was 0,48 mm. The laser tip was maintained manually perpendicularly to the disc surface at 1 mm distance, and the whole zirconia disc surface with its 6 mm diameter (0,28 cm<sup>2</sup>) was lased for 10 seconds with a speed of 2mm/s, by laser and air spray <sup>13,16</sup>. One additional specimen from each groups was prepared and sputter-coated with gold before and after  $CO_2$  laser treatment, their surface micromorphology was evaluated by electronic microscope (JSM-6335F; JEOL, Tokyo, Japan), with 1000, 3000 and 5000 magnification.

Composite resin discs (Filtek Z 250; 3M ESPE, St Paul, USA) of 3mm diameter and 2mm thick were fabricated by compressing composite in a transparent mold on a glass slap and light cured for 40 seconds (Demi, Kerr Corporation, Middleton, USA), with 1000mW/cm<sup>2</sup> light intensity.

The composite discs were cemented to the zirconia discs surface via Dual Cured resin cement (Panavia F 2.0, Kuraray, Japan co Ltd, Osaka). Following the company's instructions, first of all Primer A &B were mixed together, then applied with micro brush on ceramic and composite. 20 seconds later, A & B phase of cement were mixed and placed between ceramic and composite discs. According to previous studies, each one of composite discs was bond to a zirconia disc under finger pressure <sup>13,19,20</sup>. Cement surplus was removed, and then the whole thing was light-cured for 20 seconds. Samples were rinsed with air-water syringe and placed in distilled water at 37 degree Celsius for 24 hours before shear strength test. Shear bond strength (SBS) was determined by a mechanical test machine (DARTEC, HC10, Dartec 1td, stourbridge, England) with 0.5mm/min loading speed.

All fractured samples were analyzed by one person twice via stereo microscope (SZ40, Olympus, Tokyo, Japan) at x40 enhancement, to evaluate the fracture type (Cohesive, adhesive or mixed). Paired-T test has been performed for data analysis. The confidence interval of this test was evaluated at 95% (1- $\alpha$  0,095), the test power at 80%, and the significance level below 0,05.

## Results

The mean value and standard deviation, as well as the minimum and the maximum of SBS in both control group and laser treated group are showed in Table 1. Data normal distribution was evaluated using Kolmogorov-Smirnov test, and parametric Paired-t test for data analysis.

Data analysis showed that in comparison with the control group, surface preparation with CO<sub>2</sub> laser increase

SBS significantly between Panavia resin cement and zirconia ceramic (P value= 0,001). Considering the evaluation of fractured samples under stereo-microscope, in control group 100% of fractures happened adhesively (between zirconia and cement). But in the group treated by  $CO_2$  laser, 80% of failures were adhesive (between zirconia and cement), and 20% were mixed (a part of resin cement remained on zirconia surface). The electronic microscope images of zirconia surface in case and control groups are showed on Figures 1 and 2. The lased zirconia surface images of 3000 and 5000 times magnified are demonstrated in Figures 3 and 4.

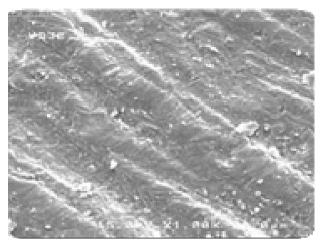


Figure 1. Zirconia surface in control group with ×1000 mangnification

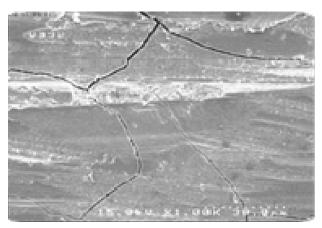


Figure 2. Zirconia surface after  $CO_2$  laser irradiation with  $\times 1000$  magnification (rough surface with scaly appearance and micro fissures)

Table 1. Comparing the shear bond strength of resin cement to zirconia ceramic in groups.

Group	Mean Bond Strength	<b>Standard Deviation</b>	Minimum Bond Strength	Maximum Bond Strength	*P-Value
Control	5.95	1.14	4.24	8.49	- 0.001
CO <sub>2</sub> Laser	12.12	3.02	9.48	21.23	
* Paired-t tes	it				

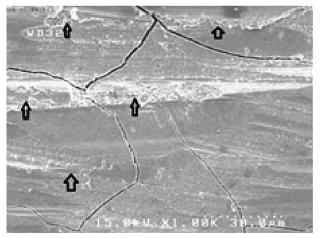
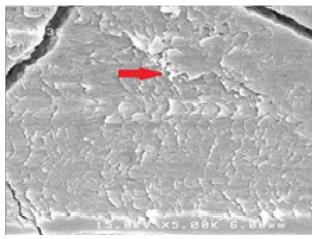


Figure 3. Zirconia surface after  $CO_2$  laser irradiation with  $\times 3000$  magnification (rough surface with scaly appearance and micro fissures)



**Figure 4.** Zirconia surface after  $CO_2$  laser irradiation with  $\times 5000$  magnification (rough surface with scaly appearance)

#### Discussion

As in clinical settings, shear stresses are the main factors involved in adhesion <sup>21</sup>, and happen easily and quickly. Therefor in this study, shear bond strength test was used. Although, in shear bond strength tests, it is difficult to completely control the applied force in order to have pure shear force on the study's subject.

All samples were immerged in 37°C distilled water for 24 hours, for completion of resin cement polymerization reaction, according to ISO 11405 TR standards <sup>22</sup>.

The results showed that  $CO_2$  laser application enhances shear bond strength of resin cement to zirconia ceramic, therefore null hypothesis of this study, which claimed that  $CO_2$  laser surface treatment of zirconia doesn't improve shear bond strength of resin cement to zirconia ceramic, is rejected.

Numerous methods have been suggested to obtain

a strong and durable bond between zirconia and resin cement <sup>11,14,23,24</sup>. Two methods, sandblasting and mechanical abrasion are able to create micro cracks in zirconia to provide retention; although these methods also weakens zirconia's mechanical properties <sup>25,26</sup>. Therefore, recently other methods such as laser etching have been introduced to create roughness on zirconia surface <sup>20</sup>. Resin cement bond strength to zirconia ceramics has been evaluated in several researches <sup>21,26-28</sup>.

In this study, in both case and control groups, Panavia cement which has ester phosphate monomer (10-methacryloyloxydecyl dihydrogen phosphate:MDP) has been used, because there has been numerous evidences showing that the bonding to Y-TZP ceramics will be improved by using products having chemical tendency to metal oxides <sup>28,29</sup>. Ester phosphate monomers such as MDP have chemical reaction with zirconium oxide and creates waterproof bond with zirconia ceramics <sup>30</sup>.

Previous experiments which studied the effect of laser treatment on shear bond strength between resin cement and zirconia, concluded that  $CO_2$  laser has the ability to enhance shear bond strength <sup>11,13,16,17</sup>. Zirconia ceramic has the ability to completely absorb  $CO_2$  laser wavelength. While absorbing the laser, multiple shell-form porosities are created due to a process of heat induction on ceramic's surface, which, by allowing resin penetration and then its hardening in the mentioned porosities, provides micromechanical bond with the ceramic <sup>16</sup>.

Through electron microscope evaluation, it was revealed that the application of  $CO_2$  laser forms a rough surface with a scaly appearance and micro cracks <sup>16</sup>, facts that confirm the present study's results (Figures 3, 4).

In contradiction with this research's results, Akin et al. <sup>22</sup> studied the effect of sandblast and also other different lasers on bond shear strength of resin cement to zirconia, and reported that  $CO_2$  laser reduces bond strength. They also reported that  $CO_2$  laser treatment creates a smooth surface without retentive capacity. But this conclusion depends on practical laser parameters. Since the main effect of laser energy is the transformation of light energy into heat, and the absorption of laser energy by material's surface is the most important interaction between material's surface and laser <sup>11</sup>, it is crucial to choose adequate laser parameters, in order to have a change in the material's surface and suitable surface treatment.

In Akin et al. study <sup>22</sup>, the conclusion could come from low density of  $CO_2$  laser (159,22J/cm<sup>2</sup>). Whereas in present study, despite a lower  $CO_2$  laser power (3W), a higher energy density (265, 39J/cm<sub>2</sub>) was applied and laser energy had a higher effect on ceramic surface to create roughness on the ceramic surface. The evaluation by electron microscope revealed that the scale shape porosities and the micro cracks created on the surface enhanced micromechanical retention and resulted in a higher shear bond resistance of resin cement to zirconia.

In control group, in which shear bond resistance was minimum (5,97Mpa), 100% of Failures were of adhesive type, and in  $CO_2$  laser treated group (12,12Mpa), 20% of Failures were of mixed type.

In addition to the value of bond strength between zirconia ceramic and resin cement, the durability of bonding has an important role in clinical success of restoration. One of this research's restrictions was that the bonding durability, has not been evaluated. Long term clinical studies are necessary to evaluate zirconia surface preparation by laser.

## Conclusion

Based on this research's results, surface treatment via  $CO_2$  laser enhances primary bond strength between resin cement and zirconia ceramic.

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