

## Original Article

# Effect of different laser treatments on the shear bond strength of zirconia ceramic to resin cement

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## ABSTRACT

**Background:** Several techniques such as sand blast, silicoating, and laser irradiation have been introduced for reliable bond between zirconia and resin cement. This study aimed to assess and compare the effect of three types of lasers on the shear bond strength (SBS) of zirconia to resin cement.

**Materials and Methods:** In this *in vitro* study, 55 zirconia disks (6 mm diameter × 3 mm thickness) were randomly divided into five groups: control (1), sandblast (2), carbon dioxide (CO<sub>2</sub>) (3), erbium-doped yttrium aluminum garnet (Er:YAG) (4), and neodymium-doped yttrium aluminum garnet (Nd:YAG) (5) laser irradiation. The surface morphology of one specimen from each group was evaluated by a scanning electron microscope. Zirconia disks were cemented to composite using Panavia F2. SBS test was performed at a crosshead speed of 1 mm/min after 24 h storage in distilled water and thermocycling. The data were analyzed by one-way analysis of variance and *post hoc* Tukey's HSD tests ( $\alpha = 0.05$ ).

**Results:** The mean SBS values of the groups such as sandblast, Er:YAG, Nd:YAG, and CO<sub>2</sub> lasers and control were 6.64 MPa, 6.63 MPa, 4.98 MPa, 4.39 MPa, and 2.32 MPa, respectively. No significant difference was observed between sandblast and Er:YAG laser and between Nd:YAG and CO<sub>2</sub> lasers.

**Conclusion:** All lasers increased SBS values of zirconia to resin cement in comparison to the untreated surface. Er:YAG laser was the most effective laser treatment on the bond strength equal to that of sandblast.

**Key Words:** Lasers, resin cements, shear strength, zirconium oxide

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## INTRODUCTION

In the course of past decade, zirconia ceramic materials have been successfully introduced to the dentistry because of their biocompatibility, high mechanical strength, good chemical stability, high toughness, and natural appearance.<sup>[1-4]</sup> Cementation is an important step to ensure the retention, marginal seal, and durability of indirect restorations.<sup>[5]</sup> Cementing

procedures are either adhesive or nonadhesive.<sup>[6,7]</sup> Short, tapered preparations will benefit from adhesive cementation.<sup>[8]</sup> One of the limitations of the zirconia ceramic is that it does not luted well with resin cements.<sup>[9]</sup> Because of polycrystalline structure of the zirconia ceramic and absence of silica content, micromechanical silica-silane bonds cannot be

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achieved. Hence, air particle abrasion and laser conditioning of these types of ceramic materials have been recommended by some authors.<sup>[9-12]</sup> Different techniques such as sandblasting,<sup>[13,14]</sup> silica coating,<sup>[14,15]</sup> silane application followed by acid etching,<sup>[16,17]</sup> and plasma spray<sup>[18]</sup> are available for conditioning ceramic surfaces to enhance the adhesion to cement or composite. Lasers have been recently introduced as an alternative means for ceramic surface treatment to improve their bond strength to cement and other materials.<sup>[19]</sup> There are many articles about the bond strength of laser-irradiated zirconia ceramic to resin cement.<sup>[9,20-24]</sup>

Aras *et al.* showed that laser treatment did not increase shear bond strength (SBS) between zirconia and resin cements.<sup>[25]</sup> Akylı *et al.* reported that air abrasion and silica coating are the most effective surface treatment methods, and carbon dioxide (CO<sub>2</sub>) and erbium-doped yttrium aluminum garnet (Er: YAG) laser irradiation can increase the SBS. However, it has been shown that neodymium-doped yttrium aluminum garnet (Nd: YAG) laser irradiation decreased the bond strength.<sup>[26]</sup>

There appears to be some controversy about the effect of laser treatment on enhancing the SBS between zirconia and resin cement. Whereas, some authors claim that CO<sub>2</sub>,<sup>[26-29]</sup> Er:YAG,<sup>[26-28,30]</sup> and Nd:YAG<sup>[30,31]</sup> laser treatments had good effects on bond strength. In contrast, some studies have demonstrated that irradiation of zirconia ceramic with CO<sub>2</sub>,<sup>[9,30,32]</sup> Nd:YAG,<sup>[26,33,34]</sup> and Er:YAG<sup>[21,33,35,36]</sup> lasers does not result in increased SBS to resin cement.

The aim of this study was to evaluate and compare the effects of three types of laser irradiation as well as sandblasting on the SBS of zirconia ceramic to resin cement. The null hypothesis was that there is no difference in the SBS between zirconia ceramic and resin cement treated with different lasers.

## MATERIALS AND METHODS

In this *in vitro* study, 55 zirconia ceramic disks (6 mm in diameter and 3 mm thick) were fabricated by a copy milling system (Zirkonzahn, Zirkonzahn GmbH, Bruneck, Italy) using prefabricated blanks of zirconia (ICE Zircon Translucent; Zirkonzahn) and then sintered according to the manufacturer's recommendations. Each specimen was finished with 600-800 grit silicon carbide paper (Matador 991A, Soflex, Starcke GmbH and Co. KG, Melle, Germany)

to standardize them. The surfaces of sintered zirconia disks were observed by an optical microscope with the  $\times 3$ , and the specimens with surface defects such as cracks or voids were replaced by intact ones. All specimens were ultrasonically cleaned in distilled water for 5 min before surface treatment and then air-dried. Afterward, the specimens were randomly divided into five groups ( $n = 11$ ), according to the surface treatment as follows:

Group 1: No surface treatment was applied in this group (control).

Group 2: Specimens were sandblasted with 50  $\mu\text{m}$  Al<sub>2</sub>O<sub>3</sub> particles (BEGO, Bremen, Germany) from a distance of 10 mm perpendicular to the specimen surface at a pressure of 2 bar for 15 s. The specimens were then cleaned with deionized water for 5 min.

Group 3: The surfaces of zirconia disks were irradiated by CO<sub>2</sub> laser (Smart US 20D, Deka, Florence, Italy). Laser beam parameters were selected based on the results of previous researches for micromechanical retention.<sup>[26,29,30,37]</sup> The wavelength of CO<sub>2</sub> laser was 10.6  $\mu\text{m}$ , with a pulse repetition of 100 Hz, pulse duration of 160 ms, output energy of 3W, and energy density of 265.39J/cm<sub>2</sub>. Laser was delivered by a 600  $\mu\text{m}$  hollow ceramic tip that was hand-adjusted perpendicular to the ceramic surface at a distance of approximately 1 mm. The whole surface of the zirconia disk was irradiated at a rate of 2 mm/s using horizontal surface scanning mode.<sup>[26,27,29,30]</sup> Air cooling was used during laser irradiating of the specimens.

Group 4: The surfaces of zirconia disks were covered with graphite powder and irradiated with Er: YAG laser (Fidelis Plus III, Fotona, Ljubljana, Slovenia). Laser parameters were set as follows: wavelength of 2.94  $\mu\text{m}$ , pulse duration of 50  $\mu\text{s}$ , output power of 2 W, pulse repetition of 10 Hz, and energy of 200 mJ. The sapphire tip was adjusted by hand at an approximate distance of 0.5 mm, perpendicular to the disk surface, and the entire zirconia disk surface was irradiated at a rate of 2 mm/s using horizontal surface scanning mode for 10 s. Fine air and water cooling was used during the irradiation of the samples.<sup>[22,26]</sup>

Group 5: The surfaces of zirconia disks were irradiated with Nd: YAG laser (Fidelis Plus III, Fotona, Ljubljana, Slovenia). Laser parameters were set as wavelength of 1064 nm, pulse duration of 300  $\mu\text{s}$ , output power of 2W, pulse repetition of 20 Hz, and pulse energy of 100 mJ.<sup>[9,26,33]</sup> Optical fiber was aligned perpendicular to the ceramic surface at 1

mm distance and the whole ceramic area was scanned for 20 s. Air cooling was used during laser irradiating of the specimens.

For evaluating the surface morphology of zirconia disk, a scanning electron microscope (SEM) (Philips xl20, Eindhoven, Netherland) was used with the magnification of 1000x. One additional specimen of each group was prepared and sputter-coated with gold. The samples were examined and photographed.

Composite resin (Filtek Z 250, 3M ESPE, St. Paul, MN, USA) was packed into transparent plastic molds (4 mm diameter and 6 mm thickness) incrementally and subsequently light-cured (Demi, Kerr, USA) for 40 s at a distance of 1 mm with 800 mW/cm<sup>2</sup>. Zirconia samples were cemented to the composite resin bases using a dual-cured resin cement (Panavia F2.0, Kuraray Noritake Dental Inc., Osaka, Japan) according to the manufacturer’s recommendations. Excess cement was removed using a microbrush and the specimens were light-cured for 20 s. Specimens were stored in distilled water at 37°C for 24 h and then thermocycled in water at temperatures between 5 and 55°C for 5000 cycles, with dwell times of 30 s in each bath and a transfer time of 2 s between baths.<sup>[34,38]</sup>

SBS test was performed at a crosshead speed of 1 mm/min by an Instron testing machine (Dartec HC10, Dartec Ltd., Stourbridge, England). Shear load was applied to the specimens statically until failure occurred. The bond strength values, expressed in MPa, were calculated using the following formula:

$$Stress = \frac{Failure\ Load\ (N)}{Surface\ Area\ (mm^2)}$$

Afterward, the specimens were examined with a stereoscopic optical magnifier (Nikon 88,286, ×40, Nikon, Kawasaki, Kanagawa, Japan) to assess the failure type. This analysis enabled three types of failures to be defined: adhesive failure at the ceramic/resin cement interface; cohesive failure in the resin or ceramic with no damage to the interface; and mixed failure by involving both the interface and the material.

### Statistical analysis

The data were analyzed using SPSS software version 18 (SPSS Inc, Chicago, IL, USA). Normal distribution of data was assessed using Kolmogorov–Smirnov test. One-way analysis of variance and Tukey’s *post hoc* tests were used to

compare SBS in the five groups.  $P < 0.05$  was considered statistically significant.

## RESULTS

The mean SBS values of five groups are presented in Table 1. There was a significant difference between groups ( $P < 0.001$ ). Groups 2 and 4 showed the highest SBS values and the lowest value was recorded for the group 1. There was no significant difference between groups 2 and 4 and between groups 3 and 5 ( $P > 0.05$ ). In group 1, the most number (100%) of adhesive failures occurred [Figure 1]. SEM images showed more irregularities in groups 2 and 4 than the other groups [Figure 2].

## DISCUSSION

The results of the present study showed that the zirconia surfaces sandblasted with 50 μm Al<sub>2</sub>O<sub>3</sub> particles and irradiated by Er: YAG laser had significant higher bond strength with the resin cement compared with other groups. Hence, the null hypothesis was rejected. Surface roughness is an important factor that enhances adhesion of zirconia to the resin cement by increasing the surface area, improving the wettability through reducing

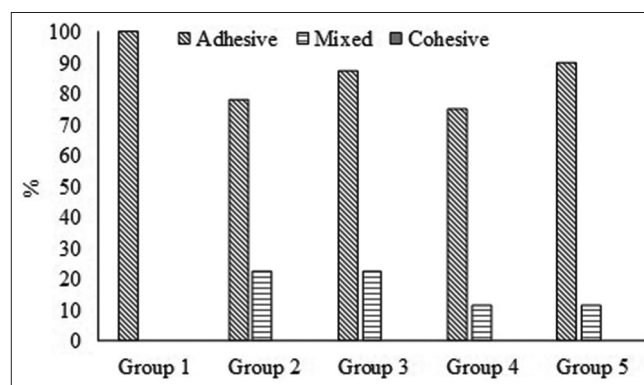
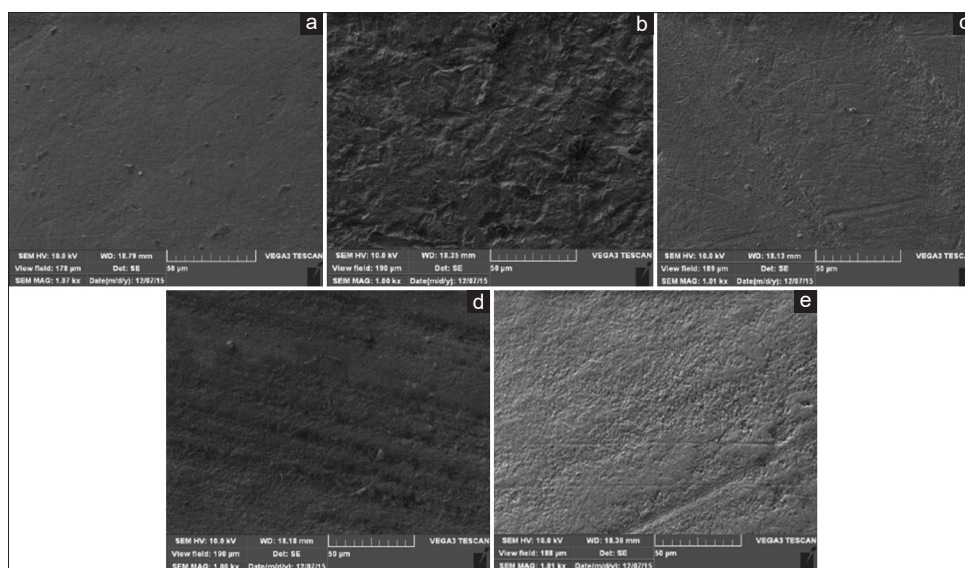


Figure 1: Distribution of failure modes in 5 groups.

Table 1: Mean and standard deviation of shear bond strength values

Groups	Shear bond strength (MPa)
Control	2.32±1.00 <sup>a</sup>
Sandblast	6.64±0.86 <sup>c</sup>
CO <sub>2</sub> laser	4.39±0.89 <sup>b</sup>
Er-YAG laser	6.63±0.82 <sup>c</sup>
Nd-YAG laser	4.98±0.97 <sup>b</sup>

The different superscript letters indicate statistically significant difference ( $P < 0.05$ ). Er-YAG: Erbium-doped yttrium aluminum garnet; Nd-YAG: Neodymium-doped yttrium aluminum garnet; CO<sub>2</sub>: Carbon dioxide



**Figure 2:** Scanning electron microscope images of zirconia surfaces: (a) Control. (b) Sandblast. (c) Carbon dioxide laser. (d) Erbium-doped yttrium aluminum garnet laser. (e) Neodymium-doped yttrium aluminum garnet laser.

the surface tension, and creating micromechanical retention.<sup>[39]</sup>

Most previous studies have found that sandblasting increases bond strength of resin cement to zirconia.<sup>[26,33,40-42]</sup> Two studies recommended that sandblasting can be used as the gold standard protocol for surface treatment of polycrystalline ceramics.<sup>[43,44]</sup>

SEM images showed remarkable differences in the surface topography of zirconia samples. These images revealed that the sandblasting and Er: YAG laser irradiation produced rougher surfaces and more irregularities (retentive pits and scratches) compared to the other two lasers. The Er: YAG laser can remove particles by microexplosions and by vaporization, a process called ablation. These findings are in agreement with those of Akyil *et al.*, who showed that Er: YAG laser irradiation at 2 W can create a rough surface similar to that of air abrasion.<sup>[26]</sup> Arami *et al.* reported the same surface roughness in zirconia samples treated by the Er: YAG laser and abraded with aluminum oxide particles.<sup>[45]</sup> In contrast, Stubinger *et al.*<sup>[39]</sup> observed that Er: YAG laser was not effective on zirconia surfaces.<sup>[46]</sup> This opposite result may be due to the application of graphite powder to cover the surface of samples in the current study. Er: YAG laser beam penetrates the zirconia material and thus is emitted from the opposite surface. It is assumed that the laser energy, at this wavelength, passes through the zirconia material without any significant energy absorption.<sup>[46]</sup> The most important laser effect is transforming the radiant energy into

heat (the thermomechanical effect). Absorption of energy by the material is the most important interaction between the material and laser.<sup>[39,46-48]</sup> This phenomenon is related to surface quality, pigmentation, and water content. Because zirconia is white opaque and has not water content, retention of laser energy is difficult.<sup>[35,39-41]</sup> Since the Er:YAG laser is not absorbed as well as the Nd:YAG and CO<sub>2</sub> lasers by the zirconia, the ceramic surfaces were covered with graphite powder to enhance its absorption.<sup>[26,27,49]</sup>

Two studies showed that the Er: YAG laser with the power setting of 200 mJ/pulse and 10 Hz for 5 s did not increase the bond strength as well as sandblasting.<sup>[21,22]</sup> This may be due to short irradiation time, while in the current study, the 10 s Er:YAG laser irradiation increased the bond strength compared to that of untreated materials.

In the present study, a mild surface alteration with shallow pits and scratch-like lines was seen by Nd: YAG and CO<sub>2</sub> laser irradiation in SEM images. No defect or microcrack was seen in the images. The lower SBS results obtained with Nd:YAG and CO<sub>2</sub> lasers can be explained by mild surface roughness observed in micrographs. These results were in line with some other studies<sup>[19,34,50]</sup> Nd:YAG laser irradiation can modify the ceramic surface by forming a glazed surface layer.<sup>[45,51]</sup> Akyil *et al.* reported that surfaces irradiated with Nd:YAG laser (1 W, 100 mJ/pulse at 10 Hz) were similar to an untreated feldspathic ceramic surface.<sup>[26]</sup> Akin *et al.*



reported that Er:YAG laser significantly increased the bond strength of resin cement to zirconia more than CO<sub>2</sub> laser. They reported smooth surfaces on CO<sub>2</sub> laser-treated zirconia samples with no retention.<sup>[30]</sup>

Some authors argued that overheating during irradiation of CO<sub>2</sub> or Nd:YAG laser may cause surface and subsurface destruction and microcracks that result in decreased SBS compared to untreated zirconia surface.<sup>[9,26,30,33]</sup> Integrity of ceramic surface was diminished after CO<sub>2</sub> laser treatment, which may affect the strength of the ceramic structure adversely.<sup>[10]</sup> Mahmoodi *et al.* reported that Nd:YAG laser can lead to thermal degradation of superficial layer of zirconia ceramic. They also found that poor connection between this layer and lower layers can cause debonding.<sup>[52]</sup> In this study, air cooling was performed during laser irradiation to prevent overheating.

Stubinger *et al.* found that CO<sub>2</sub> laser irradiation created distinct surface changes on zirconia ceramic at the power of 4-6 W. They showed that the highest roughness was produced at 4.5 W for 60 second, but observed material cracks at these powers.<sup>[46]</sup> Akyil *et al.* showed that CO<sub>2</sub> laser at power of 4 W for 50 s resulted in significantly higher bond strength of resin cement to zirconia, but microcracks and subsurface weakening could induce the adhesive mode of fracture.<sup>[26]</sup> Because high laser power settings may deteriorate the zirconia surface,<sup>[53]</sup> the low energy level of the CO<sub>2</sub> laser beam (3 W) was applied in the present study to prevent surface destruction.

Nd:YAG laser has been proposed by other researchers to increase the bond strength of zirconia ceramics to resin cements.<sup>[9,39,30,31]</sup> These findings are in contrast with other studies.<sup>[11,26,27,54,55]</sup> However, it should be noted that type of ceramic and resin cement, laser settings, test design, and subjection to artificial aging may be the source of different results.<sup>[52]</sup>

Laser settings such as power output, energy, repetition rate, pulse duration, or application time have great importance in order to prevent damage to the zirconia surface. Absorption of the laser beam energy by the material surface is the most important interaction between the laser and the material. Increasing the output energy and the pulse rate of the laser beam increase the energy density and thermal effects on the surface.<sup>[27]</sup> Higher surface roughness was seen by increasing output power and irradiation time of laser,<sup>[45,46]</sup> but greater intensities of laser are not suitable for treatment as a result of severe damage

to ceramic surface and phase transformation, causing unfavorable changes to superior mechanical properties of zirconia ceramics.<sup>[22,55]</sup> Therefore, lower power settings were selected in this study.

Furthermore, some differences in bond strength could be due to different thermocycling or cyclic loading protocols applied in studies evaluating bond stability.<sup>[19]</sup> There is no agreement on a suitable method for artificial aging,<sup>[56]</sup> but long-term water storage and thermal cycling are usually used. The ISO TR11405 standard represents that 500 cycles in water at 5-55°C is a proper aging regimen.<sup>[57]</sup> Approximately 10000 cycles reproduce 1 year of *in vivo* function.<sup>[38]</sup> In this study, all samples were stored in distilled water at 37°C for 24 h before testing, which is classified as a standard for short-term storage in ISO/TR 11405 and thermocycled for 5000 cycles. Some studies showed a reduction in bond strength of resin cement to zirconia after aging procedures. As in the research by Kasraei *et al.*, the mean SBS of Nd: YAG (18.95 Mpa) and CO<sub>2</sub> (14 Mpa) laser groups was decreased to 4.7 and 3.7, respectively, after 3000 cycles of thermocycling and 6-month water storage.<sup>[58]</sup> Kern and Wegner resulted that artificial aging with thermal cycling decreased the bond strength of resin to silicoated zirconia by approximately one-third of the initial bond. They showed that thermal cycling had a greater impact on the durability of the resin bond to zirconia ceramic than water storage.<sup>[59]</sup> Some studies reported higher bond strength than our results because artificial aging process was not done.<sup>[37,58]</sup> There was no specific value or optimum clinically SBS between resin cement and zirconia in the literature. Hence, it is impossible to compare our results with a cutoff point.

Besides the specify of the SBS values, the failure modes were analyzed to get more information about the probable outcome of treatment methods under clinical conditions. The bond quality should not be assessed only based on bond strength data. Cohesive and mixed fracture patterns are clinically preferable to adhesive mode of failure, since the last one is usually associated with low bond strength values. The failure mode results showed that all groups had a tendency to fail adhesive and most portion of their surfaces were free of cement remnants. The most adhesive failure was seen in the untreated group (100%) and in the Nd: YAG, CO<sub>2</sub>, sandblast, and Er: YAG groups. There was an increase in the frequency of adhesive failures at the zirconia–resin cement interface with a decrease in SBS values. There was no cohesive failure mode

among groups, indicating that the bond strength between resin cement and treated zirconia disks is lower than shear strength of ceramic. Although the zirconia restorations have not such a thickness (3 mm) and failure mode in the clinical situations may be different. Approximately the same level of mixed fracture (10% to 20%) was seen in the treated groups. Artificial aging may negatively affect the mode of failure. 80% of mixed failure was seen in the ceramic group irradiated by 2 W power of CO<sub>2</sub> laser without artificial aging,<sup>[37]</sup> whereas 22% of specimens failed mix at the CO<sub>2</sub> laser group in the current study.

To determine laser parameters for improving bond strength, standardized protocols should be used to reproduce clinical conditions. Furthermore, since the bonds are clinically subject to a combination of shear and tensile forces, shear and tensile tests should be performed for better analyzing of the bond strength between bonded surfaces. In future studies, degradation protocols should be applied to simulate the chemical effects of saliva and masticatory forces on restorations, which may affect the bond strength.<sup>[19]</sup> Moreover, the effect of different laser irradiation on the bond strength before and after sintering of zirconia should be evaluated. Furthermore, the effect of laser irradiation on the zirconia strength and its compositional changes must be analyzed. This study had limitations to simulate the clinical forces because the sample loading was static instead of cyclic fatigue. Furthermore, manual application of laser in this study can result in untreated or overirradiated areas. Evaluation of the bond durability of resin cement to zirconia with different treatment methods should be determined using long-term clinical studies.

## CONCLUSION

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

1. All three lasers improved SBS of zirconia to resin cement in comparison to the untreated surface
2. Er:YAG laser was the most effective laser treatment on the bond strength and its effect was equal to that of sandblast.

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### Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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