



**A COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH  
OF ZIRCONIA TO RESIN CEMENT AFTER VARIOUS SURFACE TREATMENTS  
- AN IN VITRO STUDY**

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**Abstract**

**Background:** High demand of metal-free restorations led to the development of all ceramic restorations which are widely used in dentistry due to their excellent mechanical properties and favorable esthetics. Poor bonding of ceramics to resin cement leads to the usage of surface treatment methods like sandblasting, acid etching, and laser etching.

**Aim:** To compare the effect of sandblasting with alumina particles and carbon dioxide laser surface treatment at different power outputs, 2W and 4W on the shear bond strength of Zirconia to resin cement.

**Methods:** In this in vitro experimental study 20 monolithic zirconia discs of dimension 10\*2 mm were prepared and divided into four groups based on surface treatment methods. Group 1 – No treatment was done and it served as a control group. Group 2 - Sandblasting with alumina

particles. Group 3 - Carbon dioxide laser treatment at 2watt power. Group 4 - Carbon dioxide laser treatment at 4-watt power. Composite discs were prepared of dimension 6\*2mm and then bonded with zirconia discs by resin cement. All the samples were subjected to a shear bond strength test.

**Results:** The highest shear bond strength was obtained with carbon dioxide laser irradiation at 4W power, followed by irradiation at 2W power and sandblasting with Al<sub>2</sub>O<sub>3</sub> particles. The lowest bond strength was observed in control group.

**Conclusion:** Carbon dioxide laser surface treatment method can be used as an alternative to sandblasting for improving bonding between ceramics and resin cement.

**Keywords:** zirconia surface treatment, laser etching, sandblasting, shear bond strength

**Introduction:** Zirconia restorations are the most commonly used among all-ceramic restorations due to their high mechanical strength, biocompatibility, chemical stability and favourable optical properties making it suitable to use in esthetic zone because it mimics natural teeth. Clinically it is used for fixed restorations, implant abutments, endodontic posts and orthodontic brackets. One of the limitations of the clinical use of zirconia is its bonding with resin cements.<sup>1</sup>

Various studies have shown that to have long term efficiency, ceramic restorations need a stable bonding between cement and ceramic. Bonding of resin cement to zirconia is compromised because of structural integrity of zirconia ceramic. High content of crystalline phase makes zirconia ceramics resistant to acid and non reactive. Roughened internal surface of ceramic increases surface area for resin penetration to obtain a good micromechanical bond between resin cement and zirconia ceramics. Various types of surface treatment methods have been used to enhance the bonding between resin cements and ceramics.<sup>2,3</sup>

Several surface treatments have been recently investigated but concerns still exist regarding the selection of the most appropriate zirconia surface pre-treatment. Neither hydrofluoric acid etching nor silanization result in a satisfactory resin bond to zirconia, because of the high crystalline content and the limited vitreous phase (below 1%) of this high-strength ceramic.<sup>4</sup>

Surface treatment methods that are mostly indicated for zirconia crowns are chemical modification of the surface, micromechanical interlocking through air abrasion, or a combination of both.<sup>5</sup>

The airborne-particle abrasion of zirconia ceramic surfaces with alumina particles has been found as an effective surface treatment method having a positive effect on the durability of the resin-zirconia bonding by increasing surface roughness, creating mechanical

interlocking, and simultaneously cleaning the surface. Some studies have shown that airborne-particle abrasion with alumina particles can cause damage to Y-TZP ceramic surfaces and can degrade the long-term performance of ceramic crowns. Flaws, debris, pits, microcracks, and alumina particles embedded during airborne-particle abrasion might negatively affect the durability of the resin-zirconia bonding.<sup>6</sup>

Development of the ruby laser by Maiman in 1960, lasers have become widely used in medicine and dentistry<sup>7</sup>. This resulted in the use of lasers as a surface treatment technique for zirconia Ceramics so as to enhance the bonding between ceramic and resin cement. The carbon dioxide (CO<sub>2</sub>), neodymium-doped yttrium aluminum garnet (Nd: YAG), and erbium-doped yttrium aluminum garnet (Er: YAG) lasers are the most usually used instruments for both intra-oral soft tissue surgery and hard tissue applications.<sup>8</sup> The CO<sub>2</sub> laser is compatible with the treatment of ceramic materials as a result of its emission wavelength is almost completely absorbed by the ceramic. During the process of heat induction of ceramic surfaces with a focused CO<sub>2</sub> laser, conchoidal tears—typical effects of surface warming—appear. These tears are believed to increase mechanical retention between resin composite and ceramics.<sup>7</sup> **Materials and Methods:** Monolithic zirconia samples of disc shape and of dimension 10 mm in diameter and 2mm in thickness were prepared from pre sintered blocks using CAD/CAM system. Samples were sintered in a special high temperature sintering furnace.

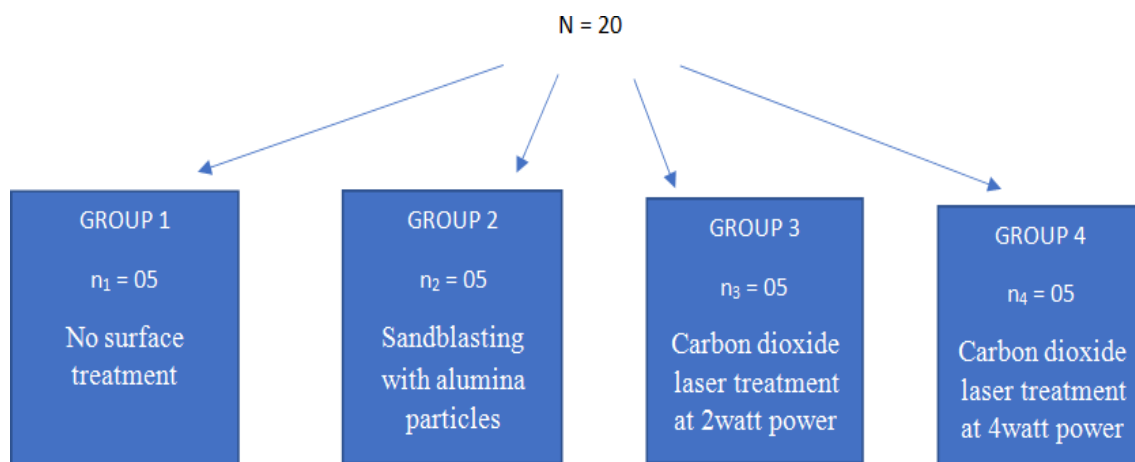
Zirconia ceramic specimen surface was polished with 600 grit silicon carbide polishing paper and then ultrasonically cleaned with distilled water for 5 minutes. Monolithic zirconia disc of size 10×2mm were embedded in the center of autopolymerizing acrylic resin block of size 12 × 12 mm (DPI RR COLD CURE)

A customized mould of polytetrafluoroethylene is prepared of size 6mm in diameter and 2mm in thickness. Composite resin (Any-Com Nano Hybrid Zirconium Composite Resin, light-cured radiopaque hybrid composite material, Made in Korea) was condensed into the mould incrementally and each layer is cured for 30 seconds using a light-polymerizing unit (LY- A180 Classic rechargeable light curing unit) with light intensity of 1200-2000mW/cm<sup>2</sup> and retrieved from mould.

All the prepared samples were segregated according to the test groups.

Total sample size = 20

There are four groups on the basis of surface treatment to be performed on monolithic zirconia disc.



### No Treatment

**Group 1 (n1=05)** samples were served as a control group. No treatment was applied on the zirconia ceramic surface.

### Sandblasting by Alumina

**Group 2 ( n2 = 05)** samples were Sandblasted by aluminum oxide powder with particle size 110- $\mu\text{m}$  at a pressure of 2.5 psi for 15 seconds at a distance of 10 mm from the surface of zirconia ceramic. The samples were rinsed thoroughly under tap water and air dried.

### Carbon Dioxide Laser Surface Treatment

**Group 3 (n3 = 05 )** – each sample was treated with carbon dioxide laser of wavelength 10600 nm at an output power of 2 watt using continuous mode with a focal spot diameter of laser beam 9 mm , scanning the whole surface of disc horizontally at a speed of 1mm / second and laser power density of 617 W/cm<sup>2</sup>

**Group 4 (n4 = 05)** - each sample was treated with carbon dioxide laser of wavelength 10600 nm at an output power of 4 watt using continuous mode with a focal spot diameter of laser beam 9 mm , scanning the whole surface of disc horizontally at a speed of 1mm / second and laser power density of 1234 W/cm<sup>2</sup>

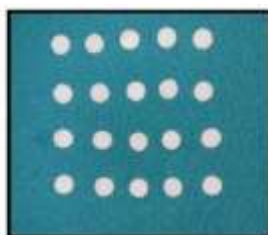


Fig.1 ZIRCONIA CERAMIC SPECIMENS



Fig.2 COMPOSITE RESIN



Fig.3 SANDBLASTING MACHINE

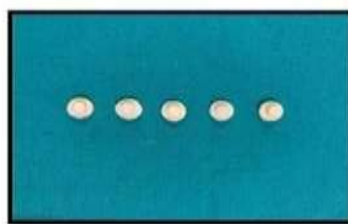


Fig.4 SPECIMENS AFTER CEMENTATION

**Cementation Procedure:** Composite resin discs were cemented to the specimen surfaces with dual- polymerized adhesive resin cement (Rely X ultimate Adhesive Resin Cement). Equal amounts of base and catalyst were mixed and applied to the composite resin block with a plastic spatula and then composite disc was bonded to a zirconium dioxide disc specimen under the finger pressure. The excess resin cement was removed with a brush and then light polymerized for 20 seconds with a curing light. Specimens were stored in distilled water at 37 °C for 24 hours before shear bond strength test.

**Shear Bond Strength Test:** A universal test machine was used for shear bond strength test with knife edge blade at a cross-head speed of 1 mm/ min. Specimen surface was placed parallel to the direction of the force applied during the shear strength test. Force was applied to the zirconium dioxide–composite interface until the failure occurred .The force recorded by the machine at the first peak of the curve was recorded as the maximum amount of force required to debond the zirconia ceramic and resin cement of each specimen. The shear bond strength values were calculated by dividing the load used until a fracture occurred (Newton, N) to the area of the composite resin ( $\pi r^2$ ).

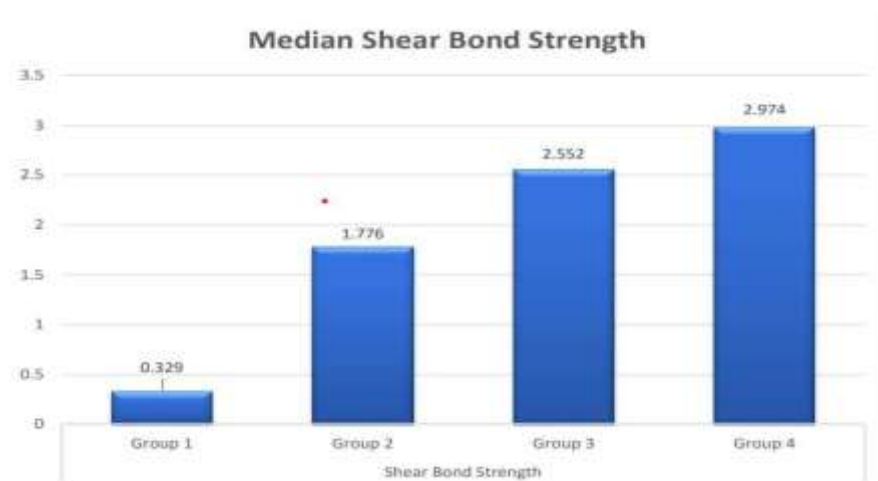
**Results and Observations:** The values of shear bond strength were subjected to statistical analysis. The data collected was entered in Microsoft Excel and subjected to statistical analysis using Statistical Package for Social Sciences (SPSS, IBM version 20.0).

#### Comparative evaluation of shear bond strength among study groups

Variable	Group	N	Mean Rank	Median	Kruskal-Wallis H	
Shear Bond Strength	Group 1	5	3.00	0.329	Value	17.860
	Group 2	5	8.00	1.776	df	3
	Group 3	5	13.00	2.552	P Value	0.000
	Group 4	5	18.00	2.974	Result	
	Total	20	10.50		Significant	

The non-parametric Kruskal Wallis test was applied which shows that the mean rank of shear bond strength of different groups was statistically significant. ( $P < 0.05$ ). The significantly higher mean rank 18.0 with median value 2.974 was for Group 4, followed by mean rank 13.0 with median 2.552 for Group 3 and mean rank 8.0 with median 1.776 for Group 2. Whereas significantly lowest mean rank 3.0 with median 0.329 was for Group 1. Individual group comparison was performed by applying Mann-Whitney U test as Post hoc test for non-parametric results.

### GRAPH 1. MEDIAN SHEAR BOND STRENGTH AMONG THE DIFFERENT GROUPS



Graph 1 shows the comparison of the shear bond strength among the different study groups. An evaluation of the median shear bond strength among the different study groups revealed highest shear bond strength with Group 4 (2.974) and lowest median shear bond strength with Group 1 (0.329).

**Discussion:** In the present study application of airborne particle abrasion, carbon dioxide laser treatment at 2W and 4W power to a zirconia ceramic surface resulted in an increase in shear bond strength values and significant differences between sandblasting, carbon dioxide laser treatment and the Control group were found. Clinically shear stresses are the main factors involved in adhesion, and happen easily and quickly. High strength is required in tension, shear and compression so as to resist stresses that occur at restoration and tooth interface. Thus, In this study, shear bond strength test was used. The highest shear bond strength was obtained with carbon dioxide laser irradiation at 4W power, followed by irradiation at 2W power and sandblasting with Al<sub>2</sub>O<sub>3</sub> particles. Lowest bond strength was observed with control group. This result is similar to the study done by Ural Ç, et al comparing the effect of sandblasting with 110 µm alumina particles and CO<sub>2</sub> laser irradiation at 3W output power on

the resin cement and zirconia ceramic shear bond strength. The results showed that both air abrasion and CO<sub>2</sub> laser enhanced the bond strength between zirconia ceramic and resin cement, bond strength obtained with CO<sub>2</sub> laser irradiation is more than the bond strength obtained with sandblasting. CO<sub>2</sub> laser etching may represent an effective method for etching zirconia to enhance bond strength and micromechanical retention, and improve the bond strength of resin cement on zirconia ceramic.<sup>8</sup>

In another study done by Ural Ç, et al compared the effect of different of carbon dioxide laser at different power outputs on bonding between zirconia ceramic surface and resin cement. zirconium dioxide core specimens were irradiated with a CO<sub>2</sub> laser at 2W,3W,4W and 5W power. The results showed that the relationship between laser output power and shear bond strength for zirconium dioxide ceramics plays a crucial role in bond strength between ceramic and resin cement. The highest shear bond strength was obtained with 2W power and irradiation with 4 W power resulted in low bond strength than 2W power which is contrary to the results of the current study.<sup>9</sup>

Goknil Ergun Kunt analyzed the surface roughness of yttrium stabilized tetragonal polycrystalline zirconia (Y-TZP) ceramics after sandblasting with alumina particles of size 50 µm, carbon dioxide laser irradiation at 3 W and 4 W power and Er: YAG laser irradiation at 150 mJ and 300 mJ . The results showed that the highest roughness values were obtained with air abrasion than with carbon dioxide laser irradiation.<sup>3,6</sup> These results are contrary to the results of the current study and this could be due to the difference in pressure and time at which air abrasion was performed and laser settings used for irradiation. The results of various lasers treatment on zirconia ceramics and particle abrasion on the surface morphology of zirconia ceramics were studied by Arami et al. and it showed that all treated surfaces were found to have higher roughness in comparison with control group.<sup>10</sup> Previous experiments which studied the effect of laser treatment on shear bond strength between resin cement and zirconia, concluded that CO<sub>2</sub> laser has the ability to enhance shear bond strength.<sup>11,12</sup>

In a study done by Farzaneh Ahrari et al to investigate the effect of air abrasion with 50 µm Al<sub>2</sub>O<sub>3</sub> particles at 2.5 bar pressure for 15 seconds, the application of universal primer (Monobond plus) and CO<sub>2</sub> laser irradiation at 10 W/10mJ, 10 W/ 14mJ and 20 W/ 10mJ on shear bond strength of Zirconia ceramic and resin cement. It was found that the highest shear bond strength value was obtained with CO<sub>2</sub> laser treatment at 20 W/ 10mJ (28.1 MPa) or 10W/ 14mJ (27.4 MPa), after that application of universal primer (Monobond plus) and air abrasion (16.2 MPa). It was concluded that carbon dioxide laser surface treatment provided the highest bond strength than Monobond plus and air abrasion, which is similar to the results of the current

study.<sup>13</sup>

Türker Akar et al evaluated the effect of shear bond strength between zirconia and resin cement by performing air abrasion with 120 µm Al<sub>2</sub>O<sub>3</sub> particles at a pressure of 200 kPa for 20 s at a distance of 10 mm and Nd: YAG laser irradiation at varying power levels –1 W, 2 W, and 3W. The laser irradiation (3 W, 1 W, and 2 W) of the pre-sintered zirconia surface resulted in the highest shear bond strength values than air abrasion.<sup>14</sup>

Varsha Murthy et al studied the effect of sandblasting with 110 µm alumina, sandblasting with 250 µm alumina, acid etching with 9.6% hydrofluoric acid, and irradiation with surgical carbon dioxide laser in a pulse mode at an average power setting of 3 W on shear bond strength between zirconia surface and resin cement. The results of the study revealed that higher shear bond strength was obtained with specimens treated with carbon dioxide laser at 3W power, followed by treatment with 9.6% hydrofluoric acid, sandblasting with 250 µm alumina particles, and 110 µm alumina particles. Surface treatments increased the bond strength between zirconia and resin cement and carbon dioxide laser could be an effective surface treatment for increasing the bond strength.<sup>15</sup>

Matani et al used airborne-particle abrasion with 80µm particles to decrease the damage to the zirconia surface that they believe would occur with larger particles. It has been shown that silica coating causes damage to the zirconia surface. Deterioration of the crystal or matrix phase may occur at high energy levels. It is therefore suggested to use low-level lasers and water spray.<sup>16</sup>

A study done by Sakineh Nikzadjamrani et al showed that the microtensile bond strength in the CO<sub>2</sub> laser-irradiated group was 40.3±6.84 MPa, almost as high as that of the Al<sub>2</sub>O<sub>3</sub> airborne-particle abraded group (43.58±10.02b) , however, it showed no statistically significant difference from the other groups.<sup>17</sup>

The Nd: YAG laser (1064 nm) and the CO<sub>2</sub> laser (10 600 nm) were evaluated for the surface conditioning of porcelain, leucite-based ceramics, and both glass-infiltrated alumina and zirconium dioxide composites, and the micromechanical retentions produced on the ceramic surface provided similar or even higher bond strength values than those obtained after conventional treatment.<sup>18,19</sup>

**Conclusion:** Surface treatment methods such as air abrasion , Irradiation with carbon dioxide laser provides sufficient shear bond strength of Monolithic Zirconia to resin cement in comparison with no treatment applied to Zirconia ceramic to enhance the bond strength between Zirconia ceramic and resin cement. Irradiation with carbon dioxide laser of wavelength 10600



nm at an output power of 2 watt and 4 watt at a continuous mode with a focal spot diameter of laser beam 9 mm, scanning the whole surface of disc horizontally at a speed of 1mm / second provides superior bond strength between Monolithic zirconia and resin cement than sandblasting the bonding surface of Monolithic zirconia ceramic with 110 $\mu$ m Al<sub>2</sub>O<sub>3</sub> particles.

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