# Shear bond strength of resin adhesive to laser treated surface of yttria tetragonal zirconia polycrystalline

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## Abstract

**Purpose:** This study evaluated the effects of surface treatment of zirconia with erbium-doped yttrium aluminum garnet (Er-YAG) laser on shear bond strength to resin cement.

**Materials and Methods:** Thirty disc-shaped zirconia specimens (10 mm in diameter and 2 mm in thickness) were fabricated, randomly assigned to 3 groups (n = 10) and treated surface with: Gr. N) no treated surface, Gr. S) sandblasted with 50 micron alumina and Gr. L) treated with an Er-YAG laser. Zirconia surface was then applied with metal/zirconia primer prior to be cemented to composite cylinder (5 mm in diameter and 4 mm in thickness) with resin cement. Shear bond strength test was performed at a crosshead speed of 0.5 mm/min. Data were analyzed by ANOVA and Tukey HSD tests.

**Results:** The means and standard deviations of shear bond strength (MPa) were: Gr. N) 10.79 ± 1.03, Gr. S) 14.15 ± 3.07, Gr. L) 14.11 ± 1.98. The treatment of zirconia surface were significantly affected to shear bond of resin adhesive to zirconia ($p< 0.05$). Treatment surface of zirconia with either Er-YAG laser or sandblast indicated significant higher shear bond strength than no treated surface ($p < 0.05$). No significant in bond strength between sandblasted surface and Er-YAG treated surface ($P > 0.05$).

**Conclusion:** Pretreatment of zirconia by Er-YAG laser improved bond strength to resin cement. The study suggested that Y-TZP ceramics should be treated surface with Er-YAG laser before cementation.

**Keywords:** Zirconia; Resin Cement; Er-YAG Laser; Shear Bond Strength.

## Introduction

The use of all-ceramic dental restorations has been increasing in recent years due to their superior aesthetic appearance and metal-free structure. Zirconia is one of the strong materials with high crystalline content that has been introduced to use as a core material for fixed partial dentures due to its unique mechanical properties in comparison to conventional alumina or lithium dislocate based ceramics when using in the stress bearing areas of the mouth.(1) Their high strength and high fracture toughness allowed for fabrication of long span all ceramic fixed partial dentures that can be either cemented with traditional cements or bonded with adhesive resin cements.

The long-term performance of ceramic fixed prostheses seems to be strongly dependent on the cementation procedure. The success of all ceramic restorations to achieve high retention, increased fracture and fatigue resistance depends on their cementation and bonding procedures to tooth structure.(2) There are some concerns for adhesive bonding of zirconia and other high strength ceramics to resin cements thus conventional cementation methods using zinc phosphate or polycarboxylate cements were recommended.(3,4) Zirconia ceramic restorations can be cemented to tooth structures by conventional cements or resin cements. However, resin cements are more preferred because they have some advantages of marginal seal, good retention and improvement of fracture resistance of ceramic materials.(5) Cementation of ceramic restorations with the resin-based materials can improve their marginal adaptation and bonding efficacy to the tooth structure. Some manufacturers have developed adhesive materials for cementation of zirconia restorations to provide cementation with high bond strength. It has been reported that good bond strength provided in cementation between zirconia restoration and resin cement is obtained by cement material containing with phosphate monomer in its composition.(6) Most of the resin-based cements used for this purpose contain phosphate monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP).

Several studies have shown that the durability of the ceramic restorations depends on the integrity of luting cement to ceramic.(7,8) A strong resin ceramic bond depends on the chemical bond between the ceramic materials and resin cement and also the micromechanical retention produced by surface roughening of ceramic materials.(9) The long term success of restorations depends on the preparation technique of the internal surfaces of ceramics prior to cementation, cement properties and bond strength between the cement and the ceramic.(10) Thus, proper ceramic surface treatment methods are necessary to produce surface roughening of ceramic materials for adhesive bonding.(11) Grinding with diamond rotary instruments, air abrasion with aluminous oxide abrasive, or etching can perform the methods by hydrofluoric acid are usually use procedure for treatment of dental ceramic.

Yttrium stabilized tetragonal zirconia polycrystalline (Y-TZP) comprises many of high crystalline content particles without glassy phase at the crystalline border.(12) Establishing a strong bond with zirconia is quite difficult. The material is resistant to aggressive chemical strong acids as hydrofluoric acid. Thus, acid etching procedure are not suitable for treatment surface of zirconia. In addition, it does not respond to common silanation procedures used with other glass containing ceramic materials.(13,14) Therefore, other surface treatment methods such as air abrasion with aluminum oxide ($Al_2O_3$) or silica coating with silica modified $Al_2O_3$ particles are frequently employed for Y-TZP restorations.(15-17)

Upon currently used ceramic surface conditioning methods, the erbium doped yttrium aluminum garnet (Er-YAG) laser is used in dentistry for many applications such as the removal of carious dentin, cavity preparation and surface treatment of indirect restorations.(18-20) Application of laser on surface preparation of zirconia ceramic can produce micro-irregular surface architecture.(21) The microscopic irregularities of ceramic restorative surface after Er-YAG laser application may play an important role in bonding. Different approaches to enhance zirconia resin bond strength were introduced to create a retentive surface where a strong durable bond between resin adhesive and with zirconia can be established, but still provide varying and controversial results.(22) Limited number of study has evaluated the effect of laser treatment on the bond strength between the zirconia ceramic and resin cements and remains controversy over the applicable surface treatments for Y-TZP ceramic materials.(23-27)

Since there is still no consensus in literature about the effective method to promote better bond strength for zirconia bonded to resin adhesive. Therefore, the purpose of this study was to evaluate the effect of zirconia surface treatment with Er-YAG laser on the shear bond strength between zirconia ceramic and resin adhesive cements. The null hypothesis was determined for zirconia surface treatment methods provide no influences on bond strength of zirconia ceramic to resin adhesive cement.

## Materials and Methods

**Zirconia Specimen Preparation:** The Y-TZP ceramic block (Cercon, Degudent GmbH, Hanau, Germany) was used in this study. The ceramic block consisted of 92% $ZrO_2$, 5% $Y_2O_3$ and <2% $HfO_2$ by weight. Thirty fully sintered and polished disk specimens (10 mm in diameter, 2 mm thickness) were prepared form Y-TZP pre-sintered blank using a precision cutting machine with diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Then, the pre-sintered specimens were sintered in the furnace (Cercon® heat furnace, Degudent GmbH, Hanau, Germany) at 1350 °C for 12 hours cycle according to the manufacturer's instructions. After sintering process, the sintered specimens were polished using polishing machine (Ecomet 3, Buehler, IL, USA) under water irrigation to obtain standardized flat surfaces. All specimens were ultrasonically cleaned for 5 minutes in 99% ethanol and distilled water to remove any surface residues and air dried prior to perform surface treatment.

**Composite Resin Disk Preparation:** The composite resin cylindrical disks were prepared in a cylindrical mold (5 mm diameter, 2 mm thickness). Composite resin (EsCom 100, Spident, Incheon, Korea) was incrementally condensed into the mold and each layer was polymerized using a light polymerizing unit (Bluephase; Ivoclar Vivadent, Schaan, Liechtenstein) for 40 seconds at a distance of 1 mm from the surface with an output power of 600 mW/cm$^2$. Thirty composite resin disks were fabricated.

**Zirconia Surface Treatments:** Zirconia disk specimens were randomly divided into 3 groups (n=10) according to the surface treatment performed:

Group C, Control group: No surface treatment was done to the surface of zirconia disk specimens for this group.

Group S, Sandblasting: Specimens in this group were sandblasted with 50 μm $Al_2O_3$ particles (Korox, Bego, Bremen, Germany) from a distance of 10 mm perpendicular to the specimen surface at a pressure of 2.5 bar for 15 second.

Group L: Laser treatment: An Er-YAG laser (AT Fidelis Er-YAG laser, Fotona, Ljubljana, Slovenia) with a wavelength of 2,940 nm was irradiated on the zirconia disk specimens using a special hand piece (R02-C, Fotona, Ljubljana, Slovenia). The Fotona non-contact R02-C hand piece was used to focus the laser beam (0.9 mm in diameter) perpendicular to the surface of zirconia disk specimen. The zirconia disk area was laser lased with water irrigation and air-cooling for 15 seconds. The laser parameters of pulse energy 400 mJ, pulse rate frequency 10 Hz, power 4 W; and MSP mode pulse width 100 μs were used in this study.

After the surface treatments were performed for each group, all samples were ultrasonically cleaned in 99 % acetone for 5 minutes and then in distilled water for another 5 minutes to remove residue particles from the surface.

**Cementation Composite Resin Disk onto Zirconia Ceramics Specimen:** After surface treatment, the zirconia ceramic specimens from each surface treatment group were bonded to composite resin disks using adhesive resin cement (Panavia F2.0/ED Primer II, Kuraray Medical Inc., Tokyo, Japan). The metal/zirconia primer (Ivoclar Vivadent, Schaan, Liechtenstein) was applied onto zirconia surface according to manufacturer recommendation and dried with a brief blast of air. The composite disk was applied with ED Primer II (Kuraray Medical Inc, Tokyo, Japan). The base paste and catalyst of resin cement (Panavia F 2.0, Kuraray Medical Inc, Tokyo, Japan) was mixed on the paper pad for 20 seconds and applied on the zirconia surface as well as the composite disk was placed on the cement. The cementation was performed under the universal testing machine (Lloyd Instruments Ltd., West Sussex, United Kingdom) at 20 newton load placed on the composite disc / resin cement / zirconia disk combination. The light-polymerizing unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) was used to cure resin adhesive cement for 2 seconds to initiate setting. Then the excess cement was removed with explorer and microbrush. Then the light-polymerization was further performed for 40 seconds. The cementation samples were stored in the distilled water at $37^0$C for 24 hours.

**Shear Bond Strength test:** The specimen was mounted in a custom made jig and submitted to shear bond strength test in a universal testing machine (Lloyd Instruments Ltd., West Sussex, United Kingdom) as shown in Fig. 1. The load was applied to the zirconia-adhesive resin interface at a constant crosshead speed of 0.5 mm/min until failure occurred. The force was concentrated on the ceramic/cement interface. The shear bond strength (σ) was calculated by dividing the maximum failure load by the circular cementation area from the following equation.

$\sigma = \frac{P}{A}$ ……………*Equation 1*

*In which P is the maximum load (Newton) required producing fracture, A is the adhesive cross-sectional area (where A =* ¶$r^2$*). The r denotes the diameter of the bonded area divided by 2, which was measured with a digital caliper.*

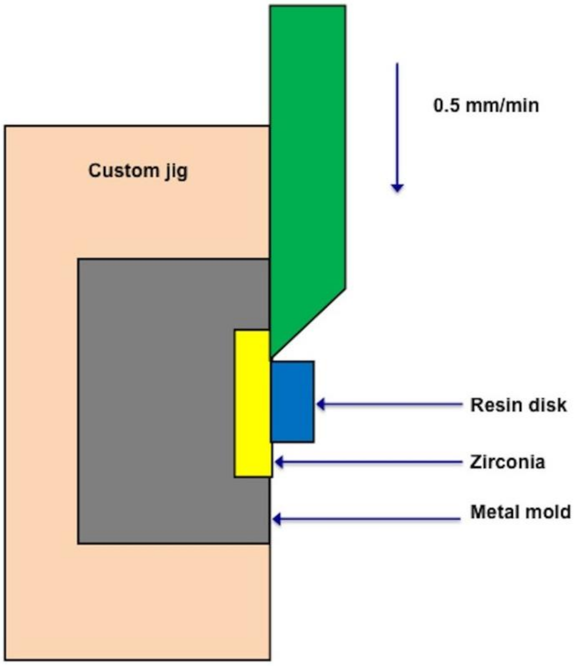

**Fig.1: indicated shear bond strength testing configuration. Specimen was mounted in custom jig and loaded at the zirconia resin interface**

**Analysis of Fracture Mode:** Following the shear bond strength test, all fractured surface of the specimens were evaluated with an optical stereomicroscope (Nikon Corp., Tokyo, Japan) at 100 magnifications to determine the mode of failure. The failure modes were evaluated and classified into one of the following types: type A, adhesive failure between zirconia ceramics and adhesive resin cement; type B, adhesive failure between zirconia ceramics and adhesive resin cement combined with cohesive failure in adhesive resin cement; and type C, cohesive failure in adhesive resin cement.

**Scanning Electron Microscope Photomicrograph Evaluation:** The zirconia ceramic specimen from each group was submitted to evaluate with scanning electron microscopy (Hitachi, Tokyo, Japan) for surface treatment analysis. The specimen surfaces were coated with gold by the sputtering technique. The scanning electron microscope photomicrographs for each surface treatment were evaluated.

**Statistical Analysis of Data:** Statistical analysis was performed using SPSS statistics system for windows (SPSS/PC, Version 17.0, SPSS, Chicago, IL, USA). In this study, parametric statistical tests were performed since data were submitted to normality test. The data of shear bond strength of different groups were analyzed by one-way analysis of variance (ANOVA). Tukey multiple comparison tests was used to determine for pairwise comparisons among groups. The statistical significance was determined at the 5% level of confidence.

## Results

The means value of shear bond strength and standard deviations (SD) of different groups were presented in Table 1 and Fig. 2. An analysis of variance (ANOVA) revealed that shear bond strength of zirconia to resin adhesive was significant effect from the method of zirconia surface treatment ($P<0.05$) as shown in Table 2. The zirconia surface treated with sandblasting exhibited the highest shear bond strength to resin adhesive compared to other groups. Post-hoc Tukey multiple comparison indicated that zirconia surface treatment with either sandblasting or Er-YAG irradiation resulted in significantly increasing shear bond strength to resin adhesive compared to non-treated zirconia surface ($p<0.05$). However, there was no significant difference in shear bond strength to resin adhesive found between the group of zirconia treated surface with Er-YAG laser and the group of zirconia treated surface with sandblasting ($p>0.05$). Upon evaluation of the mode of failure in each group showed that all of the tested samples in each group were adhesive type of failure at the interface between zirconia ceramic surface and resin adhesive (type A).

The SEM photomicrographs of zirconia ceramic surface treated with different techniques under a scanning electron microscope at ×1,000 magnifications were revealed in Fig. 3, 4 and 5. It was seen that zirconia specimen with untreated surface showed obviously flat surfaces appearance as indicated in Fig. 3. The morphology of sandblasted surface of zirconia specimen appeared to be increased in roughness and irregular pitted surface architecture as indicated in Fig. 4. The morphology of Er-YAG laser-treated zirconia specimen appeared to be increased in roughness with a scaly appearance as shown in Fig. 5. Er-YAG laser lased to zirconia surface resulted in irregularities on the surface of Y-TZP ceramic with scaly appearance under a scanning electron microscope at ×1,000 magnifications. The Er-YAG laser had ability to remove particles by micro-explosions and by vaporization in which a process called ablation at the surface of zirconia.

**Table 1: Means shear bond strength and standard deviation of zirconia bonded to resin adhesive as the effect of zirconia surface treatment**

| Groups | Surface treatment | N | Shear bond strength (Mean ± SD, MPa) |
|---|---|---|---|
| C | No treatment | 10 | 10.79 ± 1.03 |
| S | Sandblast | 10 | 14.15 ± 3.07 |
| L | Er-YAG | 10 | 14.11 ± 2.98 |

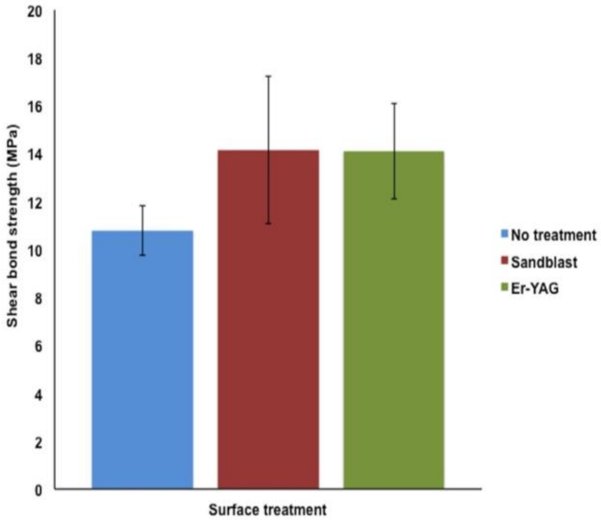

**Fig. 2: Mean shear bond strength and standard deviation of zirconia bonded to resin adhesive as the effect of zirconia surface treatment**

**Table 2: Value of one-way ANOVA test for shear bond strength**

| Source | Sum of squares | df | Mean square | F | p value |
|---|---|---|---|---|---|
| Surface treatment | 74.289 | 2 | 37.145 | 5.731 | 0.008* |
| Error | 175.001 | 27 | 6.482 | | |
| Total | 5337.547 | 30 | | | |

* The mean difference is significant at the 0.05 level

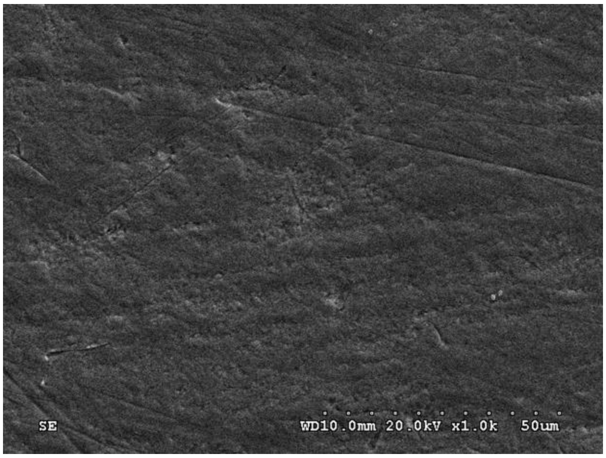

**Fig. 3: Scanning electron microscopic image of no treated zirconia surface revealed smooth surface appearance at ×1,000 magnification**

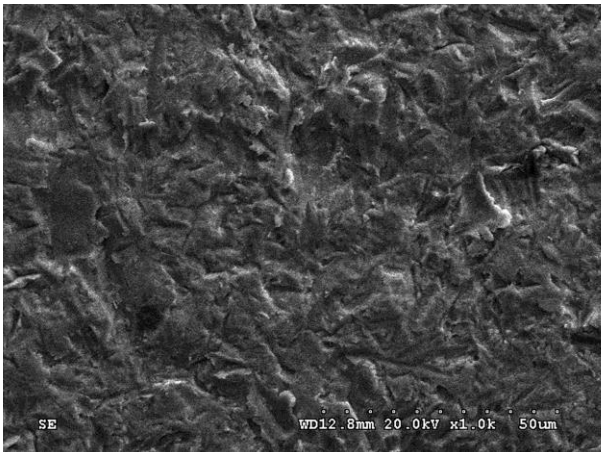

**Fig. 4: Scanning electron microscopic image of sandblasted zirconia surface revealed rough irregular pitted surface at ×1,000 magnification**

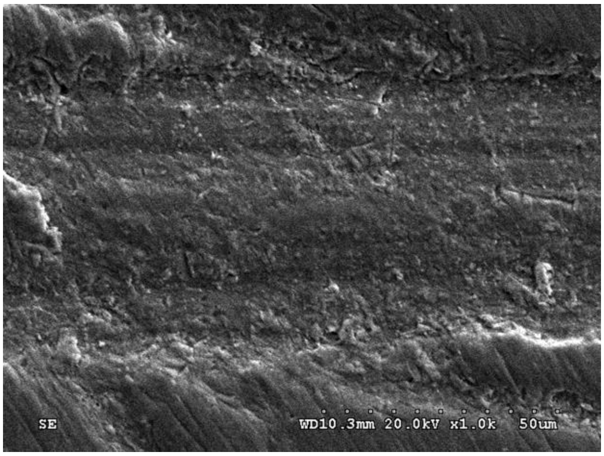

**Fig. 5: Scanning electron micrograph of Er-YAG laser irradiated zirconia surface revealed roughness and scaly appearance without microcracks at ×1,000 magnification**

## Discussion

In this study, bond strength between resin adhesive and zirconia ceramic was evaluated related to the effect of different surface treatment methods to zirconia surfaces. The treatment of Y-TZP ceramic surfaces affected shear bond strength of resin adhesive to zirconia ceramics. The surface treatment of zirconia by either sandblast or Er-YAG laser resulted in increasing surface irregularities. Upon laser technology, Er-YAG laser beam irradiation is used in various dental procedures, including roughening the surface of ceramics. In the present study, the effects of Er-YAG on surface treatment of zirconia upon shear bond strength of resin cement to zirconia ceramic were evaluated and compared to untreated surface and sandblasted surface. The results clearly showed that surface preparation of zirconia ceramic increased the shear bond strength of resin adhesive to zirconia surface ($p < 0.05$). Therefore, the null hypothesis of the study was refused. The result of this study indicated that surface treatment of zirconia either by sandblasting or Er-YAG laser treatment enhanced shear bond strength between resin adhesive cement to zirconia. In consistent with the results of the this study, other previous studies have also shown that Er-YAG lasers can increase the shear bond strength of resin cement to zirconia ceramic.(21-24)

Zirconia ceramic can absorb the energy from the Er-YAG laser beam. After absorption of laser energy, a process called heat induction produces shell-like ruptures on the zirconia ceramic surface that can provide a micro-mechanical retention between the resin adhesive and zirconia ceramic surface.(23) The micro-morphologic evaluation of the surface of zirconia ceramics has shown that the Er-YAG laser produces a rough surface with a scaly appearance on zirconia surface.(21,23) The micrograph of the zirconia disc surface in this study also showed scaly appearance as shown in Fig. 5. As seen on SEM photomicrograph, the Er-YAG laser increased surface roughness without causing micro-cracks on the zirconia ceramic surface and significantly increased the resin cement bond strength ($p < 0.05$).

In contrast to some studies have shown that irradiation of zirconia ceramic surfaces with Er-YAG lasers does not result in an increase in shear bond strength of resin cement to pretreatment surfaces.(25-27) This discrepancy in results may be attributed to differences in surface laser pretreatment methods. Absorption of the laser beam energy by the material surface is the most important interaction (thermo-mechanical effect) between the laser and the material. Increase in output energy and pulse rate of the laser beam results in increasing the energy density and thermal effects on the surface. In pulsed lasers such as Er-YAG laser, changing the pulse duration changes the peak power and its effects on the material surface.

However, in the present study the new technology of Er-YAG laser with beam parameters of pulse energy 400 mJ, pulse rate frequency 10 Hz, power 4 W, and MSP mode pulse width 100 µs for 15 seconds was used. This low pulse duration increased the laser power peak, thus, increasing the effect of the laser energy on the surface. In addition the laser energy was capable of enhance the surface roughness without micro-crack formation on the zirconia ceramic surface. The rough surface was clearly seen in SEM micrographs Fig. 5. Scaly irregularities on the zirconia surface increase micromechanical retention and improve shear bond strength of resin cement to the zirconia surface. Pretreatment of zirconia surface with Er-YAG laser resulted in a greater increase in bond strength values compared to the non treated surface ($p < 0.05$) in the present study, which can be attributed to the extent and type of the surface irregularities produced on the zirconia surface. In addition there were no micro-cracks were observed on the surfaces exposed to Er-YAG laser beam. This indicated the capability of Er-YAG laser in treatment surface of zirconia for promoting bond strength to resin adhesive. Roughening surface of Y-TZP ceramic is important for enhancing bond strength for adhesive resin cement to zirconia surface.

Upon evaluation of fracture modes in the present study showed that all of the failure specimens were adhesive type of failure. This indicated the validity in comparison the shear bond strength test in this study. The increasing in shear bond strength upon Er-YAG treated surface of zirconia indicated the durability of the bond between zirconia and resin adhesive which is more important for the clinical success of restorations. In addition, sandblasting technique is traditionally old technique used for surface preparation that induce phase transformation in zirconia leads to crack propagation. Therefore, Er-YAG laser should be introduced and recommended for the treatment surface of zirconia in order to enhance the surface roughness for bonding to resin cement without creating crack propagation on zirconia surfaces.

## Conclusion

This study indicated that surface treatment of zirconia ceramic by Er-YAG laser treatment resulted in surface roughness appropriated for bonding with resin adhesive and enhance bond strength between resin adhesive to zirconia ceramic surface. Therefore, surface treatment of zirconia with Er-YAG laser before cementation with resin adhesive is strongly recommended prior to clinical cementation with resin adhesive in fixed prosthodontic practices.

**Conflict of Interest:** None

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