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Bond strength of short-pulsed laser-irradiated zirconia to veneer ceramic

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Objective: The aim was to evaluate the effect of 1064 nm Yb-doped fiber-based nanosecond pulsed laser on surface roughness and bond strength between veneer ceramic and zirconia. **Material and methods:** Zirconia discs were divided into three groups: sandblasted (SB), laser irradiated (YL), and control ($n = 12$). YL group was treated with ytterbium laser with the setting of 85 W/25 kHz. Sandblasting was done using 50 μm Al_2O_3 particles from a distance of 10 mm for 20 s under 3.5 atm. No surface treatment was applied to the control group. The surface roughness values and SEM images of the groups were obtained. X-ray diffraction analysis was applied to a spare sample of each group to determine the monoclinic phase ratio. The samples were subjected to shear bond strength (SBS) test with a cross-head speed of 1 mm/min after being veneered. The fracture modes were evaluated. One-way analysis of variance and Tukey's HSD tests were used for statistical analysis. **Results:** The YL group had higher surface roughness than the control ($p \leq 0.0001$) and the SB group ($p = 0.007$) with a mean value of 2.90 μm . The SEM images of the groups supported this result, but formation of the microcracks was more intense for the YL group. The monoclinic phase ratio was highest for the SB group. However, the differences of SBS between SB and YL groups were not statistically significant. Mostly the combined failure of samples was observed. **Conclusions:** Ytterbium laser treatment increased the surface roughness of zirconia, but the SBS was not higher than sandblasting. Surface roughness results did not correlate with the SBS results.

Keywords: shear bond strength; surface roughness; ytterbium laser; zirconia

1. Introduction

Zirconia ceramics for the application of fixed partial dentures has expanded rapidly in the recent years. This application is popular due to its high flexural strength, chemical stability, biocompatibility, and being esthetical alternative to metal-ceramic restorations. However, recent clinical trials have shown that the most common failure in zirconia-supported ceramic restorations is the fracture occurring in veneering ceramic (chipping).[1,2]

Many factors, such as veneering surface of the framework and the mechanical retention of this surface, compatibility of thermal expansion coefficients, volumetric shrinkage of the veneering ceramic, viscosity, and wettability, can affect the bond strength between a zirconia framework and the veneering ceramic.[3,4] Although these

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factors have been identified, their exact mechanisms have not yet been defined.[5] Surface treatment methods, such as air abrasion with aluminum oxide (Al_2O_3) [6,7] or silica coating with silica-modified Al_2O_3 [6] particles, and chemical modification of the zirconia surface by use of liners,[5] are used on Y-TZP restorations. There are also limited number of studies that have evaluated laser applications of different kinds.[8–10]

Laser usage in dentistry has already become widespread. Lasers used in dental application vary extremely for different operations and purposes. Diode lasers are only suitable for cutting soft tissues. Solid state lasers, like Nd:Yag, are suitable for soft tissues that provide advantage for dental applications, but they are expensive.[11] Nd:Yag laser, much like Yb-doped fiber lasers is ranged at 1064 nm. By increasing the pulse energy, these types of lasers could become an alternative option for dental applications as they are significantly less expensive than their solid-state laser counterparts. Furthermore, fiber lasers have high optical quality, compact size, extended lifetime, and flexible mode of operation.[12–14]

The studies that have evaluated laser application to improve the bond between the zirconia framework and the veneer ceramic have utilized only CO_2 , Nd:YAG, and Er:YAG lasers which are frequently used for dental applications.[8–10] But their use to enhance the bond strength between zirconia and the veneer ceramic did not show any significant increase when compared with the sandblasting method, which can be accepted as conventional method for surface roughening; improving the bond strength between zirconia and veneer ceramic can be achieved by increasing the surface roughness. In addition to these dental lasers, ytterbium laser is a quite powerful tool mainly used in the industry. It has not been used in dentistry and so has not also been evaluated for this purpose.

The aim of the present study was to evaluate the effect of 1064 nm Yb-doped fiber-based nanosecond pulsed ytterbium laser application on zirconia framework and the bond strength between veneering ceramic and zirconia core material. The null hypothesis is that the application of ytterbium laser to the zirconia framework does not affect the bond strength between the zirconia framework and the veneer ceramic.

2. Material and methods

2.1. Preparation of samples

Partially stabilized Yttrium zirconia dioxide blocks (ICE Zirkon, Zirkonzahn, South Tyrol, Italy) were cut into discs by means of a low-speed diamond saw (Struers Ltd, Lanarkshire, UK). In total, 36 discs, $15 \times 12 \times 1.6$ mm, were used as test samples. Samples were sintered in a sintering oven (Zirkonzahn) according to the manufacturer's instructions with a heating time of approximately 3 h, standby time of 2 h, and a cooling time of approximately 18 min. After being sintered, the samples were ground by 800, 1200, and 2400 grit silicon-carbide sandpaper.

The samples were divided into three groups according to the surface treatments applied: the control group, the sandblasting group (SB), and the laser-irradiated group (YL). No surface treatment was applied to the control group. Sandblasting was performed with 50 μm aluminum oxide particles (Al_2O_3) from a distance of 10 mm for 20 s under 3.5 atm pressure in order to increase surface roughness and enhance the bond strength. For preparation of the YL group, the laser beam was directed over the zirconia oxide disc surface in a noncontact mode at a working distance of 17.8 mm. The surfaces were irradiated with 1064 nm Yb-doped fiber-based nanosecond pulsed YL (Vision, Germany) with the setting of 85 W/25 kHz. The speed of the device was

600 mm/s. The zirconia oxide disc surfaces were scanned vertically and horizontally on the fiber laser machine. All samples were then cleaned in an ultrasonic cleaner (Quantrex 90, L & R Ultrasonics, Kearny, NJ, USA) for 10 min, rinsed, and air dried.

2.2. Surface roughness measurement test

After the surface treatments, surface roughness measurements were performed for each specimen using a surface texture measuring instrument (Mitutoyo SurfTest 402 Analyzer Series 178; Mitutoyo Corporation, Minatoku, Japan). Ten measurements at different locations were recorded for each specimen and the average of these measurements was used to obtain the R_a (arithmetical mean roughness) value of each specimen.

2.3. Procedure of veneering

The translucent porcelain (CZR, Noritake Co., Kiza, Nagoya, Japan) was used to veneer zirconia discs. A metal template, with a hole corresponding to the center of the zirconia disc, was used to standardize the veneering procedure. The metal template enabled the application of veneering ceramic with a diameter of 3.5 mm and thickness of 3 mm. The ceramic veneer was then sintered according to the manufacturer's instructions: with preheat of 600 °C, holding time of 4 min, final temperature of 930 °C, heating rate of 45 °C, and drying time of 1 min.

2.4. Shear bond strength test

Zirconia discs with ceramic veneers were placed in the Universal Testing Machine (TSTM 02500 Elista Ltd., Istanbul, Turkey) and shear bond strength (SBS) values of the samples were evaluated at a speed of 1 mm/min.

2.5. X-ray diffraction (XRD) analysis

One spare specimen from each group was fabricated using the same method described above for the XRD analysis. The crystalline phases of the specimens were determined X-ray diffractometry using Cu- α radiation in the 2θ range of 20–40° with a step width of 0.02° and scan speed of 1°/min. The relative amount of monoclinic zirconia (X_m) was calculated using the Garvie and Nicholson method [15]:

$$X_m = [I_m(-111) + I_m(111)] / [I_m(-111) + I_m(111) + I_t(111)]$$

I_t and I_m represent the integrated intensity (area under the peaks) of the tetragonal (1 0 1)_t and monoclinic (1 1 $\bar{1}$)_m and (1 1 1)_m peaks. The monoclinic volume content (V_m) was then obtained using the equation proposed by Toraya et al. [16]:

$$V_m = 1,311 X_m / (1 + 0,311 X_m)$$

2.6. SEM analysis

A sample from each group was selected randomly for the surface analysis and gold coated. The samples were examined with a field emission scanning electron microscope (JSM 6300F, Joel Ltd., Japan) for the evaluation of the surface topography at magnifications of 500×, 1000×, and 5000×.

2.7. Fracture analysis

After the SBS test, fracture modes were examined by optical microscopy (Olympus SZ4045 TRPT) at magnifications of 10× and 20× to determine the types of failure. Photographs of the surfaces were also taken and fracture types were classified as adhesive (separation of zirconia and veneer ceramic), cohesive (failure in zirconia or veneer ceramic), or combined (including both cohesive and adhesive).

2.8. Statistical analysis

The 'SPSS 15.0 for Windows' software performed statistical analysis of the data. Statistical analysis was performed applying one-way analysis of variance (ANOVA) followed by *post hoc* Tukey's HSD test at a significance level set at $p < 0.05$.

3. Results

3.1. Surface roughness evaluation

Surface roughness values were shown in Table 1. YL group showed the highest surface roughness values (2.9 ± 0.69), while the control group showed the lowest surface roughness values (0.46 ± 0.10) ($p < 0.05$).

3.2. SBS test

SBS of the samples according to different surface treatments are given in Figure 1. The highest SBS value estimated was 17.14 MPa (SB group) and the lowest SBS value estimated was 13.74 MPa (control group). Both YL and SB groups showed higher SBS values than the control group, but there were not any statistically significant differences ($p > 0.05$).

3.3. XRD analysis

XRD analysis revealed changes from tetragonal to monoclinic phase in zirconia discs after surface treatments (Table 2). Figure 2 shows XRD traces for all the specimens. A decrease in the monoclinic volume was observed in the YL group. An increase in monoclinic volume was observed in SB group. Some small peaks were present in SB group that were difficult to clearly identify.

3.4. SEM analysis

The SEM analyses of the specimen surfaces are shown in Figure 3. The SB group's surface exhibited a surface texture consisting of micro-mechanical irregularities (Figure 3(b)). The SEM image revealed that Al_2O_3 , clearly, created rougher surfaces

Table 1. Surface roughness values (R_a) of the groups.

Groups	Mean	SD (\pm)	Min	Max
Control	0.41	0.10	0.28	0.57
Sandblasted	0.91	0.34	0.44	1.8
Laser irradiated	2.90	0.69	1.77	3.86

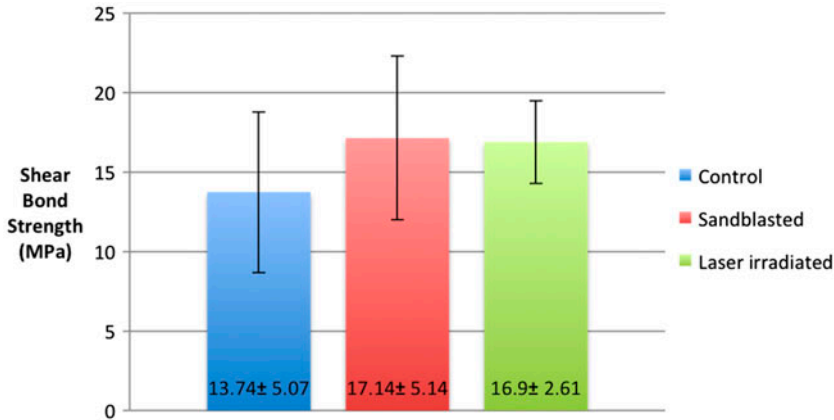


Figure 1. SBS values of groups and their standard deviations.

Table 2. Monoclinic volume fraction of the groups.

Groups	Monoclinic volume content (%)
Control	7.4
Sandblasted	15
Laser irradiated	6.7

compared to the untreated specimen (Figure 3(a) and (b)). In YL group, the zirconia oxide disc surfaces were rougher than that of all groups. It was observed that the laser irradiation created a rough surface appearance with plaque-like scaly appearance (Figure 3(c)).

3.5. Evaluation of fracture types

Fracture types between zirconia framework and veneering ceramic were observed mostly as combined. There were also a few adhesive failures, while no cohesive failure in the zirconia or veneer ceramic was observed (Table 3).

4. Discussion

Bond strength between zirconia and veneer ceramic is an important factor for success of the restoration. Clinical trials reported that veneer ceramic cracks occur up to 30%, while framework cracks stay in 1%. [1,17–19] To overcome the problem of chipping and cracking of the veneer ceramic, several studies have investigated various factors on the bonding of zirconia and the veneer ceramic. [5,8,10,20–23]

Dental laser application on zirconia is one of the surface treatment options evaluated previously to improve surface roughness and bond strength between zirconia and veneer ceramic. [8–10] The current study evaluated the effect of an industrial laser on zirconia surface. Results show that surface roughness was affected by laser irradiation (Table 1). Surface roughness is indicated as an important factor to increase the bonded area with the veneer ceramic. [24] The current study showed that laser irradiation of

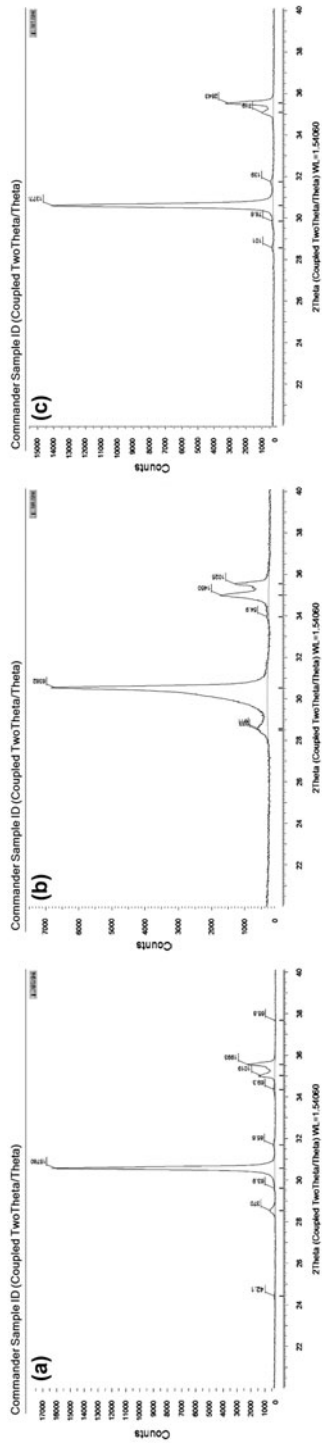


Figure 2. XRD patterns (a) control group, (b) SB group, and (c) YL group.

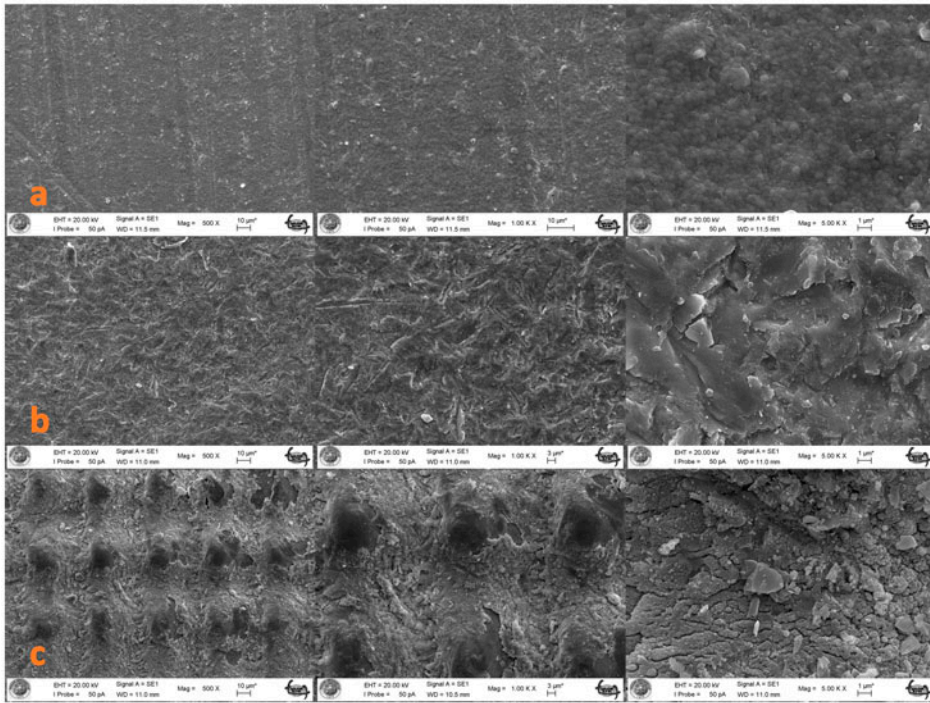


Figure 3. SEM images at 500, 1000, and 5000 K magnification (a) control group, (b) SB group, and (c) YL group.

Table 3. Fracture types of the groups.

Groups	Adhesive	Cohesive	Combined
Control	1	0	11
Sandblasted	2	0	10
Laser irradiated	2	0	10

zirconia surface increases the surface roughness significantly compared to the control group and the SB group. The SBS values were higher than the control group, but did not vary significantly from the SB group as it did in surface roughness. So, the null hypothesis claiming that ytterbium laser application would not affect the SBS was rejected. This result may depend on the heat produced on the surface of zirconia during laser irradiation.[25] The previous studies have shown that temperature rise more than 350 °C can cause M→T reverse phase transformation of zirconia.[26–28] The XRD analyses of the study have shown that laser irradiation causes a decrease in the volumetric ratio of the monoclinic phase on the zirconia surface. So, a M→T reverse phase transformation is assumed. This situation can be particularly important in terms of thermal coefficient differences and fracture toughness.

Tetragonal phase increase on zirconia surface can cause alterations in the bond of zirconia and the veneering ceramic. Minimum mismatch between the veneering ceramic and the core material is recommended for metal fused to ceramic restorations.[29] This

recommendation is also valid for bilayered ceramic restorations. Aboushelib et al. [2] have shown if a veneering ceramic has a coefficient of thermal expansion higher than that of zirconia core, delamination and massive microcracks could occur in the veneering ceramic. The coefficient of thermal expansion of monoclinic zirconia ($7.5 \times 10^{-6}/\text{K}$) is lower than that of tetragonal zirconia ($10.8 \times 10^{-6}/\text{K}$). [3,30] Although the main zirconia phase was tetragonal in both surface treatments evaluated in the current study, the monoclinic phase ratio of the SB group was higher on the veneer-facing surface of zirconia. So, a probable decrease in the SBS values could be expected due to the mismatch in the coefficient of thermal expansion. But the extent of this decrease is the topic of another study. The SBS values of the SB and YL groups were not statistically significant in our study, but there are other factors affecting the SBS and this result cannot be attributed only to the coefficient of thermal expansion mismatch.

Internal volume increase caused by T→M phase transformation reduces the relaxed surface of zirconia and forms a compressive layer. But with the M→T reverse phase transformation of laser applied samples of the current study, the compression on the zirconia surface has probably decreased, thus capability to stop crack propagation is diminished. Consequently, the fracture toughness of zirconia is probably decreased. When the SEM analysis of the YL group is considered, the need for stopping crack propagation is higher than the other groups because more microcracks exist on the YL surface (Figure 3). The SBS of the veneering ceramic adjacent to the cracked and weak interface is probably affected and this may be the reason for the lower SBS value of the YL group compared to the SB group, although the surface roughness of the YL group is significantly higher. [26,31–33]

The effect of the M→T reverse phase transformation on the zirconia–veneering ceramic surface should also be considered from the aspect of the total survival of the restoration. It has been stated that majority of the fractures start at the cracks at the core-veneering interface in the bilayered all-ceramic restorations and the interface meets high tensile stress. Also, the Weibull modulus for the interface was found to be quite lower than the free surface. [33] The increased number of microcracks in the YL group is probably decreasing the fracture toughness, especially when there is not a compressive layer in the zirconia surface. Further excess T→M phase transformation occurring by aging would not be present after the veneering procedure and the structural integrity of zirconia surface facing veneering ceramic would not be affected because contact with the aging promoting oral environment is not valid after the veneering process.

In the present study, the fracture surfaces of the test samples were also observed and it was found that they consisted of combined and adhesive types. The initiation of the fracture was observed between the veneering ceramic and zirconia framework. The bond strength between zirconia framework and veneering ceramic is weaker than the fracture resistance within the veneering ceramic or zirconia, indicating that the bond of zirconia and the veneering ceramic is the weakest link of the structure.

There are several limitations for the current *in vitro* study. Only one kind of veneering ceramic and zirconia framework material was used and only one laser setting was applied. Also, aging of laser applied zirconia and fracture strength after the laser application were not evaluated.

Future studies can evaluate the effects of repeated T→M phase transformation on SBS between zirconia and veneering ceramic by applying the laser treatment prior to sandblasting of zirconia in order to cause T→M phase transformation and form a compressive layer on the surface. Also, the effect of surface treatments of the surface facing the veneering ceramic on the fracture toughness should be explored extensively.

5. Conclusion

The results of this study demonstrate that:

- (1) Ytterbium laser treatment on the zirconia increases the surface roughness.
- (2) Both laser irradiation and sandblasting applied groups showed higher SBS values than the control group, but the difference between them was insignificant. So, it can be stated that evaluation of only the surface roughness is not enough to make assumptions about the SBS.

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