

Evaluation of Effect of Zirconia Surface Treatment with CO₂ and Nd:YAG Lasers on Shear Bond Strength between Zirconia Frameworks and Porcelain Veneers

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ABSTRACT

Aim: The purpose of this study was to evaluate the effect of zirconia surface treatment with CO₂ and Nd:YAG laser on shear bond strength (SBS) between the zirconia framework and porcelain veneering.

Materials and methods: In this *in vitro* study, zirconia blocks were converted to 50 cubes and were divided randomly into 5 groups. After sintering (S), porcelain was applied in the control group. The surface treatment of the second to fifth groups included CO₂ laser + (S), (S + CO₂), Nd:YAG laser + (S), and (S + Nd), respectively. The SBS test was done, and data were analyzed by SPSS16 software. One sample was randomly chosen from each group and the type of failure was examined under scanning electron microscope (SEM). To compare the pairs of means, the least significant difference test was used and the determined significance level was 5% ($p < 0.05$).

Results: The SBS of S + Nd group was significantly higher than the other ones, except for S + CO₂ group. The least amount of SBS belonged to CO₂ + S and the highest to S + Nd group. There were no significant differences between the other groups.

Conclusion: The bond strength of veneering porcelain to zirconia can be altered by surface treatments. It can also be affected by the type and sequence of laser and sintering application. The effect of Nd:YAG laser on the surface of zirconia, in order to create roughness to increase SBS, is better than that of CO₂ laser.

Clinical significance: Surface treatment of zirconia by certain types of lasers reduces the chipping of the ceramic veneer and increases the success rate of all-ceramic restorations.

Keywords: CO₂ laser, Nd:YAG laser, Shear bond strength, Sintering, Zirconia.

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INTRODUCTION

Esthetics play an important role in patients' satisfaction in the field of dentistry, and apart from tissue adaptation, nonmetallic ceramic restorations provide a much better appearance than their metal-ceramic counterparts. Thus, there is still an on-going search for an ideal all-ceramic restoration, and in the meanwhile, following features are looked for: the physical characteristics, strength, edge synchronization, and a perfect appearance for the anterior and posterior teeth. In many cases, all-ceramic prosthesis can potentially create a more pleasant look than a metal-ceramic restoration. In the latter, the metal infrastructure creates an opaque appearance, and the metal margins are usually observable. Its other drawbacks include risk for a metal allergy, breaking of the bond, and porcelain's color change. Reinforced all-ceramic prosthesis include a high-strength core (such as zirconia), which is covered by porcelain.¹⁻³

Zirconia is a crystalline oxide of zirconium, and its mechanical properties are quite similar to the metals and its color to that of the teeth. In 1975, Garvie showed that it has great mechanical characteristics and called it steel ceramic.⁴ Nowadays, zirconium has been mixed with computer-aided design (CAD)/computer-aided manufacturing (CAM) computer systems and has made a revolution in dentistry. For making a zirconia core, it is necessary to use a proper CAD/CAM system, which can work on zirconia. In comparison with other all-ceramic restorations, their frameworks are the most reliable.⁵ Having said that, their weakest point is chipping or cracking of the veneer. Zirconia restorations also have

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the mechanical properties for being used in the posterior areas of the mouth.⁶ For reaching a strong bonding, the mechanical properties of the core and porcelain veneer must be adaptable with each other. Having a strong bonding is necessary for making sure of the structural unity of the restoration under functional forces, and also for avoiding delamination and chipping of the veneer.² It has been reported that all-ceramic restorations, which had the

veneer ceramic attached to zirconia frame, showed a 13% chipping of the ceramic in 3 years and 15.2% in 5 years while breaking of metal–ceramics were 8–10% in 10 years. As a result, for increasing their long-term clinical success rate, it is important to increase the bonding strength between ceramic veneer and frameworks in zirconia restorations. Stress distribution in a two-phase structure is more complicated than a single-phase one, and since the interface of core and veneer is one of the weakest areas in veneer restorations, thus more factors must be considered in layered restorations.² Bonding mechanism includes chemical bonding, mechanical adaptation, and pressure stress due to difference in heat expansion coefficient between tetragonal zirconia polycrystal and ceramic veneer. However, it must be mentioned that the exact bonding mechanism between zirconia and ceramic veneer has not been fully understood yet. The strength of the mentioned bonding is affected by many factors;^{7,8} most important of which are surface finishing in core, chemical bonding strength, mechanical interlocking, type and rate of interface deficiencies, the use of porcelain liner, and wetting features along with pressure stress rate in veneer layer due to difference in heat expansion rate between zirconia and porcelain veneer.

Various methods have been introduced for improving the bond between porcelain veneer and zirconia, some of which are sandblasting, silica coating, liner, abrasion by diamond drills, and thermal preparation of zirconia's surface.^{9–12} The methods that cause roughness in zirconia surface can have devastating effects on its mechanical characteristics, such as flexural strength and durability.^{13,14}

Due to lack of silica and glass phase in zirconia ceramics, performing acid etching for creating a rough surface by using HF acid does not make a meaningful difference for improving the bond strength.¹⁵ Air abrasion is usually used for creating a rough topography on the surface of zirconia framework in order to increase the contact surface for bond and for increasing the surface energy and also wetting characteristics.^{16,17} But it must be mentioned that this method leaves tracks and other deficiencies in zirconia that would be the starting point of broken frames and thus endangering its mechanical features and longevity.¹⁸

Recently, easy-to-use and efficient surface treatment methods have been proposed using laser radiation. The laser beams that are used in dentistry depending on the wavelength, power, and the type of target tissue can either make stimulus, rupture or extracting.¹⁹ Although Maiman used the ruby laser on an extracted tooth for the first time in 1960, its actual use in dentistry started many years later.²⁰ The use of high-strength laser started in 1989 with pulsed Nd:YAG by Terry Myers.²¹ From 1990 onward, the lasers used in surgery, with little modifications, were used in dentistry as well; examples of which include argon, CO₂, Nd:YAG, and Erbium. Recently, certain types of lasers, namely, CO₂, Nd:YAG, and Er:YAG have been used for creating surface roughness in ceramics zirconia.^{17,22,23} Using radiation of CO₂ laser is an ideal preparation method for porcelain materials since its beams are almost completely absorbed by the porcelain due to its specific wavelength.

Previous studies have employed different lasers such as CO₂, Nd:YAG, and Er:YAG for the surface treatment of zirconia and reported varying degrees of success.^{24,25} So, this study was done to assess the effects of using Nd:YAG and CO₂ lasers and also the role of heat treatment of zirconia's surface for the bond between zirconia and ceramic veneer.

MATERIALS AND METHODS

Study Design

This *in vitro* research was carried out in the Dental Sciences Research Center and the Department of Prosthodontics, Tehran, Iran.

Sample Size Calculation

In this *in vitro* research, first, by using a diamond disk, zirconia blocks (YZ Cubes, Vita Zahnfabrik, badsäckingen, Germany) were cut into 50 square cubes, which measured 13.5 millimeters at each side. Those 50 cubes were randomly divided into 5 equal groups ($n = 10$). In each group, other than the 10 original cubes, 1 extra sample was added for evaluation under the electron microscope.

Preparation of Zirconia Samples

At the first step, the surface of all zirconia samples was polished under water using a silicon carbide-coated abrasive paper with a grit size of 1200 (Struers A/S, Rodovre, Denmark), and then they were cleaned using 70% ethanol alcohol and distilled water in the ultrasonic machine for 5 minutes.

Study Groups

The samples of the first group (C = control), which did not receive any surface treatment (traditional method) underwent sintering for 2 hours in a furnace at 1510 °C, based on the manufacturer's instructions. Due to a 20% shrinkage of zirconia, all the 55 samples shrank to square cubes with dimensions of 10 mm on each side. For having an even layer of porcelain on the sintered blocks, an index made of condensation silicone with a consistent Putty (Spidex, Coltene AG, Altstätten, Switzerland) was made and placed 2 mm higher than the edge of the sintered block of zirconia. And while the porcelain was being made, the zirconia block was placed inside the index and VM9 dentin porcelain with coloring code 2 M2 (Vita, Bad Säckingen, Germany) was added up to the edge of the index. Based on the manufacturing guideline, after adding the porcelain to the first group (C), it was baked in the furnace. In order to recreate the clinical conditions and for fixing the shortcomings of the first bake (bisque stage), all samples underwent a second baking. Based on the type of the laser, sequence of laser procedures, and sintering, the rest of the samples were divided as following: in the second group, first, laser was radiated on the surface of the samples using CO₂ (Smart US 20D, Deka, Florence, Italy) and afterward, they underwent sintering (CO₂ + S). Similarly, CO₂ and sintering were used in the third group, only reversing the sequence (S + CO₂). In the fourth group, scanning of the surface was done using Nd:YAG laser (Fidelis, Fotona, Slovenia) and after that they underwent sintering (Nd + S). The same laser was used in the fifth group, and, here again, the only difference was the sequence of the procedure (S + Nd).

Laser and Sintering Procedure

With a distance of 1 cm to the sample's surface, CO₂ laser was radiated with a power of 3 watts and in the form of a continuous wave and according to the manufacturer's guidelines. The Nd:YAG laser in the research had a power of 2 watts and energy of 100 mJ per pulse, in the form of LP (320 microseconds) with a frequency of 10 Hz and with a distance of 1 mm from the surface of the samples. The scanning speed for all four groups were 2 mm per second. The beam diameter in CO₂ was 0.5 mm, and 0.3 mm in Nd:YAG. Based on the scanning speed and also the area of the samples before and after sintering, the timing for the second (CO + S) and fourth group (Nd + S) was 60 seconds, and the third (S + CO) and fifth group (S +

Nd) was 45 seconds. After the application of the laser in the second and fourth groups, the sintering was performed in the furnace for 2 hours at 1510°C.

One extra sample, before application of porcelain veneer, was randomly chosen from each group in order to be analyzed under the electronic microscope (TESCAN, USA) for the effects of sintering and laser. Due to gold coating of the samples, the SBS test was not performed on them.

Finally, the adding of the porcelain for the fourth group was done similar to the control group.

Shear Bond Strength Test

Samples had to be mounted in the jig for performing the SBS test. For keeping the samples completely in place, during applying the force, and before the bond failure, they were tightly encased by three screws. The samples were put in their proper place in the universal testing machine (Zwick, Ulm, Germany, 2010), and the vertical arm of the chisel was set in a way that the force would precisely be applied to the veneer's core interface. In the next stage, the vertical arm, along with the attached chisel, started to do the forcing. The head speedcross was 0.5 mm per minute and it continued to the failure point. The applied force (in Newton) was measured and recorded and was changed into SBS based on the following formula:

$$\text{SBS (MPa)} = \text{Load (N)} / \text{Area (mm}^2\text{)}$$

In the analysis of the sample failure, after testing SBS one sample was randomly chosen from each group and the type of failure was analyzed under an SEM microscope.

Statistical Analysis

The obtained data were subjected to statistical analysis using statistical package for social sciences (SPSS) software, version 20. Data presented as mean, standard deviation, standard error, minimum, and maximum when appropriate. The variance analysis was used to compare the means between tested groups while

the LSD method was used for pairwise comparison of means. The significance level was set at $p < 0.05$

RESULTS

Based on the findings in Table 1, the SBS of zirconia for study groups including (C), (CO + S), (S + CO), (Nd + S), and (S + Nd) were 49.8, 47.3, 65.7, 49.7, and 70.8 MPa, respectively.

The results of Table 2 showed a significant difference between the preparation methods (p -value = 0.039) based on the variance analysis. Due to significant difference of SBS between zirconia and porcelain layer in different methods, the follow-up tests were performed by multiple comparisons using the LSD.

Based on the results of Table 3, intergroup comparison showed a significant difference between the SBS using Nd:YAG after sintering with the control group at the meaningful level of 5% (p -value = 0.027). Also, there was a significant difference between the SBS using CO₂ laser before sintering with Nd:YAG and after sintering at the meaningful level of 5% (p -value = 0.014). The SBS using Nd:YAG before sintering in comparison with Nd:YAG after sintering was at the meaningful level of 5% (p -value = 0.026). The SBS of S + Nd group was significantly more than other groups except S + CO₂. The lowest amount of SBS belonged to CO₂ + S and the highest was to S + Nd group. There were no significant differences between other groups.

Regarding Figures 1 to 3, the images were obtained through an SEM, after surface treatment, and before application of porcelain, and finally, photos of post-failure of the samples are shown as well.

DISCUSSION

One of the weak points of using zirconia in restorations is the interface between core and veneer. Since the natural strength of porcelain veneer is less than core and this can cause the failure points in all-ceramic restorations, so a strong bond between ceramic veneer and framework is necessary for making sure of the structural unity of the restoration under functional forces, and also

Table 1: The descriptive data on the SBS of zirconia

Group	Number	Mean	Standard deviation	Standard error	Minimum	Maximum
Control	10	49.80	23.799	7.526	19	89
CO ₂ laser before sintering	10	47.30	18.276	5.779	10	72
CO ₂ laser after sintering	10	65.70	11.586	3.664	44	82
Nd:YAG laser before sintering	10	49.70	14.229	4.500	27	73
Nd:YAG laser after sintering	10	70.80	29.389	9.294	32	117
Total	50	56.66	21.925	3.101	10	117

Table 2: Variance analysis results

Source of variance	Mean squared errors	Degrees of freedom	Mean squares	F-statistics	p-value
Intergroup (preparation methods)	4647.720	4	1161.930	2.765	0.039
Intragroup	18907.500	45	420.167		
Total	23555.220	49			

Table 3: The results of multiple comparisons in the form of LSD

<i>I-group</i>	<i>J-group</i>	<i>Mean difference</i>	<i>p-value</i>
Control	CO ₂ laser before sintering	2.500	0.786
	CO ₂ laser after sintering	-15.900	0.090
	Nd:YAG laser before sintering	0.100	0.991
	Nd:YAG laser after sintering	-21.000	0.027*
CO ₂ laser before sintering	CO ₂ laser after sintering	-18.400	0.051
	Nd:YAG laser before sintering	-2.400	0.795
	Nd:YAG laser after sintering	-23.500	0.014*
CO ₂ laser after sintering	Nd:YAG laser before sintering	16.0	0.088
	Nd:YAG laser after sintering	-5.100	0.581
Nd:YAG laser before sintering	Nd:YAG laser after sintering	-21.100	0.026*

*The level of meaningfulness: 5%.

for avoiding delamination and chipping of the veneer.²⁶ This *in vitro* study was done to evaluate the effect of zirconia surface treatment with different lasers. The SBS test was evaluated for the control group (only sintering) and other groups including (CO₂ + S), (S + CO₂), (Nd + S), and (S + N). The result of the SBS test was analyzed by SPSS16 software. The variance analysis and LSD method were used to compare the means between tested groups and pairwise comparison of means, respectively.

The SBS is the most appropriate examination for porcelain bonds.²⁷ Since the shear stress is the most common type of stress responsible for breaking of the bonding in clinical conditions and SBS is easily and quickly deployable, this was the test of choice in this study as well.²⁸ The important point about stabilizing the samples was a precise parallelism of interface with the surface of mold since any sloping of the mounted sample's interface, while applying the force, would result in an incorrect measurement of the SBS. For doing so, a different coloring than zirconia core was used for porcelain veneers in the treatment stage so that the prepared surface would be aligned with the jig and parallel to chisel while its sharp edge in the interface would be perpendicular to the horizon.

Based on the results, a significant difference was between the SBS using Nd:YAG after sintering with control group (p -value = 0.027). Also, there was a similar difference between the SBS using CO₂ laser before sintering with Nd:YAG and after sintering (p -value = 0.014). The SBS using Nd:YAG before sintering in comparison with Nd:YAG after sintering was at the meaningful level of 5% (p -value = 0.026). The effect of Nd:YAG laser on the surface of zirconia in order to create roughness to increase SBS was better than CO₂ laser. Thus, the result of this study showed that the type and sequence of laser and sintering application can affect the bond strength of veneering porcelain to zirconia. Similar to this study, there are several other studies that have examined the SBS of several materials and their compatible veneer has been analyzed.²⁹⁻³²

In this study, the SBS had their lowest figures in CO₂ + S (47.25 MPa) and their highest in S + Nd (70.30 MPa). The differences were not statistically significant except for S + Nd, which showed a significant difference with the control group. The reason for a low number in CO₂ + S, compared to others, could be due to the application of laser on zirconia samples at first and performing the sintering afterward. As shown in SEM pictures (Fig. 1A), CO₂ laser's penetration in zirconia surface was excessive and created a groovy look.³³ If laser creates deep grooves, it can damage

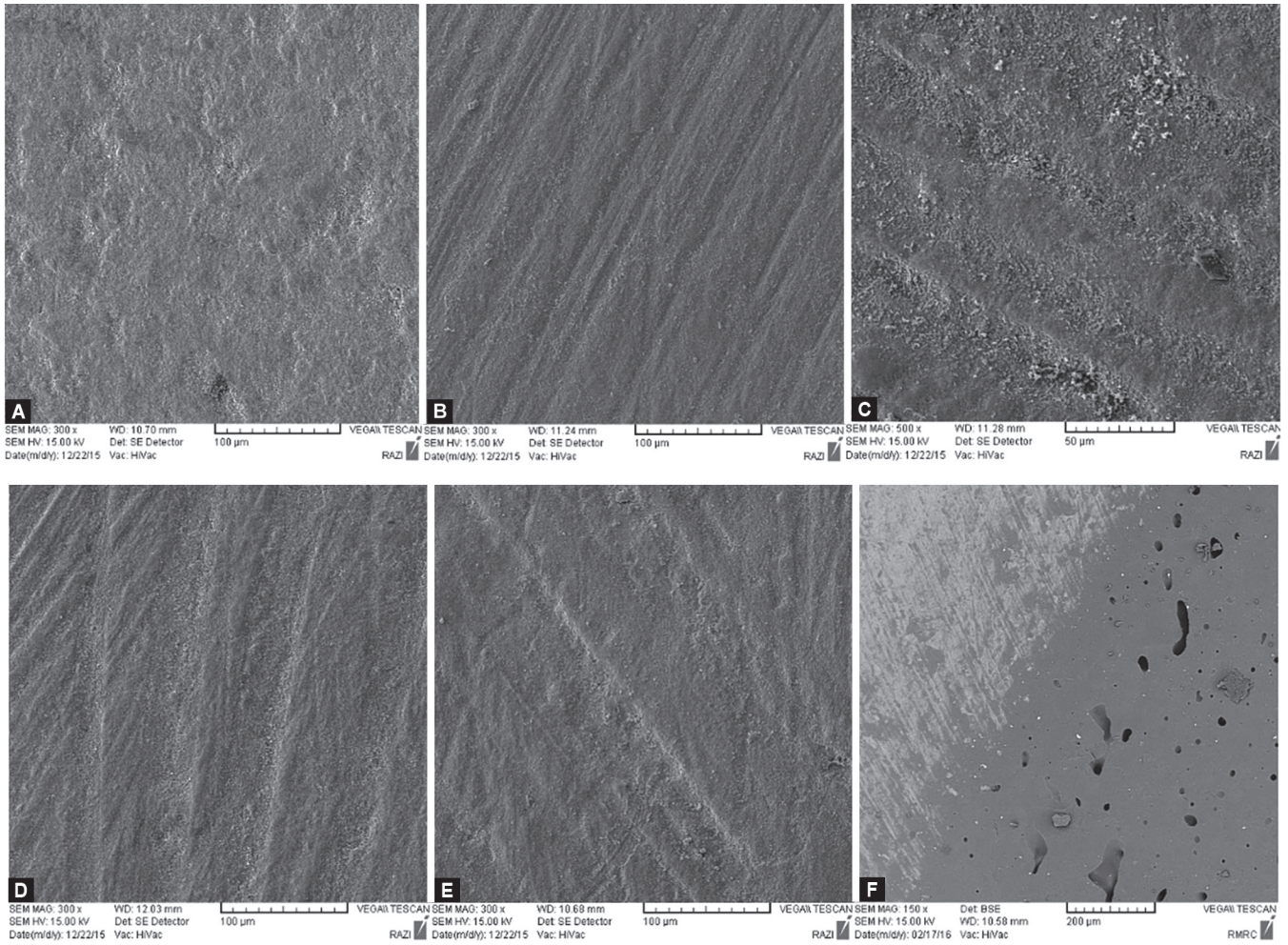
zirconia frame in fixed prostheses since the depth of their frames are 0.3–1 mm.³³

Primary studies that used the SBS test reported a range of the SBS between 9.4³⁴ and 31 mega pascal³⁵ between Y-TZP and ceramic veneer. In the current study, the figures are higher and the reason for such difference was believed to be due to the lack of any standard for sample preparation, study conditions, and analyzing methods. On the other hand, some researchers believe that the shear test results are greatly affected by the shapes and designs of the samples, and this makes comparing the results of various studies complicated.³⁶

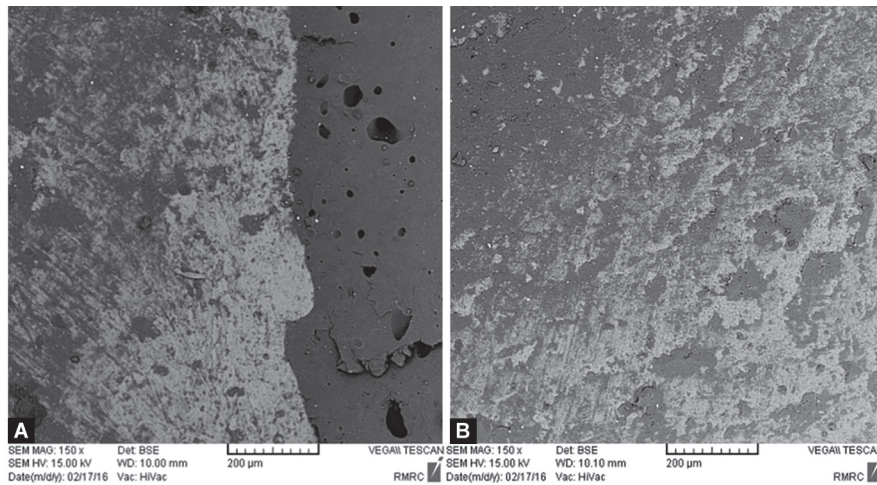
The results of this research is in agreement with other studies and they showed that CO₂, compared to other surface treatment methods such as sandblasting, Er:YAG laser, and Nd:YAG laser, was not effective for increasing the SBS and even reduced it.^{37,38} In CO₂ + S group, since sintering is performed after CO₂ procedure, it can be conceived that sintering would decrease the roughness in zirconia surface because of phase transformation of zirconia and as a result affects the SBS. SEM pictures are a confirmation to this notion.

In S + Nd group, first, the sintering was done. The sintered zirconia would probably resist better toward the undesirable changes of laser and minute explosion happening at the time of laser procedure. Those explosions leave debris behind which can attach to the melted surface of the ceramic and thus unbalance the bond.³⁹ Also, sintering increases the t-phase in zirconia, and the heat expansion coefficient between zirconia framework and porcelain veneer causes the strengthening of the bond.⁴⁰ Thus, laser procedures should be done after sintering. The pictures of SEM after the failure mode approve the point that the SBS in S + Nd had been the highest since the SEM picture of the random sample shows the failure to be completely cohesive in porcelain layer (Fig. 3A) while in other groups a range of failures was seen. In the microscopic pictures after the failure, backscatter mode was used for a better diagnosis and analysis of the layers which caused an easier differentiation between zirconia and porcelain.

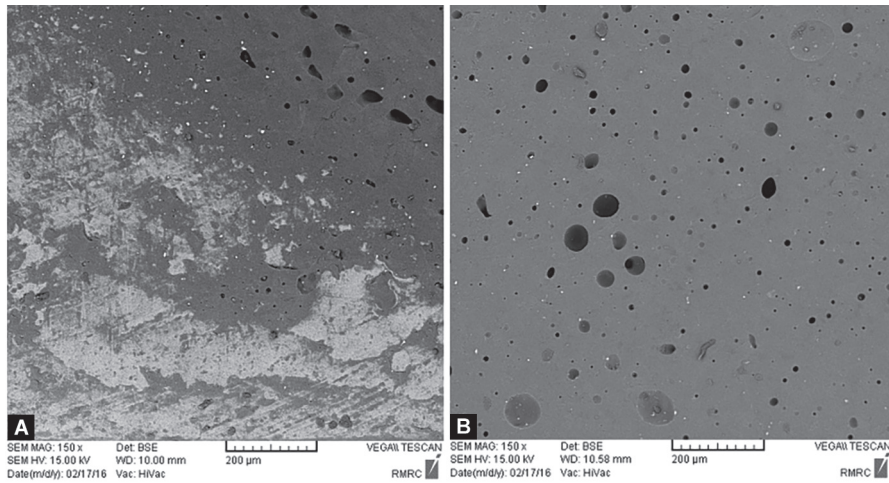
Under the conditions of performing laser after sintering, the CO₂ laser group showed a lower SBS compared to Nd:YAG (Table 1). The rate and pattern of the surface roughness created by Nd:YAG explain this difference. SEM pictures showed that the tracks caused by CO₂ were wider compared to Nd:YAG. The probable cause of the



Figs 1A to F: (A) The picture of an SEM after surface treatment of first group (group C). (B) The picture of an SEM after surface treatment of the second group, with deep tracks (group CO + S). (C) The picture of SEM after surface treatment of the third group (group S + CO). (D) The picture of SEM after surface treatment of the fourth group (group Nd + S). (E) The picture of SEM after surface treatment of the fifth group, with even and regular porosity (group S + Nd). (F) The picture of backscatter after mixed failure (group C)



Figs 2A and B: (A) The picture of backscatter after mixed failure (group CO + S). (B) The picture of backscatter after mixed failure (group S + CO)



Figs 3A and B: (A) The picture of backscatter after mixed failure (group Nd + S). (B) The picture of backscatter after cohesive failure (group S + Nd)

tracks can be attributed to higher energy of CO₂ laser and making a higher temperature and a subsequent cooling stress.^{38,41} Moreover, CO₂ laser beam has a bigger diameter compared to Nd:YAG (0.5 mm vs 320 micron), which can transfer more energy and damage the surface layer more.³⁸ The present study shows similar results to those reporting a higher bond strength for Nd:YAG compared to air-borne and silica coating.^{42,43}

Similar to the results of the present study, Paranhos et al.³⁸ and Henriques et al.⁴⁴ reported Nd:YAG laser to be meaningfully effective on the SBS, and compared to CO₂ laser, significantly increased the bond strength (14 vs 7.9 MPa).

Da Silveira et al. and Spohr et al.^{42,43} also reported results similar to the study at hand. Those two studies had laser settings similar to the present study but used graphite powder for increasing laser energy absorption since alumina-based ceramics do not absorb the 1064 nm frequency emitted by Nd:YAG laser. Zirconia ceramic can absorb the laser energy and does not need any initiators, such as graphite.

Akyil et al.¹⁷ announced that Nd:YAG and Er:YAG lasers had increased the irregularities on the zirconia surface. In contrast with the current results, they showed that Nd:YAG laser had decreased the SBS compared to control group.

Kasraei et al.⁴⁵ showed that surface treatment by CO₂ laser makes a stronger bond compared to Er:YAG laser and the reason can be attributed to the amount and type of created surface irregularities on the zirconia surface.

In accordance with this research, Bitencourt et al.⁴⁶ reported that the laser treatment is capable of improving the roughness of the zirconia surface and, consequently, the bond strength with resin cement and veneering ceramics. Er:YAG laser, however, did not present a favorable tendency toward bond strength, which was not investigated in the present research

CONCLUSION

With the limitations of this study in mind, following points can be concluded: Surface treatment can affect the strength of porcelain veneer bond to zirconia; also, the type of laser, the sequence of laser procedures, and the temperature can affect the bond strength.

Moreover, for creating surface roughness in order to increase the SBS, Nd:YAG laser on zirconia surface acts more effectively than CO₂. Thus, for increasing the mentioned strength, using laser after sintering is recommended.

Limitations and Suggestions

Restrictions in the use of thermocycling and cyclic loading lead to a lack of full-scale simulation of the examined samples compared to the clinical conditions. In future studies, the samples should be examined under of thermocycling and cyclic loading to reconstruct the conditions of the mouth.

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