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Efficacy of Surface Roughness and Bond Strength of Y-TZP Zirconia After Various Pre-Treatments

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Abstract

Objective: The aim of this study was to evaluate surface roughness (SR) and the influence of various surface treatments (STs) on the veneer ceramic shear bond strength (SBS) to Y-TZP zirconia. ***Background data:*** STs can improve the bonding properties of zirconia ceramics. However, little is known about the effect of Er,Cr:YSGG laser irradiation on SBS between zirconia and veneer ceramic/resin cement. ***Methods:*** Eighty ceramic discs of yttria-tetragonal zirconia polycrystals (Y-TZP) were prepared, and specimens were divided into eight groups ($n = 10$): untreated (control), air abrasion, and Er,Cr:YSGG laser irradiation with different energy intensities (1–6 W). Average SR of each specimen was determined with a profilometer, and a veneering ceramic recommended for zirconia was fired into cylinder-shaped Y-TZP specimens. SBS test was performed for each specimen at a crosshead speed of 1 mm/min, and the average means of SBS and SR were calculated. Fracture modes and the surface topography were evaluated with various microscopes after STs. Data were analyzed by using one way analysis of variance (ANOVA) and Tukey honest significant difference (HSD) tests. ***Results:*** The highest mean force value was observed in the air abrasion group, and followed by 6 and 5 W laser irradiations, respectively. The difference between control and air abrasion groups was found to be statistically significant ($p < 0.05$). All laser irradiations increased the SBS value, but 1 and 2 W laser irradiations showed no statistically significant differences compared with the control group. However, the SR value for air abrasion group was significantly higher than that of the control group and 1 W ($p < 0.05$), and there were no significant differences between the SR of all the laser groups and the control group. ***Conclusions:*** The result of this study showed that STs with air abrasion and high laser energy intensities (3–6W), can improve the bonding properties of Y-TZP zirconia.

Introduction

COMPUTER-AIDED DESIGN/COMPUTER-AIDED manufacturing (CAD/CAM) systems have been continuously developed and upgraded in prosthetic dentistry in association with zirconia, used primarily for the restoration of single crowns and fixed partial dentures (FPDs) in both the anterior and posterior regions. Zirconia materials, especially yttria-tetragonal zirconia polycrystals (Y-TZP), were recently introduced for prosthetic rehabilitations as a core material for single crowns,[1] frameworks for FPDs,[1,2] and, in dental implantology, as abutments,[3] because of their superior mechanical properties, such as high flexural strength (700–1200 MPa), fracture toughness (7–10 MPa $m^{1/2}$), high biocompatibility, and natural appearance.[3,4]

Bonding mechanisms between the zirconia and resin cement or zirconia and veneer ceramic are currently the subject of comprehensive research for enhancing the long-term success of zirconia restorations. Therefore, previous investigations have been focused on various surface treatments for improving the bonding potantial,[5,6] increasing the surface area, and creating a stronger micromechanical interlock.[7,8]

Researchers have described the various surface treatments to improve the adhesion of all ceramic restoration. Especially used are grinding with diamond burs,[9] air abrasion with Al_2O_3,[10–12] tribochemical silica coating (CoJet, Rocatec),[6] acid etching [typically hydrofluoric acid (HF)],[13] coupling with silane,[6] lasers,[6,10] plasma spraying with hexamethyldisiloxane,[14] selective infiltration etching (SIE),[15] experimental hot etching solution,[15] and a combination of any of these techniques. Casucci et al.[15] stated that the both SIE and experimental hot etching solution were alternative chemomechanical treatments to enhance the bonding strength of zirconia ceramic. However, some studies have shown that

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special adhesive monomers, such as 10-methacryloyloxyi-decyl-dihyidrogenphosphate (MDP), increased the bond strength to zirconia ceramics when used with various surface treatments.[12] However, acid etching application is not suitable for zirconia because it does not have a glassy phase.[6,13] Air abrasion with Al_2O_3 particles, with sizes ranging from 25 to 250 μm, is commonly used to increase the adhesion of the luting agent with the process that removes the contamination layers of the zirconia surface.[16,17]

Erbium:yttrium-aluminum garnet (Er:YAG) laser (λ = 2.940 nm) and Neodymium:yttrium-aluminum-garnet (Nd:YAG) laser (λ = 1.064 nm) were used for different clinical applications, including carious dentin removal and cavity preparations with Er:YAG laser[16,18,19] and tooth hypersensitivity,[20] bleaching,[21] and disinfecting dental tissues[22] with Nd:YAG laser. Recently, laser etching usually has been used on zirconia surface to provide a relatively safe and easy means of altering the surface of materials for improving surface roughness (SR) and shear bond strength (SBS),[10–12,23] although several authors[11,23,24] have investigated the effect of these lasers on zirconia surface for obtaining the best bonding with resin cement or veneering porcelain. Erbium, chromium: yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser (λ = 2.780 nm) has been introduced in dental clinics to remove carious dental hard tissues and to evaluate the morphological changes in human enamel and dentin that have been irradiated by it.[25] Some recent studies have evaluated the effects of the Er,Cr:YSGG laser irradiation on the SBS of resin cement to ceramic restorations;[26,27] however, a literature investigation showed that no study was found that evaluated the effect of Er,Cr:YSGG laser irradiation on zirconia.

During the past decade, the effectiveness of lasers[19,25] to some clinical applications, such as the removal of tooth hard tissues and the preparation of cavities, has undergone extensive evaluation. The aim of this study was to investigate the SR and SBS of zirconia after different surface treatments, including Er,Cr:YSGG laser irradiation of different intensities and sandblasting. The following null hypotheses were tested. (1) Surface treatments will increase the SR and SBS. (2) Er,Cr:YSGG laser irradiation of different intensities would have no statistically significant effect on SBS of the veneer ceramic/zirconia bond, at a significance of $p = 0.05$.

Materials and methods

Eighty pre-sintered Y-TZP zirconia cylinders (Noritake, Nagoya, Japan) (7 mm diameter, 3 mm height) were used in this study. Specimens were sanded with 600, 800, and 1200 grit silicon carbide abrasives (English abrasives, London, UK) by a sander machine (Phoenix Beta Grinder/Polisher, Buehler, Germany) under water for 15 sec and at 300 rev/min to be able to create a standard surface, and according to the manufacturer's instructions, all specimens were sintered at 1500°C for 8 h in a ZYrcomat (VITA Zahnfabrik, Sackingen, Germany) sintering furnace. Then, they were randomly divided into eight groups ($n = 10$) according to the surface treatments performed:

Control: No surface treatment procedure was applied.

Sandblasting: Bonding surfaces of Y-TZP zirconia specimens were treated with 120 μm Al_2O_3 particles at 2 bar pressure for 15 sec at a distance of 10 mm.

Laser irradiations (1–6 W): Bonding surfaces of zirconia specimens were irradiated by Er,Cr:YSGG laser (Millennium; Biolase Technology, Inc., San Clemente, CA) with a 2.78 μm wavelength. The optical fiber of the laser (600 μm diameter, 6 mm length) was placed perpendicularly to the surface at a distance of 10 mm. With pulse duration from 140 to 200 μs with a repetition rate of 20 Hz (pulses/sec) and pulsed laser-powered hydrokinetics, the output power of this equipment ranges from 0.25 to 6.0 W. However, the energy parameters at 1, 2, 3, 4, 5, and 6 W, respectively, and water/air flow of 55% and 65%, respectively, were used continuously during the irradiation for 20 sec.

The test protocol for all treatments in the present study is shown in Fig. 1.

Surface roughness evaluation

Following surface treatments, surface roughness (Ra, μm) of each specimen was determined with a profilometer (Mitutoyo Surftest SJ-301, Tokyo, Japan). The Ra value describes the average roughness value for a surface that has been traced by the profilometer. A lower Ra value indicates a smoother surface. Ten measurements at different locations were recorded for each specimen, and the average of these 10 measurements was used to obtain the Ra value of each specimen. In addition, one specimen from each group was selected for atomic force (AFM) and scanning electron microscope (SEM) analysis.

AFM analysis

One specimen for each group was evaluated under an AFM (Ambios Technology, Santa Cruz, CA). Digital images were taken in air. The tapping mode was performed using NSC16 (W_2C, Si_3N_4) tips oscillating at a frequency of 170 kHz. Changes in vertical position provide the height of the images, registered as bright and dark regions. The tip specimens were kept stable via constant oscillation amplitude (set-point amplitude). Fields of view at 10×10 μm scan size and a scan rate 1 Hz were considered. Then a single operator analyzed the average surface roughness (Ra) of the pre-treated ceramics, expressing it as a numeric value (in nanometers) using a specific software (Nanoscope V530R35R).

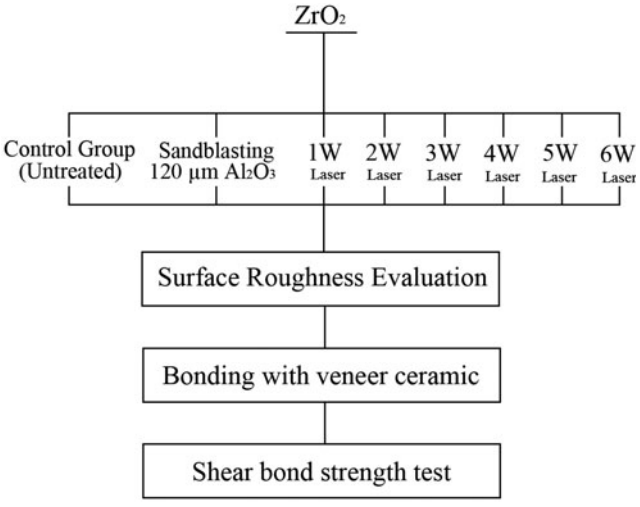


FIG. 1. The schematic test protocol.

SEM analysis

All specimens were mounted on metallic stubs, gold-sputter coated (Polaron Range SC 7620; Quorum Technology, Newhaven, UK), and evaluated with SEM (JSM-6060LV, Jeol, Tokyo, Japan) for the surface roughness and morphological features in the surface treatments applied on ZrO_2 surfaces. Images from each group were taken at 5000 × magnification.

SBS test

Zirconia specimens were then placed into a custom-made metal mold convenient (7 mm in diameter and 3 mm in thickness) for the preparation of veneering porcelain, and porcelain veneers were applied on the zirconia surface according to the manufacturer's instructions (Fig. 2). They were fired in a vacuum porcelain furnace that can be programmed to 760^0C. All zirconia-ceramic specimens were ultrasonically cleaned for 3 min and they were stored in distilled water at 37°C for 24 h. The zirconia-ceramic specimens were then submitted to an SBS test using a universal testing machine (Lloyd LF Plus; Ametek Inc., Leicester, UK), with a crosshead speed of 1 mm/min until fracture.

Fracture analysis

The surface of the fractured specimens were analyzed under a stereomicroscope (Stemi DV4, Göttingen, Germany) at ×32 magnification to assess the type of failure. The failure modes were observed as adhesive failure, in which veneer ceramic completely separated from the zirconia surface; cohesive failure, in which veneer ceramic completely fractured; and mixed failure, in which both failure types were observed (adhesive and cohesive).

Statistical analysis

The mean SBS, Ra values, and standard deviations of the specimens were statistically evaluated with one way analysis of variance (ANOVA) test in order to compare SBS and Ra values between different surface treatments, and multiple pairwise comparisons were made with Tukey's honest significant difference (HSD) test ($p < 0.05$).

Results

Table 1 presents mean and standard deviation values of the SBS and SR parameters for all groups. Results of statistical analyses indicated that all the surface treatment techniques increased the bond strength values between the veneer ceramics and Y-TZP zirconia. Sandblasting and Er,Cr:YSGG laser irradiation with high intensities (3–6 W) significantly increased the SBS values compared with the control group ($p < 0.05$); however, there was no statistical difference between sandblasting group and the 4, 5, and 6 W laser irradiation groups. But statistical differences in SBS were recorded after 3 W laser irradiation when compared to the untreated surface. Analysis of the data also revealed that no significant difference was found between the 4, 5, and 6 W groups and the 1, 2, and 3 W groups. The highest mean SBS value was observed in the sandblasting group (23.31 ± 4.10), followed by 6 and 5 W irradiations (22.99 ± 3.24 and 21.69 ± 4.00, respectively). The lowest mean bond strength value was observed in the control group (11.31 ± 5.56).

On the other hand, the mean SR value for the sandblasting group was significantly higher than for the other groups ($p < 0.05$). Except for the sandblasting group, there were no statistically significant differences within other groups. However, the highest SR was recorded for the sandblasting

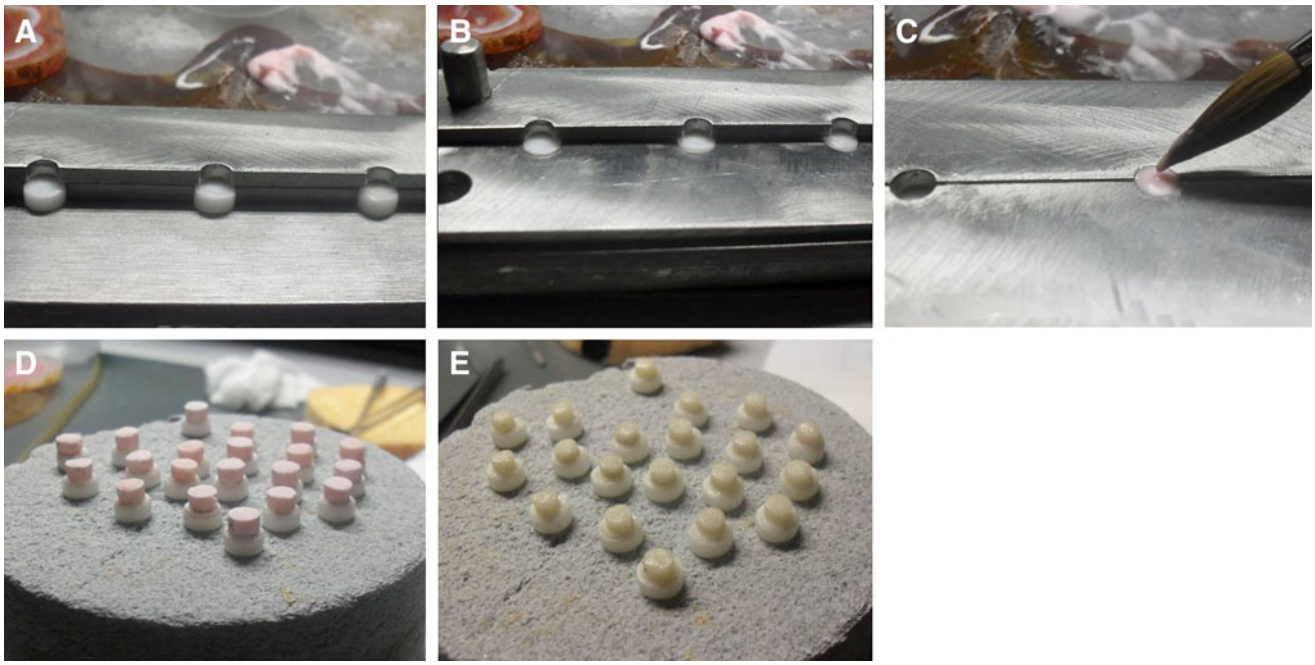


FIG. 2. Veneer application procedures. **(A)** Placement of zirconia specimen. **(B)** Placement of second metal mold for veneer ceramic. **(C)** Veneer ceramic application **(D)** before and **(E)** after firing of the zirconia-ceramic specimens.

Table 1. Mean, Standard Deviation Values of the Bond Strength (MPa), Surface Roughness (Ra, μm), and Indicence of Failure Modes

Surface treatment	*Mean (SD)*	*Roughness (SD)*	*Mode of failure of groups*		
			Adhesive failure	*Cohesive failure*	*Mixed failure*
Control	11.31 (5.56)	0.75 (0.08)	8	0	2
Air abrasion	23.31 (4.10)	0.90 (0.05)	4	1	5
1 W Laser	13.20 (2.17)	0.79 (0.07)	4	0	6
2 W Laser	13.65 (1.40)	0.82 (0.05)	4	1	5
3 W Laser	16.83 (2.81)	0.83 (0.03)	6	0	4
4 W Laser	18.50 (2.62)	0.84 (0.08)	4	1	5
5 W Laser	21.69 (4.00)	0.84 (0.06)	1	3	6
6 W Laser	22.99 (3.24)	0.83 (0.08)	4	0	6

group (0.90 ± 0.05 Ra), whereas the lowest roughness was seen in the control and 1 W laser irradiation groups (0.75 ± 0.08 and 0.79 ± 0.07, respectively).

Figures 3 and 4 show SEM and AFM images of ZrO_2 surfaces with different surface treatments. The analysis showed a variation in ZrO_2 surface topography. The resulting surface morphologic features were similar between the 2 and 6 W laser irradiation groups. Also, irregular surfaces were observed on ZrO_2 surface as melting, rare crevices and small pits formed by the laser optical fiber tip, in laser irradiation groups (Fig. 4C–H). The surfaces of specimens treated by sandblasting became rough and irregular and showed depression areas that possibly resulted from the high impact of blasting particles (Figs. 3B and 4B). This result is in accordance with the SBS and SR values for the sandblasting group.

The analysis of failure revealed that fracture occurred predominantly in the adhesive at the veneer ceramic/zirconia interface (43.75% of the failures), followed by mixed failures (both failure types – adhesive and cohesive) (48.75% of the failures), and then by cohesive failures in the veneer ceramic (7.5% of the failures).

Discussion

The results obtained in this study clearly demonstrate that the SBS and SR values of laser irradiated zirconia with different intensities and air abrasion were increased compared with the control group, by which null hypothesis one was accepted and two was rejected because the high intensities of laser irradiations significantly increased the SBS values.

Before the cementation process, cleaning the material surface and increasing the surface area are critical procedures for improving the bonding potential between the zirconia and veneer ceramic or resin cement.[12,13] Numerous investigations advocated that different mechanical and chemical conditioning methods were used to optimize bond

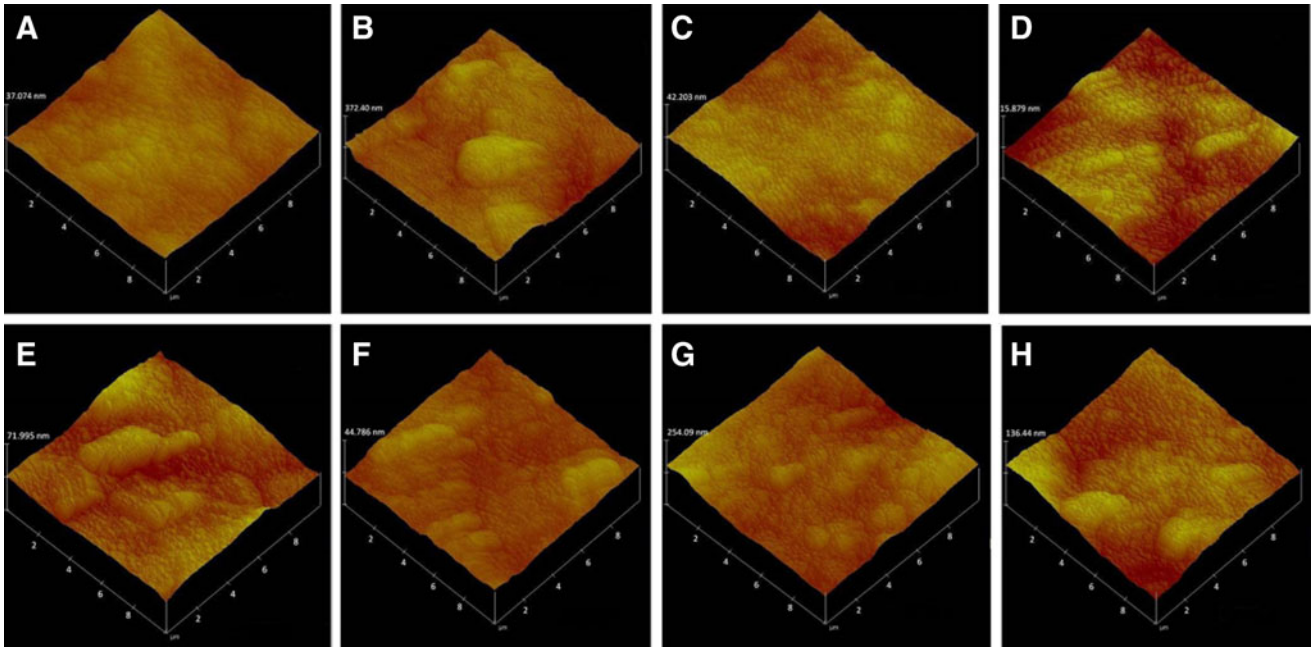


FIG. 3. Atomic force (AFM) images of different surface treatments on the surface of ZrO_2. **(A)** No treatment. **(B)** Air abrasion. **(C–H)** Laser irradiation with **(C)** 1 W, **(D)** 2 W, **(E)** 3 W, **(F)** 4 W, **(G)** 5 W, and **(H)** 6 W.

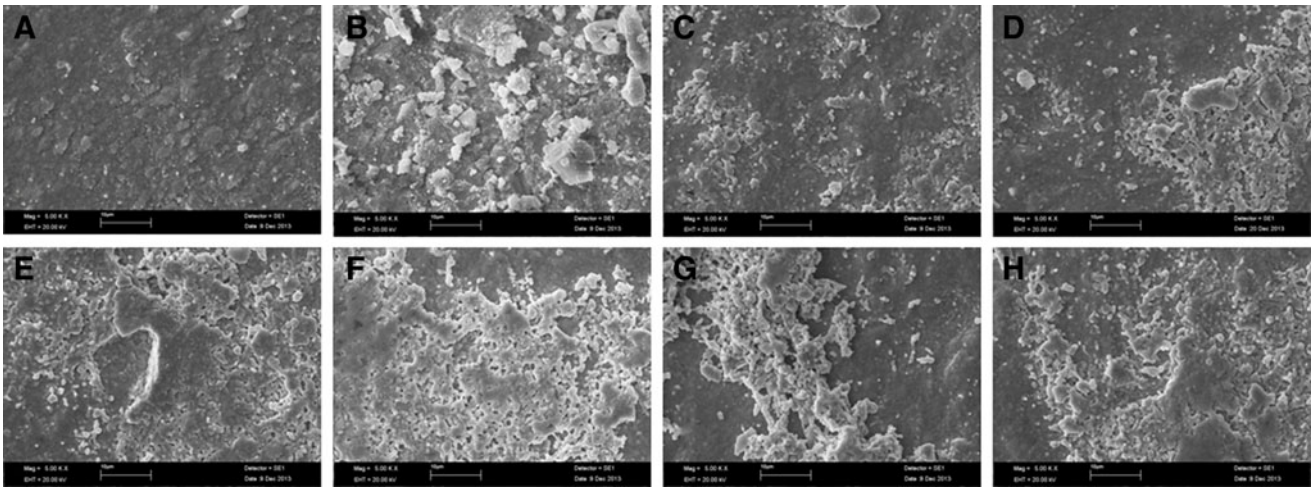


FIG. 4. Scanning electron microscopic (SEM) images of different surface treatments on the surface of ZrO_2 (×5000). **(A)** No treatment. **(B)** Air abrasion. **(C–H)** Laser irradiation with **(C)** 1 W, **(D)** 2 W, **(E)** 3 W, **(F)** 4 W, **(G)** 5 W, and **(H)** 6 W.

strength at the ceramic/cement interface.[6,27,28] With a reliable chemical bonding of resin cements to restorative materials, more tooth structures would be preserved, and more durable restorations with short clinical crowns would be performed.[11]

On the other hand, some studies[10,17] have demonstrated that sandblasting with Al_2O_3 particles is the preferred surface treatment methods for zirconia because commercial laboratories have used sandblasting by the conventional procedure, and some of them have advocated that air abrasion treatment is often performed to provide undercuts, or to prepare a rough surface to enable a strong adhesion of veneering ceramics or resin cement.[8,29] Cavalcanti et al.[12] and Subası et al.[30] demonstrated that surface treatment with sandblasting resulted in significantly higher bond strength of the resin cement to ZrO_2. There have been many studies in the literature reporting similar results.[28,31,32] Contrary to these studies, Fischer et al.[33] have stated that sandblasting is not a necessary surface treatment to improve the bond strength. However, Turp et al.[34] reported that air particle abrasion with 250 μm Al_2O_3 particles for 30 sec showed the highest surface roughness. Demir et al.[35] and Subası et al.[30] demonstrated that there were significant differences among air abrasion groups. Similarly, Casucci et al.[36] reported that airborne particle abrasion significantly improved the average surface roughness of Cercon (45.15 Ra) and Aadva Zr (51.67 Ra) ceramic compared with untreated group (7.31 and 7.27 Ra, respectively). In the present study, the effect of 120 μm Al_2O_3 particles on the surface roughness of zirconia was investigated, and this treatment resulted in statistically significantly differences. In the present study, results showed that sandblasting appears to be a more efficient method for changing the topography of zirconia surfaces, and that it yielded higher SBS and roughness values compared with the control group and laser irradiation, which uses different intensities for this study. These results agree with the previous studies mentioned. There was no correlation between SBS and SR results in any group.

Conclusions on SBS after different laser applications may be found in the literature, because many different experimental

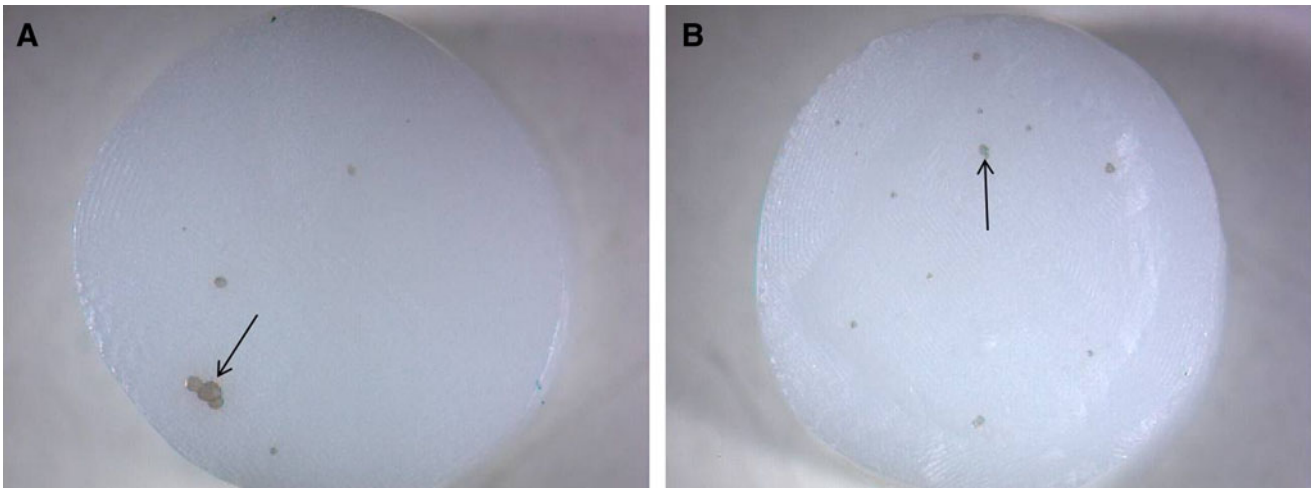


FIG. 5. Overheating areas (e.g., arrows) of the surface after application the higher energy. Sample images from **(A)** 5 W and **(B)** 6 W groups.

parameters have been used. Usumez et al.[26] evaluated the effect of Er,Cr:YSGG laser on the SBS of ceramic to resin cements, and stated that statistically significant differences were found between the laser-etched surfaces and the control group, and, similarly, Kursuoglu et al.[27] examined the untreated, HF etching, and laser etching of different intensities (1.5, 2.5, and 6 W Er,Cr:YSGG laser) on ceramic surfaces, and reported that laser irradiation at 1.5 and 2.5 W significantly affected the SBS (3.88 ± 1.94 and 3.65 ± 1.87 MPa, respectively) compared with untreated surfaces (1.95 ± 1.06 MPa). They also found that 6 W irradiation did not seem to be an efficient ceramic surface treatment technique.

In the current study, irradiation of ZrO_2 surface with different intensities of Er,Cr:YSGG laser was proposed as an alternative treatments method. The Er,Cr:YSGG laser has the ability to remove particles by a process called ''ablation,'' including microexplosions and vaporization.[26] In this study, in parallel with Kursuoglu et al.'s results, it appeared that the bond values increased in all laser groups; however, there was statistical difference between 3–6 W groups and control. Cavalcanti et al.[23] found that laser etching of ZrO_2 surfaces using an Er:YAG laser with different intensities (200, 400, and 600 mJ) was effective in obtaining the better surface roughness area, as compared with untreated surface. However, they reported that higher laser power settings might cause heat damage to the ZrO_2 structure, which can induce phase transformation and decrease bonding ability of the ceramic material. In addition, they stated that the lower energy intensity tested might be a potential method for surface roughness and result in significantly higher bond strengths than the high power setting.[23,27] In our study, the laser application of different intensities resulted in higher SR and SBS compared with the control group, and melting and some microcracks, small pits, and rare color alterations (Fig. 5) were also detected in the SEM images, but no significant loss of structure was seen. In contrast with the results of Cavalcanti et al.[23] ZrO_2 surface was not coated with graphite prior to laser irradiation and the water (55%) and air flow (65%) were used continuously during the irradiation. The reason for the difference in this result may be effective.

Demir et al.[35] experimented to improve the effect on surface modifications of zirconia after different energy intensities of an Er:YAG laser, and found that Er:YAG laser application of different intensities increased the SR, but that the differences were not statistically significant. Similarly, Subaşı et al.[30] found that Er:YAG laser irradiation changed the surface topography with formation of rare pits in the SEM images, and reported that the Er:YAG laser can be used to obtain micromechanical retention. In another study, they[34] investigated the influence of surface treatments on bonding to zirconia, and found that Er:YAG laser irradiated zirconia presented significantly higher bond strength than the untreated group. Moreover, Akın et al.[37] evaluated the effect of Er:YAG laser on bond strength and microleakage between resin cements and zirconia and reported that laser treatment is an effective method for bond strength and decreasing microleakage in the adhesive–zirconia interface. In another study, they[11] found that Er:YAG laser applications demonstrated higher bond strength than the control group. Similarly, Akyıl et al.[31] reported that the increase in SBS was more remarkable on Er:YAG laser-treated surface than on untreated surface. The findings of the present study are in agreement with these results.

The failure mode registered in the all tested groups was predominantly mixed (48.75% of the failure), whereas predominantly adhesive failure mode was seen in the control group and 3 W laser group (43.75% of the failures). Therefore, there was a correlation between SBS and failure types.

The present study proposes a new surface treatment to enhance BS and SR of a veneering ceramic to ZrO_2 treated with Er,Cr:YSGG laser irradiation of different intensities in comparison with traditional surface treatment, such as Al_2O_3 sandblasting. Although a high power setting resulted in more gentle damage than sandblasting, future investigations could focus on fracture strength and phase transformation of zirconia after different surface treatments. Evaluation of the clinical effects of each surface treatment will enhance the preferred different parameter. To evaluate the effect of temperature changes in the mouth on bonding at the zirconia-veneer ceramic interface by the thermal cycles test may be important for clinical trials.

Conclusions

Within the limitations of this study, the following could be concluded:

1. Air abrasion treatment and Er,Cr:YSGG laser irradiation with high intensities (3–6 W) provided a significant increase in bond strength.
2. 1 and 2 W laser irradiations were not effective as surface treatments to improve bond strength.
3. Air abrasion treatment created rougher surfaces on zirconia, such as rare crevices and small pits formed, than did the other treatments.

Author Disclosure Statement

No competing financial interests exist.

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