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ORIGINAL ARTICLE

Evaluation of the surface roughness of zirconia ceramics after different surface treatments

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Abstract
Objective. This study aimed to investigate the effects of different mechanical surface treatments of pre-sintered zirconium oxide (ZrO_2) in an attempt to improve its bonding potential. ***Materials and methods.*** One hundred and twenty IPS e-max ZirCAD (Ivoclar Vivadent) pre-sintered zirconia blocks (7 mm diameter, 3 mm height) received six different surface treatments ($n = 20$): Group C was untreated (control); Group E was Er:YAG laser irradiated; Group N was Nd:YAG laser irradiated; Group SB was sandblasted, Group SN was sandblasted and Nd:YAG laser irradiated; and Group SE was sandblasted and Er:YAG laser irradiated. After the surface treatments, the average surface roughness (Ra, μm) of each specimen was determined with a profilometer, then all the specimens were sintered. The surface roughness values were analysed through one-way ANOVA and Tukey's test. Changes in the morphological characteristics of ZrO_2 were examined through scanning electron microscopy (SEM). ***Results.*** Sintered sandblasted, Er:YAG laser treatment, sandblasted + Er:YAG laser and sandblasted + Nd:YAG laser irradiation resulted in a rougher surface than the other treatments. ***Conclusion.*** Nd:YAG laser irradiation alone was not effective in altering the zirconia surface morphology.

Key Words: *Y-TZP zirconia, surface roughness, surface treatment, laser treatment*

Introduction

Zirconia-based ceramics, especially yttria-tetragonal zirconia polycrystals (Y-TZP), have superior mechanical properties, such as high flexural strength (700–1200 MPa), fracture toughness (7–10 MPa m1/2), hardness, translucency [1] and high biocompatibility among other ceramic materials [2–7]. In addition, Y-TZP ceramic has aesthetic advantages because it has a colour similar to that of natural teeth [8]. Therefore, Y-TZP ceramic has a wide clinical usage as a core material for single crowns, frameworks for fixed restorations and in implant dentistry as abutments or implants, with the introduction of computer-aided design/computer-aided manufacturing (CAD/CAM) technology [9,10].

An effective bonding potential between resin cement and Y-TZP ceramic or Y-TZP ceramic to veneering porcelain is necessary to improve retention, the fracture resistance of restorations and marginal adaptation with the long-term performance of all ceramic restorations [11,12]. One of the requirements for an effective bonding potential is micromechanical attachment [13–15]. A strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface, which requires surface roughening and cleaning for adequate surface activation [16,17].

Surface treatment of ceramics increases the surface area and creates microporosities available for the penetration of resin cement or veneering porcelain, enhancing the mechanical bonding. Many researchers have investigated the effect of different surface treatments of Y-TZP ceramic in an attempt to improve its bonding potential. These attempts have included various surface treatments, such as sandblasting (Al_2O_3) [18–25], silica coating [23,24,26], hydrofluoric acid treatment [27,28] and liner treatment [18,29]. Recently, with the development of lasers used in dentistry, laser-etching [21,26,27,30–32]

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methods have been used. Acid etching does not impact the Y-TZP ceramic surface for roughness because of glassy phase [17,33–35]. Sandblasting with Al_2O_3 particles in sizes ranging from 25–250 µm is a commonly used surface treatment for Y-TZP ceramic [36]. The abrasive process increases the area available for bonding, improves the wettability of resin cement or veneering porcelain and removes loose contaminated layers [34,37]. Monaco et al. [23] reported the highest bond value with pre-sintered 110 µm Al_2O_3 sandblasting. Coinciding with Monaco et al.'s results, Fischer et al. [24] found the highest IPS e-max veneer ceramic bond values of Y-TZP ceramic with 110 µm Al_2O_3 sandblasting.

Another promising technique for the surface treatment of Y-TZP ceramic is laser irradiation [14]. Neodymium:yttrium-aluminum-garnet (Nd:YAG) lasers and Erbium:yttrium-aluminum garnet (Er:YAG) lasers have become increasingly popular in dentistry. Nd:YAG lasers have been shown to be effective for tooth hypersensitivity [38], bleaching [39], removing caries [40] and disinfecting dental tissues [41]. Er:YAG lasers have similar dental applications, including removing caries, preparing cavities, modifying ceramic surfaces and a surface treatment method for indirect restorations [42] by a process called 'ablation' that includes micro-explosions and vapourization [19]. The mechanism of ablation of dental hard tissues with erbium lasers is that it takes place by the expansion of sub-surface water resulting in micro-explosions. This micro-explosion induces strong mechanical separation of the calcified tissue. This constitutes the major principle of erbium laser ablation and produces non-uniform tissue removal with ejection of both organic and inorganic tissue microparticles, creating the micro-crater like appearance typical of lased surfaces [43].

Therefore, some researchers have investigated Nd:YAG and Er:YAG lasers to increase the roughness of Y-TZP zirconia surfaces. Akın et al. [32] reported that bond strength values between Y-TZP zirconia and resin cement after Nd:YAG laser treatment with the contact group was the highest and that Nd:YAG lasers and Er:YAG lasers increased the bond strength value.

The purpose of this study was to investigate and compare the effects of sandblasting, Nd:YAG laser irradiation, Er:YAG laser irradiation and combinations of these laser applications with sandblasting methods on the surface roughness of pre-sintered zirconium oxide. The null hypothesis was that all surface treatments used in this study would increase surface roughness.

Materials and methods

One hundred and twenty Y-TZP ceramic disc specimens were prepared (7 mm in diameter and 3 mm in thickness) of pre-sintered zirconia blocks (IPS e-max ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein) (n = 20 per group) according to the manufacturer's instructions. All specimens were sanded with 600-, 800- and 1200-grit silicon carbide abrasives (English abrasives, London, UK) by a sanding machine (Phoenix Beta Grinder/Polisher, Buehler, Germany) under water for 15 s at 300 rev/min to obtain a smooth, flat surface.

Surface treatments

Next, the specimens were randomly assigned to six groups (n = 20), according to the surface treatments applied. The study groups were allocated as follows:

Group C: Untreated (control)

No treatment was applied on the Y-TZP ceramic surfaces.

Group E: Er:YAG laser irradiated

The surfaces were coated with graphite prior to laser irradiation to increase energy absorption. An Er:YAG laser (Smart 2940D Plus, Deka Laser, Florence, Italy) was delivered using 4-µm-diameter optical fibre transmission system to the surface, scanning the ceramic surface for 20 s. The distance of application was 10 mm. The output settings were 150 mJ/pulse with a pulse repetition rate of 10 Hz, pulse duration of 700 µs, output power of 1.5 W and energy density of 119.42 J/cm^2. Moreover, the ceramic surface was cooled with a water spray at a rate of 5 mL/min during irradiation.

Group N: Nd:YAG laser irradiated

An Nd:YAG laser (λ = 1064 nm; Smarty A10) was delivered using 300-µm-diameter optical fibre perpendicular to the ceramic surface at 10 mm distance, scanning the ceramic surface during 20 s. The output settings were 100 mJ/pulse, with a pulse repetition rate of 10 Hz, pulse duration of 300 vs and energy density of 283.08 J/cm^2. Air cooling was used during laser irradiating of the specimens.

Group SB: Sandblasting

Sandblasting was performed with 120 µm Al_2O_3 particles (Blastmate II; Ney, Yucaipa, CA) at 2 bar pressure for 15 s at a distance of 10 mm. Then the specimens were washed under running distilled water and air-dried.

Group SN: Sandblasted + Nd:YAG laser irradiated

Y-TZP specimens were sandblasted at 120 µm with Al_2O_3 particles at 2 bar pressure for 15 s at a distance

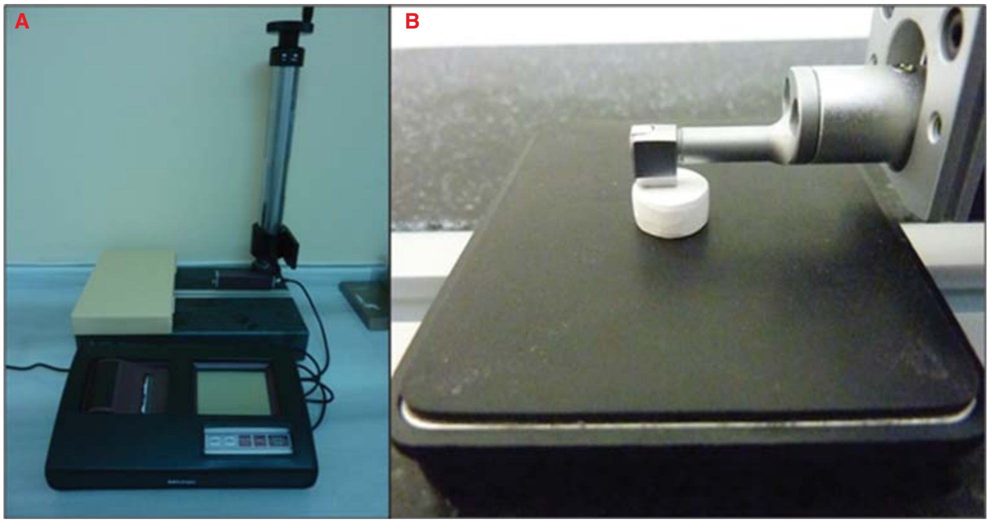

Figure 1. Evaluation of the surface roughness. (A) The photography of the profilometer. (B) Measuring surface roughness of the pre-sintered Y-TZP zirconia.

of 10 mm. Then the specimens were washed under running water and air-dried. Each sandblasted specimen was irradiated by a 100 mJ, 1 W, 10 Hz power Nd:YAG laser at 10 mm for 20 s.

Group SE: Sandblasted + Er:YAG laser irradiated

Y-TZP specimens were sandblasted at 120 µm with Al_2O_3 particles at 2-bar pressure for 15 s at a distance of 10 mm. Then the specimens were washed under running water and air-dried. Each sandblasted specimen was irradiated by a 150 mJ, 1.5 W, 10 Hz power Er:YAG laser at 10 mm for 20 s.

After the surface treatments, all specimens are ultrasonically cleaned for 3 min and specimens were stored in distilled water at 37°C for 24 h.

Surface roughness analysis

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 [44]. Three readings were taken with a travelling distance of 2 mm across the treated surface of the specimens, their average values were calculated (Figures 1 and 2). Then the specimens were sintered in

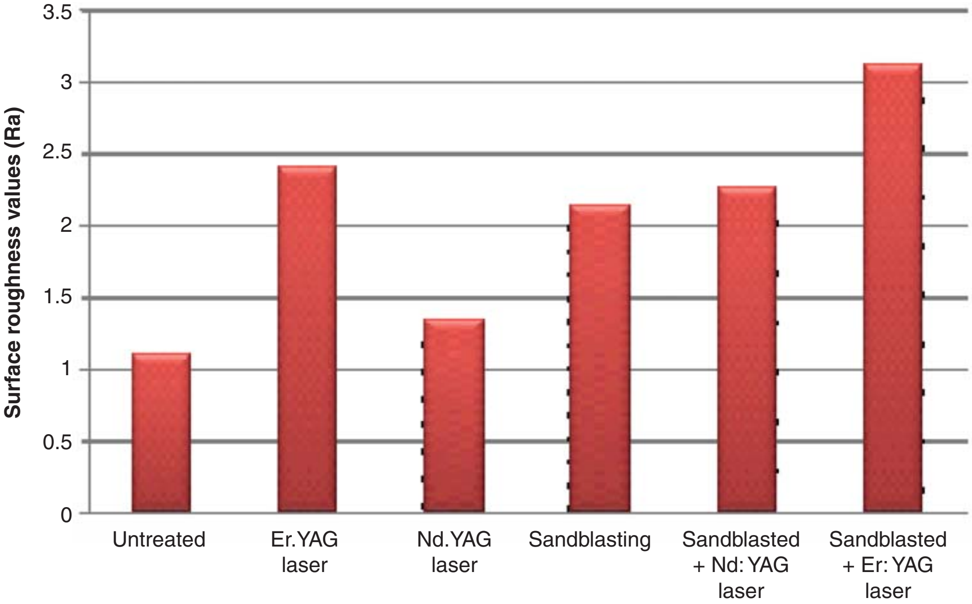

Figure 2. Surface roughness analysis of samples.

Table I. Mean value (SD) of the surface roughness (Ra, µm).

Surface treatment	Mean (SD)
Control	1.10 (0.29)
Er:YAG laser irradiated	2.40 (0.44)
Nd:YAG laser irradiated	1.35 (0.37)
Sandblasted	2.14 (0.40)
Sandblasted + Nd:YAG laser irradiated	2.26 (0.46)
Sandblasted + Er:YAG laser irradiated	3.12 (1.02)

n = 20, and the values for all the groups are significantly different from all the others ($p < 0.05$).

a ZYrcomat (VITA Zahnfabrik, Säckingen, Germany) sintering furnace according to the manufacturer's recommendation at 1500°C for 8 h.

Statistical analysis

Ra values were analysed with one-way ANOVA. Multiple pairwise comparisons were done with the Tukey post-hoc test. In order to meet requirements of the statistical test, the values were transformed into logarithm base 10. Analyses were carried out by the SAS 9.1 statistical package (SAS Institute, Cary, NC) with a $p < 0.05$ significance level.

Scanning electron microscope (SEM) analysis

All Y-TZP ceramic specimens were mounted on metallic stubs, gold sputter coated (Polaron Range SC 7620, Quorum Technology, Newhaven, UK) and evaluated under a scanning electron microscope (SEM) (JSM-6060LV, Jeol, Tokyo, Japan) at 2000× magnification to evaluate the morphological differences for the surface treatments applied on the Y-TZP ceramic surface.

Results

Some significant statistical differences were found among the surface roughness values belonging to Y-TZP ceramic specimens. All surface treatments tested produced rougher surfaces in comparison to the control groups of all specimens ($p < 0.05$). Accordingly, the highest surface roughness was recorded for the sandblasted + Er:YAG group (3.12 ± 1.02 Ra), followed by the Er:YAG laser irradiated (2.40 ± 0.44 Ra), sandblasted + Nd:YAG group (2.26 ± 0.46 Ra) and sandblasted group (2.14 ± 0.40 Ra) ($p < 0.05$). Additionally, the Nd:YAG laser (1.35 ± 0.37 Ra) irradiated presented Ra values similar to the control groups (1.10 ± 0.29 Ra) ($p < 0.05$). Comparison among the groups is shown in Table I. In SEM images, the roughest and most retentive areas were observed for the sandblasted + Er:YAG group, while the least irregularity was seen in the control and Nd:YAG laser groups (Figures 3-8).

Discussion

The bonding potential between Y-TZP zirconia and veneering ceramics or resin cements is an important factor for the clinical success of ceramic restorations [45]. This has stimulated studies to investigate the effects of various surface treatments on surface roughness. Researchers have evaluated bond strengths and morphological analyses of post sintered Y-TZP zirconia prepared using different surface treatments. However, some of them increased the risk of fracture and damage to Y-TZP zirconia by increasing the content of the monoclinic phase. Guess et al. [20] reported that after-sintering surface treatments damaged the structure of Y-TZP zirconia by causing microcracks. Similarly, Moon et al. [22] reported the monoclinic phase increased in cementation with surface treatments applied to sintered Y-TZP zirconia. Therefore, some researchers have applied surface treatments to pre-sintered Y-TZP zirconia and have reported some advantages to pre-sintered surface treatments on Y-TZP zirconia. With pre-sintered surface treatment, sharp and needle-like areas that adversely affect ZrO_2 resin connection and increase the fracture potential of ZrO_2 do not occur. Another

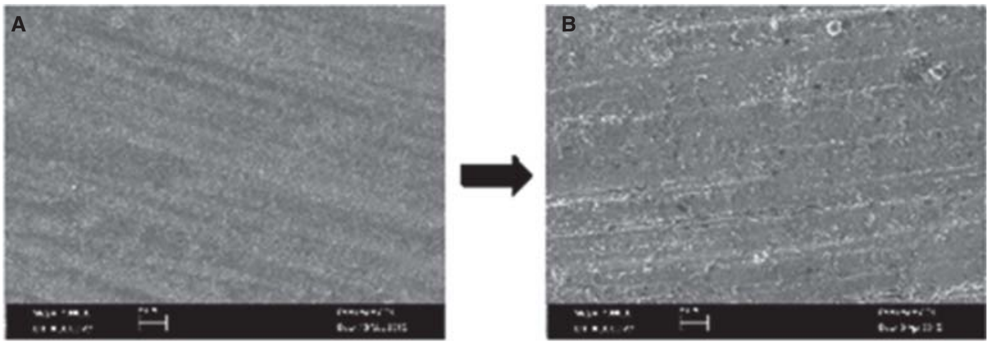

Figure 3. SEM images of different surface treatments on the surface of Y-TZP zirconia (×2000). (A) No treatment pre-sintered Y-TZP zirconia. (B) After sintering to Y-TZP zirconia.

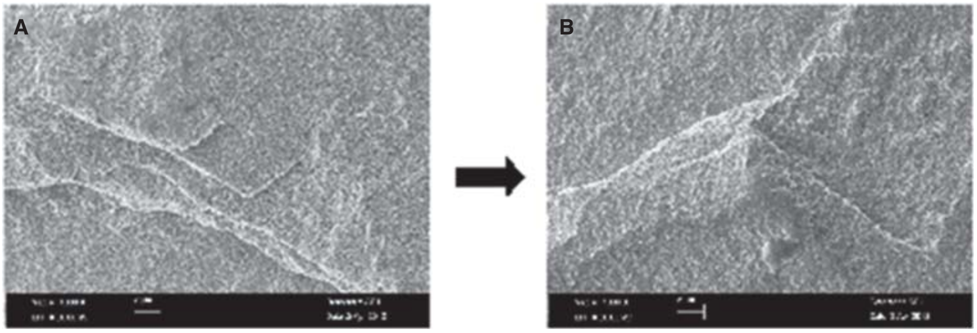

Figure 4. SEM images of different surface treatments on the surface of Y-TZP zirconia (×2000). (A) Er:YAG laser irradiated pre-sintered Y-TZP zirconia. (B) After sintering to Y-TZP zirconia.

advantage is that pre-sintering enhances the content of the tetragonal phase, which increased the mechanical features of ZrO_2.

Thus, the present study aimed to investigate the effects of sandblasting, Er:YAG laser irradiation, Nd: YAG laser irradiation and these laser applications combined with sandblasting methods on the surface roughness of pre-sintered Y-TZP zirconia. The null hypothesis was accepted. Because, all surface treatment increased the surface roughness on all ceramic groups.

Air abrasion with Al_2O_3 particles is a commonly used surface treatment for ceramic materials. Cavalcanti et al. [21] evaluated the effect of sandblasting on the surface roughness of two Y-TZP zirconias and found that air abrasion with 50-μm Al_2O_3 particles created a rougher surface compared to control surfaces (0.37 Ra) for the Cercon surfaces (2.41 Ra). Similarly, Casucci et al. [28] examined untreated (C), airborne particle abraded (S), selective infiltration etched (SIE) and experimentally etched (ST) zirconia surfaces and reported that airborne particle abrasion significantly affected the roughness compared to untreated surfaces (7.31, 7.27 Ra, respectively) for Cercon (45.15 Ra) and Aadva Zr (51.67 Ra) ceramics. Ersu et al. [46] reported that airborne particle abrasion increased the average surface roughness of Group In-Ceram Spinel (2.24 Ra) and Group In-Ceram Zirconia (1.27 Ra), compared to untreated zirconia (0.59, 0.38 Ra, respectively). In the present study, sandblasting with 120 μm Al_2O_3 particles increased the surface roughness of pre-sintered Y-TZP zirconia. These results agree with the previous studies mentioned. Representative SEM images of the treated pre- and post-sintered Y-TZP zirconia surfaces are reported in Figure 6.

The principal effect of laser energy is the conversion of light energy into heat and the most important interaction between the laser and substrate is the absorption of the laser energy by the substrate [21]. The Er:YAG laser has the ability to remove particles by a process called ‘ablation’, including micro-explosions and vapourization [19]. Cavalcanti et al. [21] experimented to improve the effect on the morphologic features of Y-TZP zirconia after different energy intensities of an Er:YAG laser and found that high values were noted after irradiation with 600 and 400 mJ. Furthermore, they reported seeing melting, some deep cracks and excessive loss of mass on the Y-TZP zirconia surface with SEM images.

Similar roughness in SEM images was seen on pre- and post-sintered Y-TZP zirconia in the present study (Figure 4). In addition, high laser power settings may deteriorate the zirconia surfaces. Therefore, we selected lower power settings in the present study. The application of the Er:YAG laser irradiation and sandblasting plus the Er:YAG laser irradiation resulted in modifications to the pre-sintered Y-TZP zirconia surface and significant increases in surface roughness. SEM images of Group E, Group S, Group

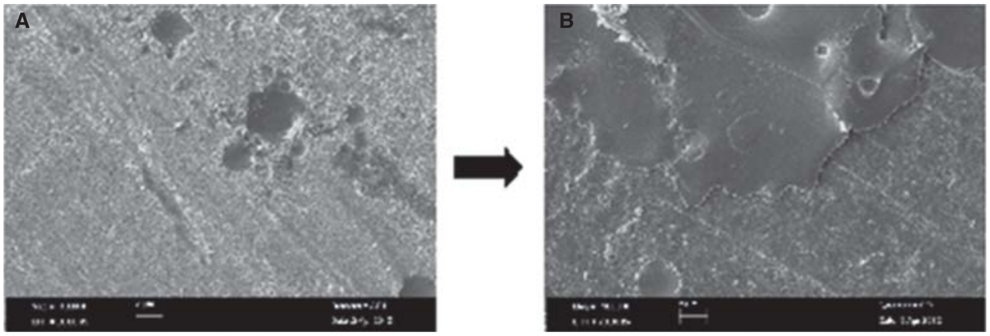

Figure 5. SEM images of different surface treatments on the surface of Y-TZP zirconia (×2000). (A) Nd:YAG laser irradiated pre-sintered Y-TZP zirconia. (B) After sintering to Y-TZP zirconia.

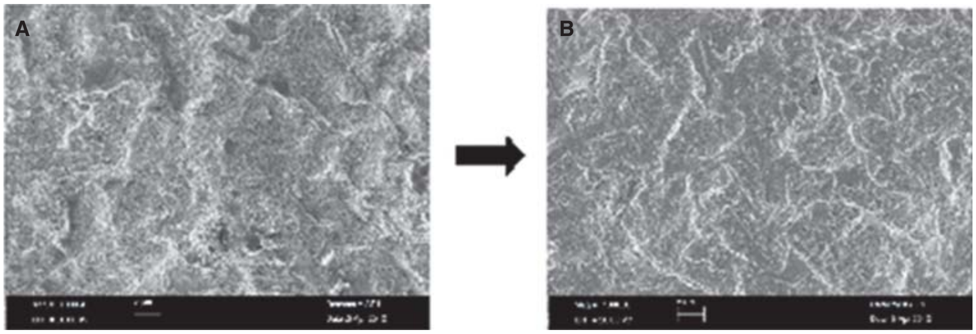

Figure 6. SEM images of different surface treatments on the surface of Y-TZP zirconia (×2000). (A) Sandblasted pre-sintered Y-TZP zirconia. (B) After sintering to Y-TZP zirconia.

SN and Group SE revealed rougher and deeper crevices on pre-sintered Y-TZP zirconia surfaces. These were also observed after sintering (Figures 4,5,6,7,8).

Spohr et al. [47] evaluated surface modification of In-Ceram Zirconia by an Nd:YAG laser, Rocatec system or Al_2O_3 sandblasting and reported that smooth blister-like bubbles with voids were surrounded by a flat and porous layer with openings of different size in SEM images. Therefore, they concluded that Nd:YAG laser irradiation surface treatments were more effective for Panavia Fluoro Cement adhesion than Al_2O_3 sandblasting treatments. Similarly, Akin et al. [32] examined the effects of different surface treatment methods on the shear bond strength (SBS) of resin cement to zirconia and found the highest SBS value was obtained in the Nd:YAG irradiated group with contact. In addition, Noda et al. [31] irradiated Y-TZP zirconia disks using an Nd:YAG laser with an applied voltage of 230 or 350 V and stated that Nd:YAG dental laser irradiation formed cracking and reduced oxygen content on the surface of zirconia. In the present study, the application of the Nd:YAG laser resulted in roughness on the zirconia surface and an increase in surface roughness. On the other hand, there was a statistically significant difference in surface roughness between Group SN and Group C, but not between Group N and Group C.

As a result, there is limited knowledge regarding the effects of various surface treatments on pre-sintered Y-TZP zirconia. The results of this study clearly indicate that sandblasting, Er:YAG laser application and laser applications combined with sandblasting are the most effective methods for increasing surface roughness on pre-sintered Y-TZP zirconia surfaces. However, the effect of changes in surface topography resulting from sandblasting and laser treatments on the surface roughness of pre-sintered Y-TZP zirconia and how long this roughness will last under clinical conditions should be the subject of further investigations.

Within the limitations of this study, the following conclusions were drawn:

- In SEM images, rougher, deeper crevices and micro-cracks were observed on pre-sintered Y-TZP zirconia in group SE. It was observed that after sintering, they did not disappear.
- Pre-sintered Y-TZP zirconia surfaces prepared with different surface treatments were more irregular than post-sintered Y-TZP zirconia surfaces.
- Sintered sandblasted, Er:YAG laser treatment, sandblasted + Er:YAG laser and sandblasted + Nd:YAG laser irradiation created rougher surfaces than untreated Y-TZP zirconia surfaces.

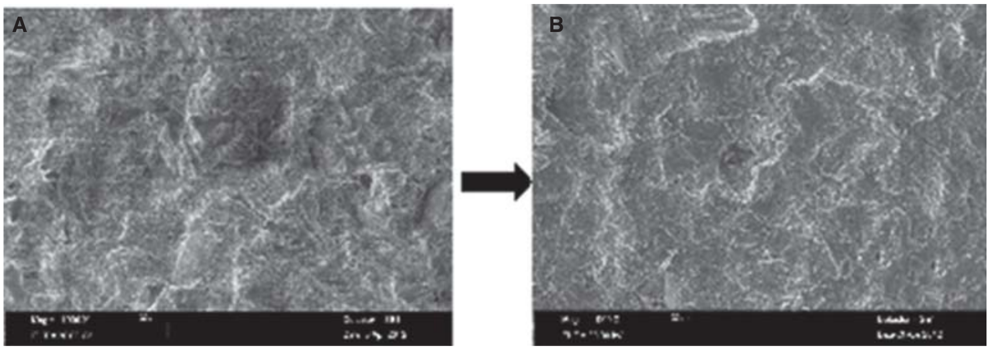

Figure 7. SEM images of different surface treatments on the surface of Y-TZP zirconia (×2000). (A) Sandblasted+Nd:YAG laser irradiated pre-sintered Y-TZP zirconia. (B) After sintering to Y-TZP zirconia.

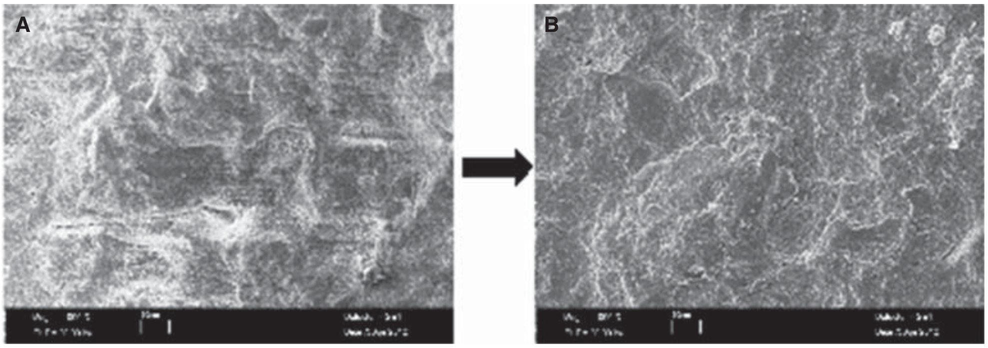

Figure 8. SEM images of different surface treatments on the surface of Y-TZP zirconia (×2000). (A) Sandblasted+Er:YAG laser irradiated pre-sintered Y-TZP zirconia. (B) After sintering to Y-TZP zirconia.

- Nd:YAG laser irradiation alone was not effective in altering the Y-TZP zirconia surface morphology.
- Sandblasting plus Er:YAG laser treatment created rougher surfaces on Y-TZP zirconia than the other treatments.

***Declaration of interest*:** The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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