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Three-dimensional profilometric assessment of Er:YAG laser irradiated unsintered zirconia

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ABSTRACT

This study evaluated the effects of different Er:YAG laser pulse width protocols on surface roughness, loss of volume of the material, and the step height formed of pre-sintered yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) by three-dimensional profilometric assessment. Blocks of pre-sintered Y-TZP were cut providing 63 standard 5-mm-thick samples which were divided by surface treatment, as follows ($n = 9$): G50 (100 mJ/10 Hz/1 W-50 µs); G100 (−100 µs); G300 (−300 µs); G600 (−600 µs); G1000 (−1000 µs); GTC (tribochemical silica coating); and GNC (untreated). After treated or not, samples were sintered according to the manufacturer's recommendations. Roughness, volume loss and step height were analyzed by 3D profilometric assessment with confocal laser microscopy. ANOVA and Tukey's test ($p < 0.05$) detected that irradiated groups showed increased roughness in the groups G50, G100, G300, and G600 when compared to GTC and GNC groups. The G1000 group showed a completely flat and unfavorable surface for retention. The groups G50, G100, and G300 showed great loss of volume and the step height formed, which can lead to a gap on the crowns. In G600 was observed satisfactory roughness with little loss of volume and the step height formed similar to GTC. Irrespective of laser protocol, any of the specimens showed the presence of cracks. It is suggested that the pulse width 600 µs (G600) is the most suitable pulse width protocol as an alternative surface treatment, promoting micro-retention, with little loss of volume of material, comparable to silica coating treatment.

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Introduction

The success of an indirect restoration is related to the material resistance, retention, and internal adaptation. In this context, good adhesion is critical for the long-term survival of the restoration. Surface treatments are applied to the internal surface of prosthetic crowns in order to increase roughness without damaging the material structure. Unfortunately, Y-TZP has the disadvantage of showing poor adhesion to luting cements [1]. The conventional surface treatment for glassy ceramics, such as porcelains and glass–ceramics involves the application of hydrofluoric acid followed by silane application [1, 2]. For Y-TZP, the high crystalline content results in an inert layer which is not susceptible to hydrofluoric acid etching. Moreover, Y-TZP does not contain a silica-based phase which hinders the use of the silane agent [3–5].

Aluminum oxide sandblasting of Y-TZP surface improves its interaction with the luting cement by means of increasing the surface energy and roughness of the material. However, Wolfart et al. and Shahin et al. [6, 7] demonstrated that the observed increase in bond strength seems to drop after long-term storage. Tribochemical silica coating of Y-TZP surfaces results in increased roughness, impregnation with silica particles, interacting with silane agents, and allowing better adherence to luting cements. Currently, this is the preferred method for surface treatment of Y-TZP [7–9], as it results in higher bond strength values compared to hydrofluoric acid and air abrasion treatments [10, 11]. However, previous works showed that the impact of alumina particles onto the Y-TZP surface damages the material surface, creating critical defects that may surpass the compression layer created by phase transformation and lead to reduced mechanical strength [9, 12].

Many studies [11, 13–16] suggested the use of high power lasers for surface treatment of Y-TZP. The erbium:yttrium–aluminum-garnet laser (Er:YAG) alters the surface of the Y-TZP with the formation of small depressions [15]. However, it was reported [12] that irradiation can cause melting, excessive loss of mass, and some deep cracks on the Y-TZP surface. Controversial literature [11, 13–18] results are due to the diversity of parameters and tested methodologies. The control of the laser parameters (power and pulse width) is of utmost importance to obtain a rough surface with little loss of volume and without cracking.

Er:YAG lasers with regulation of pulse width, such as super short pulse, have been recently developed. Shorter pulses reach a particular power with higher speed, and consequently, less energy is transformed into heat, resulting in cold ablation [17], which can cause less damage to the ceramic structure. A recent work showed that super short pulse Er:YAG laser resulted in higher surface roughness for a sintered Y-TZP [18].

Some studies [19–23] proposed that surface treatments should be applied on unsintered zirconia, claiming that after final sintering process, all crystals would remain in the tetragonal form [19, 20], avoiding unwanted phase transformations at the material surface.

Additional studies are needed to standardize the irradiation protocols of Er:YAG laser and determine the best application moment—either before or after final sintering—to increase its interaction with the luting cement. In this context, the use of the laser technology also deserves to be explored without compromising the microstructure and mechanical properties of the material. Therefore, the aim of this study was to evaluate the effect of different Er:YAG laser pulse width parameters on the surface roughness, volume loss, and the height of the step formed on unsintered zirconia specimens using three-dimensional (3D) optical profilometry. The null hypothesis was that surface treatments of yttrium-stabilized tetragonal zirconia polycrystalline do not modify zirconia roughness, volume loss, and step height.

Materials and methods

One commercial brand of Y-TZP polycrystalline material (VITA In-Ceram® YZ for inLab®; VITA Zahnfabrik, Bad Säckingen, Germany) was used in this study. This ceramic consists of 92 % ZrO_2, 5 % Y_2O_3, $HfO_2 < 3$ %, and Al_2O_3, $SiO_2 < 1$ % by weight [15]. Sixty-three ceramic specimens with dimensions of 9 × 7 × 5 mm were cut from unsintered blocks using a low-speed cutting machine (Isomet 1000 Precision Saw; Buehler Ltd, Lake Bluff, IL, USA) under water cooling at 250 rotations per min (rpm).

The ceramic surfaces were then polished in a polishing machine (Buehler Ltd., Lake Bluff, IL, USA) using a series of silicon carbide (SiC) abrasive papers in sequence (grit 600, 800 and 1,200; Buehler Ltd., Lake Bluff, IL, USA) for 20 s under water irrigation at 150 rpm to obtain standardized flat surfaces before applying the surface treatments.

After the polishing procedure, all specimens were ultrasonically (Digital Ultrasonic Cleaner; Shenzhen Codyson Electrical Co., Ltd, CHN) cleaned in distilled water for 480 s to remove any surface residues, washed in running water, and then dried with absorbent paper. Unplasticized polyvinyl chloride (UPVC) tapes were then placed on the edge of specimens' polished surfaces, leaving a central window of 7 × 3 mm square area on the ceramic surface. The specimens were then randomly divided into seven groups ($n = 9$) according to the surface treatment applied.

Laser settings: The Er:YAG laser (Fidelis III; Fotona, Ljubljana, Slovenia) with a wavelength of 2,940 nm was used to irradiate the zirconia surface using a specific handpiece (R02-C). The laser cylindrical sapphire optical fiber (0.9 mm in diameter) was placed perpendicularly to the ceramic surface at a distance of 7 mm (focused mode), and the squared ceramic area was scanned with water irrigation (60 %) and air (40 %) cooling. The laser parameters used for all laser groups were 100 mJ (energy), 10 Hz (repetition rate), 1 W (power), and 15.9 J/cm^2 (energy density). The specimens were mechanically irradiated for 10 s with an XYZ micropositioner (Model ESP 300, Newport Corporation, CA) with automatic pitch shifting, at standardized speed (6.0 mm/s) and distance (200 µm) of specimen displacement between laser pulses, thereby avoiding the creation of any gaps between the laser pulses. The energy emitted by the laser was checked by a power meter (Ophir Optronics, Wilmington, MA, USA) every three irradiated samples. The pulse width parameter varied according to experimental groups (Table 1).

Specimens were then sintered to the final dimension of 6 × 5 × 4 mm in a furnace (VITA ZYrcomat; VITA Zahnfabrik, Bad Säckingen, Germany) at 1530 °C for 7.5 h in accordance with the manufacturer's instructions.

Quantitative and qualitative changes on the Y-TZP surfaces were assessed by measuring the 3D average surface roughness (Ra), volume loss, and step height values using a 3D laser scanning confocal microscope (LEXT OLS4000, Olympus, Tokyo, JPN) at a magnification of 216 × (cut off wave length, lc = 406 nm using a Gaussian filter). This magnification was automatically set by the OLS4000 software when the objective of 10 × was used at 1 × zoom.

For the roughness measurement, confocal microscopy generated high-resolution 3D models of zirconia surfaces obtained from two different locations per specimen—one from the corners (untreated) and another one from the center (treated). Any tilting seen on the 3D models was corrected prior to the 3D surface roughness (Ra) calculation, in µm. The final roughness was calculated based on the difference between the roughness of the untreated and treated Y-TZP areas. Qualitative assessment of the Y-TZP surfaces was conducted by examining high-resolution micrographs obtained after calculating the Ra values.

After the surface treatments, a reference plan from the untreated area was defined, and the volume loss located below the reference plane was calculated in µm^3. To calculate the total of volume loss per specimen, the ratio of the volume loss (µm^3) and the area (µm^2) of treated Y-TZP was used.

To verify the step height (Rv) formed after the surface treatments, 10 readings of Rv (in µm) were carried out considering the reference surface and the treated surface. The final height of the step formed was calculated using the average of 10 readings per specimen.

Statistical analysis

Profilometric data were separately analyzed for each response variable (roughness of treated area, roughness of untreated area, volume loss, and measurement of step height formed between treated and untreated area) by one-way analysis of variance and then by Tukey's test (Minitab 17, Minitab Inc., Pennsylvania, USA) for pairwise comparisons among experimental groups ($\alpha = 0.05$).

Results

One-way ANOVA indicated statistical difference among experimental groups (Table 2; Figs. 1, 2). For the roughness of the untreated area, the expression (GTC = G300 = G50 = G1000 = G100 = G600) > G-NC is valid ($p = 0.00$), hence the group with the lowest roughness was GNC, while all others were statistically similar.

Table 1 Experimental groups considering zirconia surface treatment as main factor ($n = 9$)

Experimental groups	Zirconia surface treatment
G50	Laser Er:YAG pulse width, super short pulse mode—SSP (50 µs)
G100	Laser Er:YAG pulse width, medium short pulse mode—MSP (100 µs)
G300	Laser Er:YAG pulse width, short pulse mode—SP (300 µs)
G600	Laser Er:YAG pulse width, long pulse mode—LP (600 µs)
G1000	Laser Er:YAG pulse width, very long pulse mode—VLP (1000 µs)
GTC	Tribochemical silica coating (TC): Zirconia surfaces were abraded using an air abrasion device (Cojet; 3 M ESPE, Seefeld, Germany) filled with 30 µm Al_2O_3 particles coated with silica (Cojet Sand; 3 M ESPE, Seefeld, Germany) from a distance of approximately 10 mm and at a pressure of 2,.8 bar for 10 s, applied at an angle of 90° between the device and the ceramic surface—Positive control
GNC	Untreated—Negative control (NC)

Table 2 Results of 3D profilometric data (mean values with standard deviations in µm in µm) (roughness of treated area—Ra; roughness of untreated area—Ra; volume loss -Vl; and measurement of step height formed between treated and untreated area—Rv) per Group ($p < 0.05$)

Experimental groups	Roughness of untreated area (Sa)	Roughness of treated area (Ra)	Volume loss (Vl)	Step height (Rv)
G50	0.32 ± 0.02 a	1.32 ± 0.15 b	119.8 ± 19.6 b	2.68 ± 0.42 a
G100	0.31 ± 0.01 a	1.46 ± 0.12 b	126.2 ± 44.3 b	2.69 ± 0.75 a
G300	0.32 ± 0.02 a	1.83 ± 0.18 a	169.3 ± 39.2 a	2.75 ± 0.46 a
G600	0.30 ± 0.03 a	0.60 ± 0.03 c	76.2 ± 12.5 c	1.50 ± 0.25 b
G1000	0.31 ± 0.03 a	0.00 ± 0.00 e	0.5 ± 0.4 d	0.51 ± 0.11 c
GTC	0.35 ± 0.09 a	0.39 ± 0.06 d	27.0 ± 20.4 d	1.20 ± 0.24 b
GNC	0.23 ± 0.01 b	0.23 ± 0.01 d	0.00 ± 0.00 d	0.00 ± 0.00 c

Different letters indicate statistical difference

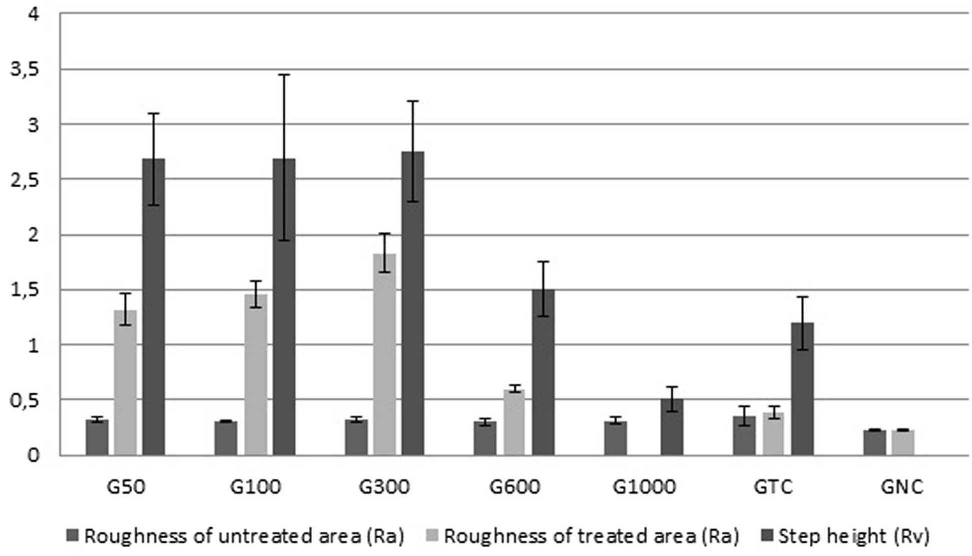

Figure 1 Effect of surface treatment on roughness and step height of Y-TZP surfaces (mean values with standard deviations in µm).

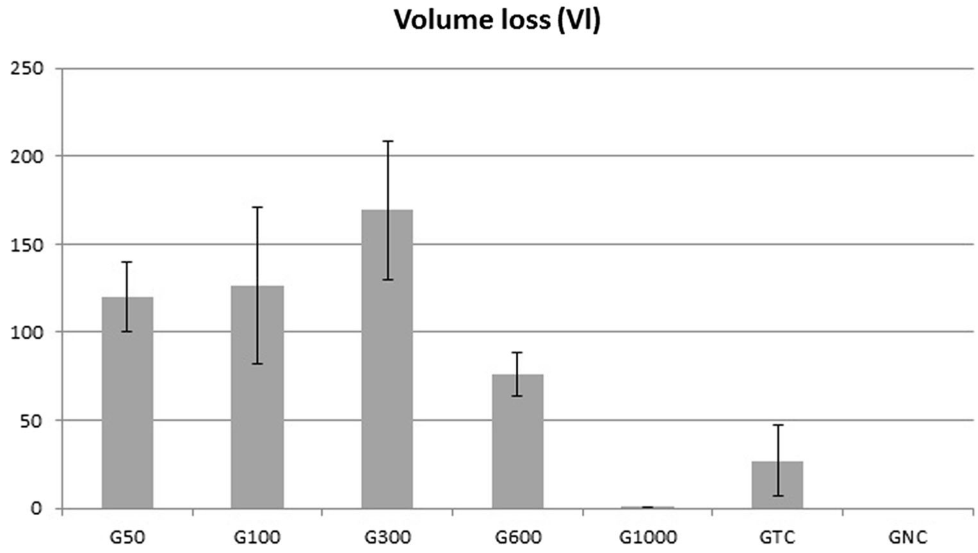

Figure 2 Effect of surface treatment on volume loss of Y-TZP surfaces (mean values with standard deviations in μm).

When the roughness of the treated area was evaluated, the following expression was valid ($p = 0.00$): G300 > (G100 = G50) > G600 > (GTC = GNC) > G1000. The highest roughness mean value for the Y-TZP surface was observed when it was irradiated with Er:YAG with a pulse width of 300 μs (G300). The roughness values obtained for the pulse widths of 50 and 100 μs (G50 and G100) were significantly lower than that of G300 and statistically higher than the roughness obtained for all other groups. G600 (pulse width of 600 μs) resulted in an intermediate roughness mean value (statistically lower than those of G50, G100, and G300, and higher than those of all other groups). Tribochemical silica coating (GTC) showed relatively low roughness mean value, which was similar to that obtained for the control (GNC), and these values were both significantly lower than those obtained from groups G50, G100, G300, and G600. The lowest roughness values were obtained for specimens irradiated with 1000 μs pulse width (G1000). The roughness mean values obtained for this group was statistically lower than those of all other groups tested.

Regarding volume loss, the expression G300 > (G50 = G100) > G600 > (GTC = G1000 = GNC) is valid ($p = 0.00$). The greater volume loss was observed when the Y-TZP was irradiated with 300 μs (G300) pulse width. The mean volume loss values obtained for G300 were statistically higher than those of groups G50 and G100 (50 and 100 μs, respectively) which had similar volume loss. The pulse width of 600 μs (G600) provided intermediate volume loss mean value, which was significantly lower than those obtained for G50, G100, and G300 and significantly higher than those of G1000, GTC, and GNC, which resulted in similar volume loss values.

For the height of the step formed, the expression (G300 = G100 = G50) > (G600 = GTC) > (G1000 = GNC) is valid ($p = 0.00$). The deepest steps formed when the Y-TZP was irradiated with 300 μs (G300), 100 μs (G100), and 50 μs (G50). These steps were statistically deeper than those obtained for groups G600 (600 μs) and GTC (tribochemical silica coating), which had similar step height. The shallowest steps were formed when Y-TZP was treated with 1000 μs (G1000) and the control (GNC).

Representative micrographs of each experimental group were obtained with magnification of 216×, in an area of 200 × 200 μm (Figs. 3, 4). The micrographs did not show any surface crack of the material in any of the experimental groups. The topography of the irradiated specimens indicated that irradiation of Y-TZP with the Er:YAG using different pulse widths promoted an effective ablation for almost all groups tested, except for G1000 which did not show any surface alteration after laser treatment.

Morphological analysis of the experimental groups revealed an ablation pattern consistent with the surface roughness values obtained in Table 2 for G50, G100, G300, and G600. G600 showed more homogeneous surface features compared to the other groups. However, G1000 showed a very flat topography, similar to that observed for control group (GNC). The

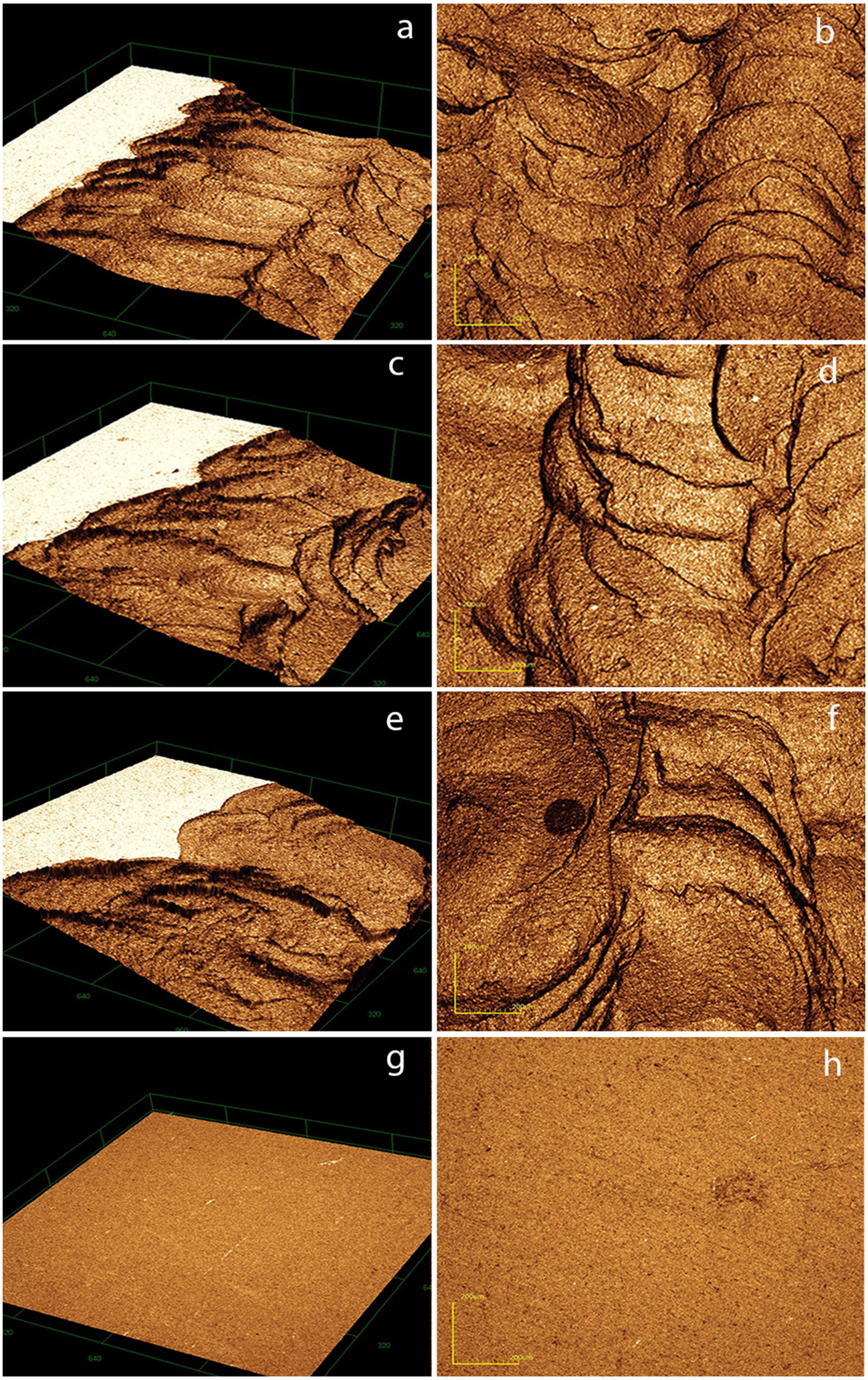

Figure 3 LSCM images illustrating the zirconia surface irradiated with the Er:YAG laser (**a**, **c**, **e**, **g**—volume loss at 3D; **b**, **d**, **f**, **h**—roughness at 2D in an area of 200 × 200 µm). **a** and **b** G50; (**c**, **d**) G100; (**e**, **f**) G300; (**g**, **h**) GNC (control–untreated).

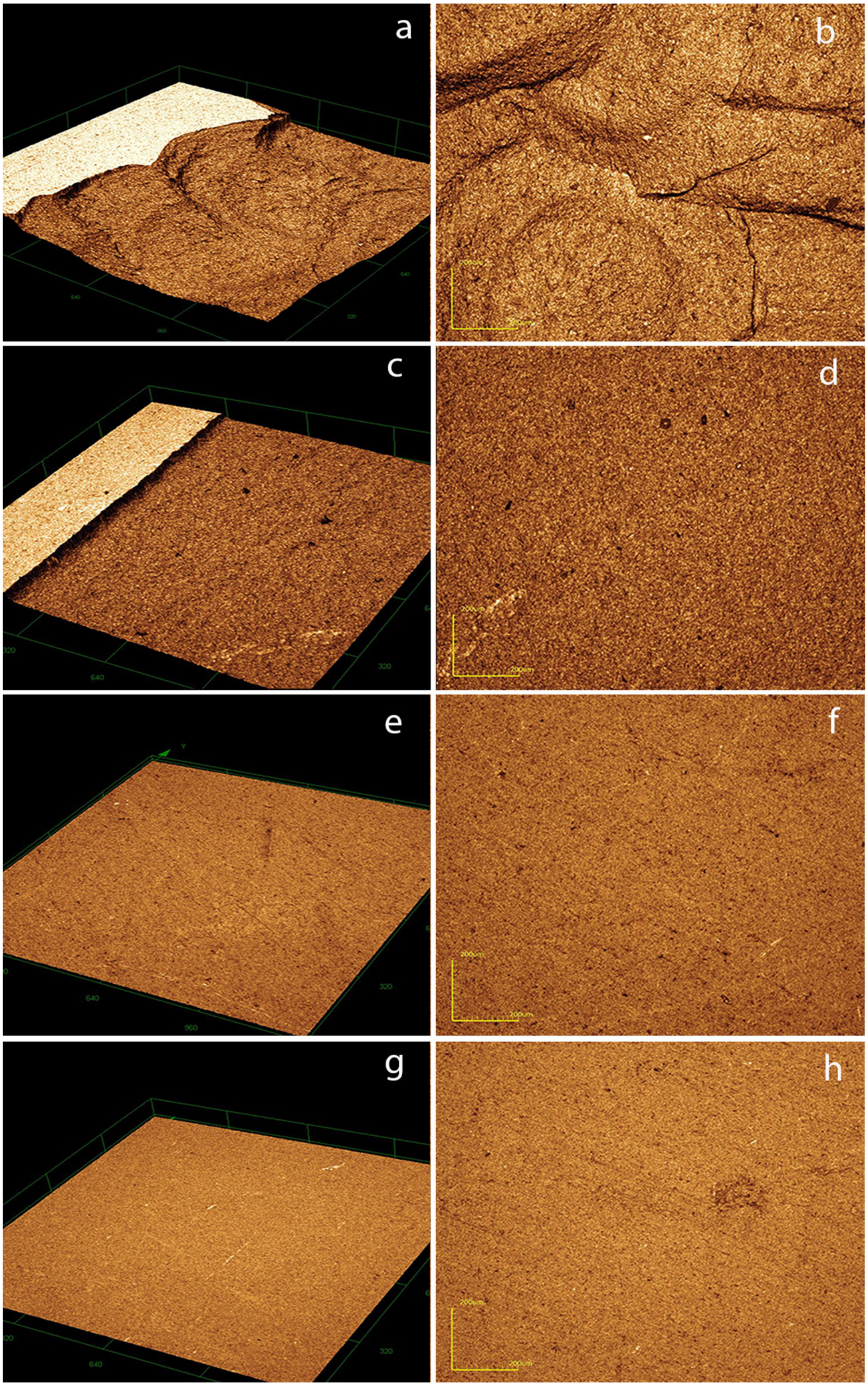

Figure 4 LSCM images illustrating the zirconia surface irradiated with the Er:YAG laser and tribochemical silica coating with 30 µm silica-coated Al_2O_3 particles (**a**, **c**, **e**, **g**—volume loss at 3D; **b**, **d**, **f**, **h**—roughness at 2D in an area of 200 × 200 µm). **a** and **b** G600; (**c**, **d**) GTC (tribochemical silica-coated); (**e**, **f**) G1000; (**g**, **h**) GNC (control–untreated).

larger the pulse width, the greater the surface irregularity, resembling multiple waves. Also, the larger the pulse width, the greater the waves dimension, due to a higher interaction period between light and substrate. GTC (tribochemical silica coating) showed a more homogeneous topography with few irregularities on the ceramic surface.

Volume loss was higher in groups G50, G100, and G300 than in groups G600, G1000, GTC, and GNC.

Discussion

The null hypothesis of the present study was rejected since the surface treatments influenced roughness, volume loss, and step height. The ideal surface treatment for zirconia restorations should generate a rough surface that favors their interaction with the luting cement without significant volume loss, which may compromise the final fitting of the restoration. It is also important to guarantee that such surface treatment should not cause significant phase transformation at the zirconia surface [13, 24], specially prior to cementation.

Er:YAG laser has been recommended for treatment of zirconia surface [13–16] because this type of irradiation can alter its morphological characteristics, increasing surface area for the luting cement. Based on literature [13, 18, 22] and on a pilot study, the current investigation used the energy density (ED) of 15.9 J/cm^2 for all experimental groups. In this way, the experimental set-up focused only on the effect of changing the pulse width on the topography of the zirconia specimens.

In this study, the irradiation of Y-TZP with the Er:YAG laser with pulse widths of 50, 100, 300, and 600 μs resulted in increased surface roughness compared to the negative (untreated) and positive (silica coating) control groups. However, in the group irradiated with pulse duration of 1000 μs, the Y-TZP surface showed extremely low roughness ($Ra = 0.00$ μm). These results are partly in agreement with those from Turp et al. [18], who showed roughness increase for pulse widths from 50 to 300 μs and decrease in roughness for 600 μs pulse width, using 1.2-mm-thick specimen. The very low roughness obtained in the current investigation for the pulse width of 1000 μs was related to the relative high thickness (5 mm) of the specimens. In fact, Sari et al. [25] concluded that the Er:YAG laser energy transmitted through ceramic specimens was 62 % of the incident energy when the thickness was 0.5 mm and 47 % for 1-mm-thick specimens. Therefore, the higher the pulse width, the greater the time available for the interaction between light and zirconia. The thinner the specimen and the higher the pulse width, the higher the probability of transmitting energy through it without absorption [26]. It is therefore essential to accurately identify the specimen thickness to choose the best irradiation parameter.

This is the first work that employed a confocal 3D laser scanning confocal microscope (LSCM) to analyze the effects of surface treatments performed on zirconia. One advantage of using LSCM is the fact that it does not require previous specimen preparation, and it is capable of capturing 3D images from various surfaces in order to evaluate topography, morphology [20], roughness [13, 27, 28], volume loss, and height of the step formed. Although several studies analyzed the change in surface roughness of zirconia with the use of lasers, none of them evaluated how much structure was lost by ablation, especially when zirconia is irradiated in the unsintered state, in which its hardness is very low.

Proper adaptation of restorations is a determining factor for a good prognosis of restorative treatment, because it can prevent, among other factors, the installation of secondary caries and retention loss [29]. Different studies [29, 30] showed that there is a significant gap between the zirconia coping and the prepared tooth structure. Such gap is higher in the occlusal area of the preparation than in the axial wall. Furthermore, Martins et al. [30] observed greater gaps for Y-TZP crowns than for porcelain fused to metal (PFM) crowns. Therefore, the surface treatment cannot cause significant loss of structure, given that the Y-TZP crown already has an internal gap that is inherent to its processing method.

With respect to volume loss and step height formed in the material after the surface treatment, there was greater volume loss when Y-TZP was irradiated with 300 μs pulse width, followed by 50 and 100 μs. The height of the step was statistically the same for these three parameters. Thus, although these pulse widths produced an increase in the roughness of the material, they also generate significant loss of structure, which can lead to greater restoration gaps. In this way, these pulse widths cannot be recommended as a surface treatment prior to cementation.

Optimal results were observed with the pulse width of 600 µs, which provided an intermediate volume loss and step height similar to that obtained for silica coating (positive control). This pulse width can be considered an appropriate parameter for surface treatment of unsintered Y-TZP, because it could improve surface roughness without compromising the internal fit due to material removal.

The gold-standard surface treatment for polycrystalline ceramics is silica coating (Cojet or Rocatec) [3, 8, 9]. This treatment uses aluminum oxide particles coated with silica of different sizes 30 or 110 µm. Since some authors [9, 31] observed that the larger the size of the abrasive particles, the greater the percentage of phase transformation, particles of 30 µm were used in the positive control group of the current study.

Tribochemical silica coating resulted in very small volume loss and irradiation with pulse width of 1000 µs resulted in even smaller volume loss. Both treatments did not alter the surface roughness of zirconia, so it was not able to generate a significantly rough surface that could improve the adhesion of Y-TZP to a luting cement. Based on the same rationale, the pulse width of 1000 µs cannot be considered an appropriate parameter for the surface treatment of zirconia.

Many studies performed the surface treatment after final sintering of the zirconia specimens [9, 11, 14–16]. However, the impact of the particles on the material surface leads to phase transformation and introduces cracks of different sizes.

The results of the current investigation indicated that the pulse width significantly affects the surface morphology of unsintered Y-TZP specimens. It is suggested that pulse width of 600 µs, with ED of 15.9 J/cm^2, is the most suitable parameter as an alternative for treatment of unsintered zirconia, in order to increase the internal roughness of the restoration, without incurring volume loss and damage to the structure of polycrystalline ceramic.

Based on 3D profilometric assessment, the following conclusions could be drawn:

Pulse width of 600 µs can be considered as a valid surface treatment for unsintered Y-TZP because it was capable of altering the surface and increasing its roughness without significant volume loss and forming a shallow step.

Er:YAG laser irradiation with pulse widths of 50, 100, and 300 µs was not suitable for surface treatment of unsintered Y-TZP because, despite promoting greater roughness values compared to the silica coating and control group (untreated), these parameters led to higher volume loss and higher step formation.

Irradiation with 1000 µs did not alter the zirconia morphology, and therefore, this parameter was disregarded as a possibility for zirconia treatment.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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