Original Article

Effect of Er,Cr:YSGG Laser Surface Treatment on Microshear Bond Strength of Monolithic Extra Translucent Zirconia to Resin Cement (An In-vitro study)

Rana A.A. Gadalla*[1]; Mohamed L. Zamzam[2]; Omnia Nabil[3]

Abstract:

Aim: The aim of this study was to evaluate the effect of Er,Cr:YSGG laser surface treatment at various power settings on the microshear bond strength between extra translucent zirconia and resin cement, comparing it to sandblasting. Methodology: Thirty-six sintered Cercon xt zirconia specimens were divided based on surface treatment (n=9): Group SB: Sandblasting with 110µm alumina particles, Group 6W: Er,Cr:YSGG laser at 6W, Group 8W: Er,Cr:YSGG laser at 8W, Group 10W: Er,Cr:YSGG laser at 10W. One sample per group was analyzed using scanning electron microscope (SEM). Subsequently, all samples were treated with zirconia primer, then resin cylinders were prepared and light cured. Samples then underwent a microshear bond test until failure. SEM was used to determine the failure mode for each group. Results: One Way ANOVA test showed no statistically significant difference between the groups ($p=0.902$). Sandblasted samples had noticeable micromechanical roughening and mixed-type bond failure. Laser-treated surfaces had reduced roughness compared to sandblasting, with predominantly adhesive failure. Conclusions: Er,Cr:YSGG laser irradiation at 6W, 8W, and 10W can be considered a valid option for treating the surface of extra translucent zirconia to generate reliable bond with resin cement.

Keywords: 5Y-TZP; Laser irradiation; Air-borne particle abrasion; µSBS

*[1] Master Candidate, Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University.
E-mail: rana.aly@dentistry.cu.edu.eg

[2] Professor Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University.

[3] Associate Professor, Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University.

INTRODUCTION:

Over the years, the concept of monolithic zirconia restorations had been sought to address the drawback of porcelain chipping in porcelain-veneered zirconia restorations, which is regarded as the most reported reason for failure. With the introduction of modern CAD/CAM technologies and the manufacturing of multilayer monolithic zirconia restorations, milling full contour restorations without veneering became an outbreak and can now deliver outstanding aesthetic results. [1,2]

The natural appearance and esthetics of a restoration are greatly influenced by the translucency of the ceramic material used to fabricate it. With the incorporation of various amounts of yttria as one of its main constituents, zirconia ceramic underwent massive evolution producing a wide range of translucencies, starting from high translucent to super translucent and ending with ultra translucent zirconia. The latter is claimed to exhibit extra high translucency with life-like esthetics, that can compete with that of lithium disilicate material. [3,4]

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) exhibits several advantageous characteristics, including biocompatibility, high compressive strength, chemical stability, and a thermal expansion coefficient closely matching that of natural dental tissues. Nonetheless, its crystalline nature, lacking a vitreous component, makes it resistant to conventional hydrofluoric acid etching and silanization surface treatments. [5,6]

In response to this challenge, adhesion to zirconia has garnered significant attention over the years. Numerous studies have explored the impact of different surface treatment methods, both mechanical and chemical, to increase the surface roughness of zirconia ceramics and, in turn, improve their bond strength with resin cement. However, the optimal surface treatment for zirconia remains a subject of debate. [7,8]

Among the various surface treatments tested, sandblasting stood out as one of the main and most practical methods utilized. It has shown to improve zirconia's bond strength to resin cements by revealing a clean contaminant-free surface, exposing zirconia's hydroxyl groups, and creating micropores permitting better micromechanical retention with resin. However, sandblasting is known to have a variety of drawbacks, including sharp scratches formation, cracks, and grain pull-outs, that were shown to deteriorate the surface of zirconia and impair the ceramic's mechanical integrity. [8]

Researchers have explored alternative surface treatments, presenting options such astribo chemical silica coating, selective infiltration-etching, plasma spraying, and laser irradiation. A variety of laser types, including CO_2, Er:YAG, Nd:YAG, and more recently, Er,Cr:YSGG lasers, have been subjected to testing and evaluation. [9]

Er,Cr:YSGG laser, being an erbium laser subgroup, possesses the property of being absorbable by both water and the hydroxyl groups of the hydroxyl-apatite crystals. Surface modifications induced by this laser result from an ablation mechanism, involving the removal of particles from the surface through micro-explosions and vaporization. During vaporization, internal pressure within the substrate increases until the inorganic component undergoes an explosion, occurring before it reaches its melting point.". [10]

A literature review revealed that while some studies have explored the effect of Er,Cr laser irradiation on zirconia's bond strength to resin cements, none have assessed the efficacy of this surface treatment method on the newly introduced extra translucent zirconia. Therefore, the aim of this in vitro study was to evaluate the microshear bond strength (μSBS) between resin cement and extra translucent zirconia after surface treatment with varying power settings of Er,Cr laser treatment and to statistically compare these results with sandblasting. The research hypothesis posits that there will be no significant difference in the μSBS values among the surface treatment methods evaluated, which include sandblasting with 110μm alumina particles and Er,Cr laser treatment at 6W, 8W, and 10W power.

MATERIALS AND METHODS:

Samples' preparation:

36 quadrangles were designed using Meshmixer software and then dry-milled from extra translucent zirconia blocks (Cercon XT, Dentsply Sirona, USA) with dimensions of 4×4×2 mm using 5-axis milling machine (inLab MC X5, Dentsply Sirona, USA) (**Figure 1**). The samples were then sintered in a sintering furnace (Sirona in Fire HTC Speed, Dentsply Sirona, USA) according to the manufacturer's instructions. For ease of handling, a customized polyvinyl chloride mold (25 mm internal diameter, 12 mm height) was used to create a resin base for each sample. The mold was filled with self-cure acrylic resin, embedding each sintered sample. After the acrylic fully set, the base was extracted and refined to remove any excess material.

Figure (1): Zirconia sample post sintering

Surface treatment protocols:

Samples were randomly divided into four groups (n=9) based on the surface treatment performed. Group SB**:** samples were air abraded with 110 µm aluminum oxide powder in a dental sandblasting unit, under 2-bar pressure, at a 90° angle and a distance of 10 mm for10 seconds; Group 6W: samples were treated using an Er,Cr:YSGG laser (WaterlaseiPlus, Biolase, San Clemente, USA) at a power of 6W; Group 8W: samples were treated using an Er,Cr laser at a power of 8W; Group 10W: samples were irradiated using an Er,Cr laser at a power of 10W. All laser groups used the same settings: 20 seconds duration at 20Hz frequency with 150mJ energy, and 140 µs pulse duration. Sandblasting and laser surface treatments were performed using custom-made 3D-printed holders designed to standardize the specified standoff distances (**Figure 2**). To evaluate the morphological surface traits post surface treatments, one sample from each group was examined with SEM at 1000 x and 10,000 x magnifications. Thereafter, the samples were cleaned using alcohol and air dried using an oil-free compressor.

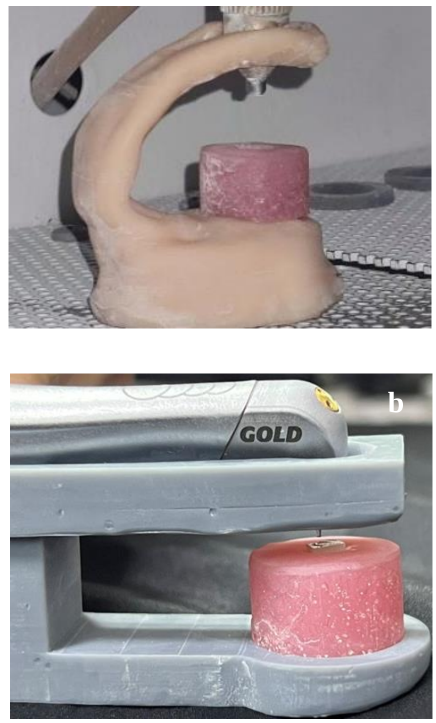

Figure (2): Custom-made 3D-printed holders designed to standardize the specified standoff distances during sandblasting (a), and laser surface treatments (b).

Bonding to resin cement:

Following the mechanical surface treatments, a zirconiaMDP-containing primer (Z-Prime™ Plus, Bisco, USA) was applied to all zirconia samples across all groups and then dried using an air syringe for 3 seconds. To aid in resin cement bonding to the conditioned zirconia samples, a polyethylene microtubule of 1.5 mm internal diameter was cut to a height of 2 mm using a sharp blade from a 6-FG Nelaton catheter and positioned in the center of each sample. The dual-cured, MDP-containing resin cement (TheraCem®, BISCO, USA) was then carefully packed into the microtubule, allowed to sit for 2 seconds, and light-polymerized at a light intensity of 21200 mW/cm²for 20 seconds. The microtubules were then carefully removed from the resulting resin cylinders using a sharp scalpel.

Microshear bond strength test (μSBS):

After a 24-hour period at room temperature, every resin-zirconia bonded sample was attached to a universal testing machine (Instron model 3345, England). A 0.14-inch diameter stainless-steel wire was looped around the sample, making contact with half of its circumference at the resin/zirconia interface. The sample was then subjected to a microshear force at a speed of 1 mm/min until the resin/zirconia interface detached. The machine software (BlueHill 3, Instron, England) calculated the SBS by dividing the force required for debonding (Newtons) by the surface area (mm²).

Failure-mode examination:

A stereomicroscope was used for examining the debonded surfaces of samples at 30X magnification, categorizing failure modes as follows: (A) failure at the interface between zirconia and resin (adhesive failure); (B) failure within resin (cohesive failure); and (C)a combination of both adhesive and cohesive failures (mixed failure,). Subsequently, the failure rates for each group were evaluated, and one randomly selected sample from each group was further examined using SEM at a magnification of 1000X.

Statistical analysis:

The statistical analysis was conducted using SPSS 2011 (Statistical Package for Social Science, IBM, USA). Following the Kolmogorov-Smirnov test and Shapiro-Wilk's test results, it was determined that the data exhibited a normal distribution and were presented as mean plus standard deviation (SD). One-Way ANOVA was used to assess differences between various evaluations. The significance level was established as ($P \leq 0.05$).

RESULTS:

Microshear stress analysis:

The highest mean value of microshear stress was recorded in the 10W group(M=13.27/SD=4.63 MPa), followed by the sandblasting group (M=12.45/SD=1.69 MPa), then the 6W group (M=11.70/SD=3.61 MPa), However the 8W group recorded the least mean value (M=11.18/SD=5.76 MPa). One Way ANOVA test revealed that the difference between the groups was not statistically significant as the recorded p-value= 0.902

Table (1): Mean values recorded in the study groups:

Study groups	Mean (MPa)	Std. Deviation	Std Error	Min.	Max.	Confidence interval		F-Value	P-Value
						Lower	upper		
Sandblasting Group	12.45	1.69	0.85	10.81	14.75	9.74	15.15	0.17	0.902
6W group	11.70	3.61	1.80	7.95	15.35	5.95	17.44		
8W group	11.18	5.76	2.88	6.29	18.09	2.01	20.34		
10W group	13.27	4.63	2.32	8.95	19.76	5.89	20.64		

SEM analysis of the impact of surface treatments on zirconia samples:

SEM observations of sandblasting-treated samples at 10,000X magnification showed the existence of scratches and edge-shaped microretentions when compared to untreated samples. Laser-treated samples, on the other hand, had less roughening or none at all compared to sandblasting samples.

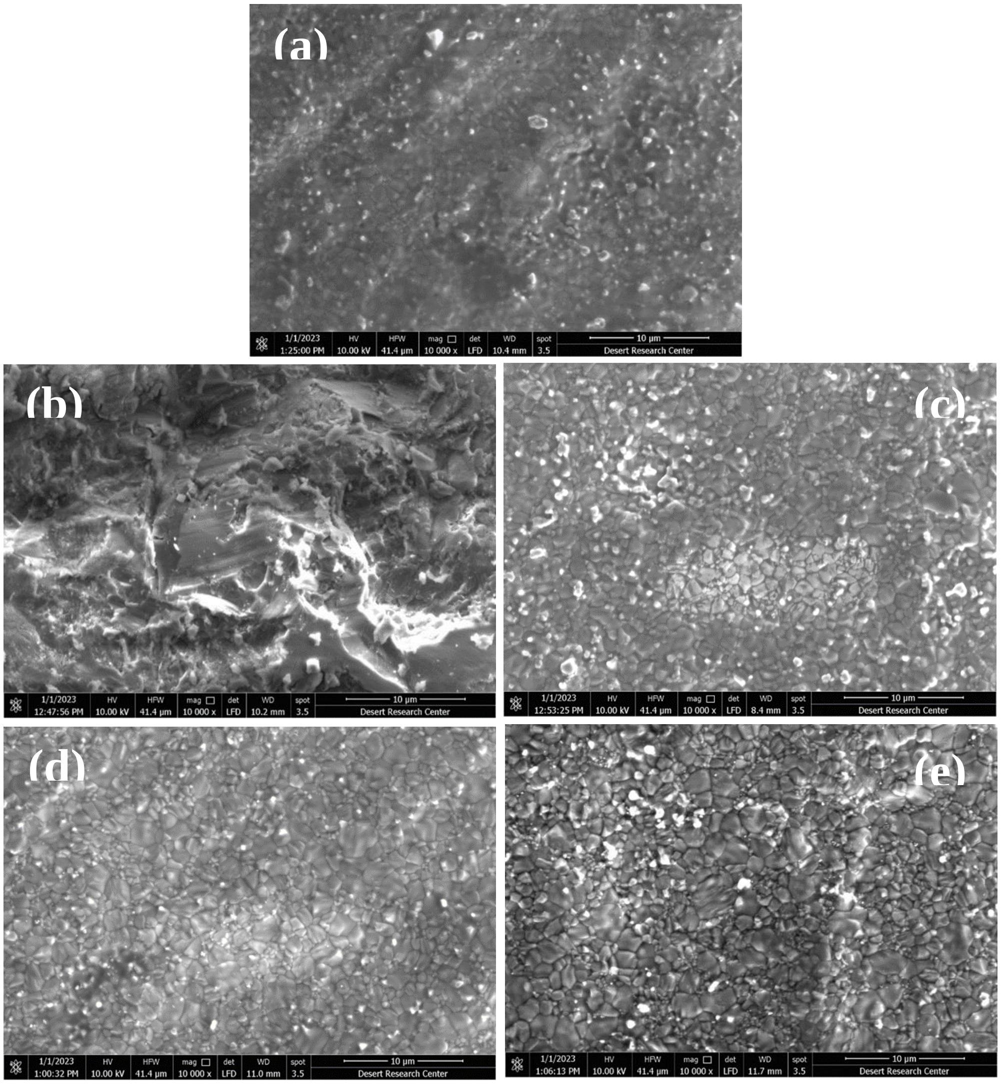

Figure (3): SEM of Cerconxt zirconia surface at 10,000 X magnification. (a) Untreated, (b) treated by sandblasting, (c) Er,Cr:YSGG laser at 6W, (d) Er,Cr:YSGG laser at 8W, (e) Er,Cr:YSGG laser at 10W.Compared to the control and laser-treated groups, the surface of the sandblasting group is noticeably rougher.

- **Failure mode analysis using SEM:**

i. *Group SB:*

At 1000X magnification, the debonded sample, Figure (4,b), showed areas of different surface morphology than that of the sample right after the surface treatment and before cementation, Figure (4,a). This indicates that a portion of the resin cement remained adhered to the zirconia surface and that the mode of failure for group SB was a mixed failure.

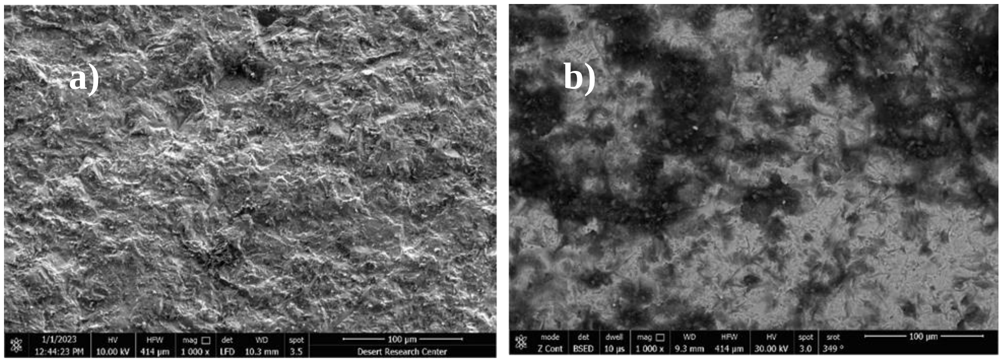

Figure (4): A sample of group SB right after surface treatment and before cementation (a), and post µSBS testing (b).

ii. Groups 6W, 8W, and 10W:

At the same magnification, the debonded samples of groups 6W,8W,10W **Figure (5b,d,f)** respectively, showed almost the same surface morphology of that of the samples right after the surface treatment and before cementation, **Figure (5a, c, e)**respectively. This indicates that the mode of failure for laser groups was an adhesive failure.

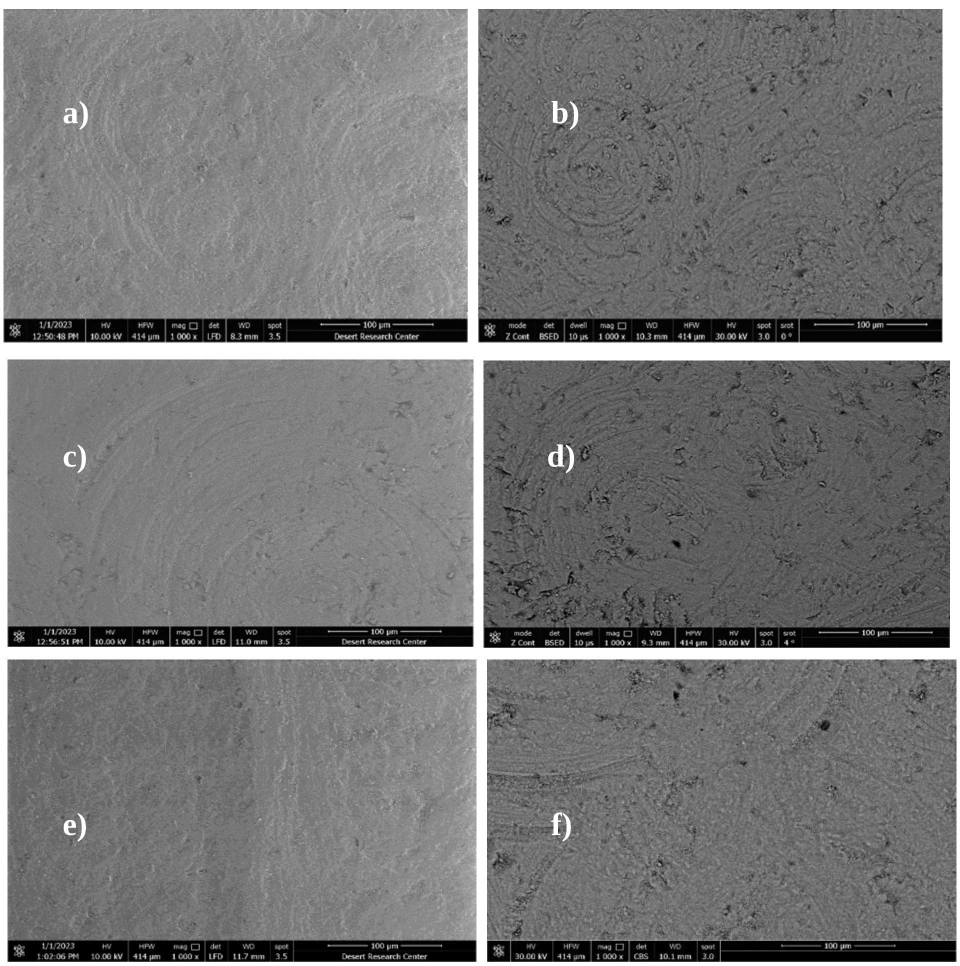

Figure (5): A sample of group 6W right after surface treatment and before cementation (a), and post µSBS testing (b). A sample of group 8W right after surface treatment and before cementation (c), and post µSBS testing (d). A sample of group 10W right after surface treatment and before cementation (e), and post µSBS testing (f).

DISCUSSION:

In this study, various surface treatments for zirconia were investigated to improve bonding with resin cement, with effectiveness assessed by measuring microshear bond strength. The highest mean microshear stress was observed in the 10W group (M=13.27/SD=4.63 MPa), followed by the sandblasting group (M=12.45/SD=1.69 MPa) and the 6W group (M=11.7/SD=3.6 MPa). The 8W group had the lowest mean value (M=11.18/SD=5.76 MPa), but no statistically significant differences were found among the groups. Consequently, the null hypothesis was confirmed.

The outcomes of the µSBS test revealed that laser-treated surfaces showed comparable results to sandblasting surfaces, with 10W group even showing higher values. This outcome aligns with findings from a prior study by **Saade et al., 2020**[9] in which the authors discovered that Er,Cr:YSGG laser surface treatment for 4Y-PSZ samples at 5.5W output power showed no significant difference in microshear bond strength values with resin cement when compared to grit blasting with 50 microns alumina particles and selective infiltration etching. Nevertheless, **Kara, 2020**[11]reported that irradiating a 4Y-PSZ zirconia surface with an Er,Cr:YSGG laser at 2W output power resulted in higher shear bond strength values with composite resin than sandblasting, with evident significant difference between the two groups.

On the contrary, **Ghasemi et al., 2014** [12]**and Akhavan Zanjani et al., 2015** [13]reported that sandblasting 3Y-TZP zirconia samples with 50 microns alumina particles resulted in higher µSBS values with resin cements than irradiating similar samples with Er Cr: YSGG laser at 2W and 3W, with a significant difference between the sandblasting and laser treatments. Also, **Aras *et al*., 2016**[14]irradiated 4Y-TZP samples with Er,Cr:YSGG laser at a power of 3W and compared it to tribo-chemical coating and sandblasting with 50 microns alumina particles, concluding that laser irradiation was insufficient to increase the µSBS between zirconia samples and resin cement. The observed discrepancies in these studies' findings could potentially be attributed to variations in the generations of zirconia employed, as well as the utilization of distinct laser parameters and various air abrasion techniques within the experimental setups, reflecting the inherent variations and heterogeneity in the methodologies employed across these studies.

SEM images post debonding revealed that the mode of bond failure between resin cement and zirconia was almost always of the adhesive type in the majority of the laser-treated samples where debonding was exclusively observed at the interface between resin and zirconia. On the other hand, the mode of failure in the sandblasting group however was predominantly mixed. These findings are consistent with other studies; **Ghasemi et al. 2014, Aras et al. 2016, and Saade et al. 2020**[9,12,14]which demonstrated that samples treated with CO_2, Er:YAG, and Er,Cr:YSGG lasers exhibited only adhesive failure mode, whereas sandblasting with 50 microns alumina particles showed mixed failure pattern.

Concerning the surface irregularities obtained post the proposed surface treatments in this study, SEM images revealed that sandblasted zirconia samples had more micromechanical roughening and irregularities than other groups. This finding has been also highlighted by previous studies; **Akyil et al. 2010, Akin et al. 2015, and Akhavan Zanjani et al. 2015**[13,15,16], in which the authors have reported that the CO2, Er:YAG and Er,Cr:YSGG lasers could create less roughening to zirconia surface when compared to sandblasting. This can pretty much explain the mode of failures observed in the tested groups. Unlike laser irradiations, sandblasting created more surface irregularities on the sample's surface where the resin cement was to lock and bond to micromechanically. Nevertheless, there was no significant rise in bond strength linked to this finding.

The limitation of the present study was that artificial aging methods to simulate oral conditions were not included. Zirconia-resin-cement bond can be significantly degraded by oral mechanical stresses, temperature, and humidity due to the susceptibility of zirconia ceramics to low-temperature degradation, which can, in turn, affect the material's mechanical behavior and bond strength. However, in this study the primary aim was to investigate the µSBS of 5Y-TZP with resin cement immediately post surface treatment without the influence of any other factor, given that this was the first time, to test zirconia surface treatment using higher levels of Er,Cr laser power settings.

CONCLUSIONS:

In the context of the current research, the following aspects can be deduced:

1) 6W, 8W, and 10W powers of Er,Cr:YSGG laser can be considered as surface treatment alternatives for enhancing the bond strength between resin cement and extra translucent zirconia.
2) Sandblasting using 110 µm Al_2O_3 particles can increase the surface irregularities of extra translucent zirconia in order to enhance its bonding with resin cement through micromechanical retention.

RECOMMENDATIONS:

1) Using artificial aging such as thermo-cycling and water-storage to further assess zirconia-resin bond strength following the surface treatments tested in this study.
2) Increased power levels of laser groups (6W, 8W, and 10W) can lead to elevated temperatures on zirconia surface, ultimately leading to a t-m phase transformation. The impact of the proposed surface treatments on the amount of monoclinic phase needs to be assessed.
3) Combining laser surface treatment with sandblasting as a dual-modality approach may enhance surface roughness and bonding properties. The effects of this combination on the bond strength and phase transformation should be evaluated.

Conflict of interests:

The authors do not have any conflicting interests pertaining to this article.

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Ethics:
This study was carried out according to the recommendation and approval of the ethics committee on in-vitro research of Faculty of Dentistry, Cairo University. No. of approval: 4 - 6 – 21.

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