Iraqi J. Laser, Issue 1, Vol., 20, pp.21-29 (2021)

IRAQI JOURNAL OF LASER

Pulsed Er,Cr:YSGG Laser For Surface Modification of Dental Zerconia Ceramic

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(Received 11/2/2021; accepted 26/5/2021)

Abstract*:* **Background:** Surface treatment of machined dental zirconia for enhancement of the adhesion to resin cement, using Er,Cr:YSGG Laser. **Materials and Methods:** Total number of 42 zirconia disc specimens (9 mm diameter, and 2 mm height) was sintered according to the manufacturer instruction. They are divided into six groups, each group of seven samples. Laser groups (Experiment parameters) were depend on laser total irradiation time, pulse duration, and power. Group (A): 20 sec., 60 µs pulse duration. Group (B): 30 sec., 60 µs pulse duration. Group (C): 40 sec., 60 µs pulse duration. Group (D): 20 sec., 700 µs pulse duration. Group (E): 30 sec., 700 µs pulse duration, with different powers used (1, 1.50, 2, 2.50, 3, 3.50, 4) W. And finally group (N): untreated samples representing the control group. Luting cement was applied to laser treated zirconia sample's surfaces and cured by light for 40 s. Then all sample surfaces were examined under a stereo microscope, for determining the depth mean values of the created pulse holes, for each specimen. The bond between the laser treated zirconia surfaces and the applied resin cement, were examined for all samples by using a universal testing machine, for determination of the shear bond strength values. Six additional specimens: one untreated sample and five other samples that were laser irradiated with the parameters that exhibited the highest shear bond strength values in each treated group, were examined for their average surface roughness by using an atomic force microscope. Another similar six specimens were also examined for their surface morphology, by using a scanning electron microscope. Both surface examination methods were performed by specialized operators. The bond failure modes were also examined by an optical microscope. Results obtained from shear bond strength test and laser pulse depth examination were statistically analyzed. **Results:** There was significant differences in the shear bond strength values, indicating a clear increase in zirconia-resin cement bonding, especially in group B (30 s, 60 µs, 4W), reaching (8.63 Mpa). Whereas, the control group had (4.49 Mpa), and an enhanced zirconia surface average roughness, that group E (30 s, 700 µs, 3.5 W) specimen had the highest value (14.5nm), among the six examined specimens. While the control specimen had the least value examined which is (3.05nm). **Conclusion:** A relation between the cement bonding efficiency and the laser treated zirconia surface roughness, laser pulse depths was detected. The Er,Cr:YSGG laser pulse duration and power is crucial parameters in the surface roughness enhancement of the zirconia ceramic.

Keywords: Er:Cr:YSGG Laser, zirconia surface treatment, Shear Bond Strength.

1. Introduction

A popular dental restoration that becomes the technique of choice is all-ceramic restorations (Zarone 2016). One of the most frequently used all-ceramic core material for fixed dental restorations is yttrium-stabilized-tetragonal-zirconia-polycrystal (Y-TZP) (Cavalcantit 2009). The clinical long-term success of Y-TZP depends on bonding strength and durability of the dental cement to ceramic substrates and teeth prepared surface that will be integrating all system parts into a coherent structure for enhancing the bonding strength of all ceramic dental restorations (Burke 2002). A strong cement-ceramic bond requires micromechanical interlocking and/or chemical bonding, via surface roughening process of ceramic surface (Borges 2003). Polycrystalline structure lacks a glass matrix making zirconia ceramic a more hydrofluoric acid-etch resistant (Guazzato 2005). Air-borne particle abrasion (Kara 2011), sandblasting, diamond bur grinding (Yoo 2015), zirconia primers, tribochemical silica coating, are widely used conditioning techniques (Valandro 2008). Airborne particle abrasion imposes significant amount of material removal, affecting the clinical adaptation. Tribochemical silica-coating system has been criticized for zirconia subcritical crack propagation possibility in thin restorations cases (Zhang 2013). In dentistry, Lasers were employed for different purposes, including restorative surfaces conditioning (Fischer 2008). Including: Nd: YAG (Kasraei 2015), Er: YAG (Kasraei 2014), CO2 (Sofi 2018), Er:Cr: YSGG (Yanardag 2018), The current study aims for investigating the effect of surface roughness on the shear bond strength (SBS) using different power parameters of Er,Cr: YSGG laser

Figure (1): Milled zirconia disc specimens.

2. Material and method:

In this experimental study, 42 discs were milled from presented zirconium oxide blocks (vita YZ HT zahnfabrik/Germany), and sintered in special furnace (Zirkonzahn, Oven600/V2, South Tyrol) at 1450 °C for 8 hours including cooling, following the manufacturer's instructions. The obtained disc dimensions were (9 mm in diameter, 2 mm in thickness) figure (1).

All bonding surfaces of zirconia discs, were polished, with water cooling, by 600, 800, 1000, and 1200 grit silicon carbide abrasive papers, consecutively, For standarization. All specimens were ultrasonically cleaned in distilled water and 7o% alcohol for 5 min. Then examined under an optical microscope (OM) at 40X magnification, for cracks or fissures, and substituted by other perfect samples when needed.

3. Specimens grouping:

42 zirconia discs were randomly divided into six groups, each of seven samples. group (L): Serve as control group of untreated samples. The laser treated groups are: group (A): 20 s, 60 µs pulse duration, group (B): 30 s, 60 µs pulse duration, group (C): 40 s, 60 µs pulse duration, group (D): 20 s, 700 µs pulse duration, and group (E): 30 s, 700 µs pulse duration. For different used powers: (1, 1.5, 2, 2.5, 3, 3.5, 4) W. Each laser irradiation parameter was repeatedly performed on two additional specimens for each sample in the laser treated groups, in order to find the mean value of the shear bond strength for each applied parameters

Each zirconia disc bonding surface was irradiated with Er,Cr: YSGG laser λ= 2,780 nm (iPlus, Waterlase, Biolase Technologies Inc., Irvine, CA, USA) at Hz: 50 (pilot study), water/air level: 65/ 55 % (Barutcigil 2019) after being fixed in a teflon mold. 600 µm quartz core tip, put at 1 mm distance (Kara 2020), and at an angle 90^0 with the sample horizontal surface, held manually to simulate technician's roughening process, that same processing was performed with all specimens as a standerization. Laser energy was delivered at the middle of each disc specimen in a circular area of 6 mm diameter, as five separated irradiation spots, creating five laser pulse microholes with varying depths. Then each zirconia disc were horizontally embedded in an acrylic mold with only 0.5 mm of the remaining height was left to be exposed for cementation procedure, figure (2). For the purpose of facilitating the SBS testing procedure, a silicon mold was constructed for molding a cylindrical adhesive resin cement to be lutted over the irradiated sample surfaces. A Silicon mold with a central circular opening of 5 mm diameter, figure (3) was positioned over the acrylic mold, while ensuring the circular opening being centered over the zirconia disc.

Figure (2) Zirconia discs were embedded in an acrylic mold

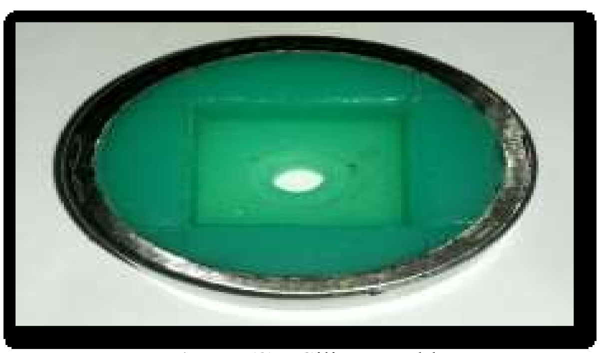

Figure (3): Silicon mold.

Figure (4): resin cementation.

Adequate amount of adhesive cement (Rely X U200 self-adhesive resin cement, 3M ESPE, Germany) was delivered into the silicon mold opening and photo polymerized by using a light curing device (Astralis5, Ivoclar Vivadent, iechtenstein) for 40 s, following manufacturer's instructions figure (4). One hour later, specimens were stored in distilled water at 37 °C for 24 hours before the shear bond strength (SBS) test.

All tested samples were examined under (OM) for determination of the five laser pulse depths mean (LPD) present at each sample surface (in micrometer at 40X mag.). Then it is subjected to shear force at the zirconia-cement interface with a universal testing machine (Instron, England) for determination of SBS. The SBS values evaluated in Mpa. Then, six samples (A: Untreated sample), and five Er,Cr:YSGG laser treated samples: B: 20s/60µs/4W. C: 30s/60µs/4W. D: 40s/60µs/3.5W. E: 20s/700µs/3.5W. F: 30s/700µs/3.5W). They showed the highest SBS values from each laser group, underwent surface roughness analysis by the atomic force microscope (AFM) (AA3000, Anstgrom Advanced. inc. USA), for an average surface roughness (Ra) evaluation, as shown in table (1). All deboned samples, of the SBS test, were evaluated under a stereomicroscope (SM) (ME, 2665, Euromex, Holland) at 40X mag, for fracture mode determination of zirconia-cement interface . The modes of failure were classified as: (1) Adhesive: de-bonding only at the cement-ceramic interface. (2) Cohesive: a rupture in the cement or zirconia ceramic. (3) Mixed (Yoshida 2006): shows both adhesive and cohesive failure modes.

4.Statistical Analysis:

This analysis was chosen depending on the needed comparison for the obtained results. It was done with SPSS software version 23/France. SBS and laser pulse depth (LPD) Data were analyzed by a one-way ANOVA-test (analysis of variance) to calculate the P-value between control and tested groups. LSD test was used to calculate the significant differences between tested means.

5.Results:

5.1 AFM surface roughness analysis:

The three-dimensional (3D) roughness measurements of different laser-treated specimens are shown in figure (5). Specimen (30 s, 700 µs, 3.5 W) showed the highest Ra (14.5 nm) value. Ra was increased with the increased laser power for all the laser groups, with the increased number of laser pulses per sec., And with the elongated laser pulse duration time (700 µs).

Table (1) demonstrates the average surface roughness values for the untreated specimen and for the five laser treated specimens that showed the highest SBS values in each laser group. Table (2) and (3) presenting the descriptive statistics of SBS, and the statistical analysis of SBS and LPD for the comparison among laser groups.

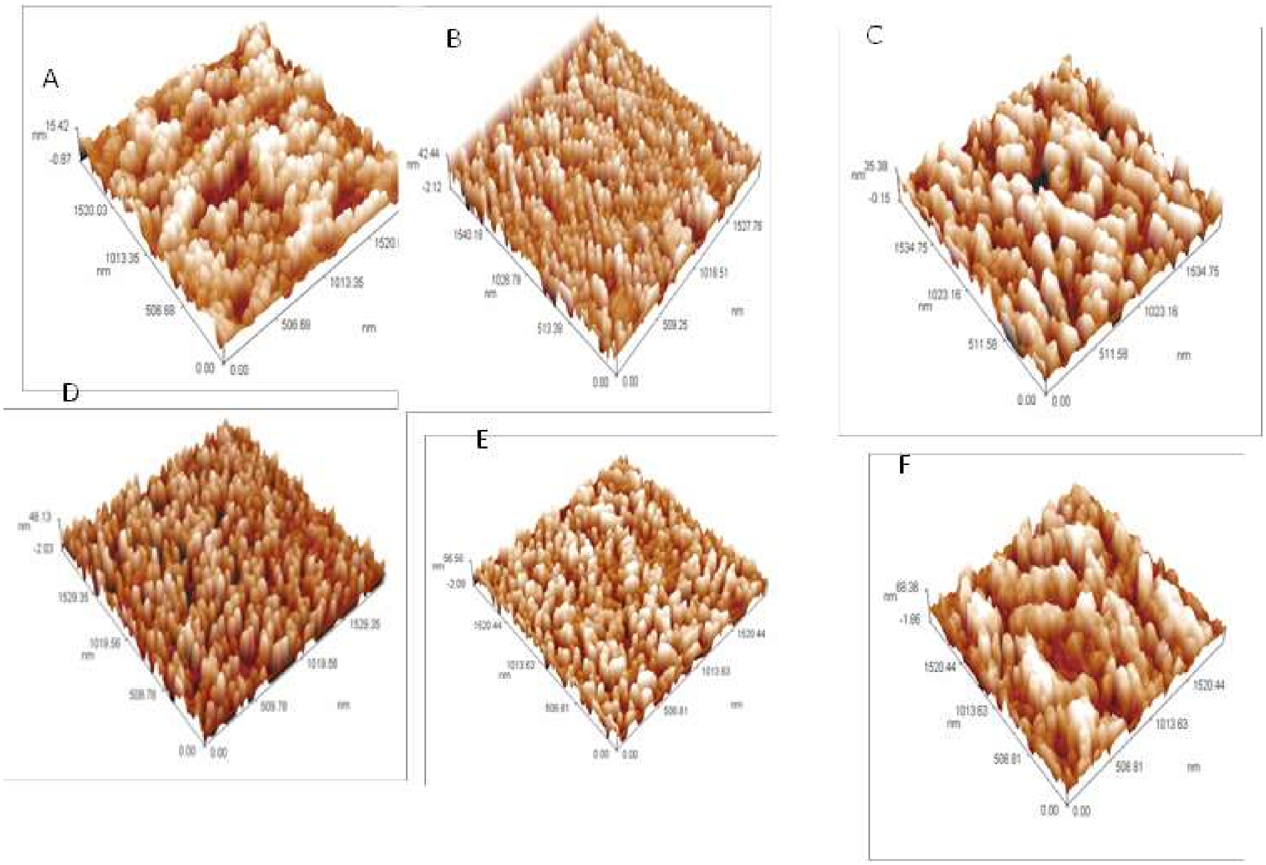

Figure (5): (3-dimentional AFM pictures). A. Untreated specimen (Ra = 3.05 nm). Er,Cr:YSGG laser treated specimens: B. (20 s / 60 µs / 4 W) Ra = 7.73 nm, C. (30 s / 60 µs / 4 W) Ra = 8.88 nm, D. (40 s /60 µs / 3.5 W) Ra = 10.2 nm, E. (20 s /700 µs / 3.5 W) Ra = 12.4 nm, F. (30 s / 700 µs / 3.5 W) Ra = 14.5 nm

Table (1): Average Surface Roughness Of Six Examined Specimens

Experiment groups	Specimens for the applied power	Average surface roughness (nm)
Control group		3.05
Group A (20sec.,60 µs)	4 W	7.73
Group B (30sec., 60 µs)	4 W	8.88
Group C (40sec.,60 µs)	3.5 W	10.2
Group D(20sec.,700µs)	3.5 W	12.4
Group E(30sec., 700µs)	3.5 W	14.5

The specimen treated with Er:Cr: YSGG laser (30s, 60µs, 4W) group exhibited the highest SBS means. The SBS mean values increased with increasing the laser power for 60µs groups. Table (2) clearly shows an increase with LPD for all groups with the power increase, except for group D. The specimen (4W, 30s, 700µs) group had The highest LPD mean and it was non-significant with the specimen; (4W, 30s, 60µs) group, however, it's SBS value was not increased with the depth increasing.

Table (2): Shear Bond Strength Measurement of Control and Laser Groups.

Power /W	20 s /60 µs		30 s / 60 µs		40 s /60 µs		20 s /700 µs		30 s /700 µs			
1	C 5.24	0.20	C 6.26	0.12	C 5.94	0.41	6.84	0.33	5.79	0.91	0.01	NS
1.5	C 5.30	0.13	B 6.76	0.30	C 5.44	0.10	6.80	0.68	6.49	0.43	0.01	NS
2	C 5.79	0.25	C 6.44	0.30	B 6.14	0.15	6.74	0.00	6.53	0.17	0.001	NS
2.5	C 5.86	0.42	C 6.33	0.46	B 6.24	0.03	6.51	0.33	6.59	0.11	0.001	NS
3	B 6.16 d	0.03	B 6.86 b	0.37	A 6.56 c	0.00	6.91b	0.42	7.44 a	0.54	0.0001	0.05
3.5	B 6.79 c	0.05	B 6.96 b	0.00	A 6.99 b	0.20	7.05 b	0.20	7.64 a	0.02	0.0001	0.01
4	A 7.23 b	0.14	A 8.63 a	0.13	C 5.74 d	0.00	6.94 b	0.02	6.48 c	0.19	0.0001	0.001
Control	4.49	0.16	4.49	0.16	4.49	0.16	4.49	0.16	4.49	0.16		
P value of C VS L.G.	0.0001		0.0001		0.0001		0.0001		0.0001			
P value of L.G	0.01		0.01		0.001		NS		NS			

C VS L.G: P value between control and tested laser groups. L. G: P value between the tested groups. LSD test was used to calculate the significant differences between tested means. std. Error calculated from laser irradiation of three samples with same parameters for each applied power. The letters (A, B, C, and D for column and a, b, c and d for rows) represent levels of significance. highly significant start from the letter (A or a) and decreasing with the last letter. Similar letters mean there are no significant differences between tested mean.

- Non-significant at P> 0.05.
- Significant at P≤ 0.05.
- Highly significant at P≤ 0.01.
- Very high significant p≤0.001

5.2 Failure Mode

The frequency of the failure modes after the SBS test for each group is shown in table (3). The results indicated that the failure modes of the groups varied with the different laser parameters used. In group A, C type (1) failure mode was frequently observed in those specimens treated with 1-3 W for both groups. On the contrary, failure mode of type 3 was mostly detected in group D, E except for (2, 2.5) W and (1, 1.50) W respectively. Type (2) had the least frequency, and observed in group B (4W) and E (3.5) W as shown in table (4).

Table (3): Laser Pulse Depth Measurements of Laser Groups.

Power/W	20 s/60 µs Pulse depth Mean /µ	20 s/60 µs Std. Error	30 s / 60 µs Pulse depth Mean/ µ	30 s / 60 µs Std. Error	40 s /60 µs Pulse depth Mean/ µ	40 s /60 µs Std. Error	20 s /700 µs Pulse depth Mean/ µ	20 s /700 µs Std. Error	30 s /700 µs Pulse depth Mean/ µ	30 s /700 µs Std. Error	P value
1	D 1.70 c	0.40	E 2.10 b	0.30	F 4.50 a	0.35	2.67 b	0.69	C 2.00 b	0.20	0.05
1.5	C 2.30 d	0.21	D 4.20 a	0.15	E 3.90 b	0.10	3.40 c	0.00	C 2.30 d	0.10	0.01
2	C 2.00 d	0.15	D 4.80 b	0.56	D 4.50 b	0.12	3.70 c	0.40	B 6.20 a	0.21	0.01
2.5	C 2.50 d	0.12	D 4.70 b	0.53	C 6.2 a	0.72	3.50 c	0.29	B 6.30 a	0.56	0 1
3	B 3.00 d	0.00	C 6.00 c	0.23	B 7.50 a	0.20	3.50 d	0.25	B 6.50 b	0.12	0.001
3.5	A 4.10 c	0.51	B 6.90 b	0.06	A 8.00 a	0.58	3.30 d	0.15	A 7.90 a	0.17	0.001
4	B 3.30 c	0.65	A 8.00 a	0.21	B 7.70 b	0.35	3.70 c	0.12	A 8.20 a	0.12	0.0001
P val	0.01		0.001		0.001		NS		0.001		

Table (4): Failure Mode Distribution.

Laser groups	Adhesive Failure -1-		Cohesive Failure -2-		Mixed Failure -3-	
	No.	%	No.	%	No.	%
A (20 s, 60 µs)	5	50			2	20
B (30 s, 60 µs)	3	30	1	10	3	30
C (40 s, 60 µs)	5	50			2	20
D (20 s, 700 µs)	2	20			5	50
E (20 s, 700 µs)	2	20	1	10	4	40

6. Discussion:

The aim of the current study is SBS enhancement of zirconia ceramic material to resin cement, without laser optical damage to zirconia surface such as crack or fracture. Many laser studies attempted investigation the effect of laser type, pulse duration; zirconia-surface laser irradiation regarding temperature elevation, zirconia ceramic type, and resin cement type, on SBS, and found conflicting results. Laser power is an important parameter in laser-material processing, that the current study aims investigating its influence. Laser energy absorption by the material's surface is the most important interaction between laser and material. Therefore, it is crucial to choose suitable laser parameters for surface treatment to achieve a desired zirconia surface changing (Kasraei 2014). Surface roughness is an important factor and could increase bonding strength values (Moon 2011). (Aboushelib2014) stated that, use of high energy pulsed Er,Cr:YSGG failed to increase the bonding strength to ceramics. The optained results showed that samples treated with Er,Cr: YSGG laser showed increased surface roughness compared with untreated zirconia samples. Regarding the 60 µs groups, the highest SBS mean value was with (4 W, 30 s) Er,Cr:YSGG laser treated specimen: (8.63 Mpa) of Ra value (8.88 nm) and LPD (8µ). While for the 700 µs groups, the highest SBS mean value was for (3.5 W, 30 s): (7.64 Mpa) of Ra value: (14.5 nm) and LPD (8.2µ). Same result found with (Gomes et al 2015) who stated an increased bonding strength values after Er:YAG laser irradiation. Comparing the results of the two (60, 700 µs) groups, the highest Ra and LPD values were for the 700 µs group, and not accompanied by SBS increasing. This could probably be due to the effect of Er,Cr: YSGG laser long pulses in roughening the zirconia bonding surface with lateral laser-heat dissipation, contributing to zirconia structure morphological changes beyond the LPD periphery, with temperature elevation in the laser affected zone, that the low powers capacity would not be effective, and the higher powers would have a loss in their heat confinement and damaging effect for the material, presenting high LPD, and increased Ra values of weakened resin-cement interlocking, as shown from the resulted SBS values.(answer for Conclusions , but it had no enhancement on the bonding strength. These findings were in good agreement with (Alhassani 2017) Who reported that surface roughness increased with increasing power and pulse duration but it had no enhance on bond strength. And with (Aras et al 2016) who reported that the increased surface area could increase adhesion but bond strength values were not higher after laser irradiation. The produced zirconia surface effect differs from the shorter pulse duration surface effect. The effect of Er,Cr: YSGG focused irradiation in ablating zirconia surface particles with concentrated, short pulse durations (60 µs) created no lateral laser-heat dissipation, nor material damage. The LPD strongly influenced by the power increase. The applied high power (4W) and the long laser exposure time (30s) facilitated for an efficient laser-zirconia ceramic interaction and later on, ablation of deeper layers of material's structure forming well defined holes of much increased depth as compared to results of samples irradiated with other laser parameters for same 60 µs group. The results increase in surface area, allowing for larger exposure areas (LPD) of zirconia surface to resin cement, facilitating resin flow and settling to be photo polymerized into a strong resin-zirconia bond with enhanced mechanical properties, thereby increasing the SBS. Same finding were with (Kunt 2018) who concluded that only 4 W CO2 laser irradiation for Y-TZP ceramics is recommended as an alternative surface treatment to sandblasting. These results disagreed with (Miranda 2015), who examined the surface roughness, on the Y-TZP surface after Er: YAG laser irradiation at 1.5 W/20 Hz and concluded that laser irradiation caused a decrease in surface roughness. This could probably be due to the fact that the author had used different laser parameters.

7. Conclusions:

The derived results had reached the following conclusions: The highest SBS of the Er,Cr:YSGG laser-irradiated specimens are obtained with the laser parameters of the 30s, 60µs, 4W. Higher surface roughness values were with the increased power and pulse duration time, but it had no enhancement on the bonding strength. The laser power and pulse duration are both vital parameters in the surface roughness of zirconia ceramic for enhancement of the bonding strength to the resin cement.

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الليزر النبضي Er,Cr :YSGG لتعديل سطح زركونيا CAD-CAM

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الخلاصة: الخلفية: تعزيز سطح زركونيا CAD-CAM لزيادة المطابقة مع الاسمنت الراتنج باستخدام ليزر Er,Cr :YSGG
الطريقة: تم تحضير40 عينه قرص زركونيا متكلس .قد قسّمت الى ستة مجاميع كل مجموعة من سبع عينات. المعالم التجريبية تعتمد على طاقه الليزر، الزمن الكلي لتشعيع الليزر، ومدة النبضة, المجموعة (A) : (20) ثانيه، مدة النبضة (60)مايكروثانية.

المجموعة (B): (30) ثانية، مدة النبضة (60) مايكرو ثانيه. مجموعة (C): (40) ثانية، مدة النبضة (60) مايكرو ثانية. المجموعة (D): (20) ثانية،(700) مايكرو ثانية. المجموعة (E): (20) ثانيه، (700) مايكرو ثانيه، باستخدام طاقات ليزر مختلفة (4, 3.5, 3, 2.5, 2, 1.5 , 1) واط. وقد تم صب الاسمنت الراتنج الى أسطح الزركونيا المعالجة. وعولجت لمدة (40) ثانيه بواسطة الضوء. العينة ذات اعلى قراءة قوة قص من كل مجموعة، ثم تقييمها بفحص قوه اللصق بواسطه اله الاختبار (عالمية)، و بقياس معدل الخشونة السطحية بإستخدام مجهرقوة الذره. نتائج قوه القص التي تم الحصول عليها، ونتائج عمق نبضة الليزر، تم تحليلها إحصائيا .وايضا تحليل اوضاع فشل السندات. **النتائج:** كانت هناك زياده واضحة في قوة القص تحديدا في المجموعة (B): (30) ثانية، W 4، مدة النبضة (60) مايكرو ثانيه وصولاً الى (paM8.63). وتحسن في خشونة سطح الزركونيا خاصة للمجموعة (E) (30) ثانية، 700 مايكرو ثانية ، 3.5 واط) وصولاً الى 14.5 نانوميتر معدل الخشونة. **الاستنتاج:** تم الكشف عن أن قوة رابطة القص مرتبطة بخشونة السطح وبعمق نبضة الليزر. مدة النبضة و طاقة الليزر Er,Cr :YSGG هي معلمه حاسمة في تعزيز سطح الزركونيا .