Effect of Ti: Sapphire and Nd: YAG Lasers on Shear Bond Strength at the Zirconia - Veneering Ceramic Juncture

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Abstract

Introduction: This study was conducted to assess the impact of nanosecond and femtosecond lasers on shear bond strength at the zirconia - veneering ceramic juncture.

Materials and Methods: The first 60 samples of partially sintered zirconia cylindrical discs measuring 7 mm diameter and 4 mm height were milled and sintered. Then they were randomly divided into three groups namely group C (control, n=10), group N (Nd: YAG laser, n=10), and group T (Ti: sapphire laser n=40) which was further divided based on the duration of laser irradiation, into 4 subgroups (n=10 each) which were 30 seconds, 1 minute, 2 minutes and 3 minutes. Surface treatment was done on sintered zirconia discs based on the group. Following the treatment, the discs were ultrasonically cleaned followed by liner application (IPS Emax Zirliner, Ivoclar-Vivadent) and veneer ceramic layering (IPS e.max Ceram, Ivoclar-Vivadent) of 1.5 mm height. Each sample underwent shear stress in the universal test machine on the mounting jig, and bond strength was evaluated. Data were assessed using ANOVA followed by Tukey's post hoc multiple comparison analyses.

Results: According to one-way ANOVA, there was a significant difference in shear bond strength between the groups. Tukey's post hoc pair wise comparison test showed a significant difference (P value=0.001) in shear bond strength of all pairs except group C and group N. The results of repeated measures, ANOVA (related and dependent groups) and Tukey's multiple pair wise comparison test showed that there was a significant difference (P value=0.001) in shear bond strength at 30 seconds and all other groups.

Conclusion: Ti: sapphire laser irradiation for 30 seconds can be used as potential surface treatment to increase shear bond strength at the zirconia-veneering ceramic juncture.

Keywords: Shear strength; Lasers; Zirconium; Femtosecond laser; Nd: YAG laser.

Introduction

In this era of cosmetic dentistry, zirconia based dental ceramics have emerged as a leading biomaterial for implants and fixed dental prostheses. Yttrium-partially stabilized, tetragonal zirconia polycrystal (Y-TZP) has excellent mechanical properties namely high flexural strength (900-1200 MPa), elastic modulus of 201 GPa, fracture toughness (7-10 MPa/m²), chemical durability, excellent biocompatibility, good optical behavior, low bacterial adhesion, low thermal conductivity, and high radiopacity.¹ Zirconia can be used as either a monolithic restoration or a substructure/core of bi-layer restoration.

Zirconia is inherently opaque; therefore, for optimum aesthetics and to match the translucency of natural teeth, a layer of translucent glass ceramic is veneered over a zirconia core. The veneering ceramic is secured onto the zirconia substructure by one of the two methods: the layering technique and the press technique.² An effective bond is entirely dependent on the micromechanical coupling between the veneering ceramic and zirconia. Contrary to Y-TZP, the veneering ceramic does not have high fracture toughness and the weak bond at the zirconia-veneering ceramic juncture leads to the chipping of veneering ceramics.³ The strength of ceramic

restoration is reliant on the weakest factor, either the core-veneer juncture or the veneer layer.⁴

Various studies have been conducted on the core-veneer bond strength by improving bonding potential, increasing wet-ability and increasing surface area. The use of laser irradiation for zirconia surface texturing has gained research interest.⁵ Researchers have investigated the effect of CO₂, Nd:YAG (neodymium-doped yttrium aluminum garnet), Er: YAG (erbium-doped yttrium aluminium garnet laser), Er: YSGG (Erbium-doped yttrium, scandium, gallium and garnet) and titanium (Ti): sapphire lasers on the zirconia surface.⁶⁻⁸ These lasers are used for porcelain etching by creating microcavities using superficial heat. Nd: YAG causes morphologic changes by superficial melting and random recrystallisation of the porcelain surface.⁹ Further studies are needed to ameliorate bond quality between the zirconia core-veneer juncture with special emphasis on the use of shorter pulse duration lasers. The effect of the Ti: sapphire laser on the zirconia-veneering ceramic juncture has never been evaluated and compared until now.¹⁰ The Ti: sapphire laser (Ti: Al₂O₃) is a tunable solid - state laser which emits red and near -infrared light in the wavelength range of 650 to 1180 nm.¹¹ It is an ultrafast femtosecond laser with high peak intensity causing material ionization and ejection without heat transfer to the environment.¹²

In this study, transformation-toughened zirconia was used as a substructure and the nano-fluorapatite glass veneering ceramic (IPS e.max Ceram)¹³ was used as the veneering ceramic. The effect of the Q-Switched Nd: YAG and Ti: sapphire laser as a mode of surface texturing on post sintered zirconia and its efficacy on shear bond strength at the zirconia - veneering ceramic juncture were evaluated.

This comparative in-vitro study was conducted to assess the impact of nanosecond and femtosecond laser surface treatment on shear bond strength at the

zirconia- veneering ceramic juncture and compare with the conventional method as control group without laser surface treatment.

Materials and Methods

A total of 60 samples in the form of a disc were prepared from a partially sintered zirconia blank (NexxZr, Sagemax, Ivoclar Vivadent, USA) of 98 mm diameter × 10 mm height by CAD/CAM technology. A stereolithographic (STL) model of the required dimension of 7 mm diameter by 4 mm length was designed for the study and uploaded on exocad® software (Germany). The software calculates the percentage of linear shrinkage and performs computer-controlled milling to create a zirconia disc of enlarged size. Computer-aided Milling was done (Roland Milling Machine). The connectors were removed using a micro-motor hand piece and a straight fissure tungsten carbide bur. As per the manufacturer's instructions, the milled zirconia discs were sintered in a Nabertherm zirconia sintering furnace at 1550 degrees Celsius for 8 hours' duration. The chemical composition of ceramics utilized in the study is depicted in Table 1. Zirconia core discs were randomly allocated into three groups, namely Group C (control n=10), group N (Nd: YAG Laser n=10) and group T (Ti sapphire Laser n=40). Group T was further subdivided into 4 subgroups based on the duration of laser exposure at 30 seconds, 1 minute, 2 minute and 3 minutes (each subgroup had 10 samples).

Laser Surface Treatment

Zirconia discs of Group N and T were coated with a layer of graphite using a soft-lead pencil because of the increased absorption of the laser beam on the darker surface (Figure 1).

The following LASER parameters were used in the study:

Group N:

Table 1. Chemical Composition of Ceramic Materials Used in This Study

Material	Type	Chemical Composition	Tg	Poisson	E	CTE
NexxZr T	Yttrium- stabilized zirconium oxide	ZrO ₂ ≥ 89%, Y ₂ O ₃ 4-6%, HfO ₂ ≤ 5%, Al ₂ O ₃ < 1%, and other oxides ≤ 0.2%,	1700	0.3	210	10.1 ≤ CTE ≤ 11.1
IPS e.max® Ceram	Low-fusing nano-fluorapatite glass ceramic	SiO ₂ 60–65%, Al ₂ O ₃ 8–12%, K ₂ O 6–8%, Na ₂ O 6–9%, ZnO 2–3%; CaO, P ₂ O ₅ and F 2–6%, other oxides 2–8.5%, pigments 0.1– 1.5%	495	0.24	60	9.8
IPS Emax® Zirliner	Nano-fluorapatite glass powder liquid all-round	Powder: SiO ₂ 50–60%; Al ₂ O ₃ 16–22%; K ₂ O 4-8%; Na ₂ O 6–11%, ZrO ₂ 1.5-3%, CaO 1.5-4%, P ₂ O ₅ 0.8-2%, F 0.2–1.5%; other oxides 0.1–1% Liquid: water > 94%, butandiol < 5%, Zinc chloride < 1%	645	0.22	65	10.4

Note: (% by weight), glass transitional temperature (Tg, °C), elastic modulus (E, GPa), coefficient of thermal expansion [CTE; (×10⁻⁶/°C)].

Figure 1. Graphite Coated Zirconia Disc.

Laser medium - Neodymium-doped yttrium aluminum garnet Crystal
 Wavelength λ - 1064 nm
 Mode of operation - Dual pulsed Q- Switched mode
 Optical Delivery - Articulated Arm
 Beam size - 7 mm
 Spot size - 7 mm diameter
 Energy density (Fluence) - 260 mJ/cm²
 Pulse duration - 5-10 nanoseconds
 Frequency - 10 Hz
 Duration of exposure - 30 seconds
 Scanned in non-contact mode
 Absence of air or water cooling
 Distance - 4 mm

The relationship between power and pulse width of Q Switched Nd: YAG LASER is shown in Figure 2. LASER surface treatment was performed using Q Switched Nd: YAG laser from Lutronic Spectra (Figure 3).

Group T:

LASER Medium - Titanium sapphire crystal
 Wavelength λ - 800 nm
 Mode of operation - Long pulsed mode
 Bandwidth ($\Delta\lambda$) - 22 nm
 Pulse duration - 50 femtosecond
 Laser energy - 6 mJ

Figure 2. Graph Showing Relation Between Power and Pulse Width of Q Switched Nd: YAG.

Frequency - 1 kHz
 Beam size - 18 mm
 Spot size - 7 mm diameter
 Energy density (Fluence) - ~12 mJ/cm²
 Scanned in non-contact mode
 Absence of air or water cooling

Based on the time duration of laser exposure: Subgroup 30 seconds, subgroup 1 minute, subgroup 2 minutes, subgroup 3 minutes.

The laser system used here is a Ti: sapphire laser system from Coherent Quantronix -Integra-C system (Hamden-CT-USA (Figure 4). It is a KHz system with the above parameters. A lens (f=50 cm) was used to partially focalize the laser beam.

Control group: Specimens of this group were not subjected to LASER surface treatment.

Liner Application

All specimen were subjected to an ultrasonic cleaner for

Figure 3. GNd: YAG Laser.

Figure 4. Ti Sapphire Laser.

3 minutes (APL ultrasonic cleaner CD-4800 PSI, Florida, USA). A coating of a liner (IPS e.max Zirliner, Ivoclar-Vivadent Schaan, Liechtenstein, Germany) on zirconia discs was sintered in a furnace (Programmat® P3010, Ivoclar-Vivadent Schaan, Liechtenstein, Germany) to 0.1 mm thickness.

Bonding With Veneer Ceramic

In this study, IPS e.max Ceram which is a nano-fluorapatite glass ceramic is used as a veneering ceramic over zirconia core discs. The creamy consistency dentine ceramic (IPS e.max Ceram, Ivoclar-Vivadent) was layered, condensed, and sintered for no more than three times to derive the final dimension of 1.5 mm (Figure 5).

Shear Bond Strength Testing

Specimens were placed on a customized mounting-jig¹⁴ and underwent a shear bond strength test by subjecting to shearing force in a universal testing machine (Instron-5565, Norwood, MA, USA) (Figure 6). Loading was done at a crosshead speed of 1mm/min until failure.¹⁵ A shear load is a force that produces sliding failure on a subject that is parallel to the direction of the force and coplanar with material cross-section. In this study the force was directed at the zirconia - veneering ceramic juncture. The force that produces a bonding failure or de-lamination of veneering ceramic from zirconia was noted. The observed shear force values were evaluated using ANOVA followed by Tukey's post hoc multiple comparison analysis. The data were statistically analyzed using SPSS 15 statistical package. The test was carried out at the significance level of 0.05.

Results

The descriptive statistics and one-way ANOVA to compare between the three groups are depicted in Table 2 which shows a significant difference in bond strength among the groups. As shown in Table 3, Tukey's pairwise comparison analyses depict a significant difference in shear bond strength of all pairs except the control group and Nd:

Figure 5. Veneer Ceramic Firing.

Figure 6. Shear Bond Strength Testing.

YAG. The repeated measures ANOVA test was performed among the subgroups of group T (Table 4). Tukey's post hoc pairwise multiple comparison analyses depict a significant difference between shear bond strength at 30 seconds and all the other groups (Table 5). Figure 7 denotes a graphical representation of the mean shear bond strength between group C (Control), group N (Nd: YAG), and group T (Ti: sapphire) at 30 seconds duration of laser exposure. The test shows that there is a highly significant difference (P value < 0.001) in bond strength between the groups owing to the greater mean bond strength in laser-

Table 2. Descriptive Statistics and One-Way ANOVA to Compare the Difference in Shear Bond Strength Between the Three Groups

Group	Mean	SD	Std. Error	95% CI for Mean		F	P Value
				Lower	Upper		
Control	343.7000	107.202	33.900	267.01	420.38	51.92	0.001
NDYAG	403.0000	66.2486	20.949	355.60	450.39		
Ti Sapphire	669.6000	39.1980	12.395	641.55	697.64		

Table 3. Tukey's Pairwise Comparison

Group (I)	(J) Group	Mean Difference (I-J)	95% CI		P Value
			Lower	Upper	
Control	NDYAG	-59.30000	-143.789	25.1892	0.209
	Ti Sapphire	-325.90000*	-410.389	-241.4108	0.000
NDYAG	Control	59.30000	-25.1892	143.7892	0.209
	Ti Sapphire	-266.60000*	-351.089	-182.1108	0.000
Ti Sapphire	Control	325.90000*	241.4108	410.3892	0.000
	NDYAG	266.60000*	182.1108	351.0892	0.000

Table 4. Repeated Measures ANOVA

Subgroups	Mean	SD	P Value
30 s	669.60	39.19807	0.001
1 min	405.60	88.94842	0.001
2 min	441.80	91.96956	0.001
3 min	377.40	130.17441	0.001

treated groups, especially group T for 30-second duration, than that of the control group.

Discussion

Zirconia is chemically inert, and it is difficult to get an effective bond with veneering ceramics.

The rate of failure for zirconia-based restoration was notified at 36% due to chipping, whereas metal ceramic

restoration accounted for 16%.¹⁶ Various factors that affect the core-veneer bond are surface texture, wetting, coefficient of thermal expansion, presence of surface irregularity and the chemical interlinking of bonded materials.¹⁴

LASER is light amplification by stimulated emission of radiation. Dr. Georgios Siviloglou has defined LASER as "A finite-energy Airy Beam that can bend and travel without scattering"¹⁷. LASER is precise and has the power to heal, create and destroy. Laser treatment works in non-contact mode and renders high flexibility for improving the surface texture of biomaterials, without changes in biochemical characteristics. It demonstrates minimal damage to biomaterials and possesses a very high repeatability rate.¹⁸ High level laser output energy was reported to produce many micro-cracks and the deterioration of zirconia surface.^{8,9} Low output laser

Table 5. Tukey's Multiple Pairwise Comparison

(I) Factor1	(J) Factor1	Mean Difference (I-J)	Std. Error	95% CI for Difference ^b		P value
				Lower	Upper	
30 sec	1 min	264.000*	32.577	154.404	373.596	.000
	2 min	227.800*	32.686	117.837	337.763	.000
	3 min	292.200*	39.242	160.181	424.219	.000
1 min	30 s	-264.000*	32.577	-373.596	-154.404	.000
	2 min	-36.200	44.827	-187.008	114.608	1.000
	3 min	28.200	60.747	-176.165	232.565	1.000
2 min	30 s	-227.800*	32.686	-337.763	-117.837	.000
	1 min	36.200	44.827	-114.608	187.008	1.000
	3 min	64.400	54.115	-117.652	246.452	1.000
3 min	30 s	-292.200*	39.242	-424.219	-160.181	.000
	1 min	-28.200	60.747	-232.565	176.165	1.000
	2 min	-64.400	54.115	-246.452	117.652	1.000

Figure 7. Graphical Representation of Mean Shear Bond Strength.

energy has the least deleterious impact and deserves further evaluation. The surface treatment of Y-TZP is done to increase the surface roughness at the microscopic level achieving high surface energy and enhanced wetting ability.¹⁹

In this study, the Nd: YAG laser is used, generating a light beam in the near-infrared region of the electromagnetic spectrum at 1064 nm. The primary property of laser energy is the transformation of light into heat. The Nd: YAG laser operates in both pulse and continuous modes. The pulse mode can further be long-pulse (minimum pulse width of 10 milliseconds) and Q Switched dual pulse mode (minimum pulse width of 5-10 nanoseconds).

In this study, the Nd: YAG laser in Q switched dual pulse mode is used because of its short pulse width and high peak power for surface treatment on zirconia core discs with minimal collateral thermal damage, and it produces selective photo thermo-lysis.²⁰ In this study, the laser surface treatment of zirconia core discs was done after sintering (post-sintered). Spohr et al²¹ and Akyil et al²² studied the effect of this laser on post-sintered zirconia, whereas Kirmali et al.^{3,23,24} evaluated its effect on pre-sintered zirconia.

Ebeid et al²⁵ assessed surface treatment on pre- and post-sintered zirconia and found increased surface roughness and bond strength in the post-sintered group. Kurtulmus et al¹⁶ studied the phase transformation of zirconia after surface treatment before and after sintering. They observed increased monoclinic phase content in the post-sintered group by X-ray diffraction analysis. They concluded that there is reverse phase transformation (monoclinic to tetragonal) of zirconia after thermal treatment, and temperature simulates the firing of the veneer.

Usumuz et al¹⁵ noticed the enhanced surface roughness of zirconia with short pulse duration (180 microseconds) when compared with long pulse duration (320 microseconds). The reduction in pulse duration caused an increase in the peak power of the laser beam and this caused higher surface roughness and bond strength.

Liu et al²⁶ studied the outcome of the Nd: YAG laser on

the zirconia ceramic by altering output power (1 W, 2 W, 3 W) and irradiation time (30, 60 and 90 seconds). An increase in output power or irradiation time was supposed to enhance the bond strength of the ceramic but the effect was opposite in this study. The irradiation time for the current study is kept at 30 seconds in accordance with that study. Air and water cooling were not used because water causes increased material deterioration as it acts as a medium for laser energy absorption and produces lower bond strength.¹⁴ The release of energy causes conditioning on the coated surface, while removing graphite as the zirconia surface is scanned with the fiber-optic tip.¹⁴

The result of this study can be explained by the fact that Laser causes pinpoint micro-explosions at the surface of zirconia leading to increased surface roughness. The high peak power and ultra-short pulse width (5-10 nanoseconds) causes increased shear bond strength of zirconia. This is in concurrence with the works done by Usumuz et al¹⁵ and Liu et al.²⁶ The result of my study does not match with the study done by Akyil et al.²² because of air and water cooling.

Erdur and Basciftci²⁷ assessed the consequence of the Ti: sapphire laser rendering a more homogeneous ablation pattern on the feldspathic porcelain surface in comparison with Er: YAG and Nd: YAG with the highest shear bond strength between the ceramic surface and the orthodontic bracket. Kara et al found that the femtosecond laser showed higher surface roughness on zirconia in comparison with Nd: YAG and Er: YAG. It showed increased bonding of resin cement and zirconia.²⁸ The Titanium sapphire laser is a highly powerful and ultra-fast laser that imparts the laser beam for picoseconds and femtosecond pulse width and causes no collateral thermal damage. The femtosecond laser seems to be a proper method for a zirconia surface modification as it did not increase the monoclinic content of zirconia evaluated with X-ray diffraction analysis.²⁹ The null hypothesis is not accepted; thus, "there is a significant difference noted in mean shear bond strengths at the zirconia-veneering ceramic juncture between group C, group N and group T". The alternate hypothesis is accepted.

The constraints of the work are as follows: the shape of the specimen did not resemble that of clinical restoration and the influence of aging and thermo-cycling³⁰ on the bonding potential of zirconia and veneering ceramics was not assessed. This study did not evaluate the effect of other lasers like carbon dioxide and Erbium: yttrium aluminum garnet. Therefore, further studies are needed.

Conclusion

After statistical analysis and thorough discussion, the following inference was made in the present study:

The titanium sapphire laser shows the highest shear bond strength at the time duration of 30 seconds. It is a promising mode of surface treatment of post-sintered

zirconia to increase the shear bond at the zirconia core-veneer ceramic juncture.

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Ethical considerations

Approval from ethical committee was received prior to the study (Approval No. 062/IEC/SS/2017).

Conflict of Interests

The authors acknowledge all participants with no conflict of interest.

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