Effect of ultrashort pulsed laser on bond strength of Y-TZP zirconia ceramic to tooth surfaces

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There is limited knowledge about the effects of ultrashort pulsed laser on zirconia ceramic surfaces. The aim of this study was to evaluate the effects of ytterbium (Yb)-doped fiber laser and other surface treatment methods —namely, sandblasting with 110 µm aluminum oxide or 30 µm silica-coated alumina on shear bond strength (SBS) of zirconia to tooth surface. A total of 128 zirconium oxide disks were made by using CAD-CAM technology. Disk surfaces were sandblasted with Al₂O₃ particles or silica-coated alumina or irradiated with Yb-doped fiber based nanosecond pulsed laser at 85W output power at 25 kHz. Disks were luted to dentin using two different resin cement. SBS of each specimen was measured. Results were statistically analyzed using two-way analysis of variance (ANOVA) and Bonferroni and Dunnett tests ($p < 0.005$). Highest bond strength was obtained when zirconia surface was pretreated with Yb-doped fiber-based nanosecond pulsed laser regardless of the resin cement used.

Keywords: Yb-doped fiber laser, Zirconia, Shear bond strength, Resin cement

INTRODUCTION

Zirconia demonstrates great potential as a substitute for myriad traditional materials in many dental applications, such as orthodontic brackets, posts, implant abutments, and frameworks for inlays, crowns, and bridges¹. The mechanical properties of zirconia are the highest ever reported for any dental ceramic, boasting of high flexural strength and toughness²⁻⁴. In prosthetic dentistry where strength and esthetics are important⁵, zirconia has become the favored material for posterior fixed partial dentures because it permits a prominent reduction in core thickness. New technologies, such as computer-aided design/computer-aided manufacturing (CAD/CAM), enable and fuel the fabrication of zirconia-based restorations for all-ceramic crowns⁴.

Different types of cements (conventional, glass ionomer, self-adhesive resin cements) have been proposed for zirconia luting. Among these, self-adhesive resin cements are the preferred choice because of their better retention and marginal fit^{6,7}. To achieve reliable adhesion to ceramics, it is typically necessary to perform surface pretreatments. Roughened ceramic surfaces allow resin cements to flow into the microretentions and create a stronger micromechanical interlock⁸⁻¹².

Unlike conventional dental ceramics, high-strength ceramics such as yttria-tetragonal zirconia polycrystal (Y-TZP) have high crystalline content and comprise many crystalline filler particles without a glass phase at the crystalline border¹³. Therefore, these ceramics are not suitable for acid etching^{8,14-16}. For this reason, surface treatment methods ranging from air abrasion with aluminum oxide (Al₂O₃)^{8,17-20} and silica coating with silica-modified Al₂O₃ particles^{9,17,18,21} to laser application are frequently used on Y-TZP restorations.

The underlying principal of laser application is the conversion of light energy into heat energy. The most important interaction between laser light and the substrate is the absorption of laser energy by the substrate^{22,23}. As far as popularity in biomedical applications goes, erbium lasers are closely followed by ytterbium fiber lasers. Possessing a rather simple energy level structure, Yb³⁺-ion transitions offer potentially high pumping efficiency due to small quantum defect, *i.e.*, energy (or frequency) difference between the pumping and lasing photons. Ytterbium lasers, operating at around 1 µm, offer small nonradiative losses, low heating, almost 80% conversion efficiency, and approximately 25% wall-plug efficiency²⁴.

Featuring high lasing efficiency and broadband tunability, ytterbium-pulsed lasers with high peak power operating in Q-switched and mode-locking regimes are a rapidly advancing area of ultrashort laser applications²⁵⁻²⁷. With radiation around the wavelength range of 1,030–1,080 nm, Q-switched ytterbium fiber lasers are becoming an attractive alternative to Nd:YAG lasers—which employs a wavelength of 1,064 nm and which traditionally occupies a large sector of the medical applications industry. Pulsed ytterbium lasers, which deliver pulses with more than 80 W of peak power and of microsecond duration, are applicable in diverse areas such as gynecology, abdominal surgery, cardiovascular surgery, and dental curing²⁸.

The aim of this study was to evaluate and compare the effects of 1,064-nm ytterbium-doped fiber laser (YbPL) on bond strength of Y-TZP zirconia ceramic to tooth surface and surface roughness of zirconia against other surface treatment methods such as sandblasting with Al₂O₃ or 110 µm silica-coated alumina (Cojet system). The hypotheses tested were that YbPL would

not influence the surface roughness of zirconia and shear bond strength (SBS) of Y-TZP to resin cement.

MATERIALS AND METHODS

One hundred and twenty molar teeth, measuring approximately 8 mm in mesiodistal length and which were free of dental caries and restorations, were cleaned and stored in saline at room temperature within 2 months after extraction. The teeth were sectioned 3 mm below their occlusal surfaces with a slow-speed diamond-saw sectioning machine (Isomet; Buehler Ltd., Lake Bluff, IL, USA) under water cooling. The crowns were embedded in an autopolymerizing acrylic resin (Meliodent; Bayer Dental Ltd., Newbury, UK). All specimens were randomly divided into eight groups ($n=15$).

For each tooth specimen, 136 zirconium oxide disk specimens (8 for SEM examination and 8 for profilometer examination) were cut from pre-sintered zirconia blocks (ZirkonZahn, Steger, Italy) using CAD/CAM technology (Yenemak, Turkey). They were sintered in a furnace at 1,500°C for 16 h to the following dimensions: 6 mm in length×4 mm in width. Disk surfaces were ground-finished with 240-, 400-, and 600 grit silicon carbide abrasive papers (3M ESPE, St. Paul, MN, USA) for 15 s using a 170-rev/min grinding machine (Minitech 233; Presi, Grenoble, France) under running water. This was followed by 3 min of ultrasonic cleaning in ethanol and deionized water, and dried with oil-free air.

All zirconium oxide disk specimens were randomly

divided into eight groups, with each group composed of 15 disk specimens according to the surface treatment and resin cement used.

Pilot study

Being the first usage of the YbPL on zirconia surfaces, a pilot study was performed to determine the optimal laser parameters. Five zirconium oxide disk samples were used for the pilot study.

Laser beam was directed over the zirconium oxide disk surface in a non-contact mode at a working distance of 17.8 mm. Zirconium oxide disk surfaces were irradiated with YbPL (Vision, Germany) at different power settings and frequencies: 60 Watt/40 kHz, 100 watt/80 kHz, 75 Watt/80 kHz, 85 Watt/25 kHz and control group with no surface treatment. For all the specimens, device speed was 4,000 mm/s.

Zirconium oxide disk surfaces were scanned once, vertically and horizontally, using the fiber laser machine at different parameters. After irradiation, specimens were sputter-coated with gold-palladium (Oxford Instruments, UK). The specimens were examined with a scanning electron microscope (SEM Oxford Instruments, UK) at ×1,000 magnification. Perceptible loss of zirconium oxide material was detected during SEM analysis of the specimen surfaces irradiated at 100 Watt/80 kHz (Fig. 1a.). Irradiation at 60 Watt/40 kHz and 75 Watt/80 kHz showed no significant surface alterations (Figs. 1b and 1c). Optimal surface roughness without cracks was achieved with the setting of 85 Watt/25 kHz (Fig. 1d), and this setting was used in this study.

Fig. 1 SEM views of the zirconia surfaces (×1,000) after laser irradiation at: (a) 100 Watt/80 kHz, (b) 60 Watt/40 kHz, (c) 75 Watt/80 kHz, (d) 85 Watt/25 kHz.

Experimental groups

Disk specimens were divided into eight groups ($n=15$) according to the surface treatment performed and resin cement used:

- Group 1 (AIP): Disk surfaces were sandblasted with Al_2O_3 particles (110 μm) for 15 s at 0.41–0.68 MPa and a distance of 10 mm, and then luted with Panavia F 2.0 (Kuraray, Japan).
- Group 2 (AIV): Disk surfaces were sandblasted with Al_2O_3 particles (110 μm) for 15 s at 0.41–0.68 MPa and a distance of 10 mm, and then luted with Variolink N (Ivoclar Vivadent, Liechtenstein).
- Group 3 (CjP): Disk surfaces were sandblasted with 30- μm silica-coated alumina (CoJet System, 3M ESPE) at a constant pressure of 280 kPa for 20 s/cm² and at a perpendicular distance of 10 mm, and then luted with Panavia F 2.0 (Kuraray).
- Group 4 (CjV): Disk surfaces were sandblasted with 30- μm silica-coated alumina (CoJet System, 3M ESPE) at a constant pressure of 280 kPa for 20 s/cm² and at a perpendicular distance of 10 mm, and then luted with Variolink N (Ivoclar Vivadent).
- Group 5 (FIP): YbPL irradiation at 85 Watt output power and 25 kHz, and then luted with Panavia F 2.0 (Kuraray).
- Group 6 (FIV): YbPL irradiation at power output 85 Watt, 25 kHz, and luted with Variolink N (Ivoclar-Vivadent).
- Group 7 (CoP): No surface treatment and luted with Panavia F 2.0 (Kuraray).
- Group 8 (CoV): No treatment was performed and luted with Variolink N (Ivoclar-Vivadent).

Two specimens were randomly selected from each group to determine the effects of different surface treatments. These specimens were sputter-coated with gold-palladium and examined using SEM at $\times 5,000$ magnification (Fig. 3).

Bonding procedure

Surface-treated disks were luted to the tooth surfaces using one of two different dual-cure resin cements: MDP-containing Panavia F 2.0 (Kuraray) *versus* Bis-GMA-containing Variolink N (Ivoclar Vivadent). Resin cements were applied to disk surfaces according to the manufacturers' instructions. Bonded specimen of

zirconium oxide disk-resin cement-tooth surface was placed under a load of 2,000 g, and resin cement was polymerized for 20 s on each side of the bonding area. Bonded specimens were then stored in distilled water at 37°C for 24 h. After 24-h water storage, specimens were thermocycled between 5°C and 55°C for 5,000 cycles at 15 s in each water bath.

SBS Test

SBS was measured using a universal testing machine (Shimadzu Corp., Japan) at a crosshead speed of 1 mm/min. A shear load was applied until failure occurred. All the specimens were examined to determine the failure type.

Analysis of failure types

Classification of failure types was done according to the adhesive remnant index (ARI)²⁹. A base remnant index (BRI) scoring system was created to evaluate the failure types on zirconium oxide disk surfaces.

Statistical analysis

Statistical analysis of the data was performed using a computerized statistical software program (SPSS 15.0 for Windows, SPSS Inc., Chicago, IL, USA). SBS values of eight experimental groups were analyzed by two-way (surface treatments, resin cements) analysis of variance (ANOVA). Multiple comparisons were performed using Bonferroni's and Dunnett's multiple comparison tests. Level of statistical significance was set at 5%.

RESULTS

SBS

Two-way ANOVA indicated that SBS values varied according to surface treatment (sandblasted with Al_2O_3 particles or silica-coated alumina, or irradiated with YbPL) and resin cement (Variolink N, Panavia F) ($p<0.05$) (Table 1, Fig. 2).

Significant differences ($p<0.05$) in SBS were seen among the experimental groups which used MDP-containing resin cement (Panavia F). YbPL-irradiated group (F1P) showed a significantly higher SBS than the other groups ($p<0.05$). Group 1(AIP) was also significantly higher than the control group. However there were no significant differences between Group 1(AIP) and Group 3 (CjP) ($p<0.05$).

Among the experimental groups which used Bis-GMA-containing resin cement (Variolink N), the SBS value of YbPL-irradiated group (FIV) was significantly higher than the other groups ($p<0.05$). No significant differences were observed among the other three groups

Table 1 SBS results of experimental groups

	Al_2O_3	CoJet System	YbPL	Control
Panavia F 2.0	6.84 $\pm$ 1.83	5.31 $\pm$ 1.71	11.91 $\pm$ 1.23	4.83 $\pm$ 1.49
Variolink N	3.55 $\pm$ 0.78	4.39 $\pm$ 0.86	11.03 $\pm$ 1.08	3.54 $\pm$ 1.28

($p < 0.05$).

Within each surface treatment groups, the MDP-containing resin cement consistently yielded a higher SBS values than Bis-GMA-containing resin cement ($p < 0.05$).

Within each resin cement groups (MDP-containing Panavia F or Bis-GMA-containing Variolink N), significant differences in SBS were observed among the different surface treatment groups ($p < 0.05$).

ARI and BRI scores

The ARI and BRI scores are listed in Table 2. In YbPL-

Fig. 2 SBS results of experimental groups (MPa).

irradiated groups, almost all the specimens were given ARI score 3 because adhesive resin cement was left on the dentine surface (13 in FIP group, 9 in FIV group). Except for YbPL-irradiated groups, sandblasted groups mainly showed BRI score 0, where abrasive resin was totally separated from the zirconium oxide disk surface.

Surface roughness

SEM view of untreated zirconium oxide disk surface is shown in Fig. 3a. Silica-coated zirconium oxide disk surface exhibited a surface texture consisting of micro-mechanical irregularities (Fig. 3b). Compared with the untreated specimen, sandblasting with Al₂O₃ particles created a clearly rougher surface (Fig. 3c). However, YbPL irradiation produced a surface roughness which exceeded the other experimental groups (Fig. 3d). Laser irradiation at 85 Watt output power 25 kHz created a rough surface with voids and plaque-like scaly appearance without any microcracks.

Figures 4a–d show the three-dimensional (3D) roughness measurements of the different surface treatment groups. Surface Roughness (*Ra*) of the YbPL-irradiated group was higher than the other surface treatment groups.

DISCUSSION

In the current study, the effects of YbPL and different surface treatments on the SBS of two resin cements to zirconium oxide surface were evaluated and compared.

Table 2 ARI and BRI of experimental groups

		Classification of the failures			
Groups		Scores			
		0	1	2	3
Group 1	AlP	13	2	0	0
Group 2	AlV	11	4	0	0
Group 3	CjP	11	3	1	0
Group 4	CjV	9	5	1	0
Group 5	FIP	0	0	2	13
Group 6	FIV	1	3	2	9
Group 7	CoP	12	2	1	0
Group 8	CoV	6	5	4	0

ARI Scores

Score 0: no luting agent left on the dentine surface.

Score 1: less than half of the luting agent left.

Score 2: more than half of the luting agent left.

Score 3: all luting agent left on the dentine surface.

BRI Scores

Score 0: all luting agent left on the zirconium oxide disc.

Score 1: more than half of the luting agent left.

Score 2: less than half of the luting agent left.

Score 3: no luting agent left on the zirconium oxide discs surface.

Fig. 3 SEM view of: (a) untreated zirconia surface; (b) Co-Jet system applied zirconia surface; (c) Al₂O₃ sandblasted zirconia surface; (d) YbPL-irradiated zirconia surface (×5,000).

Fig. 4 3-D profilometer view of: (a) untreated zirconia surface ($R_a=1.15 \mu\text{m}$); (b) CoJet System-applied zirconia surface ($R_a=1.37 \mu\text{m}$); (c) Al₂O₃-sandblasted zirconia surface ($R_a=1.64 \mu\text{m}$); (d) YbPL-irradiated zirconia surface ($R_a=2.83 \mu\text{m}$).

Particle sizes used for the CoJet system and Al_2O_3 were selected based on previous studies^{28,30,31}, and the laser parameters used were determined using a pilot study. In the pilot study, zirconium oxide disk surfaces were scanned once, vertically and horizontally, using the laser machine with different parameters and then evaluated by SEM.

Surface roughness is an important factor for adhesion. A rough surface increases the surface area, improves wettability by reducing surface tension, and creates micromechanical retention. Al_2O_3 air abrasion is one of the common methods used to increase surface roughness^{8,19,32,33}. Some investigators suggested that superior bonding to zirconia was obtained when with air-abraded surfaces³⁴⁻³⁷. However, others concluded that this method did not have a significant effect to improving the bonding of resin cements to zirconia surface^{15,37-39}. In addition, the risk of microcrack generation with the use of airborne particle abrasion method was also reported^{40,41}. In the current study, Al_2O_3 air abrasion increased the surface roughness and SBS when compared with the control group. Sandblasting with 30- μm silica-coated alumina showed similar results as Al_2O_3 .

In all surface treatment groups, luting with MDP-containing resin cement yielded higher SBS than with Bis-GMA-containing resin cement groups. This result agreed with the results of previous studies which reported higher bond strengths with the use of MDP-based primer^{8,42}. This could be due to the capability of acidic functional monomer MDP reacting with the substrate¹³⁻¹⁹.

Different laser systems have been used to increase the surface roughness of zirconia surfaces. However, the effect of laser irradiation on the mechanical properties of Y-TZP materials is controversial, and both positive and negative results have been reported in the literature. Nd:YAG laser irradiation with both short and long pulse durations significantly increased the surface roughness of Y-TZP surfaces and SBS to resin cement. On the other hand, etching the zirconia surface with Nd:YAG laser irradiation caused microcracks and increased the monoclinic content of zirconia—which unfortunately might reduce the resistance and longevity of the material⁴³.

Ural *et al.*⁴⁴ concluded that etching with CO_2 laser (3W) might be an effective method for conditioning zirconia surfaces, enhancing micromechanical retention, and improving the bond strength of resin cement (Panavia F, Kuraray) to zirconia ceramic. It was thus recommended as an alternative surface treatment technique for bonding resin cement to zirconia ceramic surfaces^{44,45}. However, Paranhos *et al.*³⁵ reported that significant microcracks were found on specimens treated with the CO_2 laser (5W) and that low SBS values were obtained with the same resin cements used in previous study.

All researchers concluded that power must be carefully considered using high-energy Nd:YAG, Er:YAG and CO_2 lasers to avoid microcracks on zirconia surfaces⁴⁴. In this current study, YbPL irradiation was

used for the pretreatment of zirconia surfaces. Positive surface roughness and SBS results were obtained. On surface roughness, YbPL irradiation created a higher level of roughness and this surface topography might be the reason for the highest SBS value of two resin cement to zirconia surface.

Interactions and possible favorable and unfavorable effects of lasers with shorter pulse durations (such as femtosecond or picoseconds) should be considered as a new investigation area for further researches.

CONCLUSIONS

Within the limitation of this study, the following conclusions were drawn;

1. Highest bond strength was obtained when zirconia surface was pretreated with YbPL.
2. Resin cement selection was not an important factor when the zirconia surface was roughened with YbPL irradiation.
3. For surface treatment with Al_2O_3 or CoJet System, resin cement selection could affect the bond strength between the resin cement and zirconia in that MDP-containing resin cements might be more suitable for cementing zirconia ceramics.

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DECLARATION OF CONFLICTING INTERESTS

All authors declare that there are no commercial associations that might create a conflict of interest in connection with the submitted manuscripts.

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