

ORIGINAL ARTICLE

The effect of different power outputs of carbon dioxide laser on bonding between zirconia ceramic surface and resin cement

ÇAĞRI URAL¹, ELIF KALYONCUOĞLU² & VEYSEL BALKAYA²¹Ondokuz Mayıs University, Faculty of Dentistry, Department of Prosthodontics, Samsun, Turkey, and ²Private Practice, Samsun, Turkey**Abstract**

Objective. The purpose of this in vitro study was to evaluate the influence of different power outputs of a carbon dioxide (CO₂) laser on shear bond strength of resin cement to zirconium dioxide-based ceramic. **Materials and methods.** Fifty zirconium dioxide core specimens (10 mm diameter and 2 mm thickness) were produced and they were embedded in the centers of auto-polymerizing acrylic resin blocks. Ten specimens served as control and no surface treatment was applied. Subsequently specimens were randomly divided into four groups, each containing 10 specimens for surface treatment with CO₂ laser with different output power; laser treated with 2 W (Group 2 W), 3 W (Group 3 W), 4 W (Group 4 W) and finally 5 W (Group 5 W). Fifty composite resin discs were fabricated and cemented with adhesive resin cement to the specimen surfaces. A universal test machine was used for shear bond strength test at a crosshead speed 1 mm/min. Data were statistically analyzed by one-way analyses of variance (ANOVA) with Post-Hoc Tukey tests ($\alpha = 0.05$). **Results.** It was found that the shear bond strength values were affected by power outputs of laser ($p < 0.05$). Highest shear bond strength values were obtained with group 2 W (21.0 ± 2.7). Lowest values were obtained with group 5 W (14.4 ± 1.6). **Conclusion.** The current study revealed that there was a relationship between laser output power and shear bond strength for zirconium dioxide ceramics. However, output power of the laser and the energy level is a critical factor on micromechanical retention.

Key Words: Laser etching, laser output power, surface treatment, zirconium dioxide

Introduction

The search for esthetic dental restorations is increasingly frequent in daily practice. [1]. Due to their high esthetic profile, mechanical properties, chemical stability and biocompatibility, all-ceramic restorations have become the focus of dental practitioners, researchers and manufacturers. With the increase of aluminum oxide content (Al₂O₃) in feldspathic ceramics, there has been a significant improvement in the mechanical properties of these materials, allowing metal-free restorations to be used more predictably [2]. One of the most commonly used all-ceramic core materials for conventional and resin bonded fixed partial dentures (FPDs) and complete coverage crown is yttrium tetragonal zirconium dioxide polycrystal (Y-TZP, zirconium dioxide) [3–6].

Some authors postulate that the advantages of adhesion of all-ceramic crowns and bridges are due

to improved retention, better marginal adaptation and fracture resistance [3]. Obtaining adhesion between a luting agent and a ceramic surface requires surface pre-treatment [7,8]. The formation of chemical bonds and micromechanical interlocking establish a strong, durable resin bond to ceramics [9]. To increase bond strength of resin materials to ceramic restorations many surface treatment methods and cementation procedures with different resin cements were evaluated [10]. These surface treatment methods are grinding, abrasion with diamond rotary instruments, airborne particle abrasion with Al₂O₃, tribochemical silica-coating, acid etching with hydrofluoric acid and combinations of any of these [10]. Tribochemical silica-coating, performed by Rocotec System, is based on silica-coated alumina particles and is designed for conditioning the ceramic substrate surface, including non-etchable alumina and zirconium dioxide, extra-orally. A Cojet system is used for intra-oral silica-coating applications.

Correspondence: Dr Çağrı Ural, Ondokuz Mayıs University, Faculty of Dentistry, Department of Prosthodontics, 55139 Kurupelit, Samsun, Turkey.
Tel: +90 362 312 19 19 / 36 90. Fax: +90 362 457 60 32. E-mail: cagriural@omu.edu.tr

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However, traditional surface treatment methods could not provide an adequate bond between resin cement and high strength ceramics like zirconium oxide [10]. In particular high-alumina or zirconium dioxide-reinforced ceramics cannot be roughened by hydro-fluoric acid etching and airborne particle abrasion, because such ceramics do not contain a silicon dioxide (silica) phase [11].

Since the development of the ruby laser by Maiman in 1960, lasers have become widely used in medicine and dentistry [12]. Many advances have been directed at the use of lasers in clinical applications as an alternative to other surface treatment methods for bonding ceramic to the tooth surface [13]. The effect of high intensity lasers on dental materials has been previously studied and includes laser irradiation of porcelain brackets, resin composite for indirect use, leucite-based ceramics and aluminous ceramic [14–16]. Only a few studies have been performed on the laser treatment of zirconium oxide ceramics [12,16].

The carbon dioxide (CO₂), neodymium-doped yttrium aluminium garnet (Nd: YAG) and Erbium-doped yttrium aluminium garnet (Er:YAG) lasers are the most generally used instruments for both intra-oral soft tissue surgery and hard tissue applications [17]. The CO₂ laser is well suited to the treatment of ceramic materials because its emission wavelength is almost totally absorbed by the ceramic. During the process of heat induction of ceramic surfaces with a focused CO₂ laser, conchoidal tears—typical effects of surface warming—appear. These tears are believed to provide mechanical retention between resin composite and ceramics [12].

Therefore, the purpose of this *in-vitro* study was to evaluate and compare the effects of different laser output power on shear bond strength of resin cement to zirconium dioxide-based ceramic. Research hypothesis was that different laser output power would affect the shear bond strength values.

Materials and methods

The materials used, compositions and manufacturing information are presented in Table I.

Forty zirconium dioxide core specimens (10 mm diameter and 2 mm thickness) were produced by a

copy-milling system (Zirconzahn, Bruneck, Italy) using pre-fabricated blanks of zirconium dioxide (ICE Zircon Translucent, Zirconzahn, Bruneck, Italy) and then sintered according to manufacturer instructions. The surfaces were cleaned with ethanol and carefully dried with air before surface treatment (Branson 2210, Branson Ultrasonics Corporation, Danbury, Ct, USA). Zirconium dioxide cores were embedded in the centers of autopolymerizing acrylic resin blocks (Meliodent; Heraeus Kulzer, Armonk, NY). Zirconium dioxide core surfaces were ground finished with a 600-grit silicon carbide abrasive (3M ESPE, St. Paul, MN) under running water on a polishing machine (Buehler Metaserv, Buehler, Germany) and ultrasonically cleaned for 3 min in ethanol and deionized water and air-dried.

Ten of the test specimens were serving as control and no treatment was applied on the zirconium dioxide ceramic surfaces. Subsequently specimens were randomly divided into four groups, each containing 10 specimens, for different laser output power and bonding surfaces of ceramic discs were irradiated by CO₂ laser (Smart US20D, Deka, Firenze, Italy). Laser energy was delivered in a pulse mode with a wavelength of 10,6 µm, pulse repetition rate of 1000 Hz and pulse duration of 160 ms at an average power settings of 2, 3,4 and 5 W.

Fifty composite resin discs (Filtek Z250, 3M ESPE, Germany) were fabricated by compacting the material into a polytetrafluoroethylene mold (Isoflon, Diemoz, France) with a hole in the center (6 mm diameter × 2 mm thickness). Composite resin was incrementally condensed into the mold to fill up the mold and each layer was light polymerized for 40 s at a distance of 1 mm using a light-polymerizing unit (Astralis 3, Ivoclar Vivadent, Schaan, Liechtenstein) with an output power of 600 mW/cm². Light intensity was measured using an internally set radiometer. For polymerization distance standardization, a plastic mold was used (Figure 1). One composite resin block was fabricated for each specimen.

Composite resin discs were cemented to the specimen surfaces with dual- polymerized adhesive resin cement (Panavia F 2.0; Kuraray Co Ltd; Osaka, Japan) containing the adhesive phosphate monomer (MDP). For cementation, equal amounts of a dual-polymerized resin luting agent paste base and catalyst

Table I. Materials used in the study.

Material	Type of material	Manufacturer
Ice Zircon	Yttrium, partially stabilized with tetragonal polycrystalline structure (Y-TZP)	Zirconzahn SRL, Bruneck, Italy
Panavia F 2.0	Dual-polymerized adhesive resin cement (BPEDMA, MDP, DMA, Ba-B-Si glass, silica, chemical and photoinitiators)	Kuraray, Okayama, Japan
Filtek Z250	Composite Resin	3M ESPE, Germany
Smart US20D	CO ₂ laser	Deka, Firenze, Italy

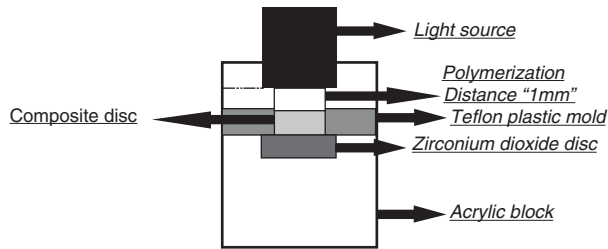


Figure 1. Schematic view of the acrylic plastic mold which was used for polymerization distance standardization.

were mixed and applied to the composite resin block with a plastic spatula. Each composite disc was bonded to a zirconium dioxide core specimen under the finger pressure. The excess resin cement was removed by means of a brush. The resin cement was then light polymerized for 20 s with a curing light (Astralis 3). A glycerin gel (Oxyguard II; Kuraray Co Ltd; Osaka, Japan) was applied on the cement layer for 10 min. The specimens were washed with air-water spray and then were stored in distilled water at 37°C for 24 h before shear bond strength test.

A universal test machine (Lloyd LRX; Lloyd Instruments PIC., Fareham, Hampshire, UK) was used for shear bond strength test at a cross-head speed of 1 mm/min. Each specimen surface was parallel to the direction of the force during the shear strength test. Force was applied to the zirconium dioxide–composite interface. The shear bond strength values were calculated in megapascal (MPa) by dividing the load used until a fracture occurred (N) to the area of the composite resin ($N/\pi r^2$). Data were statistically analyzed. The Kolmogorov-Smirnov test showed that the data was of a normal distribution ($p > 0.05$). A homogeneity of variance test was done using Levene's test ($F = 0.734$, $p > 0.05$). Means and standard deviations of bond

strengths were calculated and mean values were compared by one-way analysis of variance (ANOVA) (SPSS 12.0; SPSS Inc., Chicago, IL), followed by a multiple comparisons' test performed using a Post Hoc Tukey test ($\alpha = 0.05$).

To evaluate the effects of laser with different output power on the surface morphology of zirconium dioxide core ceramic, an additional four samples were treated with the same experimental protocol as described previously. All specimens were coated with gold using a sputter coater (S150B; Edwards, Crawley, UK) and examined under a field emission scanning electron microscope (SEM) (JSM-6335F; JEOL, Tokyo, Japan) at 15 kV. The SEM photomicrographs were developed with $\times 500$ magnification for visual inspection (Figure 2).

Results

The result of one-way ANOVA is presented in Table II. The mean shear bond strength values and standard deviations are listed in Table III. Highest shear bond strength values were obtained with group 2 W (21.0 ± 2.6) and there was no significant difference between group 3 W. Lowest values were obtained with group 5 W (14.4 ± 1.6) and there was no significant difference between group C.

Visual examination of the fractured surfaces showed that adhesive fractures were the most common types of failure for Group 5 W. Pure zirconium dioxide and pure composite resin cohesive failures were not observed. Control group specimens exhibited an average of 100% and Group 5 W specimens 90% adhesive failures between zirconium dioxide core and resin luting cement. Group 3 W specimens exhibited an average of 30%, Group 2 W specimens

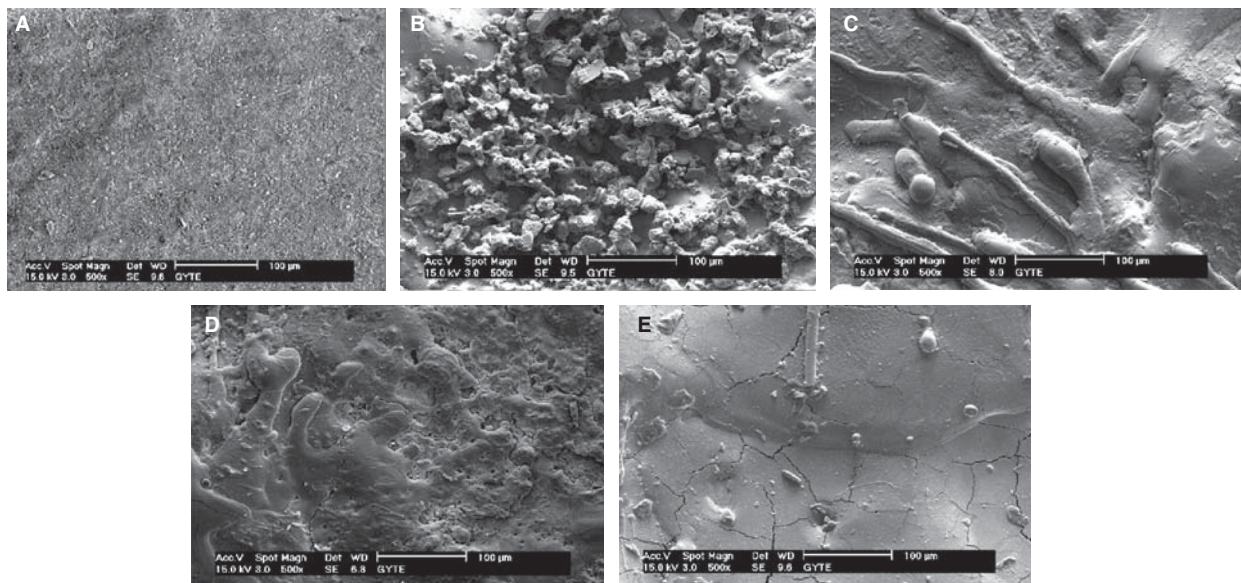


Figure 2. SEM photographs of Zirconium oxide ceramic surfaces (A), untreated; (B) 2 W laser irradiated; (C) 3 W laser irradiated; (D) 4 W laser irradiated; (E) 5 W laser irradiated ($\times 500$).

Table II. Result of one-way ANOVA test.

	Sum of squares	df	Mean square	F	Sig.
Between groups	513 363	4	128 341	16 814	0.000
Within groups	343 493	45	7 633		
Total	856 855	49			

exhibited an average of 20% adhesive failures between the zirconium dioxide core and resin luting cement, finally Group 4 W showed 40% adhesive failure (Table IV).

When the SEM photographs were evaluated, 2 W and 3 W laser irradiated ceramic surfaces showed some irregularities (Figures 1B and C). The laser-irradiated with 4 W and 5 W output power showed some micro-cracks and melted areas on ceramic surfaces (Figures 1D and E).

Discussion

The data supports the hypothesis that different output power of laser treatment affects bond strength. Different output power of the laser affected the shear bond strength values significantly ($p < 0.05$).

Dual-polymerized adhesive resin cement containing the adhesive phosphate monomer (APM) is currently recommended for all ceramic restorations for longevity and success of the restoration [3]. Some authors postulate that the advantages of adhesion of all-ceramic crowns and bridges are due to improved retention, better marginal adaptation and fracture resistance [3]. Various *in vitro* studies have shown that air-borne particle abrasion with Al_2O_3 particle is an essential step in achieving a durable bond to high-strength ceramics [18–22]. In addition etching with hydrofluoric acid or sandblasting the inner surfaces of ceramics with glassy matrix followed by the application of a silane coupling agent is an efficient conditioning method for bonding resin composite [10]. However, neither airborne particle abrasion and etching with this agent nor adding silane resulted in an adequate resin bond to some new high strength ceramics [23]. As an alternative to routinely-used surface treatment methods, high-intensity lasers have been

Table III. Mean (MPa) and standard deviations (SD) of shear bond strength values.

Groups	MPa	SD
Group C	13.4 ^a	±3.1
Group 2 W	21.0 ^c	±2.7
Group 3 W	20.9 ^c	±3.8
Group 4 W	17.2 ^b	±2.1
Group 5 W	14.4 ^{ab}	±1.6

Same letter are not statistically different ($p > 0.05$).

Table IV. Failure types.

Groups	Type of failure		
	AD (%)	CO (%)	MI (%)
C (Control)	100	—	—
Group 2 W	20	—	80
Group 3 W	30	—	70
Group 4 W	40	—	60
Group 5 W	90	—	10

AD, Adhesive failure; CO, Cohesive failure; MI, Mixed fracture at the zirconium dioxide or the composite resin surface.

investigated recently to increase the surface roughness of ceramic restorations, thus favoring ceramic adhesion to the luting cement by means of micromechanical interlocking [14]. Akova et al. [16] reported that laser irradiated ceramic surface showed conchoidal irregularities. It was hypothesized that these irregularities enhance mechanical retention between resin composite and porcelain.

Different lasers were studied in the literature and each wavelength provided various results, according to irradiation parameters and dental material irradiated. The Nd: YAG laser (1064 nm) and the CO_2 laser (10 600 nm) were evaluated for surface conditioning of porcelain, leucite-based ceramics and both glass-infiltrated alumina and zirconium dioxide composites and the micromechanical retentions produced on the ceramic surface provided similar or even higher bond strength values than those obtained after conventional treatment [14,16,24]. Li et al. [25] reported that there were no significant differences in the shear bond strength of a composite resin to ceramic after laser irradiation at power outputs of either 0.9 W or 1.2 W. In the present study lower laser output power (2–3 W) showed similar shear bond strength values and highest bond strength values were obtained with the Group 2 W.

The lower bond strength values in group 4 W and 5 W can be explained by the effect of laser on surface morphology. The irradiation of dental or ceramic substrate with laser results in the vaporization or ablation of the structure through the thermo mechanical process. The vaporization is characterized by the explosion of portions of molten material heated above the melting point at atmospheric pressure. In the thermo mechanical ablation, the water present in the material or on its surface absorbs the energy of incident photons and vaporizes quickly, causing micro-explosions that are able to eject the surrounding substrate [26]. A recent study observed that lower energy settings produced mild alterations on zirconium oxide-based ceramic surface, whereas higher parameters resulted in excessive deterioration of the material, with intense occurrence of cracks and carbonization [27]. When the SEM views of laser

irradiated ceramic surfaces with 2 and 3 W laser output power were evaluated (Figures 1B and C), as previously mentioned, micro-explosions produced irregular alterations on the irradiated ceramic surface. However, the 4 and 5 W output power of CO₂ laser created smoother surfaces, melted areas and micro-cracks (Figures 1D and E). Different reasons for micro-crack formation on ceramics after CO₂ laser irradiation have been proposed. According to a previous study, during the heating of the ceramic surface caused by the absorption of the laser radiation, there is a superficial emission of ions, electrons and atoms [12]. Due to the characteristic photo-ionization caused by the radiation, physical plasma emerges. Its formation is accompanied by the development of extremely high pressure and fluctuations in temperature in the range 10 000–50 000 K. This may cause extreme physical stress during the cooling and re-hardening of the ceramic surface [12]. The SEM analysis conducted in the present study indicates micro-cracks and melting after laser irradiation when the higher energy parameters are used. Consequently the higher shear bond strength values obtained in groups 2 W and 3 W and lower values obtained in groups 4 W and 5 W can be explained by micromechanical retention and surface irregularities on ceramic surfaces.

In this experimental study, early shear bond strength was tested but the effects of thermal cycling and long-term storage on bond strength were not evaluated. These are the limitations of this study. Further research is needed to evaluate the effects of different types of lasers and parameters like duration and pulse mode on ceramic surfaces to obtain optimum bond strength values.

Conclusion

The current study revealed that there might be a relationship between laser output power and shear bond strength for zirconium oxide ceramics. CO₂ laser etching may represent effective methods for conditioning zirconium dioxide surfaces, enhancing micromechanical retention and improving bond strength of resin cement on zirconium dioxide ceramic. However, output power of the laser and the energy level used may be one of the critical factors in micromechanical retention.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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