Original Article

Repair bond strength of composite to Er,Cr:YSGG laser irradiated zirconia and porcelain surfaces

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ABSTRACT

Background: Fracture or chipping are major concerning failures of an all-ceramic restoration. Repairing of the failure restoration using intra-oral technique is time saving and cost effective treatment modality. The present study was proposed to evaluate effect of Er,Cr:YSGG laser irradiation on shear bond strength between zirconia/porcelain and composite resin.

Methods: Thirty zirconia and thirty zirconia based porcelain disc shape specimens were prepared. Three different surface treatment procedure were applied the specimens. For control groups (Group ZC and PC), instruction manual of an intra-oral porcelain repair system was followed. Different pulse rates of Er,Cr:YSGG laser irradiation (short and long pulses) were applied to zirconia and porcelain surfaces for other groups (Group ZS, ZL, PS, and PL). Porcelain repair kit was used to repair specimens using standard cylindrical teflon mold (2 × 2 mm). Repair bond strength of the repaired specimens was tested using a universal testing machine.

Results: Highest mean bond strength value was observed at Group PC that was significantly higher than laser applied porcelain groups. Long pulse laser irradiation illustrated that increased mean bond strength compared to short pulse application on to the porcelain surface. Laser applied zirconia groups showed better mean bond strength than Group ZC, but differences between the groups were not statistically significant.

Conclusion: Different modes of Er,Cr:YSGG laser irradiation enhanced repair bond strength of the composite resin to zirconia, but these were not significant. Following the instruction manual for surface treatment on the porcelain surface was better method than Er,Cr:YSGG laser surface conditioning.

Porcelain fused to metal crowns and fixed partial dentures (FPDs) have been applied confidently for more than half a century, due to predictability, high mechanical properties and low cost [1,2]. However, high demand for metal-free and esthetic restorations, natural appearance, and favorable optical properties were increased application of all-ceramic crowns and bridges by clinicians since millennium [3]. Especially, zirconia has been widely used in restorative dentistry...
owing to superb biocompatibility and mechanical strength [4]. Although, latest advances in zirconia ceramics aimed to produce more translucent zirconia restorations with monolithic systems, it is still opaque compared to other ceramic systems. Thus, the zirconia is prepared as a core material and veneered by feldspathic ceramic to acquire superb esthetic [5].

Zirconia is a familiar polymorph and has three allotropes; monoclinic, tetragonal and cubic. The monoclinic phase is stable at room temperature up to 1170 °C. Between 1170 and 2370 °C, the tetragonal phase exists and over 2370 °C the cubic phase occurs. Melting point of the cubic phase zirconia is 2680 °C [6]. Several metallic oxides (e.g., MgO, CaO, Ce₂O₃ or Y₂O₃) were added to the zirconia in order to obtain exact molecular stability and utilize the transformation toughening to control phase transformation between the tetragonal and monoclinic crystal structures. However, yttria stabilized tetragonal zirconia polycrystalline (Y-TZP) has shown greater strength compared to other molecular types of zirconia [7].

As a high-strength crystalline content ceramic, Y-TZP has been used as core material for crowns and bridges. Apart from the good biocompatibility and esthetic feature of the Y-TZP, it exhibits brilliant mechanical properties; high flexural strength (700–1200 MPa) and high fracture toughness (7–10 MPa m¹/²) [8]. Therefore, the Y-TZP has been accepted as suitable and durable core material for FPDs [9].

Computer-aided design/computer-assisted manufacture (CAD/CAM) systems ensure to fabricate enduring and stable zirconia cores for single crown and FPD [6]. Two different zirconia milling techniques (soft-milling and hard-milling) are frequently used to produce zirconia frameworks. Partially sintered zirconia blocks are milled on the soft-milling procedure and they illustrate a linear dimensional shrinkage (20–25%) during sintering stage. The latter describes milling fully sintered zirconia blocks, so that any sintering process or heat treatment are not involved and the frameworks are milled to exact dimensions [10].

Lots of clinical studies showed that zirconia based restorations (ZBR) have illustrated high survival rate up to 5 years in mastication [11–15]. However, zirconia sintering process and structural defects, surface treatment methods such as sand-blasting, etching and grinding, design of the framework, repeated porcelain firings, type of finishing margins, luting procedures and zirconia aging may cause to possible chipping in the ZBR [16–32]. Although, Y-TZP framework shows higher strength, the veneer layer, and junction between the framework and the veneer display weaker points for the ZBR. Micro-crack propagations initiate from these layers of the restorations and chippings may occur unexpectedly. As a result of that, the underlying zirconia core may expose or the fracture may restrain in the veneering porcelain. These failure modes have been frequently encountered by the clinicians [13].

The fractured restorations should be immediately replaced with new one or repaired using appropriate repairing material [14,15]. Despite, the replacement of the ZBR has been presented as conventional method when faced to failure of the restorations, repairing the fractured restoration are taken into account, because of replacement cost, limited-time concerns, patient associated problems and other risk factors by the clinicians [3]. Contemporary intraoral porcelain repairing systems (IPRSs) increase survival rate of the all-ceramic restorations, either cost or time efficiency can be enabled using them [14,15].

Adhesion between the composite resin and feldspathic ceramic may be strongly obtained etching with hydrofluoric acid. But, glass-free polycrystalline structure of the zirconia has not been affected by hydrofluoric acid etching and bond strength of the composite resin to the zirconia may be weak [4,33]. In addition, hydrofluoric acid is a dangerous material to use intraorally [3]. Therefore, several surface treatment methods described previously to establish suitable bond strength to composite resins. Grinding with bur, tribochemical silica coating, sandblasting, acid etching, laser irradiation, and zirconia primers have been applied to surfaces for surface conditioning [2,34–40].

Lasers have been recently introduced as a surface treatment method and chair-side application utilizes easy-to-use for clinicians. Carbon dioxide (CO₂), and neodymium-doped yttrium aluminum garnet (Nd:YAG) have been applied for this purpose and several researchers analyzed effects of these on the different surfaces [10,38,41–43]. Another effective hard tissue laser is erbium chromium: yttrium scandium garnet (Er,Cr:YSGG) that was investigated effects on the zirconia substrates [44–46]. The latter has been recently used for surface treatment method on the zirconia, but adequate data was not described related to effects on porcelain and zirconia surfaces for intraoral repair.

Theory/Calculation

The core goal of this study was to investigate repair bond strength between composite resin, and zirconia and porcelain surfaces using different Er,Cr:YSGG laser pulses. The theory was that different laser pulses would not affect the repair bond strength on the porcelain surface, but would increase that on the zirconia surface.

Material and methods

Sixty zirconia disc shaped specimens were prepared. Zirconia discs (ICE Zirkon Translucent, Zirkonzahn GmbH, Gais,
Germany), 5 mm in diameter and 2 mm in height, were fabricated using CAD/CAM technology (Zirkonzahn GmbH). Randomly selected thirty zirconia discs were veneered with 1 mm porcelain layer (VITA VMK Master, Vita Zahnfabrik H. Rauter GmbH & Co. KG, Bad Säckingen, Germany) for porcelain groups (Fig. 1). The prepared specimens were cleaned at ultrasonic bath for 3 min in 96% isopropyl alcohol and steam-cleaner were applied for 10 s. Specimens at the both group were randomly divided into three sub-groups and each of them contained 10 samples.

Three different surface treatments were applied to the zirconia and porcelain surfaces. As a control, the surface of the samples was set according to manufacturer recommendations of an IPRS (Ceramic Repair N, Ivoclar Vivadent AG, Schaan, Liechtenstein). The surface of them was grinded with a diamond bur (FG 3053G, KG Sorensen, Cotia, SP, Brasil) using water irrigation.

For laser application to bonding surfaces of the samples, Er,Cr:YSGG laser (Millennium; Biolase Technology, Inc., San Clemente, CA, USA) irradiation was performed. The laser irradiation was applied at a 2.78 μm wavelength. Pulse duration was set 140 μs for short-pulse and 200 μs for long-pulse laser irradiation. Repetition rate of irradiation was 20 Hz and the output power of Er,Cr:YSGG laser equipment ranges from 0.25 mm to 6.0 W. Zirconia and porcelain surfaces were scanned perpendicularly at 10 mm distance for 20 s by a 600 μm diameter laser optical fiber. The table illustrated that tested groups in this study (Table 1).

After the surface treatments, an additional sample from each group was examined using a scanning electron microscope (SEM) (JSM 6060, JEOL USA, Peabody, MA, USA). Firstly, the samples were dried and then, a sputter-coated device (Polaron SC7620 Sputter Coater, VG Microtech, West Sussex, England) was used to coat the samples with gold and palladium. SEM images were recorded at 5,000× magnification (Fig. 2).

All specimens were ultrasonically cleaned in a bath of 96% isopropyl alcohol for 3 min and dried with oil-free air. Monobond N (Ceramic Repair N) was applied with a brush to surface of the specimens. 60 s waited to allow reaction and subsequently, dried with oil-free air. A thin layer of HelioBond (Ceramic Repair N) was brushed to the entire surface of the specimens. Any excess material was removed with compressed air and light-cured with a high performing LED polymerization light (Astralis 3, Ivoclar Vivadent AG, Schaan, Liechtenstein) at a distance of 1 mm for 10 s. A composite resin (Tetric N-Ceram, Ivoclar Vivadent AG, Schaan, Liechtenstein) was incrementally applied and adapted using suitable instruments to center of the surfaces 2 mm in height and 2 mm in diameter via standard teflon mold. Each composite resin layer was individually light-cured (Astralis 3) at a distance of 1 mm for 40 s. Cured composite cylinders were polished using a silicone polisher (Astropol, Ivoclar Vivadent AG, Schaan, Liechtenstein).

The specimens repaired with composite resin were mounted in polypropylene random copolymer (PPRC) cylinder with an acrylic resin (Paladent, Heraeus Kulzer GmbH&Co.KG, Hanau, Germany) and they were stored in distilled water at 37 °C for 24 h. Then, the specimens were placed at a universal testing machine (AGS-X, Schimadzu Co., Kyoto, Japan) and a knife shaped indenter applied at a crosshead speed of 1 mm/ min for shear bond strength (SBS) (Fig. 3). These data were used to calculate the mean failure load and standard deviation for the groups. Also, the maximum loads at failure were recorded and the following terminology was implemented. Failure modes on the repaired surfaces as adhesive failure, cohesive failure, the fracture line was confined in the composite resin; and mixed failure, both of the failure types (adhesive and cohesive) were experienced.

The obtained data from SBS test were normally distributed according to the Shapiro–Wilk test (p > 0.05) and homogeneity test of variance (p > 0.05). The results were statistically analyzed by a parametric test, one-way ANOVA. Tukey was applied to groups for pairwise comparison of SBS data. The statistical analysis was performed at a 95% confidence level using a statistical analysis software package (PSPP 1.0.1, GNU, FSF Inc, Boston, MA, USA).

**Results**

Shear bond strength values of the tested groups were statistically analyzed, and the means and standard deviations of the groups were summarized in Table 2. Control group for porcelain surface, Group PC, showed that highest mean bond strength value followed by Group ZS. The lowest mean bond strength was found in Group PS.

Group PC, is control group in porcelain groups, illustrated remarkably higher bond strength among other porcelain groups. (p < 0.05) Mean bond strength of Group PL significantly increased compared to Group PS. (p < 0.05).

For zirconia groups, laser surface treatment applied groups (Group ZS and ZL) showed higher mean bond strength than control group (Group ZC). Mean bond strength value of Group ZS was found better than long pulse laser surface treatment.
Failure modes on the surfaces were also assessed after the shear bond strength test. Observed failure types at each group were illustrated in Table 3. Failures on the composite-zirconia interface were predominantly adhesive failure (90% of failures) and followed by mixed failure of adhesive and cohesive (10% of failures). The entire observed failure modes were adhesive on the composite-porcelain interface.

Evaluation of the SEM images performed after the surface treatment procedures (grinding with bur, short-pulse and long-pulse laser irradiation) under a magnification of 5,000×. SEM evaluation of the treated surfaces showed that diverse roughness patterns. For the zirconia groups, surface of Group ZC showed uniform morphological pattern that included parallel scratches compatible with direction of the grinding bur. Group ZS illustrated that homogeneous surface roughness and micro-retentive grooves. Group ZL demonstrated parallel shallow fissures and scratches.

For the porcelain groups, the control group, Group PC, showed that irregular surface pattern and wide cracks with deep fissures and high ridges. Group PS demonstrated shallow fissures and grooves. Surface of Group PL illustrated deeper uniform fissures and grooves than Group PS.

**Discussion**

Two different clinical scenarios were evaluated in this study. Porcelain groups were represented failed restoration that fracture didn’t reach to zirconia and veneering porcelain interface. Zirconia groups were exemplified complete veneering porcelain fracture in the ZBR. Efficacy of different pulse rates of Er,Cr:YSGG laser surface treatment procedure on shear bond strength was evaluated using single IPRS. The null hypothesis of the current study was partially accepted according to the results. So, laser surface treatment procedures were not enhanced repair bond strength of the porcelain surfaces compared to control group regardless of laser pulse rates. Laser applications on the Y-TZP surfaces were increased repair bond strength, but these were not statistically significant than control group.

Nowadays, all ceramic restorations are popular on dental applications among both of dentists and patients. ZBRs come front compared to other type of all ceramics because of

| Table 2 Mean and standard deviations of shear bond strength. |
|-----------------|-------|-------|------------------|
| Material        | Group | Mean  | Standard Deviations |
| Zirconia        | Group ZC | 159.53 | 63.33 |
|                 | Group ZS | 174.77 | 48.79 |
|                 | Group ZL | 163.21 | 69.20 |
| Porcelain       | Group PC | 249.33<sup>a,b</sup> | 57.19 |
|                 | Group PS | 132.25<sup>a,c</sup> | 47.06 |
|                 | Group PL | 149.70<sup>b,c</sup> | 46.28 |

Different upperscript letters indicate statistical differences (p < 0.05).
superior mechanical properties, biocompatibility and esthetic features [5].

Zirconia frameworks ensure versatility for dentists regardless of restoration zone [9]. One of the most encountered drawback of the zirconia veneered porcelain restoration is veneer chipping or crack. Expose the underlying zirconia have still shown high incidence [11,13].

Raigrodski et al. [9] stated that 25% of zirconia ceramics were fractured at 31 months follow-up period, and Sailer et al. [12] reported that 13% of veneer ceramics were also cracked during 3 years. Lots of causes described to explain these fractures, such as unfitting framework support, surface failures, excessive loads and fatigue, low mechanical properties of the veneering porcelain, fabrication process of ZBRs (e.g. sintering, surface treatment, porcelain firing, and morphology of the restoration) [16–18].

The failed restorations should be removed and replaced with newer or repaired as soon as possible [14,15]. Complete replacement is time consuming and costly, and removal of the cracked restoration may impair to tooth and periodontal tissues. In contrast, repair of the restoration is more convenient, atraumatic and acceptable treatment modality than replacement of the restoration [47].

During the past decades, intra-oral porcelain and zirconia repairing systems have been put in the market by the dental companies. The bonding strength of porcelain or zirconia surface and composite resin is still problematic [35]. Therefore, many surface treatment methods for example, acid etching, grinding with bur, tribochemical silica coating, sandblasting, selective infiltration etching, laser irradiation, and application of metal alloy and zirconia primers were used to enhance bonding strength by researchers [1,2,34–37].

Blum et al. [47] investigated efficacy of three different IPRSs and merely CoJet(®) application on the ceramic samples and concluded that the tensile bond strength was not effected from the type of IPRS. Surface treatment with CoJet(®) system showed that statistically significant higher bond strength than use of other IPRSs. In the light of this data, solely one IPRS (Ceramic Repair N) was used to repair zirconia and porcelain surfaces.

Bond strength of the composite resin to glass-free polycrystalline structure of the zirconia needs to be improved [4,33]. Thus, surface conditioning methods may be applied to zirconia surfaces to increase surface energy and wettability [38,44]. Han et al. [39] investigated effect of CoJet(®) system on the shear bond strength of zirconia to composite resin. They reported that this particle abrasion silica coating method illustrated higher values but it was not statistically significant. Similarly, Shin et al. [40] studied same surface treatment method on the Y-TZP surface and compared with Al₂O₃ airborne particle abrasion and zirconia primer application. The authors concluded that all surface conditioning procedures increased shear bond strength but solely zirconia primer application after airborne abrasion was showed statistically significant difference.

Tribochemical silica coating and roughening with alumina particles of zirconia surfaces are still enhanced or another surface treatment method should be implemented in the zirconia repairing process [44].

Intra-oral application of laser irradiation is an innovative procedure to obtain surface roughness [48]. Thus, it was applied to the ceramic and zirconia for surface conditioning [42,43,45,46,48,49]. Nd:YAG laser was used in that manner and investigated effect on the shear bond strength in the several studies [38,42,43]. Nd:YAG laser was applied on aluminabased ceramics and effective surface roughening was presented by da Silveira et al. [42]. In another study, Nd:YAG laser was used as a surface treatment method on feldspathic porcelain and the bond strength value was found similar to hydrofluoric acid etching [49]. Usumez et al. [38] applied same laser irradiation to Y-TZP surface and suggested that this laser treatment might be used as a surface conditioning method. Paranhos et al. [48] reported that Nd:YAG laser treatment illustrated more roughness on the zirconia than other abrasive methods.

Nd:YAG laser was used with different power settings and irradiation time to roughen zirconia ceramic by Liu et al. [43] who stated that the laser irradiation was successfully roughed zirconia and ceramic surfaces. However, differences between the laser groups and control group were not found statistically significant. In addition, any significant differences of the shear bond strength were observed among laser groups. The authors concluded that increasing power of laser irradiation and increasing time might not improve repair bond strength of the ceramic and zirconia surfaces.

In the literature, apart from the Nd:YAG laser irradiation, a few study conducted using Er,Cr:YSGG laser to enhance repair bond strength of the zirconia ceramics [44–46]. Kirmali et al. [44] evaluated effect of different surface treatment methods including Nd:YAG and Er,Cr:YSGG lasers, and combination with silica coating and sandblasting on the repair bond strength between zirconia and composite resin. The researchers reported that Er,Cr:YSGG laser application and combination of Nd:YAG laser and sandblasting enhanced repair bond strength of the zirconia-composite resin interface.

Eduardo et al. [46] applied Er,Cr:YSGG laser with different power settings range from 0.5 to 5.0 W on glass infiltrate alumina. They concluded that effects of laser irradiation were acceptable. Ghasemi et al. [45] investigated effect of airborne particle abrasion and Er,Cr:YSGG laser with 2 and 3 W power surface conditioning processes on shear bond strength of sintered and non-sintered zirconia to composite. Air abrasion and Er,Cr:YSGG laser irradiation showed better bond strength values compared to control group. Previous studies changed the power of laser irradiation and showed that applied powers were desirable for surface roughening, nonetheless evaluated

| Table 3 Failure types and the specimens. | Group ZC | Group ZS | Group ZL | Group PC | Group PS | Group PL |
| Adhesive | 10 | 9 | 8 | 10 | 10 | 10 |
| Mix | 0 | 1 | 2 | 0 | 0 | 0 |
effect of pulse rates on the surface roughness and repair bond strength. In this study, Er,Cr:YSGG laser was used standard power setting, but different pulse rates were applied to the zirconia and porcelain surfaces.

In the current study, SEM analysis of the specimens showed that, all of the surface treatment procedures obtained adequate surface roughness to bond composite resin. Porcelain surface grinding with bur illustrated detailed surface topography compared to laser surface treatment methods. Short pulse laser application to zirconia surface exhibited deeper grooves and fissures than other zirconia groups. Shear bond strength test results of the study were represented as similar to SEM analysis.

Overall, short pulse laser irradiation on the porcelain samples showed that lowest bond strength and control group for porcelain illustrated highest bond strength among the tested groups. Short pulse Er,Cr:YSGG laser irradiation showed highest mean shear bond strength value for zirconia surfaces. Although short and long pulse laser irradiations illustrated higher bond strength values than control group on the zirconia, differences between the tested groups were not statistically significant.

The failure mode analysis of the tested samples showed that the fracture pattern was generally adhesive failure, apart from that several mix failures was observed on the Er,Cr:YSGG laser applied on the zirconia samples. This observations were similar to previous reports in that field [44,48].

**Conclusions**

Within the limitations of the present study, the following conclusions could be drawn:

1. Repair bond strength between zirconia and composite resin can be increased applying Er,Cr:YSGG laser with different pulses. Furthermore, short pulse laser irradiation showed better mean shear bond strength compared to long pulse rate. But, these were not statistically significant.
2. Porcelain control group, grinding with diamond bur, experienced highest bond strength among the tested groups.
3. Failure modes of the fractured patterns were observed as adhesive failure, except of several patterns of the laser applied zirconia surfaces which illustrated mix failure.
4. Er,Cr:YSGG laser irradiation may be securely used as a chairside surface treatment method when repairing of zirconia ceramics.

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**Appendix A. Supplementary data**

Supplementary data to this article can be found online at https://doi.org/10.1016/j.bj.2019.02.001.

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