Er,Cr:YSGG laser irradiation influence on Y-TZP bond strength to resin cement☆

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Received 28 April 2016 Received in revised form 25 May 2016 Accepted 27 May 2016 Available online 28 May 2016

ARTICLE INFO

Article history:
Received 28 April 2016 Received in revised form 25 May 2016 Accepted 27 May 2016 Available online 28 May 2016

Keywords:
Zirconia Bond strength Er,Cr:YSGG laser

ABSTRACT

The aim of this study was to evaluate Y-TZP surface pretreatment with different protocols on microshear bond strength (μSBS) ceramic-cement interface. One hundred and sixty pre-sintered IPS e-max ZirCAD (Ivoclar-Vivadent) blocks were randomly divided into sixteen groups according to surface treatment (n=20): G1- no treatment (control); G2- ceramic primer; G3- tribochemical silica coating; G4- tribochemical silica coating+primer; G5- airborne particle abrasion (Al2O3); G6- airborne particle abrasion (Al2O3)+primer; G7- Er,Cr:YSGG laser; G8- Er,Cr:YSGG laser + prism. All specimens were sintered before surface treatment, except lasers groups, which were sintered after laser irradiation. Ceramic blocks were bonded with Panavia F resin cement (Kuraray, Okayama, Japan) (n=10) or RelyX ARC (3M ESPE, St. Paul, MN, USA) (n=10). The μSBS tests were carried out in a universal testing machine at a speed of 1mm/min after 24 h (n=5) or 6 months storage (n=5). Differences were found for both resin cements and storage conditions in relation to μSBS values (p<0.05). However, no significant difference for interaction between factors was observed in cemented blocks with RelyX ARC. Panavia F resin cement showed significant differences for interaction between factors (p<0.05). Laser treatment was not sufficient to increase μSBS values between Y-TZP and resin cements. Tribochemical silica coating followed by primer achieved the highest immediate μSBS values. The storage did not affect negatively μSBS values to both evaluated cements.

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1. Introduction

Clinical success of polycrystalline ceramics depends on adequate adhesion to dental elements, which allows microleakage prevention, increased retention, better marginal adaptation, and higher fracture resistance [1–5]. Chemical and/or mechanical surface treatment provides a reliable adhesive bonding to resin cements and ceramic on metal-free prosthetic restorations [3,4]. However, several studies postulate about the difficulties of obtaining adhesion of these materials to dental structures because their superficial characteristics and composition compromise clinical longevity rehabilitation treatment [2–4,6–9].

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) is a biocompatible and aesthetic material that presents particularly superior mechanical properties when compared to feldspatic, leucite, and lithium disilicate ceramics [10]. Although conventional techniques for cementation with zinc phosphate and resin-modified glass ionomer cement can be applied to zirconia surface, adhesive cementation has been recommended as more appropriate to obtain marginal sealing and adequate retention [4,11]. However, to achieve a stable bond with adhesive cementation, it is necessary to prepare the ceramic surface with chemical-mechanical or mechanical treatments [12].

Ceramic surfaces without silica dioxide are not conventionally conditioned by hydrofluoric acid and constitute major problems for adhesive cementation [1,3,4,6,9]. In order to overcome this difficulty, different protocols have been used, such as airborne particle abrasion [4,13–17] tribochemical silica coating or silicatization [14,15], and selective infiltration technique [16,18].

Recently, laser treatment has been investigated, using different wavelengths, such as Er:YAG, Nd:YAG, CO2 and Er,Cr:YSGG irradiation in...
for conditioning ceramic surfaces before adhesive procedures. Akyil et al. [15] reported that Er,Cr:YSGG laser irradiation with 2 W produced similar roughness to airborne-particle abrasion, with a better bond strength than the control group. In addition, use of metallic primers [22,23], associated with phosphate monomers-based resin cements [4,23] and association of phosphate monomer-zirconate based coupling agents [24] have been recommended with the objective of improving adhesion between ceramic-resin cement-teeth. Other studies assessed surface treatment versus sintering procedure, with promising results with conditioning prior to sintering Y-TZP [25,26]. Sandblasting pre-sintered Y-TZP before sintering may increase the proportion of tetragonal structures with enhanced clinical performance of zirconia restorations [26] and the laser could be used with the same purpose.

Therefore, the aim of this study was to evaluate Y-TZP surface treatment with different protocols on microshear bond strength ceramic-cement interface. The null hypothesis was that the laser irradiation protocol does not increase bond strength values compared to other treatments.

2. Materials and methods

Blocks of pre-sintered Y-TZP (B-40 L, emax ZirCAD, Ivoclar-Vivadent, Schaan, Liechtenstein Principate) with $6 \times 6 \times 4$ mm$^3$ initial dimensions were used. In order to simulate the CAD/CAM system, a cylinder pointed bur (Sirona Bensheim, Germany) compatible with CEREC$^\text{®}$ Inlab$^\text{®}$ Compacta (Sirona, Bensheim, Germany) was mounted to low speed (Kavo, Joinville, Santa Catarina, Brazil) underwater irrigation, and the surface was standardized to all blocks.

2.1. Confocal white light microscope

Surface topography of zirconia, including ten blocks of control (n=5) and irradiated groups (n=5), was investigated using a confocal light microscope (Leica Scan DCM 3D - Leica Microsystems Ltd, Switzerland) with objective magnification 50 × in a pilot study. The purpose of the pilot study was to assess if there is roughness increase after laser treatment prior to sintering Y-TZP.

The control group was observed after cylinder pointed bur abrasion. Ceramic specimen surfaces were coated with graphite to increase energy absorption prior to laser irradiation in the experimental groups. Surfaces were then treated with Er,Cr:YSGG laser in the parameters: $\lambda=2780$ nm, 600 µm quartz core tip, 3 W, 20 Hz, 53.57 J/cm$^2$, 1 mm distance, for 30 s, focused mode and air-water cooling proportion 65%/35%. (iPlus, Waterlase, Biolase Technologies Inc., Irvine, CA, USA). Each sample was irradiated once in each direction, moving the handpiece slowly, horizontally and vertically, to promote homogeneous irradiation of entire sample area. The Sa and Ra mean values were calculated from five profiles from control and irradiated groups. Superficial micromorphology image from evaluated areas and one opposite surface area were obtained ($50 \times$).

2.2. Experimental design

One hundred and sixty blocks of Y-TZP were bonded with one of two resin cements and adhesive association (n=80) (either RelyX ARC and Adper Single Bond 2 or Panavia F and Clearfil) [27]. Table 1 describes composition of the selected materials for the study.

All specimens were sintered before surface treatment, except lasers groups, which were sintered after laser irradiation. Specimens were sintered in the Zyrcomat furnace (Vita Zahnfabrik, Germany) at 1520 °C for 2 h. The final dimensions of the blocks were $5 \times 5 \times 3$ mm$^3$, following 20% volumetric shrinkage associated with sintering. Afterwards, specimens were ultrasonically cleaned with 96% isopropanol for 3 min.

Each group (n=20) was divided into two subgroups depending on the luting system applied (either RelyX ARC and Adper Single

<table>
<thead>
<tr>
<th>Commercial brand</th>
<th>Manufacturer</th>
<th>Batch no.</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>B40-L, Vita In-Ceram Zirconia</td>
<td>Ivoclar Vivadent, Germany</td>
<td>K14590</td>
<td>Zirconium oxide (ZrO$_2$) and aluminum oxide (Al$_2$O$_3$)</td>
</tr>
<tr>
<td>RelyX$^\text{TM}$ Ceramic Primer</td>
<td>3M ESPE, Seefeld – Germany</td>
<td>N316733</td>
<td>3-methacryloxypropyltrimethoxy silane in ethanol (MPTS)</td>
</tr>
</tbody>
</table>
| RelyX ARC | 3M ESPE, St. Paul, MN, USA | 1302900656 | Cement: Bis-GMA, TEG-DMA, zirconia
Paste A: MDP; hydrophobic aromatic dimethacrylate
Past B: hydrophobic aromatic dimethacrylate; hydrophobic aliphatic methacrylate; hydrophilic aliphatic dimethacrylate; silanated; silanated colloidal silica; camphorquinone; catalysts; initiators
Conditioner: etchant (37% phosphoric acid) |
| Panavia F2.0 | Kuraray Medical Co, Okayama, Japan | 311A 185A 255A 31A | Hydrophilic aliphatic methacrylate; hydrophilic aliphatic dimethacrylate; silanated; silanated colloidal silica; camphorquinone; catalysts; initiators |
| Clearfil Porcelain Bond Activator | Kuraray, Okayama, Japan | 270A 1147A | Hydrophilic aromatic dimethacrylate; 3-Methacryloxypropyl trimethoxy silane SE Bond Primer: HEMA; MDP; hydrophilic aliphatic dimethacrylate; camphorquinone; water; accelerators; pigments |
| Clearfil Ceramic Primer Aluminum Oxide | Kuraray, Okayama, Japan | 23A 2498 | 3-methacryloxypropyl trimethoxy silane (MPTS), MDP, Ethanol |
| Rocatec Soft$^\text{®}$ | 3M ESPE, St. Paul, MN, USA | 1304010478/506373 | Aluminum oxide silicic acid modified (Al$_2$O$_3$+SiO$_2$, 30 µm) |

Abbreviations: BisGMA, bisphenylglycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; BP, biphenyl phosphate; HEMA, 2-hydroxyethylmethacrylate; DMA, dimethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA, N-Methacryloyl-5-aminosalicylic acid; BPEDMA, bisphenol-A polyethoxymethacrylate; TBB, tri-n-butyl borane catalyst.
Bond 2 or Panavia F and Clearfil). According to the experimental groups, blocks were randomly assigned to one of the eight groups, corresponding to eight surface treatment protocols (n = 10).

G1- Control group: no mechanical surface treatment before sintering.

G2- Primer: the blocks received the RelyX Ceramic Primer (3M-ESPE, Hanover, Germany) or the Clearfil Ceramic Primer (Kuraray, Okayama, Japan).

G3- Rocatec: the blocks were abraded using tribochemical silica-coating (30 μm particles) (Rocatec Soft; 3M-ESPE, St Paul, MN, USA) at 2.8-bar pressure for 15 s from a distance of 10 mm perpendicular to the treatment surface of the blocks.

G4- Rocatec+Silano: the blocks were abraded using tribochemical silica-coating (30 μm particles) (Rocatec Soft; 3M-ESPE, St Paul, MN, USA) at 2.8-bar pressure for 15 s from a distance of 10 mm perpendicular to the treatment surface of the blocks. Afterwards, blocks received RelyX Sil (3M-ESPE, St Paul, MN, USA) or Clearfil Porcelain Bond Activator (Kuraray, Okayama, Japan).

G5- airborne particle (Al2O3) abrasion: the blocks were abraded using 50 μm Al2O3 particles (Bioart, São Carlos Brazil) at 2.8-bar pressure for 15 s from a distance of 10 mm perpendicular to the treatment surface of the blocks.

G6- airborne particle (Al2O3) abrasion+Primer: the blocks were abraded using 50 μm Al2O3 particles (Bioart, São Carlos Brazil) at 2.8-bar pressure for 15 s from a distance of 10 mm perpendicular to the treatment surface of the blocks followed by the silane.

G7- Laser: the ceramic specimen surfaces were coated with graphite (pencil) to increase the laser energy absorption. Surface treatment was performed as previously described in roughness evaluation.

G8- Laser + primer: the surface treatment was similar to G7, and the blocks received the correspondent primer.

2.3. Bonding procedure

Two different adhesive resin cement systems—Panavia F (Kuraray Co. Ltd, Okayama, Japan) and RelyX ARC (3M ESPE)—were used to bond using a Tygon plastic tube mold (Tygon, Norton Performance Plastic Co, Cleveland, USA) (1-mm thickness with a 0.75-mm internal bore diameter) and applied according to the manufacturers’ instructions. Tygon was positioned in the specimens in four different regions. Cement was carefully packed into the tube against the ceramic substrate and light-polymerized for 20 s (Radii Cal Plus, SDI Limited, Bayswater, Victoria, Australia; light intensity 997 mW/cm²). Specimens were stored for 24 h or 6 months at 100% humidity storage at 37°C.

2.4. Microshear bond strength test (μSBS)

Microshear bond strength test was performed on a universal testing machine (EZ Test; Shimadzu Corp, Kyoto, Japan) after 24 h in half of the specimens from each subgroup (n = 5), and the other half after 6 months. A thin wire (0.2 mm diameter) was looped around a resin cement cylinder, making contact with half its circumference at the resin/ceramic interface, and a shear force was applied at a crosshead speed of 1 mm/min until debonding.

2.5. Failure mode

After debonding, failure modes were observed under a stereo microscope (PanTec, Panambra Ind. Técnica SA, Sao Paulo, Brazil) at 50 × magnification as fitting into one of the following categories: (A) adhesive failure at the interface between ceramic and resin; (B) cohesive failure within resin; and (C) mix failure, with 50% adhesive failure and 50% cohesive failure. The percentage of each failure mode was calculated for the control and experimental groups.

2.6. Statistical analysis

Roughness data (Ra) was statistically analyzed by two-way analysis of variance (ANOVA) at 95% confidence level (α = 0.05) and Tukey tests (p < 0.05). Microshear bond strength was recorded, and values (MPa) were submitted to statistical analysis using two-way ANOVA (α = 0.05) followed by Tukey test to assess differences between each group according to storage condition (p < 0.05) (Biostat, Maringa, PR, Brazil).

3. Results

Table 2 reports effects of Er,Cr:YSGG laser irradiation on the Ra Y-TZP material. There were significant differences in ‘surface treatment and storage condition’ for Ra values (p < 0.05), but no differences for interaction of factors (p = 0.766). After sintering and irradiation, Ra values increased (Table 2).

Regarding ANOVA, statistically significant differences were found for microshear bond strength (μSBS) values (p < 0.05) for both cements evaluated. Significant differences were observed in ‘surface treatment’ and ‘storage condition factors’ (p < 0.05), without interaction between factors for RelyX ARC resin cement. Table 3 presents mean, standard deviation, and results of Tukey test for Rely X ARC cement material.

Laser treatment before sintering was not sufficient to increase μSBS values between Y-TZP and RelyX ARC resin cement. Tribochemical silica coating, followed by primer (G4-RX), achieved the highest immediate μSBS values. The control group (G1-RX), airborne particle abrasion group (G5-RX) and laser treatment before sintering group (G7-RX) presented the lowest μSBS values, independent of storage conditions. Primer increased μSBS values between RelyX ARC and Y-TZP. Furthermore, storage had a positive influence on RelyX ARC μSBS values.

ANOVA showed statistically significant differences between ‘surface treatment’ and ‘storage condition’ and interaction of factors relative to microshear bond strength (μSBS) values (p < 0.05) for Panavia F resin cement (Table 4).

After 6 months of storage, μSBS values increased in control group (G1-PF), primer group (G2-PF), and airborne particle abrasion group (G5-PF), without differences to other groups. The Er,Cr: YSGG laser irradiation group (G7-PF) presented the lowest μSBS values, with more favorable results in the 24 h evaluation. Airborne particle abrasion, followed or not by primer, such as tribochemical silica coating followed by primer, presented higher μSBS values to Panavia F cement.

The distribution of failure modes among luting materials is shown in Figs. 1 and 2. Predominant failure mode of RelyX ARC was adhesive, with the exception of the tribochemical silica coating into one of the following categories: (A) adhesive failure at the interface between ceramic and resin; (B) cohesive failure within resin; and (C) mix failure, with 50% adhesive failure and 50% cohesive failure. The percentage of each failure mode was calculated for the control and experimental groups.

Table 2

<table>
<thead>
<tr>
<th>Parameter/ treatment</th>
<th>Pre-sinterization (n = 5)</th>
<th>Post-sinterization (n = 5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control (n = 5)</td>
<td>0.61 [0.04]</td>
<td>1.87 [0.26]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.50 [0.78] B</td>
</tr>
<tr>
<td>3 W/20 Hz/30 s</td>
<td>1.1 [0.32]</td>
<td>2.43 [0.58]</td>
</tr>
<tr>
<td>(n = 5)</td>
<td></td>
<td>1.77 [0.81] A</td>
</tr>
<tr>
<td></td>
<td>0.85 [0.33]</td>
<td>2.15 [0.48] A</td>
</tr>
</tbody>
</table>

Means followed by different letters in the row or in the column indicate statistical differences (p > 0.05); n = sample number.
coating followed by primer group (G4-PF) that presented increased cohesive failure mode (Fig. 1).

According to distribution of failure modes to Panavia F, all groups presented cohesive and mix failure mode as well as adhesive failure, with the exception of the control group, which did not present cohesive failure mode (Fig. 2).

**4. Discussion**

Optimal bond strength between resin cements and zirconia is difficult to achieve. The composition of zirconia prosthetic restorations with no vitreous component does not allow hydrofluoric acid to be applied [3,20]. Several adhesive strategies have been suggested to overcome this problem. The most common are based on the zirconia ceramic surface change, increasing roughness [13,17,28–31], and obtaining chemical bonding, as well as the use of laser [17,19,21].

Theoretically, airborne particle abrasion could increase bond strength with increased roughness [17,30]. Tribochemical silica coating is a chair side surface treatment that has in its composition alumina coated by silica particles [15,19,20,32]. This technique cleans the surface and creates a reactive silica outer layer that can
be silanized prior to bonding procedures [33], combining both micromechanical and chemical adhesion. De Castro et al. [34] study showed that the tribosilicatization bonding protocol was not affected by water storage for Panavia or Rely X ARC resin cements and those results are in accordance with the present study. It has been reported no significant changes after hydrothermal recycling with phosphate monomer (MDP) resin cement and surface pre-treatment [5,35].

Moreover, surface treatments and cements have been also proposed to improve adhesion between ceramic tooth interface, such as airborne abrasion and phosphate monomer-based resin cement combination, to obtain a stable and durable adhesion to zirconia [36]. The hydrolytic action of water on adhesive surfaces reduces up to 50% bond strength compared with the baseline [35]. The storage condition of the present study was 100% humidity and other studies methodology describe water storage [4,35]. Blatz et al. [4] did not find reduced bond strength values after long-term water storage using a ceramic primer containing the phosphate monomer MDP. Resin cements with MDP, such as Panavia, have inorganic compounds that create resistance to hydrolysis [5,35] and this can explain the microshear bond strength values after 6 months.

The surface roughness is an important parameter and could increase bonding strength values [25,26]. Laser treatment prior to sintering Y-TZP showed promising results. The present study found differences to Er,Cr:YSGG laser irradiation to Ra values (Table 3) that presented higher values compared to control group. Airborne particle abrasion also is related to increased bond strength values [26,35] and the present study also presented increased microshear values after this treatment. MDP primers enhance the zirconia bonding values of acrylic resin cements [35] and this behavior has been observed in the present study. Moreover, the addition of an MDP-containing bonding/silane coupling agent produced positive results and enhanced bonding of MDP resin cements [5].

It has been reported that phosphate-containing cements, like Panavia and non-phosphate monomer-containing cements like RelyX ARC exhibited high bond strength [5]. Tzanakakis et al. [35] results showed that the resin cements with the highest long-term bond strength contain MDP or use MDP-primers. Rely X Ceramic Primer has 3-methacryloxypropyltrimethoxy silane in ethanol (MPTS) (Table 1) in its composition. The coupling agent silane enhance the wettability capacity of an inorganic surface [35], but the increased microshear values after this treatment also were related to pre-failure test increase (Table 3). Tribochemical silica coating enhances bonding capacity, especially when silanes are applied [35] in accordance with the results of the present study.

Different laser wavelength has been described as an alternative to modify a zirconia surface, including Nd:YAG laser (1064 nm) [15,21,32], Er:YAG laser (2940 nm) [15,28,33], CO2 laser (10,600 nm) [15,21], and Er,Cr:YSGG laser (2780 nm) [19]. Zirconia surface texture changes according to the type of laser and wavelength that was used. Er:YAG has been studied as a post-sintering surface treatment [15,26,28,33], reaching increased bonding strength values [19] or not [19,33].

Foxton et al. [17] analyzed Er:YAG laser irradiation setting 200 mJ at post-sintered zirconia and observed a rough and damaged surface, including "cracks"; dark areas, probably due to "melting"...
after laser irradiation, were also described. The increased surface area could increase adhesion but bond strength values were not higher after laser irradiation. On the other hand, signs of fusion and solidification without “cracks” or increased bonding strength values had been reported after Er:YAG laser irradiation [33]. The tribochemo silica coating group presented higher μSBS values, corroborating the findings of another study [35]. Thereby, increased adhesion could be expected after physicochemical conditioning of zirconia.

According to the results, tribochemo silica coating followed by primer achieved the highest μSBS values. In turn, Er:Cr:YSGG laser irradiation treatment before sintering was not sufficient to increase μSBS values between Y-TZP and resin cements. Thus, the null hypothesis was accepted. To the best of our knowledge, further analysis of its clinical use is necessary.

5. Conclusions

Laser treatment before sintering presented micro-morphological alterations that suggest increased surface area. However, it was not sufficient to increase μSBS values between Y-TZP and resin cements. Tribochemo silica coating followed by primer achieved the highest immediate μSBS values. Airborne particle abrasion, followed or not by primer, increased μSBS values to Panavia F cement. Primer increased μSBS values between RelyX ARC and Y-TZP. Storage had no detrimental effect on μSBS values.

Conflict of interest

There was no conflict of interests among the authors.

Acknowledgments

This work was supported by the research support foundation of the state of São Paulo (FAPESP no. 2012/14839-0).

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