



The Effect of GaAlAs Laser, Sandblasting, and Primers on Bond Strength between Zirconia Ceramic and Direct Resin Composite after Thermocycling

O efeito do laser de GaAlAs, jateamento e primers na resistência de união entre cerâmica de zircônia e resina composta direta após termociclagem

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ABSTRACT

Objective: This study was conducted to evaluate the shear bond strength (SBS) of resin composite on zirconia ceramic after different surface treatments and thermocycling. **Material and Methods:** Two hundred and seven zirconia specimens were divided into 9 groups and treated as follows: Group C–no treatment (served as the control); Group PC–Clearfil Ceramic primer (CP); Group PZ–Z-Prime Plus primer (ZP); Group A–sandblasted with 50 μm Al₂O₃ at 0.25 MPa for 20 s at a distance of 10 mm; Group AC–sandblasted and coated with CP; Group AZ–sandblasted and coated with ZP; Group L–GaAlAs diode laser with 808 \pm 5 mm wavelength, 3 watts power, and 10 Hz frequency; Group LC–GaAlAs diode laser coated with CP; and Group LZ–GaAlAs diode laser coated with ZP. All specimens were directly bonded with a resin composite cylinder using Adper Scotchbond Multi-purpose. Specimens were stored at 37°C for 30 days and subjected to 2,500 thermocycles from 5°C and 55°C before the SBS was performed. One-way ANOVA and Tukey's HSD test ($\alpha = 0.05$) were performed. Surface topography changes were evaluated with a scanning electron microscope (SEM). **Results:** Sandblasting combined with CP or ZP (25.08 \pm 0.86 and 24.78 \pm 0.13 MPa, respectively) yielded the highest SBS and was significantly different from other methods ($p < 0.05$). SEM showed various degrees of changes depending on different surface treatments. **Conclusion:** Surface treatment by sandblasting combined with a CP or ZP significantly provide the highest SBS between zirconia and resin composite.

KEYWORDS

Bond strength; Resin composite; Surface treatment; Zirconia ceramic.

RESUMO

Objetivo: Este estudo foi conduzido para avaliar a resistência ao cisalhamento (SBS) de resina composta em cerâmica de zircônia após diferentes tratamentos de superfície e termociclagem. **Material e Métodos:** Duzentos e sete espécimes de zircônia foram divididos em 9 grupos e tratados da seguinte forma: Grupo C– sem tratamento (serviu como controle); Grupo PC–Clearfil Ceramic primer (CP); Grupo PZ–Z-Prime Plus primer (ZP); Grupo A - jateado com 50 μm de Al₂O₃ a 0,25 MPa por 20 s a uma distância de 10 mm; Grupo AC - jateado e revestido com CP; Grupo AZ - jateado e revestido com ZP; Grupo L – Laser de diodo GaAlAs com comprimento de onda de 808 \pm 5 mm, potência de 3 watts e frequência de 10 Hz; Grupo LC – Laser de diodo GaAlAs revestido com CP; e Grupo LZ – Laser de diodo GaAlAs revestido com ZP. Todos os espécimes foram diretamente colados com um cilindro de resina composta usando Adper Scotchbond Multi-purpose. As amostras foram armazenadas a 37°C por 30 dias e submetidas a 2.500 termociclos de 5°C e 55°C antes da realização do SBS. ANOVA unilateral e teste HSD de Tukey ($\alpha = 0,05$) foram realizados. As mudanças na topografia da superfície foram avaliadas com um microscópio eletrônico de varredura (MEV). **Resultados:** O jato de areia combinado com CP ou ZP (25,08 \pm 0,86 e 24,78 \pm 0,13 MPa, respectivamente) rendeu o maior SBS e foi significativamente diferente dos outros métodos ($p < 0,05$). SEM mostrou vários graus de mudanças dependendo dos diferentes tratamentos de superfície. **Conclusão:** O tratamento de superfície por jato de areia combinado com um CP ou ZP fornece significativamente o maior SBS entre a zircônia e a resina composta.

PALAVRAS-CHAVE

Força de união; Resina composta; Tratamento da superfície; Cerâmica de zircônia.

INTRODUCTION

Zirconia ceramics have been used in dentistry since the early 1990s as prefabricated posts, implants, and substructures for crown and fixed partial dentures. Zirconia ceramics have been filled with 2-3 mol% yttrium oxide, resulting in a tetragonal phase at room temperature. So, this type of zirconia ceramic is called yttrium-tetragonal zirconia polycrystal: Y-TZP. However, tetragonal exists in a metastable phase, which stores energy within the material and drives the phase transition from the tetragonal to monoclinic phase when stimulated. This phenomenon is called transformation toughening and results in an increasing of 3-5 per cent in volume of zirconia ceramic, causing compressive force around the crack and crack tip, and reduces or stops the propagation of cracks. The result of this phenomenon, which is not seen in other ceramics including the structure of packed crystalline ceramics, brings higher mechanical properties of zirconia ceramic than other ceramic types [1].

However, failure range up to 44% [2], and has been found in zirconia ceramic due to fractures in the veneering ceramic, either in the veneering layer or exposed zirconia substructure (bi-layered restoration) [3]. Several methods were usually searched by dentists for expanding the service life of existing restorations since restoration replacement can conduct for additional costs for patients. For this task, repairing of the fractured ceramic intraorally with a resin composite is the most commonly used method.

The success of the repairing task with resin composite is largely determined by the ability to adhere to the surface of the veneering layer or zirconia ceramic substructure. Hydrofluoric (HF) acid etching is a well accepted pre-treatment for bonding veneering ceramic surface layers [4] but not for zirconia ceramic substructures. Surface treatment of zirconia ceramic includes sandblasting with 50 μ m aluminium oxide [5], priming with MDP primer [6], a combination of sandblasting and MDP primer, silica coating [7], and CO₂, Nd:YAG or Er:YAG laser [8-11]. While previous studies have shown that diode (GaAlAs) lasers alone do not roughen the zirconia ceramic surface [12,13]. The question arose whether the

increased wattage or time duration of a diode laser combined with a primer improved the bond strength or not.

However, most research has been developed and conducted to treat the zirconia surface for indirect bonding with resin cement. Few studies for intraoral repair of zirconia ceramics with direct resin composite and thermocycles have been found. Therefore, the aims of this study were to investigate the effect of different surface treatments of zirconia ceramic for bonding with direct resin composite after thermocycling to achieve the optimal treatment and to examine the changes of zirconia surfaces after the treatments.

MATERIAL AND METHODS

Specimen Preparation

The materials used in this study and their compositions are shown in Table I. Two hundred and seven specimens were prepared according to the manufacturer's instructions. Briefly, zirconia ceramic blocks (IPS e.max ZirCAD MO block, Ivoclar Vivadent AG, Schaan, Liechtenstein) were cut with a precision saw (ISOMET 4000, Buehler, Lake Bluff, IL, USA) to specimens with 12 mm width, 12 mm length and 4 mm thickness. Crack specimens were detected under 10x magnification using a stereomicroscope (model SMZ 1500m, Nikon Instech, Kanagawa, Japan) and were excluded from the experiment. Specimens were then processed by heating to 1,500°C for 8 h (Progamat S1 high-temperature furnace, Ivoclar Vivadent AG, Schaan, Liechtenstein). Subsequently, sintered specimens with a final dimension of 9.6 mm width, 9.6 mm length and 3.2 mm thickness (18.5% to 19.6% shrinkage) were embedded in autopolymerizing clear acrylic resin (Takilon, Rodont srl, Milan, Italy), 30 mm in height and 30 mm in diameter. The specimens were then polished (model Phoenix 4000, Buehler GmbH, Düsseldorf, Germany) under running water using 600- and 1,200-grit silicon carbide paper (3M ESPE, St. Paul, MN, USA).

The specimens were randomly divided into 9 groups of 23 specimens each. The surface treatment performed on each group was as follows:

Group C was the control group (no treatment).

Group PC was coated with primer (Clearfil Ceramic Primer, Kuraray, Tokyo, Japan) for 20 s and air-dried for 20 s.

Group PZ was coated with primer (Z-Prime Plus, Bisco, Schaumburg, IL, USA) for 20 s and air-dried for 20 s.

Group S was sandblasted (Sandblaster, Dental Farm, Torino, Italy) with 50 μm Al_2O_3 at 0.25 MPa for 20 s at a distance of 10 mm and dried.

Group SC was sandblasted (Sandblaster, Dental Farm, Torino, Italy) with 50 μm Al_2O_3 at 0.25 MPa for 20 s at a distance of 10 mm and dried. Primer (Clearfil Ceramic Primer, Kuraray, Tokyo, Japan) was then coated for 20 s and air-dried for 20 s.

Group SZ was sandblasted (Sandblaster, Dental Farm, Torino, Italy) with 50 μm Al_2O_3 at 0.25 MPa for 20 s at a distance of 10 mm and dried. Primer (Z-Prime Plus, Bisco, Schaumburg, IL, USA) was then coated for 20 s and air-dried for 20 s.

Group L was treated with a diode laser (GaAlAs Laser Diode, model SL3, Philips Oral Healthcare-LA, CA, USA) with 808 \pm 5 nm wavelength, 3 watts power, and 10 Hz frequency for 2 min (modified from Kriebel [14]).

Group LC was treated with a diode laser (GaAlAs Laser Diode, model SL3, Philips Oral Healthcare-LA, CA, USA) with 808 \pm 5 nm wavelength, 3 watts power, and 10 Hz frequency for 2 min (modified from Kriebel [14]). Primer (Clearfil Ceramic Primer, Kuraray, Tokyo, Japan) was then applied for 20 s and air-dried for 20 s.

Group LZ was treated with a diode laser (GaAlAs Laser Diode, model SL3, Philips Oral Healthcare-LA, CA, USA) with 808 \pm 5 nm wavelength, 3 watts power, and 10 Hz frequency for 2 min (modified from Kriebel [14]). Primer (Z-Prime Plus, Bisco, Schaumburg, IL, USA) was then applied for 20 s and air-dried for 20 s.

Bonding Procedures

For direct bonding with resin composite,

the zirconia specimens were applied with a bonding agent (Adper Scotchbond Multi-Purpose Plus Adhesive, 3M ESPE, St.Paul, MN, USA) and light-polymerized with visible light (600 mW/cm² at a wavelength of 400 to 500 nm; XL3000, 3M ESPE, St.Paul, MN, USA) for 20 s.

A thin plastic tube (AP Extrusion, Salem, NH, USA) with 4 mm inner diameter and 2 mm thickness was placed and held at the center of each zirconia specimen with plastic pliers. The tube was filled in with resin composite (Filtek Z250XT, 3M ESPE, St.Paul, MN, USA) and light-polymerized for 40 s, and again for 40 s after the plastic tube had been cut with a blade and removed. All specimens were stored in 100% humidity at 37°C for 30 days with 2,500 thermocycles from 5°C and 55°C before shear bond strength testing was performed.

Shear Bond Strength Testing

The shear bond strength of resin composite on the zirconia specimens was tested by using a single-bladed Instron Machine (model 5583, Instron Corp., Norwood, MA, USA) at a crosshead speed of 0.2 mm/min [15]. The load at failure was recorded and converted to shear bond strength expressed in MegaPascals (MPa) as in the following formula:

$$\text{Shear bond strength} = F/\pi r^2$$

where F is a load force at fracture in Newtons and r is the radius of the resin composite cylinder in meters. The surfaces of the specimens were subsequently examined under a stereoscope (model SMZ 1500m, Nikon Instech, Kanagawa, Japan) at \times 50 magnification in order to determine the mode of failure. Mode of failure was recorded by one observer as either adhesive (between zirconia or resin composite and bonding agent), cohesive (in the zirconia, resin composite or bonding agent) or a combination of adhesive and cohesive fractures [13].

Surface Topography Analysis

Twenty seven ceramic specimens, three from each group, were selected to investigate the treated surface topography. The specimens were rinsed with distilled water for 20 s, dried and mounted onto aluminium stubs (13 mm diameter

and 10 mm height). Subsequently, specimens were sputter-coated with a gold-palladium alloy (SPI-Module sputter, SPI Supplies, West Chester, PA, USA). Observations were made under a scanning electron microscope (SEM) (model JSM-5800LV, JEOL, Tokyo, Japan) at x1000 magnification.

Statistical Analysis

Data were statistically analyzed. A one-way analysis of variance (ANOVA) was used to find differences between groups. Tukey's Honestly Significant Difference (HSD) Test was used for post hoc comparisons ($\alpha = 0.05$).

RESULTS

Results of the one-way ANOVA revealed that the shear bond strength differed significantly between groups ($p < 0.05$). The mean values of the shear bond strength of resin composite on the zirconia at the fracture, as well as the results of multiple comparisons using Tukey's HSD tests, are presented in Table II. Sandblasting combined with Clearfil Ceramic primer or Z-Prime Plus primer (25.08 ± 0.86 and 24.78 ± 0.13 MPa, respectively) yielded the highest SBS and were significantly different from other methods ($p < 0.05$).

SEM images of the differently treated zirconia surfaces are shown in Figure 1. Surfaces of the specimens in the control and laser groups showed scratches and grooves (Figures 1A and 1G). Specimens treated with sandblasting produced noticeable changes on the zirconia ceramic surface (Figure 1D), in which irregular porous surfaces with various micro-undercuts were observed over the entire surface. Specimens coated with primers presented thin layer covered.

The mode of failure of all specimen groups, evaluated under a stereoscope, was shown in Table III. Adhesive failure was noticeable in all groups in varying numbers. Combination failure was found in C, SC, and SZ groups.

Table I - Materials used in this present study

Product	Composition	Manufacturer
IPS e.max ZirCAD MO	ZrO ₂ (88-95.5%), Y ₂ O ₃ (4.5-6%), HfO ₂ (5%), Al ₂ O ₃ (1%),	Ivoclar Vivadent AG, Schaan, Liechtenstein
Clearfil Ceramic Primer	3-Methacryloxypropyl trimethoxy silane, MDP, Ethanol	Kuraray, Tokyo, Japan
Z-Prime Plus	Organophosphate monomer, carboxylic acid monomer, HEMA, ethanol	Bisco, Schaumburg, IL, USA
Scotchbond Multi Purpose Plus	Bis-GMA, HEMA, Tertiary Amines	3M ESPE, St. Paul, MN, USA
Flittek Z350XT	Bis-EMA, Bis-GMA, TEGDMA, UDMA, zirconia, silica	3M ESPE, St. Paul, MN, USA

ZrO₂: Zirconium dioxide, Y₂O₃: Yttrium oxide, HfO₂: Hafnium oxide, 10-MDP: 10-Methacryloyloxydecyl dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, Bis-GMA: Bisphenol A diglycidyl methacrylate, Bis-EMA: ethoxylated bisphenol A glycol dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate.

Table II - Mean shear bond strength \pm SD of zirconia ceramics to resin composites in MPa after ANOVA One-Way and Tukey test (5% of significance)

Group	Surface treatment	Mean shear bond strength \pm SD
C	Control (no treatment)	6.02 \pm 0.98 ^a
PC	Clearfil Ceramic Primer	16.1 \pm 0.82 ^c
PZ	Z-Prime Plus	14.65 \pm 0.73 ^c
S	Sandblasting with 50 μ m Al ₂ O ₃	15.24 \pm 0.16 ^c
SC	Sandblasting with 50 μ m Al ₂ O ₃ + Clearfil Ceramic Primer	25.08 \pm 0.86 ^d
SZ	Sandblasting with 50 μ m Al ₂ O ₃ + Z-Prime Plus	24.78 \pm 0.13 ^d
L	GaAIAs Diode Laser	8.02 \pm 0.07 ^b
LC	GaAIAs Diode Laser + Clearfil Ceramic Primer	16.07 \pm 0.72 ^c
LZ	GaAIAs Diode Laser + Z-Prime Plus	14.66 \pm 0.85 ^c

^{a-d} Group identified with different letters were significantly different ($p < 0.05$).

Table III - Mode of failure after shear bond strength testing

Group and surface treatment	Type of failure			Total
	Adhesive	Cohesive	Combination	
C: Control (no treatment)	20	0	0	20
PC: Clearfil Ceramic Primer	18	0	2	20
PZ: Z-Prime Plus	20	0	0	20
S: Sandblasting with 50 μ m Al ₂ O ₃	20	0	0	20
SC: Sandblasting with 50 μ m Al ₂ O ₃ + Clearfil Ceramic Primer	2	0	18	20
SZ: Sandblasting with 50 μ m Al ₂ O ₃ + Z-Prime Plus	2	0	18	20
L: GaAIAs Diode Laser	20	0	0	20
LC: GaAIAs Diode Laser + Clearfil Ceramic Primer	20	0	0	20
LZ: GaAIAs Diode Laser + Z-Prime Plus	20	0	0	20

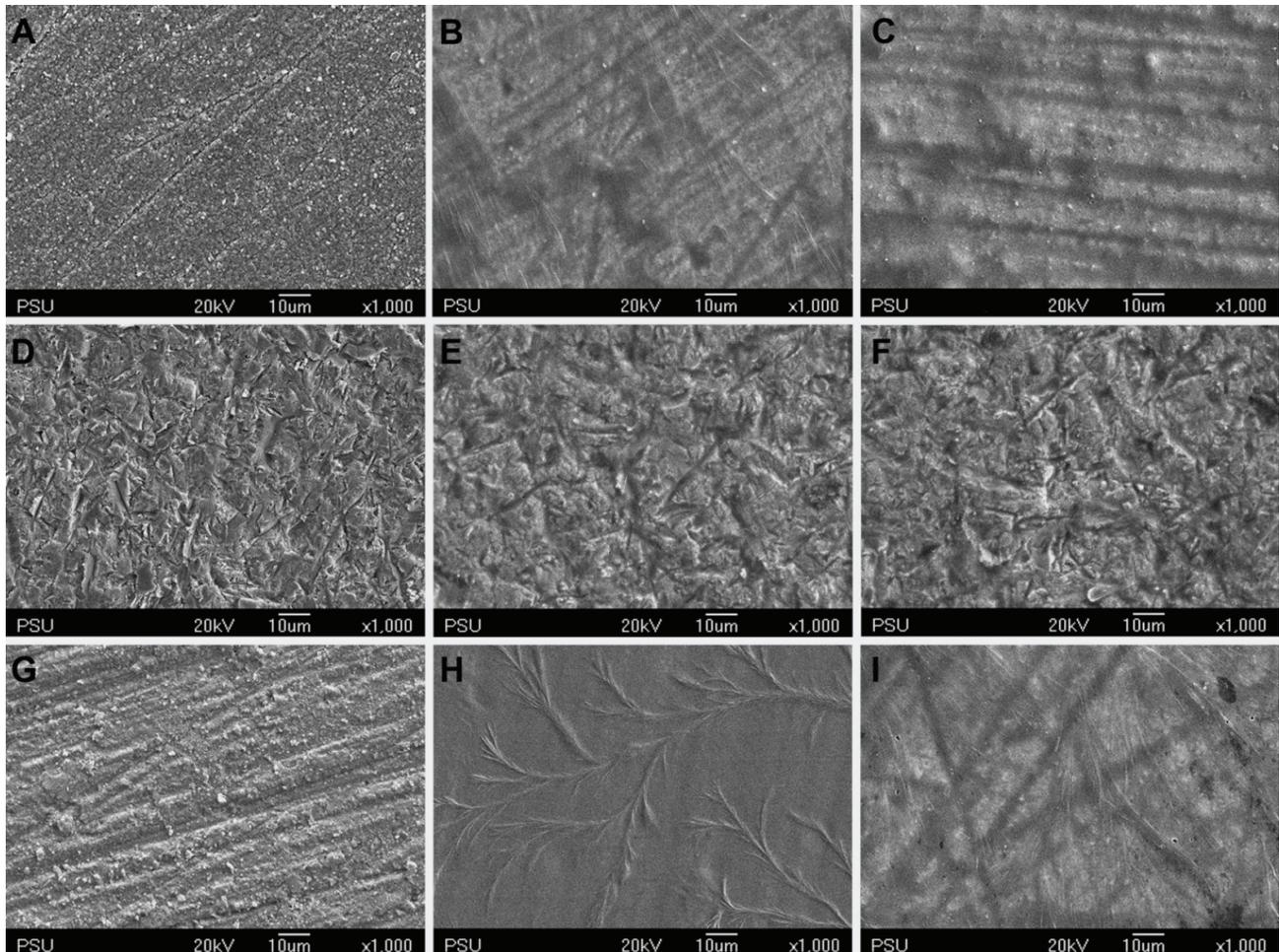


Figure 1 - SEM results of zirconia ceramic specimens (original magnification $\times 1000$). (A) Control group. (B) Clearfil Ceramic primer group. (C) Z-Prime Plus primer group. (D) Sandblasted with $50\ \mu\text{m}\ \text{Al}_2\text{O}_3$. (E) Sandblasted with $50\ \mu\text{m}\ \text{Al}_2\text{O}_3$ and coated with Clearfil Ceramic primer group. (F) Sandblasted with $50\ \mu\text{m}\ \text{Al}_2\text{O}_3$ and coated with Z-Prime Plus primer group. (G) GaAlAs diode laser group. (H) GaAlAs diode laser and coated with Clearfil Ceramic primer group. (I) GaAlAs diode laser and coated with Z-Prime Plus primer group.

DISCUSSION

The aging test or thermocycling by water immersion and temperature change is an important step in studying the effect of zirconia ceramic surface treatments on their durability on bonding to resin composites, by simulating oral conditions that include both saliva moisture and temperature changes from daily consumption habits [16,17]. Previous studies have presented that long term adhesion between zirconia ceramic and resin composite decreased after thermocycling [18,19]. A study by Gale and Darvell [20] showed that 10,000 thermocycles could be equivalent to 1 year

on oral environment. Therefore, the present study used 2,500 thermocycles which could be equivalent to a 3-month period in an oral condition. The control group showed adhesive failure after a temperature change at 5°C and 55°C for 2,500 cycles. Due to the lack of both microscopic and chemical attachments, there was water penetration and deterioration the adhesion [21].

Even though the challenges of shear bond strength test included higher bond strengths increased due to cohesive failure in the substrates and non-uniform stress distributions along tested interfaces [22], this study intended to use

shear bond strength test rather than micro-shear bond strength test due to numerous advantages such as ease of specimen preparation, simple test protocol, simplicity, and reproducible. While micro-shear bond strength test has been introduced to evaluate the bond strength of small area of substrates (1 mm² or less) to adhesives [23,24]. Some studies have reported its disadvantages, namely a technically sensitive procedure, more time consuming, and worse representing shear bond strength than shear bond strength test [22,25].

These present results suggest the hypothesis that shear bond strength is determined not only by surface roughness but also by other factors. A factor that could probably affect the bond strength is the treatment with primer compounds. Primers are a substance which have functional groups such as 4-META (4-methacryloxyethyl trimellitate anhydride), 10-MDP (10-Methacryloyloxydecyl dihydrogen phosphate), MEPS (methacryloyloxyalkyl thiophosphate derivative), and VBATDT (6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol) that can chemically attach to the oxide layer of zirconia ceramic surfaces [5,26]. The results of this study showed that Clearfil Ceramic primer products with the MDP functional group and Z-Prime Plus primer, which has an organophosphate functional group mixed with carboxylic acid, can increase the shear bond strength between zirconia ceramic and resin composite when compared to the control group. The MDP functional group has three components, namely the methacrylate group that can react with the polymerization of the resin monomer, a group of dihydrogen phosphate that promotes chemical attachment to the zirconium oxide layer on the surface, and the last decyl group is a hydrophobic element that is able to form a protective barrier for water penetration into the boundary between the dihydrogen phosphate group and the zirconium oxide layer [27]. All these three components of MDP promoted higher shear bond strength after thermocycles in the Clearfil Ceramic primer group than Z-Prime Plus, which has an organophosphate

functional group mixed with carboxylic acid, but it was not a significant difference. These results corresponded with the mode of failure with a stereomicroscope, where the Clearfil Ceramic primer group (Group PC) showed 80% adhesive failure and 20% combination failure, while the Z-Prime Plus group (Group PZ) presented 100% adhesive failure, which showed a lower shear bond strength.

Surface treatment by sandblasting with aluminum oxide powder is another method that has been used for a long time in metal-ceramic restorations. Although, initially, there is a concern about the effect of sandblasting on the phase change of zirconia ceramic from the tetragonal to monoclinic phase and that it reduces the strength. However, many studies have supported the improvement of mechanical properties by sandblasting [18,28]. There is also no clinical evidence to confirm the adverse effects of this method [29]. The present study found that surface treatment by sandblasting increased the shear bond strength between zirconia ceramic and resin composite (Group S), which were higher than specimens treated by Z-Prime Plus (Group PZ) but lower than those treated by the Clearfil Ceramic primer group (Group PC), corresponding with other studies [5,16]. When evaluating the surface topography of zirconia ceramics after sandblasting by SEM, it was found that the sandblasted surfaces caused rough surface and also increased surface area [30,31]. This is one factor that promotes the adhesion between both materials, but the average shear bond strength was lower than Clearfil Ceramic primer groups. This means that micromechanical retention alone was not enough for bonding between zirconia ceramic and resin composite after 2,500 thermocycles because there was no chemical attachment, which allows water penetration and disruption on the adhesion ability, according to Christoforides et al. [17]

Most of the previous studies have supported surface treatment by a combination of sandblasting with aluminum oxide and primers where the bond strength does not

decrease by thermocycles [19,21,32]. The present study showed the same results where combined surface treatment by sandblasting and primers provided the highest average shear bond strength. The mode of failures also showed the most combination failure. Surface treatment of zirconia ceramic by sandblasting with aluminum oxide together with primer is an effective surface treatment method to promote adhesion, both in micromechanical retention from alteration of the surface characteristics of zirconia ceramics and chemical retention from functional groups in the adhesive layer between the primer and zirconium dioxide layer [17,33]. Also sandblasting rough zirconia ceramic surfaces, increased the contact area and reduced contamination from saliva and oral bacteria [5,34]. In addition, sandblasting can increase surface energy and reduce the contact angle of the zirconia ceramic surface, which promotes wettability of the primer adhesive layer and bonding [35]. These increase the efficiency of the functional group from the primer layer to bond to the zirconium oxide layer in a zirconia ceramic both from MDP in Clearfil Ceramic primer or the carboxylic group in Z-Prime Plus primer.

For surface treatment using a diode laser without a primer, the results showed that shear bond strength slightly increased (Group L) compared to control group. However, when the primer was applied, shear bonding strength was similar to groups with solely primer treatment (Groups PC and PZ). Although previous studies have shown that diode lasers alone do not roughen the zirconia ceramic surface [12,13], this study was still conducted expecting that increased wattage or time of a diode laser combined with a primer would increase the shear bond strength. Yet from the results of this study, shear bond strength still increased in only small quantities. This may require further study by combinations with other surface treatments.

The limitation of this study was that only one zirconia ceramic brand was investigated, so it might not be able to represent all current zirconia products. In addition, even though

this study used thermocycle simulation, the oral cavity presents a distinctive environment. For instance, the presence of water, pH and temperature change in the oral cavity might also significantly influence the properties of the materials and even the thermocycles simulation. Moreover, the present study evaluated the *in vitro* effect. Therefore, further studies are required to elaborate the effect of surface treatments *in vivo*.

CONCLUSION

Under the limitations of this study, it was possible to conclude that surface treatment of zirconia ceramic play an important factor in direct bonding to resin composite. Surface treatment by sandblasting with aluminum oxide combined with ceramic primers (Clearfil Ceramic primer, with MDP functional group or Z-Prime Plus, with organophosphate functional group mixed with carboxylic acid) significantly provided the highest shear bond strength between zirconia ceramic and resin composite.

Conflicts of interest

The authors have no conflicts of interest relevant to this article.

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Regulatory Statement

None.

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