

INFLUENCE OF SURFACE TREATMENT ON VENEERING  
PORCELAIN SHEAR BOND STRENGTH TO ZIRCONIA  
AFTER CYCLIC LOADING

by

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## INTRODUCTION

For the past 40 years, the metal-ceramic restoration has been used as a reliable treatment option for prosthodontic restorations. However, patients and dentists have demonstrated an increased interest in and demand for esthetic and biocompatible restorative materials. Therefore, all-ceramic restorations have gained popularity. Since the introduction of computer- aided design/computer aided machining/milling (CAD/CAM) technology into the dental market, the use of yttria-partially stabilized tetragonal zirconia polycrystal (Y-TZP) has become more popular for dental applications. Y-TZP has excellent mechanical properties. An *in-vitro study* demonstrated a flexural strength of 900 MPa to 1200 MPa and a fracture toughness of 9 MPa to 10 MPa m<sup>1/2</sup>.<sup>1</sup> Y-TZP can be used as a coping material for all-ceramic restorations and fixed partial dentures and is further layered with veneering porcelain (VP) to optimize the esthetic outcome.

Long-term clinical results for Y-TZP all-ceramic restorations are not available at present. Several studies have evaluated Y-TZP all-ceramic restorations in short and medium terms. As a result, a high success rate for all-ceramic restorations has been reported when Y-TZP is used as a coping material.<sup>2-7</sup> However, chipping or cracking of the veneering porcelain was reported as the most common complication. Specifically, a relatively high rate (13.0 percent and 15.2 percent) of chipping or cracking failure has been observed in posterior Y-TZP all-ceramic restorations after 3-year and 5-year observation periods.<sup>3,4,8</sup> On the other hand, the rate of VP fracture on metal ceramic restorations has been reported as 2.5 percent after 5 years.<sup>9</sup> Therefore, the mechanical

integrity and bonding strength between the VP and Y-TZP coping is thought to be a key factor in making Y-TZP all-ceramic restorations as reliable as metal-ceramic restorations.<sup>10,11</sup>

Several studies have investigated the cause of increased chipping or cracking of VP on Y-TZP copings compared with metal copings. The cause of chipping or cracking of VP was reported to be multifactorial.<sup>1,10,12</sup>

A significant difference between the Y-TZP coping and metal coping is the adhesion mechanism of zirconia and metal coping materials to VP. While mechanical and chemical bonds resulting from suitable metal oxidation and interdiffusion of ions play the important role in the metal coping and VP interface, the bonding mechanism of VP to Y-TZP coping is not well understood.<sup>1</sup>

The bond strength can be compromised by residual stresses resulting from a mismatch of coefficient of thermal expansion (CTE) between the VP and coping.<sup>13</sup> VP with a slightly lower CTE than that of the metal coping is used in metal ceramic restorations because the slightly lower CTE generates compressive stresses in the veneering porcelain, a material that is weaker against tensile stress.<sup>13</sup> Likewise, this concept has been discussed in numerous studies concerning the VP and Y-TZP coping material.<sup>1,10,14,15</sup> Moreover, to generate acceptable residual stresses within the VP, dental manufacturers have made efforts to develop low-fusing veneering porcelain with similar CTE to the Y-TZP coping material. Saito et al.<sup>14</sup> demonstrated in an *in-vitro study* that the bond of VP to a Y-TZP coping can be similar to that of a metal ceramic system with matching CTEs. Therefore, similar clinical behavior can be expected for Y-TZP all-

ceramic restorations when VP with a slightly lower coefficient of thermal expansion, compared with that of Y-TZP is used.

The thermal compatibility is another important factor.<sup>16,17</sup> Whereas dental metal alloys used in metal ceramic restorations have a high thermal conductivity (in the range of  $300 \text{ W m}^{-1} \text{ K}^{-1}$  for noble alloys), thermal conductivity of the Y-TZP coping materials exhibit  $2\text{-}2.2 \text{ W m}^{-1} \text{ K}^{-1}$ . Feldspathic VP is in the same range as Y-TZP with a thermal conductivity of  $2.39 \text{ W m}^{-1} \text{ K}^{-1}$ .<sup>1</sup> The difference retards the porcelain cooling rate at the interface between VP and Y-TZP coping materials, changing the impact of CTE and resulting residual thermal stresses. Guazzato et al.<sup>16</sup> concluded that crack incidence increased with increased porcelain veneer thickness and faster cooling rates in nominally compatible porcelain/zirconia systems in the geometrically configured specimens tested. Therefore, the design of the framework and the thickness of the VP should be considered as important factors of success in Y-TZP all-ceramic restorations during the fabrication of bilayered restorations.

Pure zirconia has three different crystal structures depending on temperature. From room temperature to  $1170^\circ\text{C}$ , the crystal structure is monoclinic; the structure is tetragonal between  $1170^\circ\text{C}$  and  $2370^\circ\text{C}$ , and cubic above  $2370^\circ\text{C}$  up to the melting point. Yttrium oxide is a stabilizing oxide added to pure zirconia to stabilize it at room temperature and to generate a multiphase material known as partially stabilized zirconia (Y-TZP). The high initial strength and fracture toughness of zirconium oxide results from a physical property of Y-TZP known as transformation toughening.<sup>3</sup>

According to Aboushelib et al.<sup>18</sup> even though the pressable veneer porcelain has excellent wetting and bonding strength with the coping material when manifested by a

continuous crystalline phase, Y-TZP all-ceramic restorations demonstrated a higher percentage of interfacial failure, which showed voids and imperfect connection of crystallized phases. The authors suggested that the presence of a monoclinic phase of zirconium oxide at the core-veneer interface may be the cause of micro-spaces found at the interface.

In addition, Guazzato et al.<sup>19</sup> described the tetragonal-monoclinic transformation to be accompanied by a generation of localized stresses, which may nucleate microcracks in the glass phase of the veneer. Y-TZP all-ceramic restorations are subjected to mastication forces in the oral environment. The mastication forces are not static and high, but low and repetitive. The fatigue behavior may act on residual stresses leading to not only crack propagation but also generation of tetragonal-monoclinic transformation in Y-TZP. Moreover, stress-generating surface treatments, such as grinding or sandblasting, and firing the VP over the Y-TZP coping, are able to trigger the tetragonal to monoclinic transformation.<sup>19</sup> Some studies have mentioned that little monoclinic phase was detected in Y-TZP with heat treatment at approximately 900°C after airborne-particle abrasion.<sup>20,21</sup> De Kler concluded<sup>21</sup> that sintered tetragonal structure was converted to monoclinic up to a depth of 27 µm after airborne abrasion, and reversed back to tetragonal after porcelain veneering. In other words, heat treatment and the firing process caused reverse transformation and release of the compressive stresses. In fact, Doi et al.<sup>20</sup> concluded that there is significantly higher debonding/crack-initiation strength with heat treatment after airborne-particle abrasion than without. However, Fischer et al.<sup>11</sup> concluded that heat treatment significantly decreased the shear strength of both polished and sandblasted surfaces.

Several studies have reported that the bond strength between Y-TZP coping and veneering porcelain is significantly affected by some surface treatments such as air-borne particle abrasion, heat treatment, and use of liner porcelain.<sup>11,18,22-24</sup> However, the influence of different surface treatments on the bond strength of veneering porcelain to zirconia coping is not clear.

Another factor to consider is fatigue strength because a fatigue process can be started in localized surface areas, leading to monoclinic transformation and resulting in microcracks and surface expansion. Moreover, strength values obtained from a measurement of the failure load may be quite misleading if they are used to design a structure that is subjected to repeated or cyclic loading.<sup>13</sup> The chipping or cracking of VP on Y-TZP copings may develop progressively over many stress cycles after initiation of a crack from a flaw or surface condition on the Y-TZP coping. The overall objective of this study was to investigate the influence of different surface treatments on veneering porcelain shear bond strength to Y-TZP with and without cyclic loading.

#### PURPOSE OF THIS STUDY

The goals of this study were: 1) To investigate the influence of surface treatments on VP bond strength to Y-TZP, and 2) To investigate the influence of cyclic loading on the shear bond strength between Y-TZP and VP.

#### HYPOTHESES

The null hypotheses for this study were: 1) Shear bond strength between Y-TZP and veneering porcelain would not be influenced by heat-treatment, airborne-particle

abrasion, and heat-treatment after airborne-particle abrasion, and 2) Cyclic loading would not affect the shear bond strength between Y-TZP and VP.

The alternative hypotheses for this study were: 1) Shear bond strength between Y-TZP and VP would be increased by surface treatment using heat, airborne-particle abrasion, and heat after airborne-particle abrasion, and 2) Cyclic loading would decrease the shear bond strength between Y-TZP and VP.



REVIEW OF LITERATURE

## YTTRIA-PARTIALLY STABILIZED TETRAGONAL ZIRCONIA POLYCRYSTAL (Y-TZP)

Pure zirconia is a polymorphic material and has three different crystal structures depending on temperature. From room temperature to 1170°C, the crystal structure is monoclinic; the structure is tetragonal between 1170 °C and 2370 °C, and then cubic above 2370 °C up to the melting point. Yttrium oxide is a stabilizing oxide added to pure zirconia to stabilize it at room temperature to control the tetragonal to monoclinic phase transformation, which is accompanied by a 3-percent to 4-percent volumetric expansion leading to compressive stresses.<sup>25</sup> Tensile stresses at a crack tip will transform the tetragonal phase to the monoclinic phase, and the volumetric expansion creates compressive stresses at the crack tip that counteract the external tensile stresses. This phenomenon retards crack propagation and is known as transformation toughening.<sup>26</sup>

Initially, Y-TZP was developed for orthopedic total hip replacement because of the excellent mechanical and biocompatibility properties.<sup>27</sup> In the early 1990s, Y-TZP was introduced to dentistry for things such as all-ceramic restoration copings and framework materials for implant superstructures.<sup>28,29</sup> According to both *in-vitro* and *in-vivo* studies,<sup>30-32</sup> the excellent mechanical properties allow Y-TZP copings of fixed partial dentures to require a relatively small connector area ranging between 7 mm<sup>2</sup> and 16 mm<sup>2</sup>, which is smaller than other all-ceramic core materials, including glass-infiltrated alumina with 35-percent zirconia, and lithium disilicate,. Moreover, it is not necessary to use adhesive cementation, and conventional cements such as glass ionomer cements, resin

modified glass ionomer cements, and composite resin luting cements are available options.<sup>33</sup>

Y-TZP has a radiopacity comparable to metal copings, which enhances radiographic evaluation of marginal integrity, excess cement removal, and recurrent decay.<sup>33</sup> In addition, whereas a small percentage of the patient population is hypersensitive to dental alloy containing both noble and base metals, such as palladium and nickel, no study has reported local or systemic adverse effects from Y-TZP material.<sup>34-36</sup> Therefore, Y-TZP is thought to be attractive for restorative dentistry because of its chemical stability and biocompatibility.

On the other hand, a disadvantage of Y-TZP compared with any other all-ceramic material is its color, white or opaque, which may limit its indications from an esthetic stand point. In clinical situations, zirconia copings sometimes require a liner porcelain to mask the underlying coping color to improve the esthetics. The liner porcelain reduces veneering porcelain thickness, and some studies have reported that liner porcelain may decrease the bond strength between Y-TZP coping and VP.<sup>6, 22</sup> Several zirconia systems, such as Cercon (Dentsply Ceramco, York, PA) and Lava (3M ESPE, St. Paul, MN), have developed relative translucence and different shades to allow the restorations to be more natural.<sup>26</sup> However, Aboushelib et al.<sup>24</sup> concluded that mean VP SBS values on colored zirconia is lower than on white zirconia. They stated that the addition of coloring pigments to zirconia cores resulted in structural changes that require different surface treatment before veneering.

## THE HISTORY OF DENTAL CAD/CAM SYSTEMS

According to Miyazaki et al.,<sup>37</sup> the dental CAD/CAM system has progressed through four generations of development. In the early 1970s, Duret et al.<sup>38</sup> introduced the first generation of the dental CAD/CAM system. The intraoral abutment was scanned by an intraoral digitizer to create an optical impression. The digital data were collected as 3-D graphic information. The final crown was designed virtually on the monitor and fabricated by milling a block, using a numerically controlled machine. However, the system was not widely used because the digitizing, the computer power, and the material were not accurate enough to be applied in dentistry. Next, Mormann et al.<sup>39</sup> developed the CEREC system to produce a ceramic inlay restoration using computer-assisted technology. A compact intraoral camera was used at the chairside after the inlay preparation. Inlay preparation imaging is technically less difficult compared with crown abutments. Therefore, the CEREC system has been successful and has led to the technical term of CAD/CAM gaining popularity in dentistry. Some studies have reported satisfactory long term results of restorations fabricated with the CEREC system.<sup>40,41</sup>

In the second generation of dental CAD/CAM systems, manufacturers made much effort to fabricate a crown with an anatomical occlusal surface. To improve the accuracy, they developed different digitizer systems such as a contact probe,<sup>42</sup> laser beam with a Position Sensitive Detector (PSD), and a laser with a CCD camera.<sup>43</sup> Additionally, the conventional stone model was scanned in the second generation of the dental CAD/CAM system by newly developed digitizers instead of a direct intraoral scanner. Due to the development, both metallic and ceramic restorations could be fabricated in the second generation.<sup>44</sup>

The Procera system (Nobel Biocare AB, Göteborg, and Sandvik Hard Materials AB, Stockholm, Sweden) is a representative third-generation system. This was the first application of CAD/CAM in a specialized procedure as part of a total processing system, which networked with satellite digitizers worldwide for the fabrication of all-ceramic frameworks using industrial dense-sintered polycrystalline alumina.<sup>45</sup> In this system, the working model or wax up on the model was digitized in the dental laboratory. The information was sent to a processing center, and the coping was designed and milled on the computer. Subsequently, the milled coping was sent back to the dental laboratory and the final restoration was completed. Such networked production systems were innovative in dentistry.<sup>46</sup>

Thus, prosthetic restorations became a popular application for Y-TZP ceramic due to the development of the dental CAD/CAM system. Now, new technology is enhancing the use of Y-TZP in the clinic and the ability to obtain accurate intraoral digitizers. In fact, some products are now available on the market as the fourth generation of dental CAD/CAM system with improved technology.<sup>37</sup> However, information supporting them is still limited. Further investigation is required regarding the accuracy and long-term clinical performance.

#### DESIGN AND MANUFACTURE OF Y-TZP COPINGS

Y-TZP copings are designed by either a conventional wax-up technique or computer-assisted designing software and can be fabricated with two different materials, partially presintered or fully sintered Y-TZP.<sup>30,33,47</sup> Partially presintered Y-TZP is easy to mill or to shape, but must be sintered after milling to achieve the final strength. The milled size should be increased to compensate for the prospective shrinkage (20 percent

to 25 percent) that occurs during the final sintering.<sup>30-32</sup> The milling process is faster, and the wear and tear on the milling machine is less than that of fully sintered Y-TZP.<sup>30-32</sup> In contrast, fully sintered Y-TZP is milled to the final dimensions because no final sintering is required.<sup>47</sup> This fully sintered Y-TZP has a lower volume fraction of pores, greater strength, and improved resistance to hydrothermal aging.<sup>48</sup> For example, Lava (3M ESPE, St. Paul, MN) uses partially presintered Y-TZP. A die is scanned by a contact-free optical process, and the CAD software designs an enlarged coping.<sup>49</sup> Cercon (Dentsply Ceramco, York, PA) requires scanning of a conventional wax-up to design the Y-TZP coping made of presintered Y-TZP.<sup>33</sup> On the other hand, DCS Precident (DCS Dental AG, Allschwil, Switzerland) uses fully sintered DC Zircon ceramic.<sup>33,50</sup> Raigorodski<sup>33</sup> reported that proponents of partial sintering believe microcracks could be introduced to the copings during the milling procedure for a fully sintered blank; on the other hand, advocates of full sintering assert the marginal fits is superior, because no shrinkage is involved in the milling process.

According to Kohorst et al.<sup>48</sup> when comparing *in vitro* the load-bearing capacity of posterior four-unit fixed partial dentures (FPDs) made with two different Y-TZP ceramics, presintered or fully sintered, FPDs made from fully sintered Y-TZP had a significantly higher fracture resistance compared with FPDs made from presintered Y-TZP. However, Kohorst concluded that both types of Y-TZP could be suitable for posterior four-unit all-ceramic FPDs, because FPDs made from both materials were capable of withstanding occlusal forces as reported in the literature.

## SURVIVAL RATE AND COMPLICATION OF Y-TZP FIXED PARTIAL DENTURES (FPDS)

Long-term clinical results for Y-TZP all-ceramic restorations are not available at the present time. Such restorations have been evaluated in short-and medium-term studies. The typical survival rates for Y-TZP FPDs range from 73.9 percent to 100 percent after 3 years to 5 years in service.<sup>2-7</sup> As a result, a high success rate for all-ceramic restorations has been reported when Y-TZP is used as a coping material. On the other hand, numerous studies have reported the survival rate and complications of metal ceramic FPDs for long-term clinical results.<sup>51-55</sup> Two meta-analysis studies regarding metal ceramic FPDs have been reported.<sup>9,56</sup> According to Scurria et al.<sup>9</sup> the survival rates of metal ceramic FPDs are 92 percent and 75 percent at 10 years and 15 years, when failure was defined as FPD removal. When a broader definition of failure was used to include FPDs that are removed or that fail, the survival rates are 87 percent and 69 percent at 10 years and 15 years, respectively.

However, comparing these data is challenging because of discrepancies in the classification or definition of failure and the variability of the materials and systems present.<sup>26</sup> To compare Y-TZP FPDs and metal ceramic FPDs, further investigation is required with a more comprehensive definition of failure or a critical assessment of Y-TZP restorations.

The most common minor complication among Y-TZP FPDs that did not require remaking of the restoration was chipping or cracking limited to VP.<sup>3,4,7,8</sup> These complications obviously differ from metal-ceramic FPDs that fail primarily due to tooth fracture and caries. A randomized controlled clinical trial of short-term performance of Y-TZP FPDs and metal ceramic FPDs in posterior areas is available.<sup>7</sup> VP fracture,

including chipping, was found in 33.4 percent of the Y-TZP restorations compared with 19.4 percent of the metal ceramic FPDs. Additionally, Raigrodski<sup>3</sup> described a prospective clinical study of posterior three-unit Y-TZP FPDs over three years. This author found the survival rate of Y-TZP FPDs was 100 percent, but that five minor chippings of VP were detected among 16 samples, which was approximately 33 percent of all of the samples. Another five-year prospective study stated that 15.2 percent of 57 posterior Y-TZP FPDs showed chipping of the veneering porcelain after five years of clinical observation.<sup>4</sup> It was found that Y-TZP offers sufficient stability as a coping material, and that the success rate of Y-TZP copings was 97.8 percent. However, VP should be improved to prevent chipping complications.

On the other hand, the rate of VP fracture on metal ceramic restorations has been reported as 2.5 percent after five years.<sup>9</sup> Therefore, the mechanical integrity and bonding strength between the VP and Y-TZP coping are thought to be key factors in making Y-TZP all-ceramic restorations as reliable as metal-ceramic restorations.<sup>10,11</sup>

#### INFLUENCE OF SURFACE TREATMENT ON BOND STRENGTH

Several studies have reported that the bond strength between Y-TZP copings and VP is significantly affected by some surface treatments such as air-borne particle abrasion, heat treatment, and use of liner porcelain.<sup>11,18,22-24</sup> However, the influence of different surface treatments on the bond strength of veneering porcelain on zirconia copings is not clear.

In terms of application of liner porcelain, Aboushelib<sup>10</sup> reported that the microtensile bond strength of the Cercon coping with VP was significantly weaker if liner porcelain was not applied. On the other hand, Kim et al.<sup>22</sup> stated that the liner



porcelain application significantly decreases the shear bond strength compared with airborne particle abrasion. Tinschert et al.<sup>6</sup> stated the liner porcelain application can significantly weaken bond strength and increase the percentage of interfacial failures between coping and pressable veneer ceramic. Moreover, some studies concluded the influence of liner porcelain application on bond strength is dependent upon the zirconia material used.<sup>18,23</sup>

Airborne particle abrasion is used to increase surface roughness and the wetting ability of Y-TZP to enhance the bond strength.<sup>23</sup> Some studies have reported that airborne particle abrasion was found to decrease the percentage of interfacial failure pattern.<sup>10,22</sup> On the other hand, Fischer et al.<sup>11</sup> suggested that airborne particle abrasion was not an essential surface pretreatment to enhance bond strength. Aboushelib et al.<sup>18</sup> suggested that the presence of a monoclinic phase of zirconium oxide at the core-veneer interface resulting from stress-generating surface treatments, such as grinding or sandblasting, may be the cause of microspaces found at the interface. In addition, Guazzato et al.<sup>19</sup> found that tetragonal-monoclinic transformation is accompanied by a generation of localized stresses, which may nucleate microcracks in the glass phase of the veneer.

Some studies have investigated the effect of heat treatment after airborne particle abrasion on the Y-TZP surface area.<sup>11,20,21</sup> They stated the heat treatment at approximately 900°C and the firing process caused reverse transformation and release of the compressive stresses, so that phase transformation resulting from airborne particle abrasion does not affect the bond strength between Y-TZP copings and veneering porcelain. In fact, Doi et al. concluded<sup>20</sup> that there is significantly higher

debonding/crack-initiation strength with heat treatment after airborne-particle abrasion than without. However, Fischer et al.<sup>11</sup> concluded that heat treatment significantly decreased the shear strength of both polished and sandblasted surfaces.

Of additional interest is the influence of airborne particle abrasion on mechanical strength of Y-TZP. Numerous studies have investigated this influence.<sup>19,47,57</sup> Most of the studies describe that even though airborne particle abrasion can initiate some defects or microcracks on the Y-TZP coping resulting in influence on the mechanical strength, it is thought to be a process that can induce phase transformation that results in transformation toughening without the use of high temperatures or the creation of severe surface damage of the coping.<sup>23,25,47</sup>

## MATERIALS AND METHODS

In this study, shear bond testing was performed, investigating the influence of different surface treatments on the Y-TZP-VP bonding with or without cyclic loading.

Forty-eight yttrium-stabilized tetragonal zirconia polycrystal ceramic specimens (Diazir™ Full Contour Zirconia, Ivoclar Vivadent, Amherst, NY) (TABLE I) were manufactured using a CAD/CAM machine (Sirona InLab MC XL, Sirona Dental Systems LLC, Charlotte, NC). The zirconia material was supplied by the manufacturer in large disk-shaped blocks. Small zirconia blocks ( $10 \times 10 \times 10 \text{ mm}^3$ ) were prepared using a band saw. Next, the samples were placed in a furnace (Pyro oven, HD Justi Co., Oxnard, CA) at  $135^\circ\text{C}$  for 30 minutes to remove moisture. The zirconia samples were then milled to the required shape and dimensions (6 mm in diameter and 4 mm in height) (Figure 1, Figure 2). Subsequently, the specimens were sintered in a high-temperature furnace (Programat S1, Ivoclar-Vivadent, Buffalo, NY) at  $1500^\circ\text{C}$  for 8 hours. The material is known to experience 19.264-percent shrinkage during sintering. The computer software compensates for this shrinkage during milling to produce finished specimens of the proper size. In addition, all the materials were ground on one side with a #320 diamond paper while water-cooling in order to produce a condition similar to that of milled zirconia coping (Figure 3). The specimens were cleaned in an ultrasonic bath containing acetone for 15 minutes (Figure 4).

The specimens were divided into four groups containing 12 specimens each according to surface treatment. As a control group (C), no further treatment was applied to the specimens after grinding. Group H was heat-treated in the furnace (Cerampress

QEX, Dentsply Prosth, York, PA) with an increasing rate of 55°C/min from 650°C to 1000°C as a pretreatment according to the manufacturer's recommendations (Figure 5, Figure 6). Group S was airborne-particle abraded with 50-µm alumina (Al<sub>2</sub>O<sub>3</sub>) particles under a pressure of 0.4 MPa for 10 seconds in a direction perpendicular to the surface and at a distance of 10 mm using an airborne-particle abrasion device (Sandstorm Expert, Vaniman Co, Fallbrook, CA) moved over the surface to abrade it as evenly as possible (Figure 7, Figure 8). In the group SH, the heat-treatment was performed after the airborne-particle abrasion. All specimens were cleaned in an ultrasonic bath containing acetone for 15 minutes.

On the prepared surface of each specimen, a veneering porcelain cylinder was applied using a custom-made split silicone mold (Figure 9, Figure 10). Porcelain powder (Vintage ZR, Shofu, Japan) (Table I) was mixed with the appropriate amount of distilled water and added to build a cylinder 2.4 mm in diameter and 2 mm in height (Figure 11 to Figure 14). Excess water was removed with tissue paper. Firing was performed in a calibrated porcelain furnace with an increasing rate of 45°C/min from 650°C to 910°C according to the manufacturer's recommendations. A second firing was required to compensate for the porcelain shrinkage that occurred during the first firing (Figure 15, Figure 16).

Each specimen was embedded in an acrylic resin mold with type 4 stone. For an adequate positioning perpendicular to horizontal plane, the specimens were set up, using a custom-made plastic plate (0.5-mm thickness) and a special device (Figure 17 to Figure 20). The 12 specimens for each group were randomly divided into two subgroups of six specimens, either with or without cyclic loading, before the shear bond strength test. The

cyclic loading subgroup was subjected to fatigue testing under cyclic loading in a mechanical cycling machine (Electropuls 3000, Instron, Norwood, MA) (Figure 21). The specimens were securely mounted in a steel-supporting vice and a custom-made acrylic resin mold (Figure 22). A cylindrical loading jig with a 10-mm diameter axially induced 10 N loads for 10,000 cycles with a frequency of 1.0 Hz. The cycling was conducted at room conditions (Figure 23, Figure 24).

### SHEAR BOND STRENGTH TEST

All specimens were mounted and placed in a shear-testing device with a semicircular loading surface (2.4 mm in diameter). The shear bond strength was determined using a screw-driven universal testing machine (Sintech ReNew 1123, MTS, Shakopee, MN) at a crosshead speed of 0.5 mm/min (Figure 25 through Figure 27). The shear bond strength was calculated using the following formula: Shear bond strength (MPa) = Load (N)/area (mm<sup>2</sup>).

### The Mode of Failure

Failure mode was observed and classified into three categories: interfacial failure (at the interface between VP and Y-TZP coping), cohesive failure (within the VP), and combination failure (interfacial and cohesive failures). The observation was made with an optical microscope (Measurescope UM-2, Nikon, Japan) at X40 magnification after shear bond strength testing.

### Statistical Analyses

Summary statistics (mean, standard deviation, standard error, median, 1<sup>st</sup> and 3<sup>rd</sup> quartiles, minimum, and maximum) were calculated for the shear bond strength data for

each of the four surface treatment groups with and without cyclic loading. The effects of heat treatment, particle abrasion, and cyclic loading on shear bond strength were evaluated using three-way ANOVA, followed by pair-wise group comparisons using the Sidak multiple comparisons procedure at an overall 5-percent significance level. Due to the non-normal distribution of the bond strength measurements, the ANOVA was performed using the ranks of the data.

#### Sample Size Justification

Based on previous studies,<sup>1,11,14</sup> the standard deviations were expected to be 4 MPa for shear bond strength. With a sample size of six specimens per surface treatment group with and without cyclic loading, the study was designed to have 80-percent power to detect a difference of 9.8 MPa between any two surface treatments with and without cyclic loading, assuming two-sided tests conducted at an overall 5-percent significance level.

## RESULTS



The original mean values for each of the four surface treatment groups with and without cyclic loading are presented in Table II. There were some outliers in every group, so Table III shows the adjusted values using the ranks of data due to the non-normal distribution of the bond strength measurements. The highest mean shear bond strength was recorded for the air-particle abrasion group without cyclic loading ( $34.1 \pm 10$  MPa). The lowest mean shear bond strength was for the air-particle abrasion group with cyclic loading ( $10.7 \pm 15.4$  MPa). Two specimens in the air-particle abrasion group with cyclic loading showed complete delamination of VP during the fatigue loading, and the shear bond strength of these specimens were calculated as 0.0 MPa.

The ranks of data in the ANOVA were used to treat it essentially as a nonparametric analysis. The three-way ANOVA showed no statistically significant effect of surface treatment and cyclic loading on shear bond strength. Sidak multiple comparisons procedure was performed to make all pair-wise group comparisons among the eight subgroups in the present study (Table IV, Table V). There was no significant effect of air-particle abrasion overall ( $p = 0.39$ ) or for any heat treatment or cyclic loading combination ( $p > 0.26$ ). There was no significant effect of heat treatment overall ( $p = 0.28$ ) or for any cyclic loading or air-particle abrasion combination ( $p > 0.06$ ). Cyclic loading specimens had significantly lower shear bond strength than non-cyclic loading specimens after air-particle abrasion without heat treatment ( $p = 0.0126$ ). Cyclic loading had no effect for any other heat treatment or air-particle abrasion combination ( $p > 0.40$ ).

The results of failure mode were classified into three categories, interfacial, cohesive, and combination. All the specimens showed a combination of interfacial and cohesive failure independent of the surface treatment and with or without cyclic loading.

TABLES AND FIGURES

TABLE I

Materials used in this study

Material	Brand Name	Manufacturer	Component
Zirconia	Diazir <sup>TM</sup> Full Contour Zirconia	Ivoclar vivadent, Amherst, NY	ZrO <sub>2</sub> (>94%), Y <sub>2</sub> O <sub>3</sub> (5.15%) Al <sub>2</sub> O <sub>3</sub> (<0.1%) HfO <sub>2</sub> (<3.0%)
Veneering porcelain	Vintage ZR	Shofu, Japan,	SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , K <sub>2</sub> O, Na <sub>2</sub> O, CaO, B <sub>2</sub> O <sub>3</sub> , Pigment, and Fluorescence

TABLE II

Means of shear bond strength

Fatigue	Heat	Particles	N	Mean (SD)	Min	Q1	Median	Q3	Max
n	n	n	6	31.2 (14.5)	8.8	24.5	31.9	37.9	51.9
n	n	y	6	42.8 (6.6)	36.8	37.6	40.25	50.4	51.2
n	y	n	6	37.4 (15.2)	17.5	18.9	43.45	48.3	52.5
n	y	y	6	28.8 (13.3)	2.6	28.8	33.35	35.4	39.3
y	n	n	6	34.0 (9.6)	16.5	29.7	38.1	40.1	41.6
y	n	y	6	12.2 (18.5)	0	0	2	24.3	44.7
y	y	n	6	37.4 (11.0)	18.6	33.3	38.3	46.8	49
y	y	y	6	36.0 (13.0)	9.8	37.6	41.2	43	43.3

\*Q1 is the 1<sup>st</sup> quartile (25% of the data are less than Q1). Q3 is the 3<sup>rd</sup> quartile (75% of the data are less than Q3).

TABLE III

Means of shear bond strength – ranks of bond strength measurements

Fatigue	Heat	Particles	N	Mean (SD)	Min	Q1	Median	Q3	Max
n	n	N	6	21.3 (14.6)	6	13	17	28	47
n	n	y	6	34.1 (10.0)	24	25.5	32	45	46
n	y	n	6	31.2 (16.9)	9	11	38	43	48
n	y	y	6	17.5 (8.2)	4	15	18	21	29
y	n	n	6	23.8 (10.2)	8	16	27	31	34
y	n	y	6	10.7 (15.4)	1.5	1.5	4	12	41
y	y	n	6	28.2 (13.7)	10	18	27.5	42	44
y	y	y	6	29.3 (12.0)	7	25.5	33	38	39

\*Q1 is the 1<sup>st</sup> quartile (25% of the data are less than Q1). Q3 is the 3<sup>rd</sup> quartile (75% of the data are less than Q3).

TABLE IV

The three-way ANOVA

Effect	Num DF	Den DF	F Value	p-value
Fatigue	1	40	0.67	0.4194
Heat	1	40	1.18	0.2848
fatigue*heat	1	40	3.96	0.0535
Particles	1	40	0.76	0.3885
fatigue*particles	1	40	0.56	0.4583
heat*particles	1	40	0.67	0.4194
fatigue*heat*particles	1	40	7.44	0.0094

TABLE V

Pair-wise group comparisons  
for fatigue, heat and particles

Heat	Particles	p-value
n	n	0.9954
n	y	0.0126
y	n	0.9907
y	y	0.4082

Fatigue	Particles	p-value
all	all	0.2848
n	all	0.7750
y	all	0.0702
all	n	0.3385
all	y	0.9777
n	n	0.5794
n	y	0.1214
y	n	0.9640
y	y	0.0660

Fatigue	Heat	p-value
all	all	0.3885
n	all	0.9953
y	all	0.4503
all	n	0.9990
all	y	0.4220
n	n	0.3292
n	y	0.2655
y	n	0.2991
y	y	0.9998



FIGURE 1. Macro photograph of Y-TZP specimens.



FIGURE 2. Illustration of Y-TZP specimen surface.



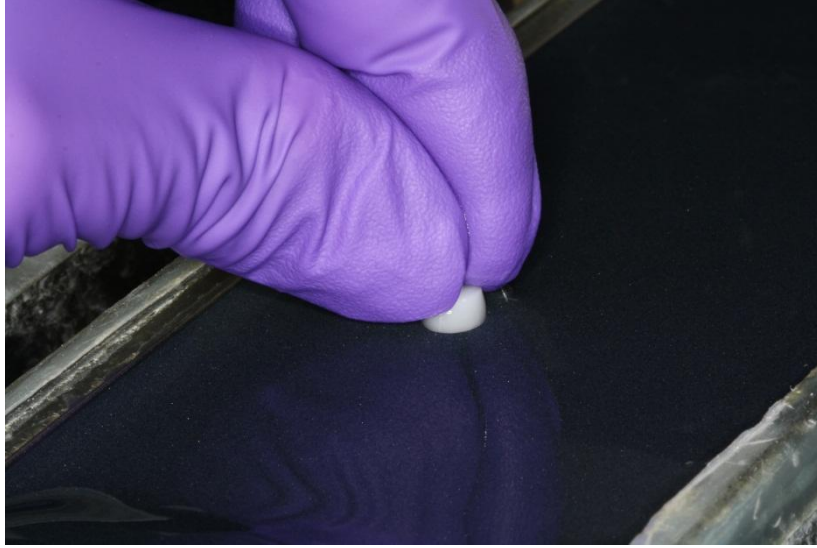


FIGURE 3. Demonstration of polishing of Y-TZP specimens with water cooling.



FIGURE 4. Illustration of cleaning of specimens in an ultrasonic bath containing acetone.

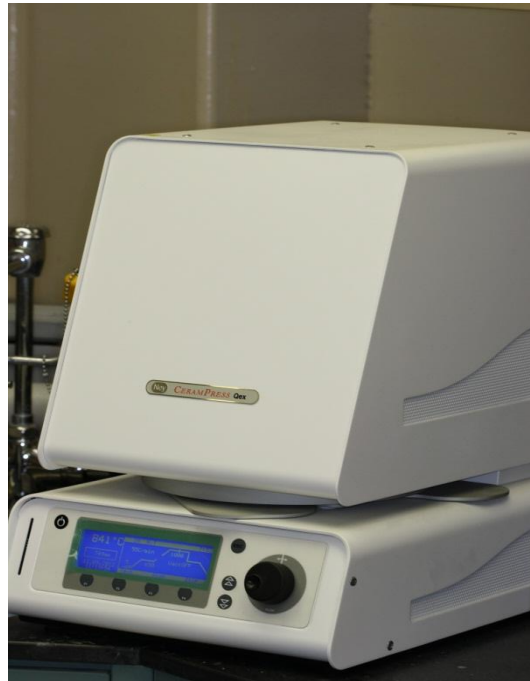


FIGURE 5. Cerampress QEX, (Dentsply Prosth) used for firing VP and heat treatment.



FIGURE 6. Illustration of heat treatment of Y-TZP specimens.



FIGURE 7. Illustration of customized plastic box to abrade Y-TZP as evenly as possible.

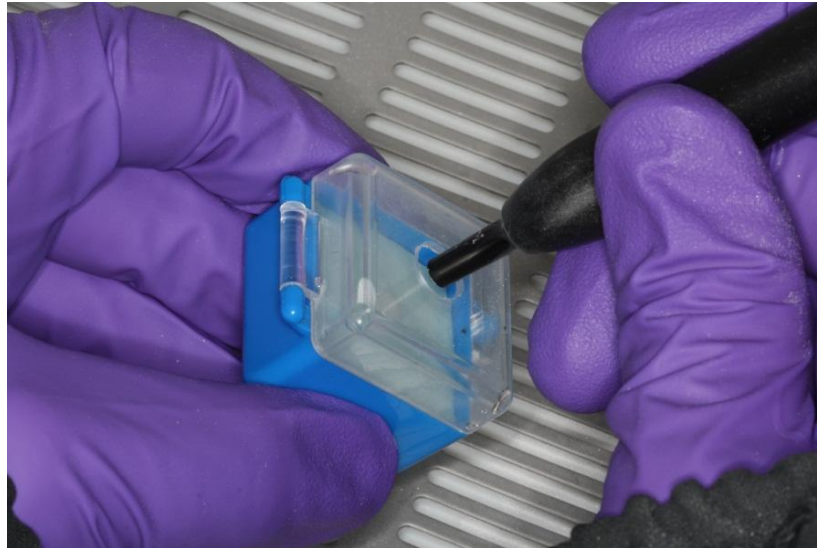


FIGURE 8. Illustration of air borne particle abrasion of Y-TZP with the customized plastic box.

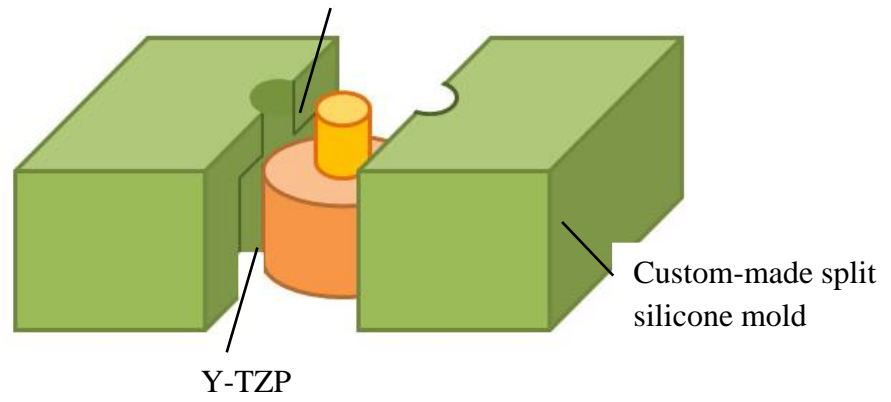


FIGURE 9. Veneering porcelain by custom-made split silicone mold.



FIGURE 10. Illustration of a custom-made split silicone mold.



FIGURE 11. Illustration of veneering porcelain (Vintage ZR, Shofu) mixed with the appropriate amount of distilled water.

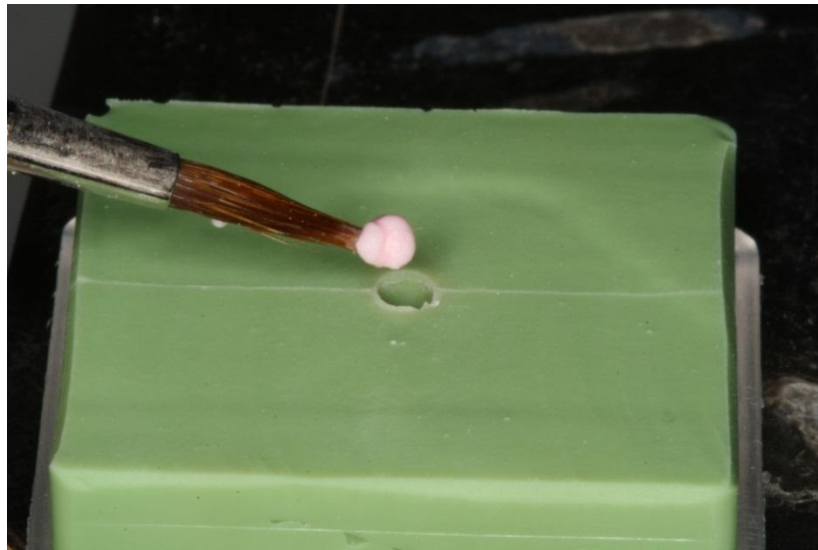


FIGURE 12. Illustration of applying of veneering porcelain with the split silicone mold.



FIGURE 13. Illustration of building up of veneering porcelain.

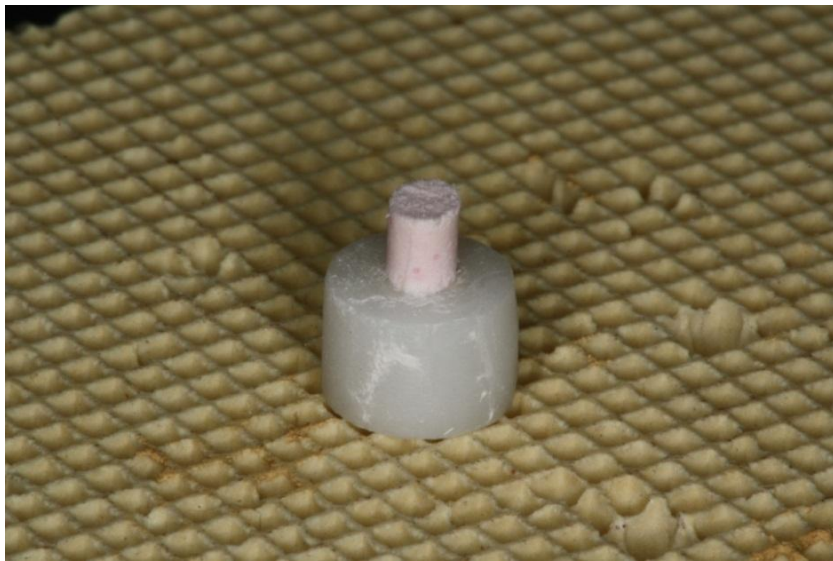


FIGURE 14. Illustration of veneering porcelain cylinder on the Y-TZP specimen.

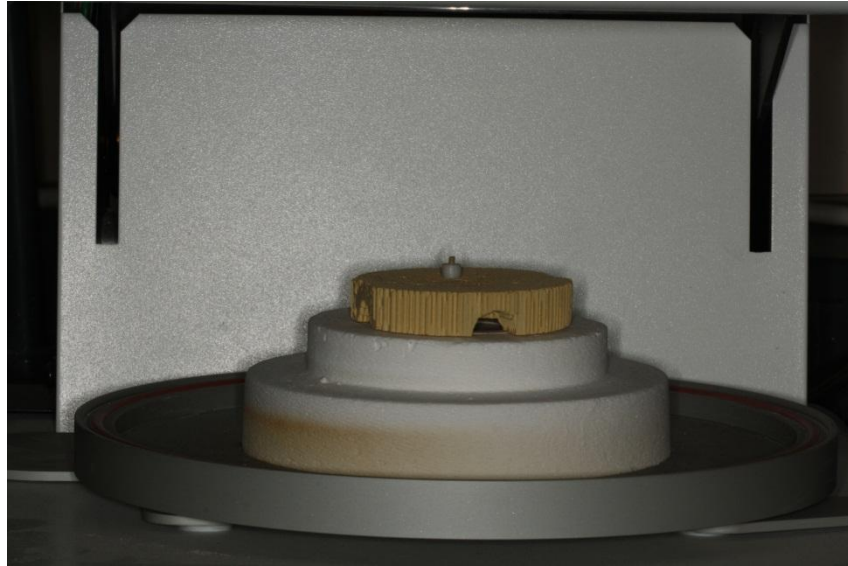


FIGURE 15. Illustration of firing the veneering porcelain.

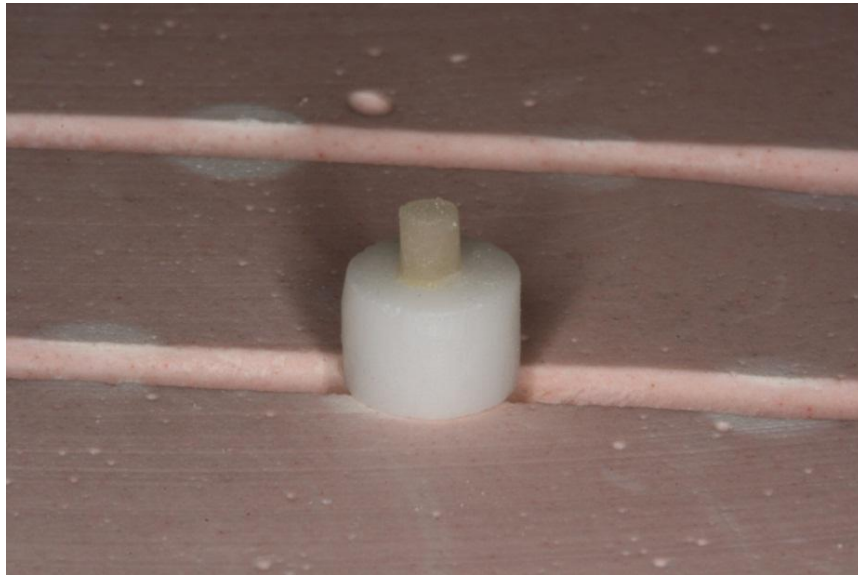


FIGURE 16. Illustration of fired veneering porcelain on the Y-TZP.

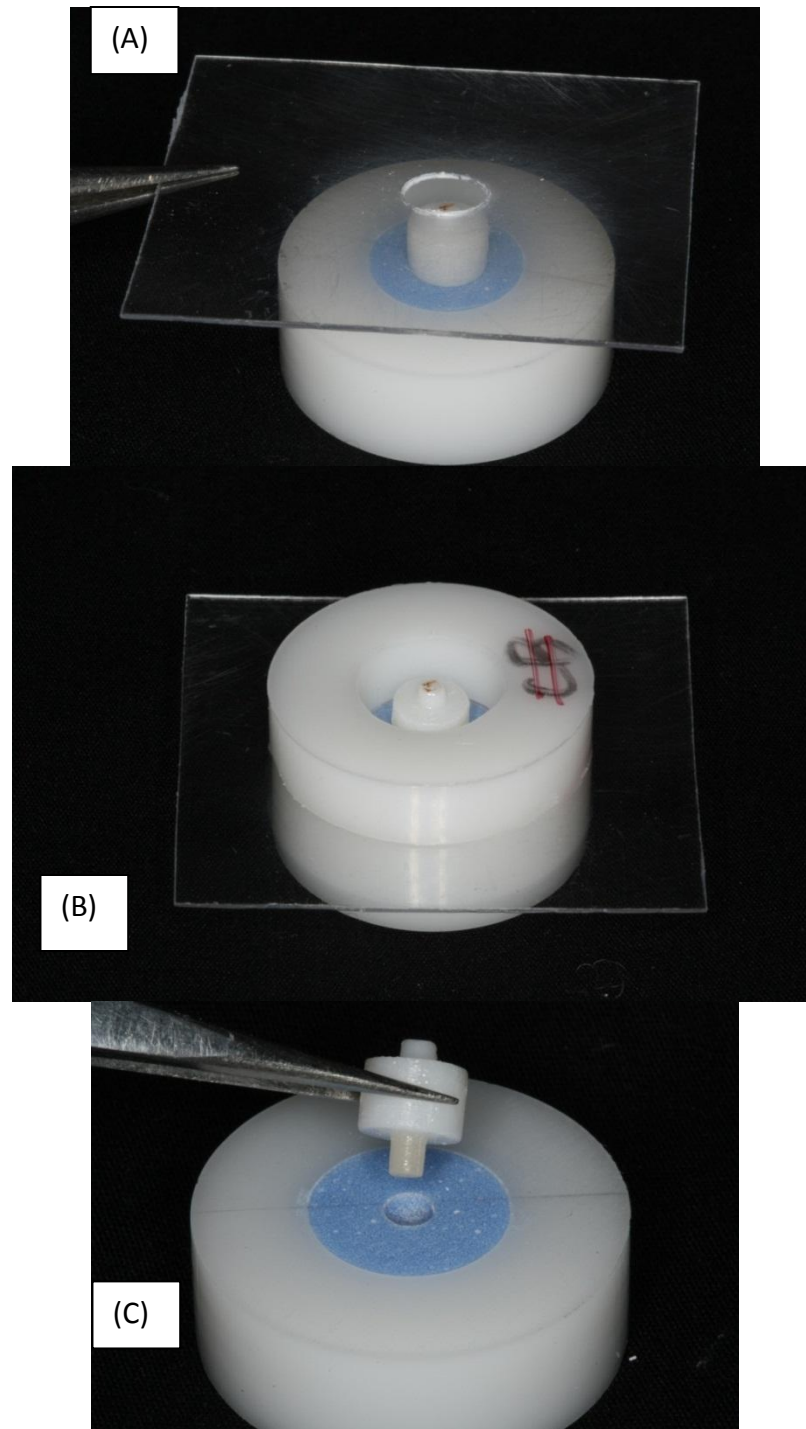


FIGURE 17. Illustrations of perpendicular positioning, using custom-made plastic plate. (A) Setting of specimen on the foundation, (B) Setting of the plastic plate, (C) Setting of the acrylic resin mold on the plastic plate.



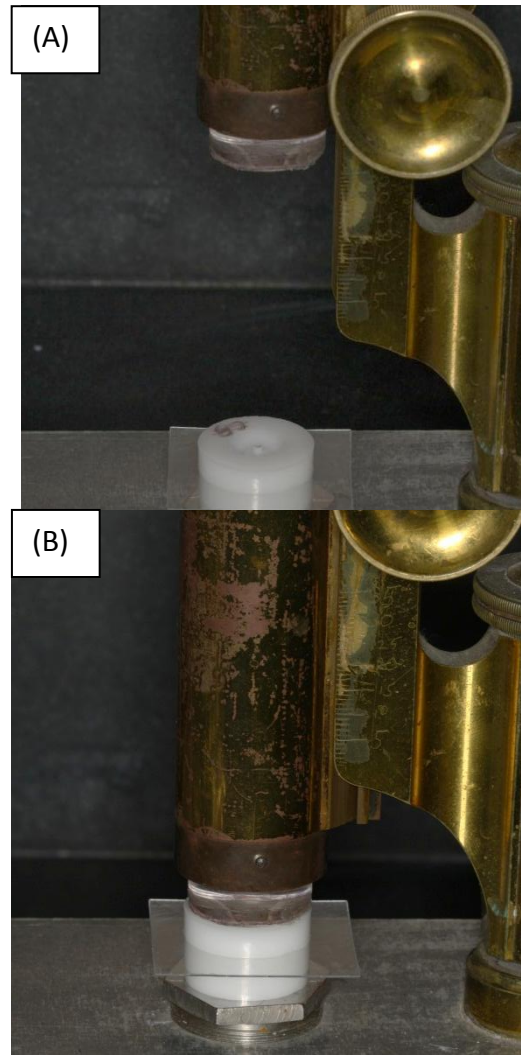


FIGURE 18. Illustrations of mounting of specimens in the acrylic resin mold by using a paralleling device. (A) Setting the specimen on the special device. (B) Mounting of the specimen.



FIGURE 19. Illustration of the specimen embedded in the acrylic resin mold with type 4 stone.

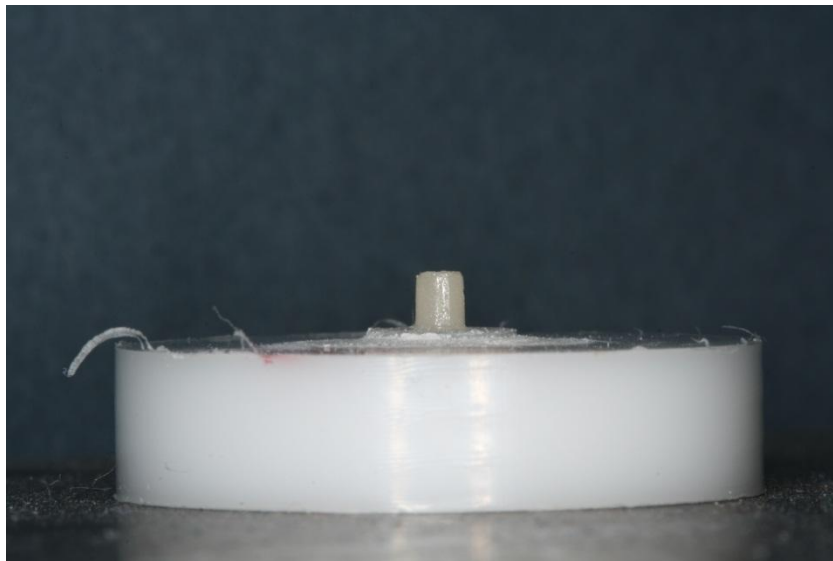


FIGURE 20. Macrophotograph of horizontal view of embedded specimen.



FIGURE 21. Illustration of mechanical cycling machine (Electropuls 3000, Instron).

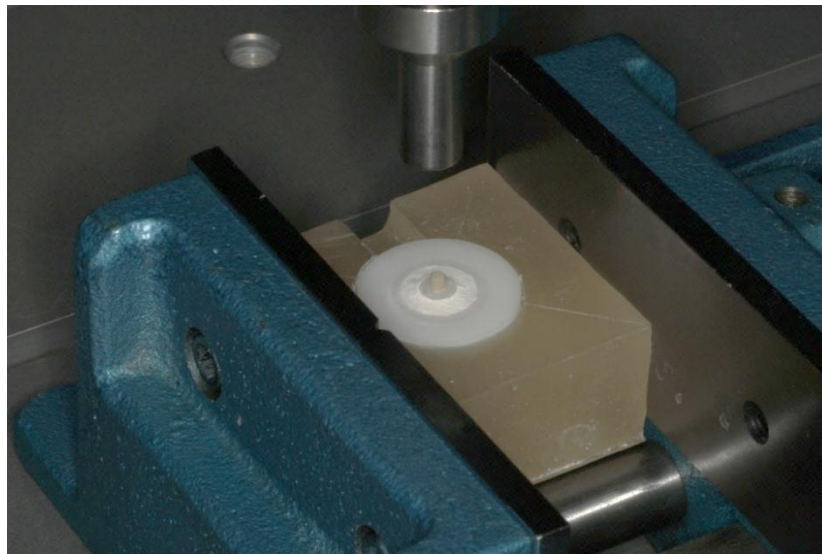


FIGURE 22. Illustration of mounted specimens secured in a custom-made acrylic resin.

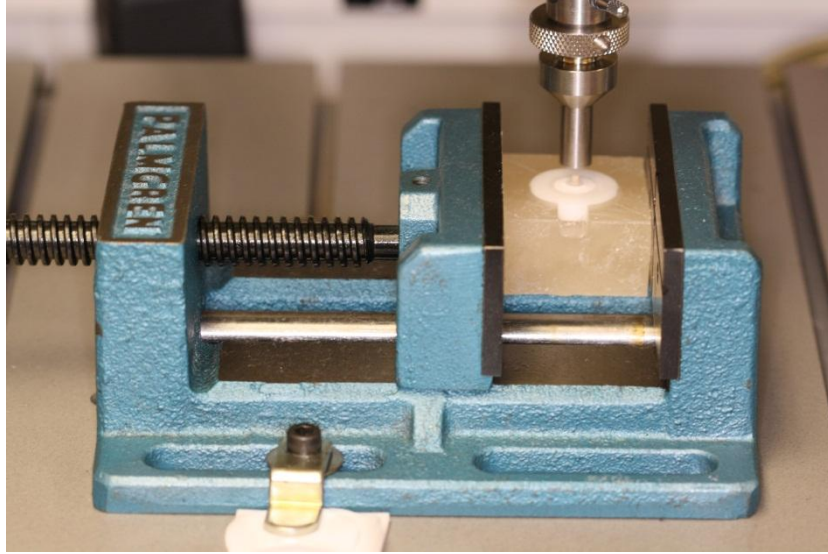


FIGURE 23. Illustration of a steel supporting vice on the mechanical cycling machine.

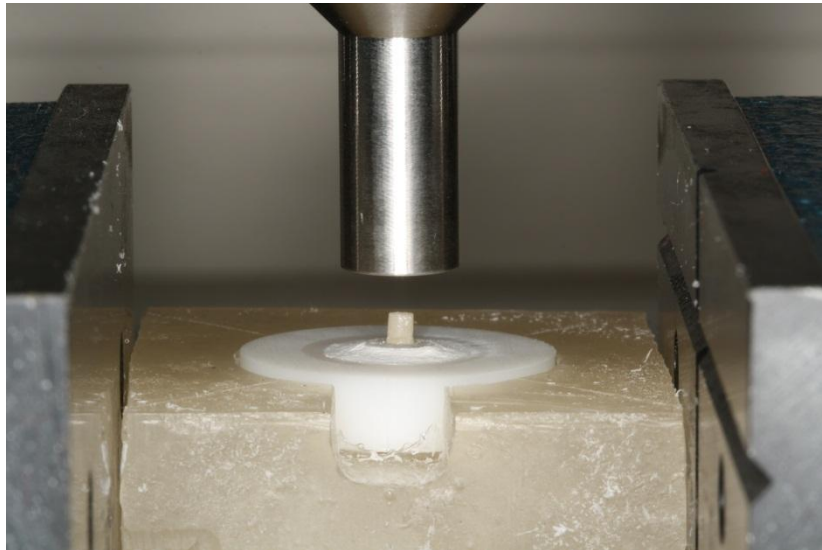


FIGURE 24. Illustration of a cylindrical loading jig.

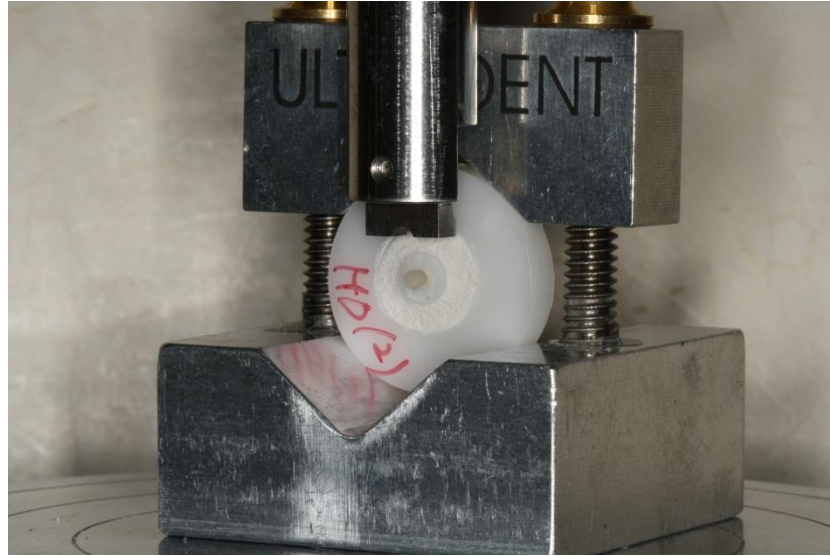


FIGURE 25. Illustration of universal testing machine (Sintech ReNew 1123, MTS) with mounted specimen.



FIGURE 26. Macro photograph of specimen placed in a shear testing device with a semicylindrical loading surface.

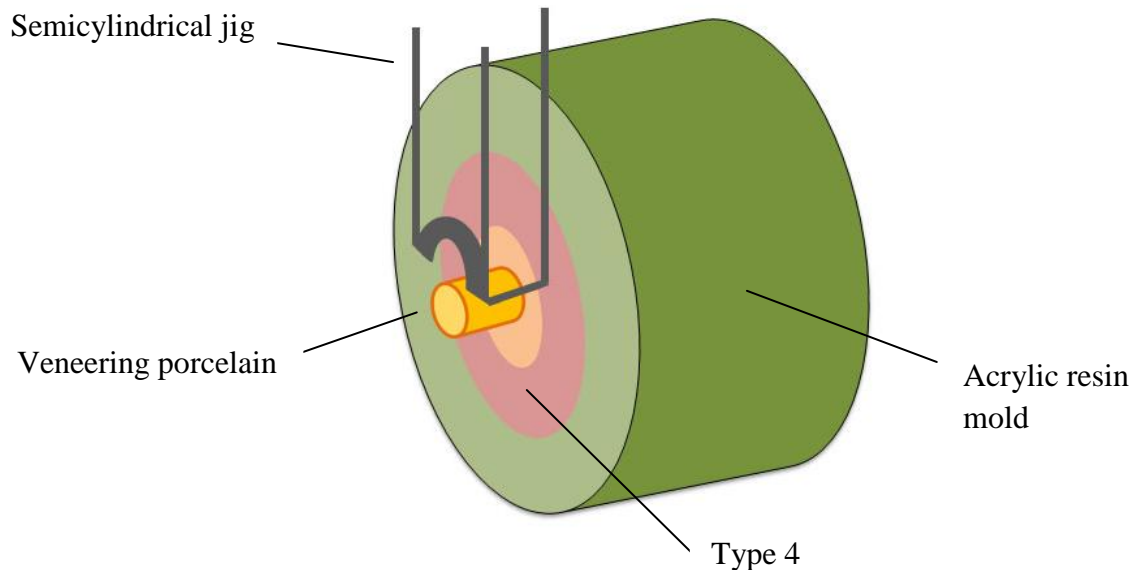


FIGURE 27. Shear bond strength test.

DISCUSSION

## SHEAR BOND STRENGTH

Adequate bond strength between veneering porcelain and metal coping of metal ceramic restorations has been considered to be greater than 25 MPa.<sup>58</sup> The bond strength measurement was determined by the Organization of Standardization by using three-point bending tests and the mean debonding strength/crack initiation strength.<sup>59</sup> However, the test setup cannot be applied to all ceramic restorations due to the brittleness of the all ceramic coping.<sup>60</sup> There are several tests to measure the bond strength of all ceramic restorations in the literature, such as the shear bond strength test, the Schmitz-Schulmeyer test, three- and four-point loading tests, the biaxial flexure strength test, and the microtensile bond strength test.<sup>1,10-12,14,18,22,61</sup> However, each test method has its advantages and disadvantages, and a common limitation of those tests is the difficulty of applying failure load to the specimen surface in the specific test setup. As a result, the standardized test for bond strength and adequate bond strength of all ceramic restorations has not been determined. In the present study, the shear bond strength test was selected because this test is relatively simple, easy to perform, and can simulate shear stresses, which are significant contributors to deterioration and bonding failure of restorative materials.<sup>61</sup> However, some aspects should be considered including storage conditions, types of substrate, specimen preparation, rate of load application, cross-section surface area, and experience of the researcher.<sup>12</sup>

In comparison with other studies<sup>12,14</sup> using the same test design and surface conditions to measure the shear bond strength, bond-strength values obtained in the



present study for the air-particle abrasion group and the heat treatment group without cyclic loading are in the same range as those reported previously. However, Kim et al.<sup>22</sup> reported the mean shear bond strength of VP to Y-TZP zirconia coping ground on the Y-TZP surface with a #320 diamond paper was 32.08 MPa, which was significantly higher than the control group shear bond strength obtained in this study. In the present study, different manufactured products of Y-TZP and VP were used, yttrium-stabilized full-contour zirconia (Diazir<sup>TM</sup> Full Contour Zirconia, Ivoclar Vivadent, Amherst, NY) and porcelain powder (Vintage ZR, Shofu, Tokyo). The CTE of the Y-TZP coping ( $10.6 \times 10^{-6}/^{\circ}\text{C}$ ) was slightly higher than that of the VP ( $9.4 \times 10^{-6}/^{\circ}\text{C}$ ). An acceptable CTE of VP was reported to be approximately  $1.0 \times 10^{-6}/^{\circ}\text{C}$  below the CTE of the Y-TZP.<sup>62,63</sup> In addition, Saito et al. stated that there was no correlation of shear bond strength with CTE mismatch between Y-TZP coping and five VP for zirconia used in the range of  $1.0\text{-}1.7 \times 10^{-6}/^{\circ}\text{C}$ .<sup>14</sup> Therefore, the combination of VP and Y-TZP products used in the present study is in agreement with those results of previous studies in terms of the CTE difference. However, the correlation between the VP and Y-TZP used in the present study with microstructural composition or properties is not clear.

The Y-TZP specimen size and shape used in a previous study were different from that of the present study, e.g. previously used square-shaped ( $5 \times 10 \times 10 \text{ mm}^3$ ) specimens versus cylindrical-shaped (6 mm in diameter and 4 mm in height) blocks in the present study. The volume of Y-TZP coping used in the cited study was four times larger than the one in this study, so that the greater volume of the Y-TZP coping may slow down the sintering process and the cooling of the VP because of the greater heat capacity. The thickness ratio of the Y-TZP coping and VP was reported as one of key factors to

prevent the failure in the development of thermal residual stresses.<sup>64</sup> Considering that the nominal Y-TZP/VP thickness ratio of fixed restorations in clinical situations is 0.5 mm to 1.0 mm, the ratio used in this study may be more relevant clinically for cooling-rate differences.

According to a literature review by Denry,<sup>65</sup> it is clear that one physical factor plays a significant role in the mechanism of chipping fracture, mainly residual stresses. There are three effects that are key contributors to the development of the residual stresses: 1) CTE mismatch, 2) Cooling rate, 3) a stress induced phase transformation of Y-TZP. Nevertheless, it was reported that an optimal Y-TZP/VP thickness ratio is difficult to establish owing to the complex geometry of Y-TZP restorations.<sup>66</sup> Therefore, the author concluded that further investigations of microstructural analyses of the interface between VP and Y-TZP coping are required to better establish the role of pre-existing proper stresses induced from the thermal expansion due to the phase transformation, as well as intergranular stresses on the transformability of Y-TZP.<sup>65</sup> The effect of microstructural composition and property of Y-TZP and VP should be considered.

## SURFACE TREATMENT

The first hypothesis, that shear bond strength between Y-TZP and VP would be increased by zirconia surface treatment using airborne-particle abrasion, heat treatment, and heat treatment after airborne-particle abrasion, was rejected, because there was no statistically significant effect for the shear bond strength according to the surface treatment in this study.

On the effect of air-particle abrasion, the mean SBS value of the air-particle abrasion group without cyclic loading was higher than the control group in this study, but there was no significant effect of air-particle abrasion overall from the results of the pairwise tests. This finding was comparable to previous studies.<sup>11,23</sup> Fischer et al. stated that no differences in SBS were found between the polished and sandblasted surfaces with three out of five ceramics. Consequently, surface roughness as created by sandblasting was not necessary to enhance bond strength.<sup>11</sup> In contrast, some studies have reported that airborne particle abrasion was found to decrease the percentage of interfacial failure pattern.<sup>10,22</sup> However, the results in the aforementioned studies could not be compared with those in this study, given that one of the authors used a different bond strength test and microtensile bond strength test. Moreover, the latter's study design did not give data for the polished surface group and the air-particle abrasion group separately.<sup>10</sup> Additionally, another study concluded that significant differences were found between air-particle abrasion groups and a liner-applied group, but the zirconia-surface-treated groups did not show a significant difference compared with the control group.<sup>22</sup>

The result of the failure modes was that all the specimens showed a combination of interfacial and cohesive failure in spite of the surface treatment and with or without cyclic loading. The interfacial failure pattern has been related to the increased stresses resulting from the difference of the elastic moduli between Y-TZP coping and VP.<sup>67</sup> Some studies described that the failure mode is greatly affected by not only the test methodology but also the mismatch of CTE.<sup>61,68,69</sup> Moreover, air-particle abrasion has been suggested to compromise the mechanical strength of the VP by initiating surface defects on the Y-TZP coping that lead to interfacial failure as stress concentration

sources.<sup>67,70</sup> Different wetting conditions or different surface energies should be considered to improve the interfacial bond strength as well.

Air-particle abrasion is likely to trigger the phase transformation of Y-TZP. The fracture toughness of the material after the phase transformation is dependent on the amount of tetragonal phase retained. In other words, the important consequence of the phase transformation is the development of surface compressive stresses at the surface.<sup>65</sup> Kosmac et al. reported that air-particle abrasion is more effective than grinding in inducing the phase transformation.<sup>25</sup> The efficacy of the phase transformation depends on the mean grain size, and larger grain size results in more.<sup>71</sup>

However, the monoclinic phase created by the air-particle abrasion or grinding was suggested to lead to tensile stress in the VP because of the quite low CTE of the monoclinic phase.<sup>11</sup> The tensile stress would cause VP fracture. Therefore, heat treatment was recommended to cause the reversal of the monoclinic phase to the tetragonal phase.<sup>72</sup> Based on the results of the present study, there was no significant effect of heat treatment overall, which is in agreement with a previous study.<sup>11</sup> In contrast, some studies have reported that heat treatment in the temperature range 850 °C to 1000 °C induces the reverse transformation as does the heat generated during grinding.<sup>73,74</sup> Therefore, Fischer concluded that even if heat treatment can relax the compressive stresses at the surface, microcracks did not close at this temperature. As a result, the overall of strength decreased, affecting the SBS of the VP adjacent to the interface.<sup>11</sup>

According to Denry,<sup>65</sup> mechanical and thermal residual stresses were shown to play an important role in the mechanical performance of Y-TZP restorations. The mechanical residual stress results from grinding or air-particle abrasion, and the thermal

residual stress is developed from the veneering process or heat treatment. It is assumed that the effect of mechanical retention from air-particle abrasion may not outweigh the influence of the mechanical and thermal residual stresses with the mechanical defects. Further investigations of how the surface treatment influences thermal and mechanical residual stress for bonding strength are required.

### CYCLIC LOADING

In the present study, cyclic loading specimens had significantly lower shear bond strength than non-cyclic loading specimens after air-particle abrasion without heat treatment. The second hypothesis, cyclic loading would decrease the shear bond strength between Y-TZP and VP, was at least partially accepted. According to Harding et al.,<sup>75</sup> cyclic loading did not affect bond strengths, regardless of surface treatment including air-particle abrasion. However, in the previous study, microtensile bond strength testing was completed with pressable veneer porcelain instead of layering veneer porcelain on the Y-TZP. As a consequence, the previous study cannot be compared with the results of the present study.

The cyclic loading parameters used in this study were a 10 N load and 10,000 cycles. Physiological chewing forces have been found to range between 20N and 120N, depending on the food consistency.<sup>76</sup> Kim et al. reported that inner cone crack formed for VP/Y-TZP bilayers at 10,000 cycles and the cracks reached the interface after 50,000 cycles.<sup>77</sup> The size of specimens was much smaller than normal crown sizes in clinical situations, and the aim of this study was to evaluate shear bond strength and the effect of surface treatment without specimen fracture prior to the shear bond strength. Therefore, the loading protocol used in the present study was developed as mentioned previously.

Failure a few years after cementation is more likely to involve subcritical crack growth (SCCG) and cyclic fatigue.<sup>65</sup> SCCG is defined as occurring at stresses below the critical value until the crack reaches its critical length leading to fast failure. Specifically, it has been reported that all ceramic restorations are indeed susceptible to the SCCG in the humidity of the oral environment.<sup>78,79</sup> As mentioned previously, air-particle abrasion induces mechanical defects, mechanical residual stresses, and phase transformations on the Y-TZP surface. When subjected to cyclic fatigue, the mechanical defects due to air-particle abrasion may be origins of failure as foci of stress concentration and residual stress within the interface. Moreover, the phase transformation due to the air-particle abrasion may cause SCCG and volumetric expansion of the Y-TZP surface as a result of the fatigue loading.

In the present study, there was a significant difference in shear bond strength with air-particle abrasion between with- and without-cyclic loading groups. This difference suggested that air-particle abrasion should be avoided in clinical situations as a surface treatment without heat treatment. Moreover, there are numerous studies concluding that bond strength of Y-TZP restorations can be comparable to one of metal ceramic restorations. Consequently, similar clinical performance of Y-TZP restorations is expected when compared with metal ceramic restorations. However, based on the results of this study, cyclic fatigue may need to be considered with measurements of the bond strength test results when attempting to predict clinical performance.

The design of the present study has several limitations for comparing the findings with the clinical situations. In the present study, the standard deviations of the shear bond strength data were greater than other studies. According to Al-Dohan,<sup>12</sup> a curved knife

wrapping around the cylinder shape of VP was recommended to minimize load concentration. However, a semi-cylinder shape of jig was used for shear bond strength test in this study. The occurrence of non-uniform interfacial stress by the semi-cylindrical shape of the jig may ascribe to the greater standard deviations of data. Moreover, some studies have reported that the application of liner porcelain affects the shear bond strength as a surface treatment as well.<sup>10,18,22,23</sup> To avoid the high chipping or cracking rates of VP on the Y-TZP coping, further investigations are necessary, such as studies on microstructural property, wetting property, residual stress, and impact fatigue.

SUMMARY AND CONCLUSIONS



Within the limitations of this *in-vitro* study, the following conclusions could be drawn:

1. Shear bond strength between Y-TZP and VP is not affected statistically by surface treatment using heat treatment, airborne-particle abrasion, and heat treatment after airborne-particle abrasion.
2. There is a significant difference of shear bond strength with air-particle abrasion between with- and without-cyclic loading groups. This difference suggests that air-particle abrasion should be avoided in clinical situations as a surface treatment without heat treatment.

REFERENCES

1. Guess PC, Kulis A, Witkowski S, et al. Shear bond strengths between different zirconia cores and veneering ceramics and their susceptibility to thermocycling. *Dent Mater* 2008;24(11):1556-67.
2. Vult von Steyern P, Carlson P, Nilner K. All-ceramic fixed partial dentures designed according to the DC-Zirkon technique. A 2-year clinical study. *J Oral Rehabil* 2005;32(3):180-7.
3. Raigrodski AJ, Chiche GJ, Potiket N, et al. The efficacy of posterior three-unit zirconium-oxide-based ceramic fixed partial dental prostheses: a prospective clinical pilot study. *J Prosthet Dent* 2006;96(4):237-44.
4. Sailer I, Feher A, Filser F, et al. Five-year clinical results of zirconia frameworks for posterior fixed partial dentures. *Int J Prosthodont* 2007;20(4):383-8.
5. Molin MK, Karlsson SL. Five-year clinical prospective evaluation of zirconia-based Denzir 3-unit FPDs. *Int J Prosthodont* 2008;21(3):223-7.
6. Tinschert J, Schulze KA, Natt G, et al. Clinical behavior of zirconia-based fixed partial dentures made of DC-Zirkon: 3-year results. *Int J Prosthodont* 2008;21(3):217-22.
7. Sailer I, Gottnerb J, Kanelb S, Hammerle CH. Randomized controlled clinical trial of zirconia-ceramic and metal-ceramic posterior fixed dental prostheses: a 3-year follow-up. *Int J Prosthodont* 2009;22(6):553-60.
8. Sailer I, Feher A, Filser F, et al. Prospective clinical study of zirconia posterior fixed partial dentures: 3-year follow-up. *Quintessence Int* 2006;37(9):685-93.
9. Scurria MS, Bader JD, Shugars DA. Meta-analysis of fixed partial denture survival: prostheses and abutments. *J Prosthet Dent* 1998;79(4):459-64.
10. Aboushelib MN, de Jager N, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. *Dent Mater* 2005;21(10):984-91.
11. Fischer J, Grohmann P, Stawarczyk B. Effect of zirconia surface treatments on the shear strength of zirconia/veneering ceramic composites. *Dent Mater J* 2008;27(3):448-54.

12. Al-Dohan HM, Yaman P, Dennison JB, Razzoog ME, Lang BR. Shear strength of core-veneer interface in bi-layered ceramics. *J Prosthet Dent* 2004;91(4):349-55.
13. Anusavice KJ, ed. *Phillips' science of dental materials*. 11<sup>th</sup> ed. St.Louis: Saunders; 2003.
14. Saito A, Komine F, Blatz MB, Matsumura H. A comparison of bond strength of layered veneering porcelains to zirconia and metal. *J Prosthet Dent* 2010;104(4):247-57.
15. Fischer J, Stawarczyk B, Tomic M, Strub JR, Hammerle CH. Effect of thermal misfit between different veneering ceramics and zirconia frameworks on in vitro fracture load of single crowns. *Dent Mater J* 2007;26(6):766-72.
16. Guazzato M, Walton TR, Franklin W, et al.. Influence of thickness and cooling rate on development of spontaneous cracks in porcelain/zirconia structures. *Aust Dent J* 2010;55(3):306-10.
17. Komine F, Saito A, Kobayashi K, et al.. Effect of cooling rate on shear bond strength of veneering porcelain to a zirconia ceramic material. *J Oral Sci* 2010;52(4):647-52.
18. Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Pt 2. Zirconia veneering ceramics. *Dent Mater* 2006;22(9):857-63.
19. Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Pt 2. Zirconia-based dental ceramics. *Dent Mater* 2004;20(5):449-56.
20. Doi M, Yoshida K, Atsuta M, Sawase T. Influence of pre-treatments on flexural strength of zirconia and debonding crack-initiation strength of veneered zirconia. *J Adhes Dent* 2011;13(1):79-84.
21. de Kler M, de Jager N, Meegdes M, van der Zel JM. Influence of thermal expansion mismatch and fatigue loading on phase changes in porcelain veneered Y-TZP zirconia discs. *J Oral Rehabil* 2007;34(11):841-7.
22. Kim HJ, Lim HP, Park YJ, Vang MS. Effect of zirconia surface treatments on the shear bond strength of veneering ceramic. *J Prosthet Dent* 2011;105(5):315-22.
23. Mosharraf R, Rismanchian M, Savabi O, Ashtiani AH. Influence of surface modification techniques on shear bond strength between different zirconia cores and veneering ceramics. *J Adv Prosthodont* 2011;3(4):221-8.

24. Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Effect of zirconia type on its bond strength with different veneer ceramics. *J Prosthodont* 2008;17(5):401-8.
25. Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. *Dent Mater* 1999;15(6):426-33.
26. Conrad HJ, Seong WJ, Pesun IJ. Current ceramic materials and systems with clinical recommendations: a systematic review. *J Prosthet Dent* 2007;98(5):389-404.
27. Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. *Biomater* 1999;20(1):1-25.
28. Jeong SM, Ludwig K, Kern M. Investigation of the fracture resistance of three types of zirconia posts in all-ceramic post-and-core restorations. *Int J Prosthodont* 2002;15(2):154-8.
29. Heydecke G, Butz F, Hussein A, Strub JR. Fracture strength after dynamic loading of endodontically treated teeth restored with different post-and-core systems. *J Prosthet Dent* 2002;87(4):438-45.
30. Filser F, Kocher P, Weibel F, et al. Reliability and strength of all-ceramic dental restorations fabricated by direct ceramic machining (DCM). *Int J Comput Dent* 2001;4(2):89-106.
31. Besimo CE, Spielmann HP, Rohner HP. Computer-assisted generation of all-ceramic crowns and fixed partial dentures. *Int J Comput Dent* 2001;4(4):243-62.
32. Suttor D, Bunke K, Hoescheler S, Hauptmann H, Hertlein G. LAVA--the system for all-ceramic ZrO<sub>2</sub> crown and bridge frameworks. *Int J Comput Dent* 2001;4(3):195-206.
33. Raigrodski AJ. Contemporary materials and technologies for all-ceramic fixed partial dentures: a review of the literature. *J Prosthet Dent* 2004;92(6):557-62.
34. Moffa JP, Guckes AD, Okawa MT, Lilly GE. An evaluation of nonprecious alloys for use with porcelain veneers. Pt 2. Industrial safety and biocompatibility. *J Prosthet Dent* 1973;30(4 Pt 1):432-41.
35. Purt R. Palladium ceramic alloys: possible health hazards? *Quintessence Dent Technol* 1987;11(1):35-41.

36. Hansen PA, West LA. Allergic reaction following insertion of a Pd-Cu-Au fixed partial denture: a clinical report. *J Prosthodont* 1997;6(2):144-8.
37. Miyazaki T, Hotta Y. CAD/CAM systems available for the fabrication of crown and bridge restorations. *Aust Dent J* 2011;56 Suppl 1:97-106.
38. Duret F, Preston JD. CAD/CAM imaging in dentistry. *Curr Opin Dent* 1991;1(2):150-4.
39. Mormann WH, Brandestini M, Lutz F, Barbakow F. Chairside computer-aided direct ceramic inlays. *Quintessence Int* 1989;20(5):329-39.
40. Reiss B, Walther W. Clinical long-term results and 10-year Kaplan-Meier analysis of Cerec restorations. *Int J Comput Dent* 2000;3(1):9-23.
41. Nakamura T, Dei N, Kojima T, Wakabayashi K. Marginal and internal fit of Cerec 3 CAD/CAM all-ceramic crowns. *Int J Prosthodont* 2003;16(3):244-8.
42. Persson M, Andersson M, Bergman B. The accuracy of a high-precision digitizer for CAD/CAM of crowns. *J Prosthet Dent* 1995;74(3):223-9.
43. Tomita S, Shin-Ya A, Gomi H, et al.. Machining accuracy of CAD/CAM ceramic crowns fabricated with repeated machining using the same diamond bur. *Dent Mater J* 2005;24(1):123-33.
44. Andersson M, Carlsson L, Persson M, Bergman B. Accuracy of machine milling and spark erosion with a CAD/CAM system. *J Prosthet Dent* 1996;76(2):187-93.
45. Andersson M, Oden A. A new all-ceramic crown. A dense-sintered, high-purity alumina coping with porcelain. *Acta Odontol Scand* 1993;51(1):59-64.
46. Oden A, Andersson M, Krystek-Ondracek I, Magnusson D. Five-year clinical evaluation of Procera AllCeram crowns. *J Prosthet Dent* 1998;80(4):450-6.
47. Sundh A, Sjogren G. Fracture resistance of all-ceramic zirconia bridges with differing phase stabilizers and quality of sintering. *Dent Mater* 2006;22(8):778-84.
48. Kohorst P, Herzog TJ, Borchers L, Stiesch-Scholz M. Load-bearing capacity of all-ceramic posterior four-unit fixed partial dentures with different zirconia frameworks. *Eur J Oral Sci* 2007;115(2):161-6.

49. Piwowarczyk A, Ottl P, Lauer HC, Kuretzky T. A clinical report and overview of scientific studies and clinical procedures conducted on the 3M ESPE Lava All-Ceramic System. *J Prosthodont* 2005;14(1):39-45.
50. Tinschert J, Natt G, Mautsch W, Spiekermann H, Anusavice KJ. Marginal fit of alumina-and zirconia-based fixed partial dentures produced by a CAD/CAM system. *Oper Dent* 2001;26(4):367-74.
51. Valderhaug J. A 15-year clinical evaluation of fixed prosthodontics. *Acta Odontol Scand* 1991;49(1):35-40.
52. Leempoel PJ, Kayser AF, Van Rossum GM, De Haan AF. The survival rate of bridges. A study of 1674 bridges in 40 Dutch general practices. *J Oral Rehabil* 1995;22(5):327-30.
53. Karlsson S. A clinical evaluation of fixed bridges, 10 years following insertion. *J Oral Rehabil* 1986;13(5):423-32.
54. Palmqvist S, Swartz B. Artificial crowns and fixed partial dentures 18 to 23 years after placement. *Int J Prosthodont* 1993;6(3):279-85.
55. Karlsson S. Failures and length of service in fixed prosthodontics after long-term function. A longitudinal clinical study. *Swed Dent J* 1989;13(5):185-92.
56. Creugers NH, Kayser AF, van't Hof MA. A meta-analysis of durability data on conventional fixed bridges. *Community Dent Oral Epidemiol* 1994;22(6):448-52.
57. Guazzato M, Quach L, Albakry M, Swain MV. Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic. *J Dent* 2005;33(1):9-18.
58. Craig RG, Power JM, Wataha JC, eds. *Dental materials*. St. Louis: Mosby; 2004.
59. 9693 I. Metal-ceramic bond characterization (Schwickerath crack initiation test). Geneva, Switzerland: International Organization for Standardization; 1999.
60. Albakry M, Guazzato M, Swain MV. Fracture toughness and hardness evaluation of three pressable all-ceramic dental materials. *J Dent* 2003;31(3):181-8.

61. Blatz MB, Bergler M, Ozer F, et al. Bond strength of different veneering ceramics to zirconia and their susceptibility to thermocycling. *Am J Dent* 2010;23(4):213-6.
62. Shell JS, Nielsen JP. Study of the bond between gold alloys and porcelain. *J Dent Res* 1962;41:1424-37.
63. Nielsen JP, Tuccillo JJ. Calculation of interfacial stress in dental porcelain bonded to gold alloy substrate. *J Dent Res* 1972;51(4):1043-7.
64. Swain MV. Unstable cracking (chipping) of veneering porcelain on all-ceramic dental crowns and fixed partial dentures. *Acta Biomater* 2009;5(5):1668-77.
65. Denry I. How and when does fabrication damage adversely affect the clinical performance of ceramic restorations? *Dent Mater* 2012.
66. Mainjot AK, Schajer GS, Vanheusden AJ, Sadoun MJ. Influence of veneer thickness on residual stress profile in veneering ceramic: measurement by hole-drilling. *Dent Mater* 2012;28(2):160-7.
67. Guazzato M, Proos K, Sara G, Swain MV. Strength, reliability, and mode of fracture of bilayered porcelain/core ceramics. *Int J Prosthodont* 2004;17(2):142-9.
68. Thompson GA. Influence of relative layer height and testing method on the failure mode and origin in a bilayered dental ceramic composite. *Dent Mater* 2000;16(4):235-43.
69. Zeng K, Oden A, Rowcliffe D. Evaluation of mechanical properties of dental ceramic core materials in combination with porcelains. *Int J Prosthodont* 1998;11(2):183-9.
70. Kern M, Barloi A, Yang B. Surface conditioning influences zirconia ceramic bonding. *J Dent Res* 2009;88(9):817-22.
71. Chevalier J. What future for zirconia as a biomaterial? *Biomater* 2006;27(4):535-43.
72. Zahnfabrik V. Veneering material VitaVM9. Working instructions. Vita Zahnfabrik, Bad Sachingen; 2007.
73. Denry IL, Peacock JJ, Holloway JA. Effect of heat treatment after accelerated aging on phase transformation in 3Y-TZP. *J Biomed Mater Res B Appl Biomater* 2010;93(1):236-43.



74. Guazzato M, Albakry M, Quach L, Swain MV. Influence of surface and heat treatments on the flexural strength of a glass-infiltrated alumina/zirconia-reinforced dental ceramic. *Dent Mater* 2005;21(5):454-63.
75. Harding AB, Norling BK, Teixeira EC. The effect of surface treatment of the interfacial surface on fatigue-related microtensile bond strength of milled zirconia to veneering porcelain. *J Prosthodont* 2012;21(5):346-52.
76. Bona AD, Anusavice KJ, DeHoff PH. Weibull analysis and flexural strength of hot-pressed core and veneered ceramic structures. *Dent Mater* 2003;19(7):662-9.
77. Kim BK HJ, Han KR. . Quantitative phase analysis in tetragonal-rich tetragonal / monoclinic two phase zirconia by Raman spectroscopy. *J Mater Sci Lett* 1997;16:669-71.
78. Gonzaga CC, Cesar PF, Miranda WG, Jr., Yoshimura HN. Slow crack growth and reliability of dental ceramics. *Dent Mater* 2011;27(4):394-406.
79. Lohbauer U, Kramer N, Petschelt A, Frankenberger R. Correlation of in vitro fatigue data and in vivo clinical performance of a glassceramic material. *Dent Mater* 2008;24(1):39-44.

ABSTRACT

INFLUENCE OF SURFACE TREATMENT ON VENEERING  
PORCELAIN SHEAR BOND STRENGTH TO ZIRCONIA  
AFTER CYCLIC LOADING

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Statement of problem: Yttria-partially stabilized tetragonal zirconia polycrystal (Y-TZP) all-ceramic restorations have been reported to suffer from chipping or cracking of the veneering porcelain (VP) as the most common complication. There is little information in the literature regarding the influence of surface treatment on VP shear bond strength to Y-TZP after cyclic loading. Purpose of this study: The goals of this study were 1) To investigate the influence of zirconia surface treatments on veneering porcelain shear bond strength, and 2) to investigate the influence of cyclic loading on the shear bond strength between VP and Y-TZP. Materials and Methods: 48 cylinder-shaped specimens (6 mm in diameter and 4 mm in height) were divided into four groups containing 12 specimens each according to the surface treatment. As a control group (C), no further treatment was applied to the specimens after grinding. Group H was heat-

treated as a pretreatment according to the manufacturer's recommendations. Group S was airborne-particle abraded with 50- $\mu\text{m}$  alumina ( $\text{Al}_2\text{O}_3$ ) particles under a pressure of 0.4 MPa for 10 seconds. In the group SH, the heat-treatment was performed after the airborne-particle abrasion. A VP cylinder (2.4 mm in diameter and 2 mm in height) was applied and fired on the prepared Y-TZP specimens. The shear bond strength was tested using a universal testing machine. Six specimens from each group were subjected to fatigue (10,000 cycles, 1.5Hz, 10N load) before testing. Results: The three-way ANOVA showed no statistically significant effect of surface treatment and cyclic loading on shear bond strength. The highest mean shear bond strength was recorded for the air-particle abrasion group without cyclic loading ( $34.1 \pm 10$  MPa). The lowest mean shear bond strength was the air-particle abrasion group with cyclic loading ( $10.7 \pm 15.4$  MPa). Sidak multiple comparisons procedure demonstrated cyclic loading specimens had significantly lower shear bond strength than non-cyclic loading specimens after air-particle abrasion without heat treatment ( $p = 0.0126$ ) Conclusion: Within the limitations of this study 1) Shear bond strength between Y-TZP and VP is not affected statistically by surface treatment using heat treatment, airborne-particle abrasion, and heat treatment after airborne-particle abrasion; 2) There is a significant difference in shear bond strength with air-particle abrasion between with and without cyclic loading groups. This difference suggested that air-particle abrasion should be avoided in clinical situations as a surface treatment without heat treatment.

## CURRICULUM VITAE

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