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# The Effect of Preceramic Soldering on Fracture Resistance of 4-Unit Zirconia Fixed Dental Prostheses

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**Keywords**
Zirconia; fixed dental prostheses; FDPs; preceramic soldering; fracture resistance; monolithic zirconia.

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## Abstract

**Purpose:** Preceramic soldering of zirconia may deliver better fitting restorations. However, there is not sufficient evidence regarding the influence of preceramic soldering of zirconia restorations on mechanical strength. The aim of this study was to evaluate the effect of preceramic soldering on the fracture load of 4-unit zirconia fixed dental prostheses (FDPs).
**Materials and methods:** Eighty samples of 4-unit FDPs between maxillary right first premolar and maxillary right second molar were prepared and two restorative materials were used as a framework (Z) and monolithic restoration (M). The samples were divided into two subgroups as control (C) and study (S). The restorations of study groups (S) were divided into two pieces and soldered with a bonding material (DCM HotBond Zirkon). The groups were divided into two subgroups for thermal cycle (T) application. After soldering and thermal cycling application, 4-point bending test was applied to the samples at a cross-head speed of 1 mm/min in a universal testing machine and the fracture load was recorded. The data was analyzed statistically, and the level of significance was set at $\alpha = 0.001$.
**Results:** Statistically significant differences were found among the groups, based on the results of maximum failure loads ($p < 0.001$). The highest mean failure load was observed in the ZCT(-) group (1094.1 + 139.77 N), while the lowest mean failure load was obtained in the ZST(+) group (627.7 + 82.14 N). No significant difference was found among the groups MC and MS, MC, and ZC groups ($p > 0.001$). Thermal aging application caused lower fracture resistance in control and soldering groups ($p < 0.001$).
**Conclusions:** The preceramic soldering applications affected zirconia group negatively. However, the values were above the clinically acceptable static load bearing capacity for posterior teeth. Soldering did not cause a statistically significant difference for the fracture strengths of monolithic zirconia groups. Thermal cycling affected the fracture strength of zirconia and monolithic zirconia restorations negatively.

The increase in esthetic expectations and technological developments in dentistry have enabled the use of different materials that are biologically compatible, esthetically acceptable, and have superior mechanical properties as an alternative to metal-supported ceramic restorations.[1–3] Zirconia has been used as a substructure material for fixed prosthetic restorations due to its high mechanical strength, biocompatibility, and color.[1–3]

Zirconia is a well-known polymorphic, metastable material that exists in three crystallographic forms: monoclinic (stable at room temperature up to 1170°C), tetragonal (stable at 1170°C to 2370°C), and cubic (stable over 2370°C to the melting point 2716°C). While cooling zirconia to room temperature, a phase transformation from tetragonal to monoclinic phase occurs which produces a volume expansion of 3% to 5%; this causes expansion stresses that can lead to the failure of zirconia.[1–3] Addition of stabilizing oxides such as CaO, MgO, and $Y_2O_3$ to the pure zirconia can stabilize it in the tetragonal phase at room temperature. $Y_2O_3$ is the most frequently used stabilizing oxide in the dental field. Yttria stabilized tetragonal zirconia polycrystals (Y-TZP) contain 2 to 5 mol % $Y_2O_3$

that is also known as partially stabilized zirconia (PSZ) due to the percentage of yttrium oxide in the composition. Y-TZP is metastable in nature and the tetragonal particles can transform to the monoclinic form spontaneously and are larger in size. This phase transformation, which is also called transformation toughening, induces compressive stresses around the crack tips and prevents its propagation by increasing its localized fracture toughness. However, a variety of factors including mechanical, physical, thermal, and chemical stimuli may increase the *t-m* phase transformation leading to a volume increase of 3% to 5% which deleteriously affects the mechanical properties known as low-temperature degradation (LTD).[1–3]

The advantages of zirconia restorations such as mechanical strength, reduced material thickness, acceptable esthetic result, and reduction in production time and cost make the material widely used in dental applications.[4] The high strength zirconia substructures prepared with computer-aided design and computer-aided manufacturing (CAD-CAM) technology can be veneered with feldspathic porcelain in order to mimic the natural teeth and overcome the main disadvantage of zirconia which is its white, opaque appearance.[3,5] However, the fit of restorations milled from presintered blocks might be affected by the sintering shrinkage and production process. The distortion of multi-unit fixed dental prostheses (FDPs) due to the sintering shrinkage can affect the accuracy of fit of restorations on natural teeth or implant abutments.[1] Poorly fitting restorations are associated with biological and mechanical problems both for the tooth-supported or implant-supported restorations.[6,7] For this aspect, preceramic soldering might help to improve the fit accuracy of zirconia restorations by reducing distortion which has been used for metal cast alloys for years. In soldering alloys, an intermediate alloy or solder is employed to unite the parts to be joined.[8] However, very limited data is available regarding the influence of preceramic soldering of zirconia restorations on mechanical strength.[9] Wimmer et al[9] investigated the load bearing capacity of soldered zirconia FDPs and concluded that soldered zirconia frameworks showed similar load bearing capacity as nonsoldered frameworks and soldering had no influence on chipping of zirconia FDPs. However, there is no study evaluating the fracture resistance of preceramic soldering of zirconia frameworks and monolithic zirconia FDPs.

The aim of this in vitro study was to evaluate the effect of preceramic soldering on the fracture load of 4-unit zirconia FDOs. The null hypothesis was that the preceramic soldering procedure would not affect the mechanical strength of zirconia.

## Materials and methods

In this study, two different restorative materials were used as a framework (Prettau Zirconia, ZirconZahn SRL) and monolithic restoration (ICE Zircon Ceramics, ZirconZahn SRL) in order to evaluate the fracture resistance of 4-unit FDPs between maxillary right first premolar and maxillary right second molar teeth soldered with a bonding material (DCM HotBond Zirkon).

Preprepared typodont teeth were used as abutments (Frasaco GmbH), they were scanned with an optical scanner (Scan In a Box, Open Technologies), and the teeth were milled from

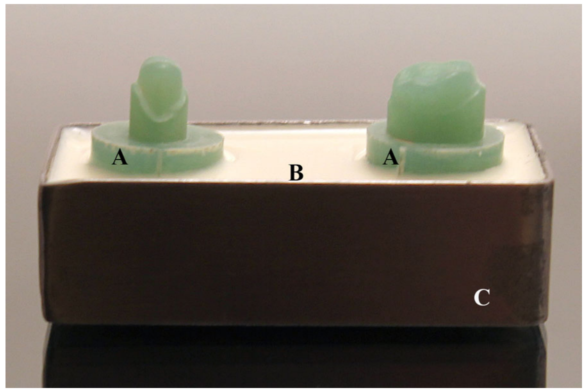


**Figure 1** Epoxy master model. (A) Glass infiltrated epoxy resin teeth; (B) polyurethane resin; and (C) metal mold.

the glass infiltrated epoxy resin material[10,11] (G-10 / FR-4, Plastform Kimya) on a CNC pantograph device (Best Marlow BWM 6050, Supertech). A metal mold was prepared in 40 × 15 × 20 mm dimensions to form a master model and the two abutment teeth were fixed in the mold, parallel to each other at a distance of 25 mm between the occlusal midpoints. The mold was filled with polyurethane resin[12–15] (Polyurock, Cendres +Métaux SA) (Fig 1). A total number of 80 study models were prepared.

The epoxy master model was scanned with an optical scanner (DentalWings7, Dental Wings Inc.) and an anatomically supported zirconia framework was designed. The first group was designed as zirconia framework (Z) while the second group was designed as monolithic zirconia FDP (M). Axial and occlusal thicknesses of framework restorations were designed as 0.8 mm and 13.2 mm$^2$ for connector areas. In the monolithic design, axial and occlusal thicknesses were 1.8 mm, connector areas were designed as 16.8 mm$^2$. In both designs, marginal margin spacing was determined as 20 $\mu$m and internal spacing as 50 $\mu$m. In the restoration design, 4 mm diameter slots were prepared on the occlusal surfaces of the first premolar and the second molar (Fig 2). Before the manufacturing of the FDPs, a sample size of n = 10 was deemed adequate by conducting a power analysis. A total of 40 frameworks were prepared from a zirconia block (Prettau Zirconia, ZirconZahn SRL) and 40 monolithic restorations were prepared from a monolithic zirconia block (ICE Zirkon Ceramics, ZirconZahn SRL) (Fig 2). The prepared restorations were sintered according to the manufacturer's instructions.

The specimens in each group were randomly divided into two subgroups as control (ZC, MC) and preceramic soldering groups (ZS, MS). Specimens of the preceramic soldering group were separated into two pieces under water cooling by means of a separator with 1mm thickness at the connection area between the second premolar and the first molar. The specimen was placed on its epoxy model and the model was stabilized on a high precision cutting machine (PIL0302L-150F, Guangdong Zhengye Technology Co. Ltd, China). The preceramic soldering space was equal to the thickness of the fiber cutting separator. The cutting surfaces were sandblasted with 50 $\mu$m aluminum oxide sand for 10 seconds from 10 mm distance

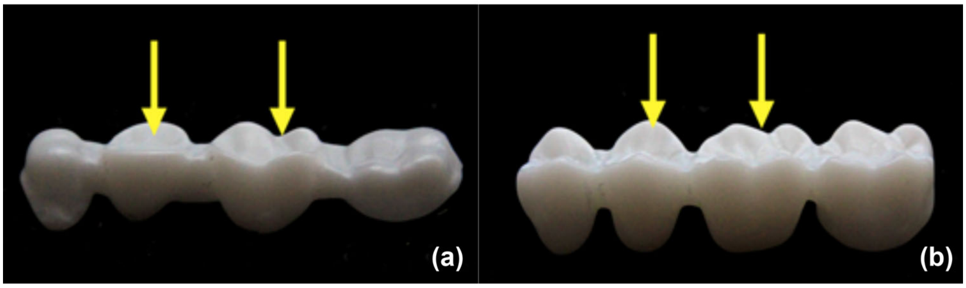


**Figure 2** Specimens of the groups (yellow arrows show the spots created). (a) 4-unit zirconia framework and (b) 4-unit monolithic zirconia restoration.

**Figure 3** Bonding material application on master model.

under 2 bar pressure. The fixed positions of the restorations on the abutment teeth were recorded with a silicone key.

Solder material was mixed in a 1:1 ratio at room temperature until cream consistency and extra attention was paid to eliminate air bubbles in the structure. The mixture was applied to the cutting surfaces with the help of brush # 12 (Pebeo 110 Series No:12; Pebeo France Art). Each restoration was placed on its own model with a silicone key. Additional soldering material was placed in between two pieces. Soldering material was dried by hot air through a drying machine (AC8002, Remington) for 30 seconds at a distance of 15cm from the buccal, lingual, and occlusal surfaces. Following a chalky appearance, the restoration was removed from the model (Fig 3). The fixing material (DCM HotBond Fix) was filled inside the anchors and moved to the oven with the restoration carrier in order to ensure that it remained stable on the carriers in the oven. The restoration with the soldering material was sintered at 450 to 1000°C in accordance with the manufacturer's instructions. After sintering, the restorations were left to be cooled at room temperature. Excess soldering material was abraded with diamond bur under water cooling.

Following the soldering process, subgroups were divided into two groups for thermal aging (n = 10) (5°C to 55°C; 1 minute each cycle) for 5000 cycles. Groups that were treated with thermal aging are shown as T(+) and without thermal aging are shown as T(-). Veneering simulation was applied to all of the specimens. Ceramic layering simulations were performed twice for zirconia group specimens. For ceramic layering simulation, the specimens were kept in a vacuum at 500°C for 6 minutes; the heating temperature was set at 55°C per minute and elevated up to 910°C. At this temperature, a 1-minute holding time was applied, and the specimens were left to return to room temperature. After the ceramic layering simulation of the zirconia group specimens, polishing simulation was performed. The specimens were heated in the oven to 900°C with a temperature increase of 80°C per minute, after which they were left to return to room temperature. For the monolithic zirconia group specimens, only the polishing simulation was applied as in the zirconia group.

The 4-unit zirconia frameworks and monolithic zirconia restorations were loaded at a cross-head speed of 1 mm/min with two balls placed on the centers of the pontics that was premarked in a universal testing machine (AVK) and the fracture load was recorded. The loading points of the four-point bending setup were designed so that they were adjustable in the vertical axis. Prior to each test, simultaneous contact and even loading were assured by precise adjustments. In order to prevent force peak and to achieve homogeneous load distribution on the pontics, a piece of teflon foil was placed between the ball and the pontic surface.

Data sets were analyzed with statistical software SPSS for Windows 21.0 (Statistical Package for Social Sciences: IBM Inc.). Three-way ANOVA was performed to examine the effects of material, group, and thermal application on the fracture load values. Post hoc analyzes were performed in order to examine the bilateral interactions with significant effects in more detail (Table 1). The level of significance was set at $\alpha = 0.001$.

The area of fracture was noted for each specimen. The fracture areas were coded with numbers 1 to 3 and defined as fracture between the first premolar and the second premolar = 1, fracture at the connector area = 2, and fracture between the first molar and second molar = 3.

## Results

According to the results obtained from the three-way ANOVA tests, the statistical model obtained as a result of the analysis was found to be statistically significant and the R-square level of the model was 0.722 [F = 26,663; $p < 0.001$; $R^2 = 0.722$]. In the model, it was determined that the material * group * thermal triple interaction was not significant, so the binary

**Table 1** Fracture loads values of the groups. (Newton)

| | | Groups | | |
|---|---|---|---|---|
| | | Control (C) Mean ±SD | Soldering (S) Mean ±SD | p |
| Materials | Zirconia (Z) | 968.70 ±164.62 | 672.95 ±84.37 | **<0.001*** |
| | Monolithic zirconia (M) | 900.25 ±153.41 | 867.10±111.00 | **0.439** |
| | **p** | **0.182** | **<0.001*** | |
| Thermal aging | T(-) | 1049.25 ±143.42 | 831.35 ±138.82 | **<0.001*** |
| | T(+) | 819.70 ±69.26 | 708.70 ±109.96 | **0.001*** |
| | **p** | **<0.001*** | **0.004*** | |

Z = zirconia group; M = monolithic zirconia group; C = control group; S = study group; T(-) = no thermal aging application, T(+) = thermal aging application.

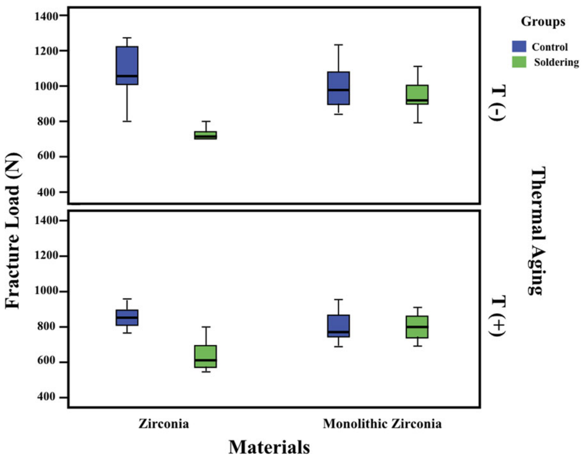


**Figure 4** Box plot graphs of the fracture loads of the groups. Fracture load values before thermal cycling application (top figure) were higher than after thermal cycling application (bottom figure).

interactions were taken into consideration. Accordingly, the effect of material *group and group* thermal app binary interactions was found to be significant, while the effect of material*thermal app was not statistically significant ($p < 0.001$, $p = 0.013$, $p = 0.797$, respectively). Post-hoc analyses were performed in order to examine the bilateral interactions with significant effects in more detail (Table 1).

According to post hoc evaluation results, there was no significant difference between the materials in the control group in terms of failure loads; the failure load of monolithic zirconia in the soldering group was greater than zirconia ($p = 0.182$, $p < 0.001$, respectively). The failure load of the soldering group was lower in the zirconia groups than the control group, and no significant difference was found between the control and soldering groups for monolithic zirconia ($p < 0.001$, $p = 0.439$, respectively). In addition, the failure load values obtained in the T(-) subgroups in the control and soldering groups were greater than the failure load values obtained in the T(+) subgroups ($p < 0.001$, $p = 0.004$, respectively). The failure load values of the soldering group in the T(-) and T(+) groups were lower than the values of the control group ($p < 0.001$, $p = 0.001$, respectively) (Table 1) (Fig 4).

The fracture area analysis is shown in Table 2. In the zirconia framework groups, many of the specimens showed fracture at the connector area (2) and between second premolar and second molar (3). In the zirconia framework control groups (ZC), 3 out of 20 specimens showed fractures that did not contain a connector area (2) fracture (Fig 5). In the zirconia framework preceramic soldered groups (ZS), only one out of 20 specimens showed fractures that did not contain a connector area (2) fracture. However, in the monolithic zirconia groups (MC and MS), all of the specimens were fractured from the connector area. When the preceramic soldering groups (ZS and MS) were evaluated, 19 out of 20 specimens showed fracture at the solder material. Only one specimen from the ZST(+) group showed a fracture of the first premolar.

## Discussion

The aim of this in vitro study was to evaluate the effect of preceramic soldering on the fracture load of 4-unit FDPs. Based on the results, the null hypothesis was partially rejected as the soldering procedure affected the mechanical strength of the zirconia group but there was no statistically significant difference with the monolithic zirconia group after soldering.

The data regarding the effect of preceramic soldering on the mechanical strength of zirconia and monolithic zirconia is limited in the literature.[9] In a previous study, preceramic soldering was applied to zirconia FDPs and load bearing capacity of the FDPs were evaluated.[9] In that study, a steel model with two abutments (first right canine and first right molar) were prepared in and 4-unit FDPs were veneered after soldering. However, in the current study, glass infiltrated epoxy resin teeth that were embedded in rigid polyurethane resin (PUR) material were preferred due to its superiority in mimicking the mechanical properties of teeth and jaw bone against high forces and veneering simulation was applied on the zirconia frameworks. Wimmer et al[9] concluded that preceramic soldering of zirconia frameworks resulted in similar load bearing capacity compared to nonsoldered frameworks. The values in their study were found to be relatively higher compared to the values of the present study. The difference between the two studies can be attributed to the difference in the study designs.

**Table 2** Fracture area analysis within groups

| | Failure area | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Group Z | | | | Group M | | | |
| | ZC | | ZS | | MC | | MS | |
| Specimen | ZCT(-) | ZCT(+) | ZST(-) | ZST(+) | MCT(-) | MCT(+) | MST(-) | MST(+) |
| 1 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| 2 | 2 | 2 | 2 | 2 + 3 | 2 | 2 | 2 | 2 |
| 3 | 3 | 2 | 2 + 3 | 2 +3 | 2 | 2 | 2 | 2 |
| 4 | 2 | 2 + 3 | 2 | 2 + 3 | 2 | 2 | 2 | 2 |
| 5 | 2 + 3 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| 6 | 2 | 2 + 3 | 2 + 3 | 2 + 3 | 2 | 2 | 2 | 2 |
| 7 | 2 + 3 | 1 + 2 | 2 | 2 + 3 | 2 | 2 | 2 | 2 |
| 8 | 2 + 3 | 2 + 3 | 2 + 3 | 2 | 2 | 2 | 2 | 2 |
| 9 | 2 + 3 | 2 | 2 | 2 + 3 | 2 | 2 | 2 | 2 |
| 10 | 2 + 3 | 1 + 3 | 2 | 1 | 2 | 2 | 2 | 2 |

(Between the first premolar and the second premolar = 1, Connector area = 2, Between the first molar and second molar = 3)
Z = zirconia group; M = monolithic zirconia group; C = control group; S = study group; T(-) = no thermal aging application, T(+) = thermal aging application.

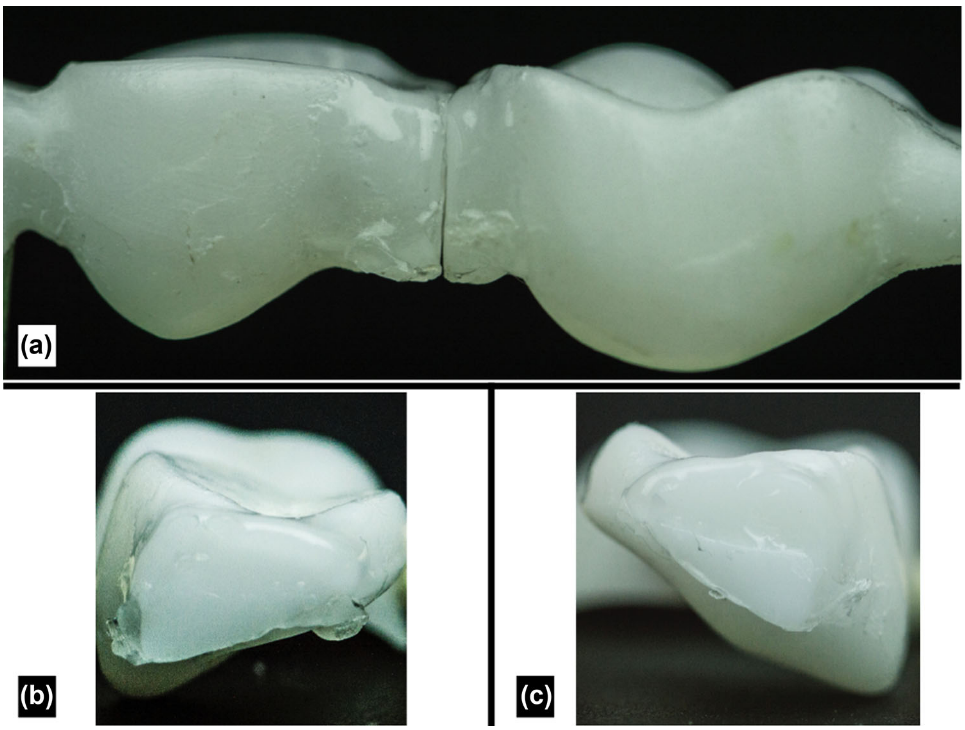


**Figure 5** Fracture surface of zirconia framework study group after thermal cycling aging specimen. Preceramic solder material showes glassy appearance and zirconia surface shows matte appearance. (a) Front view of the connector area with preceramic soldering (2). Notice the fracture line is in the soldering area. (b) Mesial surface of the fracture area. Glassy appearance is noticeable on the entire fracture surface. (c) Distal surface of the fracture area. Glassy appearance is noticeable on the entire fracture surface.

The fracture strength of all-ceramic restorations is affected by the framework material. Ideally, the underlying material should exhibit elastic behavior similar to dentine. In previous studies, the fracture strength values of the ceramics displayed higher values in the materials with higher hardness than dentin.[10,11] It has been reported that the elastic behavior and resin cement bond strength of G-10 epoxy resin is similar to hydrated dentin and has an elastic modulus (18.6 GPa) and plastic deformation similar to that of dentin (15-19 GPa).[16,17] Therefore, the use of G-10 epoxy resin can be preferred in fracture resistance studies in order to simulate the intraoral situation better.

Rigid polyurethane resin (PUR) is known as a popular test material for the evaluation of orthopedic devices and training of orthopedic surgeons. The mechanical properties of the polyurethane resin that are similar to the jaw bone have made it used in many studies evaluating the stress analysis between the implant and the jaw bone.[12–15] Therefore, glass infiltrated epoxy resin teeth were preferred in the current study so as to represent teeth according to the data in the literature that were embedded in rigid PUR used to imitate the maxillae.

Liquid environments facilitate subcritical crack growth in ceramics and cause uncontrolled transitions of Y-TZP from tetragonal to monoclinic structure.[18–21] Palmer et al[22] reported that a temperature range of 0 to 67°C occurred for food and drink, resulting in temperature changes in the teeth between 5 and 55°C. Therefore, the stress caused by thermal fatigue and the oral environment corresponded to approximately 6 months of use of the thermal cycle applied to the specimens in the present study. According to the results (Table 1), the fracture resistance values of all groups decreased, and all subgroups (ZC, ZS, MC, and MS) showed statistically significant difference after thermal cycling ($p < 0.05$). However, the results were found above the acceptable load bearing capacity for the posterior teeth. The results of the current study are supported by other studies in the literature.[23,24]

Sundh et al reported that heat treatment, in addition to veneering, also adversely affects the fracture resistance.[25] In other reports veneering was reported to increase the fracture resistance.[26] However, this effect varies according to which type of zirconia is used.[26] In the current study, the force required to fracture the superstructure ceramic of the restorations was calculated. Therefore, superstructure ceramics have not been processed on 4-unit FDPs prepared from zirconia framework. In addition, superstructure ceramics were simulated in zirconia restorations and veneering was imitated.

Studies have reported that clinical failures of all-ceramic FDPs almost always occur in the connector area.[27,28] The results of the present study are parallel with those studies. When the fracture area analysis results were evaluated, 76 out of 80 specimens showed fracture at the connector area. In addition, in most of the groups, fracture resistance of monolithic zirconia subgroups was higher than zirconia subgroups. This may be related to the fact that the connection areas of monolithic zirconia FDPs are wider than the zirconia group. Accordingly, the increase of the connection area may have positively affected the increase of the area to be soldered and therefore the fracture strength of the zirconia restorations.[29] In that study, it was reported that the connection height increased from 3 mm to 4 mm and that it reduced the stress in this area by 50%.[29]

The fracture resistance values in the study groups (ZS and MS) were lower than the control groups (ZC and MC) in all subgroups. However, the fracture resistance values were 25.4% to 118% higher than the criteria of 500 N which is the lower limit of the clinically acceptable static load carrying capacity for FDPs in the posterior region. [26]

The fracture area analysis showed that in the zirconia framework group, the number of specimens that showed fracture originating from the connector area (soldered area) increased after soldering and thermal cycling caused aging in the frameworks as the frameworks showed more multiple fracture areas (2 + 3) after thermal aging applications. However, in the monolithic zirconia group, all the specimens were fractured from the connector area. In the study groups (ZS and MS), 39 out of 40 specimens showed fracture at the connector area (2) (Table 2), which is also the area where the preceramic soldering was applied. When the fracture surfaces of those 39 specimens were examined, it was found that there was solder on both surfaces of the fracture and they were cohesive fractures formed in the solder material (Fig 5).

There are some limitations of the present study. It is hard to simulate the oral situation and the dynamic system in an in vitro study. The fracture resistance test that was applied in the current study can be considered as static loading. The thermal aging of the study can be considered equal to 6 months of use. For this reason, long-term aging experiments are needed. In addition, veneering simulation was applied to the zirconia framework group instead of veneering. Chipping of the veneering ceramics and how the solder material supports the veneering ceramics were not evaluated. The study should be supported by further studies with dynamic loading tests and in vivo studies.

## Conclusions

Preceramic soldering applications affected zirconia frameworks with 4 units negatively. However, the values were above the clinically acceptable static load bearing capacity for posterior teeth. Soldering did not produce a statistically significant difference for the fracture strengths of monolithic zirconia groups. Thermal cycle application negatively affects the fracture strength of zirconia and monolithic zirconia restorations.

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