Impact of multiple firings and resin cement type on shear bond strength between zirconia and resin cements

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PURPOSE. The aim of this study was to evaluate the effect of multiple firings on the bond strength between yttria-tetragonal zirconia polycrystals (Y-TZP) and 2 types of resin cements. **MATERIALS AND METHODS.** Sixty 3Y-TZP specimens (LAVA Frame Multi) were divided into 3 groups depending on the following firing procedures: (1) 2-firing cycles, (2) 5-firing cycles, (3) 10-firing cycles. Two samples from each group were investigated by using SEM to determine the morphological changes. All specimens were treated with 125 µm airborne-particle abrasion and the surface roughness of each specimen was measured. The specimens from each firing group were then further divided into 2 subgroups (n = 9) to apply 2 types of resin cement (MDP-free resin cement: RelyX Unicem-RU, and MDP containing resin cement: Panavia F 2.0-PA). The shear bond strength (SBS) test was performed and failure types of all the debonded specimens were classified by using a stereomicroscope as adhesive, cohesive, and mixed. The statistical analysis of surface roughness and SBS data were performed by using 1-way ANOVA and 2-way ANOVA followed by Tukey-HSD tests (α=.05). Failure modes were calculated as a percentage for each group. **RESULTS.** The bond strength of RU and PA to the specimens obtained with 2 firings were not statistically different from each other (P=.1). However, the SBS values of PA were found to be significantly higher than RU for the specimens obtained with 5 and 10 firing cycles (P=.001 and P=.02, respectively). Surface roughness analysis revealed no statistical difference between groups (P=.2). The SEM analysis of samples fired 5- and 10- times showed irregularities and boundary loss in zirconia grains, and empty spaces between zirconia grains. **CONCLUSION.** The bond strength of PA cement was higher than that of RU to the zirconia subjected to repeated firings (5 and 10 firing cycles). When zirconia is subjected to multiple firings, using MDP-containing resin cement can be recommended. ***[J Adv Prosthodont 2020;12:197-203]***

KEYWORDS: Yttriatetragonal zirconia polycrystals (Y-TZP); Zirconia; Repated-firing; Bond strength

INTRODUCTION

In recent years, yttria partially stabilized zirconia (Y-TZP - Yttria Tetragonal Zirconia Polycrystals) has attracted great attention for clinical implementations in oral rehabilitation due to its high flexural strength, fracture toughness, good biocompatibility, and longevity.[1,2] Nevertheless, the aesthetics of zirconia is still known to be quite controversial as it has a great number of crystalline particles that block transmittance and reflection of light.[3,4] In an attempt to improve aesthetics of 3 mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP), the number and grain size of the aluminum oxide (Al_2O_3) grains are reduced[5]; however, the improvements in optical properties are inadequate.[6] More recently, 4Y-and 5Y-TZP are introduced. The larger grains and increased content of yttrium oxide (Y_2O_3) in these new generation materials improved the translucency but worsened the mechanical properties.[6,7] Nevertheless, 3Y-TZP is still used as a stable framework material[7] and veneered with a traditional feldspathic ceramic to achieve an esthetic outcome.[8]

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In simple clinical procedures, zirconia restorations usually undergo 2 to 5 firings after sintering for veneering procedure.[9] However, multiple firings may be required to correct the occlusion, marginal adaptation, contours or color of the veneering porcelain.[2] Previous studies indicated that multiple firings have effects on the microhardness and flexural strength of zirconia;[10] mechanical properties and microstructure of veneering ceramics;[2] and the bond strength between veneering ceramic and zirconia framework.[11,12] Also, different firing procedures were reported to cause alterations in microstructural properties and surface characteristics of zirconia[9,13] and were associated with clinical performance and longevity of zirconia restorations.[14]

Another factor considered to influence the clinical performance of zirconia restorations is the bond strength between luting material and restoration.[15] Since both conventional cements and resin-based cements have been proposed for zirconia restorations, the choice of luting cement is up to the clinician.[15,16] However, resin cements are more preferred owing to their better retention and low solubility in the oral environment.[17] As zirconia is not regarded as suitable for acid etching due to the glass-free high crystalline content,[18] the adhesion is promoted with different methods, one of which is priming with functional monomers like 10-methacryloyloxydecyl dihydrogen phosphate (MDP).[19-21] The phosphate ester groups of MDP were reported to react with the hydroxyl groups of the zirconia, producing hydrothermally stable bonds between the resin cement and zirconia.[22,23] Previous studies indicated that MDP-containing resin cements provide higher bond strength to zirconia than that of MDP-free resin cements.[16,21,24] Also, different variables such as surface conditioning methods and aging procedures of zirconia were found to influence the bond strength of resin cements with regard to MDP content.[15-17,19-25] However, the existing literature in connection with the effects of multiple firings on the bond strength of different types of resin cement is lacking.

Therefore, the main purpose of this *in vitro* study was to evaluate the influence of multiple firings on the bond strength of 2 types of resin cement (MDP-containing and MDP-free) to 3Y-TZP. The null hypothesis was that multiple firings would not affect the bond strength between resin cement and zirconia regardless of resin cement type.

MATERIALS AND METHODS

CAD/CAM pre-sintered 3Y-TZP blocks (LAVA Frame Multi, 3M ESPE, Seefeld, Germany) were cut by using a high-speed diamond blade (Microcut 201, Metkon Instruments Inc., Bursa, Turkey) and a total of 60 specimens were obtained. All specimens were sintered according to the instructions given by the manufacturer (LAVA Furnace 200, 3M ESPE, Seefeld, Germany) and the final dimensions (10 mm × 10 mm × 1 mm) of the specimens were validated by a digital caliper (Digital Micrometer IP65, Mitutoyo Europe, Neuss, Germany). The specimens were divided into 3 groups of 20 specimens according to the firing procedure applied: 2 firings (the control group), 5 firings, and 10 firings. The different firing cycles were implemented in a ceramic furnace (Programat P510, Ivoclar, Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions (3M ESPE) for the veneering and glaze firings of LAVA Frame Multi (Table 1). Two randomly selected specimens from each firing group were investigated under a scanning electron microscope (SEM; JEOL JSM-6060LV, Tokyo, Japan) at a magnification of × 10,000 to detect the morphological alterations of the zirconia following the firing procedures. The study design and test groups were given in Fig. 1.

The specimens were embedded into self-curing acrylic resin blocks (Palapress Vario, Heraeus Kulzer GmbH, Hanau, Germany) and wet-finished with 1,000-grit silicon carbide paper (Gripo 2V Grinder-polisher, Metkon Instruments Inc., Bursa, Turkey) to standardize the surfaces. Then, airborne-particle abrasion was implemented perpendicularly to the surface of each specimen with 125 µm aluminum oxide at a pressure of 2.5 bar and a distance of 10 mm for 10 seconds.[26] Subsequently, all the specimens were ultrasonically cleaned in isopropyl alcohol for 5 min.

The surface roughness (Ra in µm) of the specimens obtained with 2 (control group), 5, and 10 firing cycles (n = 18) was evaluated by a profilometer (Perthometer, Mahr

Table 1. Firing procedure of Lava Frame Multi according to the manufacturer's recommendations

Firing cycle	Start temperature (°C)	Dry time (min)	t ↗ with vacuum (°C/min)	t ↗ without vacuum (°C/min)	Final temperature (°C)	Holding time with vacuum (min)	Holding time without vacuum (min)
1	450	4	45	-	840	1	-
2	450	4	45	-	830	1	-
3	450	6	45	-	810	1	-
4	450	6	45	-	800	1	-
5	480	2	-	45	790	-	1

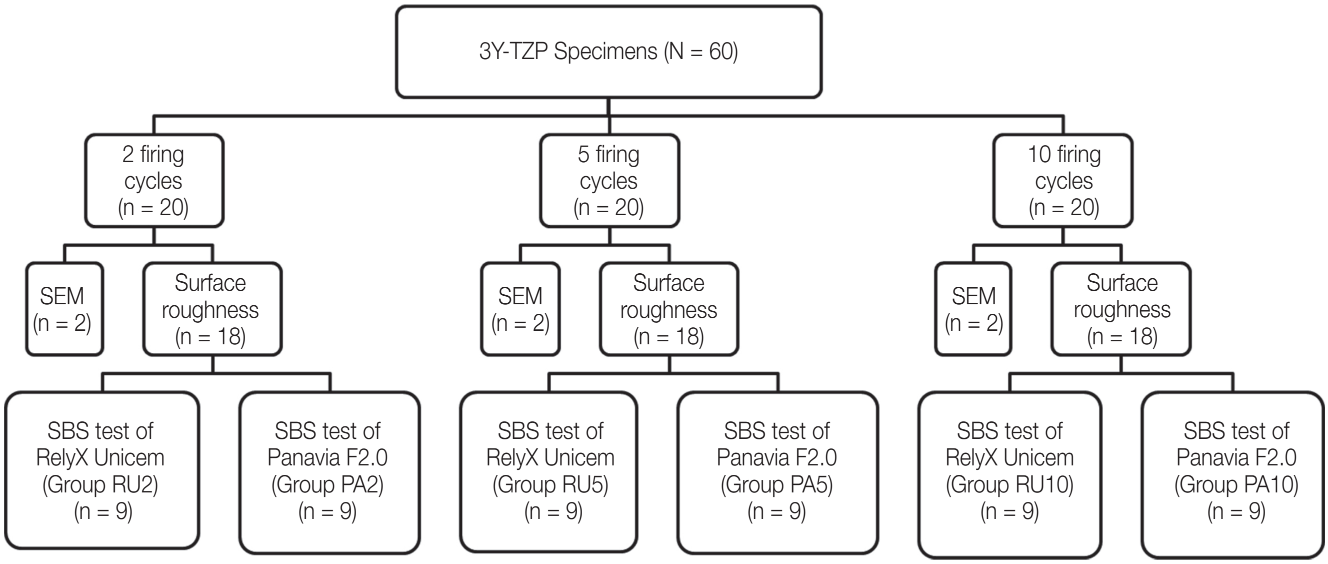

Fig. 1. Distribution of groups according to firing cycles, SEM analysis, surface roughness, and SBS (shear bond strength) tests. (RU: RelyX Unicem, PA: Panavia F2.0).

GmbH, Göttingen, Germany), which was calibrated before each measurement. Three measurements were performed at different lines, which passed through the center of each specimen, and their average was calculated to obtain the mean roughness of each sample.

The specimens obtained with 3 different firing cycles were further divided into 2 subgroups (n = 9) and 2 different types of resin cement were applied: MDP-free resin cement RelyX Unicem Clicker (RU, 3M ESPE, St. Paul, MN, USA), and MDP-containing resin cement Panavia F 2.0 (PA, Kuraray, Osaka, Japan). The resin cements were mixed according to the instructions of each manufacturer and were individually implemented into the cylindrical cavity (depth: 2 mm; diameter: 5 mm) of a polytetrafluoroethylene mold, which was fixed onto zirconia specimen's surface. The resin cement cylinders were polymerized by a LED curing unit (High power mode, Bluephase 20i, Ivoclar Vivadent, Schaan, Liechtenstein) at 1200 mW/cm^2 for 20 seconds from the top and the light intensity was controlled before each use with a radiometer (LED Radiometer, SDI Limited Dental Products, Victoria, Australia). The Oxyguard (Kuraray, Osaka, Japan) was applied to the upper surface of PA cement cylinders for 3 minutes, as suggested by the manufacturer.

The SBS tests were performed by using a computer-controlled universal testing machine (Lloyd Instruments, Fareham Hants, England) with a 1 mm/min crosshead speed. The SBS value was recorded as the peak load at failure in Newtons (N). This value was divided by the adhesive surface area (in mm^2) and the SBS in megapascals (MPa) was obtained.

The surfaces of all the debonded specimens were analyzed by using a stereomicroscope (Leica MZ 12, Meyer Instruments Inc., Houston, TX, USA) at 40 × magnification to determine the following failure modes: adhesive (A, failure of the resin cement at the zirconia surface), cohesive (C, failure within the resin cement), and mixed (M, adhesive and cohesive failures in combination). Recorded failure modes were calculated as a percentage.

The effects of firing cycles (3 levels: 2, 5, and 10 firing cycles) and resin cements (2 levels: RelyX Unicem versus Panavia F 2.0) on SBS were analyzed by the 2-way analysis of variance (ANOVA). Multiple comparisons were performed by using the Tukey-HSD test. The surface roughness data were analyzed by using 1-way ANOVA ($\alpha = .05$).

RESULTS

The means and standard deviations of SBS results are given in Table 2. The 2-way ANOVA revealed that both resin cement type and multiple firings of zirconia played an influential role in the SBS values and interactions between cement type and firing cycle were significant ($P < .05$) (Table 3). The bond strength values of RU were not statistically different regardless of firing cycles ($P > .05$). On the other hand, the SBS values of PA were found out to be significantly higher for the specimens fired 5 and 10 times than the ones fired 2 times ($P < .05$). The SBS values of RU and PA to the specimens obtained with 2 firings were not statistically different from each other ($P = 0.1$). The SBS values

Table 2. Comparison of the SBS (MPa) of resin cements to zirconia after repeated firings

Test groups	Mean	SD
RU2	5.8[A]	1.2
RU5	4.9[A]	1.3
RU10	6.4[A]	2.7
PA2	4.5[A]	1.8
PA5	14.1[B]	2.5
PA10	11.3[B]	3.0

Uppercase letters with the same superscript show no significant differences between test groups.
*RU: RelyX Unicem, PA: Panavia F 2.0.

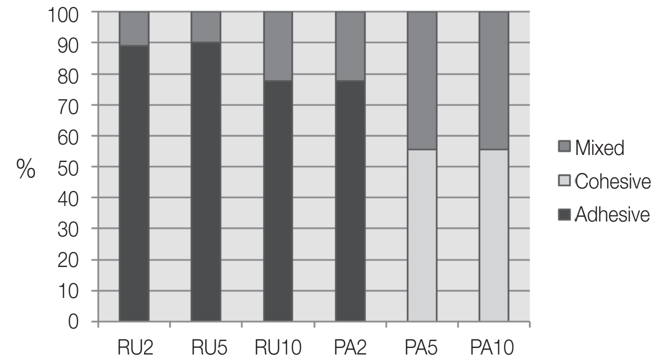

Fig. 2. Failure mode distribution of resin cements with regard to different firing cycles of zirconia.

Table 3. Two-way ANOVA results of bond strength of resin cements to zirconia after repeated firings

Source	Sum of squares	df	Mean Squares	F	*P*
Resin cement	30.973	1	30.973	4.0479	.005
Firing cycle	661.753	2	330.876	43.2419	.000
Resin cement * Firing cycle	88.489	2	44.244	5.7823	.006
Error	367.284	48	7.652		

The means ± standard deviations of surface roughness of the specimens obtained with 2, 5, and 10 firing cycles were 0.8 ± 0.02, 0.7 ± 0.03, and 0.8 ± 0.05, respectively. The 1-way ANOVA showed that there was no statistical difference between the surface roughness values of the groups (P = .2).

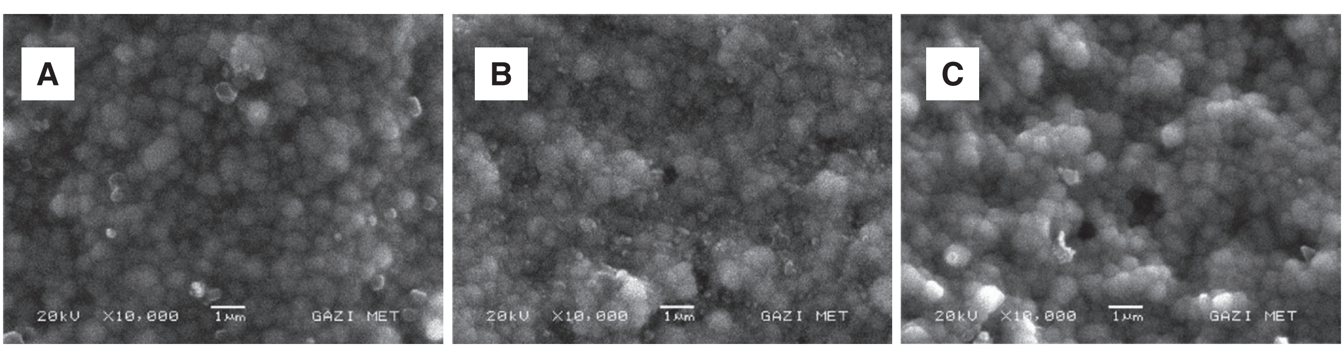

Fig. 3. SEM images (Original magnification × 10,000) of zirconia following different firing procedures. (A) 2 firings, (B) 5 firings, (C) 10 firings.

of PA were significantly higher than RU for the specimens obtained with 5 and 10 firing cycles (P = .001 and P = .02, respectively).

The distribution of failure modes is presented in Fig. 2. No cohesive failures were observed for RU cement. The failure types for RU were dominantly adhesive as 89%, 90%, and 78% for the specimens obtained with 2, 5, and 10 firing cycles, respectively. Similar to RU, PA showed no cohesive failure and failure type was dominantly adhesive with 78% for the specimens obtained with 2 firings. PA failed 56% cohesively and 44% mixed for the specimens obtained with 5 and 10 firing cycles.

SEM images showed that, compared to the control group (2 firing cycles), multiple firings (5 and 10 firing cycles) were found to cause some modifications such as irregular zirconia grain sizes and accordingly empty spaces between grains and grain-boundary continuity loss (Fig. 3).

DISCUSSION

This study examined the effects of multiple firings on SBS of 2 different types of resin cement to Y-TZP. The bond strength of RU and PA resin cements to zirconia were found similar when 2 firings were implemented, whereas PA demonstrated higher SBS to Y-TZP obtained with multiple firings. Therefore, the null hypothesis suggesting that multiple firings of Y-TZP would not affect the SBS of different types of resin cement was rejected.

Zirconia is a polymorphic material that may be present in 3 different phases: monoclinic, tetragonal, and cubic. The stable form of pure zirconia at room temperature to 1170°C is the monoclinic phase. Between 1170 - 2370°C, the tetragonal phase takes place and cubic over 2370°C. Monoclinic zirconia is the weakest of the 3 allotropes and transforms into the tetragonal structure after sintering.[27,28] However, during the cooling process after the sintering, tetragonal crystals of zirconia return to the monoclinic phase.[28] In order to stabilize the tetragonal phase at room temperature, stabilizing oxides like Y_2O_3 is added to pure zirconia.[28,29] Tetragonal Y-TZP may transform into the monoclinic phase due to the external stresses and surface treatments on zirconia or low temperature changes in the presence of water.[28,30-32] During this t-m phase transformation, the crystalline and atomic microstructures of the material are observed to change, leading to a structural and mechanical degradation on the zirconia surface.[28,30,32-34] On the other hand, t-m phase transformation caused by aging may be reversed by heat treatments at high temperatures.[28] This conversion of the monoclinic phase to tetragonal is called the reverse phase transformation.[28,35] It was reported that veneering porcelain firings of 3Y-TZP tend to trigger the reverse phase transformation that starts at 350°C and is completed at over the temperature of 550°C.[35] In our study, the Y-TZP specimens were subjected to multiple firings ranging between the temperatures of 450°C to 840°C followed by surface treatments. According to our study, PA resin cement showed higher SBS to the specimens subjected to multiple firings. This finding may be related to the reverse phase transformation of 3Y-TZP after multiple firings.[28] Cakir-Omur *et al.*[28] evaluated the reverse phase transformation of the LAVA Frame Y-TZP specimens and found that the repeated firings of zirconia reduced the monoclinic phase content and induced reverse phase transformation. On the other hand, Subaşı and Inan[15] specified that the repeated firings did not affect the monoclinic content of the VITA In-Ceram zirconia. It seems that the phase transformation of Y-TZP after repeated firings is material dependent. The correlation between bond strength and different phases of zirconia after repeated firings may be the subject of further studies.

Micromechanical retention of luting cements to zirconia can be enhanced by surface treatments which increase the surface roughness.[36] Airborne-particle abrasion with 125 µm aluminum oxide was applied to all specimens in our study. Similar to the study by Oilo *et al.*,[10] the present study showed that the surface roughness values of the specimens with different firing cycles were not found to be statistically different. However, empty spaces between Y-TZP grains and differentiation in grain size were determined with SEM images of specimens obtained with 5 and 10 firing cycles (Fig. 3B-C). Furthermore, a recent study has demonstrated empty spaces between zirconia grains, grain-boundary continuity loss, and elevated grains after different heat and surface treatments.[37] Higher bond strength to the specimens obtained with multiple firings of PA cement may be explained by functional monomer infiltration into these micro-gaps detected in SEM images.

The Panavia F2.0 contains MDP that bonds chemically to metal oxides of Y-TZP by a functional phosphate ester group.[38,39] Besides, at the resin-zirconia interface, molecular attraction arises from secondary forces such as Van der Waals forces or hydrogen bond.[40] Variations in molecular structure or surface treatments on the chemical affinity of MDP-containing materials to Y-TZP was evaluated previously.[38,40-43] The bond strength of resin cements may be influenced by alterations in the molecular structure of MDP as well as surface structural changes of zirconia.[44] After *in vitro* aging or different firing cycles, alterations in the crystalline structure of Y-TZP may occur.[14,28] Pandoleon *et al.*[45] found that a decrease in oxygen vacancies is accompanied by an increase in yttrium on the surface of Y-TZP after autoclave aging and these changes were associated with the phase transformation from tetragonal to monoclinic. They indicated that during this phase transformation, the oxygen vacancies on the surface migrated into the inner of zirconia and/or the oxygen vacancies near yttrium engaged oxygen from the ZrO_2, forming a metallic phase of Y_2O_3 on the surface with the aging process. This study indicates that surface molecular and microstructural changes of zirconia may occur due to different thermal applications. The authors of the present study assume that molecular and structural surface variations after multiple firings may be responsible for the higher bond strength of MDP-containing resin cement. However, the surface molecular and structural change of zirconia after repeated firing processes and the potential correlation between these changes and the resin cement bond strength is a matter of further research.

According to the failure mode analysis, it may be claimed that RU for all firing groups and PA for 2 times fired specimens failed adhesively, suggesting a weak bond quality.[37] On the other hand, PA showed mixed and cohesive failures for specimens obtained with 5 and 10 firing cycles. In this regard, the failure mode analysis supports the SBS results. The present findings indicate that MDP-free resin cement may not maintain a strong bonding to the Y-TZP neither with a standard firing procedure nor after multiple firings.

A shortcoming of the present study was that molecular structure and phase analysis after multiple firings of Y-TZP was not evaluated. For further investigations, analyzing the effects of structural changes and phase transformations of Y-TZP after repeated firings on bond strength seems to be necessary so as to have a better understanding of the underlying mechanisms responsible for higher bond strengths of MDP-containing resin cement to zirconia subjected to multiple firings. Furthermore, the present study evaluated a specific zirconia. Therefore, generalizing the results to other zirconia ceramics can be misleading. Additionally, airborne-particle abrasion was solely applied as the surface treatment method. The effects of different surface treatments implemented after multiple firings on resin bond strength may be the subject for further investigations.

CONCLUSION

Panavia F 2.0 and RelyX Unicem showed similar bond strength to zirconia with 2 firing cycles. However, compared to RelyX Unicem, Panavia F 2.0 showed higher bond strength to zirconia subjected to 5 and 10 firing cycles. The bond strength of RelyX Unicem to zirconia did not change regardless of firing cycles, whereas the bond strength of Panavia F 2.0 was enhanced when zirconia was subjected to multiple firings.

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