

## ORIGINAL ARTICLE

**Efficacy of ceramic repair material on the bond strength of composite resin to zirconia ceramic**OMER KIRMALI<sup>1</sup>, ALPER KAPDAN<sup>2</sup>, OSMAN TOLGA HARORLI<sup>3</sup>,  
CAGATAY BARUTCUGIL<sup>3</sup> & MEHMET MUSTAFA OZARSLAN<sup>1</sup><sup>1</sup>Department of Prosthodontics, Faculty of Dentistry, Akdeniz University, Antalya, Turkey, <sup>2</sup>Department of Restorative Dentistry, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey, and <sup>3</sup>Department of Restorative Dentistry, Faculty of Dentistry, Akdeniz University, Antalya, Turkey**Abstract**

**Objective.** The aim of this study was to evaluate the shear bond strength of composite resin in five different repair systems. **Materials and methods.** Sixty specimens (7 mm in diameter and 3 mm in height) of zirconia ceramic were fabricated. All specimen surfaces were prepared with a 30 µm fine diamond rotary cutting instrument with water irrigation for 10 s and dried with oil-free air. Specimens were then randomly divided into six groups for the following different intra-oral repair systems ( $n = 10$ ): Group 1, control group; Group 2, Cojet system (3M ESPE, Seefeld, Germany); Group 3, Cimara<sup>®</sup> System (Voco, Cuxhaven, Germany); Group 4, Z-Prime Plus System (Bisco Inc., Schaumburg, IL); Group 5, Clearfil<sup>™</sup> System (Kuraray, Osaka, Japan); and Group 6, Z-Bond System (Danville, CA). After surface conditioning, a composite resin Grandio (Voco, Cuxhaven, Germany) was applied to the zirconia surface using a cylindrical mold (5 mm in diameter and 3 mm in length) and incrementally filled up, according to the manufacturer's instructions of each intra-oral system. Each specimen was subjected to a shear load at a crosshead speed of 1 mm/min until fracture. One-way analysis of variance (ANOVA) and Tukey *post-hoc* tests were used to analyze the bond strength values. **Results.** There were significant differences between Groups 2–6 and Group 1. The highest bond strength values were obtained with Group 2 ( $17.26 \pm 3.22$ ) and Group 3 ( $17.31 \pm 3.62$ ), while the lowest values were observed with Group 1 ( $8.96 \pm 1.62$ ) and Group 6 ( $12.85 \pm 3.95$ ). **Conclusion.** All repair systems tested increased the bond strength values between zirconia and composite resin that used surface grinding with a diamond bur.

**Key Words:** zirconia, repair, composite resin, shear bond strength, Y-TZP**Introduction**

In the search for the ideal esthetic restorative material, many all-ceramic systems have been proposed. However, zirconia differs from other high strength dental ceramics with its pleasing esthetic and durable mechanical properties. Zirconia is a well-known polymorph that occurs in three forms: monoclinic, cubic and tetragonal. At room temperature, pure ZrO<sub>2</sub> has a monoclinic crystal structure and transitions to tetragonal and cubic phases at increasing temperatures. For biomedical applications, some metallic oxides, such as MgO or CaO, were added to zirconia in the early stages of the development. Today, research efforts appeared to be more focused on yttria oxide stabilized tetragonal zirconium oxide

polycrystal (Y-TZP), which has great molecular stability [1,2].

Clinical evaluations of zirconia-based bridges indicated that zirconia-based bridges perform satisfactorily over time, with no fractures to the Y-TZP framework. However, the incidence of chippings in the region of cusp tips or on the edge of occlusal surfaces increases with years and this can necessitate the reparation or replacement of restoration [3,4].

The failure of the crowns poses an esthetic and functional dilemma for the patient and dentist regarding reparation with a bonding system and composite or replacement of restoration. Replacement of a zirconia restoration is a time-consuming, costly and labored procedure, whereas reparation of a zirconia restoration is a fast, inexpensive and easy method [5].

Table I. Procedures used on the zirconia specimens in the zirconia repair groups.

| Test groups          | Repair systems and manufacturers            | Application procedures  |
|----------------------|---|---|
| Group 1<br>(Control) | No surface conditioning                     | 1. Surface grinding with a 30 $\mu\text{m}$ fine diamond rotary cutting instrument with water irrigation for 10 s and dried with oil-free air<br>2. Resin composite built-up (40 s photo-polymerization)  |
| Group 2              | CoJet System<br>(3M ESPE, Germany)          | 1. Sandblasted by silicate-coated alumina particles with a diameter of 30 $\mu\text{m}$ at a pressure of 2.3 bar ( $2.3 \times 10^5$ Pa) and from a distance of 10 mm<br>2. The tribochemical coating was completed by ESPESil (10 s)<br>3. Resin composite built-up (40 s photo-polymerization)  |
| Group 3              | Cimara System<br>(Voco, Germany)            | 1. Surface treated with Cimara grinding bur (10 strokes), removal of grind dust with a brush<br>2. Haftsilan (leave for 2 min; no air drying) was applied<br>3. Opaquer liquid (20 s photo-polymerization) was applied<br>4. Resin composite built-up (40 s photo-polymerization)                 |
| Group 4              | Z-Prime Plus<br>(Bisco Inc, Schaumburg, IL) | 1. Primer was applied and dried for 5 s in a compressed air system<br>2. Resin composite built-up (40 s photo-polymerization)   |
| Group 5              | Clearfil System<br>(Kuraray, Japan)         | 1. Surface treated with flame-shaped diamond bur (10 strokes)<br>2. K-etchant gel and rinse & dry<br>3. Mixed Clearfil SE Bond Primer and activator (5 s) Clearfil SE Bond (15 s) followed by air drying and 10 s photo-polymerization<br>4. Resin composite built-up (40 s photo-polymerization) |
| Group 6              | Z-Bond<br>(Danville, CA)                    | 1. Primer was applied for 30 s and evaporate the solvent under a gentle air stream for 5–10 s<br>2. Resin composite built-up (40 s photo-polymerization)  |

Several intra-oral all-ceramic restoration repair systems, based on different repair protocols, are on the market today [6]. The aim of this study was to evaluate the shear bond strength of five commercially available intra-oral adhesive systems for the repair of zirconia samples. The null hypothesis was that there is no significant difference in repair bond strengths among the five repair systems.

### Materials and methods

Sixty ( $n = 60$ ) Y-TZP Noritake Alliance (Noritake Co, Nagoya, Japan) specimens, 7 mm in diameter and 3 mm in height, were fabricated using Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) and sintered according to the manufacturer's instructions. The bonded surfaces of zirconia core specimens were smoothed with 600-, 800- and 1200-grit silicon carbide papers (English Abrasives, London, UK) using a polisher (Phoenix Beta Grinder/Polisher, Buehler, Germany) to obtain standardized surface roughness. Finally, zirconia specimens were ultrasonically cleaned in 96% isopropyl alcohol for 3 min and steam-cleaned for 10 s. Then, specimens were randomly divided into six groups for the following different intra-oral repair systems ( $n = 10$ ): Group 1, no conditioning surface: control group; Group 2, Cojet system; Group 3, Cimara<sup>®</sup> System; Group 4, Z-Prime Plus System; Group 5, Clearfil<sup>™</sup> System; and Group 6, Z-Bond System. Details of the repair systems procedure used for each group are summarized in Table I.

Clinically, when the fracture site is repaired with composite resin, surface grinding with a diamond bur

is a commonly used method for surface cleaning to improve mechanical bonding and remove the contamination area as standard procedure. To simulate this clinical condition, all specimen surfaces were prepared with a 30  $\mu\text{m}$  fine diamond rotary cutting instrument with water irrigation for 10 s and dried with oil-free air.

After surface conditioning, a composite resin Grandio was applied to the zirconia surface using a cylindrical mold (5 mm diameter and 3 mm length) and incrementally filled up according to the manufacturer's instructions for each intra-oral system. Each layer was light-polymerized for 40 s at a distance of 1 mm using a light-polymerizing unit (Astralis 3, Ivoclar Vivadent, Liechtenstein) with an output power of 600 mW/cm<sup>2</sup>. Then, zirconia-composite specimens were embedded in brass holders using acrylic resin for the shear bond strength test (SBS). Before the experiment, specimens were steeped in 37°C distilled water for 24 h.

The specimens were subjected to a SBS test using a universal testing machine (Lloyd LF Plus, Ametek Inc, Lloyd Instruments, Leicester, UK). A knife-shaped indenter applied the load at a cross-head speed of 1 mm/min until fracture occurred (Figure 1). SBS values were recorded in Newtons and converted into megapascals (MPa). As for the mean failure load and standard deviation for each group, they were calculated from these data.

The fractured surfaces were visually analyzed with a stereomicroscope (SMZ 800, Nikon, Tokyo, Japan) at 40 $\times$  magnification to determine the failure modes of specimens. Failure types were observed as adhesive failure, in which composite resin completely separated



Figure 1. Specimen placed in universal testing machine for shear bond strength test.

from the zirconia surface; cohesive failure, in which composite resin completely fractured; and mixed failure, in which both failure types were observed (adhesive and cohesive).

*Statistical analysis*

The bond strength values differences between the zirconia and the composite resin were evaluated with ANOVA. Pairwise comparisons were made by Tukey *post-hoc* test. Analyses were carried out by the SAS 9.1 statistical package (SAS Institute, Cary, NC) with a  $p < 0.05$  significance level.

**Results**

Figure 2 presents mean and standard deviation values of the shear bond strength test and the results of statistical analysis for all groups. The results of

statistical analyses indicated that there were significant differences between Group 1 and other five groups and there were significant differences between Groups 2, 3 and 5 and Group 6. The highest mean bond strength values were obtained in Group 3 ( $17.31 \pm 3.62$ ) and Group 2 ( $17.26 \pm 3.22$ ), while the lowest mean value was observed in Group 1 ( $8.96 \pm 1.62$ ). Besides, differences between these groups (4 and 6) and other groups (2, 3, 4 and 5) were not found to be statistically significant ( $p > 0.001$ ). All zirconia specimens exclusively showed adhesive failures between the zirconia surfaces and all repair resin.

**Discussion**

Zirconia is the most popular dental material for patients and dentists because of its superior mechanical properties such as high fracture toughness and natural appearance [7–9]. Thus, zirconia ceramic material has wide clinical usages, especially for anterior and posterior restorations by frameworks [10,11]. The effective bonding potential between zirconia and veneering porcelain is necessary with the long-term performance of all restorations. However, some authors stated that the failure of zirconia occurs mostly between zirconia and veneering porcelain [10–14]. Sailer et al. [13] reported fracture rates of 13% for veneer ceramics at the end of 3 years and Raigrodski et al. [10] reported fracture rates of 25% of zirconia for veneer ceramics at the end of 31 months. Previous investigations have been focused on different treatments for improving the bonding potential [15–17] and surface activation for chemical adhesion [18–20].

The fractures of the veneer ceramic cost patients time and money. Due to this, the need for ceramic restoration repair increases. There was a large

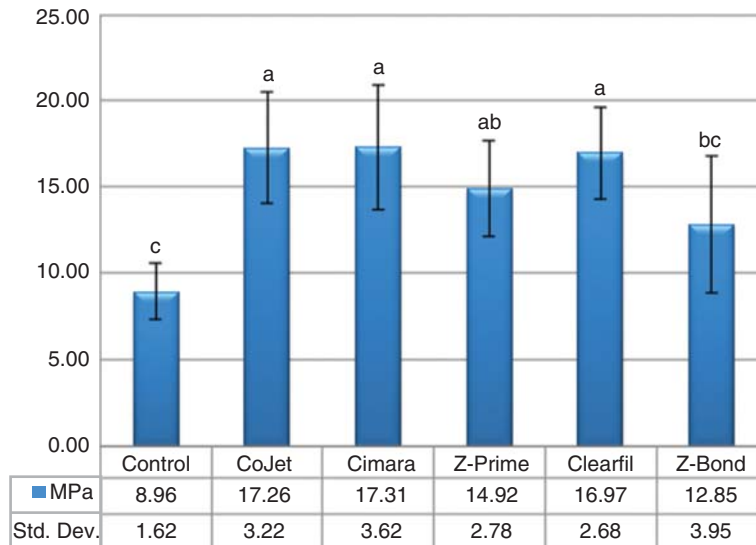


Figure 2. Mean and standard deviation value of the shear bond strengths (MPa) of composite resin to zirconia. The same letters were not significantly different ( $p > 0.05$ ).

increase in restoration repair instruction at dental schools [21]. On the other hand, intra-oral repairing methods may be a more acceptable, easy and less traumatic treatment for the patients, especially for zirconia, which is such an expensive and delicate restoration.

In recent times, some manufacturing companies have introduced intra-oral repair systems for ceramic and zirconia restorations. Researchers have evaluated the effect of these systems by the increase of the bonding strength of ceramic/zirconia surface and composite resin. So, the aim of this study was to evaluate the shear bond strength of composite resin in five different repair systems. According to the results of this study, the null hypothesis was not accepted, as all repair systems increased the bond strength.

Blum et al. [6] evaluated the effect of four intra-oral ceramic repair systems on the ceramic surface and stated that the ceramic repair system did not increase the tensile bond strength as well as when using the other repair systems. However, they found that the Cojet system significantly increased the tensile bond strength values compared to the other repair system evaluated. In addition, they reported that all repair systems impaired the bond strength of veneering ceramics toward resin cement. In our study, all repair systems tested, except Group 6, significantly increased the bond strength values. Furthermore, Z bond application increased the shear bond strength compared to no conditioning surface, but the differences were not statistically significant. This result did not coincide with the conclusions of Blum et al. [6]. The reason of the difference in these results may be happened effect of all the specimens surface prepared with diamond bur before all repair system applications in the present study.

Han et al. [22] examined the Cojet system with 30  $\mu\text{m}$  silica-modified  $\text{Al}_2\text{O}_3$ , Ceramic repair system and Signum zirconia bond applications on zirconia surfaces. They reported that the Cojet system and Signum zirconia bond applications significantly affected the bond strength compared with the Ceramic repair system. In the present study, there was a statistically significant difference between the Cojet system and no conditioning surface. Similarly, Cristoforides et al. [23] found that tribochemical silica coating (TBS) had a significantly positive effect on bond strength and reported that TBS can be used for obtaining the best bonding between Y-TZP zirconia and composite resin. Kim et al. [24] found that the values of tensile bond strength of silica coating to alumina and zirconia copings were statistically significant. Thus, the results of the present study showed similarities to those of Han et al. [22], Cristoforides et al. [23] and Kim et al. [24].

Lee et al. [5] reported that there was not a statistically significant difference in the bond strength of a

ceramic repair system compared to alumina and zirconia copings. Furthermore, they stated that porcelain repair materials increased the bond strength values of veneering ceramic surfaces. Ozcan et al. [25] indicated that the highest tensile bond strength value was seen in the Porcelain repair kit group compared to the Cojet system group and Clearfil system group in dry conditioning. The present study stated that Cojet and Clearfil system applications increased bond strength between zirconia and composite resin. This result did not coincide with the conclusions of Ozcan et al. [25]. They evaluated the durability of repair bond strength of a composite to ceramic after different repair systems. This result can be a reason of the difference. In another study, Ozcan et al. [26] found that etching the veneering ceramic, silica coating the zirconia core, followed by silane on both substrates, resulted in the highest bond strength of composite resin to zirconia core/veneering ceramic. The present study, in agreement with the Ozcan et al. [26] studies, found that the Cojet repair system was the most effective application for increasing the bond strength between the composite resin and zirconia surface.

Kim et al. [27] stated that there was a statistically significant difference in retentive force to zirconia surface when Z-Prime plus primer was used with Panavia F 2.0. Similarly, in the present study, there was a statistically significant difference between the Z-Prime plus group and no conditioning surface.

An et al. [28] evaluated of bond strengths of gingiva-colored composite resin to porcelain, metal and zirconia surfaces. For the purpose of cleaning the surfaces, they applied a porcelain repair system (Ceramic repair system) with sandblasting (50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles) and 37% Phosphoric acid, respectively, and found that zirconia surfaces showed the lowest bond strength to composite resin. Our study found that surface grinding with a diamond bur increased the bond strength values when used with all repair systems tested. Thus, the results of this study did not coincide with An et al.'s [28] results. Acid etching might be damaging to the retentive areas that are obtained with sandblasting. This can be the reason of the difference.

The result of the Z-Bond was not compared with the literature. However, no previous studies exist about this bond. The results of this study showed that Z-bond application provides good bonding strength to increase the chemical bonding strength of zirconia surface compared to no conditioning surface.

As part of the results of this study, surface applications applied on zirconia should be supported by long-term studies in order to achieve a better bond between zirconia and composite resin.

Within the limitations of this study, the following conclusions were drawn: The highest bond strength

values were recorded in Group 3 ( $17.31 \pm 3.62$ ), Group 2 ( $17.26 \pm 3.22$ ) and Group 5 ( $16.97 \pm 2.68$ ), while the lowest values were recorded in Group 1 ( $8.96 \pm 1.62$ ) and Group 6 ( $12.85 \pm 3.95$ ). All repair systems tested increased the bond strength values between zirconia and composite resin.

**Declaration of interest:** The authors have no declared financial interests in any company manufacturing the types of products mentioned in this article. The authors alone are responsible for the content and writing of the paper.

## References

- [1] Manicone PF, Rossi Iommetti P, Raffaelli L. An overview of zirconia ceramics: basic properties and clinical applications. *J Dent* 2007;35:819–26.
- [2] Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. *Biomaterials* 1999;20:1–25.
- [3] Crisp RJ, Cowan AJ, Lamb J, Thompson O, Tulloch N, Burke FJ. A clinical evaluation of all-ceramic bridges placed in patients attending UK general dental practices: three-year results. *Dent Mater* 2012;28:229–36.
- [4] Burke FJ, Crisp RJ, Cowan AJ, Lamb J, Thompson O, Tulloch N. Five-year clinical evaluation of zirconia-based bridges in patients in UK general dental practices. *J Dent* 2013;41:992–9.
- [5] Lee SJ, Cheong CW, Wright RF, Chang BM. Bond strength of the porcelain repair system to all-ceramic copings and porcelain. *J Prosthodont* 2013;31:1–5.
- [6] Blum IR, Nikolinakos N, Lynch CD, Wilson NH, Millar BJ, Jagger DC. An in vitro comparison of four intra-oral ceramic repair systems. *J Dent* 2012;40:906–12.
- [7] Vagkopoulou T, Koutayas SO, Koidis P, Strub JR. Zirconia in dentistry: part 1. Discovering the nature of an upcoming bioceramic. *Eur J Esthet Dent* 2009;4:2–23.
- [8] Akin H, Ozkurt Z, Kirmali O, Kazazoglu E, Ozdemir AK. Shear bond strength of resin cement to zirconia ceramic after aluminum oxide sandblasting and various laser treatments. *Photomed Laser Surg* 2011;29:797–802.
- [9] Pittayachawan P, McDonald A, Young A, Knowles JC. Flexural strength, fatigue life, and stress-induced phase transformation study of Y-TZP dental ceramic. *J Biomed Mater Res B Appl Biomater* 2009;88:366–77.
- [10] Raigrodski AJ, Chiche GJ, Potiket N, Hochstedler JL, Mohamed SE, Billiot S, 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;96:237–44.
- [11] 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;32:180–7.
- [12] Luthardt RG, Sandkuhl O, Reitz B. Zirconia-TZP and alumina-advanced technologies for the manufacturing of single crowns. *Eur J Prosthodont Restor Dent* 1999;7:113–19.
- [13] Sailer I, Fehér A, Filser F, Lüthy H, Gauckler LJ, Schärer P, et al. Prospective clinical study of zirconia posterior fixed partial dentures: 3-year follow-up. *Quintessence Int* 2006;37:685–93.
- [14] Kirmali O, Akin H, Ozdemir AK. Shear bond strength of veneering ceramic to zirconia core after different surface treatments. *Photomed Laser Surg* 2013;31:261–8.
- [15] Aboushelib M, Kleverlaan C, Feilzer A. Microtensile bond strength of different components of core veneered all-ceramic restorations. II. Zirconia veneering ceramics. *Dent Mater* 2006;9:857–63.
- [16] Della Bona A, Borba M, Benetti P, Cecchetti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. *Braz Oral Res* 2007;21:10–15.
- [17] Spohr AM, Borges GA, Júnior LH, Mota EG, Oshima HM. Surface modification of in-ceram zirconia ceramic by Nd:YAG laser, Rocotec system, or aluminum oxide sandblasting and its bond strength to a resin cement. *Photomed Laser Surg* 2008;26:203–8.
- [18] Sahafi A, Peutzfeldt A, Asmussen E, Gotfredsen K. Bond strength of resin cement to dentin and to surface-treated posts of titanium alloy, glass fiber, and zirconia. *J Adhes Dent* 2003;5:153–62.
- [19] Xible AA, de Jesus Tavares R, de Araujo CRP, Bonachela W. Effect of silica coating and silanization on flexural and composite-resin bond strengths of zirconia posts: an in vitro study. *J Prosthet Dent* 2006;95:224–9.
- [20] Kirmali O, Akin H, Kapdan A. Evaluation of the surface roughness of zirconia ceramics after different surface treatments. *Acta Odontol Scand* 2013;21:1–8.
- [21] Blum IR, Lynch CD, Wilson NH. Teaching of direct composite restoration repair in undergraduate dental schools in the United Kingdom and Ireland. *Eur J Dent Educ* 2012;16:53–8.
- [22] Han IH, Kang DW, Chung CH, Choe HC, Son MK. Effect of various intraoral repair systems on the shear bond strength of composite resin to zirconia. *J Adv Prosthodont* 2013;5:248–55.
- [23] Cristoforides P, Amaral R, May LG, Bottino MA, Valandro LF. Composite resin to yttria stabilized tetragonal zirconia polycrystal bonding: comparison of repair methods. *Oper Dent* 2012;37:263–71.
- [24] Kim BK, Bae HE, Shim JS, Lee KW. The influence of ceramic surface treatments on the tensile bond strength of composite resin to all-ceramic coping materials. *J Prosthet Dent* 2005;94:357–62.
- [25] Ozcan M, Valandro LF, Amaral R, Leite F, Bottino MA. Bond strength durability of a resin composite on a reinforced ceramic using various repair systems. *Dent Mater* 2009;25:1477–83.
- [26] Ozcan M, Valandro LF, Pereira SM, Amaral R, Bottino MA, Pekkan G. Effect of surface conditioning modalities on the repair bond strength of resin composite to the zirconia core/ veneering ceramic complex. *J Adhes Dent* 2013;15:207–10.
- [27] Kim SM, Yoon JY, Lee MH, Oh NS. The effect of resin cements and primer on retentive force of zirconia copings bonded to zirconia abutments with insufficient retention. *J Adv Prosthodont* 2013;5:198–203.
- [28] An HS, Park JM, Park EJ. Evaluation of shear bond strengths of gingiva-colored composite resin to porcelain, metal and zirconia substrates. *J Adv Prosthodont* 2011;3:166–71.