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Original research

Shear bond strengths of five porcelain repair systems to zirconia infrastructures

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Purpose
This study aimed to investigate the effect of five porcelain repair systems on shear bond strength in composite and zirconia infrastructures and to identify the bond failure mode after thermocycling.

Materials and Methods
Disk-shaped zirconia samples (n=50) were divided into five groups (n=10) according to repairing system type. Each repair system was applied to the zirconium samples and a hybrid composite was used for repairing. Shear bond testing of all groups was carried out using a universal testing machine after thermocycling.

Results
Repair systems demonstrated no significant difference in repairing zirconia except Single Bond. Single Bond was the weakest in repairing the infrastructures. The highest and lowest mean bond strength values for the zirconia groups were 18,91 MPa and 3,63 MPa, respectively.

Conclusion
The three repair systems, Ivoclar, Clearfil, and Bisco, were more effective than the Single Bond and Ultradent repair systems in repairing zirconia, and their bond failure modes were both mixed and adhesive.

Keywords: *Repair system, zirconia, shear bond, adhesive system, bond failure*

Introduction

Although the use of all-ceramic restorations have become widespread in recent years due to their aesthetic superiority, metal-ceramic restorations are still the most frequently used restorations due to their mechanical durability (1,2). Due to the increasing cost of gold alloys in the 1960s, the use of alternative alloys for prosthetic restorations became more popular. The mechanical properties of these materials allow for thinner but more robust restorations (3). Due to cost and rigidity nickel-chromium and cobalt-chromium alloys are preferred (4).

The increase in the aesthetic expectations of individuals in recent years has led to the development of different types of dental ceramic restorations (5–7). The chemical properties of the ceramics and their superior performance in mimicking dental tissues were the main reasons behind the widespread use of these dental materials (8). Zirconium material was first introduced in dentistry in 1990 as a crown prosthesis and as an infrastructure material in fixed prostheses (9).

Most all-ceramic materials have been developed to achieve esthetic restoration. One of the most used all-ceramic esthetic restorations is zirconia, which differs from others by its resistant mechanical properties. Zirconia has three forms: monoclinic, tetragonal, and cubic; it structure is

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monoclinic at room temperature and transforms to the cubic and tetragonal phases with increasing temperature (9).

Porcelain veneer fractures have been reported to be the most common reason for the replacement of metal-ceramic crowns and bridges (10,11). The failure of veneered porcelain may occur from inadequate metal framework design, tooth preparation and occlusal adjustment (12,13).

The most frequent complication is the small chip-off fracture of veneered ceramic. Although replacing them with ceramic restorations is a common approach, they can be repaired intraorally when they are not completely damaged (14–16). During the replacement of a fractured ceramic restoration trauma may damage the remaining teeth and tissues. This procedure costs more than repairing the chipped part (17). Numerous commercial intraoral ceramics repairing systems has been developed for repairing these kinds of restorations. On the other hand, scientific studies reveal that ceramic repair systems cannot create a persistent solution due to their weakened bond strength (18,19).

The bonding strength between the cracked restoration and the repair material must be strong enough. When the bond strength of ceramic repair systems is at clinically acceptable levels, the time and money spent on making a new restoration will be reduced (7).

In the present study, five ceramic repair systems were used to simulate chairside zirconia infrastructure repair using composite resin. The aim was to compare the effects of the five repair systems on the shear bond strength (SBS) between the composite and zirconium to analyze the mode of failure in each experimental group. The null hypothesis is as follows: there is no difference in the bond strength of the different ceramic repair systems in repairing a zirconia infrastructure.

Materials and methods

Study design and sample preparation

50 disk-shaped zirconia samples with a 10 mm diameter and a 3 mm thickness were used in the present study. The zirconia disks were prepared from presintered blocks (H.C. Starck, Berlin, Germany) according to the manufacturer's instructions using a CAD/CAM system (CORITEC T 350i loader, imes-icore, Eiterfeld, Germany) and sintered to the final required dimension (10 mm in diameter and 3 mm thick) in a special high-temperature furnace. Table 1 shows the materials used and their manufacturer's information.

All disk specimens were properly polished by a special polishing machine (Tegrapol-11;Struers, Ballerup, Germany) using wet silicon carbide paper ground with 600, 800, and 1,000 grit under cool water for 1 min. All zirconia disks were treated with airborne-particle abrasion device (Airsonic Mini Sandblaster, Hager & Warken, Duisburg, Germany) with a 50-µm particle size aluminum-oxide for 10 seconds at a pressure of 0,3 MPa and from 10 mm distance.

The repairing procedure was performed by the same operator (S.A.). Each repair system used in this study was applied to the zirconium samples according to the manufacturer's instructions, as explained in Table 2.

Hybrid composite resin was incrementally packed with a hand instrument using a specially designed epoxy glass mold (6 mm diameter and 3 mm thickness). Each layer was light-cured with a light polymerizing unit (3M Elipar S10, 3M Espe, Germany) for 40 s at a distance of 1 mm with an output of 1,000 mW/cm2. The wavelength of the light polymerizing unit was measured by a spectroradiometer (Model 77702, Oriel İnstrument, Danbury, CT, USA), power density was measured using a radiometer (Radiometer LED, Demeton/Kerr, Danbury, CT, USA) prior to every specimen curing.

The bonding process was conducted by the same operator during experiments. After polymerization, the assembly of the repaired samples was removed from the mold, and light curing was repeated in five aspects of all blocks (upper and lateral) for 20 s per side.

Experimental groups

The samples (N = 50) were divided into five groups (n = 10): zirconia with the Bisco repair system (ZB), zirconia with the Clearfil repair system (ZC), zirconia with the Ivoclar ceramic repair system (ZI), zirconia with Single Bond (ZS), and zirconia with the Ultradent repair system (ZU).

Thermocycling protocols

The samples were all stored in distilled water at 37 °C for 24 hours and then subjected to thermal cycling (Slibrus Technica Termal Siklus, İstanbul, Turkey) of 1,200 cycles at 5–55 °C, with a dwell time of 20 s at each temperature and a transfer time from one bath to the other of 10 s.

Testing protocols

All samples were fixed by chemically cured acrylic resin in a steel mold. Shear bond testing of all groups was carried out using universal testing machine (Instron 3345, Instron Corp., Norwood, Illinois, USA) at a crosshead speed of 1 mm/min. SBS values were calculated by dividing the maximum load at failure (N) by the bonding area (mm^2) and recorded in megapascals (MPa). The failure modes of the bond related to the fractured surfaces were analyzed visually by using a stereomicroscope (EMS-405, Esman, Turkey) at 20x magnification. The failure areas were classified as adhesive, cohesive, or mixed type.

Statistical analysis

Statistical analyses were performed by the Number Cruncher Statistical System 2007 (NCSS, Utah, USA) software for Windows. The Shapiro–Wilk test was used to analyze if the measured parameters met the assumptions of normal distribution. The results of the test indicated that the data were normally distributed. Therefore, data were analyzed using the one-way ANOVA and the Tukey's HSD was performed to determine the group responsible for the difference. p-values less than 0.05 were considered statistically significant.

Results

Table 3 shows maximum, minimum, mean values and standard deviation values of the SBS test for the groups. The lowest and highest mean bond strength values for the zirconia groups were 18,91 MPa and 3,63 MPa respectively. When the SBS values of the repair systems applied to the zirconia groups were compared, both Ultradent (ZU) and single bond (ZS) repair systems showed lower SBS values than the

Table 1: *Details of the materials used in the study.*

Material	Composition	Manufacturer	Lot no.
Zirconia (Z)	ZrO_2/HfO_2/ Y_2O_3>99, Al_2O_3 <0.10, Fe_2O_3 <0.10, $Na_2 O_3$<0.04)	H.C.Starck, Berlin, Germany	50574292 50575967
Clearfil repair system (C)	K-etchant gel: 40% phosphoric acid Clearfil-SE Bond Primer: 10-methacryloyloxydecyl dihydrogen phosphate (MDP), HEMA, dimethacrylate monomer, water, photoinitiator, Clearfil-SE Bond: silanated colloidal silica, Bis-GMA, 10-MDP, Clearfil Porcelain bond activator: bisphenol A polyethoxy dimethacrylate 3-methacryloyloxypropyltrimethoxy silane (MPS)	Kuraray Co., Osaka, Japan	000016
Bisco repair system (B)	9.5% Hydrofluoric acid Silane with methacrylate Solution: Alcohol One step: bis-GMA, BPDM, HEMA, CQ, p-dimethylaminobenxoic acid (co-initiator), acetone, 8.5% glass fillers	BISCO Dental Products, Illinois, U.S.A.	1700001601
Ivoclar repair system (I)	Monobond® Plus a Primer: Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate. Heliobond – a light-curing bonding agent: Bis-GMA and tri-ethylene glycol dimethacrylate (99 wt.%), initiators and stabilizers (<1%).	Ivoclar Vivadent Inc., Liechtenstein, Switzerland	T42712
Ultradent repair system (U)	Etch: 9% hydrofluoric acid, Ultradent silane: 8% methacryloxypropyl-trimethoxysilane, isopropyl alcohol, acetic acid, Peak Universal Bond: 7.5% ethyl alcohol, 0.2% chlorhexidine, methacrylic acid, 2-HEMA	Ultradent Products GmbH, Cologne, Germany	BBFC4
Single bond (S)	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond Copolymer Filler, Ethanol, Water, Initiators, Silane	3M, ESPE, St., Paul, MN, USA	604724
Filtek Z250 (shade C2)	Matrix: Bis-GMA, Bis-EMA, UDMA, TEGDMA, Filler: zirconia, Silica	3M, ESPE, St., Paul, MN, USA	N566178. N545065

Table 2: *Application procedures and contents of the repair systems used in this study.*

	Application procedures	Content
Bisco Repair System (B)	1.Apply 1 coat of Z-PRIME Plus to the exposed zirconia, dry with an air syringe for 3-5 seconds. 2.Apply a thin layer of PORCELAIN BONDING RESIN to the repair site. Spread composite evenly over the surface and light cure. 3. Repair was completed using composite resin and light cured fo 40 seconds	Porcelian etchant Porcelian primer Opaquer catalyst Opaquer Base Universal Z-Prime Plus Porcelain bonding resin
Clearfil Repair System (C)	Clearfil SE Bond Primer and porcelain bond activator were mixed for 5 seconds 2.Bonding agent was applied for 10 seconds (air drying) and photo-polymerization for 40 seconds) 3. Repair was completed using composite resin and light cured fo 40 seconds	K-etching gel Clearfil SE Bond Porcelian bond activator
Ivoclar Repair System (I)	1.Monobond Plus was applied and allowed to react for 60 seconds and after air dried. 2.Thin layer of Heliobond was applied and light cured for 90 seconds 3. Repair was completed using composite resin and light cured fo 40 seconds	IPS Empress Direct Opaque Monobond Plus Heliobond
Ultradent Porcelian Repair system (U)	1.Apply Hydrofloric acid on metal surface for 90 second 2.Apply silane and leave 1 minute 2.Apply Peak Universal bond for 10 second and light cure for 20 second	PermaFlo Dentin Opaquer EtchArrest OpalDam Peak Universal Bond Porcelain Etch Ultradent Silane
Single Bond universal adhesive (S)	1.Apply on surface of Zirconium leave it for 20 second dry with air for 5 second 2.Light cure for 20 seconds	Single bond universal adhesive

Table 3: *Shear bond strengths values (MPa) of the groups.*

Groups (n=10)	Minimum	Maximum	Mean	Std. Deviation
ZB	14,29	27,65	18,91	4,33
ZC	9,33	28,37	18,61	5,37
ZI	8,76	23,51	15,24	5,30
ZS	2,73	4,80	3,63	0,62
ZU	4,99	9,87	6,63	1,50

Table 4: *Pairwise comparisons of the repair systems.*

Tukey Multiple Comparisons Test	P
ZS/ZB	**0,0001**
ZS / ZC	**0,0001**
ZS / ZI	**0,0001**
ZS / ZU	0,447
ZB / ZC	0,999
ZB / ZI	0,249
ZB / ZU	**0,0001**
ZC / ZI	0,331
ZC / ZU	**0,0001**
ZI / ZU	**0,0001**

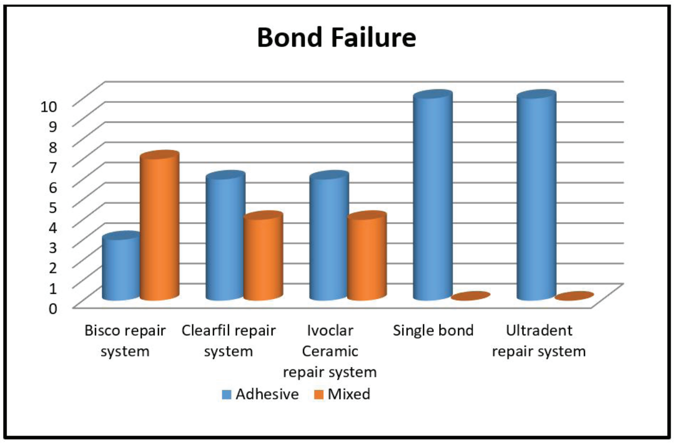

Figure 1. *Failure modes of the groups.*

remaining repair systems (p<0.0001). Bisco (ZB), İvoclar (ZI) and Clearfil (ZC) repair systems showed similar SBS values. Table 4 shows multiple comparisons of repair systems. For the failure modes of the repair systems, Ivoclar (ZI), Clearfil (ZC), and Bisco repair systems (ZB) had both mixed and adhesive failures. All the specimens of the Single Bond (ZS) and Ultradent (ZU) groups failed adhesively (Figure 1).

Discussion

Veneering porcelain fracture is a common complication that can occur in all dental ceramic systems. The incidence of chipping fractures is significantly higher for metal-ceramic and zirconia-based fixed dentures than for framework fractures. For both restorations, veneer chipping can be treated by polishing or repairing rather than replacing the restorations (20).

Five different repair systems were compared in this study. According to the results of the study, the null hypothesis was rejected. The SBS of zirconia to composite resin was significantly different among different repair systems. The samples were subjected to thermocycling for 1,200 cycles at 5–55°C. According to Gale and Darvell (21-23), 10,000 cycles could represent a year of service, as 20–50 cycles are equal to one day. The thermal cycle procedure has been applied similarly in many other studies, and thus we used the same protocol to compare our results with theirs.

Generally, shear bond tests or tensile bond tests are used to measure bond strength. The tensile bond strength test is extremely affected by sample form and the formation of non-uniform stress distributions throughout load applications. The shear bond strength test is the most widely used test for bond strength because of its simple usage, clear test protocol, and rapid production of the test result (23,24). For these reasons, the shear bond tests were performed to evaluate the bond strength in our study as in previous studies (23-25).

Sandblasting provides micromechanical retention and a stronger composite-metal bond when performed on zirconia. In comparing the different repair systems in this study, the surface treatments were not changed, and the sandblasting process, which is one of the most effective methods, was performed for all groups (25). Matsumura *et al.*(26) reported the SBS value of 10 MPa as the minimal to obtain clinically acceptable results. According to the results of the present study, all the repair systems, except ZS and ZU, have exceeded 10 MPa. Statically significant differences were found between the repair systems, and thus the null hypothesis was rejected. Kocaagaoglu and Gurbulak (27) evaluated the SBS between two porcelain repair kits and zirconia or a nonprecious metal alloy. After thermocycling for 1,200 cycles, the SBS of the zirconia group using the Clearfil repair system was 8.80 MPa, and the SBS of metal was 19,75 MPa. We evaluated the SBS of the five porcelain repair systems and the zirconia infrastructure materials after thermocycling for 1,200 cycles. The result of the Clearfil repair system was 18,61 MPa. The difference may be due to the use of a rotary cutting instrument, as 30 µm is not sufficient to roughen a zirconia surface. Zirconia has superior hardness and needs to be ground with coarse diamond rotary instruments (28). Goncalo et al. tested the effect of a surface treatment and primer application on the composite SBS to zirconia. The zirconia prime plus group, which is present in the Bisco repair system and is similar to our ZB group, was higher, and the scores were similar to those in our study (29,30).

Han *et al.* (30) investigated the effects of three intraoral ceramic repair kits on the bond strength between composite resin and zirconia. The SBS was found to be 3.21 MPa for a ceramic repair system (Ivoclar), 7,80 MPa for a CoJet repair system, and 8,98 MPa for a Signum Zirconia Bond (30). The SBS of the ceramic Ivoclar repair system was weak even without thermocycling. The score for Ivoclar of the ZI group in our study was 15,24 MPa, which was higher than that in the previous study. This may be due to the fact that a zirconia surface should be sandblasted, which is a more effective method for roughening a zirconia surface. Kocaoğlu *et al.* (31) examined the effect of three intraoral ceramic repair systems on the bond strength between composite resin and zirconia (31). The SBS was 10,85 MPa for the Clearfil repair system and 12,64 MPa for the Bisco intraoral repair kit. Compared with the results of our study, the results were lower in Kocaoğlu *et al.'s* study (31). We sandblasted the zirconia samples before application, and this could have enhanced the retention.

No significant difference was found among Clearfil, Bisco, and the ceramic repair system used for repairing the zirconia infrastructure properly because the three repair systems, which contained bonding agents and organo-phosphate monomers (e.g., 10-methacryloxydecyl dihydrogen phosphate [MDP]), were developed to improve the bond strength of resin-based materials to a silica-free zirconium structure (32, 33). Previous studies showed that commercial phosphate-monomer-containing zirconia primers, improved both the initial and long-term resin bond strength to zirconia ceramic sutructures significantly (34–40). The pretreatment of zirconia with MDP-containing adhesive systems can lead to satisfactory adhesion between the different composite resins and ceramic surfaces even after the artificial aging process (41). Moreover, similar to our study, the surface treatment of air-abrasion and phosphate-monomer-containing primer application improved the durability of zirconia-resin bond strength (42–45).

A significant difference was found among the three repair systems (Bisco, Ultradent, and Clearfil) and between the other two repair systems (Ultradent and Single Bond adhesive), probably because Ultradent and Single Bond adhesive depend on silane as a surface treatment. Silane materials were often used for coupling with silica-based ceramics through the formation of a chemical covalent bond to obtain a chemical bond between resin and zirconia, which have silica-free and relatively nonpolar surfaces. They are chemically much more stable than silica-based ceramics, and thus traditional silane chemistry is not usually effective for zirconia (41).

Evaluating the mode of failure of specimens is important to demonstrate the quality of the bond to treated zirconium and composite resins. In this study, the tested specimens exhibited adhesive failure with the Ultradent and Single Bond repaired specimens, indicating that the Single Bond adhesive and Ultradent repaired specimens obtained a weak bond with the composite. As in previous studies, a higher mean SBS value is related to the predominance of mixed failure modes (45–46).

None of the repair methods resulted in cohesive failures in the zirconia specimens. This may be due to the effect of thermocycling on the bond between zirconia and composite resin.

No significant difference was observed in the mode of bond failure of the Bisco, Ultradent, and Clearfil repair systems. This result may be related to the presence of MDP, which increases the bond between zirconia and composite resin.

This study has the following limitations: the number of thermal cycles was limited, only one surface roughness method was investigated, and the experimental device can not fully simulate the oral environment. Further studies with higher number thermal cycles and those focusing on other types of surface roughness should be performed to provide more reliable information about repair systems.

Conclusion

Within the limitations of the present experimental study, the three repair systems, namely Ivoclar, Clearfil, and Bisco, could be used effectively for repairing chipped veneered porcelain for zirconia infrastructures. The observed failure modes indicates that Ivoclar, Clearfil, and Bisco repair systems could have advantages over others.

Türkçe Özet: *Beş farklı porselen tamir sisteminin Zirkonya alt yapılara bağlanma dayanımının karşılaştırılması. Amaç: Bu çalışmanın amacı, termal siklus sonrası beş farklı porselen tamir sisteminin kompozit ve zirkonya altyapıları arasındaki bağlanma dayanımı üzerindeki etkisini ve meydana gelen ayrılma tiplerini araştırmaktır. Gereç ve Yöntem: Disk şeklinde oluşturulmuş örnekler (n=50), Z'den yapıldı, tamir sistemi tipine göre beş alt gruba (n=10) ayrıldı. Herbir tamir sistemi zirkonyum numunelerine uygulandı ve onarım için hibrit kompozit kullanıldı. Tüm grupların bağlanma dayanımı testleri, termalsiklüs sonrasında Universal test makinesi kullanılarak gerçekleştirildi. Bulgular: Zirkonya tamirinde Single bond tamir sistemi hariç diğer tamir sistemlerinde anlamlı bir farklılık belirlenmedi. Single bond alt yapıların tamirinde en zayıf olarak bulunmuştur. Zirkonya grupları için en yüksek ve en düşük ortalama bağlanma dayanımı değerleri sırasıyla 18.91 MPa ve 3.63 MPa olarak ölçülmüştür. Sonuç: Zirkonya tamirinde üç tamir sisteminin (Ivoclar, Clearfil ve Bisco), Single bond ve Ultradent tamir sistemine göre daha etkili olduğu saptanmış ve ayrılma tipleri mixed ve adeziv olduğu görülmüştür. Anahtar kelimeler: Tamir sistemi; zirkonya; bağlanma dayanımı; adeziv sistem; ayrılma tipleri.*

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Author contributions: SA, FE, and MÇG designed the study. SA, FE, and MÇG generated data for the study. SA, FE, and MÇG gathered data. SA, FE, and MÇG analyzed the data. SA, FE, and MÇG wrote the majority of the original draft of the paper. SA, FE, and MÇG wrote the manuscript. All authors approved the final version of this paper.

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