Micro-Shear Bond Strength of Indirect Composite Resin to Three Different Computer-Milled Cores After Thermocycling

Somayeh Zeighami¹, Safoura Ghodsi¹, Naghmeh Musapoor²

¹ Associate Professor, Dental Research Center, Dentistry Research Institute and Department of Prosthodontics, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran
² Postgraduate Student, Department of Prosthodontics, School of Dentistry, Gilan University of Medical Sciences, Rasht, Iran

Corresponding author: Naghmeh Musapoor, Postgraduate Student, Department of Prosthodontics, School of Dentistry, Gilan University of Medical Sciences, Rasht, Iran
naghmomoosapoor@yahoo.com
Received: 16 May 2020
Accepted: 10 Sep 2020

Abstract

**Background and Aim:** The aim of this study was to evaluate the micro-shear bond strength of an indirect composite resin to three different types of cores.

**Materials and Methods:** In this in-vitro study, 14 blocks (5×5×2 mm) were designed and milled by computer from cobalt-chromium (Co-Cr) alloy, zirconia, and polyether ether ketone (PEEK). Each sample was treated according to the manufacturer’s instructions, and the appropriate primer, and a layer of Crea.lign opaquer were applied on the surface. For micro-shear bond strength test, a plastic tube with an internal diameter of 1 mm and height of 2 mm was placed on each block, and Crea.lign paste was condensed in it. The Crea.lign paste and opaquer were light-cured separately for 180 s, and finally for 360 s. All samples were placed in 37°C distilled water for 24 h and thermocycled for 5000 cycles (5 to 55°C). The microtensile tester machine was used to measure the micro-shear bond strength. The samples were also evaluated for failure modes. Data were analyzed using one-way ANOVA (P<0.05).

**Results:** According to one-way ANOVA, the mean micro-shear bond strength (MPa) in Co-Cr alloy, zirconia, and PEEK groups was 26.09±5.23, 23.49±5.48, and 20.58±5.68 MPa, respectively. There was no significant difference in micro-shear bond strength of the three groups (P= 0.099). The most frequent mode of failure in all three groups was adhesive, followed by mixed, and cohesive.

**Conclusion:** Applying the standard procedure, type of core material had no significant effect on the micro-shear bond strength of Crea.lign composite veneer.

**Key Words:** Chromium Alloys, Composite Resins, Polyetheretherketone, Shear Strength, Zirconium Oxide

Introduction

Metal-ceramic restorations are still used due to their optimal clinical longevity and biocompatibility. This type of prosthesis is mainly used in long-span cases. They benefit from predictable clinical performance, and lower cost (1). However, based on the increasing esthetic demands, the tendency to use metal-free materials has increased over the years. Zirconia is chemically stable in the oral environment, has high fracture resistance, and is known as a metal-free core and an esthetic alternative for metal-based restorations (2). These restorations provide optimal strength and esthetics at the same time (3). The bond
strength of zirconia to layering porcelain is not high enough (2). Recently, a bio-high performance polymer based on polyetheretherketone (PEEK) was introduced to dentistry because of its superior physical and biological properties, good esthetics, and low plaque accumulation. Some applications of PEEK in dentistry include dental implants, implant abutments, healing caps, implant-supported bars, orthodontic bite sticks, and above all, frameworks for fixed partial dentures (4,5). The grayish and opaque color of PEEK necessitates a veneering. Composite resin is the material of choice for veneering of PEEK, but the core-veneer bond strength is low due to the inert chemical performance, low surface energy, and resistance to surface treatment (5).

Layered dental restorations consist of a core and a veneering. Cobalt-chromium (Co-Cr) alloy, zirconia, and PEEK are three materials frequently used as core in fixed restorations. The veneering materials consist of porcelain, polymethyl methacrylate, and composite resin (4). Feldspathic ceramic, a commonly used veneering material, provides optimal abrasion resistance and translucency, with acceptable esthetics and mechanical properties. However, the main drawback of ceramics is their brittleness, and therefore they are susceptible to fracture (6,7). Composite resins have become a suitable alternative to porcelain to diminish impact forces, decrease prosthesis weight, and reduce treatment costs specifically for long-span fixed partial dentures and complete-arch fixed implant-supported prostheses (4,8).

However, the bond strength between the composite veneer and the metal core is relatively low, and it has been considered a major challenge in clinical application of metal resin restorations. Fracture of the veneering material (chipping or delamination) is the most common prosthetic complication in fixed restorations (4). The bond strength of indirect composite to different cores [PEEK (4,5), titanium (4,9), nickel-chromium alloy (Ni-Cr) (8), noble alloy (9,10), zirconia (9,11)] has been evaluated in various studies.

Crea.lign ceramic-filled composite was introduced for veneering of metal, ceramic, and polymer substratures, and its manufacturer claims that it provides permanent chemical bond to all framework materials (12). Thus, application of composite veneering systems has become a research hotspot. There is no study comparing the bond strength of routinely used cores to Crea.lign indirect composite veneers. Since the core-veneer bond strength has high importance in clinical success of bi-layer restorations, this in-vitro study aimed to evaluate the micro-shear bond strength of Crea.lign composite to three types of cores namely Co-Cr alloy, zirconia and PEEK. The null hypothesis was that there would be no significant difference in bond strength of the veneering composite to different core materials.

Materials and Methods
This was an in-vitro study. Details and composition of the materials used in this study are presented in Table 1. According to the results of Yoo et al, (13) considering the standard deviation of 3.51 MPa, $\alpha = 0.5$, $\beta = 0.1$ and effect size=0.676, the minimum sample size in each group was calculated to be 11; however, for more accurate results, 14 samples were considered in each group. Fourteen blocks of each material (Co-Cr, zirconia, and PEEK) were fabricated in 2 mm thickness and 5 mm width and length. Computer-aided design/computer-aided manufacturing system (CORITEC 340i, Imes-icore GmbH, Eiterfeld, Germany) was used for milling of the blocks. Zirconia specimens were sintered in Programat® S1 1600 sintering furnace for 2 h and 55 min and at 1600°C according to the manufacturer’s instructions (ethical code:1396.2077).

The surface of each block was polished with a series of #600, #700, and #800 silicon-carbide abrasive papers (Struers, RotoPol 11, Struer A/S Rodovre, Denmark) for 1 min, and ultrasonically cleaned with distilled water (Quantrex 90 WT, L&R Manufacturing Inc., Kearny, NJ, USA) for 6 min. The samples were sandblasted with 110 µm aluminum oxide particles at 45° angle from 3 cm distance at 4
Table 1. Materials used in this study

| Material       | Brand          | Manufacturer                                      | Composition                                                                 |
|----------------|----------------|---------------------------------------------------|-------------------------------------------------------------------------------|
| PEEK           | BioHPP         | Bredent GmbH & Co.KG, Witzighausen, Germany       | Ceramic filled (20%) PEEK                                                     |
| Zirconia       | IPS e.max ZirCAD LT | Ivoclar Vivadent, Schaan, Liechtenstein          | ZrO2 (88.0 - 95.5%), Y2O3 (> 4.5 ≤ 6.0%), Al2O3 (≤ 1.0%), Other oxides (≤1.0%) |
| Co-Cr          | CORiTEC CoCr Disc | Imes-icore GmbH, Eiterfeld, Germany               | Co (>62%), Cr(>28%), W(>8.45%), Si(>1.65%), Mn, Fe, C (>0.5%)               |
| Primer         | Visio. Link    | Bredent GmbH & Co.KG, Witzighausen, Germany       | MMA, PETIA, Photo-initiators                                                  |
| Primer         | MKZ primer     | Bredent GmbH & Co.KG, Witzighausen, Germany       | Ethanol                                                                      |
| Composite       | Crea.lign opaquer | Bredent GmbH & Co.KG, Witzighausen, Germany     | bis-GMA composite with microfillers                                          |
| Composite paste | Crea.lign paste | Bredent GmbH & Co.KG, Witzighausen, Germany       | bis-GMA composite with microfillers                                          |

In the next step, all the specimens were coated with 0.1 mm Crea.lign opaquer, and polymerized for 180 s. A plastic tube with 1 mm internal diameter and 2 mm height was placed on each core sample, filled with Crea.lign paste, and cured for 180 s. Finally, both Crea.lign opaquer and Crea.lign paste were cured for 360 s. All the procedures were performed according to the manufacturer’s instructions and by an expert technician. Plastic tubes were removed, and all the specimens were placed in distilled water at 37°C for 24 h to complete the polymerization, and the samples were thermocycled for 5000 cycles between 5 to 55°C with a dwell time of 15 s and a transfer time of 10 s, which was equivalent to 6 months of clinical function (14). In order to evaluate the micro-shear bond strength, a micro tensile tester machine (Bisco Inc., USA) was employed at a speed of 1 mm/min. By dividing the resulted quantity by the cross-section of each sample ($\pi r^2=0.785$), the bond strength was calculated in megapascals (MPa) according to the formula $S= F (N)/A$.

The broken specimens were checked under a stereomicroscope (SZX9; Olympus, Japan) at x4 magnification to evaluate the failure mode, which was divided into cohesive (in Crea.lign composite), adhesive (between composite and block), or mixed (combined fracture) (15,16) (Figures 1-3).

Data were analyzed by one-way ANOVA using SPSS version 25 (SPSS Inc., IL, USA) at $P< 0.05$.

Results
Fourteen samples were investigated for microshear bond strength and failure mode in
each experimental group. The results of microshear bond strength are summarized in Table 2. Although Co-Cr core showed greater bond strength compared with other groups, one-way ANOVA showed no statistically significant difference between the groups (P=0.099). All microshear bond strength values were within the acceptable clinical range (5 MPa). The frequency of failure modes is summarized in Table 3. In all groups, adhesive failure accounted for most of the failures, followed by mixed and cohesive failures. The failure modes of the three groups were compared using the Fisher's exact test. There was no significant difference in failure modes between the groups (P=0.947).

**Discussion**
One of the most common in-vitro tests for dental materials is evaluation of bond strength.
Table 2. Mean ± standard deviation of microshear bond strength in Co-Cr, zirconia, and PEEK (n=14)

| Group   | Mean (MPa) ± std. deviation | Minimum (MPa) | Maximum (MPa) |
|---------|-----------------------------|---------------|---------------|
| Co-Cr   | 26.09 ± 5.23                | 20.84         | 35.16         |
| Zirconia| 23.49 ± 5.48                | 15.13         | 31.05         |
| PEEK    | 20.58 ± 5.68                | 14.33         | 33.04         |

Table 3. Frequency of failure modes in different groups

| Group   | Adhesive Count % | Mixed Count % | Cohesive Count % |
|---------|------------------|---------------|------------------|
| Co-Cr   | 11 (78.5%)       | 2 (14.3%)     | 1 (7.2%)         |
| Zirconia| 10 (71.4%)       | 3 (21.4%)     | 1 (7.2%)         |
| PEEK    | 9 (64.3%)        | 3 (21.4%)     | 2 (14.3%)        |
| Total   | 29 (69%)         | 9 (21.4%)     | 4 (9.6%)         |

Since many of the failures in the oral cavity arise from shear and tensile loadings, these forces have the most common application in bonding evaluations (17,18). The main purpose of this study was to determine the microshear bond strength of Crea.lign indirect composite veneer to three different types of cores namely Co-Cr, zirconia, and PEEK. The null hypothesis was confirmed since the difference in bond strength between the three groups was not statistically significant (P>0.05).

The mean microshear bond strength of Co-Cr, zirconia, and PEEK groups was reported to be 26.09±5.23, 23.49±5.48, and 20.58±5.68 MPa, respectively. The shear bond strength of zirconia core to various types of composite veneers under the same surface preparations has been reported to be 9.5 to 13.7 MPa in different studies (19-22). While, for Ni-Cr alloy and PEEK core, the composite veneer bond strength was measured between 10.04 to 16.55 MPa (8,23,24), and 5.3 to 13.5 MPa (5,25,26), respectively. In the present study, the bond strength values were almost twice the previous rates in all three groups, which could be related to the type of evaluation made. Considering the smaller structural defects in micro samples used for micro-shear tests (27), the bond strength is expected to be 2-3 times higher than the amount obtained in shear bond strength test. Furthermore, Visio.link contains acrylates like pentaerythritol triacrylate or pentaerythritol tetracrylate. These materials are more reactive compared with methacrylates (28), and can potentially increase the chemical bond strength.

According to the standards for dentistry-polymer-based crown and veneering materials (International Organization for Standardization, 2018), the minimum core to veneer shear bond strength should be 5 MPa to achieve clinical satisfaction (29). Considering this evidence, it can be concluded that the results were within the clinically acceptable range. The shear bond strength of zirconia to ceramic has been reported between 23.3 to 26.9 MPa (19), which...
is in line with the present study with the difference that, in the present study the micro-shear bond strength test was performed. This comparison shows that zirconia has almost the same bond strength to ceramic and composite veneers through micromechanical bonding. In a study by Hatta et al, the bond strength of Nobel Rondo Zirconia Dentin as layering porcelain to zirconia blocks polished by #600 papers was reported to be 23.3 MPa (19), which was close to the result of the current study. It can be concluded that proper surface treatment can provide similar bond strength of indirect composite to zirconia compared with the veneering porcelain. However, the shear bond strength of Ni-Cr to veneering ceramic was reported to be 42.9 MPa in a previous study (8), which was significantly higher than that of Crea.lign composite in the present study. This result can be justified based on the formation of oxide layer on the metal surface (by firing in an oxidizing atmosphere) which has the most important role in chemical bonding between metal and ceramic (30). Therefore, higher bond strength to metal is expected in ceramic compared with composite resin.

The most frequent mode of failure was adhesive in all three groups, and the lowest was cohesive which was in accordance with other studies (9,19-22). Komine et al. reported no cohesive failure between indirect composite and zirconia in a number of studies, and the most frequent type of failure was reported to be the adhesive type (9,20,22). Other studies reported adhesive type of failure as the most prevalent failure type in bonding of veneering composite to zirconia (19,21). However, there are studies with different results. In studies on Ni-Cr, the failure mode of samples was reported to be 75%-100% cohesive, and 0%-25% adhesive (8,23,24). The frequency of failure modes in PEEK samples was reported to be 44-43% adhesive, 50-87% cohesive, and 30% mixed (5,25,26). These controversial results can be attributed to the difference in the type of materials, and bond strength tested (27). Previous studies analyzed the bond strength of either direct composites (19-22), or resin cement (8,23,24). Crea.lign is a laboratory indirect ceramic-filled composite with high degree of conversion, and higher structural strength (16), that causes less cohesive failure within the material mass.

According to a study by Komine et al, thermocycling significantly decreased the shear bond strength, and the maximum mean value of bond strength in zirconia specimens was 12.1 MPa; also, tendency to cohesive failure increased after 20,000 thermal cycles (9). The different results of their study compared with the current study could be due to the difference in the number of thermal cycles and type of surface preparation and materials.

The present study confirmed the reliability of bond strength between Crea.lign ceramic-reinforced composite veneer and different computer-milled core materials. The limitations of this in-vitro study were related to the technical sensitivity of the microshear bond strength test, and inability to completely simulate the clinical condition. Further studies focusing on different surface treatments, and different types of laboratory and clinical composites are suggested.

**Conclusion**

Given the limitations of the current study, the following conclusions could be drawn:

1. The highest microshear bond strength value was noted in Co-Cr group, followed by zirconia, and PEEK groups.
2. There was no significant difference in microshear bond strength of the groups. The microshear bond strength of indirect composite was not affected by the type of core material. In all three groups, adhesive failure was more prevalent than mixed and cohesive failure modes.

**Acknowledgement**

This study was financially supported by the Dental Research Center, Dentistry Research Institute, Tehran University of Medical Sciences, Tehran, Iran (Grant No: 95-03-70-34238). This study was derived from a DDS thesis by Dr. Naghmeh Musapoor (No: 6228).

**References**

1. Motta AB, Pereira LC, da Cunha AR. All-ceramic and porcelain-fused-to-metal fixed...
partial dentures: a comparative study by 2D finite element analyses. J Appl Oral Sci. 2007 Oct; 15(5):399–405.
2. Zeighami S, Mahgoli H, Farid F, Azari A. The effect of multiple firings on microtensile bond strength of core veneer zirconia-based all-ceramic restorations. J Prosthodont. 2013 Jan;22(1):49–53.
3. Tabatabaian F. Color in zirconia-based restorations and related factors: a literature review. J Prosthodont. 2018;27(2):201–11.
4. Jin HY, Teng MH, Wang ZJ, Li X, Liang JY, Wang WX, et al. Comparative evaluation of BioHPP and titanium as a framework veneered with composite resin for implant-supported fixed dental prostheses. J Prostheth Dent. 2019 Oct;122(4):383–8.
5. Zhou L, Qian Y, Zhu Y, Liu H, Gan K, Guo J. The effect of different surface treatments on the bond strength of PEEK composite materials. Dent Mater. 2014;30(8):e209–15.
6. Sreekala L, Narayanan M, Eerali SM, Eerali SM, Varghese J, Zainaba Fathima AL. Comparative evaluation of shear bond strengths of veneering porcelain to base metal alloy and zirconia substructures before and after aging - An in vitro study. J Int Soc Prev Community Dent. 2015;5(Suppl 2):S74–81.
7. Mattiello RDL, Coelho TMK, Insaurralde E, Coelho AAK, Terra GP, Kasuya AVB, et al. A review of surface treatment methods to improve the adhesive cementation of zirconia-based ceramics. ISRN Biomaterials. 2013:1–10.
8. Machado AL, Pavarina AC. Shear bond strength of aesthetic materials bonded to Ni-Cr alloy. J Dent. 2003;31(3):205–11.
9. Komine F, Koizuka M, Fushiki R, Taguchi K, Kamio S, Matsumura H. Post-thermocycling shear bond strength of a gingiva-colored indirect composite layering material to three implant framework materials. Acta Odontol Scand. 2013 Sep;71(5):1092–100.
10. Petridis H, Garefis P, Hirayama H, Kafantaris NM, Koidis PT. Bonding indirect resin composites to metal: Part 1. Comparison of shear bond strengths between different metal-resin bonding sand a metal-ceramic system. Int J Prosthodont. 2003 Nov-Dec; 16 (6):635–9.
11. Kubochi K, Komine F, Fushiki R, Yagawa S, Mori S, Matsumura H. Shear bond strength of a denture base acrylic resin and gingiva-colored indirect composite material to zirconia ceramics. J Prosthodont Res. 2017 Apr;61(2):149–57.
12. Crea.lign freestyle manual. Senden-Germany: the bredent group; 2019. available at: https://www.visiolign.com/crealign/
13. Yoo JY, Yoon HI, Park JM, Park EJ. Porcelain repair - Influence of different systems and surface treatments on resin bond strength. J Adv Prosthodont. 2015 Oct;7(5):343–8.
14. Morresi AL, D’Amario M, Capogreco M, Gatto R, Marzo G, D’Arcangelo C, et al. Thermal cycling for restorative materials: does a standardized protocol exist in laboratory testing? A literature review. Mech Behav Biomed Mater. 2014 Jan; 29:295–308.
15. Kobayashi K, Komine F, Blatz MB, Saito A, Koizumi H, Matsumura H. Influence of priming agents on the short-term bond strength of an indirect composite veneering material to zirconium dioxide ceramic. Quintessence Int. 2009 Jul-Aug;40(7):545–51.
16. Su N, Yue L, Liao Y, Liu W, Zhang H, Li X, et al. The effect of various sandblasting conditions on surface changes of dental zirconia and shear bond strength between zirconia core and indirect composite resin. J Adv Prosthodont. 2015 Jun;7(3):214–23.
17. Sirisha K, Rambabu T, Shankar YR, Ravikumar P. Validity of bond strength tests: A critical review: Part I. J Conserv Dent. 2014;17 (4):305–11.
18. Sirisha K, Rambabu T, Ravishankar Y, Ravikumar P. Validity of bond strength tests: A critical review-Part II. J Conserv Dent. 2014 Sep-Oct; 17(5):420–426.
19. Hatta M, Shinya A, Yokoyama D, Gomi H, Valliuttu PK, Shinya A. The effect of Surface treatment on bond strength of layering porcelain and hybrid composite bonded to zirconium dioxide ceramics. J Prosthodont Res. 2011 Jul;55(3):146–53.
20. Komine F, Koizuka M, Fushiki R, Iwasaki T,
Kuboči K, Matsumura H. Effect of various surface preparations on bond strength of a gingiva-colored indirect composite to zirconia framework for implant-supported prostheses. Dent Mater J. 2015;34(3):402-9.

21. Shimoe S, Tanoue N, Kusano K, Okazaki M, Satoda T. Influence of air-abrasion and subsequent heat treatment on bonding between zirconia framework material and indirect composites. Dent Mater J. 2012;31(5):751-7.

22. Komine F, Fushiki R, Koizuka M, Taguchi K, Kamio S, Matsumura H. Effect of surface treatment on bond strength between an indirect composite material and a zirconia framework. J Oral Sci. 2012;54(1):39-46.

23. Seimenis I, Sarafianou A, Papadopoulou H, Papadopoulos T. Shear bond strength of three veneering resins to a Ni-Cr alloy using two bonding procedures. J Oral Rehabil. 2006 Aug; 33(8):600-8.

24. Ciftçi Y, Canay S, Hersek N. Shear bond strength evaluation of different veneering systems on Ni-Cr alloys. J Prosthodont. 2007 Jan-Feb; 16(1):31-6.

25. Schwitalla AD, Botel F, Zimmermann T, Sütel M, Müller WD. The impact of argon/oxygen low-pressure plasma on shear bond strength between a veneering composite and different PEEK materials. Dent Mater. 2017 Sep; 33(9): 990-4.

26. Schmidlin PR, Stawarczyk B, Wieland M, Attin T, Hämmerle CH, Fischer J. Effect of different surface pre-treatments and luting materials on shear bond strength to PEEK. Dent Mater. 2010 Jun; 26(6):553-9.

27. Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: a critical literature review. Dent Mater. 2010 Feb; 26(2):e78-93.

28. Stawarczyk B, Krawczuk A, Ilie N. Tensile bond strength of resin composite repair in vitro using different surface preparation conditionings to an aged CAD/CAM resin nanoceramic. Clin Oral Investig. 2015 Mar; 19(2):299-308.

29. International Organization for Standardization. (2018). Dentistry - Polymer-based crown and veneering materials_ (ISO/DIS Standard No. 10477:2018). Retrieved from https://www.iso.org/standard/68235.html.

30. Abrisham SM, Fallah Tafti A, Kheirkhah S, Tavakkoli MA. Shear bond strength of porcelain to a base-metal compared to zirconia core. J Dent Biomater. 2017 Mar;4(1):367-72.