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

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#### 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 ( $5 \times 5 \times 2$  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  $26.09\pm5.23$ ,  $23.49\pm5.48$ , and  $20.58\pm5.68$  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

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#### 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

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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, abutments, implant healing caps, implant-supported bars, orthodontic bite sticks. above frameworks and all, 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 longspan 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 substructures, 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/computeraided 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  $\mu$ m aluminum oxide particles at 45° angle from 3 cm distance at 4

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%), HfO2 (≤5.0%), Al2O3 (≤1.0%), Other oxides (≤1.0%)
Co-Cr	CORiTEC CoCr Disc Non-precious alloy	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 opaque	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

#### Table 1. Materials used in this study

bar pressure for zirconia and PEEK, and 2 bar pressure for Co-Cr. Afterwards, the surfaces were cleaned using a brush and alcohol. MKZ primer was applied on the surface of zirconia and Co-Cr blocks, and allowed to evaporate for 30 s. For PEEK, Visio.link primer was used and cured (Labolight LV-III) for 90 s (300-400 nm). 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 ( $\pi$ r2=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

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Figure 1. Cohesive failure type in Co-Cr (A), PEEK (B), and zirconia (C) groups



Figure 2. Adhesive failure type in Co-Cr (A), PEEK (B), and zirconia (C) groups



Figure 3. Mixed failure type in Co-Cr (A), PEEK (B), and zirconia (C) groups

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.

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

**Table 2.** Mean ± standard deviation of microshear bond strength in Co-Cr, zirconia, and PEEK (n=14)

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

**Table 3.** Frequency of failure modes in different groups

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  $26.09\pm5.23$ ,  $23.49\pm5.48$ , and  $20.58\pm5.68$  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 Furthermore, Visio.link test. 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 dentistrypolymer-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

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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 ceramicreinforced 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.

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