Evaluation of Fracture Resistance and Color Stability of Crowns Obtained by Layering Composite Over Zirconia and Polyetheretherketone Copings Before and After Thermocycling to Simulate Oral Environment: An In Vitro Study

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Background: Most common material used for the fabrication of an implant restoration is full-ceramic crown or an all-ceramic crown. Frequent chipping of the ceramic under occlusal load has posed a great problem to the clinician and the patient. Composites have been layered over zirconia successfully in the recent past to overcome this problem. This study, thus, aimed to evaluate and compare the fracture resistance and color stability of crowns obtained by layering composite over zirconia and polyetheretherketone (PEEK) copings before and after thermocycling to simulate oral environment. Materials and Methods: A total of 40 crowns (20 per group) were obtained by layering composite of A3 shade over computer-aided design/computer-aided milling milled zirconia and PEEK copings. Thermocycling of the 10 out of the 20 crowns was performed in a thermocycler (5000 cycles, in water temperature of 5°C and 55°C with dwell time of 30 s), and then they were kept in hot and cold beverages for 24 h each, to simulate oral environmental conditions. After thermocycling, the crowns were divided into four groups of 10 samples each: Group Z, ZT, P, and PT. Shade evaluation of all the crowns was performed using digital shade guide (VITA Easyshade® Advance; VITA Zahnfabrik, Bad Säckingen, Germany) and VITAPAN classical (VITA Zahnfabrik). Fracture strength was tested for all the crowns in a universal testing machine. Fracture strength in megapascal and the applied occlusal load in kilograms were recorded. Data obtained were statistically evaluated by one-way analysis of variance and post hoc test. Results: The final shade of the crowns obtained by layering A3 shade composite in the groups Z was A3, ZT was B3, P was C3, and PT was D2. The value of mean fracture strength of crowns of groups Z was 1142.3 MPa, ZT was 1034.57 MPa, P was 2134.64 MPa, and PT was 1765.01 MPa. Conclusion: Thermocycling affected the shade of all the crowns. The mean fracture strength of the crowns having PEEK copings was significantly higher than that of zirconia copings. Thermocycling did not have a significant effect on the mean fracture strength.

Keywords: CAD/CAM, layered composites, PEEK, zirconia

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performance of removable prosthesis and predictable successful results of the implant-supported prostheses.[1] To rehabilitate implants with full contoured restorations, various materials have been used such as precious and non-precious alloys, acrylic, metal acrylic, metal ceramic, and porcelain. Metal has its color as the biggest disadvantage; chipping of ceramic is the most common problem with metal ceramic and all ceramic crowns. Acrylic resin alone and its modifications have also been used, but due to poor mechanical properties its use is now limited to provisional restorations. Computer-aided design and computer-aided milling (CAD/CAM) has made it possible to mill newer materials precisely and accurately such as titanium, zirconia, high-performance ceramics, and polymers. It has already changed the scenario of dentistry and is replacing more and more of the traditional or conventional techniques in the fabrication of dental restorations. Crown and bridge fabrication using CAD/CAM is gaining popularity in dentistry. Zirconia is a crystalline dioxide of zirconium. Its mechanical properties are very similar to those of metals and its color is similar to the tooth. In 1975, Garvie proposed a model to rationalize the good mechanical properties of zirconia, by virtue of which it has been called “ceramic steel.” The use of zirconia in dentistry dates to more than two to three decades. Zirconia layered with ceramic is very hard and does not absorb the occlusal loads, sometimes resulting in a fractured prosthesis. Failures in zirconia-veneered restorations are due to a very weak porcelain material (flexural strength of 90 MPa) having masticatory forces exerted upon it. The zirconia substructure with a flexural strength of 1000 MPa remains intact, but the failure of the layering porcelain ultimately causes a failure of the restoration. To overcome this, layering of composite resin on the zirconia substructure with the help of a primer has been tried successfully in the recent past.[2]

Polyetheretherketone (PEEK), a relatively newer material, has emerged as a promising alternative in implant prosthetics. It is a linear, aromatic, semicrystalline thermoplastic polymer and belongs to the family polyaryletherketone. It has excellent chemical resistance and biomechanical properties. In its pure form, Young’s modulus of PEEK is around 3.6 GPa. But when carbon reinforced (CFR-PEEK), it can go up to 19–150 GPa. Its tensile strength is 110 MPa and compressive strength is 138 MPa. It is a very lightweight material with low molecular weight.[3] These properties make PEEK an ideal material for implant prosthesis. But the problem with PEEK is that it is snow-white or grayish in shade and thus has to be layered with some tooth-colored material like composite.

Zirconia layered with ceramic or composite is used extensively as an implant prosthesis. The absence of periodontal fibers around the implant makes it more rigid; the forces acting on them are greater than that on the natural tooth. And a material as hard as zirconia will transfer more amount of load to the underlying implant. Also zirconia layered with ceramic being very hard, there is repeated frequent chipping or fracture of the ceramic layer. To overcome this disadvantage of ceramic, layering of composite over zirconia coping was tried, and as literature suggests, the performance in terms of fracture strength of these crowns was at par with that of zirconia ceramic crown.[4,5] The advantage of these crowns was that it had the strength of the zirconia substructure and a resilient layering of composite. It also had the advantage of easy handling and intraoral repair.[6] A material with plastic properties, elastic modulus comparative to bone, and adequate strength has the potential to come out as an ideal material for implant prosthesis. The previously mentioned properties of PEEK suggest that it can prove to be a better material than zirconia when used as coping. Framework for implant-supported prosthesis has been successfully fabricated using PEEK in the recent past. But there is paucity of literature on the use of PEEK as a coping material layered with an aesthetic material like composite. So this study will be evaluating and comparing the fracture resistance and shade of composite veneered over PEEK copings to that of zirconia copings, before and after subjecting to artificial oral environmental conditions.

**Aims and objectives**

The aim of this study was to evaluate and compare the fracture resistance and color stability of crowns obtained by layering composite over zirconia and PEEK copings before and after thermocycling to simulate oral environment.

**Settings and design**

*In vitro* study.

**Materials and Methods**

A stainless-steel master die of 6mm diameter at the occlusal surface and 10mm at the margin was fabricated to simulate a prepared lower molar tooth. Its one surface was flat to incorporate anti-rotational feature. The axial surface of the master die had a taper of 6° and a height of 7.0mm. The finish line was designed as a deep chamfer having curvature radius of 1.0mm [Figure 1]. The die was sprayed with scanning spray Telescan (DFS, Germany)
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and then scanned with a CAD/CAM scanner (Ceramill® Map 400 scanner; Amann Girbach, Koblach, Austria). A total of 40 copings, 20 copings of 0.5 mm thickness of each zirconia (Group Z) and PEEK (Group P), were designed using Ceramill Mind software (Amann Girbach). This design was nested on to 12 mm Ceramill zirconia blanks (Ceramill CAD/CAM Material—ZI 12 mm; Amann Girbach) and 13 mm Ceramill PEEK blank (Ceramill CAD/CAM Material; Amann Girbach). Dry milling for the zirconia copings and wet milling for the PEEK copings were performed using a milling machine (Ceramill Motion 2; Amann Girbach) [Figure 2]. Once the milling was completed, the copings were de dusted and the zirconia copings were placed inside the sintering furnace (Ceramill Therm; Amann Girbach) and a sintering cycle of 12 h was performed with an average rise in temperature of 8°C/min and peak temperature of 1450°C and holding time of 2 h. Then the copings were sand-blasted in a sand blasting unit (Renfert). After that it was cleaned in an ultrasonic cleanser for 1 min. AZ-primer (Shofu, Koyoto, Japan, San Marcos, CA) was applied on all the surfaces of the zirconia copings using an applicator tip and was left to dry for 10 s, and Cera Resin Bond (CRB)-1 (Shofu) was applied over PEEK copings and was left to dry for 10 s. CRB-2 was applied after that and was light cured in the light cure unit (Labolight LV®; GC) for 20 s. Layering of an A3-shade composite (Ceramage; Shofu) over the copings was performed to achieve a uniform thickness of 2 mm with the help of a clear shell [Figure 3]. It was cured in the light cure unit for 30 s. Polishing of the obtained sample was carried out using sandpaper strips and rubber discs (Soflex spiral). Shade evaluation of the obtained sample was compared by layering composite over zirconia and PEEK was performed using digital shade guide (VITA Easyshade® Advance; VITA Zahnfabrik, Bad Säckingen, Germany) [Figure 4]. A manual evaluation of the shade was also conducted using VITAPAN classical (VITA Zahnfabrik) shade guide [Figure 5]. To mimic oral environmental conditions, thermocycling of 10 out of 20 crowns of zirconia and PEEK copings was carried out. The samples were exposed to 5000 thermal cycles between 5°C and 55°C water temperature and a dwell time of 30 s per bath in a thermocycler (BIOBEE®). After that, the crowns were kept in hot and cold beverages for 24 h each. After thermocycling, the crowns of the zirconia and PEEK coping group were termed as Group ZT and PT, respectively. Evaluation of shade was performed again to check the effect of thermocycling on it. For fracture resistance test, the master die with the sample was mounted on a universal testing machine (Tec-Sol India, Chennai, India), and a stainless-steel ball indenter of 9.0 mm in diameter was placed on the occlusal aspect with a crosshead speed of 1.0 mm/min. Compressive load was applied till the samples fractured [Figure 6]. Reading was obtained from universal testing machine software. Fracture strength in megapascals and the applied occlusal load in kilograms were recorded when the samples fractured under compression.

**Statistical analysis used**

SPSS software, version 23.0 (IBM, Armonk, NY) was used for analyzing the data. Mean and standard deviation of fracture strength values of all groups were obtained. Independent t test was performed to check for fracture strength within the group. One-way analysis of variance (ANOVA) was conducted to check for significant difference of fracture strength between the test groups. Tukey’s post hoc test was performed to check for intergroup significance of fracture strength.

**Results**

This study compared the shade and fracture resistance of composite layered over zirconia copings to that of PEEK copings. The groups were further divided on the basis of thermocycling as Group Z (without thermocycling), Group ZT (after subjecting to thermocycling). The groups were further divided on the basis of thermocycling as Group Z (without thermocycling), Group ZT (after subjecting to thermocycling), Group P (without thermocycling), and Group PT (after subjecting to thermocycling). The results showed that the fracture strength of zirconia crowns was significantly higher than that of PEEK crowns. The fracture strength of zirconia crowns was also significantly higher after thermocycling.

**Figure 1:** A metallic master die simulating a prepared lower molar
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thermocycling), Group P (without thermocycling), and Group PT (after subjecting to thermocycling). The final shade of all the crowns was checked using digital shade guide (VITA Easyshade Advance) and VITAPAN classical shade guide, after layering with composite of A3-shade [Table 1]. To observe the effect of thermocycling, the shade after thermocycling was also checked. The comparison of shade between the Group Z and Group ZT; Group P and Group PT was performed.

Figure 2: Coping design (Ceramill Mind software)

Figure 3: Layered composite to obtain a uniform crown of 2 mm

Figure 4: Digital shade guide (VITA Easyshade Advance)
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Highest mean load and fracture strength were recorded in composite layered over PEEK copings (Group P), followed by composite layered over PEEK copings after thermocycling (Group PT), composite layered over zirconia copings (Group Z), and then composite layered over zirconia copings after thermocycling (Group ZT; [Table 2] and [Graphs 1 and 2]).

Independent sample t test was performed to compare load and strength within the groups.

As P value is >0.05, there is no significant difference within the groups.

One-way ANOVA was performed to compare load and strength between the groups (Group Z and P). And it was observed that there is a significant difference in load and strength of composite layered over zirconia copings to that of PEEK copings.

**DISCUSSION**

This study was conducted in CAD/CAM dental laboratory of Yenepoya Dental College, Yenepoya Deemed to be University, Karnataka, India, to evaluate and compare the fracture resistance and color stability of crowns obtained by layering composite over zirconia and PEEK copings before and after thermocycling to simulate oral environment.

The evaluation of shade was performed for all the groups (control groups [Group Z and Group P] and test groups [Group ZT and Group PT]) using a digital shade guide (VITA Easyshade Advance) and was noted. For confirmation of the shade, a manual evaluation of all the samples was also performed using VITAPAN classical shade guide by three different experienced evaluators. The final shade of the crowns obtained by layering an indirect composite of A3 shade over zirconia copings was A3 and B3 in the control group (Group Z) and the test group (Group ZT), respectively. The final shade of the crowns obtained by layering an indirect composite of A3 shade over PEEK copings was C3 in the control group (Group P) and D2 in the test group (Group PT). The reason for the change in shade of the crowns obtained by layering of composite over PEEK copings could be because of the innate grayish color of the PEEK itself. There was a change observed in the shade of the samples after thermocycling, which was because of the nature of the layered composite to absorb stains and discolor when kept in various beverages. Several studies conducted by Douglas,[8] Nasim et al.,[9] Erdemir et al.,[10] and Al Kheraif et al.[11]...
show that composite takes up stain when kept in some media or beverages.

After the evaluation and comparison of shade, all the samples were subjected to compressive load to test their fracture strength. It was observed that there is statistically significant difference between the mean fracture strength of the crowns obtained by layering composite over zirconia copings to that of PEEK copings. Samples of the control group of the crowns obtained by layering composite over PEEK copings (Group P) had the highest mean fracture strength of 2134.64 MPa when a mean load of 217.67 kg was applied. These findings are comparable to the mean fracture strength of 2354 N for a three-unit fixed partial denture of PEEK layered with composite in a study conducted by Stawarczyk et al.\cite{13}

The mean fracture strength for the test group of the crowns obtained by layering composite over zirconia copings (Group ZT) was 1034.57 MPa under mean load of 105.5 kg, and for the test group of the crowns obtained by layering composite over PEEK copings (Group PT) it was 1765.01 MPa, when a mean load of 179.98 kg was applied. There was a decrease in values of the mean fracture strength observed in both the groups when compared to their control groups (Group Z and Group P), respectively, but it was not statistically significant. So within the groups, there was no statistically significant difference in the fracture resistance. This is in accordance with the findings of Komine et al.\cite{2} and Komine et al.\cite{6}, who concluded in their study that there is no effect of thermocycling on the fracture strength of zirconia primed to composite, and Stawarczyk et al.\cite{14} who concluded that there is no change in fracture strength of composite veneered to PEEK after subjecting to thermal aging in an artificial environment. Overall the mean fracture strength of crowns obtained by layering composite over PEEK copings was higher than that of zirconia copings.

In the control and test groups (Group Z and Group ZT) of crowns obtained by layering composite over zirconia copings, all the samples fractured along with the copings, suggestive of cohesive failure. In the control group (Group P) of crowns obtained by layering composite over PEEK copings, only 1 out of the 10 samples showed both adhesive and cohesive failure. In the test group (Group PT) of crowns obtained by layering composite over PEEK copings, 4 out of the 10 samples showed cohesive failure. So in the composite layered over PEEK copings, the fracture is mostly due to adhesive failure or the cohesive failure of the composite itself, which is in accordance with the findings of a study done by Fuhrmann et al.\cite{15} Kern and Lehmann,\cite{16} Silthampitag et al.\cite{17} and Stawarczyk et al.\cite{18} Even though there is no delamination of composite layered over zirconia copings, the substructure itself is getting fractured, whereas composite layered over PEEK copings shows failure mostly at the interface. There is less fracture of the PEEK substructure, even on application of almost approximately double the load

| Groups | Mean load (SD) | Mean strength (SD) |
|--------|---------------|--------------------|
| Z      | 116.49 (36.05)| 1142.3 (353.58)    |
| ZT     | 105.50 (15.48)| 1034.57 (151.84)   |
| P      | 217.67 (49.15)| 2134.64 (481.99)   |
| PT     | 179.98 (77.19)| 1765.01 (756.95)   |

SD = standard deviation
\( ^a \text{in kg} \)
\( ^b \text{in MPa} \)

Graph 1: Bar chart showing mean load applied on the four different groups

Graph 2: Bar chart showing mean fracture strength of the four different groups

Table 2: Descriptive statistics of mean and standard deviation of load and strength
applied to fracture the crowns of composite layered over zirconia copings. The reason for non-fracture of most of the PEEK copings could be due to the flexible nature of the material.

The crowns were obtained by layering composite over the zirconia or PEEK substructure to make them aesthetic. The other advantage of this was the strength of the substructure and a resilient layering of composite. It also had the advantage of easy handling and intraoral repair, making them ideal crown and bridge material for implants. The crown obtained by layering composite over PEEK copings can be used as implant restoration in the non-aesthetic posterior region, where the occlusal forces are heavier. Further studies are needed to improve the color properties of PEEK to use it as a coping material. BioHPP (Bredent, Chesterfield, UK) has a white shade, which makes it more closer to that of dentin. It can be tried as a coping material in an implant restoration in the aesthetic zone.

The following were the limitations of this study:

- The shade of the PEEK material used in this study was grayish.
- The load applied by the universal testing machine does not simulate the clinical loading condition.
- The thermocycling procedure performed does not exactly simulate the oral environmental condition.
- The primers used to bond composite over PEEK and zirconia copings were different in composition and properties. So the bond strength was also different in the two groups.

**CONCLUSIONS**

Within the limitations of this in vitro study, the following conclusions can be drawn:

- Thermocycling affects the shade of crowns obtained by layering composite over zirconia copings and crowns obtained by layering composite over PEEK copings.
- The mean fracture strength of crowns obtained by layering composite over PEEK copings was significantly higher than that of crowns obtained by layering composite over zirconia copings.
- Thermocycling does not affect the fracture strength of crowns obtained by layering composite over zirconia copings and that of crowns obtained by layering composite over PEEK copings.

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**Conflicts of interest**

There are no conflicts of interest.

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