Three-unit reinforced polyetheretherketone composite FDPs: Influence of fabrication method on load-bearing capacity and failure types

Bogna STAWARCZYK1, Marlis EICHBERGER1, Julia UHRENBACHER1, Timea WIMMER1, Daniel EDELHOFF1 and Patrick R. SCHMIDLIN2

1 Department of Prosthodontics, Dental School, Ludwig-Maximilians-University Munich, Goethestrasse 70, 80336 Munich, Germany
2 Clinic of Preventive Dentistry, Periodontology and Cariology, Center of Dental Medicine, University of Zurich, Plattenstrasse 11, 8032 Zurich, Switzerland

To investigate the influence of different fabrication methods of three-unit reinforced polyetheretherketone composite (PEEK/C) fixed dental prostheses (FDPs) on fracture load. Forty-five three-unit anatomically supported PEEK/C FDPs were fabricated as follows: i. milled using a CAD/CAM system from an industrially fabricated PEEK/C blank, ii. pressed from industrially fabricated PEEK/C pellets, and iii. pressed from granular PEEK/C. Fracture load was measured and data were statistically analysed (p<0.05). CAD/CAM fabricated FDPs (2,354 N) presented a higher mean fracture load than those pressed from granular PEEK/C material (1,738 N) (p<0.001). CAD/CAM milled FDPs and those pressed from PEEK/C-pellets showed spontaneous and brittle fractures near the pontic without deformation of the FDP. In contrast, granulate pressed FDPs showed some plastic deformation without fracture. CAD/CAM fabricated FDPs, and FDPs pressed from PEEK/C pellets showed higher Weibull moduli compared to FDPs pressed in granular form. Industrial pre-pressing of blanks (CAD/CAM/pellet) increased the stability and reliability of PEEK restorations.

Keywords: PEEK/C, FDPs, CAD/CAM, press technique, fracture load

INTRODUCTION

Due to their specific mechanical properties including, e.g. damping effects, composite-based reconstructions are finding increased application, especially in implant borne restorations1,2). Industrial manufacturing of CAD/CAM (Computer Aided Design/Computer Aided Manufacturing) blanks for composite and PMMA-based reconstructions was shown to significantly improve mechanical properties3-5). Especially CAD/CAM composites can be applied as alternative materials to ceramic single tooth restorations3-8). In addition, a previous clinical study found that composite inlays had even had a significantly better color match after three years than glass-ceramic inlays9). Although the CAD/CAM composite materials are very promising, there are also disadvantages, especially with regard to their abrasion resistance. In this context, laboratory and clinical studies report increased wear after only short loading periods10-12). To overcome the latter problem, a new generation of composites in dentistry, known as PAEK (polyaryletherketone) materials or as PEEK (polyetheretherketone) or PEKK (polyetherketoneketone) or reinforced PEEK/PEKK materials with inorganic fillers may be a suitable alternative with comparable wear properties in the range of ceramics as shown in some pilot trials (unpublished data). Additionally, PEAK is biocompatible and resistant to nearly all organic and inorganic chemicals and has also shown adequate mechanical properties13,14). Based on these excellent physical and biological properties—despite the lack of clinical long-term studies—this composite material seems to be suitable for superstructures in dentistry, i.e. for dental implants, provisional abutments, fixed dental prostheses (FDPs), as well as implant-supported bars and clamps for removable prostheses15-19). PEEK represents a high performance thermoplastic polymer within this material group with a low melting temperature of 343°C and can therefore be processed in various ways. One possibility is pressing the material with a special vacuum-pressing device in a dental technical laboratory. For this purpose, PEEK is used either as industrially pre-pressed pellets or in granular form. For the pressing process, the preheated muffle (with the press plunger) is placed into the vacuum-pressing device and pressed. Another option is the milling using CAD/CAM technologies where PEEK blanks are pressed industrially under standardized parameters such as pressure, temperature and time. All these fabrication methods can be applied using the same raw material for PEEK FDPs. So far, however, results of mechanical stress tests with these materials are limited and the available literature varies considerably in terms of investigated prosthetic applications. Therefore, the objective of this study was to test and compare the impact of three typical above mentioned different fabrication methods on the fracture load of 3-unit PEEK/C FDPs. The null hypothesis was that CAD/CAM PEEK/C FDPs show similar fracture
load and failure types compared to PEEK/C FDPs pressed from granular or pellet material.

**MATERIALS AND METHODS**

In this study, the fracture load of anatomically constructed PEEK FDPs was tested. For all FDPs, the identical raw reinforced PEEK composite (PEEK/C) material containing 20 wt% inorganic fillers was used, but FDPs were fabricated differently as follows: i) CAD/CAM milled from PEEK/C blanks which were originally pressed from granular PEEK/C (breCAM Bio HPP blank; Bredent, Senden, Germany, LOT 381115), ii) pressed from industrially fabricated pellets which were also pressed from granular PEEK/C (Bio HPP Pellets; Bredent, LOT 381125), and iii) pressed from granular PEEK/C (Bio HPP Granular; Bredent, LOT 379806) (Fig. 1).

In order to produce standardized FDPs, a steel model with two abutments simulating a bridge between a canine and a first molar was used. The abutments of this model had flat occlusal surfaces and a ball end. They were cylindrically shaped (height: 5 mm; diameter canine: 7 mm; molar: 8 mm) with a 1-mm circular shoulder and 6° taper, and were surrounded by a 0.75 mm thick plastic covering, which simulated the periodontal ligament. The holder of the test set-up was made of aluminium alloy with cylindrical holes with a distance of 16.5 mm. After scanning (Ceramill Map 400; AmannGirrbach, Koblach, Austria), an anatomically supported FDP was constructed (Ceramill Mind; design software, AmannGirrbach). The connectors had a cross-sectional area of 16 mm², an occluso-gingival height of 4.45 mm, and a bucco-lingual width of 3.60 mm. The pontics showed a cavity in their middle congruent to the loading stainless steel ball (diameter 5 mm) ensuring a 3-point-contact between the steel ball and the occlusal surface at loading.

In total, fifteen PEEK/C (breCAM Bio HPP, Bredent, LOT 381115) and thirty wax FDPs (breCAM. wax; Bredent, LOT 380089) were milled (ZENO 4030 M1; Wieland+Dental, Pforzheim, Germany). The wax FDPs were randomly divided into two groups (n=15 per group), i.e. pressed using either PEEK/C pellets (Bio HPP Pellets; diameter: 20 mm, Bredent, LOT 381125) or granular PEEK (Bio HPP Granular; Bredent, LOT 379806). Afterwards, wax FDPs were embedded (Brevest for 2 press investment material; Bredent, LOT 121012) in a muffle according to the manufacturer’s instruction. After 20 min, the muffle was heated up to 630°C for 60 min (for PEEK/C granular) or 90 min (for PEEK/C pellets) and then cooled to 400°C at a cooling rate of 8°C/min. Subsequently, the pre-heated muffle was filled with PEEK/C granular/pellets, and kept in the preheating oven for 20 min at 400°C. As the next step, FDPs were pressed at a pressure of 2.3 bar (for PEEK/C granular) or 4.5 bar (for PEEK/C pellet) in a special vacuum-pressing device (Vacuum pressing device for 2 press; Bredent). For this pressing process, one plunger for each muffle was used and the pressing process lasted for 25 min. After cooling, the investment material was removed in an aluminium oxide blasting unit (Fine-blasting type FG 3; Sandmaster, Zofingen, Switzerland) using 105 µm Al₂O₃ (Hasenfratz, Sandstrahltechnik, Abling, Germany) at a pressure of 2 bar. The FDPs were finished using a silicone polisher (Ceragum Wheel; Bredent) and polishing paste (Abraso-Starglanz; Bredent) for 3 min. Before fracture load testing, all FDPs were ultrasonically cleaned in distilled water for 5 min (Ultrasonic T 14, Kearny, NJ, USA). Subsequently, the uniformity of all different fabricated FDPs was checked using a silicon mold, which was based on a CAD/CAM fabricated FDP, served as the master model for the silicon mold. Thus, all FDPs showed comparable overall thickness.

**Fracture load measurement**

FDP specimens were then cemented with a resin composite cement (Variolink II, Vivadent-Ivoclar, Schaan, Liechtenstein, LOT R35481/P84939) on corresponding steel abutments according to the manufacturer’s instructions. Before cementation, the inner surfaces of the restorations were treated as mentioned above in the text (air-abrasion and cleaning ultrasonically). The metal abutments were not pre-treated. Variolink II was mixed in a 1:1 ratio for 10 s and then applied on the inner surfaces of FDPs. The restorations were placed on the model and excess cement was removed. A special cementing device was used to ensure that the pontic was loaded centrally at a force of 0.98 N for 10 min.

After 24 h water storage (37°C), the cemented FDPs were subjected to testing and were loaded in a universal testing machine (Zwick/Roell 1445; Zwick, Ulm, Germany) at a cross-head speed of 1 mm/min with a steel ball (diameter 5 mm) placed on the centre

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Fig. 1 Reinforced PEEK material as CAD/CAM blank for milling as well as pellet and granular for pressing technique.
of the pontic. To achieve an even force distribution, a 0.5 mm tin foil (Dentaurum, Ispringen, Germany) was placed between the pontic and the loading ball. The measurement stopped as soon as fracture load decreased by 10% of the maximum load (Fmax). An illustration of the test set-up is shown in Fig. 2.

![Fig. 2 FDP cemented on steel abutments during fracture load measurement.](image)

Afterwards, a fracture types analysis was performed. Failure modes were defined as having occurred either as “complete fracture” or by deformation.

**Statistical analysis**

Fracture load results were presented descriptively by means, standard deviation (SD) and the corresponding 95% confidence intervals (95% CI). Normality of data distribution was tested using Kolmogorov-Smirnov and Shapiro-Wilk tests and one-way ANOVA together with the Scheffé post-hoc test was applied in order to investigate the fracture load differences between the groups. In addition, Weibull statistics (shape, scale) were computed. In all tests p-values smaller than 5% were considered to be statistically significant. The data were analyzed using a software package for statistical analysis (SPSS Version 20, SPSS INC, Chicago, IL, USA).

**RESULTS**

The fracture load results of each group are presented in Table 1 and Fig. 3. Figure 4 shows a typical load-displacement curve for each PEEK/C material. Kolmogorov-Smirnov and Shapiro-Wilk tests indicated no violation of the assumption of normality for all tested groups. The milled CAD/CAM FDPs (2,354±422 N) showed significantly higher fracture load than those pressed from granular PEEK/C (1,738±439 N) (p<0.001).

No differences were observed between FDPs pressed using PEEK/C pellets (2,011±353 N) and the other groups.

According to the Weibull statistics, CAD/CAM milled FDPs (2,527 N) showed significantly higher (p<0.001) characteristic fracture load (scale) than bridges pressed from granular PEEK/C (1,902 N). No further differences were found. PEEK/C FDPs pressed with granular PEEK/C (4.63) showed a significantly lower (p<0.001) characteristic fracture load (shape).

### Table 1 Results of the fracture load evaluation and Weibull statistic analyses for all tested groups

|                        | Normal distribution (N) | Weibull distribution |
|------------------------|-------------------------|----------------------|
|                        | mean        | SD           | 95% CI         | min      | median    | max      |
| CAD/CAM milled PEEK    | 2,354<sup>a</sup> | 422          | 2,118;2,588    | 1,571    | 2,384     | 3,169    |
| pressed pellet PEEK    | 2,011<sup>ab</sup>| 353          | 1,814;2,208    | 1,388    | 2,026     | 2,660    |
| pressed granular PEEK  | 1,738<sup>a</sup> | 439          | 1,494;1,981    | 1,187    | 1,591     | 2,631    |
| scale (N)              | shape       |              |                |          |           |          |
| CAD/CAM milled PEEK    | 2,527<sup>a</sup> |              | 6.27<sup>a</sup> |          |           |          |
| pressed pellet PEEK    | 2,155<sup>ab</sup>|              | 6.49<sup>a</sup> |          |           |          |
| pressed granular PEEK  | 1,902<sup>a</sup> |              | 4.63<sup>a</sup> |          |           |          |

* Different superscript letters (a, b) represent a significant difference according to post hoc test between the different fabricated FDPs.
Weibull modulus (shape) compared to CAD/CAM milled PEEK/C (6.27) and pellet-pressed PEEK/C (6.49).

During fracture load tests CAD/CAM milled FDPs and pellet-pressed FDPs usually fractured at the pontic whereas the FDPs pressed from granular PEEK/C usually showed plastic deformation of the bridge, without complete fracture (Fig. 5).

DISCUSSION

This study assessed the influence of different fabrication methods of three-unit reinforced PEEK/C FDPs on fracture load. The data obtained in this study supports the rejection of the null-hypothesis, since the fracture load of differently fabricated FDPs was statistically different. In general, the industrially pre-pressed and consequently milled CAD/CAM FDPs showed higher fracture loads than those pressed from granular PEEK. It can therefore be assumed that the industrial pre-pressing process for the CAD/CAM blanks and for the pellets increases the mechanical properties. Additionally, CAD/CAM blanks, after industrial fabrication under optimal conditions display a reduced risk of porosities within the restorations and therefore show improved mechanical properties. In contrast, the mechanical properties of pressed FDPs are more operator-dependent: The preheating method, the vacuum pressing device and other factors may influence the overall quality of the specimens. According to the fracture types, information regarding the brittleness of the material can be won: FDPs fabricated from pre-pressed CAD/CAM blanks and FDPs pressed from pellet PEEK/C showed complete fractures at the pontic, whereas FDPs pressed from granular PEEK/C usually displayed a plastic deformation without complete fracture. Hence, it can be supposed that an additional industrial pre-pressing of PEEK/C blanks/pellets does not only increase the flexural strength of the material, but also reduces its elastic deformability.

In spite of its relatively rigid molecular chain structure, thermoplastic PEEK material demonstrated considerable ductility and can accommodate a wide range of plastic deformation, in both uniaxial tension and compression\textsuperscript{14). However, the material used in this study was optimized in its mechanical properties due to the combination with inorganic fillers (20 wt\%). The mean fracture load for the granulate group was deformed at

Fig. 3 Boxplots for the fracture load of all three tested FPD groups.
*The small circles in the boxplot denote an outlier.

Fig. 4 Typical load-displacement curves for the three tested PEEK/C materials: (a) CAD/CAM PEEK/C, (b) PEEK/C Pellets, (c) PEEK/C Granular.
1,738 N, however plastic deformation and no fracturing was found and slight deformation was initially observed at approximately 1,600 N. Maximum occlusal forces for up to 909 N have been recorded in the molar region. Of course, this function depends on varying factors such as restoration type, measurement process, diet, and gender variation. However, it can be assumed that the FDPs analyzed in this study would withstand clinical applications without restrictions.

In the present study, the connector area of the FDPs was set at to 16 mm². An increased connector surface area would have probably additionally increased the results. Another study investigating the fracture load of CAD/CAM milled non-veneered three-unit PEEK FDP frameworks with a connector area of 7.36 mm² observed a fracture load of 1,383 N with a deformation at approx. 1,200 N. The CAD/CAM milled PEEK/C FDPs in the present study showed a higher mean fracture load of 2,554 N. The manufacturer published data regarding CAD/CAM milled three-unit PEEK FDPs, veneered with resin composite, which showed a mean fracture load of 2,055 N. These values are comparable to those in the present study (1,738–2,354 N). This was even higher than values found for three-unit-FDPs made of lithium disilicate glass-ceramic (950 N), In-Ceram Alumina (851 N), In-Ceram Zirconia (841 N), and zirconia (981–1331 N). In contrast, composite- and PMMA-based three-unit FDPs showed much lower mean fracture loads ranging from 268 N to 467 N.

In the present study, Weibull statistics were also applied to characterize the structural reliability of the materials. In doing so, a lower Weibull modulus indicated a greater variability and thereby less reliability in strength, most probably due to flaws and defects in the material. Significant differences in Weibull moduli were observed: FDPs pressed from granulate showed a lower modulus compared to CAD/CAM milled FDPs and FDPs pressed from PEEK/C pellets. Again, the statement can be confirmed that an additional industrial pre-pressing of PEEK/C increases the Weibull moduli and thus leads to improved reliability of the material.

An increase in fracture load was found when FDPs were non-rigidly mounted and when the elastic modulus of the abutment was higher. The clinical situation could be simulated by investigating non-rigidly mounted abutments with an elastic modulus similar to that of natural teeth. Although the physiological mobility of teeth was simulated with an emulated periodontal ligament, it must still be emphasized that steel abutments with an elastic modulus of 180 GPa were used, whereas natural teeth only have an elastic modulus ranging approximately from 15 (dentin) to 85 (enamel) GPa. It could therefore be assumed that the stability of the FDPs in a clinical situation might therefore be slightly lower. As the specimens in this study were not aged, further investigations with additional aging through chewing simulation or thermal cycling are required for more longitudinal clinical aging data or at least trends. Of course, clinical studies are also needed to support the use of PEEK for long-term restorations.

CONCLUSIONS

Within the limitations of this in-vitro study, it can be summarized that, based on the findings in this study, milled PEEK/C FDPs displayed advantages over FDPs pressed from granular PEEK/C. Therefore, it can be concluded that—PEEK/C reinforced with other inorganic fillers can be potentially used as crown and bridge material—Industrial pre-pressing of blanks (CAD/CAM/pellet) increases the stability and reliability of PEEK/C restorations.

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