Aus dem

Poliklinik für Zahnärztliche Prothetik

Klinikum der Ludwig-Maximilians-Universität München

# Polyaryletherketone in prosthetic dentistry - in vitro studies for fixed and removable dental prostheses

Dissertation
zum Erwerb des Doctor of Philosophy (Ph.D.)
an der Medizinischen Fakultät der
Ludwig-Maximilians-Universität München

vorgelegt von

Danka Mićović Soldatović

aus

Serbien

Jahr
2023

Mit Genehmigung der Medizinischen Fakultät der
Ludwig-Maximilians-Universität München

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Tag der mündlichen Prüfung: 24.10.2023

*Dedicated to my family for unconditional love and support*

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# List of abbreviations

| | |
|---|---|
| AKP | Aryl ketone polymer |
| Au | Gold alloy |
| CAD/CAM | Computer-aided design and computer-aided manufacturing |
| CoCrMo | Cobalt-chrome-molybdenum |
| FDP | Fixed dental prostheses |
| PAEK | Polyaryletherketone |
| PEKK | Polyetherketoneketone |
| PMMA | Polymethylmethacrylate |
| RDP | Removable dental prostheses |
| STL file | Standard Tessellation Language, Stereolithography |
| $TiO_2$ | Titanium dioxide |
| TMC | Thermomechanical cycles |
| $ZrO_2$ | Zirconia |

# List of publications

### Journal publications

**Micovic Soldatovic, D.**, Liebermann, A., Huth, K. C., Stawarczyk, B., Fracture load of different veneered and implant-supported 4-UNIT cantilever PEEK fixed dental prostheses. J Mech Behav Biomed Mater, 2022. 129: p. 105173.

**Micovic Soldatovic D**, Bitter M, Meinen J, Huth C. K, Liebermann A, Stawarczyk B. Impact of material combinations and removal and insertion cycles on the retention force of telescopic systems. Clin Oral Investig, 2023. 27(7): p. 4007-4016.

Mayinger, F., **Micovic, D.**, Schleich, A., Roos, M., Eichberger, M., Stawarczyk, B., Retention force of polyetheretherketone and cobalt-chrome-molybdenum removable dental prosthesis clasps after artificial aging. Clin Oral Investig, 2021. 25(5): p. 3141-3149.

**Micovic, D.**, Mayinger, F., Bauer, S., Roos, M., Eichberger, M., Stawarczyk, B., Is the high-performance thermoplastic polyetheretherketone indicated as a clasp material for removable dental prostheses? Clin Oral Investig, 2021. 25(5): p. 2859-2866.

### Presentation at conferences

**Micovic Soldatovic D**, Liebermann A, Huth C. K, Stawarczyk B. Stability of Veneered and Implant-supported PEEK FDPs with Free-end Unit. BSODR British Society for Oral and Dental Research, Annual Meeting 2021, 1 - 3 September, University of Birmingham, Birmingham, UK.

# Author`s contribution to the publications

## 1.1 Contribution to publication 1

- Developed research question and study design together with supervisors
- Planned the experiment with the main supervisor
- Manufactured specimens, performed aging in mastication simulator and fracture load tests
- Collected and organized data and prepared them for statistical analyses
- Participated in statistical analysis and produced tables and graphs to present the results
- Wrote the main draft of the manuscript, created tables and figures and revised manuscript according to the other authors inputs

## 1.2 Contribution to publication 2

- Planned the experiment together with the main supervisor and other authors
- Conducted the experiment, manufactured specimens, performed retention load measurements and artificial aging
- Collected and organized data
- Performed statistical analyses with main supervisor
- Wrote the main draft of the manuscript, created tables and figures and revised manuscript according to the other authors inputs

## 1.3 Contribution to publications 3 and 4

- Participated in figures and graphs creation for presentation of the results
- Performed statistical analyses with main supervisor
- Wrote the main draft of the manuscript and finalized it together with other authors

The entire process of writing, submitting and revising the manuscript of publications III and IV was carried out together with PD Dr. Felicitas Mayinger, so that the shared first authorship in these two publications is the consequence of equal work and equal contribution.

# 2. Introductory summary

## 2.1 Introduction

Polyarylretherketone in prosthetic dentistry

- in vitro studies for fixed and removable dental protheses

*The following project was performed at the Department of Prosthetic Dentistry, University Hospital, Ludwig Maximilian University, Munich, supervised by Prof. Bogna Stawarczyk, Prof. Anja Liebermann and Prof. Karin C. Huth.*

This dissertation comprises four publications based on performed experiments in which behavior of different PAEK materials for different indications in prosthetic dentistry (fixed dental prostheses, clasps for removable protheses and telescopic crowns) was examined.

The term polyaryletherketone (PAEK) is common name for closely related high-performance semicrystalline thermoplastic materials such as polyetheretherketone (PEEK), polyetherketoneketone (PEKK) [1] and recently developed aryl ketone polymer (AKP) [2, 3]. They differ among themselves in number of functional ether- and keto- groups and this slightly different composition indicates different properties and indication areas [4]. Due to the notable mechanical characteristics [5] and material composition which besides conventional (pressing) [6-8] allows fully digital processing (milling and 3D printing) [6, 9, 10] this material group is gaining on its popularity in manifold applications in restorative dentistry.

Historically, PAEK was introduced as biomaterial three decades ago and since than it is considered as important material group in orthopedic and maxillofacial surgery [11]. PEEK was, for instance, used as a spinal implant material [12] and its properties have been changed over time [13] by adding fillers to improve mechanical stability or surface modifications for better osseointegration [14].

A decade later, this material group was introduced in dentistry and since than is being investigated and improved for possible dental applications as material for implants [15], abutments [16], long-term provisional restorations, crowns [17] and bridges [6], removable dental prostheses [18], telescopic crowns [19, 20], root canal posts, brackets, etc.

PAEK is characterized by semi-crystalline structure, which is a combination of crystalline and amorphous areas inside the material [12]. Crystalline means ordered structure and is produced by heating, while amorphous is a wild structure and is produced by quenching the temperature. Both proportions can be influenced by the processing of this material which affects its mechanical performances [21]. For instance, pressed restorations made of PAEK have more elastic properties than milled ones, whereas milled restorations are showing better mechanical stability [22].

For dental purposes, PAEK materials are produced as filled or unfilled. Unfilled PEEK is due to the lower strength mainly used for removable dental prostheses. On the other hand, a filler content of 10-30 weight percent (mostly inorganic fillers like $TiO_2$) increases the mechanical stability [6] and broadens the spectrum of indications for both FDPs and RDPs.

The white or greyish color and non-transparency make the PAEK materials non-esthetic, so the monolithic PAEK restorations are not acceptable, and veneering is required. The inert and chemically stable surface of those materials was an impediment for bonding between PAEK and veneering material, but also for bonding between the restoration and tooth/abutment. According to the recent investigations, those problems could be solved by conditioning the PAEK surface using for instance airborne-particle abrasion with $Al_2O_3$, than MMA containing adhesives and opaquer layer [17, 23-26].

Today, alloy-based and ceramic materials are still predominantly used in prosthetic dentistry due to their proven mechanical properties and satisfactory esthetics. However, ever-increasing demands, especially in terms of esthetics and biocompatibility, may threaten the position of alloys, for instance as implant material and promote the trend towards metal-free thermoplastic PEEK materials [11]. As PEEK is showing favorable biocompatibility [14] and similarity to the human bone [27] it can be considered as a concurrent implant material [11].

This was an example that shows a possible future of PAEK materials for indications where conventionally used materials cannot meet the expected requirements.

Moreover, the PAEK family has not only established itself as an implant material in medicine and dentistry, but also as a restorative material for various applications. Due to the novelty of those materials in the dental field, the literature on them is limited. And this was exactly the motive and the purpose of this PhD project, to contribute the knowledge by providing more information on the in vitro behavior of PEEK and PEKK when used for fixed and removable dental prostheses as well as telescopic crowns.

### 2.1.1 Publication 1. Fracture load of different veneered and implant-supported 4-UNIT cantilever PEEK fixed dental prostheses

In our first investigation, we compared the mechanical stability of FDPs with frameworks which were milled and pressed from two differently filled PEEK materials and veneered in three different methods, initially and after mastication simulation.

Implant-supported restorations are nowadays mostly made of metal-ceramic and zirconia [28]. As this is the era of thermoplastics, we decided to investigate PEEK properties for this this purpose and even more, to test it beyond its recommended indication area - in cantilever design. Our aim was to investigate the ability of PEEK to withstand the occlusal forces in the posterior region as well as the endurance of the veneering material.

In order to simulate the clinical situation as good as possible, the FDPs were anatomically manufactured, finished, polished and bonded to the implant abutments using the same procedure as for the clinical usage. Furthermore, FDPs were aged in mastication simulator to predict the behavior of the material after 5 years of clinical use. During the testing and aging, FDPs were loaded with individually made antagonists to apply forces on each unit of 4-UNIT FDPs [6].

The results showed that filler content in PEEK compound has a great influence on the fracture load values. Increasing the wt% of fillers leads to an enhancement in the mechanical stability of the material. As the pressed PEEK material filled with 30 wt% $TiO_2$ was an experimental material used for the first time in this investigation, the results are not comparable with previously published papers where milled and pressed PEEK were investigated [22].

Regarding the veneering method, the conventional veneering using resin composite showed the lowest fracture load values, which was consistent with previous investigations [29, 30]. In contrast, the best performance in our investigation was recorded for prefabricated veneers, followed by digital veneering, which contradicts the mentioned papers where digital veneering was described as mechanically the most stable.

Nevertheless, both types of PEEK compound as well as all three veneering methods showed higher fracture load values than the described occlusal forces in posterior region. It can be concluded that PEEK frameworks can be a viable alternative to conventional materials, even for cantilever constructions, if the proper framework material and appropriate veneering technique are selected.

### 2.1.2 Publication 2. Impact of material combinations and removal and insertion cycles on retention force of telescopic systems

The aim of the second investigation in this project was to evaluate and compare retention load values of different material combinations used for telescopic crowns initially and after different aging regimes. To ensure long-term satisfactory results, patients should be provided with a restoration whose retention forces are predictable and remain constant over time.

The materials used for telescopic crowns are mostly gold alloys and cobalt-chromium-molybdenum. As alloy-based materials are used for this indication for a long period of time, a lot of in vitro, but also in vivo studies confirm their reliability as telescopic crown material [31, 32]. The current trend towards non-metallic restorations, but also the use of new technologies (milling, 3D printing), which allow faster and more reliable fabrication, have prompted new materials for this indication: zirconia and PAEK [33]. So far, the literature on $ZrO_2$ telescopic crowns delivers contradictory findings [34] and further in vitro and in vivo investigations should provide clinicians with adequate information for clinical use.

Speaking about polymer-based materials for this indication, a few studies demonstrated promising characteristics of PEEK in terms of sufficient retention forces and low wear rates [19, 35]. On the other hand, information about PEKK as telescopic material is limited and based on a few available in vitro investigations [4, 36]. However, these investigations revealed optimistic results that PEKK has a similar behavior to conventionally used double crown materials.

As described, there is a gap in knowledge about the long-term behavior of novel materials for telescopic systems processed with CAD/CAM technology, which motivated us to investigate this topic further.

In this investigation we compared retention load values of twelve different material combinations, initially and after three aging regimes. Aging was performed in mastication simulator and the telescopic systems were exposed to 500, 5 000 and 10 000 removal and insertion cycles, which simulates 6 months, 5 years and 10 years of clinical use (respectively). Pull-off tests were prepared on universal testing machine with artificial saliva between contact surfaces to approximate clinical conditions [20].

According to our results, both the material combination and the simulated aging affected the retention force values. Repeated removal and insertion cycles led to a decrease in retention forces for CoCr and $ZrO_2$ secondary crowns. On contrary, an increase of retention forces was observed for PEEK and PEKK secondary crowns.

Recent studies investigated PEEK and PEKK as double-crown materials have yielded similar results. These authors suggest that polymeric materials could be an alternative for metal components [36, 37].

As limitations of this investigation should be mentioned the in vitro study design and the evaluation of the retention of a single telescopic crown (assuming that multiple telescopic systems may show different behavior of retentive forces). Nevertheless, it can be concluded that the digital workflow and novel materials can provide a suitable alternative to conventional methods. However, improvements in milling design and spacing between crowns are needed, as well as investigations on material combinations and taper under clinical conditions.

### 2.1.3 Publications 3. and 4.

**Is the high-performance thermoplastic polyetheretherketone indicated as a clasp material for removable dental prostheses?**

**Retention force of polyetheretherketone and cobalt-chrome-molybdenum removable dental prosthesis clasps after artificial aging**

In these two investigations the performance of PEEK as a clasp material for removable dental prostheses was evaluated.

Different processed PEEK materials (milled and pressed) were compared to the CoCr control group. Results from Publication 3 are showing the lower retention force of PEEK in comparison to CoCr. On the other hand, PEEK clasps presented stability of the retention forces over time which remained constant after aging and pull-off tests. This finding may indicate possible clinical application [7]. Aging was here simulated by storage of specimens in artificial saliva for 90 or 180 days.

The set-up for investigation 4 was similar to investigation 3, with slight difference in aging regimes. Specimens were exposed to 30 days storage and 10 000 thermal cycles or 60 days storage with 20 000 thermal cycles. The results are not consistent with previous investigation, as both the clasp material and artificial aging significantly affected the retention force. Milled PEEK clasps and CoCr clasps showed higher retention force values and less influence of artificial aging than the pressed PEEK clasps. However, all tested materials exhibited sufficient retention to be cautiously recommended for clinical use [8]. This finding is supported by the recently published review article confirming potential clinical use of shape-optimized PEEK clasps [13].

# 3. Publication 1

## Fracture load of different veneered and implant-supported 4-UNIT cantilever PEEK fixed dental prostheses

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J Mech Behav Biomed Mater, 2022. 129: p. 105173.

DOI:10.1016/j.jmbbm.2022.105173.

journal of the mechanical behavior of biomedical materials 129 (2022) 105173

Contents lists available at ScienceDirect

Journal of the Mechanical Behavior of Biomedical Materials

journal homepage: www.elsevier.com/locate/jmbbm

# Fracture load of different veneered and implant-supported 4-UNIT cantilever PEEK fixed dental prostheses

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ARTICLE INFO

*Keywords:*
PEEK compound
Veneering technique
Implant-supported restorations
Aging
Fracture load

ABSTRACT

*Objectives:* To determine the fracture load before and after artificial aging of implant-supported 4-unit cantilever fixed dental prostheses (FDP) with frameworks made of two differently filled polyetheretherketone (PEEK) compounds and veneered using three different techniques.

*Methods:* A total of 120 duplicate 4-unit FDP frameworks were produced (n = 60 milled from PEEK, 20% $TiO_2$ filled and n = 60 pressed from PEEK, 30% $TiO_2$ filled) and veneered using three different techniques: (i) digital veneerings, (ii) conventional resin composite veneerings and (iii) prefabricated veneers (n = 20 per subgroup). The FDPs were adhesively bonded to titanium abutments and the fracture load was measured in a universal testing machine (1 mm/min) before and after artificial aging in a mastication simulator (1,200,000 cycles, 50 N, TC: 5/55 °C, 6000 cycles). The fracture patterns were analyzed using digital microscopy. Data were analyzed using the Kolmogorov-Smirnov test, two-way ANOVA, post hoc Scheffé, $Chi^2$-test ($p < 0.05$), and Weibull modulus *m*, as well as fracture patterns using the Ciba-Geigy table.

*Results:* Veneering technique and filler content significantly affected the fracture load ($p < 0.001$). Prefabricated veneers showed higher fracture load ($p \leq 0.001$) whereas digital and conventional veneerings were similar ($p = 0.451$). PEEK with 30% filler content presented higher fracture load ($p < 0.001$) compared with PEEK with 20%. Aging showed no effect on fracture load ($p = 0.176$). Regarding fracture types, no significant differences were found among the groups ($p = 0.055$).

*Conclusions:* Filler content of PEEK compound as well as veneering technique influenced fracture load while aging had no effect on fracture load. FDPs made of PEEK with 30% of filler content veneered using prefabricated veneers had the highest fracture resistance.

## 1. Introduction

Implant-supported fixed dental prostheses (FDPs) can be considered as an effective treatment option for partially edentulous patients, improving their long-term oral health-related quality of life (Ali et al., 2019). Currently, the most commonly used materials for implant-supported fixed dental prostheses are metal-ceramic and zirconia (Sailer et al., 2018).

Despite constant improvements in materials and technologies, both have shortcomings which affect the long-term outcome of implant-supported FDPs. Veneered zirconia FDPs have been reported to be prone to chipping and delamination of the veneering ceramic, leading to the popularity of monolithic zirconia FDPs (Stawarczyk et al., 2017; López-Suárez et al., 2018). Monolithic zirconia restorations have esthetic limitations, and obtaining a perfect tooth-like color is problematic (Tabatabaian, 2019).

Even though metal-ceramic FDPs show better long-term stability compared with veneered zirconia, they also have limitations, including compromised esthetics and discoloration of marginal gingiva (Poggio et al., 2017) as well as potential allergy to the metal. Therefore, all-ceramic restorations have become popular compared to metal-based restorations (Heintze and Rousson, 2010; Sailer et al., 2018).

In addition to metal-ceramic and zirconia, a relatively novel approach in implant-supported prosthodontics is the use of thermoplastic materials, specifically polyaryletherketone (PAEK). In dentistry, the PAEK material class includes a variety of thermoplastics, including

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https://doi.org/10.1016/j.jmbbm.2022.105173
Received 25 January 2022; Received in revised form 4 March 2022; Accepted 7 March 2022
Available online 9 March 2022
1751-6161/

polyetheretherketone (PEEK), polyetherketoneketone (PEKK), and aryl-ketone-polymer (AKP) (Marie et al., 2019).

Polyetheretherketone (PEEK) is the most commonly used of these PAEK thermoplastics family in dentistry. With excellent mechanical properties, biocompatibility, chemical stability, X-ray translucency, polishability, and good wear resistance (Papathanasiou et al., 2020), PEEK is particular promising for the restorative dentistry. Moreover, its modulus of elasticity (3–4 GPa) similar to that of bone (Kurtz and Devine, 2007) provides PEEK restorations with a damping effect (Parmigiani-Izquierdo et al., 2017; Papathanasiou et al., 2020) that produces a reducing stress shielding effect (Lee et al., 2012). In vitro investigations have reported PEEK to be suitable for frameworks for fixed and removable dental prostheses (RDPs), clasps for RDPs, occlusal splints, intraradicular posts, interim restorations, and implant abutments (Najeeb et al., 2016; Bathala et al., 2019; Papathanasiou et al., 2020). One recent clinical study, comparing PEKK with cobalt-chromium restorations on natural teeth reported no differences between the materials and suggested using PEKK in anterior regions to improve esthetics but not in the posterior regions because cantilever FPDs were prone to fracture (Klur et al., 2019). Clinical case reports have suggested PEEK frameworks as a suitable alternative to currently used materials for implant-supported restorations because of PEEK's biocompatibility and cushioning effect and because its wear is similar to that of natural teeth (Parmigiani-Izquierdo et al., 2017; Zoidis, 2018). However, results from clinical trials including PEEK compounds are still scarce, and long-term clinical evidence is required before recommending PEEK as a suitable material for implant-supported FDPs (Zoidis, 2018; Jovanović et al., 2020; Papathanasiou et al., 2020).

PEEK can be milled from extruded blanks using computer-aided design (CAD) and computer-aided manufacturing (CAM) technology. Another option is pressing in special vacuum-pressing devices, and, for this purpose, PEEK is produced in pellets or granular form. CAD-CAM processed PEEK has been reported to have higher fracture resistance than PEEK pressed from the granular form (Stawarczyk et al., 2015). Furthermore, in recent years, processing of PEEK through 3D printing has been used to produce especially detailed or patient-individualized PEEK objects (Honigmann et al., 2018; Zanjanijam et al., 2020). This is a technology with great potential, but one that requires further investigations to improve quality and solve current problems that include extensive post-processing and the dependence of mechanical properties on printing directions (Prechtel et al., 2020a, 2020b). PEEK compounds are available unfilled or filled with titanium in order to reinforce PEEK and improve mechanical properties (Panayotov et al., 2016). The higher the filler content in the PEEK compound, the higher the stiffness, hardness, and elastic modulus of the material. As the longevity of dental restorations is of a great importance, the behavior of PEEK compounds after aging is significant. PEEK is not susceptible to a degradation process during artificial aging and successfully maintains its original properties after thermocycling (Niem et al., 2020) or mastication simulation (Prechtel et al., 2020b).

The disadvantages of PEEK include its white to grayish color, opacity, and low translucency, which restrict its usage for monolithic restorations. PEEK also requires veneering, especially in the esthetic zone (Stawarczyk et al., 2016; Taufall et al., 2016), which can be done with a digital process, with the application of prefabricated veneers, or conventionally with resin composite. Digital veneering has the highest fracture load resistance compared with conventional resin composite veneering or the use of pre-manufactured veneers that reduce complex manual steps during manufacturing (Taufall et al., 2016).

From systematic reviews, implant-supported cantilever FDPs in the posterior region have been reported to have a high survival rates (Storelli et al., 2018) and are reliable as a long-term treatment option (Schmid et al., 2020) independent of restoration material. One further investigation reported the similar survival rates of cantilevered and fixed-to-fixed FDPs made of ceramic in the posterior region (Passia et al., 2019).

The authors are unaware of previous investigations on PEEK cantilever implant-supported FDPs. Therefore, the aim of the present investigation was to examine the fracture load of different veneered and implant-supported cantilever FDPs with frameworks made of two differently filled PEEK compounds after artificial aging. The null hypotheses was that the filler content of the framework material, different veneering methods, and artificial aging would have no impact on the fracture load values.

## 2. Materials and methods

The fracture load of FDPs fabricated from two differently filled PEEK framework materials (approximately 20% and ca. 30% of $TiO_2$ fillers) and veneered with three different techniques (digital veneering, conventional veneering and prefabricated veneers) was examined before and after artificial aging (Fig. 1, Table 1). A total of 120 specimens shaped as implant-supported 4-unit cantilever FDPs were manufactured.

### 2.1. Specimen preparation

A gypsum master cast was made with two implants (SKY implant system: SKY fast & fixed laboratory analog (made of titanium, grade IV KV) and SKY fast &fixed prosthetic coping transversal screw-retained, bredent) replacing teeth 34 and 36. A total of 120 acrylic resin implant casts were fabricated from the master cast (Fig. 2). All light-cured materials were polymerized using bre.Lux PowerUnit 2 (bredent) and wavelength of 370 nm.

A master PEEK framework for 4-unit implant-supported FDPs from a first premolar to a second molar (34–37) was designed. The implant abutments were in the position of the first premolar (34) and the first molar (36), with the second premolar as a pontic (35) and a free-end unit (37). The master cast was scanned (Ceramill Map 400, Amman Girrbach, Koblach, Austria) and digitalized. The obtained digital cast was used for framework construction (CAD software- Ceramill Mind, Amann Girrbach). The thickness of the framework was 1 mm (buccal and occlusal) and 0.8 mm (mesial) for the tooth 34. The thickness of the connectors was:

- between 34 and 35: 19.25 $mm^2$ (5.5 mm vertical and 3.5 mm horizontal dimension);
- between 35 and 36: 15.12 $mm^2$ (4.2 mm vertical and 3.6 mm horizontal);
- between 36 and 37: 20.58 $mm^2$ (4.9 mm vertical and 4.2 mm horizontal).

Sixty PEEK (breCAM.BioHPP, bredent) and 60 wax frameworks (Ivoclar wax blanks, Schaan, Liechtenstein) were milled (Ceramill Motion 2, Amann Girrbach, Koblach, Austria), and detached, and the connectors were removed with a laboratory handpiece (KaVo, Biberach an der Riss, Germany) and a tungsten carbide bur (H1S 104 018; Komet Dental, Lemgo, Germany).

The wax frameworks were embedded (Brevest for 2 press, bredent) and pressed. The muffle was heated according to the manufacturers' instructions in a furnace (KaVo Ewl typ 5636, Lemgo, Germany) using a precise preheating process (290 °C 45 min, 580 °C 45 min, 900 °C 60 min). After cooling the muffle to 400 °C, PEEK granules and pellets were melted (20 min). For each pressing process, one PEEK pellet was placed on the bottom of the heated muffle to close the gap and prevent the granules from falling into the sprues. Above the pellet, PEEK granules were added to the exact amount calculated by using the formula: g (wax) × 2 + 1 g (2 g). Melted PEEK was subsequently pressed under vacuum for 3 min 40 s under a pressure of 3.2 bar and under a pressure of 4.2 bar for the next 45 min (for 2 press, bredent). After devesting, the pressing channels were separated and frameworks were ready for veneering (Fig. 3).

To achieve the shape congruency of all examined FDPs, an anatomic

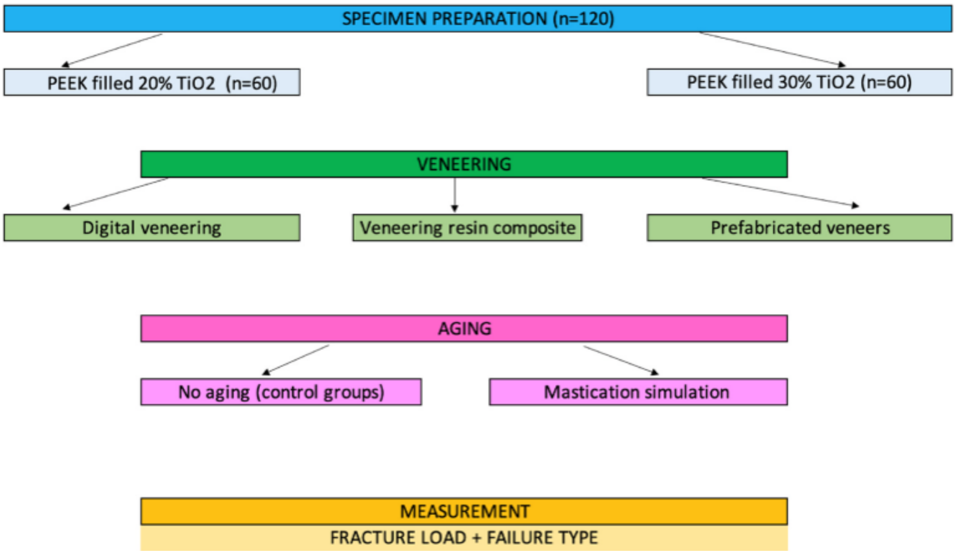


**Fig. 1.** Study design.

**Table 1**
Summary of used materials, their composition and lot numbers. Manufacturer: bredent GmbH & Co. KG, Senden, Germany.

| Material | Product name | Composition | Lot.No | Flexural strength | Modulus of elasticity |
|---|---|---|---|---|---|
| Framework materials | BioHPP breCAM | Polyetheretherketone, filled with ca. 20% $TiO_2$ | 494209 | ≥160 MPa | ≥4200 MPa |
| | BioHPP plus Pellets | Polyetheretherketone, filled with ca. 30% $TiO_2$ | 484074 | N/A | N/A |
| | BioHPP plus Granulat | Polyetheretherketone, filled with ca. 30% $TiO_2$ | 488087 | N/A | N/A |
| Veneering materials | breCAM.HIPC | high-performance polymer, composite (matrix: PMMA, filler: $SiO_2$) | 495547 | ≥110 MPa | ≥2200 MPa |
| | visio.lign | high-impact PMMA composite | Z9179071 | ≥140 MPa | ≥3000 MPa |
| | crea.lign | Bis-GMA composite with microfillers, tetramethylene dimethacrylate | N193296 | ≥145 MPa | N/A |

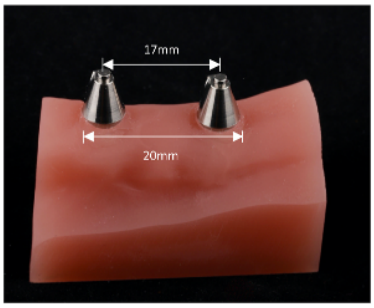


**Fig. 2.** Acrylic resin implant cast.

master FDP veneered using prefabricated veneers (visio.lign, bredent) was made and served as a master for all three veneering groups.

All frameworks were prepared as followed:

1. Airborne-particle abrasion (110 µm $Al_2O_3$, 0.2 MPa, 45° angle, 10-mm distance);
2. Adhesive system (visio.link, bredent) - applying a thin layer with a microbrush and polymerization for 90 s;
3. Opaquer (crea.lign, bredent) - applying a thin layer with a brush and polymerization for 360 s.

For manufacturing of the digital veneerings, the PEEK framework on a cast and the master FDP on a cast were scanned (Ceramill Map 400) and milled (breCAM.HIPC, bredent; Ceramill Motion 2). The subtraction method was used in the CAD software program (Ceramill Mind, Amann Girrbach), resulting in the design of digital veneerings. After detaching and removing the connectors, digital veneerings were airborne-particle abraded (intaglio surface: 110 µm $Al_2O_3$ at 0.2 MPa, 45° angle, 30-mm distance) and conditioned (visio.link) by applying a thin layer with a microbrush and polymerizing for 90 s. The digital veneering was then bonded on the pretreated framework. The veneer was filled with dual-curing luting resin composite (combo.lign, bredent), pressed on a framework, polymerized for 180 s, and cleaned of excess.

To bond prefabricated veneers (Visio.lign, bredent), a transparent silicone mold reinforced with putty silicone was made according to the master FDP. Each veneer was ground manually by using a silicone mold as a guide. Subsequently, they were airborne-particle abraded (110 µm $Al_2O_3$ at 0.2 MPa, 45° angle, 30 mm distance) and conditioned (visio.link) by applying a thin layer with a microbrush and polymerized for 90 s. The veneers were then positioned into the silicone mold, which was filled with luting resin composite (combo.lign). The prepared FDPs were temporarily fixed on the casts and pressed into the mold and polymerized for 180 s. Subsequently, a resin composite (crea.lign) was added where required.

For the conventional veneering, a translucent silicone mold (visio.sil, bredent) was manufactured according to the master FDP. The pretreated framework was temporarily fixed on the cast with wax (Supradent - Klebewachs, Bonn, Germany). The silicone mold was filled with the veneering resin composite (crea.lign) and the cast with the framework was pressed into the mold. After polymerization for 180 s, the resin composite surface was cleaned (crea.lign surface cleaner, bredent) (Fig. 4).

All FDPs were polished to a high gloss with brushes and polishing pastes (Acrypol and Abraso Starglanz; bredent).

The examined FDPs were bonded on the implant models as follows:

1. Airborne-particle abrading both surfaces using alumina powder 110 µm, 0.2 MPa, a 45-degree angle, 10-mm distance, 10 s;

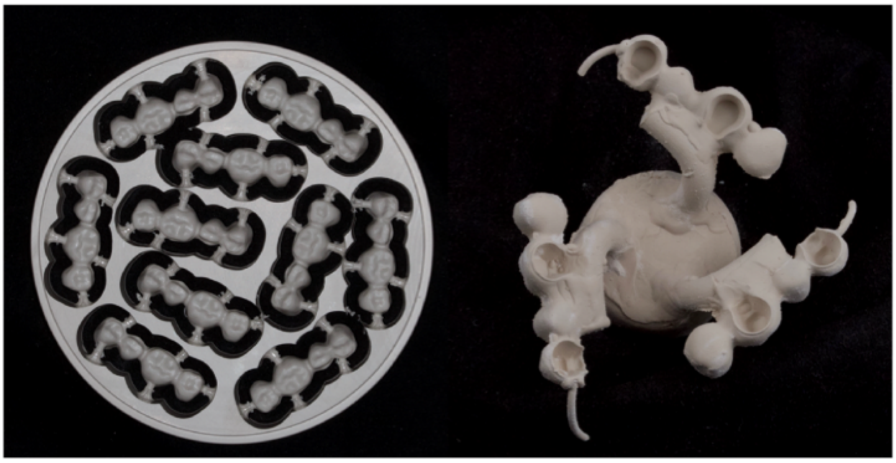

**Fig. 3.** Framework fabrication: milled and pressed FDP frameworks.


**Fig. 4.** Veneering procedure using veneering resin composite (from the bottom: airborne-particle abraded framework; adhesive layer; opaquer layer; veneering resin composite - finished FDP).

2. MKZ primer applied on titanium surface with a microbrush and dried for 1 min;
3. Adhesive system (visio.link) applied on PEEK surface with a microbrush for 5 s, followed by 90-s polymerization;
4. Thin film of opaque applied with a microbrush on the titanium surface, followed by 360-s polymerization;
5. Dual-curing luting composite resin (combo.lign bredent) applied directly into crowns from the mixing tip, followed by 180-s polymerization.

### 2.2. Fabrication of antagonists

The antagonists were prepared for mastication simulation to apply force on each tooth of four-unit FDPs. The four-contact antagonist prototype was made by waxing the fabricated antagonists onto an acrylic resin holder. Once fabricated, the occlusion was adjusted in a parallelometer, and the prototype was scanned in order to obtain an STL file. The STL file was nested and than milled (Ceramill Motion 2) from Co–Cr–Mo alloy (Sintron, Amann Girrbach). After detaching and cutting the connectors, antagonists were sintered in an argon atmosphere (Ceramill Argotherm, Amann Girrbach). The impact of the abutment and antagonist material for the aging simulation had been tested in a previous study (Schmeiser et al., 2022), with the authors reporting, that metal antagonists showed wear behaviors comparable to those of enamel antagonists. Therefore, in this study metal antagonists for the aging regimes in a mastication simulator were used.

### 2.3. Aging, measurement of fracture load and fracture type analysis

Before performing aging or initial measurements, the examined FDPs were stored in deionized water at 37 °C for 24 h in an incubator (HERAcell 150, Thermo Scientific, Waltham, USA). The FDPs were allocated into groups (per framework and per veneering technique). Half of each subgroup was used for initial measurements, and the other half was aged, 1.200.000 cycles in a mastication simulator (SD Mechatronic, Feldkirchen-Westerham, Germany; 50 N at 1.5 Hz, vertical movement of 2 mm) and simultaneously thermocycled for 6000 cycles between 5° and 55 °C (distilled water, dwell time 60 s).

The fracture load measurements were performed in a universal testing machine (Zwick 1445, Zwick/Roell, Ulm, Germany). When the specimens and the antagonists were positioned, a 0.3-mm-thick tin foil, (Dentaurum, Ispringen, Germany) was placed between the contact surfaces to avoid force peaks. The vertical force was applied with a crosshead speed of 1 mm/min. A moment when the force dropped by 10% from the maximum was considered the failure point.

The fracture types were analyzed using digital microscope (Keyence VHX-970F, Keyence, Osaka, Japan). Fracture types were classified as cohesive (fracture within veneering material), adhesive fractures (fracture between framework and veneering), and complete (fracture of framework and veneering) (Fig. 5).

### 2.4. Statistical analysis

The assumption of normality was tested with the Kolmogorov-Smirnov test. Global univariate analysis (two-way ANOVA) with partial eta squared ($\eta_{p2}$) followed by the Scheffé post hoc test was used to verify the impact of aging on fracture load.

The Chi square test ($chi^2$) and Ciba-Geigy tables were used to analyze the relative frequencies of fracture types together with the corresponding 95% confidence intervals (CI) ($\alpha = 0.05$ for all tests) A statistical software program (IBM SPSS Statistics, version 26.0.0.1, IBM Corp, Armonk, NY, USA) was used for the statistical analysis. The Weibull modulus $m$ was calculated using the maximum likelihood estimation

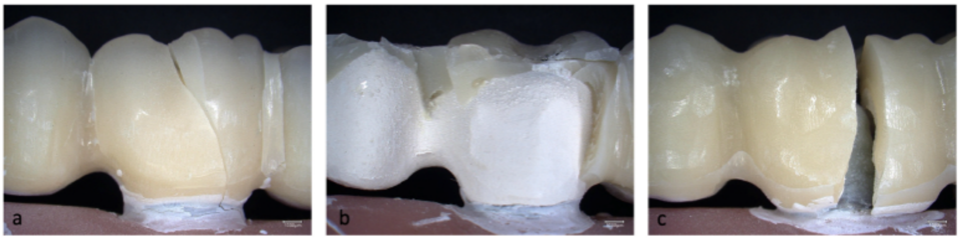

**Fig. 5.** Fracture types: a - cohesive, b - adhesive, c - complete.

method and 95% confidence interval (95% CI) (Bütikofer et al., 2015).

## 3. Results

The descriptive statistics are summarized in Table 2. One of twelve tested groups deviated from normal distribution (9% of all tested groups). Hence, the parametric tests were performed as no violation of normality assumption was indicated.

Veneering technique and filler content in PEEK compound significantly affected the fracture load ($p < 0.001$), with PEEK compound showing a slightly higher effect ($\eta_p{}^2 = 0.208$) than the veneering technique ($\eta_p{}^2 = 0.206$). PEEK with 30% filler content presented a higher fracture load ($p < 0.001$) compared to PEEK with 20% filler content. Aging presented no effect on fracture load results ($p = 0.176$). Regarding the veneering technique, the prefabricated veneerings showed a higher fracture load than the digital and conventional veneerings ($p \leq 0.001$), whereas the digital and conventional veneerings were similar ($p = 0.451$).

The Weibull statistics are presented in Table 3 for the framework material, veneering technique, and aging procedure. Prefabricated veneerings initially and after aging showed the highest Weibull modulus, followed by digital veneering and conventional veneerings for both PEEK materials. In PEEK with 30% filler content, the Weibull modulus after aging was highest for prefabricated veneerings, followed by conventional veneerings, and digital veneerings. This was in contrast with PEEK with 20% filler content with the highest Weibull modulus for digital veneerings followed by prefabricated veneerings, and conventional veneerings.

Regarding the fracture types (Fig. 5), no significant differences ($p = 0.055$) between the groups were observed for the different subgroups analyzed; they are described separately in the following section (Table 4).

Concerning initial fracture types, complete fractures were observed the most for digital followed by prefabricated, and conventional veneering for both PEEK framework materials tested. Adhesive fractures were seen the most for conventional, followed by prefabricated and digital veneerings for PEEK with 30% filler content and for conventional, followed by prefabricated veneerings for PEEK with 20% filler content. No adhesive failures occurred for digitally veneered PEEK with 20% filler content. Cohesive fractures occurred the most for prefabricated followed by digital and conventional veneerings for PEEK with 30% filler content. This was in contrast with digital followed by prefabricated veneerings for PEEK with 20% filler content. No cohesive fractures were found for conventionally veneered PEEK with 20% filler content.

**Table 2**
Descriptive statistics for fracture load [N] of different framework materials and veneering techniques at varying aging levels. Given are the means and standard deviations (SD) and the 95% confidence interval (CI).

| Framework material | Veneering technique | Initial | | After mastication simulation | |
|---|---|---|---|---|---|
| | | Mean ± SD | 95% CI | Mean ± SD | 95% CI |
| **PEEK** filled with 30% $TiO_2$ | **Digital veneering** | 3908 ± 207 | [3438; 4378] | 3544 ± 309 | [2844; 4234] |
| | **Veneering resin composite** | 3506 ± 337 | [2743; 4269] | 4217 ± 207 | [3747; 4686] |
| | **Prefabricated veneering** | 4548 ± 216 | [4058; 5038] | 4462 ± 118 | [4193; 4331] |
| **PEEK** filled with 20% $TiO_2$ | **Digital veneering** | 3232 ± 130 | [2938; 3526] | 3390 ± 237 | [2851; 3928] |
| | **Veneering resin composite** | 2449 ± 217 | [1958; 2940] | 3045 ± 311 | [2340; 3749] |
| | **Prefabricated veneering** | 3768 ± 146 | [3436; 4100] | 3884 ± 307 | [3884; 4580] |

**Table 3**
Weibull modulus of framework materials and veneering techniques investigated before and after artificial aging. Means with the 95% confidence interval are given.

| Framework material | Veneering technique | Weibull modulus | | | |
|---|---|---|---|---|---|
| | | Initial | | After mastication simulation | |
| **PEEK** filled with 30% $TiO_2$ | **Digital veneering** | 6.6 | [3.3; 12.8] | 4.1 | [2; 7.9] |
| | **Veneering resin composite** | 3.2 | [1.6; 6.2] | 7.1 | [3.6; 13.8] |
| | **Prefabricated veneering** | 7.5 | [3.8; 14.4] | 13.6 | [7; 26.2] |
| **PEEK** filled with 20% $TiO_2$ | **Digital veneering** | 8.5 | [4.3; 16.4] | 5.7 | [2.9; 11] |
| | **Veneering resin composite** | 4.4 | [2.2; 8.5] | 3.1 | [1.5; 6.1] |
| | **Prefabricated veneering** | 8.8 | [4.5; 16.9] | 3.7 | [1.8; 7.1] |

Concerning fracture types after aging, complete fractures were seen the most in digital followed by prefabricated and conventional veneerings for PEEK with 30% filler content and were seen the most for digital, followed by prefabricated, and conventional veneerings for PEEK with 20% filler content. Adhesive fractures were seen the most for conventional, followed by prefabricated and digital veneerings combined with PEEK with 30% filler content compared with PEEK with 20% filler content where most adhesive fractures occurred for conventional followed by digital, and prefabricated veneerings. No cohesive fractures were seen for conventional veneered PEEK with 30% filler content and for digitally and conventionally veneered PEEK with 20% filler content. For PEEK with 30% filler content, cohesive fractures were seen the most for digitally followed by prefabricated veneerings. For PEEK with 20% filler content, cohesive fractures occurred only for prefabricated veneerings.

No correlation between fracture types and fracture load was detected ($p = 0.303$).

## 4. Discussion

All examined FDPs survived mastication simulation, and the mechanical strength of the material was shown to be high enough to resist the masticatory forces which can reach up to 900 N in the posterior

**Table 4**
Fracture types of framework materials and veneering techniques investigated before and after artificial aging. The means and standard deviations (SD) and the 95% confidence interval (CI) are given.

| Framework material | Veneering technique | Fracture type | | | | | |
|---|---|---|---|---|---|---|---|
| | | Cohesive in veneering | | Adhesive between veneering and framework | | Complete fracture | |
| | | Mean ± SD. | 95% CI | Mean ± SD. | 95% CI | Mean ± SD. | 95% CI |
| Initial | | | | | | | |
| **PEEK** filled with 30% $TiO_2$ | **Digital veneering** | 20 | [2; 57] | 20 | [2; 57] | 60 | [25; 89] |
| | **Veneering resin composite** | 10 | [0; 45] | 80 | [43; 98] | 10 | [0; 45] |
| | **Prefabricated veneering** | 30 | [6; 66] | 30 | [6; 66] | 40 | [11; 75] |
| **PEEK** filled with 20% $TiO_2$ | **Digital veneering** | 10 | [0; 45] | 0 | | 90 | [54; 100] |
| | **Veneering resin composite** | 0 | | 90 | [54; 100] | 10 | [0; 45] |
| | **Prefabricated veneering** | 10 | [0; 45] | 40 | [11; 75] | 50 | [18; 82] |
| After artificial aging | | | | | | | |
| **PEEK** filled with 30% $TiO_2$ | **Digital veneering** | 50 | [18; 82] | 20 | [2; 57] | 30 | [6; 66] |
| | **Veneering resin composite** | 0 | | 90 | [54; 100] | 10 | [0; 45] |
| | **Prefabricated veneering** | 30 | [6; 66] | 40 | [11; 75] | 30 | [6; 66] |
| **PEEK** filled with 20% $TiO_2$ | **Digital veneering** | 0 | | 30 | [6; 66] | 70 | [34; 94] |
| | **Veneering resin composite** | 0 | | 90 | [54; 100] | 10 | [0; 45] |
| | **Prefabricated veneering** | 50 | [18; 82] | 20 | [2; 57] | 30 | [6; 66] |

region (Waltimo and Könönen, 1995; Varga et al., 2011).

The two aspects of the null hypotheses concerning the framework materials and veneering techniques were rejected as the different filler content in the framework material and veneering technique led to differences in the fracture load of implant-supported 4-unit PEEK FDPs. The third aspect of the null hypothesis was not rejected, as aging had no impact on fracture load.

FDPs with frameworks pressed from experimental BioHPP filled with 30% of inorganic fillers showed a higher fracture load than FDPs with frameworks milled from BioHPP blanks filled with 20% of filler content. FDPs made of materials with the same percentage of fillers but differently processed in the laboratory have been reported to exhibit different fracture resistance. Milled BioHPP has been reported to be more resistant in than pressed BioHPP in three-unit FDPs (both filled with 20% of fillers) (Taufall et al., 2016) while pressed and 3D printed BioHPP were in the same value range (Prechtel et al., 2020b) but tested as inlays. Nevertheless, in the present study, two different PEEK materials were tested and even though experimental BioHPP was processed by pressing, it showed a higher fracture load than the milled one. The better stability of this material might be explained by material composition with a higher percentage of inorganic fillers.

A tendency for the fracture load values to increase with highly filled FDPs was detected. However, since these FDPs are pressed, there could be a further increase when higher filler content is incorporated into milled materials. Furthermore, although all examined FDPs were prepared and veneered according to the same procedure, the FDPs veneered using prefabricated veneerings showed a higher fracture load than digital and conventional veneerings. This finding was not consistent with the results of previous studies where digital veneering provided the highest fracture resistance (Taufall et al., 2016; Preis et al., 2017). In addition, the conventional veneering using resin composite showed the lowest results, which was consistent with these previous studies. The explanation may come from the process of fabrication of CAD-CAM blocks or prefabricated veneerings under controlled and standardized conditions with defined pressure and temperature, which leads to reduced residual monomer and therefore increased mechanical properties (Preis et al., 2017).

Weibull statistics have been used as a convenient tool in dental material science to assess reliability (Quinn and Quinn, 2010). Higher Weibull modulus $m$ indicates higher structural stability and reliability of the material. The lowest Weibull modulus was found with the FDPs with frameworks made of PEEK with 20% filler-content conventionally veneered with resin composite after mastication simulation. This can be explained by the lower percentage of fillers in the framework material and the poor mechanical properties of the veneering resin composite. FDPs with frameworks pressed from PEEK with 30% filler-content with prefabricated veneers exhibited the highest values. The explanation may come from the high filler percentage in the framework material composition and in the use of prefabricated veneerings which were manufactured under controlled industrial conditions.

Relative frequencies of fracture types were analyzed using the Ciba-Geigy table, with no typical fracture type observed for different framework materials or veneering techniques at different aging levels. This finding was inconsistent with that of a previous investigation that reported that the fracture usually occurred in the pontic region (Taufall et al., 2016). The explanation may come from the antagonist design and the manner of applying force during the mastication simulation or testing of the fracture load. In the present investigation, the force was applied on each unit of 4-unit FDPs, unlike the Taufall et al. (2016) experiment where the force was applied directly on the pontic.

A limitation of this study is the lack of fractography of the fracture types. This should be carried out in following investigations to provide further information.

Mastication simulation was used to predict the behavior of the restorative material after use under clinical conditions. FDPs were exposed to 1.2 million cycles, which simulated 5-years of clinical use (Rosentritt et al., 2006, 2008). When selecting the appropriate material, the fatigue and fracture resistance could essentially contribute to the long-term success of the restoration (Preis et al., 2017). This experiment exhibited no impact of artificial aging on the fracture load of the examined FDPs; thus, one part of the null hypothesis that aging would have no effect on fracture load was not rejected.

Implant-supported fixed dental restorations might be more susceptible to overloading in comparison with tooth-supported restorations because of the absence of a periodontal ligament to absorb the masticatory forces (de Kok et al., 2015). The lack of a periodontal ligament explains why chipping and fractures are reported more often on implant-supported than on tooth-supported fixed restorations (Preis et al., 2017). More elastic materials, such as PEEK, may be beneficial in implant-prosthodontics to overcome the lack of a periodontal ligament.

The present investigation was consistent with systematic reviews (Storelli et al., 2018; Schmid et al., 2020) reporting that implant-supported FDPs could withstand occlusal forces in the posterior region, given the limitations of in vitro study design. Another investigation reported no significant differences in survival rates between cantilevered and fixed-to-fixed posterior ceramic FDPs, even after a follow-up period of 13 years (Passia et al., 2019), which may encourage the use of cantilever FDPs and promote further research in this direction and into other materials or support forms (natural teeth or implants).

The present investigation shows that PEEK has promising characteristics as an appropriate material for implant-supported FDPs. Despite the fact that the design of the FDPs used in this investigation is beyond the recommended indication area, the results showed its reliability even in a cantilever design. Further investigations should include more PEEK and composite materials, as well as fabrication methods (3D-printed PEEK).

## 5. Conclusions

Within the limitation of the study, the following conclusions were drawn:

- All implant-supported 4-unit PEEK FDPs showed a higher fracture load than the maximum occlusal forces in the posterior region.
- Mastication simulation had no impact on the fracture resistance of the examined PEEK FDPs.
- A higher percentage of inorganic fillers in the PEEK compound led to better mechanical stability of the examined PEEK FDPs.
- The veneering technique highly influences the long-term stability of implant-supported 4-unit PEEK FDPs. Selecting the appropriate veneering method can improve the long-term success of bi-layered structures.

## CRediT authorship contribution statement

**Danka Micovic Soldatovic:** Writing – original draft, Methodology, Investigation. **Anja Liebermann:** Writing – review & editing, Supervision. **Karin C. Huth:** Writing – review & editing. **Bogna Stawarczyk:** Writing – review & editing, Supervision, Methodology, Data curation, Conceptualization.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Acknowledgements

The authors would like to thank bredent for supporting the investigation with materials and equipment.

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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# 4. Publication 2

## Impact of material combinations and removal and insertion cycles on the retention force of telescopic systems

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Clin Oral Investig. 2023 Jul;27(7):4007-4016.

DOI:10.1007/s00784-023-05027-w.

Clinical Oral Investigations
https://doi.org/10.1007/s00784-023-05027-w

RESEARCH

# Impact of material combinations and removal and insertion cycles on the retention force of telescopic systems

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Received: 2 January 2023 / Accepted: 16 April 2023


## Abstract

**Objectives** A variety of dental materials are available for the fabrication of telescopic crowns. The aim was to investigate the impact of material combinations and removal and insertion cycles on their retention forces.

**Materials and methods** CAD/CAM-fabricated cobalt–chromium–molybdenum (CoCr) and zirconia ($ZrO_2$) primary crowns were combined with polyetheretherketone (PEEK), polyetherketoneketone (PEKK), CoCr, and $ZrO_2$ secondary crowns (four combinations included PEEK/PEKK secondary crowns in a thickness of 0.5 mm bonded to the CoCr tertiary construction), resulting in 12 different material combinations: CoCr–PEEK; CoCr–PEKK; CoCr–$ZrO_2$; CoCr–CoCr; CoCr–PEEK 0.5; CoCr–PEKK 0.5; $ZrO_2$–PEEK; $ZrO_2$–PEKK; $ZrO_2$–$ZrO_2$, $ZrO_2$–CoCr; $ZrO_2$–PEEK 0.5; and $ZrO_2$–PEKK 0.5 ($n = 15$ pairings per material combination). Pull-off tests were performed with a universal testing machine initially and after 500, 5000, and 10,000 removal and insertion cycles in a mastication simulator. Descriptive statistics with the Kolmogorov–Smirnov, Kruskal–Wallis, and Mann–Whitney $U$ tests were computed ($\alpha = 0.05$).

**Results** The tested parameters, material combination, and removal and insertion cycles had significant impact on the retention force values ($p < 0.001$). An increase in removal and insertion cycles was associated with a decrease in retention forces within CoCr and $ZrO_2$ secondary crowns, regardless of the primary crown material. In contrast, PEEK and PEKK secondary crowns presented higher retention load values after 10,000 cycles than initially.

**Conclusion** Different material combinations behaved differently after simulated removal and insertion regimens. This difference should be considered during treatment planning.

**Clinical relevance** Telescopic crown systems should be made of materials with predictable retention forces that do not deteriorate with time. The implementation of new materials and technologies facilitates reproducibility and time-saving fabrication.

**Keywords** Double-crown system · PEEK · PEKK · Zirconia · Artificial aging · Retention force measurements

## Introduction

Telescopic crown-retained removable partial dentures (RPDs) provide a suitable treatment option for partially edentulous patients with multiple missing teeth or in combination with extended edentulous ridges where fixed dental restorations are not indicated. These RPDs could be tooth-supported or tooth-implant-supported [1], depending on the individual situation. According to the clinical evaluations, telescopic crown-retained RPDs have been reported to show higher survival rates than conventional clasp-retained RPDs [2].

A telescopic crown system consists of a primary crown (fixed to abutment tooth or implant) and a secondary crown which is a part of the denture [3]. The contact surfaces of the

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primary and secondary crowns can be made almost parallel (taper angle very close to 0°). Another option is the provision of conical crowns with a recommended convergences angle of 2 to 6°. Taper, height of contact surfaces, and materials directly influence the retention values of telescopic crown systems [4–6].

In the past, precious alloys (mainly gold) were used for manufacturing primary and secondary crowns. Gold alloys have excellent biocompatibility, can be easily processed by a dental laboratory technician, and allow the required retention forces to be adjusted [7]. Due to the high cost of precious alloys, non-precious alloys, for example, cobalt–chromium–molybdenum (CoCr), have been used for this application. CoCr crowns can be conventionally manufactured using the lost-wax casting technique, but, due to the higher modulus of elasticity (≈ 210 GPa) in comparison with gold alloys (≈ 150 GPa), the process of fabrication and adaptation is more difficult and error-sensitive. Recently, with the help of computer-aided design and computer-aided manufacturing (CAD/CAM), CoCr telescopic crowns can be milled or even 3D printed. Although this alloy exhibited satisfactory characteristics regarding retentive behavior [7], precise fitting, and flexural strength [8], its biocompatibility is questionable. The combination of CoCr alloy with other metal alloys in wet oral conditions could lead to the dissolution of metal ions and galvanic corrosion [9]. The current trend towards non-metallic restorations and increased esthetic demands, as well as the high number of allergy-prone patients, has led to the introduction of new prosthodontic materials.

Advanced CAD/CAM dental technologies led to rapid and cost-effective production, overcoming the problems of conventional casting [5]. CAD/CAM technology also enabled the use of improved ceramic and polymer-based materials, including zirconia and polyaryletherketone (PAEK).

Zirconia has excellent esthetic and mechanical properties, biocompatibility, and long-term stability, all of which make it suitable for implants, abutments, frameworks for fixed dental restorations, and monolithic fixed prostheses [10, 11]. Zirconia has been reported to be a suitable primary crown material [12], especially in combination with electroformed gold secondary crowns [13]. However, the combination of zirconia with non-precious alloy secondary crowns has been reported to cause significant wear and loss of friction [5]. Studies testing zirconia as a secondary crown material have reported contradictory findings [14], and further studies are necessary for more accurate results and for providing reliable recommendations.

Polymer-based dental materials, including PAEK, have become popular with CAD/CAM systems. The PAEK family consists of a variety of high-performance thermoplastic polymers which differ in the number of functional ether- or keto-groups. These include polyetheretherketone (PEEK), polyetherketoneketone (PEKK) [15], and the recently developed high-performance aryl-ketone polymer (AKP) [16]. Because of their slightly different composition, their properties and thus also the indication area differ [17]. PEEK has been previously tested as part of a telescopic crown system in a few in vitro investigations and was reported as a suitable material for this indication [3, 18]. However, the authors are only aware of a case report [19] and an in vitro study [20] that examined PEKK as a telescopic crown material, reporting promising results for this indication. Excellent mechanical properties, biocompatibility, chemical stability, low plaque adhesion, and a broad range of processing options (milling, pressing, 3D-prinitng) make PAEK materials attractive for wider implementation in prosthetic dentistry [21].

Another traditional approach is the electroplating of secondary crowns. In this procedure, gold ions are deposited under electric current to produce accurately fitting gold copings [22]. Crowns made in this way do not need to be adjusted, as do conventionally cast secondary crowns, but are intraorally bonded to the tertiary structure, ensuring a passive fit of the restoration with excellent stress distribution. However, the technically demanding and time-consuming fabrication process leads to an expensive dental restoration, and whether milled PEEK or PEKK copings could be an affordable alternative to gold is unclear. Milled PEEK or PEKK might overcome the drawbacks of the electroplating technique by facilitating the fabrication of reproducible copings and the passive fit of the tertiary structure. If the retention force changes over time, a PEEK or PEKK coping could be easily replaced without fabricating a completely new restoration.

The goals of this investigation were to examine and compare the behavior of the retention forces of different material pairings, simulating function with artificial aging (removal and insertion cycles). The null hypotheses were that material combinations would not impact retention force on one aging level and that thermomechanical aging would not impact the retention force values of one material combination.

## Materials and methods

The retention load of telescopic crowns made of different materials was investigated in the present investigation (Table 1). Cobalt–chromium–molybdenum (CoCr) alloy and zirconia ($ZrO_2$) were used as primary crown materials. For each material, 15 secondary crowns were produced using polyetheretherketone (PEEK), polyetherketoneketone (PEKK), CoCr alloy, and $ZrO_2$.

Four groups were designed as three-element systems with tertiary constructions where the primary crowns were made of CoCr/$ZrO_2$ and secondary PEEK/PEKK in a thickness of

**Table 1** Summary of used materials

| | Material | Manufacturer | LOT number |
|---|---|---|---|
| • Primary crown | Cobalt–chromium–molybdenum (CoCr), Ceramill sintron | Amann Girrbach | 1303045, 1700661 |
| | Zirconia ($ZrO_2$), Ceramill ZI | Amann Girrbach | 1303002 |
| • Secondary crown | Polyetheretherketone (PEEK), BioHPP | bredent | 504894, 496211, 495767, 486101 |
| | Polyetherketoneketone (PEKK), Pekkton | Cendres+Métaux | 204280, 211144, 211145 |
| | Cobalt-chromium (CoCr) | a.m. | a.m. |
| | Zirconia ($ZrO_2$) | a.m. | a.m. |
| • Tertiary crown | Cobalt-chromium (CoCr) | a.m. | a.m. |
| • Bonding | AGC Cem Automix System | C. Hafner | 220868 |
| | visio.link | bredent | 193211 |
| | MKZ primer | bredent | 494986 |

0.5 mm simulating the electroplated copings and CoCr tertiary crowns (Fig. 2). This resulted in 12 groups of material pairings with 15 specimens per group (Fig. 1).

## Specimen manufacturing

### Primary crowns

To obtain a basis for the abutments, a prepared plastic model of the maxillary first molar (26) was duplicated with a silicone mold (Adisil blau 9:1, Siladent). Thirty wax abutments were manufactured and converted into CoCr abutments (Remanium GM 800+, Dentaurum) using the conventional lost-wax technique.

Each of these abutments was scanned (Ceramill map 300, Amann Girrbach) and digitized in a CAD software program (Ceramill Mind, Amann Girrbach). Based on this, parallel primary crowns (cone angle 0°) with chamfer preparation were designed and subsequently milled from CoCr alloy (Ceramill Sintron, Amann Girrbach) and zirconia (Ceramill ZI, Amann Girrbach). The primary crowns were sintered according to the manufacturer's instructions: CoCr crowns in a protective atmosphere (argon: 1 bar, compressed air: 1.2 bar; Ceramill Argotherm, Amann Girrbach) and zirconia crowns following the program: heat up to 1450 °C (5–10 K/

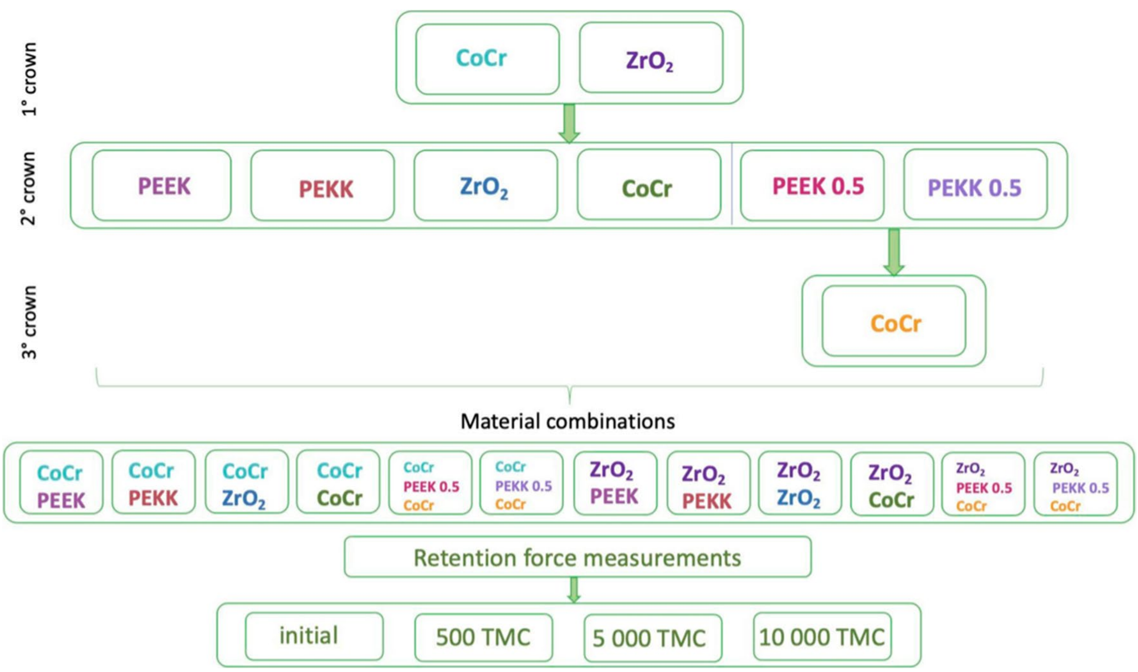


**Fig. 1** Study design

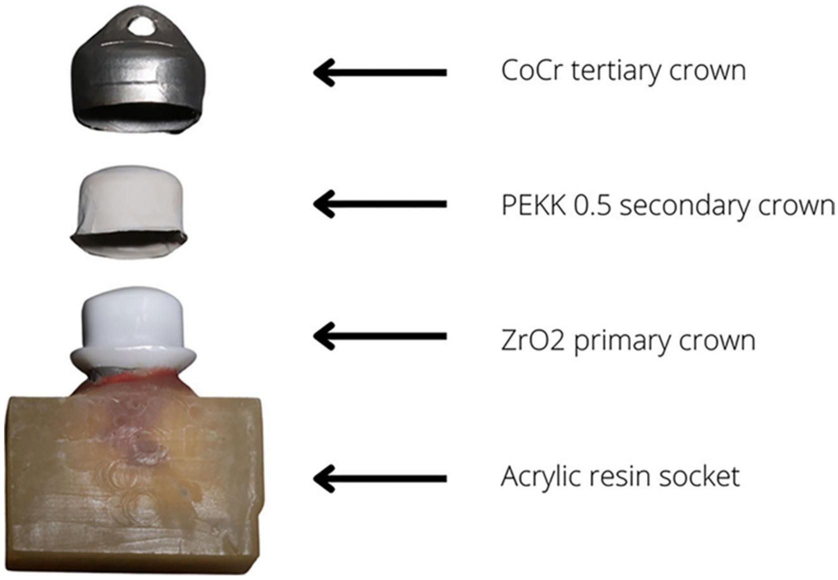


**Fig. 2** Three-element system components

min), with a dwell time of 2 h and a cooling rate of 5 K/min until room temperature.

Both types of sintered primary crowns were adhesively bonded to the abutments (RelyX Unicem 2, 3M). All bonded primary crowns were parallel mounted in acrylic resin sockets (Scandiquick, Scan-Dia) to ensure stability during pull-off tests and subjected to artificial aging. The insertion direction was defined using a turbine (W&H Perfecta 900; W&H Dentalwerk) positioned in a parallelometer (F4 basic, DeguDent) with constant water cooling. All primary crowns ($n$ = 15 CoCr, $n$ = 15 $Zr0_2$ crowns) were high-gloss polished.

### Secondary crowns

A total of 180 secondary crowns were manufactured using four different materials. Each primary crown was scanned (Ceramill map 300) to construct a corresponding secondary crown with a CAD software program (Ceramill Mind). All secondary crowns were constructed individually without cement spacer or block outs, with a 2-mm thickness and a ridge on the occlusal surface (provided to make a hole to perform pull-off tests and mount specimens in the mastication simulator). The same STL data were used to mill (Ceramill Motion 2) PEEK (breCAM.BioHPP, bredent) and PEKK (Pekkton, Cendres+Métaux) secondary crowns due to the similarity of materials. In order to mill CoCr (Ceramill Sintron, Amann Girrbach) and $ZrO_2$ (Ceramill ZI, Amann Girrbach) secondary crowns, the parameters, like cement spacer, were adjusted so that all types of secondary crowns had at the baseline retention force of 10–15 N.

Previously used STL files were optimized to mill PEEK and PEKK secondary crowns which were used in combination with CoCr tertiary crowns. All the parameters were the same except for the thickness of the crown (reduced to 0.5 mm), and they were constructed without an occlusal ridge: PEEK 0.5 and PEKK 0.5.

This resulted in 12 different groups of material pairings (Table 1; Fig. 1).

### Tertiary crowns

For four groups (CoCr–PEEK 0.5; CoCr–PEKK 0.5; $ZrO_2$–PEEK 0.5; and $ZrO_2$–PEKK 0.5) tertiary crowns were fabricated (Fig. 2). The secondary PEEK/PEKK 0.5 crown was positioned on its corresponding primary crown and then scanned and digitized. The tertiary construction (with occlusal ridge) was milled from CoCr, sintered, and high-gloss polished using polishing brushes and paste (Komet Dental; Abraso-Starglanz, bredent).

Prior to initial retention force measurements, secondary PEEK 0.5/PEKK 0.5 crowns were adhesively bonded to tertiary CoCr crowns and incubated for 24 h at 37 °C (HERAcell 150, Thermo Scientific).

The bonding procedure consisted of airborne-particle abrasion of both surfaces (PEEK/PEKK and CoCr) with 50-µm $Al_2O_3$, with a pressure of 2 bars, cleaning in an ultrasound bath, applying a thin layer of MKZ primer (bredent) on the CoCr intaglio surface, drying for 60 s, applying a thin layer of visio.link (bredent) on the outer PEEK/PEKK surface and polymerization for 90 s (bre.lux power unit, bredent), filling CoCr crown with AGC autopolymerizing compomer cement (AGC Cem Automix system, C Hafner), and pressing onto the secondary crown which had been positioned on corresponding primary crown which had been previously isolated with a thin layer of Vaseline.

The fit of every secondary crown was tested, and the initial retention force was adjusted by grinding the intaglio surface of the secondary crown to obtain 10–15 N for each pairing. After the adjustment, the intaglio surfaces were polished, and the crowns made ready for the initial retention force measurements.

### Retention force measurement and artificial aging

Retention force measurements were performed in a universal testing machine (Zwick 1445, Zwick/Roell). The primary crown on its acrylic resin base was fixed in the machine. The secondary crown was wetted with an artificial saliva spray (Glandosane, cell pharm) and fitted onto the primary crown. Using a hook through the hole in the occlusal ridge of the secondary crown, pull-off tests were done at a speed of 50 mm/min (Fig. 3). The experimental setup was already proven in several investigations [8, 13, 23, 25, 26].

According to the study design (Fig. 1), each specimen was exposed to 500, 5000, and 10,000 thermomechanical cycles in a mastication simulator (SD Mechatronic). The cycles corresponded to approximately 6 months, 5 years, and 15 years (respectively) in clinical conditions when patients remove the restoration three times per day [3, 18, 20]. A mechanical load of 50 N was applied, and the thermal cycles consisted of temperature changes between 5 and 55 °C with a 60-s dwell time.

A parallelometer was used to ensure the specimens were in the same position each time they were mounted in the mastication simulator. Acrylic resin sockets with primary crowns and secondary crowns were fixed for the antagonistic parts of the mastication simulator, securing the path of insertion during aging. Pull-off tests and loading during aging were executed in an axial direction, parallel to the insertion direction and perpendicular to the model base. After each aging interval, five pull-off tests per pairing were performed and retention force values were recorded.

### Statistical analyses

For power analysis, the results from a prior study [23] on the initial retention load of primary and secondary zirconia crowns (17.63 ± 5.16 N) were used for calculation (nQuery + nTerim, Version 3.0, Statistical Solutions). The aim of this power analysis was to determine cross-sectional differences after aging using chewing simulator between the tested secondary crowns. A sample size of 15 in each of 12 material combinations had a 95% power to detect a difference in retention load means of 8.81 N (50% reduction), assuming that the common standard deviation of retention load was 5.16 N using a two group $t$-test with a Bonferroni corrected two-sided significance level ($\alpha = 0.008$).

The assumption of normality was tested with the Kolmogorov–Smirnov test. The descriptive statistics mean, standard deviation (SD), and 95% confidence interval (CI) were computed. The Kruskal–Wallis test was used to disclose differences in mean retention load between 12 tested material combinations. The Mann–Whitney $U$ test was performed to estimate the effect of material combination and removal and insertion cycles on retention load values. A statistical software program (IBM SPSS Statistics, version 26.0.0.1, IBM Corp) was used for the analyses ($\alpha = 0.05$).

## Results

As the measured data deviated from normal distribution (64.6%), non-parametric tests were performed (Table 2). The tested parameters, material combination, and removal and insertion cycles were shown to impact the retention force values ($p < 0.001$). The highest impact showed material combination ($\eta_p^2 = 0.542$), followed by interaction between material

**Fig. 3** Retention force measurement setup

**Table 2** Descriptive statistics. All values for retention load in Newton (N)

| Material combinations | | | Removal and insertion cycles | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Initial | | 500 TMC | | 5000 TMC | | 10,000 TMC | |
| 1° | 2° | 3° | Mean ± SD | [95% CI] | Mean ± SD | [95% CI] | Mean ± SD | [95% CI] | Mean ± SD | [95% CI] |
| CoCr | PEEK | | $13.1 \pm 2.9^{*\mathrm{ef\,A}}$ | [12.2; 13.8] | $12.0 \pm 4.2^{\mathrm{cd\,A}}$ | [10.9; 13.0] | $17.3 \pm 5.6^{\mathrm{df\,B}}$ | [15.8; 18.6] | $20.3 \pm 4.6^{\mathrm{f\,C}}$ | [19.1; 21.4] |
| CoCr | PEKK | | $11.6 \pm 2.5^{*\mathrm{cde\,A}}$ | [10,9; 12,1] | $10.4 \pm 3.5^{*\mathrm{c\,A}}$ | [9.5; 11.3] | $18.3 \pm 5.2^{\mathrm{f\,B}}$ | [17.0; 19.6] | $19.6 \pm 5.5^{*\mathrm{ef\,B}}$ | [18.2; 21.0] |
| CoCr | $ZrO_2$ | | $10.4 \pm 2{,}8^{*\mathrm{abc\,D}}$ | [9.3; 10.8] | $6.1 \pm 3.3^{\mathrm{ab\,C}}$ | [5.2; 6.9] | $3.9 \pm 2.9^{*\mathrm{a\,B}}$ | [3.1; 4.7] | $2.2 \pm 1.9^{*\mathrm{a\,A}}$ | [1.6; 2.7] |
| CoCr | CoCr | | $12.2 \pm 2.3^{\mathrm{def\,B}}$ | [11.6; 12.8] | $7.6 \pm 2.7^{\mathrm{b\,A}}$ | [6.9; 8.3] | $7.7 \pm 3.9^{*\mathrm{bc\,A}}$ | [6.6; 8.7] | $9.1 \pm 4.7^{*\mathrm{bc\,A}}$ | [7.9; 10.3] |
| CoCr | PEEK 0.5 | CoCr | $13.1 \pm 2.1^{*\mathrm{ef\,A}}$ | [12.6; 13.7] | $12.4 \pm 3.2^{*\mathrm{cde\,A}}$ | [11.6; 13.2] | $16.8 \pm 5.2^{\mathrm{df\,B}}$ | [15.5; 18.1] | $15.8 \pm 5.6^{\mathrm{d\,B}}$ | [14.4; 17.2] |
| CoCr | PEKK 0.5 | CoCr | $13.3 \pm 2.0^{\mathrm{f\,A}}$ | [12.7; 13.8] | $15.7 \pm 3.7^{*\mathrm{f\,B}}$ | [14.7; 16.6] | $18.5 \pm 4.7^{*\mathrm{f\,C}}$ | [17.3; 19.7] | $15.9 \pm 3.9^{*\mathrm{d\,B}}$ | [14.9; 16.9] |
| $ZrO_2$ | PEEK | | $9.1 \pm 2.2^{*\mathrm{a\,B}}$ | [8.4; 9.6] | $7.4 \pm 2.3^{*\mathrm{b\,A}}$ | [6.7; 8.0] | $9.7 \pm 2.8^{*\mathrm{c\,B}}$ | [8.9; 10.1] | $11.1 \pm 3.8^{\mathrm{c\,C}}$ | [10.1; 12.1] |
| $ZrO_2$ | PEKK | | $12.1 \pm 2.6^{*\mathrm{def\,A}}$ | [11.3; 12.8] | $10.8 \pm 3.4^{*\mathrm{cd\,A}}$ | [9.8; 11.6] | $15.0 \pm 4.1^{*\mathrm{d\,B}}$ | [13.9; 16.0] | $18.1 \pm 7.8^{*\mathrm{def\,C}}$ | [16.1; 20.1] |
| $ZrO_2$ | $ZrO_2$ | | $9.8 \pm 2.3^{*\mathrm{ab\,D}}$ | [9.1; 10.4] | $4.6 \pm 2.5^{*\mathrm{a\,C}}$ | [4.0; 5.3] | $2.7 \pm 2.8^{*\mathrm{a\,B}}$ | [2.0; 3.5] | $1.6 \pm 1.3^{*\mathrm{a\,A}}$ | [1.2; 2.0] |
| $ZrO_2$ | CoCr | | $10.9 \pm 2.0^{\mathrm{bcd\,B}}$ | [10.4; 11.5] | $4.8 \pm 2.5^{*\mathrm{a\,A}}$ | [4.1; 5.5] | $5.4 \pm 4.5^{*\mathrm{ab\,A}}$ | [4.2; 6.5] | $6.1 \pm 5.0^{*\mathrm{b\,A}}$ | [4.9; 7.4] |
| $ZrO_2$ | PEEK 0.5 | CoCr | $13.0 \pm 2.3^{\mathrm{ef\,A}}$ | [12.4; 13.6] | $13.0 \pm 3.3^{*\mathrm{de\,A}}$ | [12.1; 13.9] | $15.0 \pm 5.1^{*\mathrm{d\,A}}$ | [13.7; 16.2] | $16.1 \pm 6.4^{*\mathrm{de\,B}}$ | [14.5; 17.7] |
| $ZrO_2$ | PEKK 0.5 | CoCr | $13.7 \pm 1.5^{\mathrm{f\,A}}$ | [13.3; 14.1] | $14.3 \pm 3.5^{\mathrm{ef\,A}}$ | [13.4; 15.2] | $18.8 \pm 5.1^{\mathrm{f\,B}}$ | [17.5; 20.1] | $18.5 \pm 4.3^{\mathrm{def\,B}}$ | [17.3; 19.6] |

*Not normal distributed groups

$^{\mathrm{a,\,b,\,c,\,d,\,e,\,f}}$Different homogeneity material combination

$^{\mathrm{A,\,B,\,C,\,D}}$Different homogeneity removal and insertion cycles combination and removal and insertion cycles ($\eta_p^2 = 0.266$) and removal and insertion cycles ($\eta_p^2 = 0.079$). Descriptive statistics are summarized in Table 2. All material combinations showed differences in retention load values regardless of removal and insertion cycles ($p < 0.001$) (Table 2).

Within initial measurements, $ZrO_2$–PEEK showed lower values compared with $ZrO_2$–CoCr, CoCr–PEKK, $ZrO_2$–PEKK, CoCr–CoCr, $ZrO_2$–PEEK 0.5, CoCr–PEEK, CoCr–PEEK 0.5, CoCr–PEKK 0.5, and $ZrO_2$–PEKK 0.5 ($p < 0.001$). The highest values were found for $ZrO_2$–PEKK 0.5 and CoCr–PEKK 0.5. These groups showed higher retention load values than CoCr–PEKK, $ZrO_2$–CoCr, CoCr–$ZrO_2$, $ZrO_2$–$ZrO_2$, and $ZrO_2$–PEEK ($p < 0.001$).

After 500 removal and insertion cycles, $ZrO_2$–$ZrO_2$ and $ZrO_2$–CoCr showed similar retention load values ($p = 0.625$) which were significantly lower in comparison with $ZrO_2$–PEEK, CoCr–CoCr, CoCr–PEKK, $ZrO_2$–PEKK, CoCr–PEEK, CoCr–PEEK 0.5, $ZrO_2$–PEEK 0.5, $ZrO_2$–PEKK 0.5, and CoCr–PEKK 0.5 ($p < 0.001$). The highest retention load values after 500 cycles were for the CoCr–PEKK 0.5 material combination which were similar to the $ZrO_2$–PEKK 0.5 ($p = 0.097$) but differed significantly ($p < 0.001$) from all other material combinations.

After 5000 removal and insertion cycles, the lowest retention load values were for $ZrO_2$–$ZrO_2$ and CoCr– $ZrO_2$ compared with CoCr–CoCr, $ZrO_2$–PEEK, $ZrO_2$–PEKK, $ZrO_2$–PEEK 0.5, CoCr–PEEK 0.5, CoCr–PEEK, CoCr–PEKK, CoCr–PEKK 0.5, and $ZrO_2$–PEKK 0.5 ($p < 0.001$). The highest values were measured for $ZrO_2$–PEKK 0.5 which were similar to CoCr–PEKK 0.5 ($p = 678$), CoCr–PEKK ($p = 0.453$) and CoCr–PEEK ($p = 0.067$) material combinations. They differed significantly from $ZrO_2$–PEEK, CoCr–CoCr, $ZrO_2$–CoCr, CoCr– $ZrO_2$, and $ZrO_2$–$ZrO_2$ ($p < 0.001$).

After 10,000 removal and insertion cycles, CoCr–PEEK exhibited the highest values, which were similar to those of CoCr–PEKK ($p = 0.232$). Between CoCr–PEKK and $ZrO_2$–PEKK 0.5, there was also no significant difference ($p = 0.519$). $ZrO_2$–$ZrO_2$ and CoCr–$ZrO_2$ had the lowest retention load values after 10,000 cycles, which was significantly different from those of all other material combinations ($p < 0.001$).

The behavior of all tested material combinations after different aging regimens is illustrated in Fig. 4.

An increase in removal and insertion cycles showed differences in retention load values independent of material combinations ($p < 0.01$) (Table 2; Fig. 5). Within CoCr–PEEK, CoCr–PEKK, CoCr–PEEK 0.5, CoCr–PEKK 0.5, $ZrO_2$PEKK, and $ZrO_2$PEKK 0.5 material combination, the initial retention force and after 500 cycles showed lower values than after 5000 and 10,000 cycles. In addition, within CoCr–PEEK and $ZrO_2$–PEKK, material combination retention force increased between 5000 and 10,000 cycles and within CoCr–PEKK 0.5; no differences were found between 500 and 10,000 cycles. Within $ZrO_2$–PEEK 0.5 material combination after 10,000 cycles, higher values

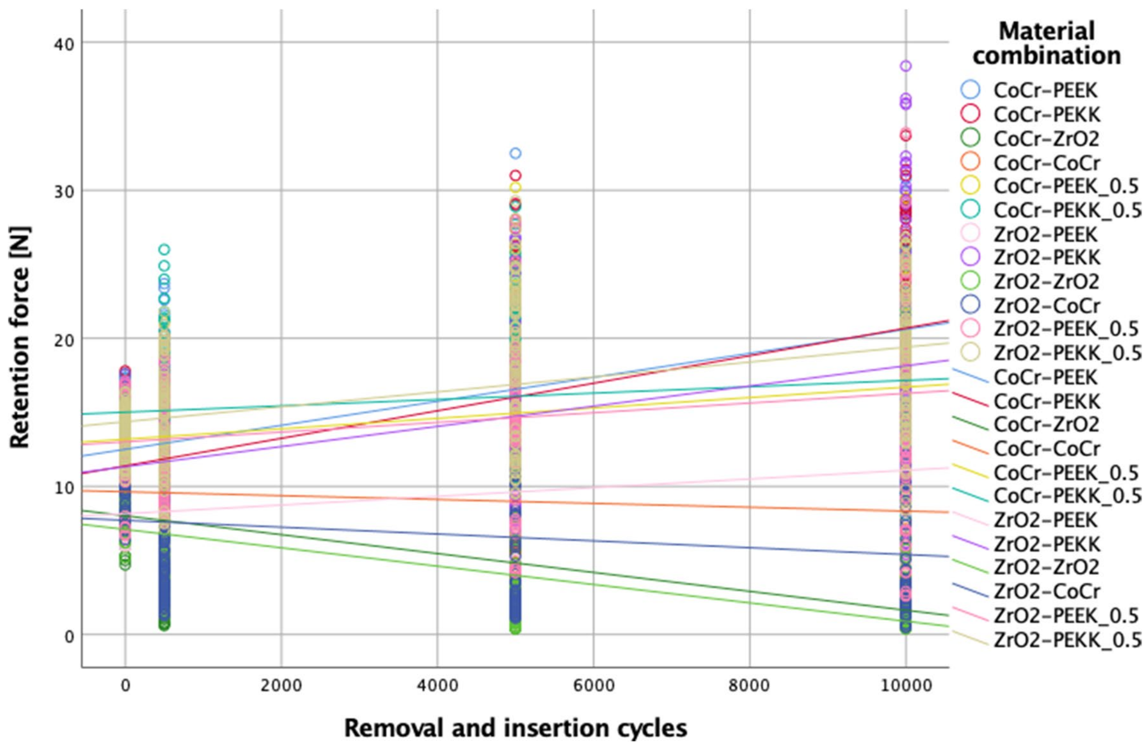


**Fig. 4** Retention load values of different material combinations measured initially and after 500, 5000, and 10,000 removal and insertion cycles

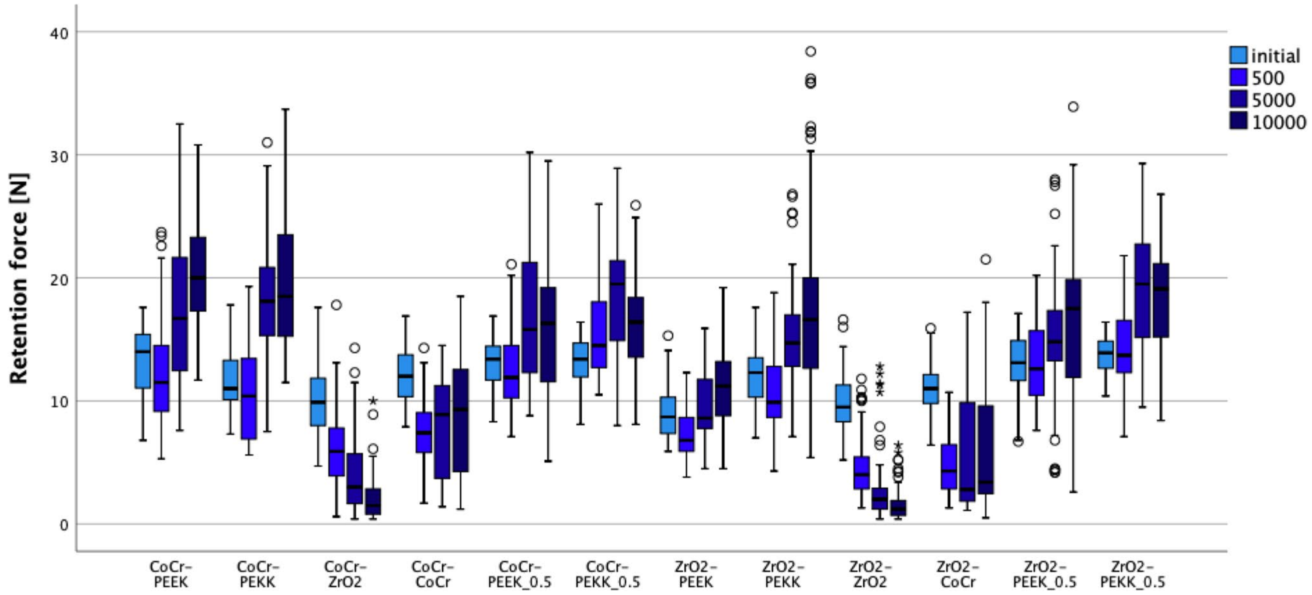


**Fig. 5** Performance of different material pairings initial and after 500, 5000, and 10,000 removal and insertion cycles

were found compared with initial and 500 cycles. Within the $ZrO_2$–PEEK material combination, the lowest values were found after 500 and the highest after 10,000 cycles.

In contrast, within the CoCr–$ZrO_2$ and $ZrO_2$–$ZrO_2$ material combination, a decrease in retention load was observed with an increase in removal and insertion cycles. Within the CoCr–CoCr and $ZrO_2$–CoCr material combination, initial values showed higher retention force than after removal and insertion cycles (Fig. 5).

## Discussion

This investigation examined the influence of material combinations and removal and insertion cycles on the retention load values of telescopic systems. Specimens exposed to 500, 5000, and 10,000 removal and insertion cycles in combination with temperature changes simulated a clinical lifetime of more than 15 years.

The obtained results showed that material combination as well as removal and insertion cycles significantly impacted the retention load values (Fig. 4); therefore, both null hypotheses were rejected. The retention forces declined constantly and significantly when zirconia secondary crowns were tested on both types of primary crowns. Similar results presented for CoCr secondary crowns where initial retention forces were higher than after thermomechanical cycling. This retention reduction might be explained by wear from the friction between the contacting surfaces during the removal and insertion cycles.

Within material combinations which included PEEK or PEKK, an increase of retention forces was observed between baseline and 10,000 cycles. This increase could be explained by the elasticity and adaptability as well as by the reduced wear of polymer materials and was consistent with a previous investigation [24] which reported that a PEEK–PEEK combination remained constant during aging but that PEEK secondary crowns in combination with $ZrO_2$ primary crowns exhibited an increase in retention force. On the other hand, the increase of retentive forces may be the consequence of interfacial wear or deformation leading to settling of the components of telescopic system. This can result in tight fit beyond that which is clinically acceptable. Hence, the increase of retention forces cannot be always understood as an advantage, and further investigations, including SEM imaging, shall provide us with more information. The retention force of PEEK crowns was also raised by increasing the number of pairings tested simultaneously.

The authors are unaware of a previous study that tested PEEK or PEKK secondary crowns as part of a three-system prosthesis. The hypothesis was to determine whether PEEK/PEKK crowns in a thickness of 0.5 mm could replace gold copings as part of a three-system prosthesis to reduce costs, avoid technically demanding procedures, and achieve reproducibility and completely metal-free restorations. According to the obtained results, retention forces measured within the material combinations, including PEEK/PEKK_0.5, showed comparable behavior with that of the PEEK and PEKK secondary crowns, indicating that an increase in retention force values was observed with increased removal and insertion cycles.

The high retention load values obtained during this experiment might be explained by the relatively large friction surface of 175 $mm^2$. The high retention load values of PEEK/PEKK crowns may be a result of the oversized contact surfaces and because a dimensional reduction of the crowns would decrease the retention load values, making them more clinically relevant. However, the enlarged contact area was used to ensure increased retention forces to obtain comparable values. In addition, the results obtained were comparable with those of previous investigations with a similar experimental design [8, 23, 25, 26].

All secondary crowns were produced by milling, and the results were consistent with those of an investigation that stated that the digital workflow might provide predictable retention forces and be a suitable alternative to the conventional workflow [5]. Retention force measurements were performed under wet conditions using artificial saliva, whereas distilled water was used for removal and insertion cycles. According to previous investigations, moist conditions are important for generating hydraulic forces between primary and secondary crown (like saliva in the clinical situation), and no differences were found between artificial saliva and distilled water [3].

Artificial saliva and thermomechanical loading in a mastication simulator, with removal and insertion cycles as well as temperature changes, were attempts to simulate oral conditions. However, limitations of this investigation included the in vitro study design, oversized specimens, and retention load measurements that were always performed on only one material pairing, which does not correspond to the clinical situation, as telescopic prostheses consist of at least two or more telescopic crowns. Further investigations should use different tapers of telescopic crowns, increase the number of telescopic systems acting simultaneously, and use specimens with tooth like dimensions to improve recommendations for clinical application.

## Conclusions

Within the limitations of this investigation, it was concluded that different material combinations have different retention forces, which should be considered during treatment planning. Furthermore, the simulation of approximately 15 years of clinical use resulted in a decrease in the retention forces for $ZrO_2$ and CoCr secondary crowns on both types of primary crowns, while an increase in retention load values was demonstrated for PEEK and PEKK secondary crowns. This increase of retention forces should be further investigated.

**Acknowledgements** The authors would like to thank bredent and Cendres+Métaux for supporting this investigation with materials.

**Author contributions** Danka Micovic Soldatovic: methodology, validation, investigation, data curation, formal analysis, writing — original draft.
Maximilan Bitter: methodology, writing — review and editing.
John Meinen: resources, technical support during specimen manufacturing and performing the experiment, writing — review and editing.
Karin C. Huth: supervision, writing — review and editing.
Anja Liebermann: supervision, writing — review and editing.
Bogna Stawarczyk: conceptualization, methodology, validation, formal analysis, supervision, writing — review and editing.

**Funding** Open Access funding enabled and organized by Projekt DEAL.

## Declarations

**Ethical approval** This article does not contain any studies with human participants or animals performed by any of the authors.

**Informed consent** For this type of study, formal consent was not required.

**Conflict of Interest** The authors declare no competing interests.



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## 5. Additional contributions:

### Publication 3

### Retention force of polyetheretherketone and cobalt-chrome-molybdenum removable dental prosthesis clasps after artificial aging

Felicitas Mayinger, Danka Micovic, Andreas Schleich, Malgorzata Roos, Marlis Eichberger, Bogna Stawarczyk

Clin Oral Investig, 2021. 25(5): p. 3141-3149.

DOI:10.1007/s00784-020-03642-5.

### Publication 4

### Is the high-performance thermoplastic polyetheretherketone indicated as a clasp material for removable dental prostheses?

Danka Micovic, Felicitas Mayinger, Sebastian Bauer, Malgorzata Roos, Marlis Eichberger, Bogna Stawarczyk

Clin Oral Investig, 2021. 25(5): p. 2859-2866.

DOI:10.1007/ s00784-020-03603-y.

Clinical Oral Investigations
https://doi.org/10.1007/s00784-020-03642-5

ORIGINAL ARTICLE

# Retention force of polyetheretherketone and cobalt-chrome-molybdenum removable dental prosthesis clasps after artificial aging

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Received: 29 April 2020 / Accepted: 9 October 2020


## Abstract

**Objectives** To examine the retention force of removable dental prosthesis (RDP) clasps made from polyetheretherketone (PEEK) and cobalt-chrome-molybdenum (CoCrMo, control group) after storage in water and artificial aging.

**Materials and methods** For each material, 15 Bonwill clasps with retentive buccal and reciprocal lingual arms situated between the second pre- and first molar were manufactured by milling (Dentokeep [PEEKmilled1], NT digital implant technology; breCAM BioHPP Blank [PEEKmilled2], bredent), pressing (BioHPP Granulat for 2 press [PEEKpressed], bredent), or casting (remanium GM 800+ [CoCrMo], Dentaurum); $N = 60$, $n = 15$/subgroup. A total of 50 retention force measurements were performed for each specimen per aging level (initial; after storage [30 days, 37 °C] and 10,000 thermal cycles; after storage [60 days, 37 °C] and 20,000 thermal cycles) in a pull-off test. Data were statistically analyzed using one-way ANOVA, post hoc Scheffé and mixed models ($p < 0.05$).

**Results** Initial, PEEKpressed (80.2 ± 35.2) and PEEKmilled1 (98.9 ± 40.3) presented the lowest results, while PEEKmilled2 (170.2 ± 51.8) showed the highest values. After artificial aging, the highest retention force was observed for the control group (131.4 ± 56.8). The influence of artificial aging was significantly higher for PEEK-based materials. While PEEKmilled2 and PEEKpressed showed an initial decline in retention force, all other groups presented no impact or an increase in retention force over a repetitive insertion and removal of the clasps.

**Conclusions** Within the tested PEEK materials, PEEKmilled2 presented superior results than PEEKpressed. Although CoCrMo showed higher values after artificial aging, all materials exhibited sufficient retention to recommend usage under clinical conditions.

**Clinical relevance** As RDPs are still employed for a wide range of indications, esthetic alternatives to conventional CoCrMo clasps are sought.

**Keywords** PEEK · Cobalt-chrome-molybdenum · Clasp · Removable dental prosthesis · Retention force

## Introduction

Removable dental prostheses (RDPs) are commonly used to treat patients with large or multiple edentulous areas. Indications furthermore include the replacement of missing teeth in patients with severely damaged periodontal tissue, an excessive loss of alveolar bone limiting the possibility for implantation or the use as interim restorations for patients awaiting extensive treatments like bone augmentation [1–3]. In addition, psychological and financial factors play an important role in choosing between RDPs and alternative treatment options like multi-unit fixed dental prostheses (FDPs) or implants.

Felicitas Mayinger and Danka Micovic contributed equally to this work.

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Published online: 16 October 2020

RDPs are usually manufactured of a PMMA base with acrylic or ceramic teeth in combination with cobalt-chrome-molybdenum (CoCrMo) clasps. The tried and tested CoCrMo clasps show excellent mechanical properties, such as a promising long-term stability and reliability [4–8] and high retentive capabilities, even when manufactured in small dimensions to improve patient comfort [4]. However, CoCrMo's silver color is nowadays becoming more and more unacceptable for patients with high esthetic requirements, especially when employed in the visible region. Moreover, the biocompatibility of metal clasps is viewed as controversial [9–11]. In the oral cavity, non-precious metals like CoCrMo can cause galvanic corrosions as metallic ions solved in saliva interact with amalgam or gold restorations [12]. In this context, patients have specified a metallic taste in connection with a new removable prosthesis manufactured of CoCrMo or shown allergic reactions of the oral mucosa [9–11],

These disadvantages called for the implementation of new dental compositions such as high-performance thermoplastic polymers as clasp materials in the treatment with RDPs. Polyetheretherketone (PEEK), a member of the polyaryletherketone (PAEK) family, possesses a high biocompatibility, excellent mechanical characteristics, a high chemical stability, and a high temperature resistance [13–16]. Due to its high flexibility, PEEK RDPs induce less stress on abutment teeth and may be less prone to deformation or fracture than standard alloy counterparts [17, 18]. PEEK furthermore possesses a low weight, an important factor for RDPs of the maxilla, and allows for an individual adaption of the clasp color to the patients' natural tooth color. As of today, PEEK materials are available in a multitude of shades, from classic pearl white to a wide variety of different enamel colors. To reduce extensive surgical procedures for FDP treatment of patients presenting with deficiencies of soft and hard tissues in the esthetic zone and enable RDPs to be manufactured solely from PEEK, a pale-pink shade option has been developed to imitate the color of the gum. A recent case report describing the long-term outcome of a treatment with a PEEK RDP has observed the patient to perceive this restoration as more acceptable and easier to assimilate to than alloy alternatives [19]. PEEK materials are nowadays employed for a wide range of restorations in prosthetic dentistry, from dental implants, abutments, FDPs, frameworks of RDPs to clasps, or telescopic prostheses [20–22]. In implant dentistry, flexible PEEK frameworks can reduce excessive masticatory forces occurring due to a lack of proprioception [23]. PEEK restorations can be produced employing the conventional lost-wax technique by pressing from pellets or granules, or via computer-aided design and computer-aided manufacturing (CAD/CAM) by milling from blanks. The use of CAD/CAM allows for a fully digital workflow that entails numerous advantages like an increased material homogeneity and the ability to reproduce restorations, for example, when elderly patients misplace their prostheses.

One property of utmost importance for a clasp is its retention force, which will keep the dental prosthesis in place during function such as eating or speaking. This point strongly affects the patients' contentment with their restoration. One way to measure retention force in an in vitro study set-up is the pull-off test, where specimens are removed from abrasion-resistant models under constant measurement conditions.

The aim of the present study was thus to examine the retention force of clasps made from different PEEK materials in comparison with a CoCrMo control group after storage in water and artificial aging with thermocycling. The study tested the null hypothesis that neither the clasp material, the manufacturing process of the PEEK specimens, artificial aging nor a repetitive insertion and removal of the clasps on an abrasion-resistant CoCrMo model showed an impact on the retention force.

## Materials and methods

The retention force of clasps made from three differently manufactured PEEK materials (Dentokeep [abbreviation: PEEKmilled1], NT digital implant technology, Karlsruhe, Germany; breCAM BioHPP Blank [PEEKmilled2] and BioHPP Granulat for 2 press [PEEKpressed], bredent, Senden, Germany) and a CoCrMo alloy (control group; remanium GM 800+ [CoCrMo], Dentaurum, Ispringen, Germany) was examined in a pull-off test at different aging levels (Table 1 and Fig. 1).

### Specimen fabrication

For each material, 15 specimens were manufactured ($N = 60$; $n = 15$/subgroup; Fig. 2).

The second pre- and first molar of a dental arch model (Frasaco Mandible 119, A-3, Franz Sachs & Co, Tettnang, Germany) were prepared to incorporate a Bonwill clasp. A master clasp was produced from CoCrMo (remanium GM 800+) by casting (Globucast, Krupp AG, Essen, Germany) with the lost-wax technique (Finowax, DT, Bad Kissingen, Germany). The casting channel, which was positioned in the insertion direction of the Bonwill clasp, was cut to a height of 15 mm to allow for a later positioning in the pull-off test. The specimen was air-particle abraded (basis Quattro IS, Renfert, Hilzingen, Germany) with 110 μm $Al_2O_3$ (Korox 110, Bego, Bremen, Germany) at 0.2 MPa and subsequently polished with a silicone polisher and a polishing brush (Komet, Gebr. Brasseler GmbH & Co. KG, Lemgo, Germany) before scanning (Ceramill map V2.5.02, Amann Girrbach, Koblach, Austria) was performed to create a master STL file (Table 2).

Clasps made of PMMA (Zeno® PMMA cast Disc, Wieland Dental + Technik, Pforzheim, Germany; $n = 30$) and PEEK (Dentokeep and breCAM BioHPP Blank; n = 15/

**Table 1** Materials, abbreviations, manufacturers, compositions, and lot. no. used

| Material | Abbreviations | Shade | Manufacturers | Compositions | Lot. no. |
|---|---|---|---|---|---|
| Dentokeep | PEEKmilled1 | Pearl white | NT digital implant technology, Karlsruhe, Germany | Polyether ether ketone, inorganic fillers (20%) | 11DK18001 |
| breCAM BioHPP Blank | PEEKmilled2 | | bredent, Senden, Germany | | 380149 |
| BioHPP Granulat for 2 press | PEEKpressed | | | | 379806 |
| Remanium GM 800+ | CoCrMo | | Dentaurum, Ispringen, Germany | Co (58.3%), Cr (32.0%), Mo (6.5%), W (1.5%), Si (1.0%) | 816 |

subgroup) were then manufactured with CAM software (Zenotec CAM, V2.2.009, Wieland Dental + Technik) using a milling machine (i-Mes 4030, Wieland Dental + Technik).

PEEKpressed specimens were produced by carefully embedding the PMMA clasps (Brevest for 2 press, bredent). The investment ring was then heated closely following the manufacturer's instructions (ARCA 20, Schütz Dental, Rosbach, Germany) and Granulat was pressed under vacuum (for 2 press, bredent; Fig. 3).

Following the same workflow, CoCrMo specimens (remanium GM 800+) were produced by embedding PMMA clasps (JET2000, Siladent, Dr. Böhme & Schöps GmbH, Goslar, Germany). The investment ring was then heated closely following the manufacturer's instructions (KaVo EWL 5636, KaVo Dental GmbH, Biberach/Riß, Germany) before clasps were cast at 1410 °C with a pressure of 0.45 MPa (Globucast).

After outbedding, PEEKpressed and CoCrMo specimens were air-particle abraded with 105 µm $Al_2O_3$ at 0.2 MPa (Hasenfratz, Fine-blaster type FG 3, Sandmaster, Zofingen, Switzerland).

Connectors and casting channels were cut to a height of 15 mm before specimens were polished with a goat hairbrush and buffing wheel using polishing paste (Universal-Polierpaste, Ivoclar Vivadent, Ellwangen, Germany). All specimens were then fitted on CoCrMo models using occlusion foil (Hanel Okklusions-Folie 12 µm, Coltène/Whaledent AG, Altstätten, Switzerland).

## Measurement of the retention force

Retention force was determined at different aging levels:

1. Initial,
2. After storage in distilled water for 30 days at 37 °C in an incubator (Hera Cell 150, Heraeus, Hanau, Germany) and artificial aging with 10,000 thermal cycles (Thermocycler THE-1100, SD Mechatronik, Feldkirchen-Westerham, Germany), with specimens remaining in each bath set to 5 °C and 55 °C for 20 s, simulating 1 year in clinical conditions [24], and
3. After storage in distilled water for 60 days at 37 °C and artificial aging with 20,000 thermal cycles (Thermocycler THE-1100) simulating a clinical period of 2 years.

For the pull-off test, models were carefully positioned in the insertion direction before casting channels/connectors were inserted in an individually manufactured stainless steel adapter (SD Mechatronik GmbH, Feldkirchen, Germany; Fig. 4). Pull-off force was applied with a crosshead speed of 5 mm per minute employing the universal testing machine (Zwick 1445, Zwick GmbH & Co. KG, Ulm, Germany) until the

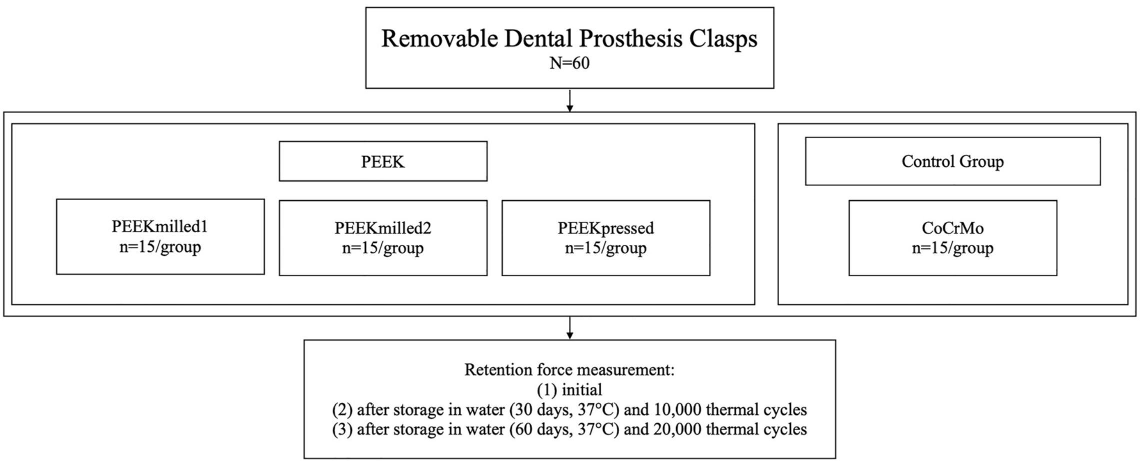



**Fig. 1** Study design

**Fig. 2** RDP clasp specimens made of CoCrMo, PEEKmilled1, PEEKmilled2 and PEEKpressed

maximum force dropped by 10%. For each specimen, 50 measurements were performed at the three different aging levels.

## Statistical analysis

Prior to performing this study, a power analysis had been computed using nQuery Advisor (Version 6.04.10, Statistical Solutions, Saugaus Mass, USA). For this calculation, retention force values of the control group (163 ± 55 N) were used. A sample size of 15 in each group would have a power of 97% to detect a difference of 81.5 N using a two-group $t$ test with a significance level of $\alpha = 0.05$. The Bonferroni correction would furthermore have a power of 92% under identical conditions.

Statistical evaluation of the data was performed with descriptive analysis followed by Kolmogorov-Smirnov for testing the violation of normal distribution. One-way ANOVA followed by the Scheffé post hoc test was performed to determine the influence of the material and aging level on the retention force. To determine global retention force values within the tested groups and potential changes of these values at different aging levels and measurement intervals, as each clasp was measured 50 times leading to dependent measurements, linear mixed models were computed.

All $p$ values below 0.05 were construed as statistically significant. Data were analyzed with SPSS version 25.0 (IBM, Armonk, NY, USA).

# Results

The results of the descriptive analyses are presented in Table 3. As no violation of normality assumption was indicated, parametric tests were performed.

The clasp material showed an influence on the retention force ($p < 0.001$). Initial, PEEKpressed and PEEKmilled1 showed the lowest values, while PEEKmilled2 presented the highest results. The control group led to results in the same value range as both PEEKmilled1 and PEEKmilled2. After artificial aging with storage in water (30 days, 37 °C) and 10,000 thermal cycles, PEEKpressed and PEEKmilled1 presented significantly lower retention force values than PEEKmilled2 and CoCrMo. After additional artificial aging (storage in water [60 days, 37 °C] and 20,000 thermal cycles),

**Table 2** Dimensions of the Bonwill clasp

| | Length (mm) | Height (mm) | Width (mm) | Undercut (mm) |
|---|---|---|---|---|
| Retentive arm, overall (external dimension) | 19.0 | | | |
| Reciprocal arm, overall (external dimension) | 16.2 | | | |
| Retentive arm, short (inner dimension) | 4.9 | 2.33 | 1.76 | 0.75 |
| Retentive arm, long (inner dimension) | 10.5 | 2.9 | 1.72 | 1.0 |
| Reciprocal arm, short (inner dimension) | 5.5 | 1.79 | 1.73 | |
| Reciprocal arm, long (inner dimension) | 8.7 | 2.91 | 1.89 | |
| Support | | 2.0 | 4.8 | |
| Connector | | | 4.5 × 4.92 | |

Retentive arm (buccal), reciprocal arm (lingual), short arm (premolar), and long arm (molar)

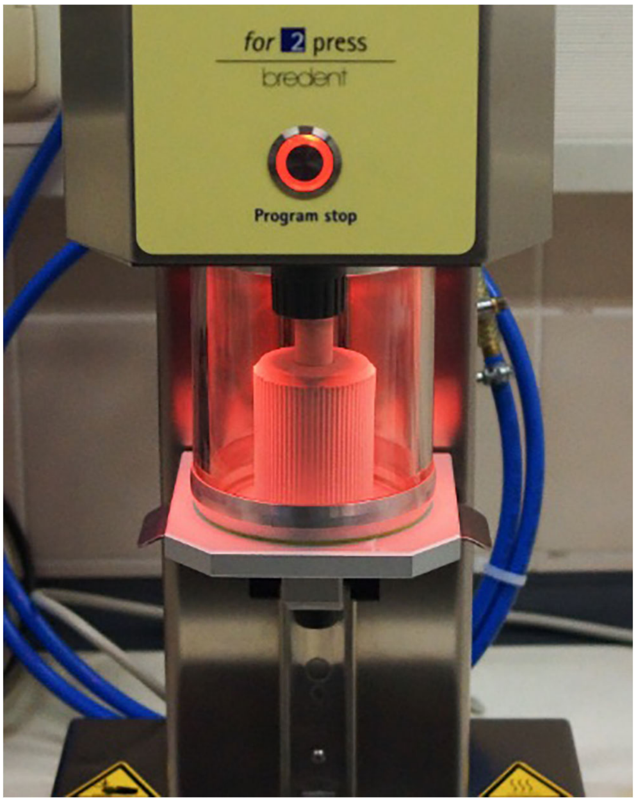



**Fig. 3** Pressing process for clasps made from PEEKpressed (for 2 press, bredent)

PEEKpressed and PEEKmilled1 showed lower retention force values than the control group, while PEEKmilled2 presented results in the same value range as PEEKmilled1.

Initially, values for PEEKmilled1 (9.5 N [0.0; 18.5]; $p$ = 0.04) and CoCrMo (11.2 N [8.9; 13.4]; $p$ < 0.001) increased over the repetitive insertion and removal of the clasps on the abrasion-resistant CoCrMo models, while PEEKmilled2 (− 2.9 N [− 4.3; − 1.5]; $p$ < 0.001) and PEEKpressed (− 3.1 N [− 4.3; − 2.0]; $p$ < 0.001) showed a decline in retention force. After the first artificial aging level, all groups but PEEKpressed that showed a rise in retention force (2.9 N [2.2; 3.6]; $p$ < 0.001) showed no impact of a repeated insertion and abrasion on the retention force. After artificial aging with 60-day storage in water at 37 °C and 20,000 thermal cycles, all groups presented an increase in retention force (PEEKmilled1: 6.1 N [5.4; 6.7]; PEEKmilled2: 13.6 N [13.0; 14.3]; PEEKpressed: 5.0 N [4.5; 5.6], CoCrMo: 18.8 N [17.3; 20.4]; $p$ < 0.001) over the repetitive insertion and removal of the clasps.

Mixed models defining the control group as baseline showed no significant difference between CoCrMo and PEEKmilled2 ($p$ = 0.051) initial, while PEEKmilled1 (− 44.2 N [− 73.8; − 14.6]; $p$ = 0.004) and PEEKpressed (− 62.7 N [− 92.2; − 33.1]; $p$ < 0.001) presented lower retention force values.

The influence of artificial aging was significantly higher for PEEK-based materials (PEEKmilled1: − 20.2 N [− 27.7; − 12.6]; PEEKmilled2: − 41.0 N [− 48.5; − 33.4]; PEEKpressed: − 15.4 N [− 22.9; − 7.8]; $p$ < 0.001) than for the control group (Fig. 5).

## Discussion

The aim of this study was to examine the retention force of clasps made from different PEEK materials in comparison with a CoCrMo control group after storage in water and artificial aging with thermocycling to approximate a clinical situation. The tested null hypothesis had to be rejected, as the choice of material, artificial aging, and the repetitive insertion

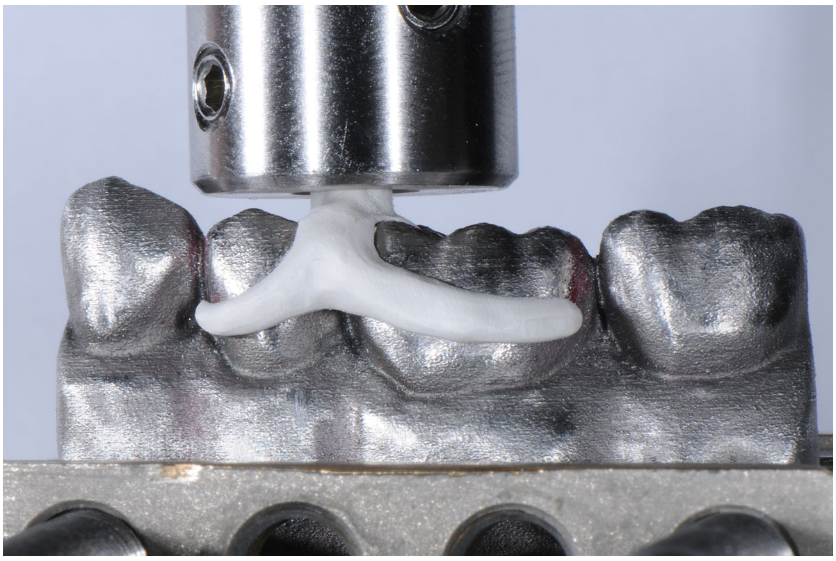


**Fig. 4** Retention force measurement (Zwick 1445, Zwick GmbH & Co. KG)

**Table 3** Descriptive statistics for the retention force [N] of the different clasp materials at varying aging levels

| Aging level | PEEKmilled1 | | PEEKmilled2 | | PEEKpressed | | CoCrMo | |
|---|---|---|---|---|---|---|---|---|
| | Mean ± SD | 95% CI | Mean ± SD | 95% CI | Mean ± SD | 95% CI | Mean ± SD | 95% CI |
| 1. Initial | 98.9 ± 40.3[a,b] | [76.6; 121.3] | 170.2 ± 51.8[c] | [141.5; 199.0] | 80.2 ± 35.2[a] | [60.6; 99.7] | 139.7 ± 57.4[b,c] | [107.8; 171.5] |
| 2. After storage in water (30 days, 37 °C) and 10,000 thermal cycles | 76.3 ± 27.9[a] | [60.8; 91.8] | 134.2 ± 44.0[b] | [109.7; 158.6] | 63.2 ± 26.4[a] | [48.5; 77.9] | 147.6 ± 54.8[b] | [117.2; 178.0] |
| 3. After storage in water (60 days, 37 °C) and 20,000 thermal cycles | 50.3 ± 21.2[a,b] | [38.5; 62.1] | 80.0 ± 31.4[b] | [62.6; 97.4] | 41.2 ± 14.0[a] | [33.3; 49.0] | 131.4 ± 56.8[c] | [99.9; 162.9] |

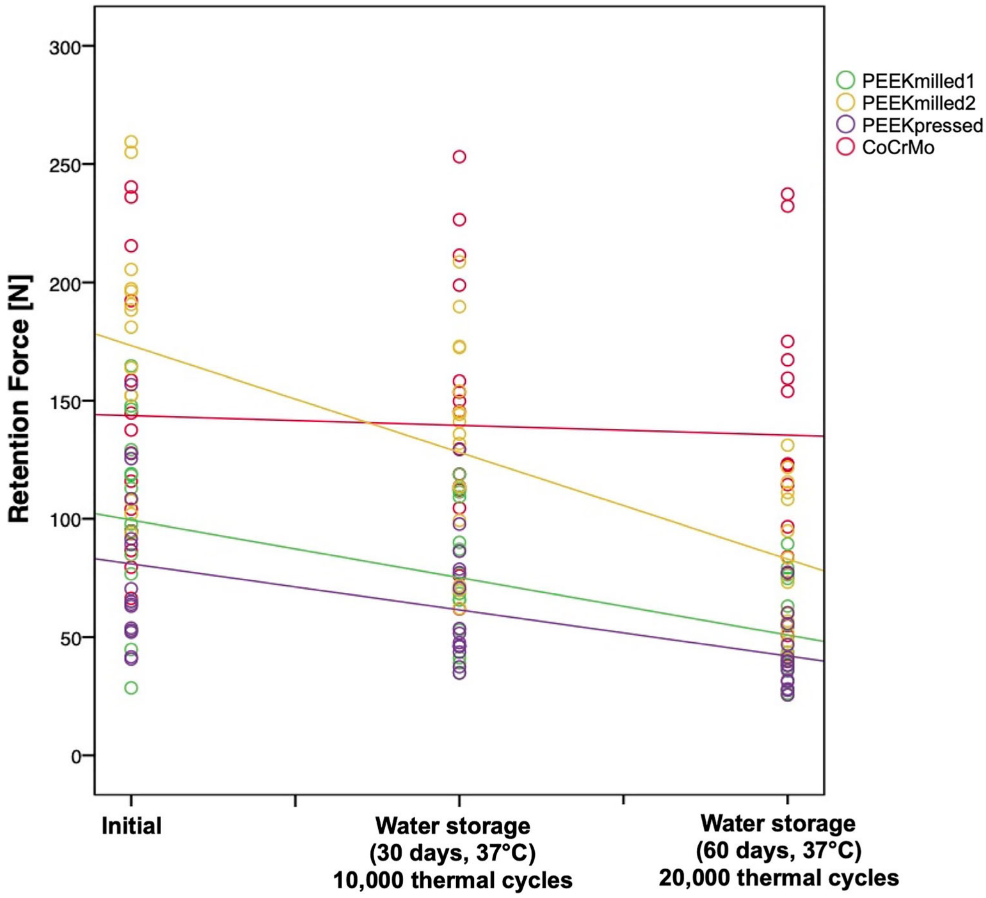
[a,b,c] Different letters present significant differences between the different materials within one aging level

and removal of the clasps on the abrasion-resistant CoCrMo model showed an impact on the retention force.

The present study observed PEEK clasps to present significantly lower retention force values than CoCrMo after artificial aging. In a recent study, the mean retention force of PEEK clasps (2.06–3.67 N) was also reported to be smaller than values observed for CoCr (8.25 N) [17]. As the aspired retention force per clasp has, however, been described as 5–10 N [25, 26], a clinical application of PEEK clasps may be cautiously recommended [27]. Yet, one crucial parameter in this context is stress phenomena occurring during the insertion and removal of RDP clasps. With the choice of material dictating the clasp design, flexible PEEK can require a deeper undercut to ensure sufficient retention force [17]. During removal, high stress levels may thus exceed the strength of the material itself [28]. Further studies are necessary to determine in how far PEEK can represent a clinically valid alternative to established alloy clasps and define an optimum clasp design for this material group.

When regarding the different PEEK materials, it can be reported that PEEKmilled2 presented higher values than PEEKpressed. This might be explained by the differing

**Fig. 5** Depiction of the influence of artificial aging on the retention force [N] of all tested materials



manufacturing process. While PEEKmilled2 specimens were fabricated from standardized blanks using CAD/CAM technology, clasps pressed from Granulat are more prone to outside influences and application errors, as this manufacturing process entails intricate steps such as the initial embedding, heating, and cooling of the muffle, pressing of the heated material under vacuum, or the subsequent air-abrasion. Following the different steps of this manufacturing process might thus result in an impaired homogeneity of the material [29]. Moreover, the fabrication process of PEEK blanks and PEEK Granulat differs, as PEEK blanks undergo an industrial prepressing procedure, which could increase the mechanical properties of the final product [29]. Contrary to expectations, PEEKmilled1 and PEEKmilled2 presented disparate results in the initial stage. A possible explanation for this puzzling observation may be provided by variations in the industrial manufacturing of the prepressed blanks. After artificial aging, results for the two groups did, however, align. Future investigations are needed to examine this point further.

Although PEEKmilled2 presented higher values than the control group initial, the observed values declined in the course of artificial aging. Artificial aging with 20,000 thermal cycles is supposed to correspond to a clinical situation after 2 years in vivo [24]. The present findings are in agreement with the results of a recently published study that reported specimens milled from PEEK blanks to show decreased mechanical properties after artificial aging [30]. Even though CoCr clasps are reported to show a permanent deformation after aging, they still present higher retention force values than resin clasps due to their high material stiffness and elastic modulus [5].

While PEEKmilled2 and PEEKpressed showed an initial decline in retention force, all other groups presented no impact or an increase in retention force over the repetitive insertion and removal of the clasps on the abrasion-resistant CoCrMo models at the different aging levels. A decline in retention force might be explained by an occurring material fatigue of the PEEKmilled2 and PEEKpressed clasps. Due to PEEK's low elastic modulus (4 GPa) in comparison with a CoCrMo alloy (240 GPa), it may not be rigid enough to withstand the occurring forces during a repetitive insertion and removal [31]. To counteract this, PEEK clasps could be manufactured to be bulkier and designed with a greater undercut to provide sufficient retentive force [31]. For CoCrMo, the effect of fatigue is seen controversial. While some studies observed a decrease in retention force due to a permanent deformation of the alloy [5], others showed no impact of aging on the retentive values [31]. This might be explained by the differing study set-up, where specimens were rigidly fixed and compromising torqueing forces were thus aimed to be excluded [31]. An increase in retention force, especially for PEEKmilled2 and PEEKpressed specimens that previously showed a decline of the retention force, is, however, unexpected. One possible explanation might be that the repetitive insertion and removal of the clasp specimens entails a better fit through either a minor abrasion of the model or an improved adaption of the clasps through the removal of any imperfections on the inside of the clasp arms. This idea has been described in a previous study, where an increased friction between the two components due to the wear phenomena of the materials was observed in the initial phase of a repetitive insertion and removal of the claps, while an increased wear and decreased retention was reported later on [31].

As of today, only few clinical case reports documenting the behavior of PEEK clasps in vivo are available. One study with a 2-year follow-up showed promising results regarding retention force, color stability, and plaque affinity [19]. The use of PEEK clasps can thus contribute to a healthier periodontium, an important factor for periodontally damaged dentitions, as the low plaque affinity prevents bacterial adhesion [21], while PEEK's high flexibility entails a low stress on the abutment teeth [17]. These advantages are mirrored in the high satisfaction of both patient and clinician in terms of functional and esthetic results [32]. PEEK clasps can furthermore preserve the existing dentition, with a clinical report describing an absence of scoring phenomena on silicate ceramic or enamel surfaces that are routinely seen for CoCrMo clasps [33]. The low weight of PEEK prostheses, combined with the tooth-similar color and appropriate fit and retention can make these restorations easy to assimilate to [32].

When regarding the findings of the present investigation, PEEK's promising results during the repetitive insertion and removal of the clasps, and its overall sufficiently high retention force, even after artificial aging, have to be noted. The mechanical properties of PEEK RDP clasps might thus allow the many advantages to be gained from its manufacturing process, from a fully digitalized workflow to a standardized manufacturing process entailing a high material homogeneity. As future material compositions might lead to improved mechanical properties, especially in regard to PEEK's poor performance in the course of artificial aging, this technique could behold a promising future. The present findings do, however, have to be seen in regard to their limitations, as this in vitro study only examined a limited number of tested materials. Moreover, the rigid model used in this study does not represent the clinical situation accurately, where the periodontal ligament permits a minor flexibility of the natural tooth. As the retention force correlates with the friction coefficient, the different friction coefficients of human enamel, dental restorative materials such as silicate ceramics, and the metallic model employed in the present study have to be considered [34]. This underlines the importance of an individual planning of the clasp geometry, as both the abutment and clasp material hold a decisive impact on the necessary undercut [17, 34]. The microscopical analysis of wear features could provide additional information on the observed differences between PEEK groups [35]. Thus, clinical studies with a long-term follow-up investigating a wider range of PEEK materials are warranted.

## Conclusions

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

1. Within the tested PEEK materials, PEEKmilled2 presented superior results than PEEKpressed.
2. Artificial aging led to a significant decline in retention force for all PEEK-based materials.
3. Overall, groups presented an increase in retention force due to a repetitive insertion and removal of the clasps.
4. Although CoCrMo showed higher values after artificial aging, all materials exhibited sufficient retention to recommend usage under clinical conditions.

**Acknowledgments** The authors would like to thank NT digital implant technology, bredent, and Dentaurum for supporting this study with material.

**Funding** Open Access funding enabled and organized by Projekt DEAL.

### Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

**Ethical approval** This article does not contain any studies with human participants or animals performed by any of the authors.

**Informed consent** For this type of study, formal consent was not required.



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Clinical Oral Investigations
https://doi.org/10.1007/s00784-020-03603-y

ORIGINAL ARTICLE

# Is the high-performance thermoplastic polyetheretherketone indicated as a clasp material for removable dental prostheses?

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Received: 23 April 2020 / Accepted: 23 September 2020


## Abstract

**Objectives** To investigate the retention force of polyetheretherketone (PEEK) removable dental prosthesis clasps in comparison with a cobalt-chrome-molybdenum control group after storage in artificial saliva.

**Materials and Methods** Clasps were milled (Dentokeep (PEEKmilled1), NT digital implant technology; breCAM BioHPP Blank (PEEKmilled2), bredent), pressed (BioHPP Granulat for 2 press (PEEKpressed), bredent), or cast (remanium GM 800+ (cobalt-chrome-molybdenum), Dentaurum); $N = 60$, $n = 15$/subgroup. Retention force was examined 50 times/specimen in a pull-off test using the universal testing machine (Zwick 1445), where pull-off force was applied with a crosshead speed of 5 mm/minute until the maximum force dropped by 10%, at different aging levels: (1) initial, after storage in artificial saliva for (2) 90 and (3) 180 days. Statistical analysis was performed using one-way ANOVA followed by post hoc Scheffé-test and mixed models ($p < 0.05$).

**Results** Cobalt-chrome-molybdenum presented the highest retention force. No differences were observed between polyetheretherketone materials. Cobalt-chrome-molybdenum showed a significant decrease of its values after artificial aging, while polyetheretherketone materials presented similar results over the course of aging. Regarding a repetitive insertion and removal, even though PEEKmilled2 and cobalt-chrome-molybdenum showed an initial increase, ultimately, a decrease in retention force was observed for all tested groups.

**Conclusions** Although the control group showed significantly higher results, the retention force of polyetheretherketone materials indicate a potential clinical application. Neither the manufacturing process nor artificial aging showed an impact on the retention force of polyetheretherketone clasps.

**Clinical relevance** Mechanical properties of novel removable dental prosthesis clasp materials devised to meet the growing esthetic demands of patients need to be investigated to ensure a successful long-term clinical application.

**Keywords** PEEK · Cobalt-chrome-molybdenum · Clasp · Removable dental prosthesis · Retention force

## Introduction

Due to recent leaps in implant and restorative dentistry, fixed dental prostheses (FDPs) allow highly esthetic results for a wide majority of patients. In some cases, an extensive replacement of missing teeth does, however, still require treatment with removable dental prostheses (RDPs) because of reduced health, challenging anatomical situations, physiology, or financial reasons [1].

Clasps can be used as retention elements to attach a prosthesis to the remaining teeth, thus ensuring functional stability during enunciation and mastication. In the course of time, a wide variety of clasps have been designed to tailor to various indications. Clasps traditionally consist of a retentive arm that

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Published online: 07 October 2020

passes over the prosthetic equator and comes to a rest in an undercut, while the reciprocal arm undertakes the task of opposing lateral forces during insertion and removal [2]. The depth of the undercut as well as the elastic modulus of the clasp material directly affects the retention of RDPs [3].

Metal alloy has for a long time been the material of choice for RDP clasps, as its outstanding mechanical properties are well documented [4–12]. The alloy most commonly used is cobalt-chrome-molybdenum (CoCrMo) [13]. Numerous studies have observed significantly higher retention load values of CoCrMo clasps than seen for alternative materials such as titanium [11, 12]. With ever rising esthetic demands, research activities have focused on tackling the main drawback of alloy clasps: their metallic color. To eliminate the esthetically disadvantageous retentive arm, lingual retentions or rotational paths were investigated as alternatives to conventional clasp designs [14, 15]. Others aimed to modify the alloy claps itself by etching and veneering said materials with tooth-colored resin composite [16].

One relatively new approach is to manufacture clasps of a tooth-colored thermoplastic material, such as polyoxymethylene [17], polycarbonate and polyamide [3], or polyaryletherketone (PAEK) [18]. The term "PAEK" comprises a number of closely related high-performance thermoplastics, from polyetheretherketone (PEEK) over polyetherketoneketone (PEKK) to aryl ketone polymer (AKP), that convince with notable mechanical properties and manifold applications in the field of dentistry [19, 20]. In prosthodontics, PEEK is employed as a framework material for fixed and removable dental prostheses and the manufacturing of clasps and implant abutments [21, 22]. PEEK may also hold a promising future in dental implantology. While unmodified PEEK is less osseoconductive and bioactive than titanium [23], dental implants made from PEEK have been shown to exhibit less stress shielding when compared with titanium [21]. Studies have furthermore observed a high biocompatibility and chemical stability of PEEK to both organic and inorganic chemicals [24, 25]. This finding is of special importance for patients prone to allergies. In a dental technical laboratory, PEEK can be processed by pressing the extruded material with a special vacuum-pressing device. For this purpose, PEEK is used either as pellets or in its granular form. Computer-aided design (CAD) and computer-aided manufacturing (CAM) technology enable an alternative manufacturing process by milling PEEK restorations from prepressed blanks. The industrial prepressing of blanks has been observed to increase the stability and reliability of PEEK restorations [26]. While all these fabrication methods allow using the same raw PEEK material, results of mechanical stress tests for these materials are very limited [19, 26] and the available literature varies considerably in terms of the investigated prosthetic applications.

Therefore, the aim of this study was to examine the retention force of RDPs' clasps made from three different PEEK materials in comparison with a CoCrMo control group after storage in artificial saliva. One important aspect when conducting an in vitro study is the close approximation to the clinical situation. To test the specimens' long-term performance, artificial aging was thus included in the study design [27]. The study tested the null hypothesis that neither the clasp material, the different manufacturing processes for the PEEK clasps, artificial aging, nor a repetitive insertion and removal of the clasps on an abrasion-resistant CoCrMo model showed an impact on the retention force.

## Materials and methods

The retention force of RDP clasps made from three differently manufactured PEEK materials (Dentokeep (abbreviation: PEEKmilled1), NT digital implant technology, Karlsruhe, Germany; breCAM BioHPP Blank (PEEKmilled2) and BioHPP Granulat for 2 press (PEEKpressed), bredent, Senden, Germany) and a CoCrMo alloy (control group; remanium GM 800+ (CoCrMo), Dentaurum, Ispringen, Germany) was examined in a pull-off test at different aging levels (Table 1, Fig. 1).

### Specimen fabrication

To produce 15 RDP clasp specimens from each material ($N = 60$; $n = 15$/subgroup; Fig. 2), a hollow form for a Bonwill clasp was prepared between the second pre- and first molar of a dental arch model (Frasaco Mandible 119, A-3, Franz Sachs & Co, Tettnang, Germany). By casting (Globucast, Krupp AG, Essen, Germany) with the lost-wax technique (Finowax, DT, Bad Kissingen, Germany), a master clasp was fabricated from CoCrMo (remanium GM 800 +). To allow for a later positioning in the pull-off test, the casting channel, which had been positioned in the insertion direction of the Bonwill clasp, was cut at a height of 15 mm. The model specimen was subsequently air-particle abraded (basis Quattro IS, Renfert, Hilzingen, Germany) with 110 µm $Al_2O_3$ (Korox 110, Bego, Bremen, Germany) at 0.2 MPa and polished with a silicone polisher and a polishing brush (Komet, Gebr. Brasseler GmbH & Co. KG, Lemgo, Germany). A master STL file (Table 2) was then created by scanning (Ceramill map 300, Amann Girrbach, Koblach, Austria) the model CoCrMo specimen.

Employing CAM software (Zenotec CAM, V2.2.017, Wieland Dental + Technik, Pforzheim, Germany) and a milling machine (i-Mes 4030, Wieland Dental + Technik), PMMA (Zeno PMMA cast Disc, Wieland Dental + Technik; $n = 30$) and PEEK (Dentokeep and breCAM; $n = 15$/subgroup) clasps were manufactured.

**Table 1** Materials, abbreviations, Young's modulus, manufacturers, compositions, and lot. no. used

| Material | Abbreviations | Young's modulus | Manufacturers | Compositions | Lot. no. |
|---|---|---|---|---|---|
| Dentokeep | PEEKmilled1 | 4 GPa | NT digital implant technology, Karlsruhe, Germany | Polyether ether ketone, inorganic fillers (20%) | 11DK18001 |
| breCAM BioHPP Blank | PEEKmilled2 | 4 GPa | bredent, Senden, Germany | | 380149 |
| BioHPP Granulat for 2 press | PEEKpressed | 4 GPa | | | 379806 |
| remanium GM 800+ | CoCrMo | 230 GPa | Dentaurum, Ispringen, Germany | Co (58.3%), Cr (32.0%), Mo (6.5%), W (1.5%), Si (1.0%) | 816 |
| Zeno PMMA cast Disc | | 2.4 GPa | Wieland Dental Pforzheim, Germany | Polymethylmethacrylate | 1304 |

Afterwards, PMMA clasps were embedded (Brevest for 2 press, bredent) in a muffle according to the manufacturer's instruction (Fig. 3). The investment ring was heated at 8 °C/s to 630 °C (ARCA 20, Schütz Dental, Rosbach, Germany) and then cooled to 400 °C. Subsequently, the pre-heated muffle was filled with Granulat and kept in the preheating oven for 20 min. As the next step, Granulat was pressed at 0.45 MPa under vacuum (for 2 press, bredent).

The remaining PMMA clasps were embedded (JET2000, Siladent, Dr. Böhme & Schöps GmbH, Goslar, Germany) in a similar workflow, before the investment ring was heated at 6 °C/s to 900 °C (KaVo EWL 5636, KaVo Dental GmbH, Biberach/Riß, Germany). CoCrMo specimens (remanium GM 800 +) were then cast at 1410 °C with a pressure of 0.45 MPa (Globucast).

After cooling, the investment material was removed from PEEKpressed and CoCrMo specimens using a blasting unit (Fine-blaster type FG 3, Sandmaster, Zofingen, Switzerland) with 105 µm $Al_2O_3$ (Hasenfratz) at a pressure of 0.2 MPa.

Clasps were subsequently polished with a silicone polisher and a polishing brush (Komet). High gloss was achieved with a goat hairbrush and buffing wheel using polishing paste (Universal-Polierpaste, Ivoclar Vivadent, Ellwangen, Germany). The fit of the clasp specimens on CoCrMo models was adjusted and verified with occlusion foil (Hanel Okklusions-Folie 12 µm, Coltène/Whaledent AG, Altstätten, Switzerland).

## Retention force measurement

Retention force was determined at three different aging levels:

(1) Initial
(2) After storage in artificial saliva for 90 days at 37 °C in an incubator (Hera Cell 150, Heraeus, Hanau, Germany)
(3) After storage in artificial saliva for 180 days at 37 °C in an incubator (Hera Cell 150)

Artificial saliva was prepared according to Fusayama Meyer et al. [28] (components: potassium chloride [0.4 g/l], sodium chloride [0.400 g/l], calcium chloride dihydrate [0.906 g/l], monosodium phosphate dihydrate [0.690 g/l], sodium sulfide nonahydrate [0.005 g/l], urea [1.000 g/l]; pH = 4.7) and replaced every 14 days.

Casting channels/connectors were inserted in an individually manufactured stainless steel adapter (SD Mechatronik GmbH, Feldkirchen, Germany; Fig. 4) after CoCrMo models were positioned in the insertion/removal direction of the Bonwill clasp. Using the universal testing machine (Zwick 1445, Zwick GmbH & Co. KG, Ulm, Germany), pull-off force was applied in direct extension of the casting channel/connector with a crosshead speed of 5 mm/min until the maximum force dropped by 10%. At the three different aging levels, 50 retention force measurements were performed for each clasp at each aging level.

**Fig. 1** Study design

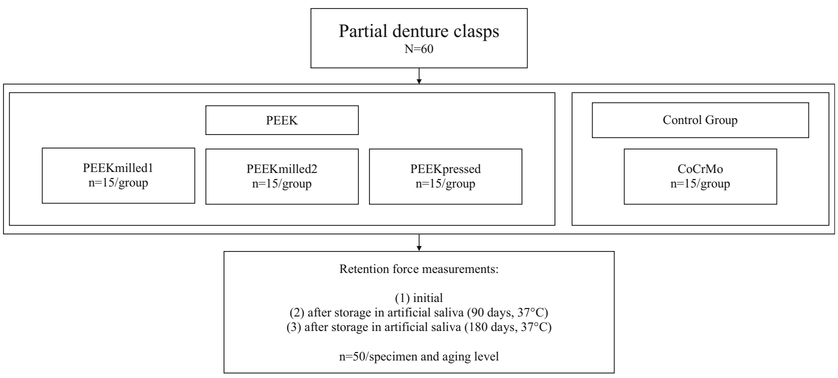

**Fig. 2** RDP clasp specimens made of CoCrMo, PEEKmilled1, PEEKmilled2, and PEEKpressed

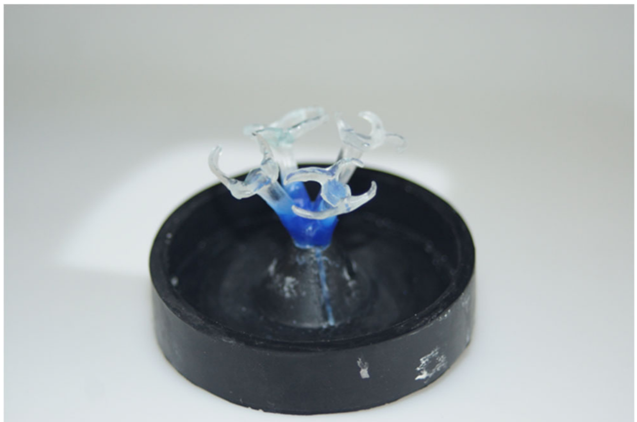

**Fig. 3** PMMA clasps prior to embedding during the manufacturing process of PEEKpressed and CoCrMo specimens

### Statistical analysis

A power analysis using the retention force values of the control group (163 ± 55 N) had been computed using nQuery Advisior (Version 6.04.10, Statistical Solutions, Saugaus Mass, USA) prior to performing this study. Employing a two-group $t$ test with a significance level of $\alpha = 0.05$ showed that a sample size of 15 in each group would have a power of 97% to detect a difference of 81.5 N. Under identical conditions, a Bonferroni correction would have a power of 92%.

A statistical evaluation of the data was performed using descriptive analysis followed by Kolmogorov-Smirnov for testing the violation of normal distribution. To determine the influence of the material and the aging level on the retention force, one-way ANOVA followed by Scheffé post hoc test was computed. Because each clasp was measured 50 times, leading to dependent measurements, linear mixed models were applied to determine global retention force values within the tested groups and potential changes of these values at different aging levels.

All $p$ values below 0.05 were construed as statistically significant. Data were analyzed with SPSS version 25.0 (IBM, Armonk, NY, USA).

## Results

The results of the descriptive analyses are presented in Table 3. Parametric tests were performed, as no violation of normality assumption was indicated.

The choice of clasp material presented a significant impact on the retention force, with the control group showing higher values than the three PEEK materials ($p < 0.001$). No differences in retention force were observed between different PEEK materials ($p = 0.412$–$0.607$).

Artificial aging showed an influence on the retention force of the different materials, with the control group presenting a significant decrease of its values ($p < 0.01$, Fig. 5). There is no evidence that PEEK materials show any decrease over the course of aging ($p = 0.236$–$0.401$).

The repetitive insertion and removal of the clasps led to a reduction of the retention force of PEEKmilled1 ($p < 0.001$–$0.048$; Table 4) and PEEKpressed ($p < 0.001$) specimens at all aging levels. For PEEKmilled2 and CoCrMo, an increase of retention force was observed initially ($p < 0.001$), before values decreased with a repetitive insertion and removal of the RDP clasps at the subsequent aging levels ($p < 0.001$–$0.199$).

**Table 2** Dimensions of the Bonwill clasp

| | Length (mm) | Height (mm) | Width (mm) | Undercut (mm) |
|---|---|---|---|---|
| Retentive arm, overall (external dimension) | 19.0 | | | |
| Reciprocal arm, overall (external dimension) | 16.2 | | | |
| Retentive arm, short (inner dimension) | 4.9 | 2.33 | 1.76 | 0.75 |
| Retentive arm, long (inner dimension) | 10.5 | 2.9 | 1.72 | 1.0 |
| Reciprocal arm, short (inner dimension) | 5.5 | 1.79 | 1.73 | |
| Reciprocal arm, long (inner dimension) | 8.7 | 2.91 | 1.89 | |
| Support | | 2.0 | 4.8 | |
| Connector | | | 4.5 × 4.92 | |

Retentive arm (buccal), reciprocal arm (lingual), short arm (premolar), long arm (molar)

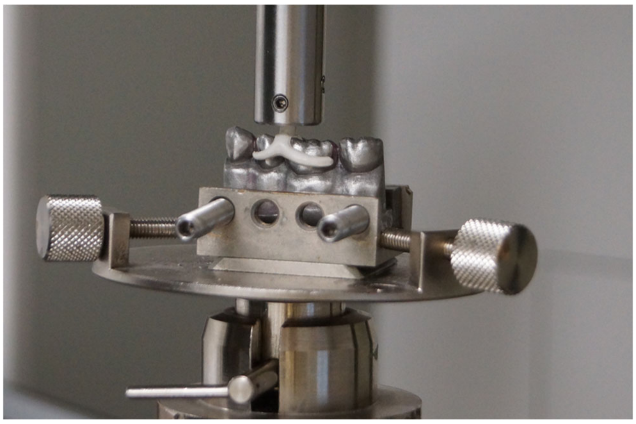

**Fig. 4** Retention force measurement (Zwick 1445, Zwick GmbH & Co. KG)

## Discussion

The aim of this study was to examine the retention force of RDP clasps made from different PEEK materials in comparison with a CoCrMo control group after storage in artificial saliva to imitate clinical conditions. The null hypothesis had to be rejected, as the results showed all tested parameters to affect the retention force.

When regarding the choice of clasp material, the control group showed superior retention values compared to the three PEEK materials. These results are in line with previous examinations investigating the retentive force and fatigue resistance of both PEEK and CoCr clasps [18, 29, 30]. Even though PEEK clasps presented lower values, they might provide enough retention for a clinical usage, as they exceed the suggested retention force of 5–10 N per clasp [31, 32]. As excessive retentive forces can overstrain the remaining abutment teeth, especially in periodontally compromised dentitions [33], PEEK materials could represent a valid alternative. As all PEEK materials showed similar results over the course of aging, the manufacturing process does not seem to hold an influence on the resulting mechanical properties. In the present study, two PEEK materials were milled using CAD/CAM technology, while one material was pressed. As most dental laboratories nowadays have access to high-end milling machines, this elegant process regarded to be less time-consuming and prone to manual mistakes should be preferred [34].

Artificial aging also presented an impact on the retention force. The control group showed a high decrease of its values, while PEEK clasps presented similar results before and after the aging process. A high decrease in the retention force of the control group can be explained by alloy corrosion taking place in wet environments, which has previously been reported to lead to a reduced fatigue strength of CoCr [35]. While the three PEEK materials also presented a decline in retention force, this was not significant. These results are consistent with a previous study investigating the behavior of PEEK during artificial aging with different saliva solutions that reported the thermoplastic to show a great structural stability and little or no impact of varying pH values on its nanomechanical properties [36].

The repetitive insertion and removal of the clasps led to a reduction of the retention force of PEEKmilled1 and PEEKpressed specimens at all aging levels. For PEEKmilled2 and CoCrMo, an increase of retention force was observed initially, before values decreased with a repetitive insertion and removal of the RDP clasps at the subsequent aging levels. An initial increase in retention force might be explained by abrasion phenomena of both the model and clasps resulting in an improved fit of the clasps and in consequence, an increased retention force. A previous examination investigating the retentive force of thermoplastic resins and cobalt-chrome over a simulation period of 10 years reported similar findings with an initial increase in values during the first period of cycling that was later on substituted by a continuous decrease [18]. The elastic modulus plays an important role in fatigue testing, as a material with a high elastic modulus is able to assume its prior structure without permanent deformation. CoCrMo, which possesses a high elastic modulus of 220 GPa [37], should thus in theory be less prone to a decrease in retention force due to a repetitive insertion and removal of the clasps than PEEK, which only holds an elastic modulus of around 4 GPa [38]. In contrast to this idea, a recent study observed polymer-based clasps to act more consistently over

**Table 3** Descriptive statistics for the retention force [N] of the different clasp materials at varying aging levels

| Aging level | PEEKmilled1 | | PEEKmilled2 | | PEEKpressed | | CoCrMo | |
|---|---|---|---|---|---|---|---|---|
| | Mean ± SD | 95% CI | Mean ± SD | 95% CI | Mean ± SD | 95% CI | Mean ± SD | 95% CI |
| (1) Initial | 58.1 ± 18.8[a] | [47.6; 68.5] | 43.9 ± 22.6[a] | [31.1; 56.4] | 50.8 ± 17.9[a] | [40.8; 60.8] | 163 ± 55.2[b] | [132; 193] |
| (2) After storage in artificial saliva (90 days, 37 °C) | 43.0 ± 14.4[a] | [35.0; 51.0] | 40.3 ± 20.4[a] | [29.0; 51.6] | 45.6 ± 14.9[a] | [37.3; 53.9] | 127 ± 40.4[b] | [104; 149] |
| (3) After storage in artificial saliva (180 days, 37 °C) | 36.4 ± 9.50[a] | [31.1; 41.7] | 33.5 ± 13.3[a] | [26.0; 40.9] | 35.7 ± 13.2[a] | [28.3; 43.0] | 102 ± 29.3[b] | [86.2; 119] |

[abc] Different letters present significant differences between the different materials within one aging level

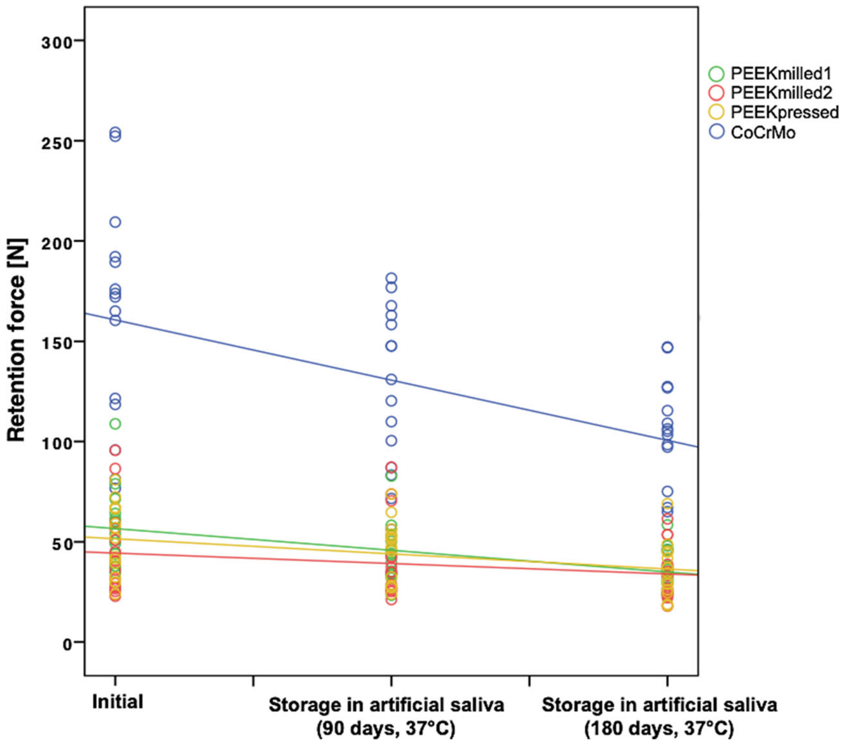


**Fig. 5** Graphical illustration of the retention force [N] of the different clasp materials at the three aging levels

a prolonged aging process, which included cycles of repeated insertion and removal along both ideal and non-ideal paths in artificial saliva, while exhibiting inferior retention forces in comparison to conventional CoCr clasps [20].

Regarding clinical implications, PEEK materials might therefore represent the material of choice for anterior abutment teeth that possess little anatomical undercut and in consequence require little deformation during insertion and removal, while CoCrMo could be the material of choice for the posterior regions, where molars provide a large retentive area and high masticatory forces demand superior retentive capacities and functional stability [2]. Individual patient situations might thus call for individualized treatment planning regarding the choice of clasp material.

As of today, only few reports about PEEK's behavior in clinical conditions are available. According to one recently published case report with a 2-year follow-up period, PEEK shows promising results, as few color and texture changes of PEEK were found macroscopically. The clasp arm still fitted well without any deformation and a high subjective satisfaction was expressed by both the practitioner and the patient [39]. Further advantages include the low weight of PEEK prostheses, the tooth-similar color, a reportedly good fit and high retention [40, 41], and a protective effect on the periodontal ligament [42]. However, the indication of PEEK as a framework material remains controversial, as its stability in a free-end situation under masticatory forces is not conducive for a RDP's stability [42].

While this study observed promising results for PEEK materials in regard to their potential use as RDP clasps and their high resistance against artificial aging in saliva, this in vitro study does entail several limitations. Only a small number of

**Table 4** Influence of a repetitive insertion and removal on the retention force [N] of the different clasp materials at varying aging levels

| Aging level | PEEKmilled1 | | PEEKmilled2 | | PEEKpressed | | CoCrMo | |
|---|---|---|---|---|---|---|---|---|
| | Mean ± SD | 95% CI | Mean ± SD | 95% CI | Mean ± SD | 95% CI | Mean ± SD | 95% CI |
| (1) Initial | − 1.7[c] | [− 3.4; − 0.02] | 7.5[b] | [6.4; 8.6] | − 4.8[c] | [− 6.0; − 3.5] | 14.8[c] | [11.3; 18.3] |
| (2) After storage in artificial saliva (90 days, 37 °C) | − 9.1[b] | [− 9.9; − 8.2] | − 0.5[b] | [− 1.2; − 0.2] | − 1.3[b] | [− 1.8; − 0.7] | − 11.8[b] | [− 14.4; − 9.1] |
| (3) After storage in artificial saliva (180 days, 37 °C) | − 0.7[a] | [− 1.2; − 0.2] | − 6.4[a] | [− 7.3; − 5.5] | − 4.3[a] | [− 5.0; − 3.6] | − 6.8[a] | [− 8.7; − 4.8] |

[abc] Different letters present significant differences between aging levels within one material

materials were tested in the present study. As the dental market moves quickly and new compositions are introduced each year, future studies will have to examine a wider range of materials. The use of artificial saliva furthermore only imitates one part of the manifold influences RDPs are exposed to during function, such as temperature changes or variations of the pH value. In the present study, only one clasp design, namely the popular Bonwill clasp, was examined. To allow the implementation of PEEK as a clasp material to a bigger extent, it is necessary to convey further examinations including a wide variety of clasp designs and geometries. As the environment has been reported to show varying effects on dislodging a clasp according to the type of clasp [43], and deformations differ due to the design of a clasp, future studies should focus on determining in how far PEEK materials could present a valid alternative to CoCrMo in specific situations, such as the esthetic anterior region presenting with little undercut, or periodontally damaged dentitions prone to the negative effects of excessively high retention forces [33]. The use of PEEK clasps could pave the way for a fully digital workflow in the treatment of patients with RDPs, from the digital impression to manufacturing using CAD/CAM technology [34]. Highly time- and resources-consuming laboratory processes in the fabrication of CoCrMo clasps that due to their manual background are furthermore prone to mistakes could hereby be replaced by machine processing of PEEK materials ensuring a high homogeneity of the material and promising great esthetic results. Moreover, due to the high surface resistance of PEEK material, its low reactivity, and a highly inert behavior in the oral cavity, these materials could have a good prognosis for allergy prone patients [19, 24]. Further clinical as well as laboratory studies are necessary to confirm the present findings.

## Conclusions

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

1 Although the control group showed significantly higher results, the retention force values observed for PEEK materials indicate a potential clinical application.
2 The manufacturing process of PEEK did not influence the retention force.
3 While the control group was susceptible to artificial aging, PEEK materials presented constant results.
4 Ultimately, a repetitive insertion and removal of the clasps resulted in decreased retention force values.

**Acknowledgments** The authors would like to thank NT digital implant technology, bredent, and Dentaurum for supporting this study with material.

**Funding** Open Access funding enabled and organized by Projekt DEAL.

## Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

**Ethical approval** This article does not contain any studies with human participants or animals performed by any of the authors.

**Informed consent** For this type of study, formal consent was not required.



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# Acknowledgements

I would like to express my sincere gratitude to all who supported me during my Ph.D. studies.

First of all, I am extremely grateful to **Prof. Dr. Bogna Stawarczyk** for her continuous support, motivation and encouragement. Her comments and suggestions have guided me throughout every stage of my research project, while her support and belief in me motivated me to finish.

Special thanks to **Prof. Dr. Anja Liebermann**, my second supervisor for her invaluable advices, patience, kindness and for always having time for me.

My thanks and appreciation to my third supervisor, **Prof. Dr. Karin C. Huth** for her great supervision, inspiring comments and invaluable support.

I would like to thank **Prof. Dr. Daniel Edelhoff** for providing me with the opportunity to conduct my Ph.D. project in the Department of Prosthetic Dentistry and for leading the great team in which I felt very welcome during my studies.

Special thanks to **Prof. Dr. Jan Kühnisch** for sharing his knowledge in field of literature research and presentation skills as well as for insightful discussions in our Ph.D. seminars.

Furthermore, I would like to offer special thanks to **Marlis Eichberger** and **John Meinen** for their technical support during the laboratory phases of my projects, for finding the solution to every obstacle and making my projects feasible.

My special thanks to the entire **Material research group** and e-learning colleagues for the kind help and support, coffee breaks and everything we did together besides science. It is you who have made my studies a wonderful time.

I would like to extend my sincere thanks to **BAYHOST** (Bayerisches Hochschulzentrum für Mittel-, Ost- und Südost Europa, Regensburg, Germany) and **Fund for Young Talents of the Republic of Serbia** (Government of the Republic of Serbia, Ministry of Youth and Sports, Belgrade, Serbia) for providing me with the scholarship and facilitating me to conduct my research.

Special appreciation to **my closest friends** for always being by my side and believing in me. Finally, my most sincere gratitude to **my beloved husband, my sister and parents** for all the love, endless support and understanding. You are the ones who encouraged me to accomplish what I have started and never give up on my dreams.