

## RESEARCH AND EDUCATION

# Effect of adhesive system, resin cement, heat-pressing technique, and thermomechanical aging on the adhesion between titanium base and a high-performance polymer

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## ABSTRACT

**Statement of problem.** Even though polyetheretherketone (PEEK) has become popular for various prosthetic indications, a standard adhesive protocol to bond the PEEK to titanium bases has not been yet established. How the heat-pressing technique performs in this respect is also not clear.

**Purpose.** The purpose of this in vitro study was to investigate the effect of an adhesive system-cement combination, the heat-pressing technique, and thermomechanical aging on the retention force between titanium bases and PEEK specimens.

**Material and Methods.** Sixty 9×11×20-mm PEEK specimens with a titanium base slot integrated into the design were milled to simulate an implant-supported PEEK framework for a cantilevered fixed prosthesis. The specimens were assigned to 8 groups (n=10) according to the titanium base primer (MKZ or Monobond) and resin cement (DTK or Multilink hybrid) used and with or without thermomechanical aging. Twenty PEEK specimens were directly heat-pressed on titanium bases, and half of the specimens were not subjected to thermomechanical aging (n=10). For nonaged groups, the PEEK specimen complex was tightened to an implant analog and secured on a custom-made pull-off device. Retention forces were measured by using the pull-off tensile test in a universal testing machine, and the maximum tensile bond strength (MPa) was calculated. The aged groups were subjected to 5000 cycles of thermal aging (5 °C to 55 °C), and the specimens were clamped to load the extension (cantilever) for 1 200 000 cycles with 120 N and 200 N at 1.5-Hz frequency. After aging, the pull-off test was performed for those specimens that survived thermomechanical aging. A nonparametric Kruskal-Wallis test was used to determine whether there was a difference among the groups, followed by pairwise Wilcoxon rank tests with Bonferroni correction. The Wilcoxon rank test was used to analyze the effect of thermomechanical aging in each adhesive system-cement or heat-press group ( $\alpha=.05$  for all tests).

**Results.** None of the specimens failed during cyclic loading. According to the Kruskal-Wallis test, the effect of the PEEK-Ti base bonding technique on the retention force in the nonaged ( $P=.019$ ) and thermomechanically aged groups was significant ( $P=.010$ ). In the nonaged groups, the heat-pressing technique resulted in a higher retention force than when the specimens were bonded by using the Monobond-Multilink hybrid combination ( $P=.031$ ). Thermomechanical aging did not significantly affect the results ( $P>.241$ ). All failures were adhesive, with cement remaining only on the Ti-bases.

**Conclusions.** All bonding protocols tested resulted in a stable bond between PEEK and Ti-bases, as all specimens survived thermomechanical aging. The heat-pressing technique resulted in mean bond strength values similar to those obtained with the tested adhesive system-cement combinations with 1 exception; the nonaged heat-pressed groups presented higher bond strength than the Monobond-Multilink hybrid combination. Failure types indicated that the weaker bond was between the PEEK and the cements tested rather than between the titanium base and the cements, regardless of the adhesive system-cement combination. (J Prosthet Dent 2022;■:■-■)

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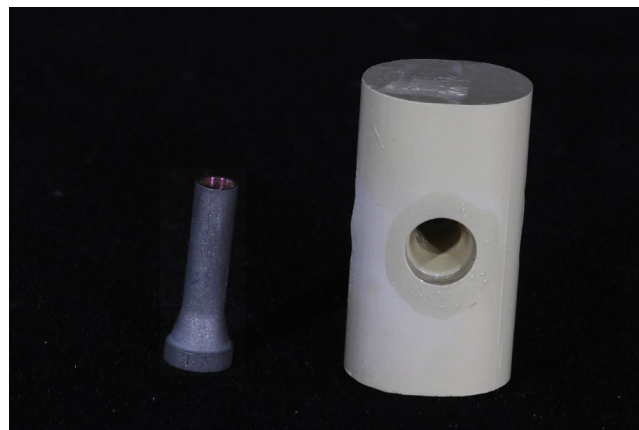
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## Clinical Implications

Heat-pressing PEEK directly on titanium bases resulted in a tensile bond strength similar to that obtained with titanium base cemented to PEEK after thermomechanical aging. Therefore, heat-pressing PEEK on titanium bases is a promising technique for clinical application. When the tested adhesive system-cement pairs are used, the bond between the PEEK and the cement can be considered the weak link.

The polyaryletherketones, also named polyketones (PAEKs), are a relatively new family of linear aromatic molecular chains interlinked by ketone and ether functional groups.<sup>1</sup> Popular polymers in this family are polyetheretherketone (PEEK), polyether ketone (PEK), and polyetherketoneketone (PEKK).<sup>2</sup> PEEK has been explored for its use as dental implants, healing and interim implant abutments, implant-supported bars or clasps,<sup>3</sup> and frameworks for removable dental prostheses.<sup>4-6</sup> PEEK has also been reinforced by adding approximately 20 wt% of inorganic filler,<sup>4</sup> which allowed PEEK to serve as an alternative material for long-span fixed partial dentures (FPDs).<sup>3,7</sup> Improved distribution of impact stresses, reduction in denture weight, and lower treatment costs are some of the benefits that have been reported.<sup>7</sup> Three manufacturing methods are currently available for PEEK: heat-pressing technology, milling, or printing by using computer-aided design and computer-aided manufacturing (CAD-CAM).<sup>8,9</sup>

Because of PEEK's inherent properties, durable bonding to PEEK is still problematic. In a prospective cohort clinical study, Malo et al<sup>3</sup> reported on a total of 37 patients rehabilitated with 49 complete-arch prostheses with PEEK substructures veneered with pink acrylic resin and acrylic resin denture teeth. The authors reported mechanical complications, bonding to the PEEK substructure being the most problematic. The authors also reported the need to incorporate a titanium base (Ti-base) to avoid damage to the PEEK infrastructure during titanium prosthetic screw tightening in the pilot phase of the study. The 3-year follow-up of patients from the same clinic recently reported on problems with bonding to PEEK frameworks.<sup>10</sup> To improve bonding to PEEK polymers, several surface modifications have been made and reported to diversify the functional groups of the PEEK polymer.<sup>11</sup> The implementation of mechanical and chemical surface treatments in conjunction with the application of adhesive primers has been proposed to bond to PEEK.<sup>9</sup> The selection of an adhesive system is as important as the micromechanical surface topography in establishing a strong bond between PEEK polymers and



**Figure 1.** Titanium base (left) and polyetheretherketone specimens (right).

composite resins.<sup>12-14</sup> Successful bonding to PEEK has been credited, in part, to the content and solvents found in the adhesive systems. Adhesive systems containing methylmethacrylate (MMA) were reported to provide durable micromechanical interlocking and a potential chemical bond between PEEK and resin.<sup>5,15,16</sup> visio.link, An MMA-containing primer, has been used to bond composite resins to PEEK.<sup>11</sup> However, the ideal surface treatment to use on the Ti-base surface is still unclear.<sup>17</sup> Manufacturers recommend the airborne-particle abrasion of titanium surfaces with alumina ( $\text{Al}_2\text{O}_3$ ) or tribochemical silica ( $\text{SiO}_2$ ) particles of different sizes, at different pressures, and using different methods, followed by different adhesive systems.<sup>11,18-26</sup> PEEK can be heat-pressed to Ti-bases without a cement or adhesive system. The performance of the bond between PEEK and Ti-base when the heat-pressing technique is used has not been investigated thoroughly.

The adhesion between the PEEK and Ti-bases is crucial for the success of PEEK implant-supported prostheses. Since the strength of this bond is still controversial, the aim of the present study was to assess the effect of the cement-adhesive system and the heat-pressing technique on the retention force between Ti-bases and PEEK frameworks. The null hypotheses were that the adhesive system-cement combination would not affect the retention force between the Ti-base and PEEK framework and that the retention force between the Ti-base and PEEK framework would not be affected by thermomechanical aging.

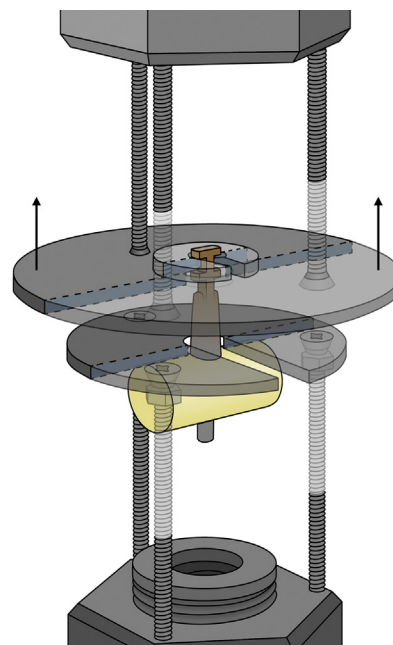
## MATERIAL AND METHODS

PEEK specimens (N=80) (9×11×20 mm) (BioHPP; bre-dent GmbH & Co KG) and multiunit Ti-bases (Sky uni.cone Prosthetic cap CAD/CAM; bre-dent GmbH & Co KG) (N=80) were used in the present study. The Ti-base design was included in the PEEK specimens to simulate

**Table 1.** Overview of tests groups

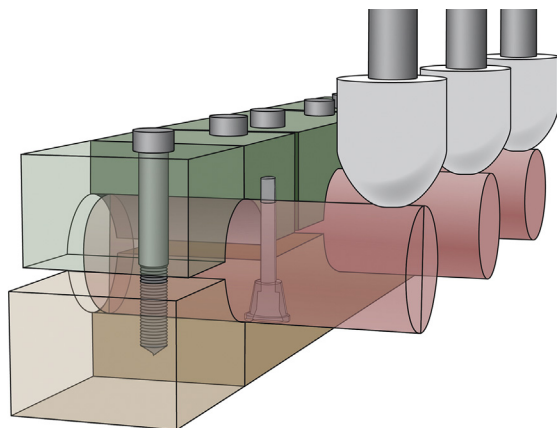
Group (n=10 per Group)	Ti-Base Primer	Cement	PEEK Primer	Thermomechanical Aging
MKZ-ML	MKZ primer	Multilink hybrid abutment	visio.link	Yes
MB-ML	Monobond Plus	Multilink hybrid abutment	visio.link	Yes
MKZ-DTK	MKZ primer	DTK adhesive	visio.link	Yes
P (Heat-pressed)	None	None	None	Yes
MKZ-ML	MKZ primer	Multilink hybrid abutment	visio.link	No
MB-ML	Monobond Plus	Multilink hybrid abutment	visio.link	No
MKZ-DTK	MKZ primer	DTK adhesive	visio.link	No
P (Heat-pressed)	None	None	None	No

an implant-supported PEEK framework for a cantilevered fixed dental prosthesis by using a computer-aided design (CAD) software program (Geomagic freeform; 3D Systems). The cement gap was defined as 20  $\mu\text{m}$ . Sixty PEEK specimens were milled by using a computer-aided manufacturing (CAM) software program (Modellier; Zirkonzahn) and a milling machine (M1; Zirkonzahn) (Fig. 1). The surface of the PEEK specimens to be bonded to the Ti-base was airborne-particle abraded with 110- $\mu\text{m}$  alumina at a 10-mm distance at 0.2-MPa pressure. A resin primer (visio.link; bredent GmbH & Co KG) was applied in a uniform single coat and polymerized according to the manufacturer's recommendations. The Ti-bases were airborne-particle abraded with 110- $\mu\text{m}$  alumina at a 10-mm distance and 0.2-MPa pressure, thoroughly cleaned with a brush with alcohol, and dried by using oil-free compressed air for 1 minute. The titanium surface was then conditioned with either a primer agent (Monobond Plus; Ivoclar Vivadent AG) or MKZ primer (bredent GmbH & Co KG) for 60 seconds. The bonding surface between the Ti-bases and the specimens was 80  $\text{mm}^2$ . The Ti-bases and PEEK were assigned to 6 different groups (n=10) according to the primer and the resin cement used and whether the bonded specimens were submitted to thermomechanical aging or not (Table 1). For the specimens assigned to the DTK-adhesive group, the cement was mixed with the mixing cannula and applied directly to the PEEK and Ti-base surfaces that were being bonded. This procedure was conducted by the same operator (D.G.) with firm finger pressure for 2 minutes and continuous light pressure for 6 minutes at room temperature. Meanwhile, a photopolymerization unit (Essentials Curing Light; Essentials Healthcare Products) was used to accelerate and ensure complete polymerization of the cement. The excess cement was cleaned with a microbrush, and the specimen was placed in its assigned group. For the application of Multilink hybrid abutment cement (Ivoclar Vivadent AG), the cement was dispensed with a mixing tip directly onto the PEEK and Ti-base surface to be bonded, and the parts were tightly pressed together with firm finger pressure for 5 seconds. Glycerin gel (Liquid Strip; Ivoclar Vivadent AG) was applied at the

**Figure 2.** Schematic drawing of pull-off test. Maximum retention force of titanium bases in polyetheretherketone specimens recorded while device moved upward.

cementation joint to prevent the formation of an oxygen inhibition layer, and the parts were held with continuous light pressure for 7 minutes. Once the polymerization was completed, the glycerin was cleaned with a microbrush, and excess cement was carefully removed from the cementation joint.

Twenty specimens were fabricated by using the heat-pressing technique. The specimens were first milled (M1; Zirkonzahn) from wax blanks (breCAM.wax; bredent GmbH & Co KG) based on the standard tessellation language (STL) data set used for the milled PEEK specimens and then connected to the Ti-bases (Sky uni.cone prosthetic cap; bredent GmbH & Co KG) by melting modeling wax between the milled wax and the Ti-base. The specimens were embedded in a flask to burn out the wax at 650  $^{\circ}\text{C}$ . PEEK granules (BioHPP; bredent GmbH & Co KG) were melted and then pressed into the flask at 400  $^{\circ}\text{C}$  by using a press machine (For2Press;

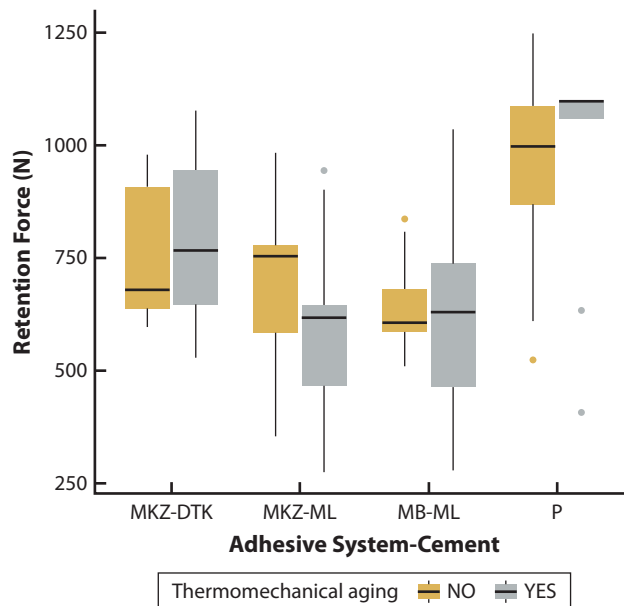


**Figure 3.** Schematic drawing of cyclic loading. Load applied 12 mm from clamp, simulating loading prosthesis cantilever.

breident GmbH & Co KG) at a pressure of 0.4 MPa. After cooling to room temperature, the test specimens were deflasked. For the groups not subjected to thermomechanical aging, the specimens were stored dry for 24 hours after cementation. Each Ti-base-PEEK framework complex was tightened to an implant analog (SKY uni-cone laboratory analogue; breident GmbH & Co KG) and secured on a custom-made pull-off device (Fig. 2).

Retention forces were measured by using the pull-off test in a universal testing machine (Instron Model 5566; Illinois Tool Works) with a 10-kN calibrated load cell at a crosshead speed of 1.0 mm/min. The maximum retention force ( $F_{\max}$ ) was recorded in newtons (N) and saved for the statistical analysis. Then, the maximum bond strength (MPa) was calculated from  $F_{\max}$  (N)/bonding area ( $\text{mm}^2$ ). The thermomechanical aging of the specimens started with thermocycling (5 °C to 55 °C, 5000 cycles) followed by mechanical cyclic loading (1 200 000 cycles at 120 N and 1 200 000 at 200 N and 1.5 Hz). The load was applied on the extension, 12 mm away from the clamped part of the specimens to simulate a cantilevered prosthesis (Fig. 3). The specimen and test design were based on previous studies.<sup>27-30</sup> After thermomechanical aging, the specimens were evaluated under  $\times 10$  magnification by the same operator (D.G.) to check for any micromovement or fracture.

If any micromovement between the Ti-base and the PEEK framework was noted, the specimen was assigned to bond failure. The specimens that survived the thermomechanical aging were then subjected to the pull-off test to record the retention forces and to calculate the tensile bond strength according to the protocol used for nonaged specimens. After the pull-off tests, the PEEK surface and the Ti-bases were inspected for remaining cement and failure mode classification. Failure modes were classified as adhesive (cement left either only on Ti-base or PEEK), cohesive (cement left homogeneously on



**Figure 4.** Boxplot of retention force versus adhesive system-cement. DTK, DTK adhesive; MKZ, MKZ primer; ML, Multilink hybrid abutment; P, heat-press.

both Ti-base and PEEK), or mixed adhesive-cohesive (cement left on both Ti-base and PEEK, leaving parts exposed on both structures).

A nonparametric Kruskal-Wallis test was used to test whether there was a difference in retention force in the nonaged and thermomechanically aged groups. In case of significant differences, pairwise Wilcoxon rank test was used with Bonferroni correction to find significant pairwise differences. To analyze the effect of thermomechanical aging in each adhesive system-cement or heat-pressing group, the Wilcoxon rank test was performed ( $\alpha=.05$ ).

## RESULTS

None of the specimens failed during cyclic loading. A significant difference was found in the retention force within the nonaged groups ( $P=.019$ ) based on the Kruskal-Wallis test. The pairwise Wilcoxon rank test with Bonferroni adjustment indicated a significant difference between the MB-ML and P-group (adjusted  $P=.031$ ). A significant difference was also found among the thermomechanically aged groups based on the Kruskal-Wallis test ( $P=.010$ ). According to the post hoc Wilcoxon rank test, the retention force for P-group was similar to that of MKZ-ML (adjusted  $P=.060$ ) (Fig. 4). No statistically significant effect of thermomechanical aging was detected in the adhesive system-cement or pressed-on groups ( $P>.241$ ). The maximum retention forces and bond strength for all groups are presented in Table 2. All failures were adhesive in mode, and cement was retained only on the Ti-base surfaces.



**Table 2.** Maximum retention force and bond strength

Study Outcomes	MKZ-DTK (Nonaged)	MKZ-ML (Nonaged)	MB-ML (Nonaged)	Pressed (Nonaged)	MKZ-DTK (Aged)	MKZ-ML (Aged)	MB-ML (Aged)	Pressed (Aged)
Max. retention force (N), median [IQR]	680.50 [638.75, 907.00]	755.00 [585.75, 776.00]	608.00 [588.50, 680.50]*	998.50 [870.75, 1086.75]*	767.00 [648.00, 944.00]	618.00 [467.75, 644.00]	630.00 [465.50, 736.00]	1094.00 [1061.00, 1098.00]
Max. bond strength (MPa), median [IQR]	8.6 [8.0, 11.4]	9.5 [7.4, 9.8]	7.6 [7.4, 8.6]	12.6 [11.0, 13.7]	9.7 [8.2, 11.9]	7.8 [5.9, 8.1]	7.9 [5.9, 9.3]	13.8 [13.4, 13.8]

\*Groups significantly different.

## DISCUSSION

The null hypothesis that the use of varying bond system-cement combinations or the heat-pressing technique would not affect the retention force between the Ti-base and the PEEK frameworks was rejected, as a significant effect of the adhesive system-cement combination and heat-pressing technique was observed before thermo-mechanical aging. The second null hypothesis was not rejected because the effect of thermomechanical aging on the retention force between the Ti-base and PEEK framework was not significant.

The authors are unaware of a previous study that investigated different cementation protocols and the resulting retention force between PEEK specimens and Ti-bases. However, the adhesion of composite resin cements to PEEK has been investigated.<sup>13,22,23</sup> Schmidlin et al<sup>13</sup> reported bond strength values ranging from 8.7 to 19 MPa by using different surface pretreatments, including acid etching, airborne-particle abrasion, and silica coating combined with 2 types of resin cements. Other surface treatments for PEEK that resulted in comparable or higher bond strength values included the use of cold atmospheric plasma and an erbium-doped yttrium aluminum garnet laser.<sup>14,21</sup> These methods were not available for the present study, and because the bond strength reported in those studies were in a range similar to that of alumina airborne-particle abrasion, the PEEK specimens and Ti-bases were pretreated by using alumina airborne-particle abrasion for standardization. Airborne-particle abrasion has been recommended for both bonding on PEEK and bonding on Ti-bases to obtain surfaces with improved adhesive properties.<sup>24-26</sup> As airborne-particle abrasion is used for many other bonding procedures, this should also warrant its broad applicability in clinical practice. The results of the present study showed that the heat-pressing of PEEK on Ti-base results in a retention force similar to that obtained with cementation. However, the heat-pressing technique should be compared with the other PEEK pretreatment protocols.

Previous studies that investigated different bonding protocols for PEEK on different substrates have mainly focused on different types of adhesive systems. The use of visio.link for PEEK surface conditioning, as applied in the present study, has been reported to be a reliable

method.<sup>24</sup> Furthermore, the primers and resin cements used in the present study to bond the PEEK specimens to Ti-bases have been reported to provide durable bond strength values, even after artificial aging.<sup>14</sup> With regard to the adhesive systems for Ti-bases, one of the most widely used adhesive systems (Monobond Plus) and the adhesive system recommended by the PEEK manufacturer (MKZ primer) were used.<sup>11,21</sup> However, the authors are unaware of studies using either DTK adhesive or Multilink hybrid abutment to bond PEEK to Ti-bases, suggesting the lack of knowledge on bonding of PEEK on Ti-bases. The widespread use of different bonding protocols on Ti-bases may also explain why, in the present study, the failure modes all showed uniform cement adhesion to Ti-bases but not to the PEEK test specimens, suggesting that the weak link is the inferior adhesion between the cement and the PEEK. This link needs to be improved in order to recommend PEEK bonded to Ti-bases for clinical use.

Currently, PEEK is not as frequently used as zirconia or lithium disilicate bonded to Ti-bases for implant-supported prostheses. Sung et al<sup>31</sup> evaluated different bonding procedures of lithium disilicate to Ti-bases, reporting shear bond strength results ranging between 7.16 and 8.92 MPa. Similar to the present study, they used thermocycling for artificial aging and reported a significant effect of thermocycling when the Multilink hybrid abutment was used; this was also analyzed in the present study. Oddbratt et al<sup>22</sup> reported bond strengths of 1.3 to 9.3 MPa for zirconia specimens bonded on Ti-bases by using different protocols after thermocycling. Considering similar, or in some situations higher, bond strength values in the present study compared with a previous study<sup>22</sup> with zirconia and without artificial aging, all tested bonding protocols for PEEK may be considered suitable for clinical use. However, factors such as the physical, optical, and veneering properties of PEEK need to be further clinically evaluated to better understand the performance of PEEK fixed prostheses with Ti-bases. The authors are aware of only 1 study<sup>32</sup> that investigated the physical properties of PEEK with respect to processing methods. The findings suggested that the heat-pressing technique was inferior to milling from industrially manufactured PEEK blocks in terms of maximum fracture load for 3-unit fixed dental prostheses. However, the authors concluded that pressed PEEK was

also suitable as a framework material, as the maximum fracture loads were >1700 N, which exceeds loads that can be expected even in posterior sites.<sup>32</sup>

The specimen design and the arrangement for cyclic loading in the present study were similar to those of a previous study focusing on load-to-failure tests for cantilevered reconstructions on Ti-bases.<sup>27</sup> Dynamic loading on the cantilever results in higher stresses on the bond between the abutment and the reconstruction than direct (compressive) loading of an implant crown, tested according to ISO 14801:2016.<sup>33</sup> For the thermal aging process, a commonly used arrangement for various materials was applied. However, for cyclic loading, the number of cycles and the force exceeded 1 200 000 cycles with a 49-N or 98-N force, as are commonly applied.<sup>28-30</sup> Even though relatively higher loads were applied in the present study, the fact that no failures occurred during cyclic loading may indicate the clinical suitability of all techniques tested.

Limitations of this study included that no sample size was calculated. Performing a sample size analysis based on previously available data in the literature was not possible, as the authors are unaware of a previous study that compared the interaction of PEEK primers, Ti-base primers, and adhesive resin cements with the direct heat-pressing technique. Studies using similar testing conditions and specimen shapes included smaller sample sizes.<sup>27,34</sup> Nevertheless, statistically significant differences were demonstrated with the sample size used in the present study, particularly for intergroup comparisons. The PEEK specimens and Ti-bases were from one manufacturer, and different results may be obtained with materials from other manufacturers. As the tested Ti-base did not contain mechanical grooves, the retention force may be higher with Ti-bases containing additional mechanical retention elements.

## CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The retention force between PEEK and the titanium base was not affected significantly by thermo-mechanical aging for all combinations tested.
2. Before thermomechanical aging, the heat-pressing technique led to higher retention force than the Monobond adhesive-Multilink cement group.
3. The tested adhesive system-cement combinations and heat-pressing technique resulted in a similar retention force between PEEK and titanium base after thermomechanical aging.
4. Adhesive failures of the resin cement from the PEEK surfaces indicate the weak link in adhesion to this material.

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