# Comparing shear bond strength of a composite to PEEK, PEKK and titanium

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A research report submitted to the Faculty of Oral Health Sciences in partial fulfillment of the requirements for Master of Dentistry degree in Prosthodontics

# DEDICATION

This research project is dedicated to my loving husband Sivesonke Khanyile, my son Tshephang, my parents, siblings, and family in law, whose unwavering love and support have been the driving force behind my academic pursuits.

I would also like to dedicate this work to my supervisors, Dr. V.J. Hoods-Moonsammy, Dr. D.V Bwerinofa and Prof. J.L Shackleton, whose expertise and guidance have been instrumental in shaping my research skills and knowledge. Their mentorship has been an invaluable asset to my academic and professional growth.

Above all, I would like to express my gratitude to God for giving me the wisdom, fortitude, and health to make this achievement possible. It is my hope that the study will contribute to the advancement of knowledge and have a positive change in the field of prosthodontics.

# DECLARATION

I, Maditi Louisa Maphutha, declare that this research paper is solely my original work. This work has been submitted for the Master of Dentistry degree in Prosthodontics at the University of the Witwatersrand, Parktown, Johannesburg, South Africa. The work has not been previously submitted for any other academic degree or examination at this university or any other institution.

Signature: ____________ Date: 11 April 2025

# ACKNOWLEDGEMENTS

This research report has been conducted as a component of the Master of Dentistry degree in Prosthodontics. My sincerest gratitude is extended to Mr. Leith Cumming from Southern Implants (PTY) LTD, Irene, South Africa, for generously providing the milled titanium and PEEK specimens, as well as for his expertise in milling the PEKK specimens used in this research. Gratitude is extended to Sandile from Sandz Laboratory and Mr Nxumalo from Wits Laboratory for their invaluable support. Lastly, I would like to acknowledge Wits Dental Laboratory for generously allocating space in their laboratory to facilitate the preparation of my specimens.

# ABSTRACT

## Purpose

This *in vitro* study aims to compare the shear bond strength (SBS) of veneering composite to PEEK, PEKK, and titanium, as well as their failure modes (adhesive, cohesive, mixed).

## Materials and Method

Specimens of milled titanium, PEEK, and PEKK (n = 105) were abraded using 600-1000 grit silicon carbide paper. The specimens underwent surface treatment using sandblasting with 110μm aluminium oxide particles. The titanium specimens were bonded with a methacryloyloxydecyl dihydrogen phosphate-based (MDP) adhesive (Plafique Universal Bond), whilst the PEEK and PEKK specimens were bonded using a methyl methacrylate-based (MMA) adhesive (PEKKbond, AnaxDent, Ardmore, USA). A composite (Gradia® Plus Heavy body paste, GC, Europe) was bonded to the prepared surface of the specimens. An Instron universal testing machine (Instron, UK) was used to assess the SBS of titanium, PEEK, and PEKK when bonded to composite. The sheared interface of all specimens was evaluated with a stereomicroscope (Nikon SMZ 1500) at 16X magnification

## Results

A comprehensive narrative analysis of material performance compared titanium, PEEK, and PEKK, focusing on their SBS to composite, including their failure modes. The data revealed substantial differences among the mean SBS of titanium, PEEK, and PEKK when bonded to composite. Titanium shows a high mean SBS of 28.60 MPa, with a 95% confidence interval (CI) (SD = 9.84 MPa). PEKK shows a moderate mean compressive stress of 25.80 MPa, with a 95% confidence interval (SD = 5.44 MPa). This mean value indicates that PEKK has a higher SBS to composite, closer to that of titanium.

PEEK has a lower mean SBS of 16.83 MPa, with a 95% confidence interval (SD = 5.71). This lower mean value indicates that PEEK has a lower SBS to composite compared to titanium and PEKK. A three-sample t-test revealed a difference of 3.77 MPa between titanium and PEEK ($p < 0.0001$), 2.87 MPa between PEKK and PEEK ($p < 0.0001$), and 0.90 MPa between titanium and PEKK ($p = 0.1449$).

Titanium predominantly showed mixed failure mode, with 88.6% of failures involving mixed failures and 11.4% of adhesive failures. PEEK was more prone to adhesive failures, with 82.9% of failures occurring at the adhesive bonds. PEKK was also prone to adhesive failures, with 57.1% being adhesive and 42.9% being mixed failures.

## Conclusions and Recommendations

The current study's results allow for the drawing of the following conclusions within its limitations:

When bonded to composite, PEKK exhibited comparable SBS to titanium. In terms of their bond to composite, both PEKK and titanium were superior to PEEK. Therefore, PEKK may serve as a viable substitute for titanium, offering both cost-effectiveness and improved aesthetic results. While titanium and PEKK exhibited comparable SBS, their failure mechanisms varied. Titanium primarily displayed mixed-type failure, with only three specimens experiencing adhesive failure, while PEKK displayed predominantly adhesive-type failure. The PEEK specimens exhibited primarily adhesive failure. None of the specimens demonstrated cohesive failure. However, additional studies considering the oral environment and ageing effects are recommended to assess the long-term success.

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# List of abbreviations

**SBS** Shear bond strength

**PAEKs** Polyaryletherketones

**PEEK** Polyetheretherketone

**PEKK** Polyetherketoneketone

**MDP** Methacryloyloxydecyl dihydrogen phosphate

**MMA** Methyl methacrylate

**MPa.** Megapascals

**GPa.** Gigapascals

**NiCr** Nickel-chromium alloy

**CpTi** Pure titanium grade 1

**$Al_2O_3$** Aluminium oxide

**BisGMA** Bisphenol A-glycidyl methacrylate

**TEGDMA** Triethylene glycol dimethacrylate

**UDMA** Urethane dimethacrylate

# CHAPTER 1: INTRODUCTION AND LITERATURE REVIEW

## 1.1 Introduction

Rigid metal alloys such as titanium and titanium alloys have been the preferred materials for superstructures or frameworks of dental prostheses. Their low specific weight, strength, biocompatibility, and corrosion resistance make them ideal for replacing dental hard tissues that sustain heavy loads (Lee et al., 2009; Al-Rabab'ah et al., 2019; Koizumi et al., 2019). However, their main disadvantage involves aesthetic complications because of the need to mask their greyish colour with either composite or porcelain and the metal allergy that limits their use in some patients (Jin et al., 2019).

The recent trends towards aesthetically pleasing and biologic restorations that are cost and time-efficient resulted in the introduction of tooth-coloured framework materials such as zirconia and Polyaryletherketone materials (PAEKs) as alternatives to metal alloys (Rosentritt et al., 2015). Compared to zirconia, titanium and its alloys, PAEKs provide improved aesthetics without the increased cost and complex fabrication techniques. Additionally, PAEKs have a low modulus of elasticity (4 GPa), which is close to that of bone and oral hard tissues, in contrast to that of titanium (110 GPa). Thus, PAEK polymers act as stress breakers, reducing the forces transferred to the restorations and the underlying alveolar bone (Caglar et al., 2019; Barto et al., 2021). Furthermore, PAEK polymers are chemically inert; hence, they are suitable substitutes for patients allergic to titanium and other metals (Lee et al., 2017). Their repairability (Barto et al., 2021), high polishing qualities, low plaque affinity, and good wear resistance are additional advantages of PAEKs.

However, despite these advantages, their low translucency and greyish colour limit their use as monolithic restorations, especially in the aesthetic areas (Rosentritt et al., 2015). Therefore, like titanium, they require additional veneering with an aesthetic material such as porcelain, polymethyl methacrylate (PMMA) or composite. Although porcelain veneering provide superior aesthetics, good abrasion resistance and lower relative surface free energy compared to composites, making them less susceptible to staining, they are brittle, difficult to adjust or repair chair-side, and are costly compared to PMMA and composite. Composites are used as alternatives to porcelain because they are more cost-effective, absorb some occlusal stresses preventing them from being transferred to the framework substructure, and their lightweight decreases the weight of the prostheses. The material is also comparatively more aesthetic than PMMA (Jin et al., 2019).

One of the common mechanical complications for bi-layered prostheses include fracture of the veneering material. Therefore, the achievement and maintenance of adequate bond strength between veneering material and framework/substructure materials are one of the requirements for successful clinical outcomes and long-term stability of dental prostheses (Rosentritt et al., 2015; Lee et al., 2017; Gouveia et al., 2022).

Shear bond strength is the compressive force necessary to break a bonded interface between two dental materials or between a substance and a tooth. The limitations of the SBS test include the reported non-uniformity of stress distributions at the tested surfaces and the reported stress concentration within the substrate, which may cause premature failure. Furthermore, although shear loading is used during testing, the stresses caused by the bending moment are thought to be responsible for fracture initiation (Placido et al., 2007). However, the SBS was chosen for its simplicity and ease of specimen processing. Bher et al. (2003) recommended the Shear test for the assessment of dental materials because shear forces are dominant during function, and therefore, the test best represents the clinical scenario.

## 1.2 Literature review

### 1.2.1 Bonding to Titanium

Various surface treatments have been recommended for titanium alloys to increase their bond strength. These include the application of acidic compounds, primers, adhesives, and sandblasting. Almilhatti et al. (2013) evaluated the SBS of commercially pure titanium grade 1 (cpTi) to composite veneers. The study's results demonstrated increased SBS of composite to titanium when a combination of sandblasting and metal conditioners containing functional monomers derived from phosphoric acid, specifically methacryloyloxydecyl dihydrogen phosphate (MDP), were used. These results were supported by Kodaira et al. (2022), who highlighted the advantages of MDP-containing adhesives in promoting the bonding characteristics between titanium and composite.

Yanagida et al. (2002) assessed the adhesive efficacy of cast titanium to composite using eight different metal conditioners. They found that a metal conditioner comprising hydrophobic phosphate functional monomer MDP demonstrated a SBS of 22.2 Megapascals (MPa). A SBS of 10 MPa or greater is considered a clinically acceptable bond strength (Behr et al., 2011).

### 1.2.2 Bonding to PAEKs

PAEKs are high-performance thermoplastic polyaryletherketones that can be classified as Polyetheretherketone (PEEK) and polyetherketoneketone (PEKK) (Lee et al., 2017; Taha et al., 2022). They differ based on the proportion of their keto- and ether groups, glass transition temperature, and melting points (Taha et al., 2022). An increased ratio of keto-groups correlates with enhanced rigidity and elevated melting temperature of the material (Fokas et al., 2019). Consequently, PEKK has 80% higher compressive strength than PEEK. PEEK and PEKK display high tensile, fatigue, and flexural strengths, dimensional stability at high temperatures, and high wear resistance (Lee et al., 2017; Fokas et al., 2019).

Adhesion to PAEKs is challenging due to their stable chemical structure and unreactive and inert surface character. According to Adem et al. (2022), an important factor for durable adhesion is the adequate spread of the adhesive material on the surface of the substrate. Therefore, for increased adhesion, the substrate's surface-free energy should be higher than that of the adhesive. However, PAEKs have low surface-free energy, resulting in poor bonding properties. Two methods have been described to improve the bonding performance between composite and PAEKs. These include the mechanical alteration of PAEK surfaces and the conditioning using adhesives to allow chemical interactions (Caglar et al., 2019).

Mechanical modification treatments include airborne particle abrasion, etching the surface with sulphuric acid or piranha solution (a mixture of sulphuric acid and hydrogen peroxide), plasma pre-treatments, and laser etching. Chemical adhesive materials include methyl methacrylate (MMA)-containing adhesives and MDP-containing adhesives (Stawarczyk et al., 2014; Taha et al., 2022).

## 2.2 a) PEEK

Caglar et al. (2019) examined the impact of various adhesive systems and surface pre-treatments on the bond strength between PEEK and composite. They reported superior surface pre-treatment of PEEK with sandblasting and silica coating compared to laser pre-treatment. The use of MMA-containing adhesives in combination with sandblasting demonstrated higher SBS values than the other pre-treatments. Their results were supported by Stawarczyk et al. (2014) and Rosentritt et al. (2015), who reported reliable SBS between PEEK and composite after surface treatment that combined roughening by airborne particle abrasion with aluminium oxide ($Al_2O_3$) and the application of an MMA-containing adhesive.

Chopra et al. (2023) evaluated the SBS and failure modes between PEEK and composite (3M ESPE Filtek 350 XT St. Paul, MN, USA). The authors conducted a comparative analysis of three different surface treatments: 95% sulphuric acid etching, airborne abrasion using 110 µm aluminium-oxide, and 99% acetone. All the specimens were bonded with a resin composite (3M ESPE) using 3M ESPE Single Bond Universal Adhesive before undergoing SBS testing. Each group included fifteen specimens that were evaluated for failure mode analysis using a scanning electron microscope. The results of the study showed superior SBS with the mechanical surface treatment group (110-µm aluminium oxide) compared with the chemical surface treatment groups (sulphuric acid and acetone). The air-abraded group showed the highest frequency of the mixed-type fracture mode, which suggests a strong bond between composite and PEEK after air abrasion.

### 2.2 b) PEKK

Lee et al. (2017) evaluated the SBS of veneered composite to PEKK using three different surface treatments. Their findings showed that the combination of air abrasion and an adhesive agent increased SBS. No difference was found between the adhesive materials used. Dede et al. (2021) reported increased SBS between PEKK and composite when tribochemical silica airborne-particle abrasion with 30-µm silica-modified $Al_2O_3$ was used in combination with an MMA-based adhesive material.

Another study by Arvai et al. (2024) looked at the SBS of PEKK to composite using 110-µm aluminium oxide and silica as pre-treatments. The bonding protocols included in their study were Visiolink, PEKKbond, and a combination of PEKKbond and Visiolink following pre-treatment with either 110-µm aluminium oxide or silica. Their results showed superior SBS of PEKK to veneered composite when 110-µm aluminium oxide was used as a pre-treatment, followed by the application of both PEKKbond and Visiolink adhesives. They also reported higher SBS when 110-µm aluminium oxide was used in combination with PEKKbond compared with Visiolink.

The present study used an MMA-based adhesive system for PEEK and PEKK, following the manufacturer's instructions. Previous studies have also reported on the improved SBS of PEEK to composite with MMA-containing adhesive systems (Stawarczyk et al., 2014; Rosentritt et al., 2015;

Caglar et al., 2019). MMA is also reported to cause swelling of PEEK surfaces, allowing the dimethacrylate monomers to bond to the composite via methacrylate groups (Barto et al., 2021; Adem et al., 2022).

## 2.2 c) Comparing SBS of PEEK and PEKK materials

Gouveia et al. (2022) evaluated the SBS of composite (Gradia Revolution 2) to PEEK and PEKK. Thirty specimens each of pressed PEKK, milled PEKK, and milled PEEK were used in the investigation. These specimens were divided into six groups (n = 13) based on the surface treatment and manufacturing method. The specimens underwent airborne-particle abrasion using 110-µm aluminium oxide or were left untreated. Their results showed that airborne-particle abrasion with 110-µm aluminium oxide/alumina improved the SBS between veneering composite to both PEEK and PEKK polymers. Furthermore, the manufacturing process (milled or heat-pressed) did not affect the SBS when subjected to the same bonding technique.

There is limited literature comparing titanium, PEEK, and PEKK materials in terms of bonding properties. Furthermore, because PEEK and PEKK have similar chemical surface properties, with only changes in the proportion of the keto and ether groups, some authors presume that surface treatments will have the same effect on both materials (Fuhrmann et al., 2014; Stawarczyk et al., 2017). Alsadon et al. (2023) compared the SBS of composite bonded to PEKK, PEEK, zirconia, and nickel-chromium alloy (NiCr) substructure materials. The study looked at 40 samples from the four materials (n = 10). Although all materials in their study met the minimum acceptable limit for SBS of 10 MPa, the authors observed that PEKK exhibited lower SBS than PEEK. However, PEEK and PEKK exhibited superior SBS compared to zirconia and alloy materials. Their findings indicated adhesive failure as the most prevalent, with an equal number of specimens demonstrating cohesive and mixed failure.

# Chapter 2: Aims and Objectives

## 2.1 Aim of the study

The aim of this *in-vitro* study was to compare the shear bond strength (SBS) of veneering composite to PEEK, PEKK, and titanium, as well as their failure modes (adhesive, cohesive, mixed).

## 2.2 Objectives

1. To measure the SBS of composite with PEEK, PEKK and titanium using a universal Intron testing machine.
2. To compare the shear bond strengths of composite bonded to PEEK, PEKK and titanium
3. To determine the failure modes of composite bonded to PEEK, PEKK and titanium using a stereo microscope.

## 2.3 Research Statement

The null hypothesis is that no statistically significant difference exists between the SBS of a composite bonded to titanium, PEEK, and PEKK.

# CHAPTER 3: MATERIALS AND METHOD

UNIVERSITY OF THE WITWATERSRAND, JOHANNESBURG

HUMAN RESEARCH ETHICS COMMITTEE (MEDICAL)

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**REF:** *R14/49*

**PROTOCOL NO:** W-CBP-230323-01 (This is your ethics application study reference number. Please quote this reference number in all correspondence relating to this study)

**PROJECT TITLE:** *Comparing sheer bond strength of a composite resin to PEEK, PEKK and titanium*

Please find attached the Ethics Waiver Certificate for the above project. I hope it goes well and that an article in a recognized publication comes out of it. This will reflect well on your professional standing and contribute to the Government funding of the University.

MSWorks2000/Iain0007/ClearScanWaiver.wps

| UNIVERSITY OF THE WITWATERSRAND, JOHANNESBURG | HUMAN RESEARCH ETHICS COMMITTEE (MEDICAL) |
|---|---|

**Office of the Deputy Vice-Chancellor (Research & Innovation)**

23/03/2023

Ref: W-CBP-230323-01

**TO WHOM IT MAY CONCERN**

**Waiver:** This certifies that the following research does not require clearance from the Human Research Ethics Committee (Medical)

**Investigator:** Dr M Maphuta, et al
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**Supervisor:** Dr VJ Hoods-Moonsammy

**School:** Oral Health Sciences
**Department:** Prosthodontics
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**Project title:** *Comparing sheer bond strength of a composite resin to PEEK, PEKK and titanium*

**Reason:** In vitro laboratory study
No human participants will be involved in the study

CB Penny

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## 3.1 Study design

The study is an *in-vitro* descriptive study.

## 3.2 Sample size calculation

The sample size for this study was calculated using estimates for a one-way analysis of variance (ANOVA). Data from a pilot study conducted in the Prosthodontics Department (Wits University), which measured the same variables to obtain the mean and standard deviation (SD) for SBS for titanium, PEEK, and PEKK, was used to calculate the sample size. The pilot study found the estimates for titanium (25.9 ± 6.12) and PEEK (20.76 ± 6.90). Because PEKK is stronger than PEEK and weaker than titanium (Lee et al., 2017), it was estimated that the mean for PEKK would be an average of the two (23.38) materials. The calculation was performed to allow for within-group variance, which was estimated as a square of the SD for titanium and PEEK (var = 37.5 – 47.6). The sample size for the largest within-group variance was set at 35 specimens per group. The calculation was performed in Stata version 15 (StataCorp, USA) at a significance level (alpha) of 5% and a power (1-beta) of 80%.

## 3.3. Specimen preparation and data collection

The study design ensured that quality data were collected and biases were minimised or eliminated. The same operator performed SBS testing and data recording. The mixture of the acrylic resin was performed as per the manufacturer's instructions, and the mould was standardised for all specimens. The Instron machine from the School of Oral Health Sciences was used for shear bond testing and was calibrated prior to data collection. Access to the Instron room was only permitted to the operator for the entire investigation duration. The inspection of the specimens was performed at the School of Anatomical Sciences using a stereo microscope to determine the mode of failure. The microscope's settings and orientation were kept consistent for all specimens.

## 3.4. Methods

### 3.4.1. PEEK, PEKK and titanium disc preparation

Titanium, PEEK, and PEKK specimens were milled. The specimens were standardised to 16 mm in diameter and 11 mm in thickness (Figure 1a). The specimens were subsequently embedded in auto-polymerizing acrylic resin, increasing their dimensions to 18mm in diameter and 12mm in thickness (Figure 1b). This was conducted to confirm the appropriate fit of the specimen within the stainless-steel ring designed to secure it in the universal testing machine (Figure 1c).

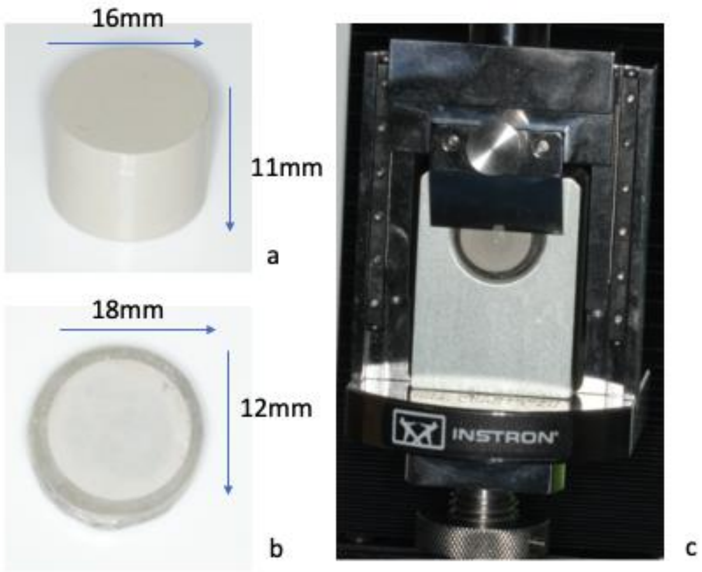



**Figure 1:** a) Milled PEEK specimen with standardised dimensions of 16mm diameter and 11mm thickness b) PEEK specimen with dimensions of 18mm diameter and 12mm thickness, modified using auto-polymerising acrylic resin c) PEEK specimen mounted within a stainless-steel frame, positioned in the Instron universal testing machine

An impression of the socket within the stainless-steel frame was taken using laboratory putty (Figure 2a, b, c). This was cast using type 3 dental stone (yellow plaster) (Figure 3a). After the plaster had hardened, the putty was extracted (Figure 3b). Fifty percent of the concavity formed by the putty was filled with auto-polymerizing acrylic (Figure 3c). PEEK, PEKK, and titanium specimens were inserted, leading to an overflow of surplus acrylic. After setting, the PEEK, PEKK, and titanium specimens were taken out from the plaster, and the acrylic was subsequently trimmed to fit into the stainless-steel frame (Figure 3d).

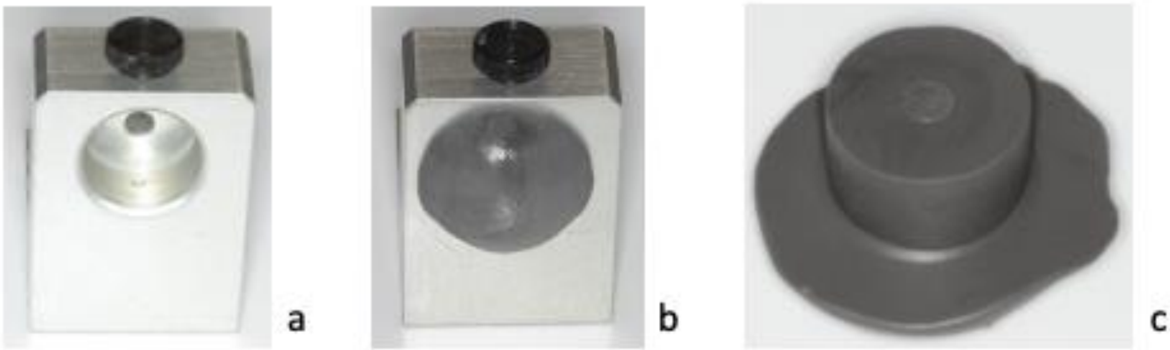


**Figure 2:** a) The Instron machine's stainless steel frame holds the specimens. b) Putty impression material was inserted into the socket in the stainless-steel frame. c) Putty impression of the socket of the stainless-steel frame

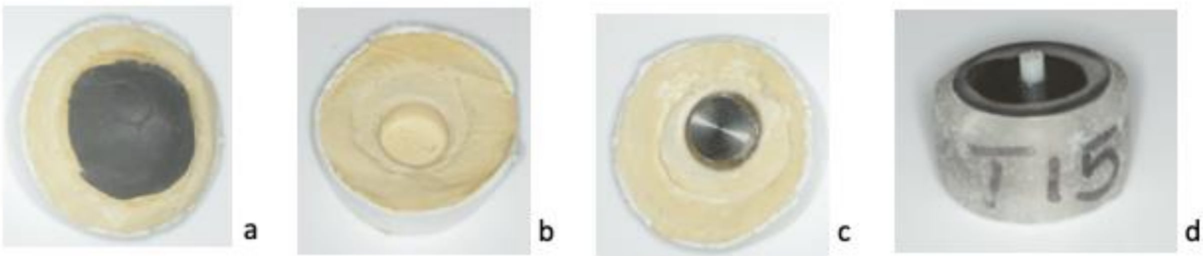


**Figure 3:** a) Putty impression poured in dental stone. b) Concavity following removal of putty impression
c) Titanium disc embedded in auto-polymerising acrylic. d) The trimmed titanium specimen with bonded composite

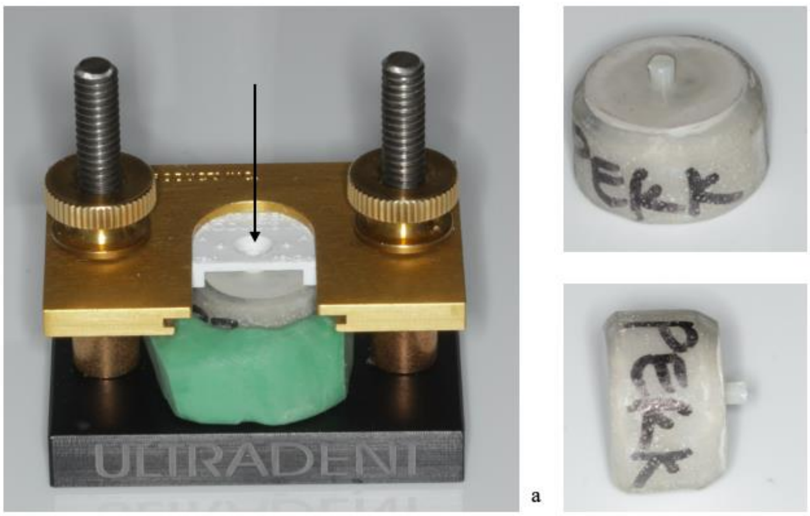
### 3.4.2. Surface treatment for PEEK, PEKK and titanium

All specimens (PEEK, PEKK and titanium) underwent grinding using silicon carbide paper with grit sizes of 600 and 1000. The surfaces underwent sandblasting using alumina particles of 110μm grain size and then lightly air-blown. The sandblasting process involved applying an air pressure of 0.45 MPa, with the nozzle positioned 10 mm away from the specimen, for 15 seconds (Fuhrmann et al., 2014; Lee et al., 2017). Next, an adhesive containing MMA (PEKKbond, AnaxDent, Ardmore, USA) was applied to the PEKK and PEEK surfaces according to the manufacturer's instructions (Gouveia et al., 2021).

The titanium specimens were bonded using a primer and adhesive containing MDP (Plafique, Universal Bond). Adhesives (PEKKbond for PEEK and PEKK, and Plafique, Universal Bond for titanium) were applied to the surface of the specimens using a disposable brush for 20 seconds, followed by gentle air drying for 5 seconds using oil-free compressed air. This was succeeded by light curing using an LED polymerisation unit with a light output exceeding 800 mW/cm² for 10 seconds. This step was undertaken to prepare the specimens for bonding to composite material.

### 3.4.3. Bonding of composite

The specimens were bonded with the composite (Gradia® Plus Heavy Body Paste, GC, Europe) using an Ultradent bonding mould attached to an Ultradent bonding clamp (Ultradent, South Jordan, UT, USA). The composite material was inserted into the central opening of the bonding mould using the smaller end of a non-serrated amalgam plugger. It was then cured at an angle perpendicular to the surface for 40 seconds. A composite specimen measuring 2 mm in diameter and 3 mm in height was fabricated. Prior to the shear bond test, the specimens were stored in distilled water at a temperature of 37 degrees Celsius for 24 hours (Gouveia et al., 2021; Adem et al., 2022).




b

a c

**Figure 4:** a) The PEKK specimen mounted in the bonding mould and bonding clamp. The arrow indicates the space in the bonding mould through which the composite was packed. b) and c) PEKK specimen with a bonded composite of 2mm diameter and 3mm height, guided by the bonding mould

### 3.4.4. Shear testing

Each specimen was secured to a test base clamp on the Instron testing machine (Instron, UK). The SBS was assessed using an Instron shear test instrument fitted with a 2kN load cell. The shearing load was exerted parallel to the bonded surface at a loading distance of 1mm (fig 5) and a crosshead speed of 1 mm/min (Lee et al., 2017). The force was continuously applied until the composite material was entirely sheared from the PEEK, PEKK, or titanium specimens.


**Figure 5:** PEKK specimen securely contained within the stainless-steel frame of the Instron universal testing machine. A chisel-shaped rod fitted with a 2kN load cell directed at the adhesive interface

### 3.4.5. Evaluation of failure mode

Subsequent to shear failure, all specimens were examined using a stereo microscope (Nikon SMZ 1500, Johannesburg, South Africa) at a magnification of 16X to determine the failure mode (adhesive, cohesive, or mixed). This procedure was used to investigate the fractured interface resulting from the shear test. Adhesive failure occurs at the interfaces between the composite and PEEK/PEKK/titanium materials. Cohesive failure describes bond failure within the composite material as a result of the shearing force (GPT- 10), whereas mixed failure involves a combination of adhesive and cohesive failures. True-colour images of the specimens were produced by the microscope.

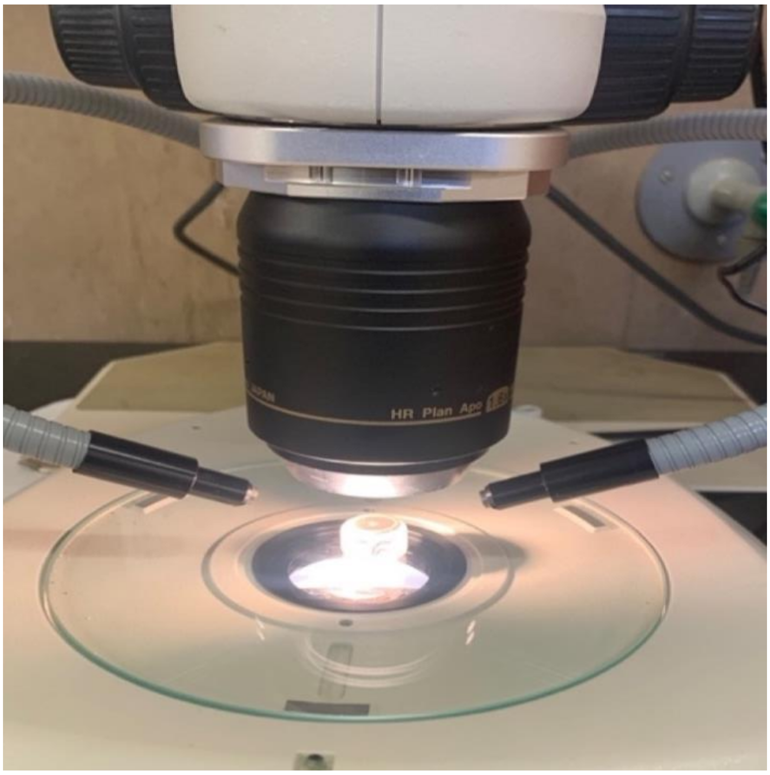


Figure 6: Failure mode being assessed on a PEEK specimen using a Stereo Microscope

## 3.5. Data analysis

Data was captured in the Research Electronic Data Capture (REDCap), which is an electronic database for clinical research. Continuous data (SBS) was assessed for normality, and where appropriate, data was summarised as means and standard deviations. A one-way analysis of variance (ANOVA) was used to assess differences in SBS between titanium, PEKK, and PEEK bonding materials, with multiple comparison tests using Bonferroni. Differences in bonding failures between the materials were evaluated using the Pearson chi-square test. All analyses were performed at the 5% significance level in the latest version of Stata (StataCorp, USA).

**Table 1:** Analysis plan

| **Objective** | **Method** |
|---|---|
| To measure the SBS of composite with PEEK, PEKK and titanium using a universal Intron testing machine. | Presented as mean and standard deviation |
| To compare the SBS of composite with PEEK, PEKK and titanium | Compared with a one-way ANOVA with Bonferroni multiple comparisons |
| To determine failure modes of composite to PEEK, PEKK and titanium using a stereo microscope. | Compared with Pearson chi-square test |

# CHAPTER 4: RESULTS

## 4.1 Descriptive statistics for SBS in MPa

Measurements of the compressive stress of PEEK, PEKK, and titanium at maximum compressive load (SBS) were summarised using the mean and standard deviation (Table 2 and Figure 7).

**Table 2:** Mean SBS (MPa) and standard deviations of titanium, PEEK and PEKK bonded to veneering composite

| Material | Mean (MPa) | SD | 95% CI | Range |
|---|---|---|---|---|
| Titanium | 28.60 | ±9.84 | 25.20 - 32.00 | 8.03 - 49.06 |
| PEEK | 16.83 | ±5.71 | 14.90 - 18.80 | 6.30 - 31.98 |
| PEKK | 25.80 | ±5.44 | 23.90 - 27.70 | 12.67 - 42.59 |

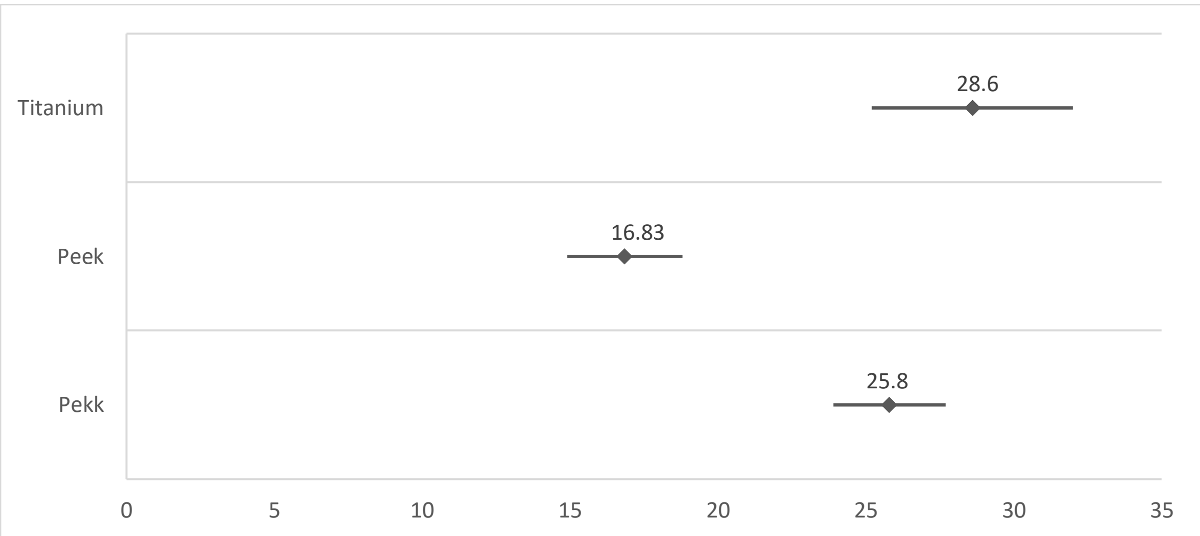



**Figure 7:** Forest plot showing the mean SBS of titanium, PEEK and PEKK

Titanium shows a high mean SBS of 28.60 MPa, with a 95% confidence interval (CI) ranging from 25.20 to 32.00 MPa and a standard deviation (SD) of 9.84 MPa. PEEK has a lower mean SBS of 16.83 MPa, with a 95% confidence interval between 14.90 and 18.80 MPa and a standard deviation of 5.71 MPa. PEKK shows a moderate mean compressive stress of 25.80 MPa, with a 95% confidence interval from 23.90 to 27.70 MPa and a standard deviation of 5.44 MPa. This mean value indicates that PEKK has a higher SBS to composite, closer to that of titanium.

## 4.2 Analysis of Variance (ANOVA) for Maximum SBS

An ANOVA test was done to determine any statistically significant differences between the means of the research materials (titanium, PEEK, and PEKK). In this test, the null hypothesis that the mean SBS of all three materials is equal was assessed.

**Table 3:** ANOVA comparisons for SBS

| **Source** | **Partial SS** | **df** | **MS** | **F** | **Prob > F** |
|---|---|---|---|---|---|
| Model | 271.69 | 2 | 135.84 | 24.97 | 0.0000 |
| Material | 271.69 | 2 | 135.84 | 24.97 | 0.0000 |
| Residual | 554.83 | 102 | 5.44 | | |
| Total | 826.52 | 104 | 7.95 | | |

## 4.3 Two-Sample T-Tests for Maximum SBS

To further investigate the differences between specific pairs of materials, we conducted two-sample t-tests.

**Table 4:** Comparison of SBS (in MPa) between the material groups

| **Group 1** | **Group 2** | **Mean Difference** | **Std. Error** | **98.3% CI** | ***p-value*** |
|---|---|---|---|---|---|
| Titanium | PEEK | 3.77 | 0.62 | 2.26 - 5.28 | *< 0.0001* |
| Titanium | PEKK | 0.90 | 0.61 | -0.60 : 2.39 | *0.1449* |
| PEEK | PEKK | 2.87 | 0.43 | -3.92 : -1.82 | *< 0.0001* |

The difference in means between titanium and PEEK is 3.77 MPa. The t-value is 6.12, and the p-value is less than 0.0001. These results confirm that titanium has a significantly higher mean compressive stress than PEEK. The difference in means between titanium and PEKK is 0.90 MPa. The t-value is 1.47 and the p-value is 0.1449, which exceeds the 0.05 significance level. These results indicate that the difference in mean compressive stresses between titanium and PEKK is not statistically significant. The difference in means between PEEK and PEKK is 2.87 MPa. The t-value is -6.73, and the p-value is less than 0.0001. These results confirm that PEKK has a significantly higher SBS to composite than PEEK.

## 4.4 Failure Modes by Material

**Table 5:** Failure mode distribution of titanium, PEEK and PEKK

| **Material** | **Adhesive** | | **Cohesive** | | **Mixed** | |
|---|---|---|---|---|---|---|
| | **Frequency** | *%* | | | **Frequency** | *%* |
| ***Titanium*** | 4 | *11.4%* | 0 | 0% | 31 | *88.6%* |
| ***PEKK*** | 20 | *57.1%* | 0 | 0% | 15 | *42.9%* |
| ***PEEK*** | 29 | *82.9%* | 0 | 0% | 6 | *17.1%* |

p-value <0.001

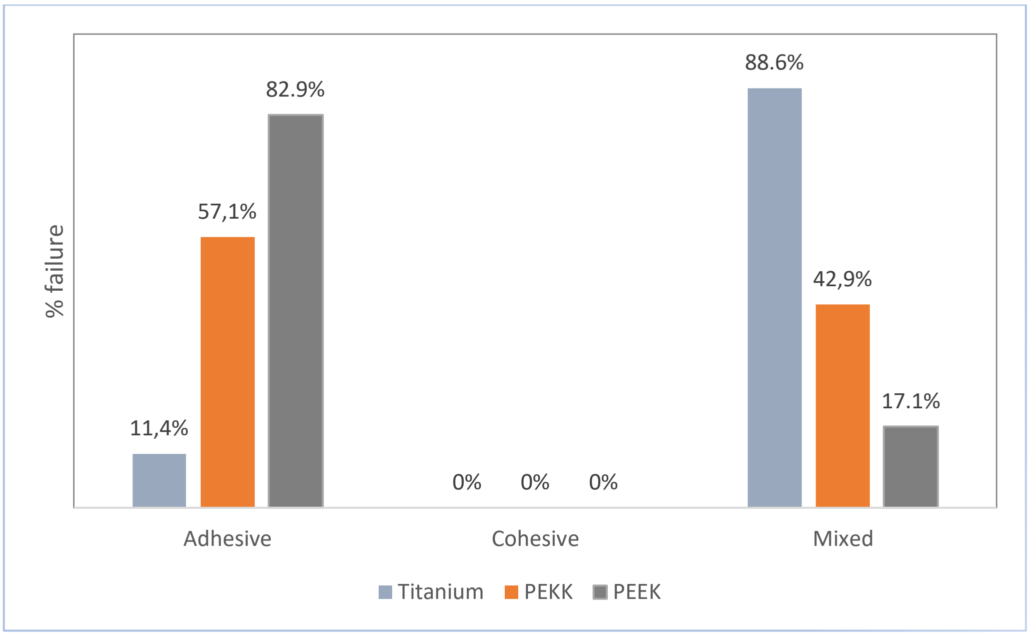
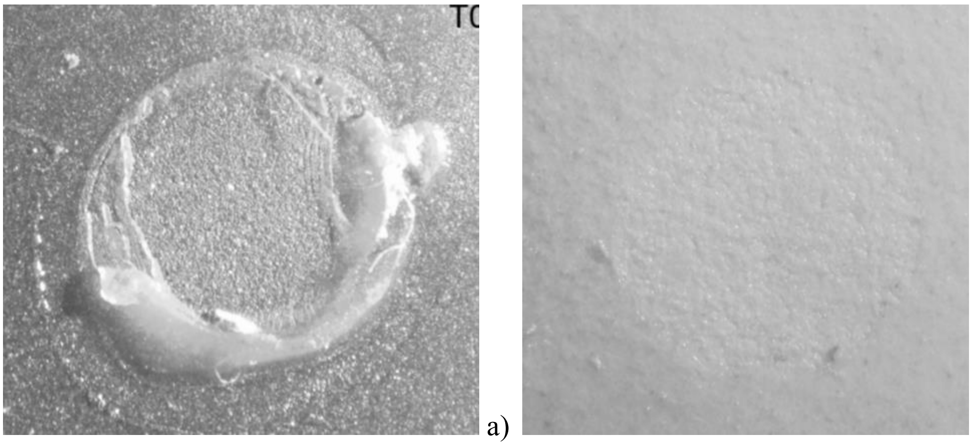



**Figure 8**: Bar graph depicting the failure modes for Titanium, PEEK and PEKK



a) *b)*

**Figure 9:** a) View of titanium specimen at 16X magnification showing mixed failure b) PEKK specimen showing adhesive failure. None of the specimens showed cohesive failure

Titanium predominantly shows mixed failure modes, with 88.6% of failures involving mixed failures and 11.4% of adhesive failures. PEEK is more prone to adhesive failures, with 82.9% of failures occurring at the adhesive interface and only 17.1% showing mixed failures. PEKK also presented with predominantly adhesive failure, with 57.1% of failures being adhesive and 42.9% being mixed failures.

# CHAPTER 5: DISCUSSION

The present study focused on comparing the SBS of PEEK, PEKK, and titanium to composite material, while also examining their respective failure modes. The study aimed to give a comprehensive assessment to direct material selection according to performance requirements and failure characteristics. A focus was placed on assessing the potential correlation between the SBS and the failure modes of the study materials.

An essential finding from the data was that the confidence intervals range for PEKK (23.90 to 27.70 MPa) and Titanium (25.20 to 32.00 MPa) overlap, suggesting that their average SBS values are not statistically different. The presence of this overlap suggests that there is no definitive difference in SBS between titanium and PEKK within the confidence limits, and any observed differences might not be statistically significant. Conversely, the confidence interval for PEEK (14.90 to 18.80 MPa) is not concurrent with that of titanium or PEKK, suggesting a significant difference in the mean SBS. The absence of an overlapping confidence interval emphasises the inferior performance of PEEK in comparison to the other two materials. Therefore, the study's findings led to the rejection of the hypothesis that the materials would exhibit comparable SBS.

Two methods exist for processing PAEK polymer materials, including hot pressing and milling. This study selected milled PEEK, PEKK, and titanium materials for bond strength testing based on the established lack of statistical variations in the optical and mechanical characteristics between pressed and milled materials (Gouveia et al., 2022). Previous research has shown that both surfaces can be treated similarly, with similar bond strengths (Gouveia et al., 2022; Alsadon et al., 2023).

## 5.1 Shear bond strength

The present study's findings are consistent with those of Lee et al. (2009), who similarly documented a greater SBS, greater than 20 MPa of titanium alloy to veneered composite when sandblasting was used in conjunction with metal conditioner. This SBS is similar to the value reported in the present study (28.60 MPa). Furthermore, the authors documented only adhesive failure at the interface of all their specimens, in contrast to the mostly mixed failure modes seen in the present study. However, although the predominant failure mode for titanium specimens was mixed in the present study, their surface assessment revealed a more significant proportion of adhesive rather than cohesive failure (fig 9a).

The average SBS of PEEK and PEKK samples, as reported in a recent study by Alsadon et al. (2023), was 16.23 MPa and 14.32 MPa, respectively, with Visiolink used as an adhesive. The present investigation showed a similar mean SBS of 16.83 MPa for PEEK. However, the SBS for PEKK was greater in the present study, measuring 25.80 MPa. Thus, PEKK bond and Visio-link adhesives yield comparable outcomes for PEEK materials following 110-µm aluminium oxide airborne-particle abrasion. However, when PEKKbond was used as an adhesive instead of Visio-link for PEKK, it exhibited significantly superior SBS. This study aligns with the findings of Arvai et al. (2024), who noted an increased SBS of composite to PEKK when 110-µm aluminium oxide airborne particles were used with PEKKbond as a bonding agent, in contrast to their use with Visio-link bonding agent. Similarly, Lee et al. (2017) reported a SBS of 16.33 MPa for PEKK when subjected to surface treatment with 110-µm aluminium oxide airborne particles and Visio-link bonding agents. This result is lower than the mean SBS values of 25.80 MPa observed in the present study using PEKKbond.

Adhesive systems influence the chemical bond between the veneering composite and the framework material. Gouveia et al. (2022) reported that the SBS of PEEK and PEKK were similar when subjected to the same surface treatments. This finding contrasts with the results of the current study, which showed significantly higher SBS for PEKK when surface treatment with 110-µm aluminium oxide was used in combination with PEKKbond adhesive compared to PEEK. Variations in composites and the chemical bonding agent used in the investigations may account for the observed differences.

PEKKbond adhesive systems contain methyl methacrylate, which dissolve the superficial PAEK surface. This dissolution enables the methylmethacrylate monomers to expand within the layer, enhancing bonding to the composite (Adem et al., 2022; Gouveia et al., 2022; Alsadon et al., 2023). The results are in line with those of Lee et al. (2017), who found that when bonded to composite, PEKK had a higher SBS than PEEK.

## 5.2 Failure modes

An important factor in understanding SBS measurements and results is failure mode analysis. Failure modes were classified according to the relative proportion of the veneering composite that remained on the substructure samples following shearing forces. The analysis provides insights into how each material fails under stress. The failure modes are categorised into adhesive, cohesive and combination failures. Understanding these modes helps predict how materials will behave under various conditions and informs decisions about their use in different clinical applications.

According to the failure mode analysis data, mixed failures were the most common type of failure for titanium (88.6 %), while PEEK and PEKK showed predominantly adhesive failures of 82.9% and 57.1%, respectively. There was no evidence of cohesive failure in any of the materials. The results were consistent with the findings reported by Alsadon et al. (2023), who also observed the lack of cohesive failure and the predominant adhesive failure linked to PEKK material, consistent with the findings of the present investigation. Fuhrmann et al., 2013 also reported a predominant adhesive failure associated with bonding composite to PEEK, despite the use of an MMA- containing primer.

Clinically, the ideal failure mode positively associated with increased SBS would be cohesive in nature. That is, the bond between the substrate and the veneering composite should be greater than the cohesive strength of the composite. Barto et al. (2022) found that the SBS of repaired composite-veneered PEEK, exhibiting both adhesive and mixed failures, was comparable to that of the original composite-veneered PEEK when airborne-particle abrasion and an MMA-based adhesive was used.

Behr et al. (2011) proposed that a 10 MPa or higher SBS is necessary to guarantee durable material bonding and attain clinical acceptability. The authors demonstrated that an adhesive bond strength of 10 MPa is required to withstand shear forces for a bonding interfacial area of 20 $mm^2$ and a maximum anterior occlusal force of 200 N. The stronger the bond between the substructure and veneering composite, the better it will resist the stresses imposed by resin polymerisation and oral function.

The findings of this study reinforce that significantly higher bonding strengths with values above the minimal clinically acceptable values can be achieved by combining surface treatment with 110-µm aluminium oxide with an MMA-based bonding agent for PEEK and PEKK, and an MDP-based bonding agent for titanium. In addition, the data indicate that PEKK has a bond strength to composite similar to titanium and significantly greater than PEEK. Therefore, this promotes its application as a core material and framework material in implant-supported prostheses. Since it has 80% higher compressive strength than PEEK (Lee et al., 2017; Fokas et al., 2019), it can be regarded as a viable treatment alternative to titanium when replacing dental tissues in cases of high aesthetic demands. Although the predominant failure mode was adhesive in nature, the repairability of composite veneers bonded to PAEK materials allows for chairside repair of adhesive and mixed failures with clinically acceptable bond strengths (Barto et al., 2022).

## 5.3 Limitations of the study

Because the bond strength/adherence of the veneering composite to the framework has proven to be an important factor in the long-term performance of the restorations, the bond strength results gained from this *in vitro* study can provide useful information about the behaviour and predictability of the veneered PAEK restorations. However, the bond strength evaluation was conducted under static loading conditions despite mastication being a dynamic process. The effect of saliva was also not included in the study. Therefore, the results must be interpreted cautiously. Studies considering the oral environment and clinical trials are recommended. Furthermore, clinical factors such as parafunction and opposing occlusion should also be considered.

# CHAPTER 6: CONCLUSION

Within the limitations of the study, the following conclusions can be drawn according to the results of the current study:

1. The SBS of composite to titanium was similar to that of composite bonded to PEKK.
2. PEKK and titanium materials could be considered superior to PEEK in terms of adhesion strength to veneered composite.
3. The combination of 110-µm aluminium oxide airborne-particle abrasion with PEKKbond leads to a significantly higher adhesive strength between composite and PEKK compared to PEEK polymer.
4. All the study materials exceeded the minimum acceptable limit for SBS of 10 MPa.
5. Although titanium and PEKK demonstrated similar SBS, titanium presented a predominantly mixed type of failure. Only three titanium specimens showed adhesive failure compared to twenty of the PEKK specimens, indicating a stronger bond of composite to titanium than PEKK.
6. PEEK demonstrated predominantly adhesive failure.
7. None of the specimens demonstrated cohesive failure.

Therefore, PEKK may serve as a viable substitute for titanium, offering cost-effectiveness, ease of repair and improved aesthetic results.

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# Appendix 1: Ethics clearance

UNIVERSITY OF THE WITWATERSRAND, JOHANNESBURG

HUMAN RESEARCH ETHICS COMMITTEE (MEDICAL)

**Office of the Deputy Vice-Chancellor (Research & Innovation)**

**TO:** Dr M Maphuta, et al
School: Oral Health Sciences
Department: Prosthodontics
Dental School
University

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**CC:** Supervisor: Dr VJ Hoods-Moonsammy <Yvonne.Hoods-Moonsammy@wits.ac.za>
and <HREC-Medical.ResearchOffice@wits.ac.za>

**FROM:** Mr Iain Burns
Human Research Ethics Committee (Medical)
Tel: 011 717 1252

E-mail: Iain.Burns@wits.ac.za

**DATE:** 23/03/2023

**REF:** *R14/49*

**PROTOCOL NO:** W-CBP-230323-01 (This is your ethics application study reference number. Please quote this reference number in all correspondence relating to this study)

**PROJECT TITLE:** *Comparing sheer bond strength of a composite resin to PEEK, PEKK and titanium*

Please find attached the Ethics Waiver Certificate for the above project. I hope it goes well and that an article in a recognized publication comes out of it. This will reflect well on your professional standing and contribute to the Government funding of the University.

MSWorks2000/Iain0007/ClearScanWaiver.wps

| UNIVERSITY OF THE WITWATERSRAND, JOHANNESBURG | HUMAN RESEARCH ETHICS COMMITTEE (MEDICAL) |
|---|---|

**Office of the Deputy Vice-Chancellor (Research & Innovation)**

23/03/2023

Ref: W-CBP-230323-01

**TO WHOM IT MAY CONCERN**

**Waiver:** This certifies that the following research does not require clearance from the Human Research Ethics Committee (Medical)

**Investigator:** Dr M Maphuta, et al
Student No. (if appropriate): 05 0397 8X
Staff No. (if appropriate): A0064390

**Supervisor:** Dr VJ Hoods-Moonsammy

**School:** Oral Health Sciences
**Department:** Prosthodontics
Dental School
University

**Project title:** *Comparing sheer bond strength of a composite resin to PEEK, PEKK and titanium*

**Reason:** In vitro laboratory study
No human participants will be involved in the study

Dr CB Penny
Chairperson: Human Research Ethics Committee (Medical)

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# Appendix 2: Calibration Certificates

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## Speed and Displacement Verification Certificate

Version 2.21

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Form No. CAL02B

| | |
|---|---|
| Certificate Number | 719DS |
| Customer Name | University of Witwatersrand (Medical School) |
| Address | 35 Jubilee Rd<br>Parktown<br>Johannesburg South Africa |
| Physical Calibration location | On-site<br>Instron Room Level 3 |
| Make, Model / Serial No | Instron 3344 K6219 |
| Machine Travel | 1000.00 mm |
| Max speed verified | 100.00 mm/min |
| Max displacement verified | 200.00 mm |

| | |
|---|---|
| Calibration date | 16 Nov 22 |
| Date of Issue | 24 Nov 22 |
| Start Temperature | 24.7 Deg C |
| End Temperature | 27.6 Deg C |
| Calibration Technician | Nhlanhla Ntombela |
| Technical Signatory | Sheldon Keet |

| Standard used | Displacement | Speed | 3 |
|---|---|---|---|
| Device | Digital Caliper | Encoder | |
| Serial Number | 2303192319 | 6244410 | |
| Traceable cert No | G 7143 | DMDIM-4064 | |
| Travel | 300 mm | 2000 mm | |
| Readout used | On Device | 83242 | |
| Units | mm | mm | |
| Resolution | 0.0100 | 0.0100 | |
| Procedure Used | P01B | P01B | |
| Machine output calibrated | PC | | |

### Displacement verification check

runs in mm for crosshead / actuator travel

| Cal Steps mm | Standard Used | Output Resolution | Pre cal Run | Run 1 | Run 2 | Run 3 | Run 4 | Run 5 | Average disp mm | Error mm | Error % | Std Dev % | Range % | Uncertainty +/- |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 50 | 2303192319 | 0.01 | 49.99 | 49.99 | 49.99 | 49.99 | | | 49.99 | -0.01 | -0.02% | 0.00% | 0.00% | 0.5% |
| 100 | 2303192319 | 0.01 | 99.99 | 99.99 | 99.99 | 99.99 | | | 99.99 | -0.01 | -0.01% | 0.00% | 0.00% | 0.5% |
| 150 | 2303192319 | 0.01 | 149.98 | 149.98 | 149.98 | 149.98 | | | 149.98 | -0.02 | -0.01% | 0.00% | 0.00% | 0.5% |
| 180 | 2303192319 | 0.01 | 180.00 | 180.00 | 180.00 | 180.00 | | | 180.00 | 0.00 | 0.00% | 0.00% | 0.00% | 0.5% |
| 200 | 2303192319 | 0.01 | 199.99 | 199.99 | 199.99 | 199.99 | | | 199.99 | -0.01 | 0.00% | 0.00% | 0.00% | 0.5% |

### Speed verification check

| | Standard Used | Output Resolution | Pre cal Run | Run 1 mm/min | Run 2 mm/min | Run 3 mm/min | Run 4 mm/min | Run 5 mm/min | Average mm/min | Error mm/min | Error % | Std Dev % | Range % | Uncertainty +/- |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 100 | 6244410 | 0.01 | 0.00 | 99.99 | 100.00 | 100.00 | | | 100.00 | 0.00 | 0.00% | 0.01% | 0.01% | 2.1% |
| 200 | 6244410 | 0.01 | 0.00 | 199.98 | 199.88 | 199.88 | | | 199.91 | -0.09 | -0.05% | 0.02% | 0.04% | 2.1% |
| 300 | 6244410 | 0.01 | 0.00 | 299.98 | 299.68 | 299.87 | | | 299.84 | -0.16 | -0.05% | 0.05% | 0.11% | 2.1% |
| 400 | 6244410 | 0.01 | 0.00 | 399.74 | 400.03 | 400.19 | | | 399.99 | -0.01 | 0.00% | 0.06% | 0.11% | 2.1% |
| 500 | 6244410 | 0.01 | 0.00 | 499.81 | 500.20 | 500.00 | | | 500.00 | 0.00 | 0.00% | 0.04% | 0.08% | 2.1% |

**Notes**

1 The reported expanded uncertainty is based on a standard uncertainty multiplied by a coverage factor k = 2 providing a level of confidence of approximately 95%, the uncertainty of measurement has been estimated in accordance with the principles defined in the GUM, Guide to Uncertainty of Measurement, ISO, Geneva, 1993

2 This calibration is valid at the time of issue of the certificate. The continued retention of accuracy will depend on the care with which the instrument is handled and used.

3 Standards used in the execution of this calibration have been certified by SANAS accredited laboratories and through the above provide an unbroken chain of traceability to national standards by NMISA National Metrology Institute of South Africa.

End of Certificate

| | | |
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| ShareCall: [illegible]<br>ShareFax: [illegible]<br>Page 1 of 1 | A D I Scientific (Pty) Ltd. t/a Advanced Laboratory Solutions<br>P O Box [illegible], Bryanston, 2021<br>[illegible] Angus Crescent, Northlands Business Park<br>Newmarket Road, Northriding, 2189, South Africa<br>Registration Number: [illegible] | E-mail: support@advancedlab.co.za<br>Web: www.advancedlab.co.za |

| | | |
|---|---|---|
| sanas | **Force Calibration Certificate**<br>Version 2.21 | advanced laboratory SOLUTIONS |

Form No. CAL02A

| Certificate Number | 4719 | | |
|---|---|---|---|
| Customer Name | University of Witwatersrand (Medical School) | | |
| Address | 35 Jubilee Rd | | |
| | Parktown | | |
| | Johannesburg | 2193 | South Africa |
| Physical Calibration location | On-site | | |
| | Instron Room Level 3 | | |
| Make, Model / Serial No | Instron 3344 | K6219 | |
| Frame Capacity | 2.00 | kN | |
| Load cell Range | 2.00 | kN | |
| Serial number | 83363 | | |
| Calibrated range | 2.00 | kN | |
| Selected units (N or kN) | kN | | |

| | |
|---|---|
| Calibration date | 16 Nov 22 |
| Date of issue | 24 Nov 22 |
| Start Temperature | 24.7 Deg C |
| End Temperature | 27.6 Deg C |
| Calibration Technician | Nhlanhla Ntombela |
| Technical Signatory | Sheldon Keet |

| Standard used | 1 | 2 | 3 |
|---|---|---|---|
| Serial Number | WEIGHTS PIMA 1 | 63540003 | |
| Traceable cert No | 184-22 | 1462 | |
| Capacity (kN) | 180 (b) | 3 | |
| Readout used | None | 83241 | |
| Full scale | None | 2.50 | |
| Units | N | kN | |
| Resolution | 0.0000 | 0.0001 | |
| Procedure Used | P 01A | P 01 | |
| Constant force (indicated / true) | CTF | CIF | |
| Machine output calibrated | PC | Machine adjusted ? NO | |
| | | Change over point 1 (kN) 0.1 | |

## Calibration performed in Tension

| Cal Steps kN | Standard Used | Method Used | Output Res | Pre cal Run | Run 1 | Run 2 | Run 3 | Run 4 | Run 5 | Average kN | Error kN | Error % | Std Dev kN | Range kN | U % | U kN |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Runs in kN | | | | | | | | | | | |
| 0.000 | WEIGHTS PIMA 1 | CTF | 0.0001 | 0.000 | 0.0002 | 0.0002 | 0.0002 | | | 0.0002 | -0.0002 | | 0.000 | 0.000 | | |
| 0.020 | WEIGHTS PIMA 1 | CTF | 0.0001 | 0.020 | 0.0200 | 0.0200 | 0.0200 | | | 0.0200 | 0.0000 | 0.00% | 0.000 | 0.000 | 0.58 | 0.000 |
| 0.040 | WEIGHTS PIMA 1 | CTF | 0.0001 | 0.040 | 0.0400 | 0.0400 | 0.0400 | | | 0.0400 | 0.0000 | 0.00% | 0.000 | 0.000 | 0.40 | 0.000 |
| 0.100 | WEIGHTS PIMA 1 | CTF | 0.0001 | 0.100 | 0.1002 | 0.1002 | 0.1002 | | | 0.1002 | -0.0002 | -0.20% | 0.000 | 0.000 | 0.40 | 0.000 |
| 0.100 | 63540003 | CIF | 0.0001 | 0.101 | 0.0995 | 0.0999 | 0.0999 | | | 0.0998 | -0.0002 | -0.24% | 0.000 | 0.000 | 0.72 | 0.001 |
| 0.200 | 63540003 | CIF | 0.0001 | 0.202 | 0.1990 | 0.1998 | 0.1998 | | | 0.1996 | -0.0004 | -0.22% | 0.000 | 0.001 | 0.82 | 0.002 |
| 0.400 | 63540003 | CIF | 0.0001 | 0.403 | 0.4002 | 0.3999 | 0.3997 | | | 0.3999 | -0.0001 | -0.02% | 0.000 | 0.001 | 0.40 | 0.002 |
| 0.800 | 63540003 | CIF | 0.0001 | 0.806 | 0.7986 | 0.7986 | 0.7986 | | | 0.7986 | -0.0014 | -0.18% | 0.000 | 0.000 | 0.40 | 0.003 |
| 1.200 | 63540003 | CIF | 0.0001 | 1.207 | 1.2014 | 1.2012 | 1.2010 | | | 1.2012 | 0.0012 | 0.10% | 0.000 | 0.000 | 0.40 | 0.005 |
| 1.600 | 63540003 | CIF | 0.0001 | 1.606 | 1.6013 | 1.6013 | 1.6013 | | | 1.6013 | 0.0013 | 0.08% | 0.000 | 0.000 | 0.40 | 0.006 |
| 2.000 | 63540003 | CIF | 0.0001 | 2.005 | 2.0015 | 2.0022 | 2.0017 | | | 2.0018 | 0.0018 | 0.09% | 0.000 | 0.001 | 0.40 | 0.008 |
| 0.000 | 63540003 | CIF | 0.0001 | 0.001 | -0.0010 | -0.0012 | -0.0007 | | | -0.0010 | -0.0010 | | 0.000 | 0.000 | | |

Notes:

1 The reported expanded uncertainty is based on a standard uncertainty multiplied by a coverage factor K = 2 providing a level of confidence of approximately 95%, the uncertainty of measurement has been estimated in accordance with the principles defined in the GUM, Guide to Uncertainty of Measurement, ISO, Geneva, 1993.

1st Zero
2nd Zero
Max-min

2 On this certificate the actual target load for each calibration step is listed in the first column.
Dead weight calibrations are performed with constant true force. Runs using load cell force standards are usually performed using constant INDICATED force from the machine under test. The method for each calibration point is given in column 3 as CTF (constant true force) or CIF (constant indicated force).
Error calculations are adjusted accordingly

3 This certificate is issued in accordance with the conditions of the approval granted by the South African National Accreditation System.
It is a correct record of the measurements made. Copyright of this certificate is jointly owned by SANAS and Advanced Laboratory Solutions. This certificate may not be reproduced other than in full, except with the prior written approval of SANAS and of Advanced Laboratory Solutions

4 This calibration is valid at the time of issue of the certificate. The continued retention of accuracy will depend on the care with which the instrument is handled and used.

5 This calibration has been performed with EN ISO 7500 part 1AS A GUIDE. No instrument classification has been made based on the results achieved. Neither have hysteresis checks unless specifically stated.

6 Standards used in the execution of this calibration have been certified by SANAS accredited laboratories and therefore provide an unbroken chain of traceability to national standards maintained by NMISA-National Metrology Institute of South Africa held in Pretoria

7

| Classification according to ISO 7500-1 | Class |
|---|---|
| Over the range 80% to 100% of full scale this force measuring system meets the requirements of | 0.5 |
| Over the range 40% to 100% of full scale this force measuring system meets the requirements of | 0.5 |
| Over the range 20% to 100% of full scale this force measuring system meets the requirements of | 0.5 |
| Over the range 10% to 100% of full scale this force measuring system meets the requirements of | 0.5 |

End of Certificate

# Appendix 3: Turnitin Report

## Research report MDent Turnitin report-2.docx

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