



THE UNIVERSITY of EDINBURGH
School of Engineering

Performance of 3D printed discontinuous fibre reinforced polymer composites

CCUS Innovation 2.0

Key Knowledge Deliverable 2.2

December 2023

Key Knowledge Deliverable Cover Sheet

This Key Knowledge Deliverable (KKD) has been produced by The University of Edinburgh as part of the Department for Energy Security and Net Zero £1bn Net Zero Innovation Portfolio (NZIP) - CCUS Innovation 2.0 programme. The document is reflective of the status of the project at the time of writing. The material presented could have been subject to change as the project matured. These documents should not be considered a full representation of the final project.

Supercritical CO₂ power cycles are a novel process currently under consideration across the energy sector, including for carbon capture, usage, and storage (CCUS). Compared with conventional steam-driven systems, they boast higher cycle efficiencies, reduced emissions, and compact turbomachinery, resulting in reduced fuel and water consumption and lower capital expenditures. Through this project, John Crane—in collaboration with Cranfield University and the University of Edinburgh—aims to develop an innovative, uncooled high-temperature dry gas seal solution for supercritical CO₂ power cycles by using new simulations, material recipes and testing validations. This innovation, expected to work at high temperatures and pressure values, should significantly reduce leakages and potentially even enable the inclusion of an additional turbine expansion stage. Through a supercritical CO₂ cycle efficiency improvement, overall emissions will be significantly reduced, cutting the cost of CCUS and accelerating the adoption of supercritical CO₂ power cycles into existing and future power plants.

This KKD involves with the advancement of PEEK based composites for the polymer seal with a service temperature up to 200 °C through a fused deposition modelling (FDM) based 3D printing technique.

KKDs to be released in full:

D2.2 Performance of 3D printed discontinuous fibre reinforced polymer composites.

D3.1 Conceptual design of the rig

D3.2 Component commissioning of CO₂ source (subsystem 1)

D3.3 Integrated sub-systems test - Shakedown test

D4.1 Commissioning of rig at John Crane

D4.2 Integrated system + seal rig commissioning at Cranfield University

KKDs to be released after being modified with critical information removed:

D3.4 Design and build of static/ dynamic test cell for 300 bar, 250 °C

D3.5 Design and build of static/ dynamic test cell for 200bar, 600 °C (CO2)

D4.3 Test of rig operating at 200 bar, 300 C (CO2)

D4.4 Testing to 200 bar, 250 °C

D4.5 Testing to 200 bar, 500 °C (CO2)

D5.1 Design/ build test seal with new seal head module/ bd-module

D5.2 Dynamic seal testing/ Benchmark testing, 140 m/s, to 200 bar, to 200 °C

D5.3 Dynamic Seal Testing, 140 m/s, to 200 bar, to 200 °C



Klaus-Dieter Meck, 5th December 2023

Director Core Technology, John Crane UK Ltd.



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1.Introduction

1.1 Project Overview

Supercritical CO₂ (sCO₂) power cycles represent a cutting-edge development in the energy sector, with broad applications that extend to carbon capture, usage, and storage (CCUS). These cycles are gaining increasing attention for their potential to revolutionise power generation and environmental impact. Compared to conventional steam-driven systems, sCO₂ power cycles offer a range of compelling advantages, including high cycle efficiency, reduced emissions, compact turbomachinery, and reduced fuel and water consumption. In pursuit of these benefits and to overcome critical challenges, John Crane, in collaboration with Cranfield University and the University of Edinburgh, has embarked on a visionary project. The project's objective is the development of an innovative, uncooled high-temperature dry gas seal solution tailored specifically for supercritical CO₂ power cycles. This endeavour involves cutting-edge simulations, advanced material recipes, and rigorous testing validations.

1.2 Project Scope

The project scope includes the design, fabrication, and laboratory testing of critical DGS components (balance diameter sealing module) and system prototypes for application in rotating equipment (turbine and compressors) used in sCO₂ power cycles up to typical pressures and temperatures of 200 barg and 500 °C. The project also includes the design and commissioning of test equipment to verify and demonstrate the innovative DGS concept/prototype. Modular testing will be conducted at John Crane Slough and Cranfield University (CU) using the University's existing sCO₂ test loop modified to the requirements of this project. The project scope excludes dynamic seal testing of the DGS prototype at sCO₂ conditions but in air at maximum velocity (140 m/s) and pressure rating (200 barg), which is expected to occur following the completion of this project.

This report provides a comprehensive overview of the development of PEEK-based composites for a polymer seal, designed to operate at temperatures up to 200 °C, utilising a fused deposition modelling (FDM) based 3D printing technique. Commercially available PEEK composite filament with carbon fibre reinforcement has been procured from suppliers, featuring filaments containing 10% and 20% short carbon fibre reinforcement.

Following a series of trials for both filaments, the decision has been made to proceed with the 10% short carbon fibre-reinforced PEEK (sCF/PEEK). This choice is based on considerations of printability, printing quality, and the performance of the printed composite in meeting the first benchmark target temperature, for this first project phase of 200 °C. The report delves into the specifics of printing parameters and post-processing conditions, presenting characterisation results for microstructures and thermal-mechanical properties of the 3D printed sCF/PEEK composites. Additionally, a full-size seal demonstrator has been successfully printed.

2. Characterisation and optimisation of 3D printed sCF/PEEK composites

2.1 Optimisation of sCF/PEEK printing parameters and post-processing by hot-press

During the optimisation of the sCF/PEEK composite printing process, critical parameters were meticulously calibrated to ensure the production of the highest quality output, as outlined in Table 1. The nozzle temperature was set as 400 °C to facilitate optimal extrusion flow. To maintain structural integrity during the print, the build platform temperature was maintained at 140 °C, effectively minimising the risks of thermal distortion. The printing speed was determined to be 20 mm/s, achieving a balance between print efficiency and layer adhesion. A layer thickness of 0.2 mm was employed to improve the surface finish and enhance detail accuracy. Additionally, both the nozzle diameter and extrusion width were both set at 0.4 mm, a decision that promotes precision in the deposition of material.

The impact of these optimised parameters is clear in the printed sCF/PEEK samples, and their quality was assessed in relation to extrusion width variations shown in Figure 1 (a). The samples reveal a discernible relationship between extrusion width and print quality. The highest quality is observed in the sample with an extrusion width of 0.4 mm, which aligns with the optimised extrusion width parameter. Notably, the sample that underwent additional hot-press post-printing exhibited enhanced quality, highlighting the potential advantages of post-processing techniques.

The quality improvements from the samples with higher extrusion widths (0.8 mm and 0.6 mm) down to the optimised 0.4 mm underlines the critical importance of precise control over the extrusion parameters. Furthermore, Figure 1(b) illustrates the cycle of the hot-press program.

Table 1: Key parameters for sCF/PEEK composite 3D printing optimisation

Optimised Printing parameters	Value
Nozzle temperature	400 °C
Build platform temperature	140 °C
Printing speed	20 mm/s
Layer thickness	0.2 mm
Nozzle diameter	0.4 mm
Extrusion width	0.4 mm

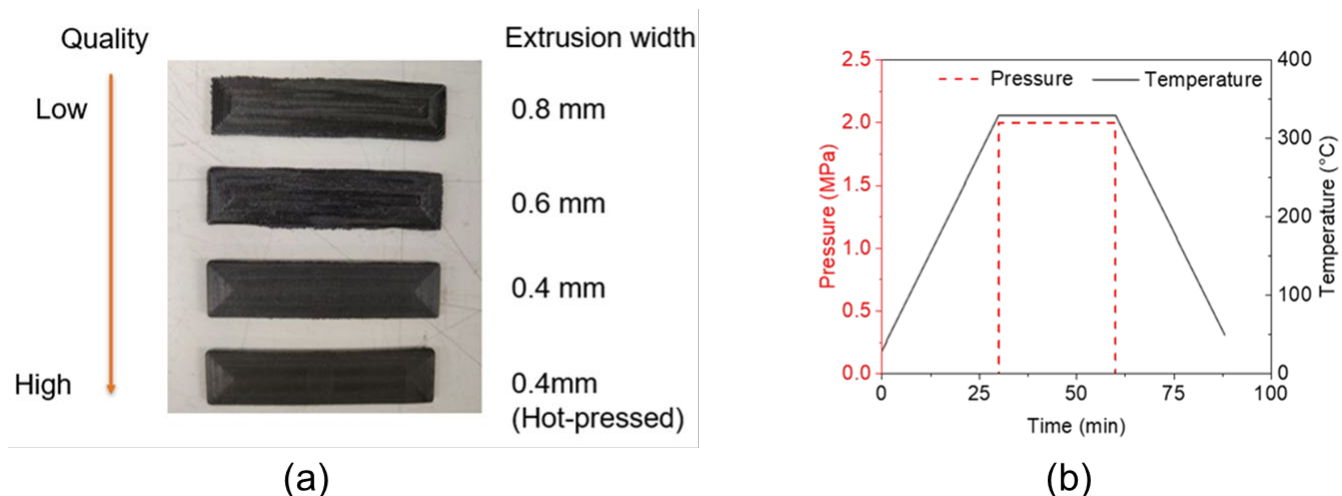
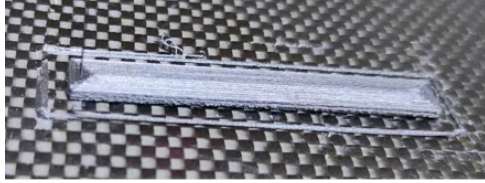
Figure 1 : (a) Influence of extrusion width on sCF/PEEK composite quality with/without post-processing (b) Hot-press processing window for 3D printed sCF/PEEK.

Figure 2 displays two sets of samples fabricated using sCF/PEEK material. The upper set of images showcases a 3D-printed part with distinct layer lines on the left, which is a common characteristic of additive manufacturing. On the right side, two adjacent samples are presented before post-processing, with a ruler indicating their approximate length of 54 mm. In contrast, the lower set of images presents the same case after undergoing a hot-pressing treatment. The post-processing technique is clearly transformative, visibly altering the appearance of the 3D-printed part. The surface becomes smoother, and mechanical properties are enhanced through improved inter-layer adhesion. The central part exhibits a more consolidated and less textured surface compared to its pre-hot-pressed state. While the hot-pressing process has induced some dimensional changes to the piece, typically in thermally treated polymers, this transformation from a layered structure to a densified one confirms the effectiveness of the hot-

pressing method in enhancing the surface quality and structural integrity of printed sCF/PEEK composites.

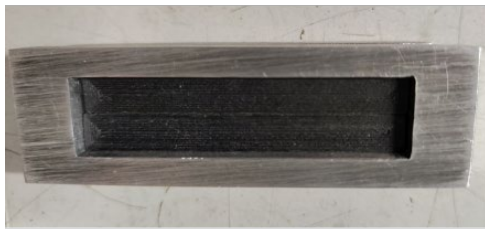
Figure 2: Comparative display of sCF/PEEK samples: 3D-printed and post-processing specimens



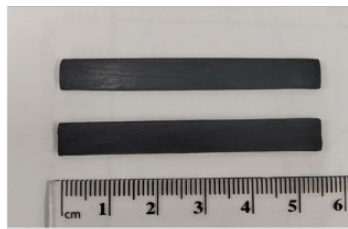
Specimen on the bed



Printed specimens



Two specimens in the mould



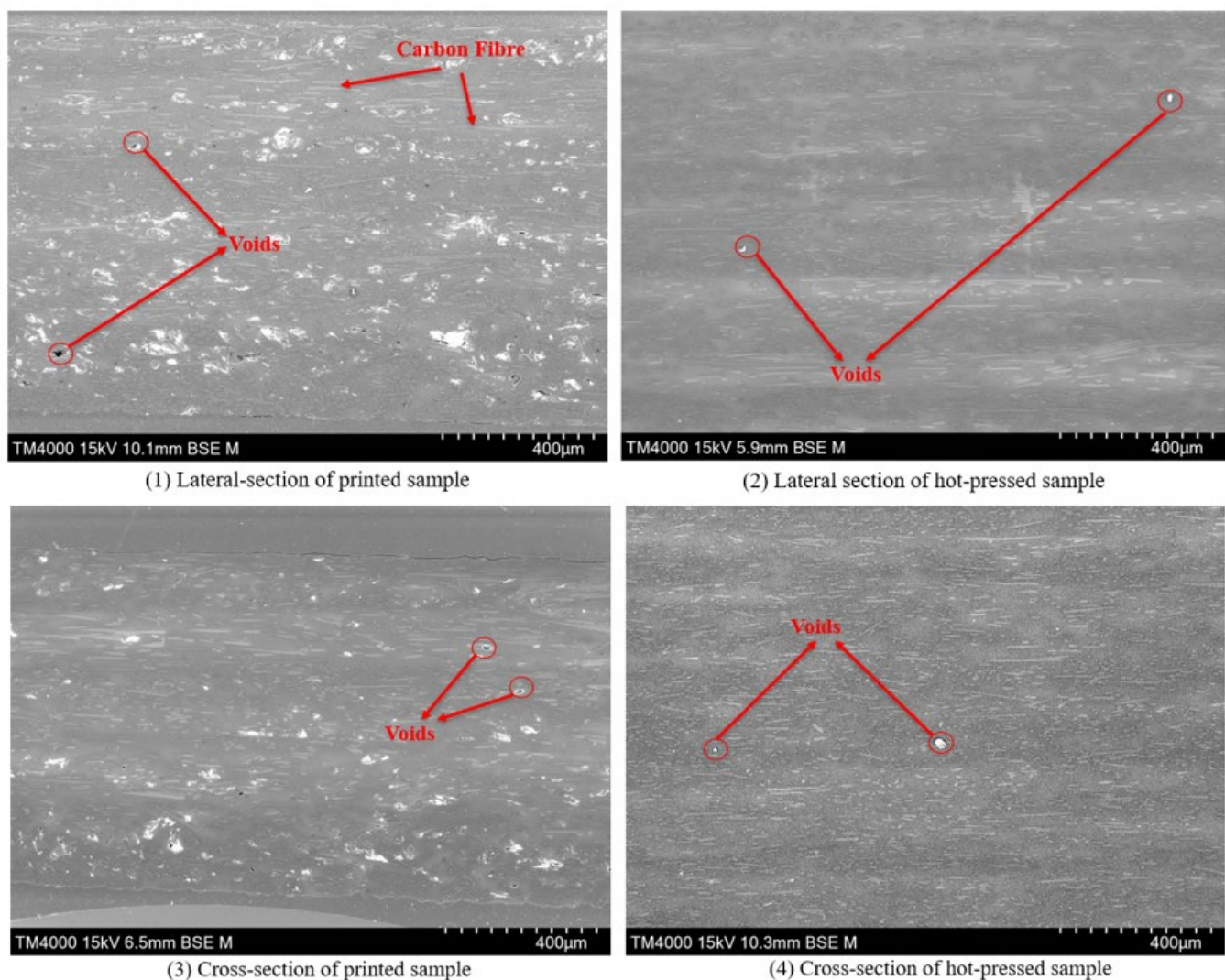
Hot-pressed specimens

2.2 Characterise the void content and fibre orientation by SEM

As shown in Figure 3 (1&2), the initial scanning electron microscope (SEM) image reveals a side view of a 3D-printed composite before and after hot press, highlighting the alignment of carbon fibres in relation to the printing direction. The composite without hot press post-processing shows numerous voids (highlighted with red circles) and less consistent fibre alignment. Following the hot press, the composite shows significantly fewer voids, indicating a more compact composite with potentially superior quality. This suggests that the hot-pressing technique may reduce the void presence and promote a more even fibre distribution.

In Figure 3 (3), the cross-sectional view of the printed composite again reveals obvious voids, marked to indicate potential weak spots in fibre-matrix adhesion. Conversely, the cross-section of a hot-pressed specimen in Figure 3 (4) displays far few voids, suggesting a more homogeneous and tightly consolidated composite. This could ultimately enhance the mechanical properties due to a more effective fibre-matrix bonding.

Figure 3: Microstructural images of 3D printed sCF/PEEK with and without hot press



These images illustrate the impact of the manufacturing process on the internal structure of CF/PEEK composite materials. The hot-pressed samples reveals a more uniform fibre distribution and reduced void content, characteristics that may correspond to enhanced mechanical performance. Fewer voids generally result in improved load transfer between the matrix and fibres. Given that voids can be considered a type of manufacturing defect, their reduction is crucial, as they have the potential to impair the creep and fatigue performance of the sCF/PEEK composite seal in real service conditions. Therefore, minimising voids is imperative for ensuring optimal performance and durability.

3. Thermal-mechanical analysis by DMA

3.1 Thermal-mechanical behaviour with temperature ramp

The Dynamic Mechanical Analysis (DMA) analysis of sCF/PEEK composites was conducted under specific conditions as outlined in the Table 2, utilising a double cantilever testing method

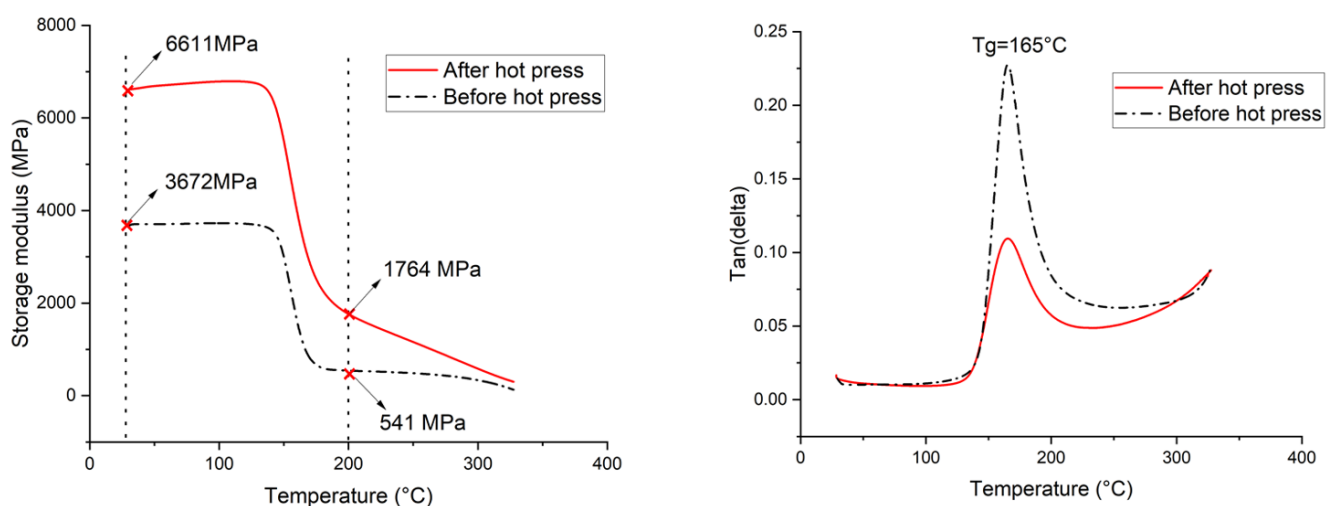
suitable for measuring the flexural properties of the material. The temperature range for the analysis was set from 30 °C to 330 °C, covering the ambient temperature up to the point near the melting temperature of PEEK. A constant frequency of 1 Hz was selected to simulate standard testing conditions, with the amplitude fixed at 30 μm to ensure measurement sensitivity. These parameters were crucial to ensuring the reliability and consistency of the DMA test results for the sCF/PEEK composite materials.

The DMA curves, presented in Figure 4 distinctly illustrate the thermal-mechanical behaviour of sCF/PEEK composites before and after the hot-pressing process. Notably, the hot-pressed specimen presents enhanced stiffness at both 30 °C and 200 °C, as evidenced by the higher storage modulus values of 6611 MPa and 1764 MPa, respectively. This increase in stiffness indicates a significant improvement in the load-bearing capacity of the composite material. Additionally, the glass transition temperature (T_g) of the CF/PEEK composite with 10% CF is identified at 165 °C. This critical point marks the material transits from a hard and relatively brittle state into a more ductile and malleable form. The T_g is an essential thermal property for sCF/PEEK composites, defining the temperature range within which the material can be effectively used in applications requiring thermal resistance.

Table 2: DMA testing parameters for sCF/PEEK composite evaluation

Parameters	Value
Mode	Double cantilever
Temperature range	30-330°C
Frequency	1 Hz
Amplitude	30 μm

Figure 4 DMA curves illustrating thermal-mechanical behaviour of sCF/PEEK composites

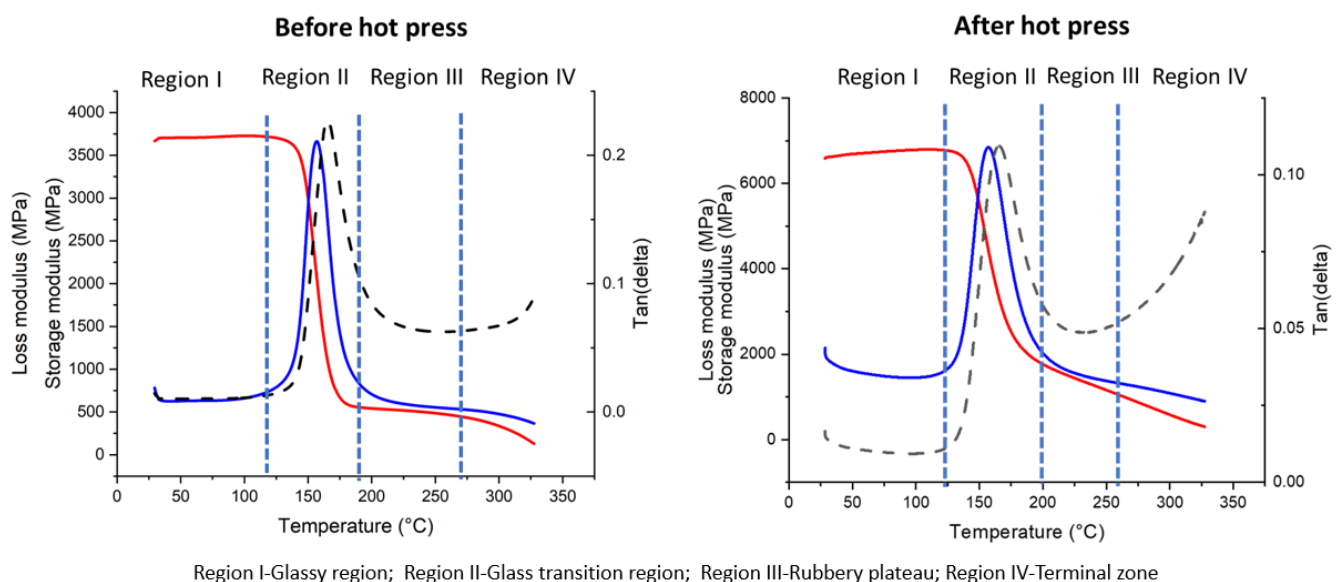


The DMA graphs shown in Figure 5 provide a comprehensive view of the sCF/PEEK composite's thermal-mechanical transition phases, both before and after hot pressing. Each graph outlines four distinct regions characterised by their mechanical behaviour in response to increasing temperature.

- Region I (Glassy State): this is where the material exhibits its maximum stiffness.
- Region II (Glass Transition Region): this indicates a steep decline in the storage modulus, corresponding to the softening of the composite as it approaches and passes the glass transition temperature.
- Region III (Rubbery Plateau): this shows a levelling-off of the storage modulus, indicating a temporary stability in the material's elasticity.
- Region IV (Terminal Zone): this is where the material experiences a drastic reduction in stiffness, leading to flow.

Comparing the two graphs, it is evident that the hot-pressed samples maintain higher stiffness within the first three regions. This suggests an improvement in thermal stability and mechanical performance due to the hot-pressing process. The enhanced stiffness in these crucial transition phases implies increased resistance to deformation and improved overall material integrity, supporting the efficacy of the hot-pressing technique in enhancing the thermal-mechanical properties of the sCF/PEEK composite.

Figure 5: Thermal-mechanical transition phases of sCF/PEEK composites pre and post hot pressing



3.2 Creep-recovery behaviour

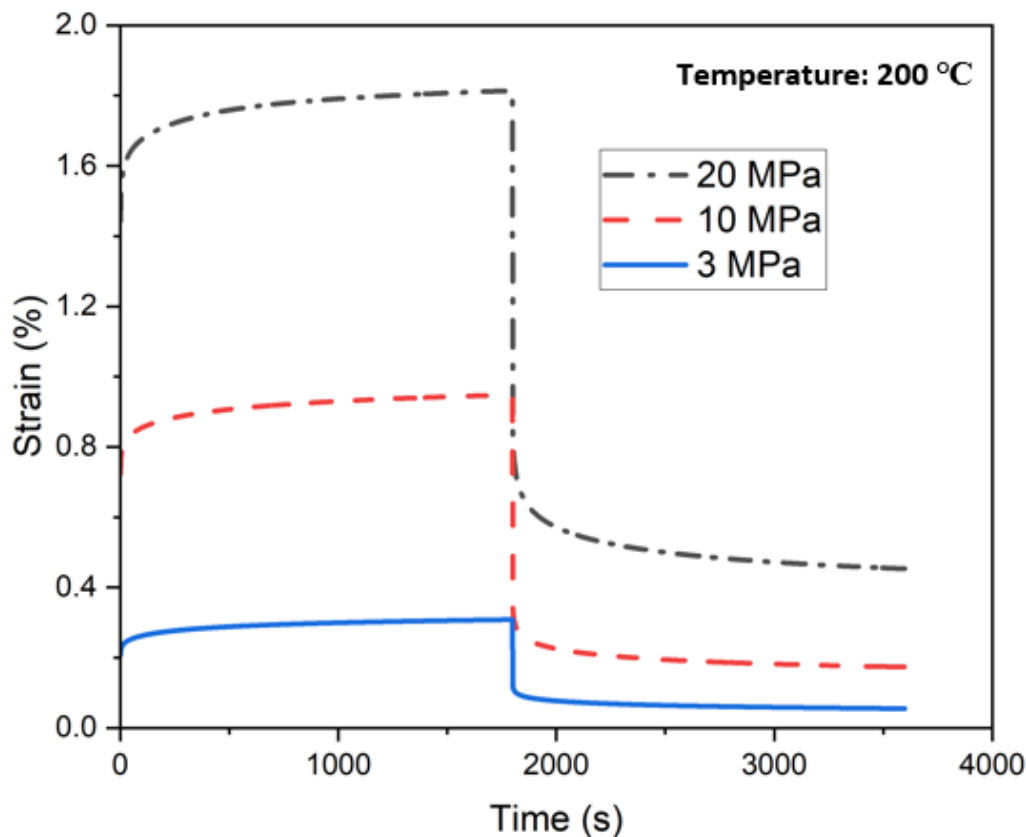
This section of the report investigates how a combination of PEEK and carbon fibres withstand heat and pressure over time, specifically focusing on the material's tendency to slowly deform or stretch, a phenomenon known as creep. This understanding is crucial for ensuring the suitability of these materials in applications where long-term durability is essential.

Figure 6 depicts the creep-recovery behaviour of the material subjected to different stress levels (20 MPa, 10 MPa, and 3 MPa) at a constant temperature of 200 °C. The graph illustrates several key observations:

- **High Strain at High Stress:** Under high stress (20 MPa or 200 bar), the material experiences substantial creep. The black dashed line indicates a steep increase to its peak, reaching a strain of 1.81% after approximately 30 minutes, followed by a plateau.
- **Good Recovery at Low Stress:** When the stress is lower, at 3 MPa (30 bar), the material exhibits a very good recovery from the strain. The blue solid line shows that considerably lower strain, almost returning to its original position, with a minimal permanent set of about 0.06%. This suggests excellent resilience of the material at this stress level.
- **Presence of Recoverable (Elastic) and Non-recoverable (Plastic) Strain:** At the high-stress level of 10 MPa and 20 MPa, even after a recovery period of 30 minutes, there is a noticeable amount of non-recoverable strain. Specifically, the black dashed line for 20 MPa shows that, despite some recovery, approximately 0.45% of the strain does not recover, indicating a plastic deformation component in the material's response.

The result from Figure 6 clearly demonstrates the effect of stress level on both the immediate strain response of the material (creep) and its ability to recover (elasticity). As stress increases, the material undergoes more permanent deformation. At lower stress levels, the material behaves more elastically with a greater ability to recover its original shape after the load is removed.

Figure 6: Creep-recovery behaviour of sCF/PEEK composite materials under different stress levels: 20 MPa, 10 MPa, and 3 MPa.



3.3 Comparative creep response over short and prolonged durations

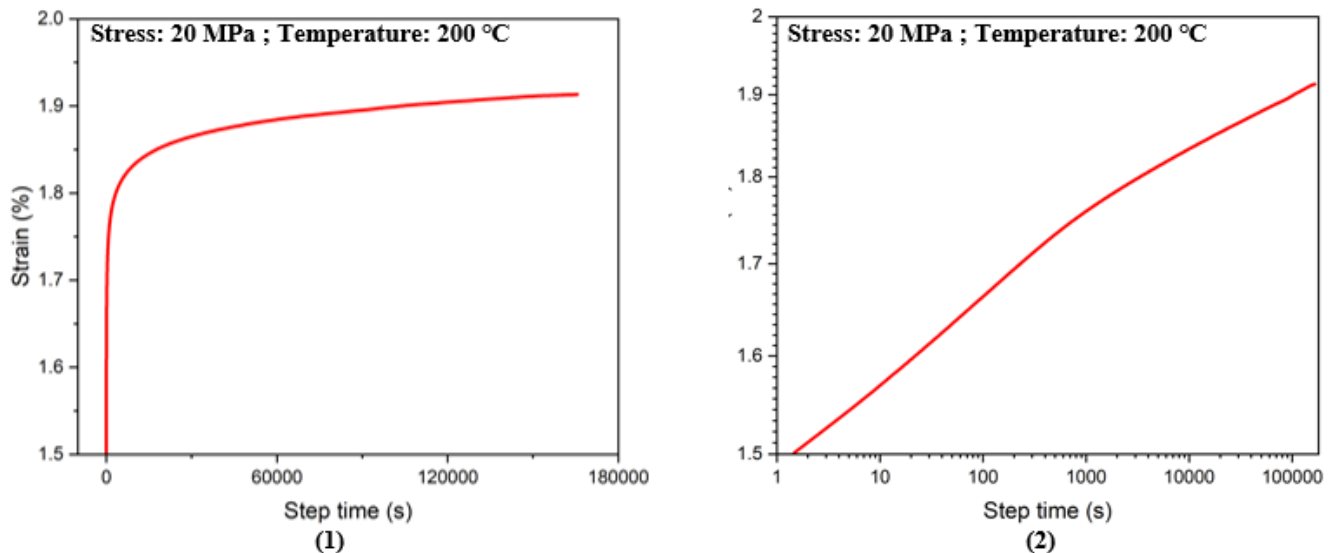
Figure 7 provides a visual comparison of the long-term creep behaviour of sCF/PEEK composites under a constant stress of 20 MPa at a high temperature of 200°C. The left chart displays time in a regular way, while the right chart stretches the time axis to show longer-term changes. This analysis gives a more detailed discussion of the material's mechanical stability and durability under prolonged stress and thermal conditions.

The curve on the left graph shows a rapid increase in strain within the initial 30 minutes, reaching 1.78%, followed by a gradual ascend to 1.91% over the course of 46 hours. This indicates a stable long-term creep behaviour, where after the initial strain, the material exhibits a low and decelerating rate of deformation, suggestive of the onset of a slow secondary creep phase.

On the right graph, plotted on a logarithmic timescale, a detailed view of the creep behaviour of the composite material under the same conditions. The near-linear trend in the log-log plot corresponds to the secondary phase of the creep, confirming a steady-state creep phase. The gentle slope of this phase reflects a low rate of creep, indicative of the material's positive long-term performance and its ability to sustain load without significant deformation over time.

In both graphs, the creep curves plateau towards the end of the observation period, a highlighting the composite's potential for structural applications where long-term mechanical stability under thermal and load stress is crucial.

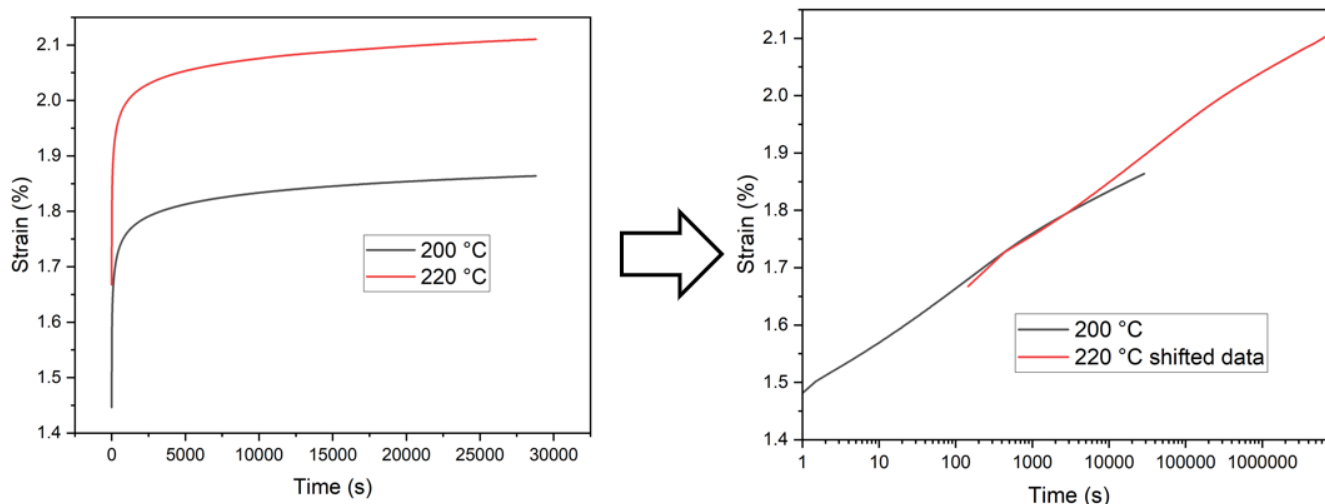
Figure 7: Creep response of sCF/PEEK to constant stress at elevated temperature.



3.4 Insights into ultra-long-term creep behaviour

Figure 8 displays two graphs providing a dual-analysis of sCF/PEEK composite's creep behaviour under a constant load of 20 MPa within a high-temperature regime. The left graph shows that sCF/PEEK exhibits stable creep behaviour up to 220°C, with the strain increment slowing down as it approaches 2.1%. This stability is crucial for applications that involve sustained heat exposure, indicating that the material maintains its mechanical integrity under prolonged high-temperature conditions. The right graph employs time-temperature superposition principles to predict long-term behaviour, suggesting that the strain could reach around 2.1% at 200°C over an extended period of 115 days. This predictive modelling is essential for designing components that require long-term reliability at elevated temperatures and under continuous mechanical stress.

Figure 8: Application of time-temperature superposition principle for long-term creep prediction in sCF/PEEK composites



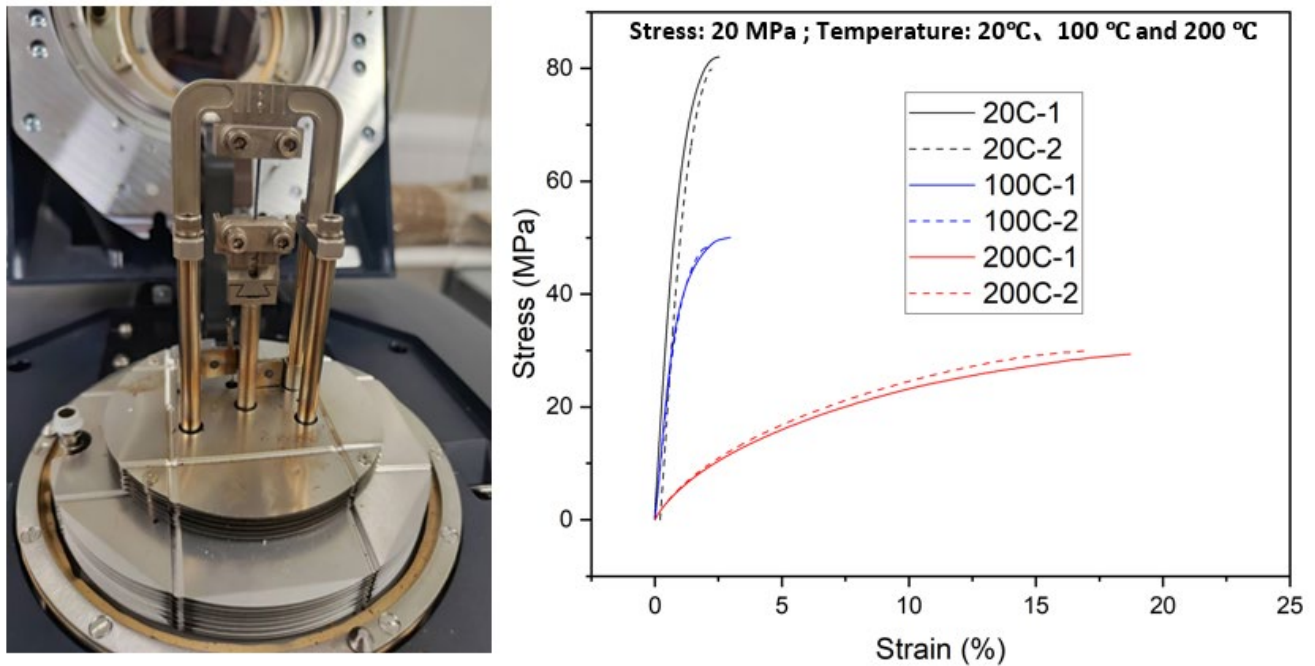
4. Tensile properties of sCF/PEEK composite at elevated temperatures

In preparation for the subsequent finite element analysis of the seal under real service conditions involving heat and pressure, it is essential to characterise the tensile properties of the printed sCF/PEEK. These properties typically serve as input parameters for finite element analysis (FEA). Given that the maximum load capacity of the available DMA is 18 N, a single printed sCF/PEEK stripe is tested to obtain the thermal-mechanical response of the material at different temperatures. The tested results (i.e. stress-strain curves) will be extracted and imported into a FEA model for thermal-mechanical analysis to aid in optimising the seal design.

The tensile behaviour of sCF/PEEK as depicted in the Figure 9 shows its typical thermoplastic characteristics, displaying an increase in ductility and a decrease in stiffness and strength with rising temperatures. Notably, even at the temperature of 200°C, sCF/PEEK maintains a significant level of strength and ductility. This performance is indicative of its suitability for high-temperature applications, reaffirming its status as a high-performance material.

The stress-strain curves at 20°C, 100°C, and 200°C provide a clear representation of the material's response to thermal stress, confirming its robustness and reliability in various operating conditions. This data is particularly valuable for applications where sCF/PEEK components are subjected to high thermal loads, as it demonstrates the material's ability to retain critical mechanical properties under such demanding circumstances.

Figure 9: Temperature-dependent tensile behaviour of CF/PEEK printed paths

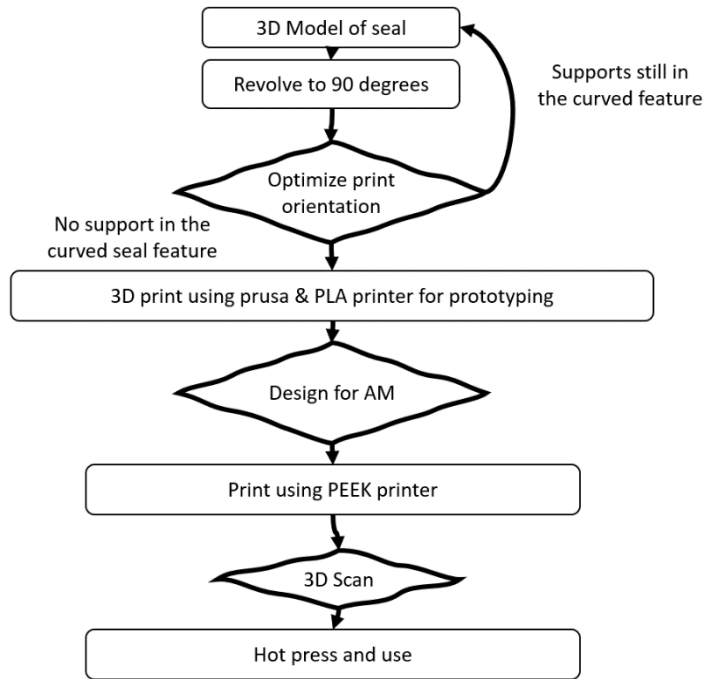


5. 3D printing of full-size sCF/PEEK composite seal

5.1 Geometrical design and printing strategy

The flowchart shown in the Figure 10 describes a streamlined 3D printing process for a seal component, highlighting efficiency in material use and manufacturing time. The process begins by adjusting the 3D model of the seal to a specific orientation, strategically minimising the need for support structures, particularly in the curved features—an essential aspect of the design. This optimisation of print orientation directly aligns with the objective of reducing material consumption and eliminating surface imperfections that may arise during support structure removal. This systematic approach not only enhances the overall efficiency of the 3D printing process but also contributes to the production of high-quality seal components with minimised material waste.

Figure 10: Optimisation of support structures in 3D printing of a seal component



The prototyping phase employs a Prusa 3D printer with PLA material, printing only a quarter of the seal design. This critical step assesses print accuracy and ensures minimal support requirements. The objective is not only to verify the print quality but also to test the structural integrity with minimal supports. This practice is observed for both PLA and 10% sCF/PEEK composite printing.

Within the Design for Additive Manufacturing (DfAM) strategy, a challenge arises with the curved portion under the seal where the initial 30% infill density for supports is inadequate. Two potential solutions were considered: either increase the support infill density to 60% or slightly modify the design to make it self-supporting. This decision is pivotal in maintaining a balance between structural support and efficient material use.

After resolving the design constraints, the component is printed using a PEEK printer. Subsequently, a 3D scanning process verifies dimensional accuracy, confirming the success of the minimal support strategy. The final step involves hot pressing of the printed part, preparing it for its intended application. This process exemplifies the intricate balance between design, material efficiency, and functional integrity within the realm of additive manufacturing. The careful consideration of support structures and material choices highlights the holistic approach taken to ensure the successful production of the seal component.

5.2 Optimising seal design from 2D sketch to 3D prototype

The initial phase of 3D printing begins with the creation of a 2D sketch in SolidWorks, serving as the basis for generating the corresponding 3D model. This model is the foundation for the subsequent printing stages.

To conserve materials and increase efficiency, the first physical iteration of the seal is printed using PLA material, which is known for its ease of printing and cost-effectiveness. The printing is conducted on a Prusa printer. Before committing to a full-scale print, a section of the seal is enlarged and printed to study the optimal placement direction of the print on the print bed. This step is crucial as it determines the amount of material used and affects the surface finish of the printed object. Slicer software aids in this process, enabling a detailed examination of the material usage and surface cleanliness. This analysis guides the fine-tune the printing parameters for best possible outcome. The full-scale PLA prototype seal shown in Figure 11 is a complete ring, illustrating the initial prototype made with economical PLA material on a Prusa printer. This stage allows for the study of material behaviour and print orientation without the high costs associated with PEEK filament.

In the pursuit of a more durable and heat-resistant final product, a quarter-scale version of the seal is printed using a high-temperature printer and PEEK filament. This exploratory phase is essential to assess the printing effects of the chosen material and equipment. Once satisfied with the results of the quarter-scale print, a full-scale 3D printed seal is produced from the 10% sCF/PEEK composite material. The full-scale seal was printed at nozzle temperature of 400 °C, bed temperature of 140 °C, and print speed of 240 mm/min. The final product represents the culmination of careful planning, material consideration, and parameter optimisation, all aimed at achieving a high-quality, high-pressure seal.

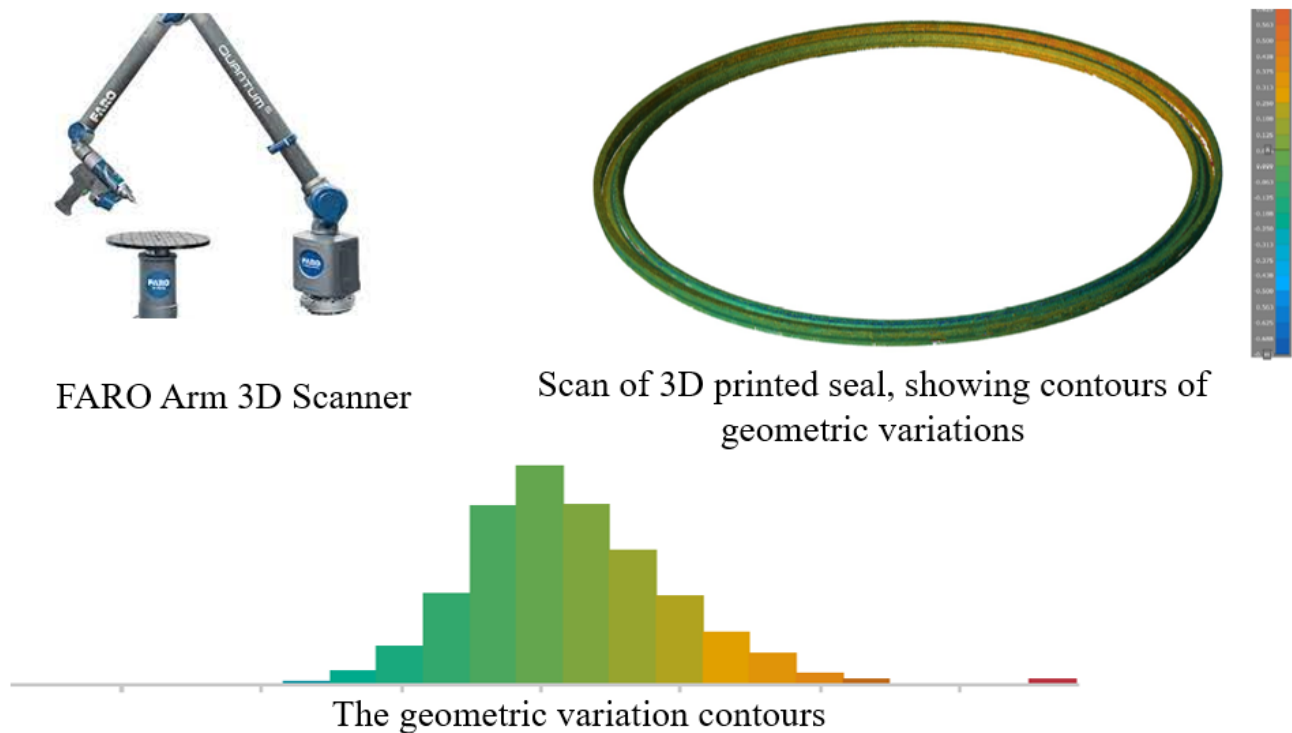
5.3 Verifying dimensional accuracy with 3D FARO Arm scanning

To validate and verify the print geometry, a comprehensive examination of the dimensional precision of the 3D printed seal was conducted using a FARO Arm 3D Scanner. The scanner, equipped with a high resolution of 0.2 µm, features a complex multi-jointed arm and a scanning probe, enabling a thorough scan of the seal. The scanner captures a dense cloud of data points that precisely represent the seal's geometry.

The image on the right side of Figure 12 shows the scan output of the 3D printed seal, revealing the contours that map geometric variations against the expected dimensions. The actual 3D model was given to the FARO arm for benchmarking, allowing for a comparison of the geometric variations. The scanned point cloud was superposed to the 3D model and the geometric variations were quantified. A histogram below the scanner image quantifies the scan's accuracy, with a significant count of 47,689,15 data points, resulting in a performance accuracy of 88.7%. However, it was observed that the printed seal was oversized from the 3D model by 0.063 mm. Figure 12 highlights the similarity between the printed seal's dimensions

to the digital 3D model, demonstrating the effectiveness of the printing process and the scanner's capability to ensure that the seal adheres to rigorous quality and tolerance specifications.

Figure 12 Precision analysis of a 3D Printed seal via FARO arm



Conclusions /Recommendation:

The printing, testing and characterisation results confirmed good thermo-mechanical properties of sCF/PEEK composites with 10%wt short carbon fibres for potential use as polymer seals under a service temperature of 200°C and a pressure of 200 barg. Optimal printing parameters and post-processing window have been identified for sCF/PEEK composites and a full-size composite seal has been successfully demonstrated with good geometrical accuracy. For real application of the sCF/PEEK seal, there may be tribological requirement, such as friction coefficient, which needs to be addressed. This will be investigated after the test of the present prototype, and potential blending of other lubricant additives could be explored to further enhance the tribological performance of the 3D printed sCF/PEEK composite seal.

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