OPTIMISING GRIP DESIGNS FOR THE TENSILE TESTING OF FIBRE-REINFORCED POLYMER COMPOSITES AT CRYOGENIC TEMPERATURES

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Abstract
Advanced materials underpin the development of storage and distribution technologies for liquid hydrogen a fuel on board an aircraft. Though it is necessary to have in place fit-for-purpose testing and characterisation methods that can accurately measure the required properties at the associated conditions. These need to include mechanical, thermal as well as transport properties of the associated materials. When it comes to mechanical testing, the method of the load introduction to the specimen is overly critical. In this work, a novel gripping system was developed, addressing issues that have been observed with traditional wedge action gripping, thus allowing for tensile testing of fibrous composite materials at cryogenic temperatures. Overall, the grips have been performing well when it comes to ease of use and function, however they were found to be susceptible to local grip-face damage when testing harder materials.

1. Introduction

The aviation industry's transition to using hydrogen as a sustainable fuel is undoubtedly pushing the boundaries when it comes to materials that can withstand the cryogenic temperatures that liquid hydrogen (LH2) must be sustained at. To develop such materials, it is imperative that testing and characterisation methods are fit-for-purpose and accurately measure the required properties at the associated environmental conditions.

For decades, there has been a lot of research generated around the nuances of cryogenic mechanical testing of fibre-reinforced composite materials, for the space/aerospace industries, as well as for characterising superconductive materials for particle physics. The typical test setups consist of a load frame (commonly a universal test machine) and the device that is responsible for cooling the test space down to the desired cryogenic temperature. Regarding the latter, a standard environmental test chamber can be used in conjunction with liquid nitrogen evaporative cooling to achieve temperatures as low as ~100K. For reaching lower temperatures, a cryostat operating with liquid nitrogen or helium is typically required. When it comes to mechanical testing however, the method of the load introduction to the specimen is also critical. Ensuring that the specimen is well aligned with regards to the load direction and that the load is exerted fully on the specimen without any relative motion or compliance throughout the fixture is of outmost importance. Gripping the specimen is especially challenging at cryogenic temperatures, as the fixtures and jigs typically used for room temperature testing, are not suitable for low temperature use. Therefore, there are numerous issues that can be observed when testing with non-cryogenic rated equipment at cryogenic temperatures. The most common, would be the slipping of specimens in the grips because of trapped moisture that has iced, as well as loosening of any threaded and load bearing connections because of thermal contraction of the different components that are commonly made from dissimilar materials.

In this work, a novel gripping system has been developed, allowing for tensile testing of fibrous composite materials at cryogenic temperatures. Two distinctive design approaches have been proposed, optimising the test procedure, and accommodating for coupon geometries prescribed by international standards (i.e., respecting the minimum representative volume element of the material encompassed by the specimen geometry). The performance validation was done in two tests setups. The first consists of an environmental chamber where gaseous nitrogen is used to achieve a target temperature of 110K, while the second one is in a liquid flow cryostat where the test space is submerged in liquid nitrogen at 77K.

2. Design Considerations

Unlike metals, fibre-reinforced composite materials can be more challenging to grip since coupons cannot be miniaturised due to the need to test a representative volume of material. In addition, it is not possible to machine more convenient tensile geometries that are used for metals e.g., button-headed, or threaded specimens. Typically wedge-action grips are used for testing of composite materials however these have been proven problematic when testing at low temperatures, as highlighted in a previous NPL report [1]. Common issues include the formation of ice from trapped moisture between the grip faces and the material, leading to slippage of the specimen or locking of the wedges in their housing, as well as practical challenges with tightening and loosening the grips post-testing, extending the test duration, down-time between tests and cryogen consumption. To address these issues, several modifications to wedge-action gripping were proposed with the main priorities being to:

- Reduce points of contact and potential for wear,
- Minimise the number of and reduce the friction between moving parts,
- Minimise the size and thermal mass of the grips where possible,
- Improve serviceability between test runs,
- Achieve an acceptable level of test specimen alignment.

A key factor was to design the grips to be modular thereby enabling the use of most grip components for both tension and compression by interchanging only the grip-faces and reversing the design concept for load introduction between tension and compression.

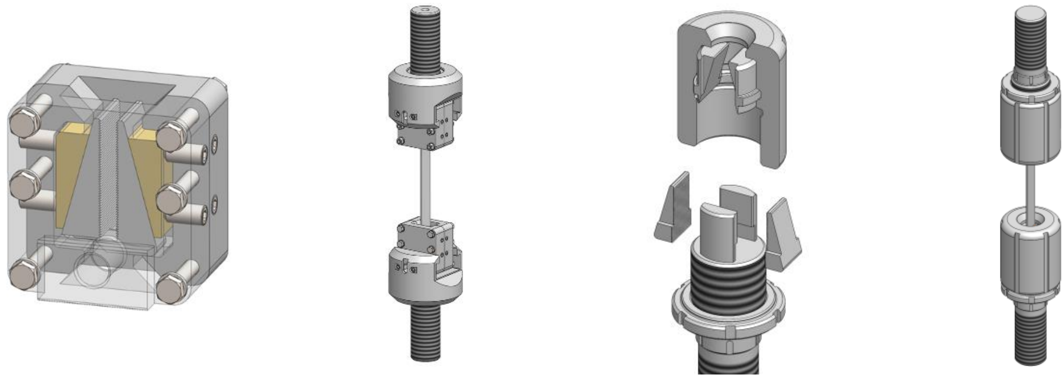
2.1. Cubic design variant

The cubic variant was designed to offer better gripping at lower temperatures using four preload screws on the side. A key advantage of this design is the potential to achieve a faster turnaround time of specimens. By having the grip holders permanently attached on the pull-rod an additional set of grip faces pre-attached to the specimen can be used so that the tested specimen can be slid out and the new specimen slid in. This is especially helpful for batch testing, as having a duplicate set of grips means one can expedite the cooldown time by having one grip set cooling down in the test chamber, while another set is held at low temperature in a separate dewar. Unfortunately, the cubic design ends up being comparable to traditional grips when it comes to weight, although more compact. In addition, the high number of moving parts and fasteners is concerning, as their behaviour at cryogenic temperatures is hard to predict. For this reason, manufacturing and validation of the cubic variant was de-prioritised.

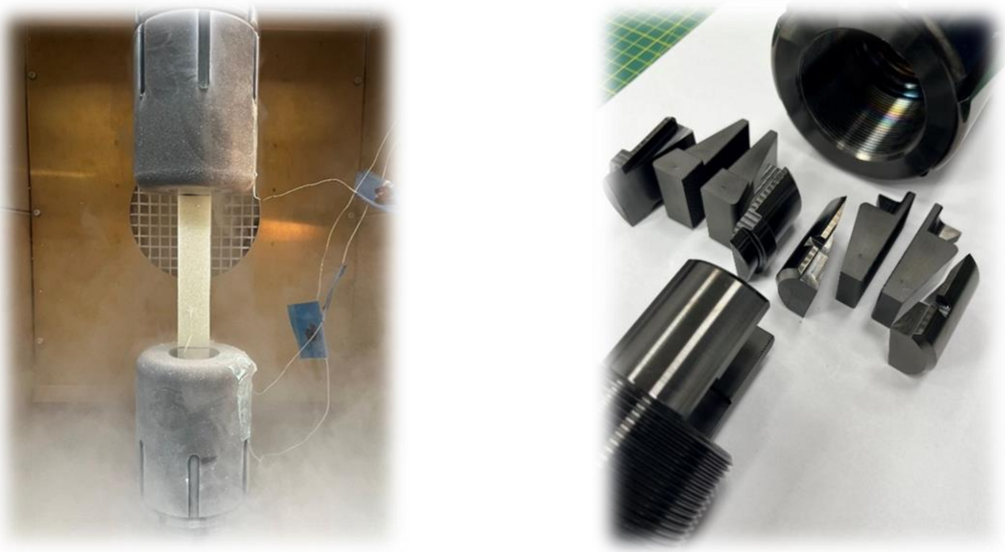
2.2. Cylindrical design variant

This variant comes with several strengths over the cubic one, with the only major weakness over the latter being the absence of a potentially faster specimen change. The cylindrical grip design is a smaller and lighter concept, with improved ergonomics for tightening/loosening and a self-aligning function due to its axisymmetric geometry that ensures that the specimen is always centred about the loading line in the through-thickness direction. Depending on the thread size on the main body, the load rating can be 100 or 250kN. Like the cubic variant, the change from tension to compression configuration only requires swapping four components in each grip and a 3D printed tool makes the assembly on the grip easy enough to perform within an environmental chamber.

Materials that were considered for the construction of the grips focussed on metals with known cryogenic performance including AISI 316 and 316LN stainless steels, Nitronic 40 and 50 manganese-strengthened alloys, and Inconel 718 which is a nickel-chromium superalloy. These materials have suitable properties at cryogenic temperatures with Inconel 718 considered favourably by the cryogenics community. As a key priority of the grip design was to minimise the friction between components, different low friction coating options were investigated alongside resistance to wear and low temperatures. Inconel 718 was selected as it offers the best compatibility with the selected coating which is a graphitic carbon-based coating for highly loaded wear components.



(a) (b)



(c) (d)

Figure 1. (a) Design of cubic and (b) cylindrical variants; (c) Grips installed within an environmental chamber and (d) subcomponents of the grip laid out before assembly. Both tension (left) and compression (right) configurations are visible.

3. Preliminary Validation Testing

For the preliminary validation of the performance of the cryogenic grips, a series of tension tests on a woven glass fibre-reinforced plastic (GFRP) material were undertaken at 108K (-165ºC) using an environmental chamber cooled via evaporation of liquid nitrogen. The grips were mounted on steel pull-rods and then the load-train was preloaded to 60 kN, using a tool steel bar (250 mm×25 mm×5 mm) to lock the loading collars, which prevent the loading-train from becoming loose under load.

Five tensile tests were undertaken on Tufnol® 10G/40 specimens. The first test was undertaken on a specimen with reinforcement orientated at ±45º to the longitudinal axis of the specimen, whilst the remaining four specimens were machined with reinforcement at 0°/90º to the longitudinal axis. All five specimens were straight rectangular coupons (dimensions of 250 mm×25 mm×5 mm) and were gripped without additional end-tabs. Three tests were performed at room temperature (RT) and two tests at -165ºC.

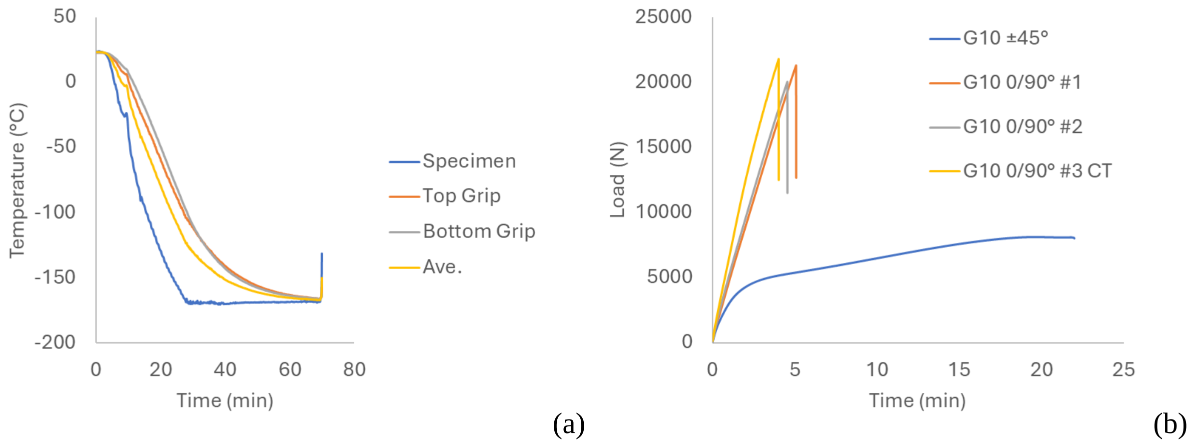


Figure 2. (a) Temperature profiling graphs for the grips and specimen temperatures and (b) the load vs displacement curves from the validation tests of the grips

Each coupon was marked adjacent to the end of the grips to monitor for slipping during loading. These marks also helped in ensuring that specimens were mounted symmetrically inside the grips, maximising the contact patch with the grip-faces. Three Type-T thermocouples were used to measure temperature during the test, one mounted on each grip and one in direct contact with the specimen. The cooldown time to -165ºC was found to be approximately one hour at which point the grips and specimens had reached the target temperature (Figure 2(a)).

Specimens were loaded in displacement control at 2 mm/min until failure. The measured strengths were as expected and compared well with previously measured data for this material. Failure was observed between or just outside the grips; these were acceptable failure modes for rectangular, non-end tabbed specimens.

However, it was found that following use of the steel tool bar for preloading the load train slippage occurred in a few cases. Upon thoroughly inspecting the grip-faces, a discoloration between the teeth serrations was observed, indicating that the coating might have been removed. By employing microscopical analysis it was evident that the teeth had been heavily damaged along the length of the face, with more damage accumulating on the bottom grip faces (Figure 3)

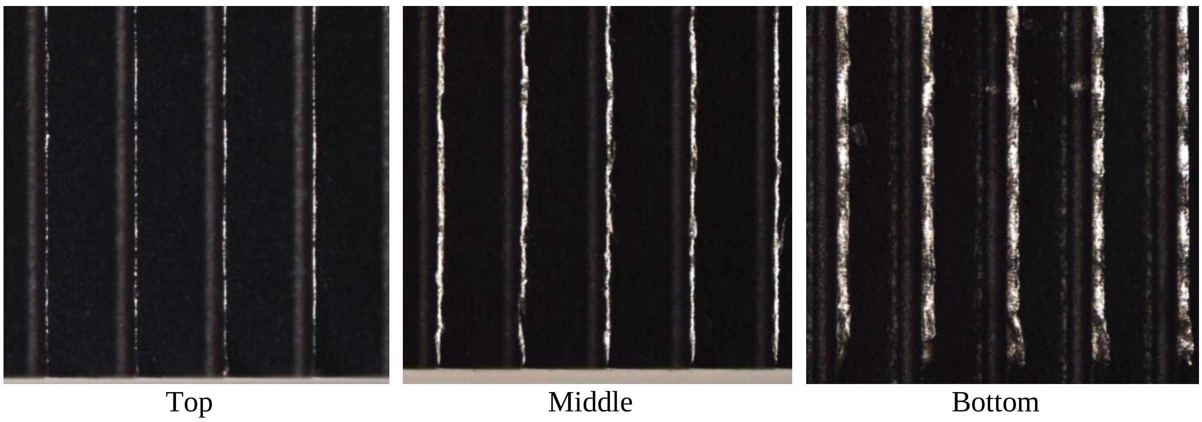


Figure 3. The damage accumulation at the grip-face's serrations

4. Conclusions

Overall, the grips have been performing well when it comes to ease of use and function compared to the traditional options. Most of the design targets set at the beginning were achieved. However, damage on the faces was found went loading the hard metallic bar and possibly when loading the glass fibre thermoset material uses in the initial validation. Compared to these tests on materials with resin rich surface layers did not present any problems with gripping and slippage. A revised variant of the grip faces has been designed addressing the issues of this initial validation.

Acknowledgments

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